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Assessment of active tectonics based on GIS and statistical model: a case study of upper Alaknanda catchment (western Himalaya, India)

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Research Article

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Tectonics.

ABSTRACT

Due to continuing deformation of the Earth's crust, degradation and aggradation processes, the upper part of the Alaknanda basin lying in the Garhwal Himalaya, India, is considered to be tectonically active. Active tectonics in this region made a considerable impact on the drainage system and the topographic expression. Using a digital elevation model (DEM) with a resolution of 30 m * 30 m and based on eight geomorphic indices [hypsometric integral (HI), drainage texture (Dt), asymmetry factor (AF), ruggedness number (Rd), valley floor width/height ratio (Vfw), lemniscate coefficient (k) and basin shape index (Bs)], we evaluated active tectonics. Application of Index of Active Tectonic (IAT) and clustering model disclose the sub-watersheds in the southern part covering watersheds 16, 17 and near Badrinath sub-watershed 4, 9 and 8 are tectonically highly active (Class-1, 1.500-1.781). Cluster analysis defines two prominent clusters of sub-watersheds: 2, 4, 14 and 15 in cluster 1; and 6, 7, 8 and 18 in cluster 2. Cluster 1 includes sub-watersheds 2, 4, 14, 15, and 16 and very high, high, and moderate IAT magnitudes. Sub-watersheds 6, 7, 8, and 18 are included in Cluster 2, which has very high and high tectonic activity.

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1. Introduction

In the Himalayan landscape, tectonics is the most important factor forming the present-day topography, which could have an additional role of erosion/denudation (Harkins et al., 2005; Andermann and Gloaguen, 2009; Perez-Pena et al., 2009). In the upper Alaknanda basin, crystallines of the Lesser and the Higher Himalaya exist at south and north, respectively (e.g., Metcalfe, 1993). This section of Alakanada catchment is composed of phyllites, slates and other metamorphic rocks (e.g., Celerier et al., 2009). It is possible to retrieve geomorphic evidence of active tectonics from topo-sheets, aerial photos, satellite

information and through morpho-tectonic parameters (Horton 1945; Keller and Pinter, 2002). Morphometric investigation presents quantitatively the geometry of basins to comprehend initial slant or differences in the drainage basin's topography, geomorphic history, structural control, recent tectonics as well as rock hardness (Strahler, 1964). The Alaknanda River Basin is a tectonically active area with a history of destructive landslides and earthquakes. Landslides and/or seismicity has been reported frequently from locations such as Chamoli, Birahi, Pipalkoti and Rudraprayag. This area is traversed by several significant tectonically active thrusts e.g., the Main

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Central Thrust (MCT)- I, also known as the Vaikrita Thrust; MCT II, which is also known as the Munsiri Thrust; MCT III, also known as the Ramgarh Thrust, and the North Almora Thrust. (Shukla et al., 2014). Therefore morphological study in this area is an important exercise.

Even though several geomorphologic works have been undertaken on the Alaknanda river and the

surroundings (review in Table 1), a morphometric study such as the preset work remained a due. This work involves hierarchical clustering of drainages and estimates Index of Active Tectonics (IAT) for different watersheds of the area taken into consideration. These are performed in order to understand the different degrees of tectonic activeness within the considered area.

Table 1- Geomorphologic research done on Alaknanda river valley that partially or completely covers the study area of this work.

Sl no.	Reference	Aim	Key approach	Software used	Salient result
1	Bhattacharya and Jugran (1982)	To evaluate land subsidence as a landslide cause.	Ground truth has been used in conjunction with aerial photo interpretation.	-	The important factor in soil subsidence is seepage.
2	Barnard et al. (2001)	To comprehend the causes of landslides, both natural and anthropogenic.	Major landslides were mapped and samples from boulders on the surface were taken for cosmogenic radionuclide dating.	-	Human activity was responsible for ~ 2/3 of the landslides, primarily by removing toes of the slope at road cuttings.
3	Joshi and Kumar (2006)	To determine the relationship between historical occurrences of natural disasters and very heavy rainfall.	Daily rainfall data analysed statistically.	-	Frequent extreme rainfall is one of the main causes of landslides.
4	Sarkar et al. (2006)	To identify unstable slopes along the National Highway in the Alaknanda valley of Garhwal Himalaya.	Through photo documentation, probable slope failure zones were demarcated.	-	Mitigation of landslide-prone zones recommended.
5	Sati et al. (2007)	To attribute the development of the morphologic evidence along the North Almora Thrust.	Comprehending the Late Quaternary seismicity in the Alaknanda Valley near Srinagar from the morphologic evidence of Uttarak Fluvial terraces, entrenched stream channels, landslide-induced ponding, and active and stabilised landslide deposits.	-	NAT and its related lineaments are active in the Late Quaternary Period.
6	Sundriyal et al. (2007)	To carry out observations on lake deposits that have been impacted by landslides at the south-dipping, E-W and NW-SE trending North Almora Thrust.	Application of lithofacies, geochemistry and optically stimulated luminescence chronology.	-	Second and third order tributaries of the Alaknanda river were temporarily dammed due to activation of crumpled and unstable phyllite-dominated slopes. Seasonal deposition is indicated by sedimentary patterns of the succession under a transient lacustrine environment.
7	Kundalia et al. (2009)	To set rainfall thresholds for landslide initiation.	To determine the connection between rainfall amounts and the onset of a slope failure, an analytical approach is used.		Precipitation is the primary mechanism for slope failure.
8	Tyagi et al. (2009)	To locate areas of substantial surface uplift in the Alaknanda valley .	Application of steepness index.	ERDAS IMAGINE	Convex river profiles and high steepness index values identify areas that are experiencing quicker surface uplift. The zone of high uplift (incision) are the Main Central Thrust (MCT). South of the MCT, at Chamoli, Nand Prayag, and Karn Prayag.

Table 1- Continued.

9	Juyal et al. (2010)	To understand geographic and temporal variability in stages of aggradation/incision in response to variations in climate and seismicity over the Late Quaternary in the Alaknanda valley.	Fill and strath terraces were mapped using total station survey. Paleoenvironmental reconstruction employed the fill sequences' sediment texture, structures and vertical lithostratigraphy. Infrared stimulated luminescence applied on feldspar grains.	-	Debris flow terraces formed during the early part of pluvial Marine Isotopic Stage 3 are the earliest fluvial landforms retained in the south of the Main Central Thrust.
10	Onagh et al. (2012)	To produce a landslide susceptibility map .	Multiple linear regression analysis using slope, aspect, lithology, land cover, rainfall, distances from fault, river, and road .	ILWIS 3.31 Academic, Arc GIS 9.3, Global Mapper 13.0 and Excel	The study area has been divided into Low, Moderate, High, and Very High relative landslide susceptibility classifications.
11	Parmar and Purohit (2013)	Mapping landslide hazard zones in Alaknanda Valley.	Lithology, structure, slope morphometry, relative relief, landuse and land cover, and hydrological conditions considered.		Shear zones, faults, heavily weathered and saturated sediments with unfavourable discontinuities characterize very high hazard zones.
12	Sarkar et al. (2013)	To evaluate the level of risk in the probable landslide zones, an effort has been made to quantify landslide intensity.	By assessing the severity of the present and possible landslides, landslide hazard assessment has been done to define the hazard levels. Individual landslides were initially subjected to hazard assessments in order to determine the possible risk of the research region.	ArcGIS (<i>version not referred</i>)	Most of the time, rock fall, rock slide and debris flow fall into the very high and high risk categories. To categorize the zones into different classes, the cumulative impact of landslide hazards was considered.
13	Shukla et al. (2014)	To assess the tectonic activeness using morphometric techniques and GIS .	Stream length ratio, bifurcation ratio, transverse topographic symmetry factor, asymmetry factor, drainage density, stream frequency, drainage texture form factor, circulatory ratio, and elongation ratio, valley width to valley height ratio, roughness number, the relief ratio, and the hypsometric integral have been computed on 8 sub basins.	Arc GIS 9.3, ERDAS 9.1 and MicroDEM software	The regions surrounding the MCT II (Munsiari Thrust), MCT III (Ramgarh Thrust), and North Almora Thrust, which are located in sub-basins 2, 3 and sections of 5 are tectonically quite active.
14	Chaturvedi et al. (2014)	Regional landslide susceptibility analysis using remote sensing.	A landslide susceptibility map was created involving lithology, fault, lineament, geomorphology, drainage, slope, aspect, land-use and land cover, soil texture, and soil depth.	-	After field validation, this method for susceptibility evaluation is fairly reliable and exact.
15	Devrani and Singh (2014)	To recognize the significance of location and regional elements in the valley filling process in an active mountain belt.	Hypsometry along with the lithofacies analysis were conducted .	ArcGIS 9.3	Sedimentation in the Pipalkoti valley stretch was mostly caused due to local reasons.
16	Gupta and Tandon (2014)	To identify the probable zones of instability, which causes frequent rock fall.	For each of the 23 locations where <i>in-situ</i> rocks are found, a kinematic rockfall hazard analysis was done.	-	The HH is mostly of low- to moderate-hazard, whereas the LH has a moderate- to high-hazard potential.
17	Chaudhary et al. (2015)	To understand the genesis of (paleo)valley.	The geomorphic configuration, sedimentological character, sediment source, flow velocity, and Optical Stimulated Luminescence (OSL) chronology have all been emphasised by authors.	ArcGIS 9.1	In the Alaknanda valley in the Central Himalaya, paleovalleys formed in two ways- (1) by significant valley aggradation, and (2) locally through landslides and lake breaching. Most of the Alaknanda valley's paleovalleys evolved between 15 to 8 ka, during significant valley aggradation.

Table 1- Continued.

18	Sajwan and Sushil (2016)	To examine the factors causing slope instability and landslides.	510 landslides identified in field. Landslides categorised as per rigidity and rockmass movements and debris that constitute the slid mass.		The atmospheric precipitation that causes landslides and flash floods can both be anticipated if real-time rainfall data is available.
19	Sundriyal et al. (2015)	To determine the type and reasons for the valley damage in June 2013.	Geochemical analysis of flood sediments.	-	Sediments came from two main sources: landslides from the Higher and Lesser Himalaya, and moraines and alluvial fans from the Trans and Higher Himalaya.
20	Chandak et al. (2016)	To highlight the locations at risk for landslides.	Information Value Method was adopted for landslide hazard mapping.	Integrated Land and Water Information System (ILWIS)	The unexpected dislodgment of small to large jointed blocks leads to rock avalanches / debris flows.
21	Rana et al. (2016 a,b)	To analyze the morphometric indices for neotectonic implications in a fluvial erosion-dominated regime.	Application of morphometric indices e.g., stream order, stream number, bifurcation ratio, drainage density, stream frequency, texture ratio, circularity ratio, basin hypsometry and elevation-relief ratio to assess neotectonic imprint upon the study area.	ArcGIS 9.3	The findings point to two high deformation/uplift zones in the valley: the first is in the Inner LH, close to the Main Central Thrust (MCT), and the second is in the Outer LH, south of the Tones Thrust.
22	Rana et al. (2016 a,b)	To categorize the seismic and aseismic categories for the fluvio-lacustrine sediment successions and valley-fill terraces.	Analyzing Soft sediment deformation structure and brittle deformation features in fluvial and fluvio-lacustrine deposits in the Alaknanda Valley.	-	Most of the deformation structures produced presumably in aseismic manner, although the terrain lies in the active seismic zone of the Central Himalaya.
23	Maurya et al. (2017)	To map probable landslide hazard zones in Badrinath, Garhwal Himalaya.	Application of GIS techniques .	ArcGIS 9.2	54% of the area has a high chance of landslides. Risk is moderate in 25% of the area.
24	Mehta et al. (2017)	To assess the change in terrain as an effect of 2013's disaster in Alaknanda and Mandakini basin.	The use of a field survey and a GIS technique allowed us to evaluate the changes in the topography.		Debris of 0.3×10^6 and 0.72×10^6 m ³ deposited at Govindghat and Pulna Village, respectively.
25	Prasad and Pandey (2017)	To evaluate the effects of LULC change, climate change, and their effects on hydrological hazard.	Both qualitative and quantitative methodologies. Peoples' perception about the origin of hazards, trend, frequency, magnitude, and mitigation measures were analyzed using a Likert-scale.		Households in the away district headquarter (ADH) village are more vulnerable than those in the near district headquarter (NDH) village. Due to their traditional and local knowledge of the responses to climate change, the NDH households had more potential for adjustment within the framing system.
26	Khanduri (2018)	To analyse and evaluate the effects of landslides in the Chamoli district.	Understanding the historical landslip episodes and resulting damages by overlying several thematic layers.	Arc View 9.3	These slides have been made possible in large part by the combination of heavy rainfall and the weak shear strength of the rocks.
27	Sharma et al. (2018)	To assess the relative active tectonics of the Alaknanda basin of Garhwal Himalaya.	Active tectonics in 8 sub-basins of the Alaknanda basin using DEMs based on seismicity and 8 geomorphic indices.	ArcGIS 10.2	The middle portion of the tectonically most active sub-basin I exhibits movement rate of 41.1 mm y ⁻¹ in accordance with GNSS data.

Table 1- Continued.

28	Chauniyal (2018)	To locate the areas that have recently experienced uplift and displacement along the NAT in the Lesser Garhwal Himalaya's Lower Alaknanda Valley.	To identify places of neotectonic activity along N-S transverse faults.	-	Neotectonic movement along the NAT were noticeable between 15 to 9 ka and demonstrated recurring tectonics. Minor structures characteristic of Quaternary sediments identified.
29	Pant et al. (2020)	To identify the soil erosion and flood hazard zones using morphometric techniques.	Six primary morphotectonic parameters were examined on the upper Alaknanda basin and its sub-basins.	ArcGIS and SPSS (v.22)	Future development in the region should consider soil erosion, tectonic activity and potential for flash floods.
30	Khanduri and Rautela (2021)	To recognise the primary causes of slope instability.	In a GIS environment, the distribution of landslides has been connected with numerous parameters affecting their occurrence.	Arc Info 9.3	Landslides are most strongly influenced by proximity to the road and drainage system.
31	Meghanadh et al. (2021)	To generate a of landslide susceptibility map between Rudraprayag and Srinagar.	Multi-temporal interferometric synthetic aperture radar (MT-InSAR) and AHP approaches.	Sentinel Application Platform (SNAP) and Stanford Method of Persistent Scatterer (StaMPS)	There have been found 44.5% new landslide susceptible zones (LSZs).
32	Kumar et al. (2022)	To assess the vulnerable sites of slope instability along the NH-58 from Dhari Devi to Rudraprayag.	Kinematic analysis, geologic strength index, uniaxial compressive strength, rock quality designation, rock mass rating, slope mass rating and continuous slope mass rating are considered to assess slope instability.	-	Active landslides are caused by the Kaliasuar Fault in Uttarakhand. The Kaliasaur landslide is unstable due to pre-existing tectonic fracture and a plunging fold.
33	Joshi and Biswakarma (2023)	To compare models regarding landslide susceptibility mapping in the Alaknanda catchment area.	Application of Frequency Ratio Model, Analytic Hierarchical Process and Information Value Method to evaluate landslide susceptibility.	ArcGIS, Earth Resource Data Analysis System (ERDAS) IMAGINE	The Information Value Method yields the most precise results.
34	Clubb et al. (2023)	To evaluate sediment-storage mechanisms and identify the main influences on valley-floor morphology.	Application of the valley-floor width ratio throughout the Himalaya.	LSDTopoTools software	Long-term tectonically induced exhumation, not water discharge or bedrock erodibility, controls valley-floor width.
35	Raha et al. (this work)	To assess the active tectonics of the Upper Alaknanda Catchment.	Morphometric parameters such as Hypsometric integral (HI), Drainage texture (Dt), Asymmetry Factor (AF), Ruggedness number (Rd), Circularity Ratio (Rc), Valley Floor Width/Height Ratio (Vfw), Lemniscate Coefficient (k) and Basin Shape Index (Bs) are taken into consideration for the calculation of IAT. Cluster analysis has been performed on the sub-watersheds using dendrogram.	ArcGIS 10.3 and SPSS software (v26)	Tectonic activity is particularly high in the southern region where the NW-SE tectonic lineaments intersect with MCT II, encompassing sub-watersheds 16 and 17, and where numerous lineaments are present in various directions.

2. Geology

The upper catchment of the Alaknanda watershed lies under the Tethys Himalaya (TH) and is composed of Badrinath Granite and mostly the Central Crystallines. Towards south it is characterized by the Tethys Himalaya, the Higher Himalaya (HH), and a portion of the Lesser Himalaya (LH). Trans-Himadri Fault (THF) or the South Tibetan Detachment System (STDS) separates THS from HH. Main Central Thrust (MCT) is the boundary between the HH and the LH (Figure 1). The southern boundary of the LH is the Main Boundary Thrust (MBT). To the north and south of the Dudatoli syncline, respectively, the North Almora Thrust (NAT) and South Almora Thrust (SAT) divide the LH (Yin, 2006; Mukherjee, 2013). NAT is an out-of-sequence thrust and is presently active (review in Mukherjee, 2015).

With a variety of lithologies, dissection and drainage developments, the region is intricate. The litho-units in the study area are the Ramgarh Formation (lower crystalline zone) between the Munsiyari and

Ramgarh Thrusts, the Munsiyari Formation (middle crystalline zone) between the Vaikrita and Munsiyari Thrusts, and the Vaikrita Group (upper crystalline portion lying between the STDS and the Vaikrita Thrust) (Srivastava and Mitra, 1994; Valdiya, 1995, 2001; Bhattacharya, 2008; Ray and Srivastava, 2010). In detail, the study area comprises of ten major geologic units- Badrinath Granite/Tourmaline Granite, Baijanath Formation, Central Crystallines, Garhwal Group (Pithoragarh), Garhwal Group (Bering), Martoli Formation, Rakcham Granite, Rawali Bagar Group, Sumna Group and an unamapped area (Figure 2).

3. Data acquisition and methods

From the Digital Elevation Model (DEM) of 30 m * 30 m spatial resolution of the SRTM the drainage lines were delineated. Based on the DEM (taken on 11-Feb-2000, released on 23-Sept-2014), watersheds were identified, evaluated and morphometric indices were generated (Table 2).

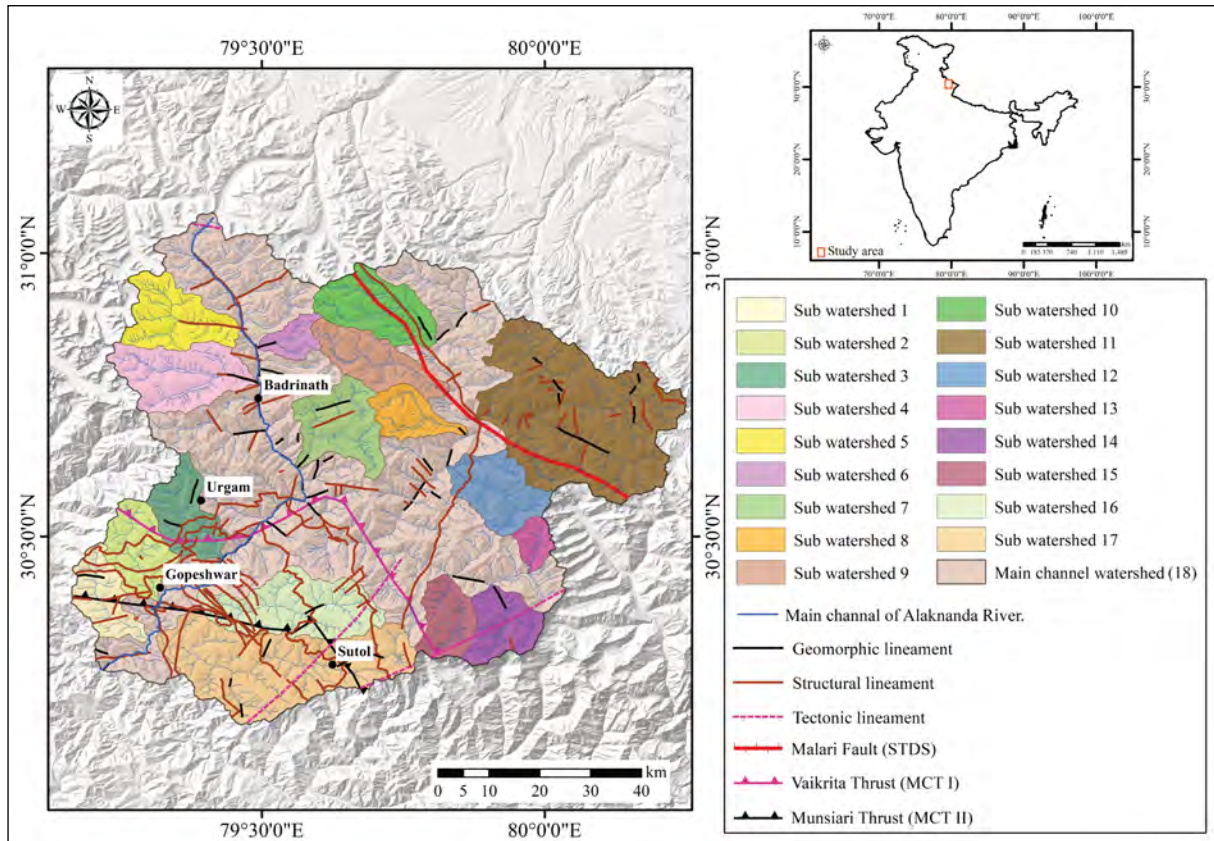


Figure 1- Location of the study area upper part of Alaknanda River with 17 delineated sub-watersheds and major tectonic features.

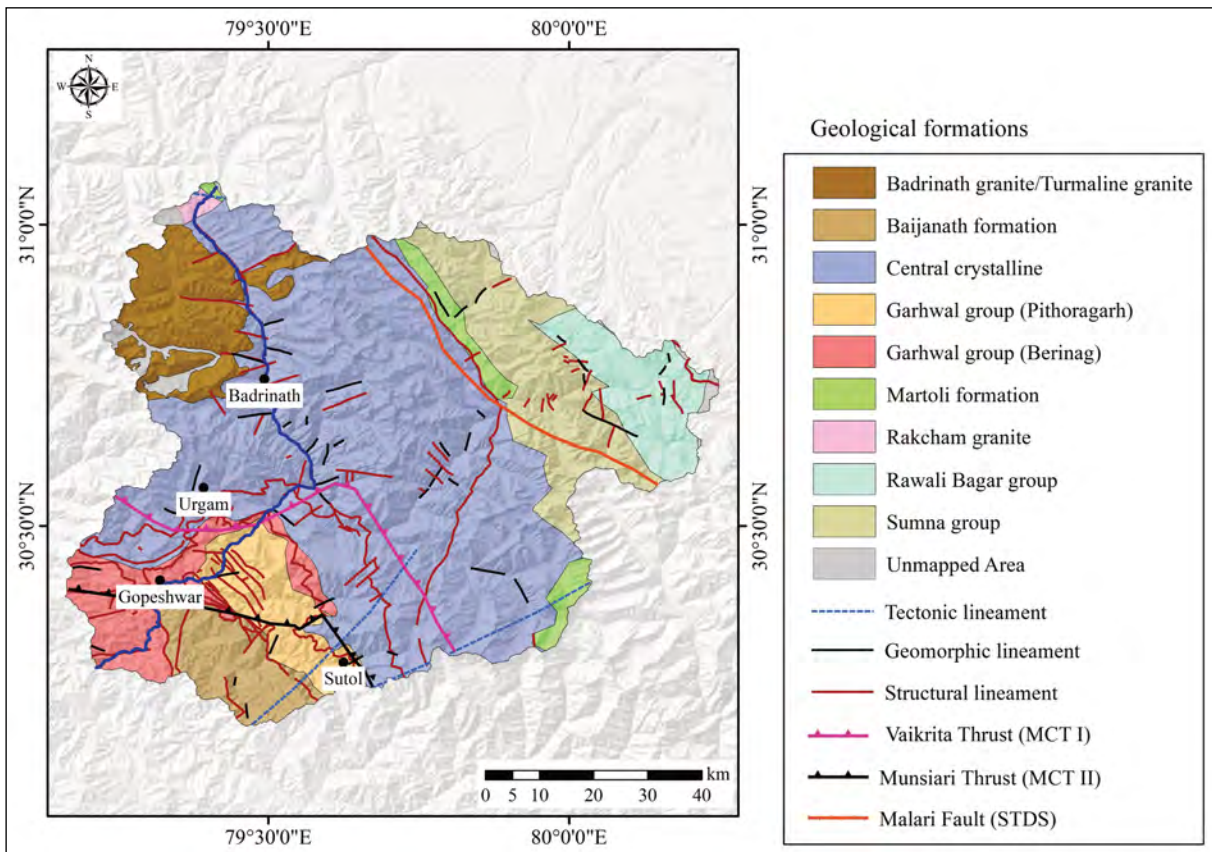


Figure 2- Map of general geology of the study area.

There were no clouds present when the photographs were taken. Therefore, no atmospheric correction was necessary. Several areal, linear and relief are assessed using the ArcGIS of the version of 10.3 (2014). The desired threshold value was utilized to create and extract the stream network using the D8 Python program incorporated in the ArcGIS itself. Slope directions have been deduced from each pixel to the eight-neighbour pixels. The watershed limits delineated by discrete flow angles and a single flow direction. The extracted drainage lines were mapped in the ArcGIS 10.3 (2014) platform. Seventeen sub-watersheds were delineated along the main channel of Alaknanda. The delineation of the sub-watersheds is based on the order of streams as per Strahler (1964). The streams of and above third order are considered for the delineation of the sub-watersheds. The lower order streams are not considered as the sub-watersheds do not have a considerable areal extent to understand the tectonic imprint upon them.

After careful review of the past literature, we have selected eight morphometric indices incorporating relief, areal aspects. The selected parameters are: Hypsometric integral (HI), Drainage texture (D_t), Asymmetry Factor (AF), Ruggedness number (R_d), Circularity Ratio (R_c), Valley Floor Width/Height Ratio (V_{fw}), Lemniscate Coefficient (k) and Basin Shape Index (B_s). The details of all the parameters along with its relevance in morpho-tectonic studies are listed in the Table 2. These are considered to calculate the IAT and to perform the clustering analysis using dendrogram for the considered watersheds. IAT ranks the watersheds as per their tectonic prioritization into four classes- very high, high, medium and low through mutual comparison. Magnitudes of these parameters have been grouped into four classes 1 to 4. The watersheds are categorized under low, moderate, high and very high classes in respect to tectonic activeness. The previously mentioned indices have been adopted for the selected seventeen sub watersheds as well as on the main channel watershed. The relation is:

Table 2- Details of equations and results of considered spatial and linear parameters for the study.

Sl. No.	Formula for basin scale indicators (unitless)	Equations & meaning of symbols	Explanations	References
1.	Hypsometric integral (HI)	$\frac{(Ele_{avg} - Ele_{min})}{(Ele_{max} - Ele_{min})}$ Ele_{avg}: Average of elevation Ele_{min}: Minimum elevation Ele_{max}: Maximum elevation	The hypsometric integral is a dimensionless number that allows different sub-basins to be compared regardless of the scale. Hypsometric Integral indicates both tectonic activeness and lithological control. The rate of relief uplift is positively correlated with the hypsometric integral. HI value varies between 0 and 1 and a value nearer to 1 indicate a tectonically very active basin, 0.5 HI value is an indicator of equilibrium (mature) state and a value nearer to 0 indicates tectonically less active basin. Pike and Wilson (1971) mathematically derived the simpler form of hypsometric integral and shown proved that it is a valid approximation of actual hypsometric integral.	Strahler (1952) e.g. Chen et al. (2003); e.g. Pike and Wilson (1971); (Hamdouni et al., 2008).
2.	Elongation ratio (R_e)	$Re = 1.128 \sqrt{\frac{A}{L_b}}$ A: signifies the area of the basin, L_b: denotes the length of the basin.	Elongation ratio is influenced by geology and climate. The values vary from 0 to 1 (e.g. Wołosiewicz, 2018). As the landscape evolves, the river basin becomes circular and the value tends to be 1.	Schumm (1956); e.g. Wołosiewicz (2018)
3.	Lemniscate coefficient (k)	$k = \frac{\pi L_b^2}{4A_B}$ Where, A_B is the area of basin and L_b is the length of the basin.	Lemniscate coefficient based on expression at basin with Lemniscate curve (i.e. ratio of basin area to its length) in order to determine the shape of the basin. Higher Lemniscate values indicate an elongated basin, which in term denote tectonically very active drainage basin, whereas the low Lemniscate values are associated with less tectonically active basin.	Chorley (1957)
4.	Asymmetry factor (AF)	$AF = \left(\frac{A_r}{A_t}\right) * 100$ A_r: area of the basin (km²) to the right of the main channel facing downstream and A_t: total area (km²) of the basin.	An indicator to measure how much a river basin is tilted due to tectonics. Tectonic activity causes the main stream to change course, sloping away from the basin's midline.	Hare and Gardner (1985); e.g. Anand and Pradhan (2019)
5.	Ruggedness number (Hd)	$Hd = R * D$ R = Basin relief D= Drainage density of the basin	Surface unevenness as topographic differentiation is measured.	Horton (1932)
6.	Valley floor width height ratio (V_{fw})	$V_{fw} = \frac{2V_{fw}}{[(E_{ld} - E_{rd}) + (E_{rd} - E_{sc})]}$ Here V_{fw}: width of the valley floor, E_{ld} and E_{rd}: elevations of the divide on the left and right side of the valley, respectively. E_{sc}: average elevation of the valley.	This index differentiates between valleys with a wide floor relative to the height of valley dividers with a “U” shape compared to narrow, steep valleys with a “V” shape. Valleys with a U shape generally have high values of V_p while V-shaped valleys with relatively low values. Since uplift is associated with incision, the index is thought to be a cause for dynamic tectonics where low values of V_f are associated with higher rates of uplift and incision. This index may be a degree of incision and not uplift; but in an equilibrium state, incision and uplift are nearly coordinated.	Bull and McFadden (1977)

Table 2- Continued.

7.	Drainage texture (D _t)	$D_t = N_u/P$ Nu: Number of streams of a given order. P: Perimeter (km) of the watershed.	The underlying lithology, infiltration potential, and relief characteristics of the terrain all affect drainage texture. D _t is the total number of stream segments in all orders along the watershed's edge (Horton, 1945). This is the relative channel spacing in fluidly prepared terrain, depending on many natural factors such as climate, precipitation, vegetation, rock / soil type, permeability, undulations, and watershed development stages. On the other hand, T is the product of D _d and F. It is calculated by multiplying the drainage density by the current frequency. If less than 4, T is classified as a coarse drainage texture. Intermediate drainage structure if it is between 4 and 10; fine drainage structure if it is between 10 and 15; ultrafine drainage structure if it exceeds 15 (Smith 1950).	Cox (1994)
8.	Basin shape index (Bs)	$B_s = \frac{Bl}{B_w}$ Bl: measured length from headwater to the point on the mouth of the basin, B _w : measured width at the widest point on the basin.	In the tectonically active area, the basin tends to be elongated.	E.g. Ramírez-Herrera (1998); Bull and McFadden (1977); Anand and Pradhan (2019)

$$IATc = \sum p_n(1r + 2r + 3r \dots + nr)/N \quad (\text{equation 1})$$

IATc: class of Index of Active Tectonics, p_n : parameter number, r: rank, N: number of parameters.

Clustering can discriminate signals (grouping signals) from noise (source signals). This technique works on unsupervised classification. Therefore, no prior assumptions are needed. This enables a better research outcome from the heterogeneous value ranges of the concerned indicators. In this study, hierarchical clustering is performed based on same indicators in sub-watershed scale. Using the Euclidean-based dissimilarity method, each segment was compared to its neighbours to assess how similar they were. The procedure was carried out using the following formula:

$$dR = (\sqrt{\sum_{i=0}^n (X_i - Y_i)})/n \quad (\text{equation 2; Clubb et al., 2014})$$

Here n: number of segments, X_i: distance between the segments, Y_i: difference in the steepness indices between the two profiles, i: component of the array, and dR: Euclidean-based dissimilarity. Using Ward's minimum variance method, hierarchical clustering was carried out using the dR values in the SPSS software (version 26, 2019). To show how each

segment mutually relates, a dendrogram is created. Information about stress patterns is gathered from the World Stress Map (Internet reference 2: <https://www.world-stress-map.org/> (Accessed on 16-Aug-2023).

4. Results

IAT was computed by taking the mean of the different classes of the areal and relief parameter. IAT values were sorted into four classes (Table 3) and mapped. As per the IAT analysis, sub-watersheds 4, 8, 9, 16 and 17 come under the tectonically very highly active region where IAT values under the range of 1.500-1.781. Sub-watersheds 6, 7, 10, 15 and 16 are under the magnitude of high tectonic activity (IAT = 1.782-2.063). The IAT range from 2.064-2.344 includes 1, 2, 3 and 14 sub watersheds, which are moderately active. Sub-watersheds at the NE portion as 11, 12 and 13 and at the NW part as 3 and 5 are under low range of active tectonic with IAT = 2.345-2.625 (Table 3). It is clear from the IAT map (Figure 3) that (i) tectonically active regions (watersheds 16 and 17) are characterized by the presence of Munsiyari Thrust (MCT II) that extends east-west. Besides there are NE-trending tectonic lineament and several multidirectional structural lineaments. (ii) Tectonically

Table 3- Class ranges and results of IAT analysis with magnitude.

Sub-watershed	Asymmetry Factor (AF)	Basin Shape (Bs)	Elongation Ratio(Re)	Lemniscate Coefficient (k)	Hypsometric Integral (HI)	Drainage Texture (Dt)	Ruggedness Number (Rd)	Valley Floor Width/ Height Ratio (Vfw)
1	6.955	1.836	0.709	1.989	0.383	1.963	21.462	0.030
2	4.619	2.029	0.683	2.143	0.426	2.135	29.896	0.016
3	1.666	2.089	0.656	2.323	0.465	1.795	47.841	0.164
4	8.518	2.046	0.672	2.214	0.469	4.621	52.415	0.070
5	1.324	1.248	0.819	1.488	0.509	3.476	32.595	0.420
6	13.396	1.422	0.679	2.167	0.474	1.760	18.309	0.576
7	19.142	1.096	0.780	1.643	0.511	4.146	49.232	0.125
8	18.105	1.618	0.629	2.522	0.524	3.401	45.456	0.098
9	0.695	2.645	0.549	3.319	0.460	2.361	58.393	0.209
10	16.025	1.856	0.729	1.878	0.398	3.996	53.190	0.402
11	4.388	1.219	0.923	1.172	0.461	5.704	24.294	0.173
12	0.127	1.434	0.790	1.601	0.523	4.443	31.643	0.230
13	6.122	1.432	0.697	2.057	0.426	1.454	36.302	0.704
14	8.260	1.436	0.806	1.537	0.367	2.641	58.522	0.026
15	10.702	1.937	0.745	1.799	0.513	2.741	54.558	0.272
16	4.579	2.216	0.604	2.740	0.367	2.342	46.025	0.044
17	2.736	2.348	0.608	2.701	0.313	5.037	45.065	0.023
Main channel watershed (18)	26.304	1.247	0.907	1.214	0.479	15.824	29.045	0.019

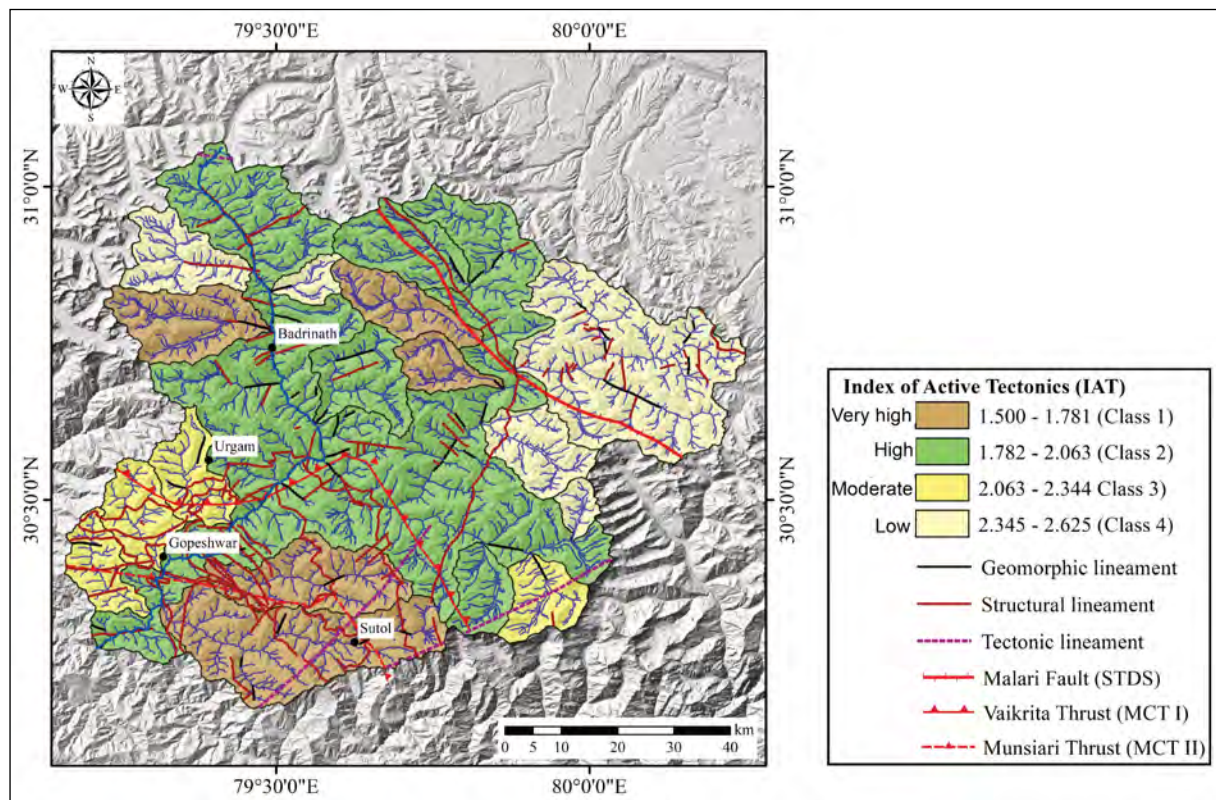


Figure 3- Map of Index of Active Tectonics (IAT) with 4 classes and 4 magnitudes as very high, high, moderate and low.

less active regions are distinguished by fewer presence or even absence of structural lineaments / faults.

The prepared dendrogram from the dR values enabled clustering of the calculated indicators of all watersheds (Figure 4). Cluster investigation of waterway profiles can compare the groups of similar waterway profiles inside heterogeneous scenes. The dR values are calculated to create a symmetric nearness lattice (Table 4). There are two major clusters as per the rescaled remove cluster combination. These are cluster 1 (sub-watersheds 2, 4, 14, 15 and 16) and cluster 2 (sub-watersheds 6-8 and 18) (Table 4).

5. Discussions

Comparison between two methods as IAT and clustering done in this work indicate a positive similarity in assessing active tectonics of Alaknanda sub-watersheds. Cluster 1 covers watersheds 2, 4, 14, 15 and 16 belong to very high (1.500-1.781), high

Table 4- Class ranges and results of IAT analysis with magnitude.

Class range	Magnitude	Sub-watersheds
1.500-1.781	Very High	4, 8,9, 16, 17
1.782-2.063	High	10,18,15
2.064-2.344	Moderate	1,2, 3, 14
2.35-2.625	Low	11,12,13

(1.782-2.063) and moderate (2.064-2.344) magnitudes of IAT. Cluster 2 covers sub-watersheds 6, 7, 8 and 18, which are under very high (IAT = 1.500-1.781) and high (IAT = 1.782-2.063) tectonic activeness (Table 5a,b).

Active thrust faults in the southern direction of the considered area covers sub watersheds 1, 2, 7, 16, and 18 (Figure 5). These watersheds belong to class 1 and 3 according to the IAT classes. Along the main channel of Alaknanda river near 20 km upstream of Gopeshwar, a hitherto unnamed strike-slip fault

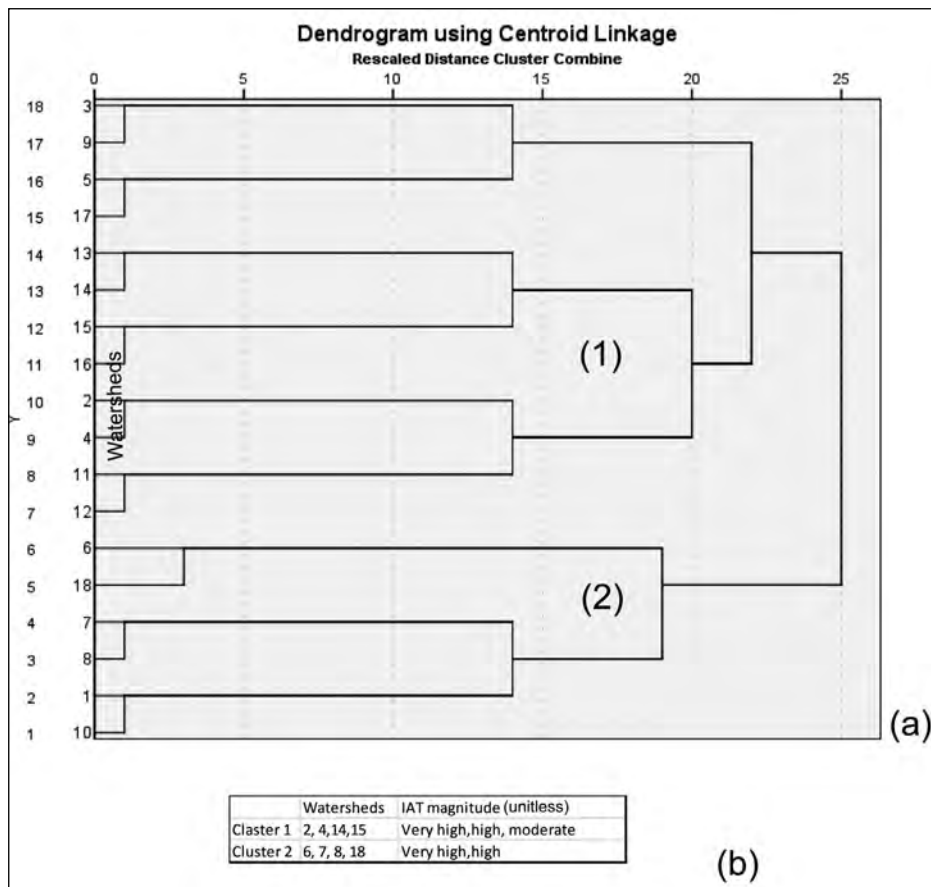


Figure 4- a) Dendrogram showing two specific clusters, b) Considered watersheds under class 1 and 2.

Table 5a- Results of agglomeration schedule for calculating cluster 1 and 2.

Agglomeration Schedule						
Stage	Cluster	Combined	Coefficients	Stage Cluster First Appears		Next Stage
	Cluster 1	Cluster 2		Cluster 1	Cluster 2	
1	5	12	3.358	0	0	6
2	16	17	11.599	0	0	4
3	4	15	13.126	0	0	7
4	3	16	14.503	0	2	13
5	7	8	16.956	0	0	10
6	2	5	17.929	0	1	9
7	4	14	25.790	3	0	11
8	1	11	29.720	0	0	12
9	2	13	34.458	6	0	14
10	7	10	36.056	5	0	15
11	4	9	60.159	7	0	13
12	1	6	71.818	8	0	14
13	3	4	97.740	4	11	15
14	1	2	129.028	12	9	16
15	3	7	149.008	13	10	17
16	1	18	262.847	14	0	17
17	1	3	468.580	16	15	0

Table 5b- Proximity Matrix (similarity) of 18 sub-watersheds.

Proximity Matrix																		
Case	Correlation between Vectors of Values																	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	1.000	0.986	0.960	0.987	0.957	0.929	0.994	0.995	0.954	0.998	0.972	0.943	0.987	0.985	0.992	0.976	0.964	0.813
2	0.986	1.000	0.993	0.999	0.991	0.854	0.967	0.966	0.991	0.986	0.984	0.983	0.999	0.999	0.997	0.999	0.994	0.725
3	0.960	0.993	1.000	0.991	0.998	0.792	0.934	0.933	1.000	0.962	0.974	0.994	0.991	0.993	0.986	0.998	0.997	0.650
4	0.987	0.999	0.991	1.000	0.991	0.862	0.973	0.971	0.987	0.989	0.990	0.983	0.998	0.999	0.998	0.997	0.993	0.747
5	0.957	0.991	0.998	0.991	1.000	0.786	0.933	0.930	0.998	0.961	0.984	0.998	0.987	0.991	0.983	0.996	0.999	0.670
6	0.929	0.854	0.792	0.862	0.786	1.000	0.955	0.958	0.777	0.926	0.843	0.756	0.866	0.856	0.882	0.829	0.799	0.914
7	0.994	0.967	0.934	0.973	0.933	0.955	1.000	1.000	0.925	0.996	0.960	0.915	0.973	0.969	0.981	0.954	0.939	0.855
8	0.995	0.966	0.933	0.971	0.930	0.958	1.000	1.000	0.924	0.995	0.956	0.911	0.972	0.968	0.980	0.953	0.937	0.851
9	0.954	0.991	1.000	0.987	0.998	0.777	0.925	0.924	1.000	0.955	0.971	0.995	0.987	0.990	0.981	0.996	0.997	0.635
10	0.998	0.986	0.962	0.989	0.961	0.926	0.996	0.995	0.955	1.000	0.976	0.946	0.989	0.987	0.994	0.977	0.966	0.815
11	0.972	0.984	0.974	0.990	0.984	0.843	0.960	0.956	0.971	0.976	1.000	0.982	0.980	0.984	0.982	0.981	0.987	0.784
12	0.943	0.983	0.994	0.983	0.998	0.756	0.915	0.911	0.995	0.946	0.982	1.000	0.977	0.982	0.972	0.989	0.997	0.652
13	0.987	0.999	0.991	0.998	0.987	0.866	0.973	0.972	0.987	0.989	0.980	0.977	1.000	0.999	0.999	0.997	0.989	0.728
14	0.985	0.999	0.993	0.999	0.991	0.856	0.969	0.968	0.990	0.987	0.984	0.982	0.999	1.000	0.999	0.998	0.992	0.726
15	0.992	0.997	0.986	0.998	0.983	0.882	0.981	0.980	0.981	0.994	0.982	0.972	0.999	0.999	1.000	0.994	0.986	0.755
16	0.976	0.999	0.998	0.997	0.996	0.829	0.954	0.953	0.996	0.977	0.981	0.989	0.997	0.998	0.994	1.000	0.997	0.693
17	0.964	0.994	0.997	0.993	0.999	0.799	0.939	0.937	0.997	0.966	0.987	0.997	0.989	0.992	0.986	0.997	1.000	0.685
18	0.813	0.725	0.650	0.747	0.670	0.914	0.855	0.851	0.635	0.815	0.784	0.652	0.728	0.726	0.755	0.693	0.685	1.000

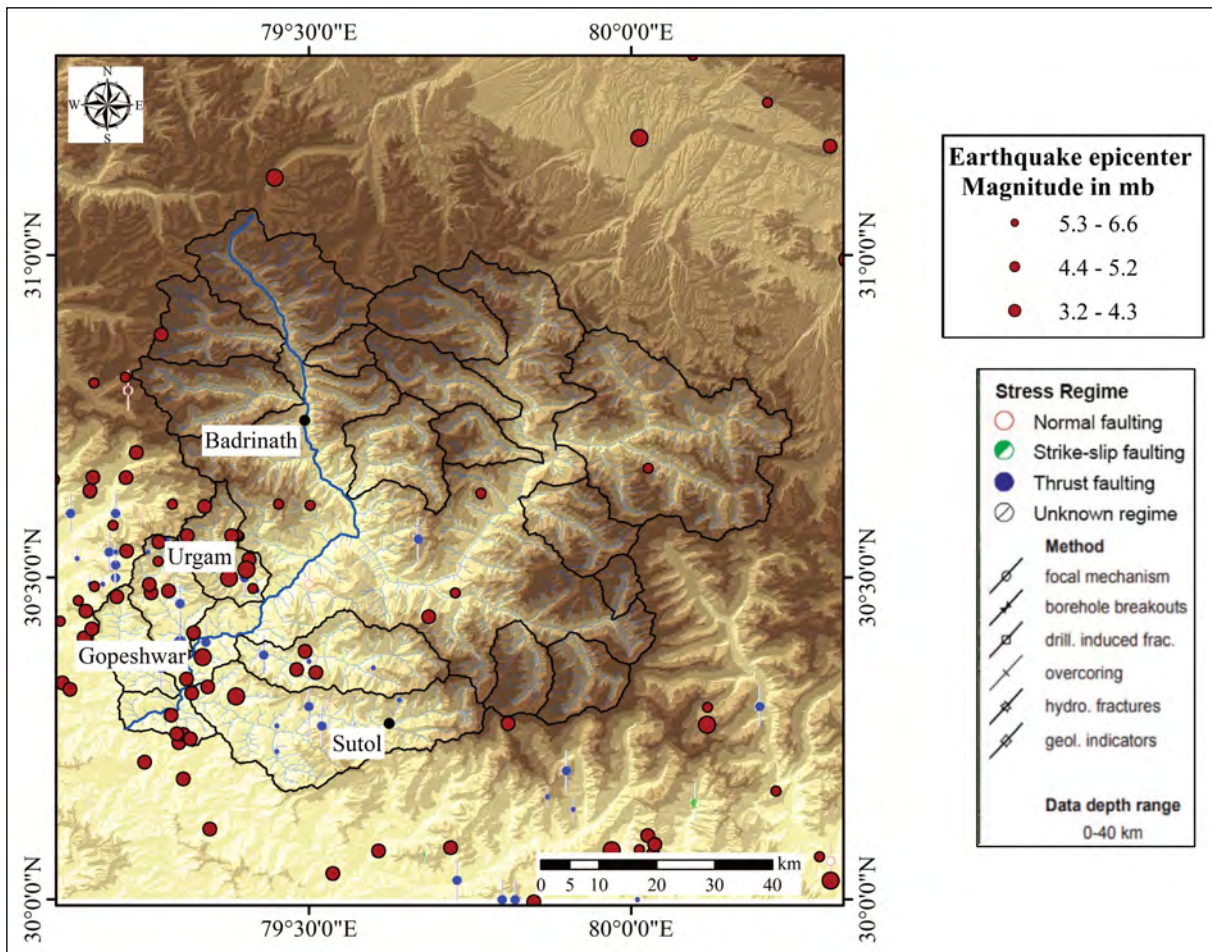


Figure 5- World stress map of the study area with 18 watersheds and IAT classes marked as C1-4.

exists (Figure 5). GPS time series data acquired near Gopeshwar disclose a high rate of displacement of 24.72 mm y^{-1} (Sharma et al., 2018). Major strike-slip and thrusts are located between the MCT I and MCT II (Figure 5).

Conclusions

For morphometric assessment of active tectonic, geomorphic indices that can be easily produced from the high resolution DEMs presently available through satellite observation are used. Such indices can be used to analyze data that covers a broader area quickly. We study 18 sub-watersheds of the upper Alaknanda basin in this work. Sub-watersheds 16 and 17 at the southern portion where NW-SE tectonic lineaments cross MCT II and where several lineaments exist in multi-direction, tectonic activity is very strong. High-to-moderate activeness was observed at the places on

the periphery of the very high activity zone. This is also confirmed from cluster analysis.

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Effect of calculated dry rock bulk modulus on uncertainty in compressional wave velocities derived from Gassmann fluid substitution theory and fluid mixing law in a CO₂ sequestration case

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Research Article

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ABSTRACT

This study examines the effect of variations in dry rock bulk modulus (K_{dry}) on compressional wave velocity (V_p) using Gassmann fluid substitution theory and various fluid mixing laws in the context of CO₂ sequestration in the Utsira formation at the Sleipner Field. Significant changes in V_p , exceeding 600 m/s, were observed, particularly when applying the Gassmann-Wood law, which demonstrated heightened sensitivity to saturation variations. In contrast, the Gassmann-Brie approach, utilizing an empirically selected constant, produced more stable and reliable results. This study emphasizes that relying solely on the Gassmann-Wood equation for K_{dry} calculations may result in inaccurate predictions, particularly for CO₂-saturated formations. To mitigate this, laboratory measurements of K_{dry} are recommended to enhance the reliability of predictions. Furthermore, initial saturation conditions critically affect the V_p calculations, underlining the importance of selecting an appropriate mixing law to ensure accurate assessments during CO₂ injection and storage.

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1. Introduction

Since the industrial revolution, the effect of greenhouse gases in the atmosphere has dramatically increased. Taking its effect on the environment and human health into account, CO₂ is obviously the most harmful greenhouse gas with a high proportion (64%) in the atmosphere (Bachu, 2000). The main reason for this situation is the consumption of fossil fuels as a primary energy resource all around the world (Omeregbe et al., 2020). Only in 2019, the total emission of CO₂ by the U.S. refining industry is nearly 166×10^7 tonnes (EPA, 2020). In order to reduce the concentration of CO₂ gases in the atmosphere, carbon capture and storage (CCS) projects have been widely implemented (Yao et al., 2018; Metz et al., 2005).

Sleipner project is the first industrial scale carbon capture and storage project that has been approved and successfully operated in the North Sea since 1996 (Chadwick et al., 2014; Eliasson et al., 2014; Ghosh et al., 2015; Eliasson and Romdhane et al., 2017). Understanding the structural model is critical to increase success rate of CO₂ sequestration. Overall, 16 Mt CO₂ has been injected and stored into the Utsira Sand successfully by 2017 (Park et al., 2013; Kvamme and Aromada, 2018) and 17.8 Mt by 2019 (Ahmadinia and Shariatipour, 2020). Project target of 20 Mt CO₂ injection (Chadwick et al., 2004).

Geophysical techniques, including AVO (Amplitude versus offset) analysis, are powerful tools to estimate CO₂ saturations in the target formation

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(Roverud, 2017). After CO₂ injection, initial condition and final condition can exhibit different elastic properties. So, the injection zone is selected to include highly porous and permeable sand units which are sealed by impermeable cap rocks, like shale or limestone (Wisman, 2012). Porous media includes chemical substance based pore fluids. The security of a CO₂ injection process is indirectly influenced by the estimation of brine and CO₂ saturations in the reservoir while in-situ fluid mixture properties are changing during the injection (Mavko et al., 2009). Therefore, for monitoring the leakage risks, modelling the fluid mixture properties and the rock elastic properties at initial condition are two critical stages. However, modelling the fluid mixture is almost impossible if one of the constituents is CO₂ (Yam and Schmitt, 2011).

The temperature and pressure of the target formation are critical because the compressibility and density characteristics of CO₂ are totally changed if the pressure and temperature of the formation are higher than the critical pressure (~7.4 MPa) and critical temperature (~31 C) (Yam and Schmitt, 2011). In this situation, the phase of CO₂ transforms from gas to liquid or supercritical state. Yam and Schmitt (2011) also showed that the phase of CO₂ changes from gas to liquid instantly around the critical values. Seismic signatures of the target rock are widely varied corresponding to the different phases of injected CO₂ (Njiekak et al., 2013). Thus, identifying the phase of CO₂ has a significant effect on the results.

For a CO₂ injection case, the properties of the final fluid mixture are completely different from those of each constituent. (Vera, 2012; Khalid and Ghazi, 2013; Maurya and Singh, 2019). Recommended mixing models in the literature can be listed as Gassmann-Wood (Gassmann, 1951), Gassmann-Brie (Brie et al., 1995), Gassmann-Hill (Hill, 1963) and Voigt averaging (Voight, 1910). These models provide a theoretical way to estimate the bulk density and Bulk modulus of CO₂-fluid mixture, which is essential to reveal the seismic signature of saturated rock (Wisman, 2012; Njiekak et al., 2013; Ivandic et al., 2018; Vasco et al., 2019). In fact, mixing laws are not suitable for every formation. Defining an appropriate mixing law for target formation is quite significant for reliable

V_p responses on final (Mavko et al., 2009; Roverud, 2017). Gassmann-Wood mixing law is commonly used in the literature to model CO₂ and brine mixture. This approach has been applied in several studies including those by Si et al. (2016), Zhang et al. (2017), Papageorgiou and Chapman (2017). Falcon-Suarez et al. (2018) specifically applied the Wood's law, which is the case of Gassmann equation, to Utsira formation.

Gassmann-Hill approximation is another traditional model used for fluid mixing scenarios. This approach is suggested in the literature if patchy saturation is assumed. Similar to Voigt averaging, mass and heat transfer between the fluids are assumed. Gassmann-Hill approximation is used by many authors and to some cases (Pabst et al., 2015; Eid et al., 2015).

The perspective of Gassmann Brie techniques is a bit different from the other methods because of the 'e' empirical constant in the equation. 'e' constant is a variable that can be defined considering geological details of the porous formation. (Brie et al., 1995; Papageorgiou et al., 2016; Si et al., 2016).

1.1. Geological Settings of Sleipner Field

To increase the reliability of the results, rock and fluid properties are applied to the models in Utsira formation of Sleipner field where is almost the best-known injection site in the world.

The injection location within Utsira Formation is approximately 6 km away from Sleipner Vest field as indicated in Figure 1 (Karstens et al., 2017). Utsira sand is lying between Hordland (bottom) and Nordland (top). The Utsira sand deposition occurred during the Mio-Pliocene period, spanning a significant distance of nearly 400 km in a south-north trending manner. Formation surface is generally smooth, but it exhibits some small undulations, valleys and domes within the depth range of 550 m to 1500 m depths. Formation is dipping mostly towards the south and thicknesses are varying along 200 km from the north (200 m) to the south (300 m). The formation contains about 12 m height domes, which are suitable for CO₂ injection. Within the Utsira Formation, there are mud diapirs accompanied by thrust faults that dip towards the base of the formation (Chadwick et al., 2004; Dupuy et al., 2016).



Figure 1- Location of Sleipner field around Utsira formation (Modified from Labat et al., 2012).

Regarding to the caprock properties, thicknesses are around a few hundred meters and capillary pressure is ranging from 2 to 5.5 MPa that is capable to seal a few hundreds of meters thick CO₂ column. The estimated porosity of the caprock is approximately 0.11% which means that it is managed to store 16 billion tons CO₂ considering less than 12 km migration way (Chadwick et al., 2004; Karstens et al., 2017).

Utsira formation has mostly homogenous sand with a few meters' thick shale layers (Dupuy et al., 2016). The Utsira Sand reflector is typically observed at depths ranging between 800 and 1000 meters. Porosity refers to the fraction of pore spaces within the rock, and these values indicate a significant volume

of available pore space for fluid storage, including CO₂. The estimated porosity is between 30% and 40%. Permeability of the sand is ranging from 1 to 5 Darcy. These values clearly indicate the great storage potential of Utsira sand (Dupuy et al., 2016; Karstens et al., 2017).

Utsira formation also includes poorly cemented, fine-to-medium grain size, mostly moderate sorted 1-6 m thick mudstone layers with 10 to 40 meters overall thickness. Laboratory studies show that Utsira sand consist of multiple constituents including quartz grains, K-feldspars, calcite, plagioclase, shell fragments, calcite, albite, aragonite, muscovite and biotite (Dupuy et al., 2016; Karstens et al., 2017).

1.2. Gassmann Fluid Substitution

Gassmann's fluid substitution theory is applied to estimate P (V_p) and S (V_s) wave velocities of a saturated rock using elastic properties of fluid and rock matrix (Gassmann, 1951). Gassmann's equations use bulk modulus of dry rock that are derived from initial case parameters or sometimes from laboratory measurements.

To estimate dry rock Bulk modulus, the equation given in the Gassmann's theory is expressed as;

$$K_{dry} = \frac{K_{sat\_init} \left(\frac{\phi K_m}{K_{fl\_init}} + 1 - \phi \right) - K_m}{\frac{\phi K_m}{K_{fl\_init}} + \frac{K_{sat\_init}}{K_m} - 1 - \phi} \quad (1)$$

In the equation (1), K_m is Bulk modulus of rock matrix, ρ_{init} is initial bulk density, ϕ is porosity, K_{fl_init} initial Bulk modulus of fluid mixture and V_{p_init} is P wave velocity in initial state (Russell et al., 2001; Bacon et al., 2003).

One of the critical parameter is Bulk modulus of the dry rock (K_{dry}) here because this is derived from the is initially predicted values. In the next step, calculated K_{dry} is used to estimate final saturated rock. Bulk modulus which is essential for traditional compressional wave velocity theory (Telford et al., 1990).

Bulk modulus of the saturated rock in final case can be calculated using equation (2)

$$K_{sat\_fin} = K_{dry} + \frac{(1 - \frac{K_{dry}}{K_m})^2}{\frac{\phi}{K_{fl\_fin}} + \frac{1 - \phi}{K_m} - \frac{K_{dry}}{K_m^2}} \quad (2)$$

One can see in the equations above (Bacon et al., 2003), Bulk modulus of the initial and final fluid mixture are the deterministic inputs that can be estimated from a range of mixing laws.

1.3. Fluid Mixing Laws

Each mixing laws are not suitable for every formation. Therefore, assuming saturation behaviour of the formation is the key parameter to prefer appropriate mixing law. Some mixing laws are based a consideration that the fluid is flowing inside the pore spaces homogenously (uniform saturation), while some of them assume heterogeneous distribution

which is also called patchy saturation (Gassmann, 1951; Mavko et al., 2009; Si et al., 2016). Uniform saturation is expressed if formation reaches equilibrium (relaxed state). In other word, formation fluids are homogenously distributed into the pore spaces of the rock due to a long geological time. This situation is seen mostly in hydrocarbon cases. On the other hand, most of the CO₂ sequestration cases have a different perspective. If any external process, such as CO₂ injection, is applied to the formation, saturation model is frequently assumed as heterogeneous (unrelaxed state formation).

1.3.1. Gassmann-Wood

First essential assumption to consider that Wood's law is formation is saturated homogenously and constituents must remain unrelaxed or frozen state. In other word, any mass or heat transfer between fluids must be neglected (Khalid and Ghazi, 2013). Dry rock Bulk modulus of the fluid mixture are calculated using

$$\frac{1}{K_f} = \sum_i^N \frac{S_i}{K_i} \quad (3)$$

In the equation (3), S_i and K_i are volume fraction, elastic modulus and density of i^{th} fluid respectively (Wood, 1955; Mavko et al., 2009).

1.3.2. Voigt Averaging

This method can also be called Hill's equation or arithmetic average in Si et al. (2016). On the other hand, this equation was called Voigt average in Hill (1963). Bulk modulus of fluid mixture can be calculated using equation (4)

$$\frac{1}{K_f} = \sum_i^N S_i K_i \quad (4)$$

Voigt average has been suggested for heterogeneously saturated formations. In contrast to the Wood's law, due to the patchy saturation model, it is assumed that heat and mass manage to be transferred between fluids. Voigt averaging are applied to CO₂ sequestration cases in Si et al. (2016). Voigt average is also easily applicable to mix three or more fluid constituents.

1.3.3. Gassmann-Brie

Success of this approach directly depends on e empirical constant which is generally between 2 and

5 (Papageorgiou et al., 2016). In Gassmann-Brie approach (Brie et al., 1995), Bulk modulus of fluid mixture (K_{fl}) is derived from equation (5)

$$K_{fl} = (K_w - K_{CO2})S_w^e + K_{CO2} \quad (5)$$

Here, K_w and K_{CO2} are the Bulk modulus of brine and supercritical state CO_2 . S_w and S_{CO2} are the saturation of water and supercritical state CO_2 respectively. Recommended e empirical constants associated with porosity and permeability are given in Si et al. (2016) (Table 3).

However, Papageorgiou et al. (2016) suggested series of equations to calculate e constant in terms of only saturations and capillary pressure (P_{cap}) which is given in equation (6)

$$e = \frac{\log\left(\frac{S_w}{S_w + \alpha(1-S_w)}\right)}{\log(S_w)} \quad (6)$$

And α constant can be calculated using equation (7)

$$\alpha = \frac{K_w - S_w(1-S_w)P_{cap}(S_w)}{K_g - S_w(1-S_w)P_{cap}(S_w)} \quad (7)$$

Here, P_{cap} is the capillary pressure. Capillary pressure between the brine filled formation and injected CO_2 is defined by Chadwick et al. (2005)

$$P_{cap} = 810.35(1 - S_g)^{-0.948} \quad (8)$$

Equation (8) is suggested only if gas in the fluid mixture is CO_2 instead of hydrocarbon

1.3.4. Gassmann-Hill

Gassmann-Hill mixing law (Hill, 1963) takes shear modulus of the patchy saturated rock (μ) into account. So, bulk modulus of fluid mixture can be calculated using equation (9)

$$K_{fl} = \left(\frac{S_w}{K_w + \frac{4}{3}\mu} + \frac{S_{CO2}}{K_{CO2} + \frac{4}{3}\mu} \right)^{-1} - \frac{4}{3}\mu \quad (9)$$

Here, K_{CO2} and S_{CO2} are bulk modulus and saturation of CO_2 respectively.

2. Workflow

General workflow is illustrated in a flowchart given in Figure 2 that shows 2 main stages calculating K_{fl} and approximating V_p . In the first calculations, Fluid mixing laws are applied with regards to the range of scenarios from fully brine to fully CO_2 saturated Utsira sand. In order to estimate V_p by Gassmann fluid substitution equation, needed K_{dry} parameter is both calculated from XRD results

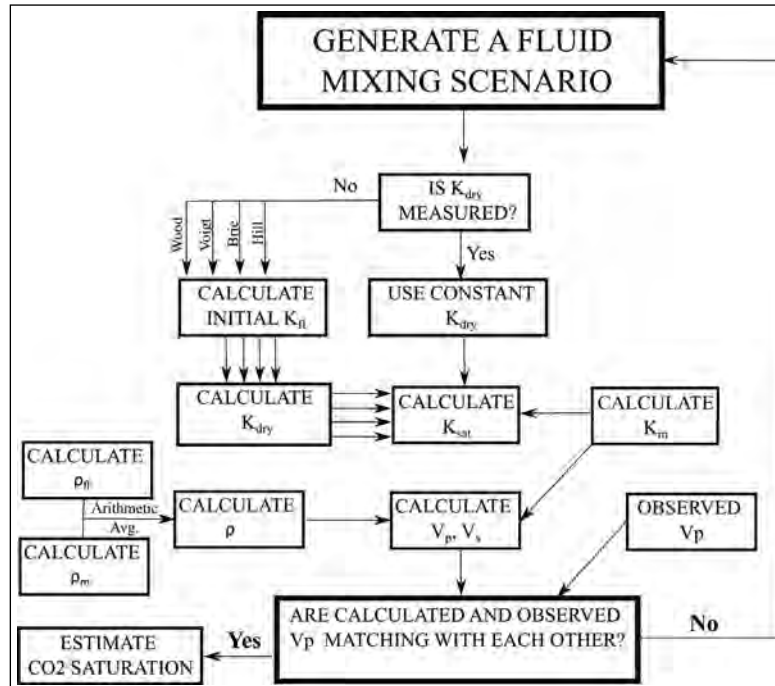


Figure 2- Flowchart of the study. K_m , K_{dry} and K_{sat} represent Bulk modulus of matrix, dry and saturated rock respectively. Also, ρ_f , ρ_m and ρ are densities of fluid mixture, matrix and saturated rock respectively. V_p and V_s are the seismic velocities.

(Chadwick et al., 2005), given in the Table 1, and directly taken from measurements (Dupuy et al., 2017). Calculations are done in MATLAB (Matrix Laboratory) software of Mathworks®. It helps to interpret the results comparatively. Calculated V_p values are checked if it matches with each other and the V_p suggested by Chadwick et al. (2005) which is called ‘observed’ data hereinafter. It helps to analyse the suitability of the mixing laws and observing sensitivity of K_{dry} calculation on final V_p .

3. Field Data and Model Parameters

In this study, observed rock elastic properties and seismic characteristics of Utsira sand within Sleipner CO₂ injection site is used as a field data (Table 2 and Table 3).

As it was expressed, temperature and pressure effecting on the formation are determinant factors to estimate phase of injected CO₂. Critical temperature and pressure of CO₂ are given as 31.1°C and 7.39 MPa respectively in Ravazzoli and Gomez (2014). Temperature and pressure of Utsira sand is measured as nearly 116 °C and 26.2 MPa respectively (Ahmed et al., 2016). Thus, injected CO₂ in this formation condition is turned into liquid phase (Ravazzoli and Gomez, 2014).

On the other hand, sand and shale matrix are measured using XRD analyse from core samples

(Chadwick et al., 2004) and given in Table 2. Using matrix constituents and its volumes, Bulk modulus and density of rock matrix are also derived from Voigt-Reuss-Hill averaging method in this study (Table 3). Elastic properties of the minerals are taken from Bacon et al. (2003) and Pabst et al. (2015).

Relaxation state and saturation model of a formation are dependant to the time needed for equilibrium. In hydrocarbon case, gas or oil inside the formation is distributed homogenously (uniformly) during a long geological time. This is called relaxed state. Contrarily, injected CO₂ cannot be distributed homogenously due to the limited time (Eid et al., 2015; Zhang et al., 2017). This is called unrelaxed state. In this situation, patchy saturation is considered as saturation model of the formation. Defining the saturation model is essential to choose the suitable mixing law. In this study, relaxed and unrelaxed state is regarded to illustrate the differences between results. Observed values from Chadwick et al. (2004) and Dupuy et al. (2017) indicate that full of brine water saturated Utsira sand patch has 2050 m/sec and 636 m/sec P and S wave velocities respectively. Moreover, Bulk density of sand patch is measured as 2.078 g/cc (Table 3). Also, Chadwick et al. (2004) suggested that P wave velocity reduces to 1420 m/sec in the case of whole CO₂ saturated sand that is consistent with

Table 1- Mineral constituents of Utsira sand and Utsira shale (Chadwick et al., 2004).

	Quartz	Calcite	K-feldspar	Albite	Aragonite	Smectite	Kaolinite	Mica	Other
Utsira Sand	75%	3%	13%	3%	3%	-	-	3%	
Utsira Shale	30%	3%	5%	2%	-	3%	14%	30%	11%

Table 2- Utsira formation rock and fluid properties. Porosity (ϕ), Shear modulus (μ), Bulk modulus of rock matrix (K_m), P wave velocity (V_p), S wave velocity (V_s), bulk density of rock (ρ), Bulk modulus of fluids (K_f), density of fluids (ρ_f) and saturation of fluids (S) (Chadwick et al., 2005; Dupuy et al., 2017; Roverud, 2017; Falcon-Suarez, 2018).

		ϕ [%]	μ [GPa]	K_m [GPa]	V_p [m/sec]	V_s [m/sec]	ρ [g/cc]
Formation	Utsira Sand	37.5	0.84	39.29	2050	636	2.078
	Utsira Shale	13	2.4	22.6	2450	1050	2.2
		K_f [GPa]		ρ_f [g/cc]		S [%]	
Fluids	Water	2.3		1.03		100	
	CO₂	0.075		0.7		0	

Table 3- Calculated parameters used to calculate final P wave velocities. While final saturations are stayed the same, initial saturations of fluids are varied to observe the effect of mixing laws on final P wave velocities.

S_{w_init}	S_{g_init}	S_{w_fin}	S_{g_fin}	Methd	K_{fl_init}	K_{fl_fin}	K_{sat_init}	K_{dry}	K_{sat_fin}	r_m	r_{fin}	V_{p_fin}
Initial water saturation	Initial CO ₂ saturation	Final water saturation	Final gas saturation	Mixing Law	Bulk modulus of fluid mixture in initial case	Bulk modulus of fluid mixture in final case	Bulk modulus of saturated rock in initial case	Bulk modulus of dry rock	Bulk modulus of saturated rock in final case	Density of matrix	Density of the rock in final case	P wave velocity in final case
[]	[]	[]	[]	[]	[Gpa]	[Gpa]	[Gpa]	[Gpa]	[Gpa]	[g/cc]	[g/cc]	[km/sec]
1	0	0.00	1.00	Wood	2.3	0.075	7.61	2.72	2.89	2.71	1.95	1.43
				Voigt	2.3			2.72	2.89			1.43
				Brie	2.3			2.72	2.89			1.43
				Hill	2.3			2.72	2.89			1.43
0.998	0.002	0.00	1.00	Wood	2.1712	0.075	7.61	3.05	3.22	2.71	1.95	1.49
				Voigt	2.2955			2.73	2.91			1.44
				Brie	2.2956			2.73	2.91			1.44
				Hill	2.2873			2.75	2.93			1.44
0.99	0.01	0.00	1.00	Wood	1.7738	0.075	7.61	4.03	4.19	2.71	1.96	1.65
				Voigt	2.2777			2.78	2.95			1.44
				Brie	2.2782			2.78	2.95			1.44
				Hill	2.2375			2.88	3.06			1.46
0.95	0.05	0.00	1.00	Wood	0.9262	0.075	7.61	5.88	6.02	2.72	1.96	1.91
				Voigt	2.1887			3.01	3.18			1.48
				Brie	2.1909			3.00	3.17			1.48
				Hill	2.0087			3.46	3.63			1.56
0.9	0.1	0.00	1.00	Wood	0.5798	0.075	7.61	6.56	6.70	2.73	1.97	1.99
				Voigt	2.0775			3.29	3.46			1.53
				Brie	2.0817			3.28	3.45			1.52
				Hill	1.7632			4.05	4.21			1.65
0.8	0.2	0.00	1.00	Wood	0.3317	0.075	7.61	7.02	7.15	2.75	1.98	2.05
				Voigt	1.855			3.83	4.00			1.61
				Brie	1.8635			3.81	3.98			1.60
				Hill	1.372			4.94	5.09			1.77

Dupuy et al. (2017). This expression is considered to define ‘expected values’ that must be matched with the model results with an acceptable uncertainty range. However, formation saturation associated with these velocities has some uncertainties. Measuring or estimating exact P wave velocities of fully CO₂ saturated sand is not possible in practice. In any case, these published V_p values are defined as the closest values to the V_p of fully CO₂ and water saturated sand case (Figure 3). Thus, these values are used to test the theoretical approaches in this study.

4. Result and Discussion

The main objective in the study is to understand the effect of Bulk modulus of dry rock on V_p . Therefore, initial results focussed on the Bulk modulus of fluid mixture behaviour depending on the fluid saturation variations. As it can be seen in the Figure 4, Bulk modulus of fluid mixture within the Utsira sand is decreasing exponentially from nearly 2.2 GPa to 0.1 GPa with ascending the CO₂ saturations. This module forms the Bulk modulus of the saturated Utsira sand proportionally and linearly between 2.7 and 7.6 GPa in Figure 5. Relevant graph shows also V_p is slowing

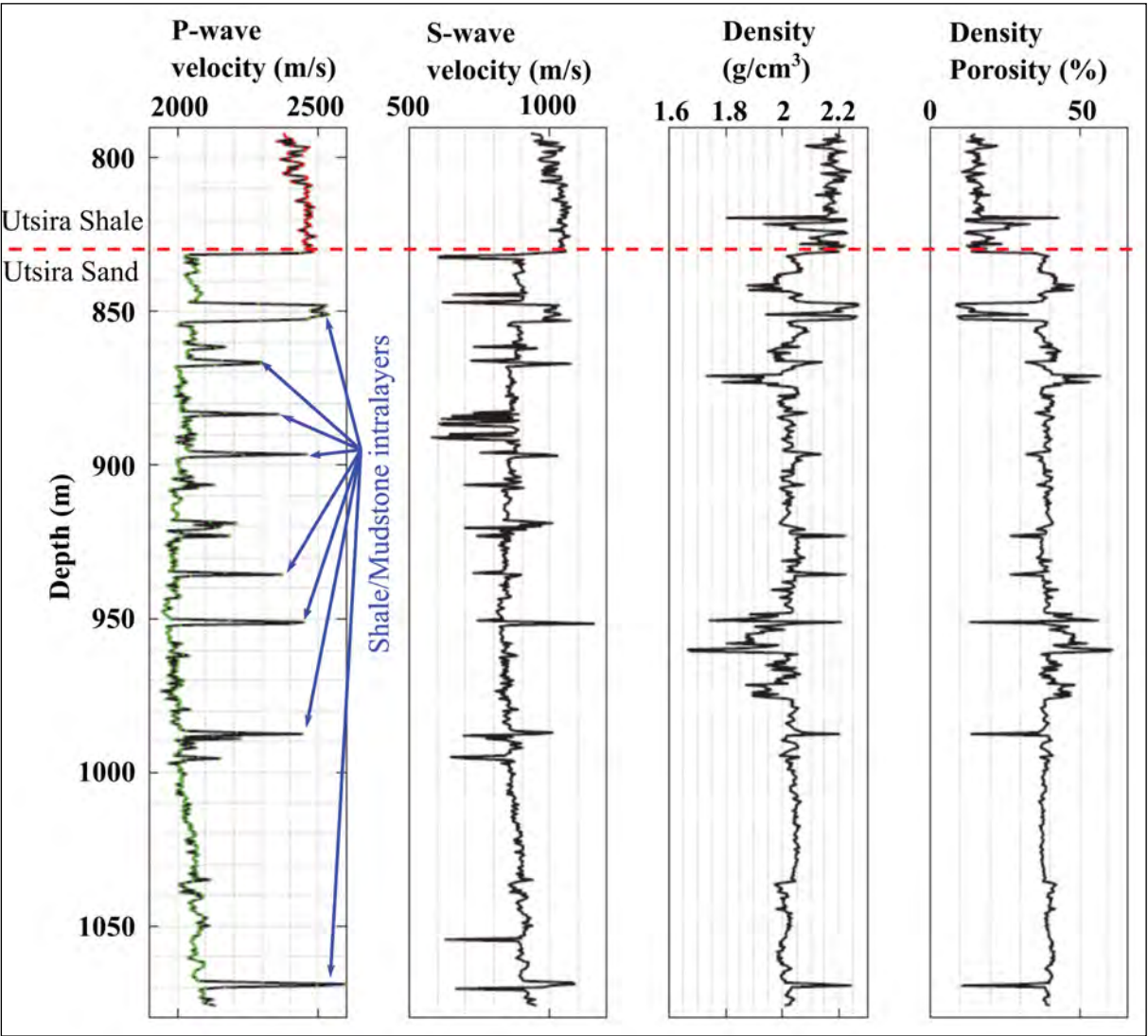


Figure 3- Seismic velocities, density and porosity logs of Utsira formation between 790 m and 1080 m depth (Modified from Dupuy et al., 2017).

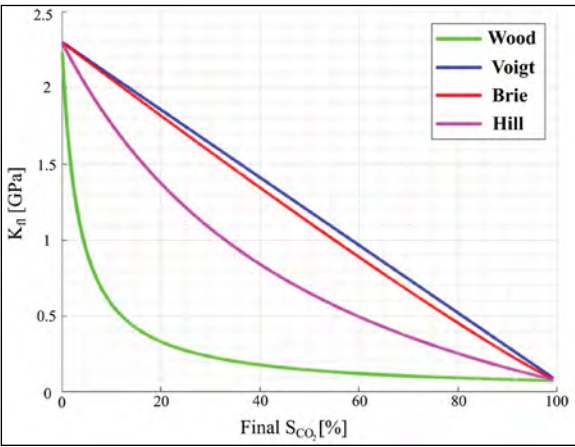


Figure 4- Changing of Bulk modulus of fluid mixture within the pore spaces of the rock as CO₂ saturation increases.

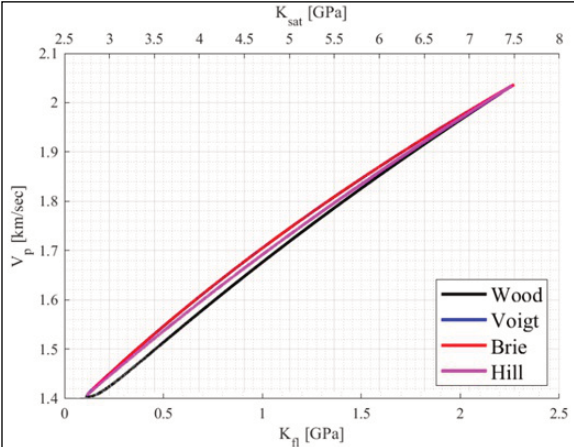


Figure 5- Bulk modulus of the fluid within the rock (K_f) versus P wave velocity (V_p) of the rock graph.

down from nearly 2.05 km/sec to 1.4 km/sec if Utsira sand CO_2 saturation increases during the injection.

In order to analyse the behaviour of V_p in detail during a CO_2 injection scenario, each mixing law is applied separately for calculating Bulk modulus of fluid mixture (Figure 6). Particularly, Brie mixing law is applied multiple time because its results are being changed dramatically by e empirical constant. As it is illustrated in Figure 7, 30% CO_2 saturated Utsira sand gives a range of Bulk modulus of fluid mixture between 0.1 GPa and 1.65 GPa regarding to e empirical constant changing from 10 to 1. These range is relevant to 1.4 km/sec and 1.85 km/sec in the same formation respectively (Figure 5). However, suggested maximum value of e empirical constant is lesser than 5 (Papageorgiou and Chapman, 2017) which point out maximum 1 GPa fluid mixture Bulk modulus around 50% CO_2 saturation scenario (Figure 7). Therefore, 3 different scenarios for Brie mixing law are taken into account in Figure 6 to analyse the results of the mixing

laws comparatively. Plots in Figure 6 is also derived from different initial CO_2 saturation estimation (0%, 1%, 5% and 20% respectively). Results clearly show that initial fluid saturation estimations dominate the estimated V_p for a CO_2 sequestration scenario. While fully brine and fully CO_2 saturated Utsira sand indicates the same V_p (2.05 km/sec and 1.45 km/sec respectively) in Figure 6a, finally full of CO_2 saturated Utsira sand scenario shows a wide range of V_p (between 2.05 km/sec and 1.6 km/sec) for an initially 20% CO_2 saturated Utsira sand in Figure 6d. Similarly, the same range is seen in finally full of brine saturated Utsira sand case. This indicates a question mark on reliability of the results derived from different fluid mixing laws. In Figure 6d, for dully CO_2 saturated Utsira sand, while almost 2.03 km/sec V_p is read from Wood's curve, 1.6 km/sec is seen on Voigt average and Brie's law ($e=1$). Practically, the mixing models showing exponential behaviour in the graph plot response CO_2 saturation changes more sensitively than the ones

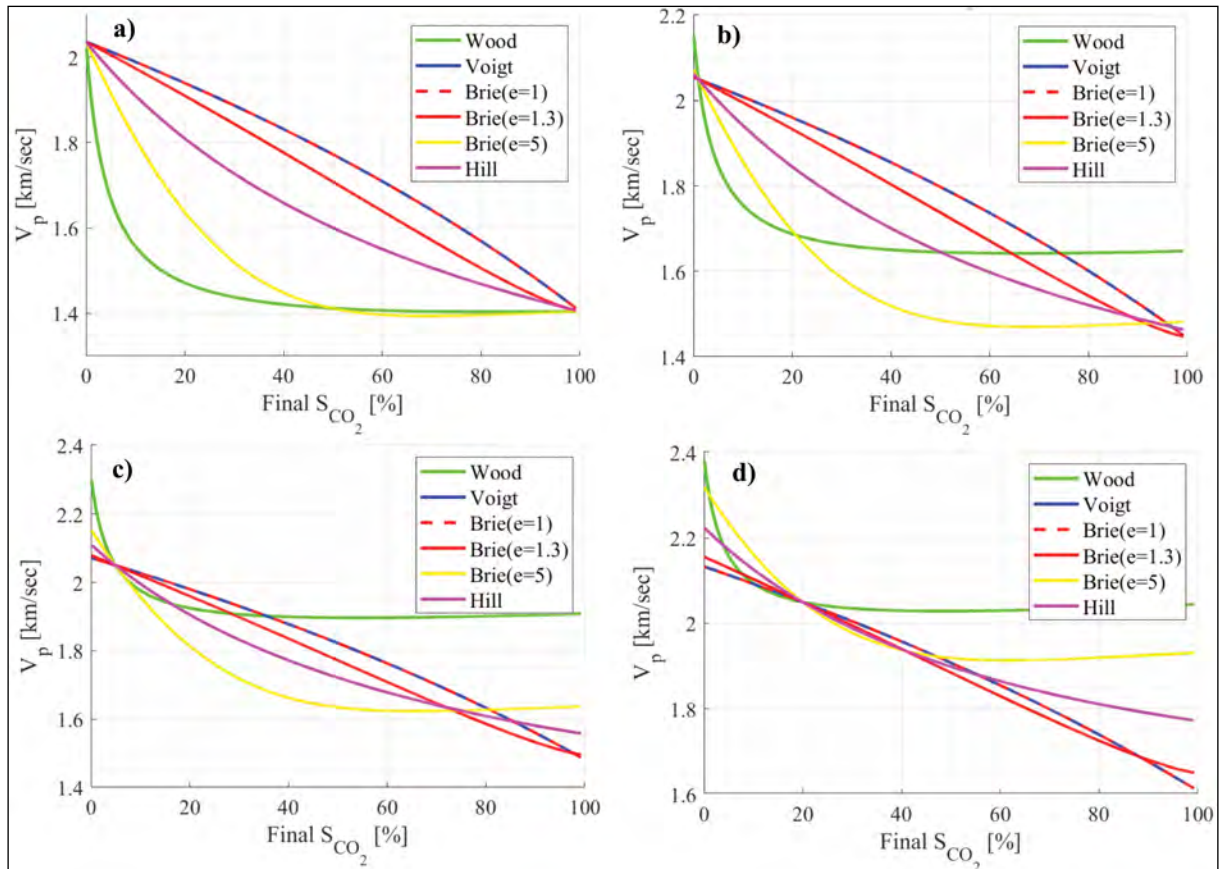


Figure 6- P wave velocity versus final CO_2 saturation graphs, a) Bulk modulus of dry rock is calculated using different mixing laws. Initial CO_2 saturations are entered 0%, b) 1%, c) 5%, d) 20%.

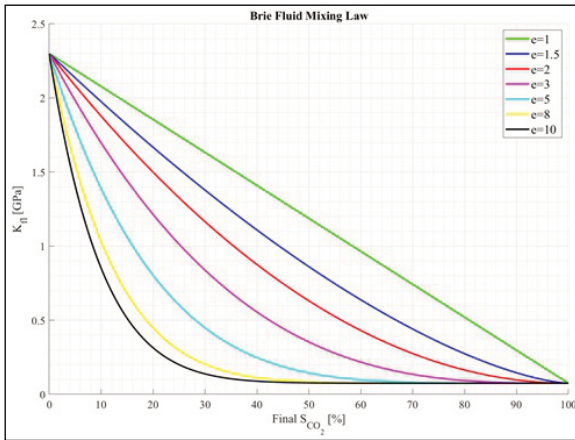


Figure 7- Change of bulk modulus of fluid mixture with increasing final CO₂ saturation of the rock. Bulk modulus of fluid mixture is derived from Brie mixing law with a range of e empirical constants.

that show linear behaviour. Similar V_p range is also seen in the Brie mixing laws derived from $e=1$ and $e=5$. Curve behaviours are obviously coming from the mathematical equation of Bulk modulus of fluid mixture of each mixing law. Overall results reveal the significance of defining accurate initial saturations if dry rock Bulk modulus is not measured in laboratory tests.

Initial CO₂ saturation dependency of dry rock Bulk modulus is also modelled in Figure 9. Final CO₂ saturation is set as 100% for an Utsira sand sample while initial saturations are varied. It is clearly seen that Wood's law is quite sensitive to the CO₂ saturation changes if its saturations assumed to be less than 20%. Considering initial CO₂ saturation between 0% and

20%, dry Utsira sand Bulk modulus is ranged from 2.7 GPa to 7 GPa. On the other hand, if initial CO₂ saturation is higher than these critical values (around 20%), dry Utsira sand Bulk modulus is not changing remarkably. Consequently, attention must be paid if Wood's law is preferred for a scenario CO₂ injected into initially brine saturated formation.

Sensitivity of each mixing law is also seen in Table 3 that includes Bulk modulus of fluid mixture, Bulk modulus of dry Utsira sand, Bulk modulus of saturated Utsira sand and P wave velocity respectively. Only initial CO₂ saturation assumption is ranged to observe the sensitivity of mixing laws. While initial CO₂ saturation is increasing from 0% to 20%, difference between Wood's response and the other mixing law response are increasing exponentially. Differences in Wood's law from initially Bulk modulus of fluid mixture to V_p on final is folding with each variable derivation. On the other hand, linear behaviour mixing laws response similarly ($V_p \approx 1.6$ km/sec), such as Voigt average and Brie's law ($e=1$).

It is also possible to measure K_{dry} in the laboratory. In this point, in order to dig in the K_{dry} dependency of the mixing laws, constant and measured K_{dry} are used in the equations to analyse the estimated V_p velocities comparatively. Regarding to the observed K_{dry} (2,56 GPa) (Dupuy et al., 2017), V_p versus CO₂ saturation graphs plotted in Figure 8a and 8b that clearly show the limits and behaviour of the V_p are not dependant to the initial saturation assumptions. However,

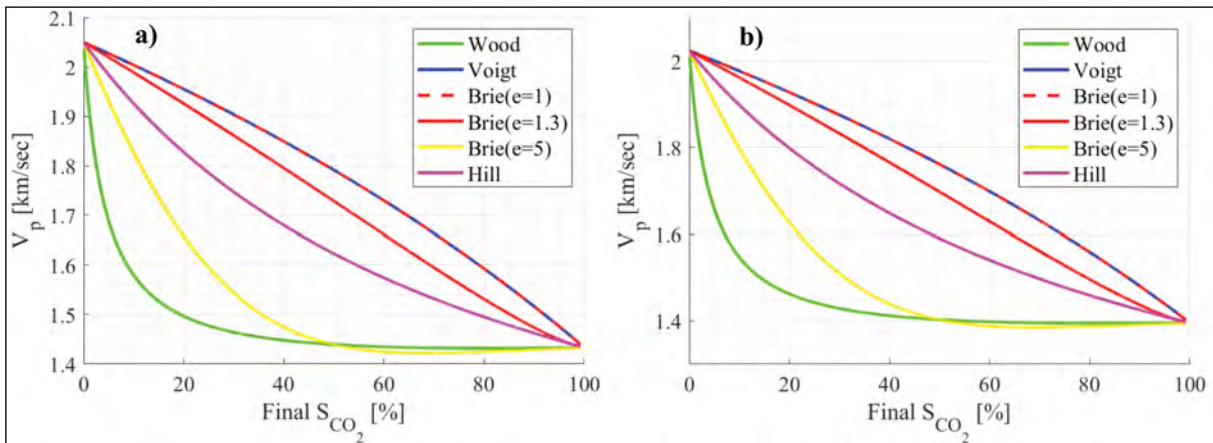


Figure 8- P wave velocity versus final CO₂ saturation graphs, a) Measured Bulk modulus of dry rock (2.56 GPa) (Dupuy et al., 2017) is used for all mixing laws. Initial water saturations are 100%, b) 80%.

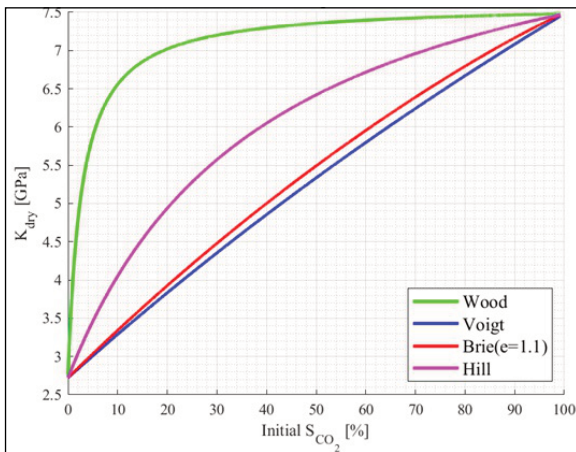


Figure 9- Initial saturation dependency of the K_{dry} values derived from finally full of CO_2 saturated Utsira sand sample.

unrecognizable descend can be seen in Figure 8b because of diminished average density of the fluid mixture. Furthermore, V_p of the fully brine and CO_2 saturated sand is almost 2.05 km/sec and 1.4 km/sec respectively in every mixing model. These values are matching not only with each other but also with the suggested results for Utsira sand case in Chadwick et al. (2004) and Dupuy et al. (2017) (Figure 3).

V_p curve characteristics differences between the results derived from observed K_{dry} (Figure 8) and calculated K_{dry} (Figure 6) are dramatically increasing with increasing CO_2 saturation of the sand. For a scenario of fully CO_2 saturated Utsira sand which is initially 20% CO_2 saturated, approximately 2.05 km/sec V_p is estimated from Wood's law if dry Utsira sand Bulk modulus is calculated using Gassmann fluid substitution theory. 2.05 km/sec indicates sand is still fully brine saturated, considering the suggested V_p for a fully CO_2 saturated Utsira sand (Figure 6d). Consequently, these misleading results show that using calculated dry rock bulk modulus in a CO_2 leakage monitoring study is risky and unreliable. Not only Wood's law but also even Voigt average and Brie's law ($e=1$) cannot give the reliable results ($V_p \approx 1.6$ km/sec). On the other hand, V_p is estimated 1.4 km/sec (Figure 8b) from the observed and constant dry Utsira sand bulk modulus that is matching with the literature (Chadwick et al., 2004; Dupuy et al., 2017). Saturation behaviour of a formation can be uniform or patchy which mean the seismic signature of the formation is changing suddenly or in time. In other

words, V_p - S_{CO_2} graph gives a clue about the unrelaxed state (suitable for patchy saturation) or relaxed state (suitable for uniform saturation) of the formation.

Exponential response of V_p indicates Gassmann Wood or Brie mixing law ($e>3$) as a suitable model. Otherwise, Voigt or Brie mixing law ($e=1$) suggest more reliable V_p . Hill's law is particular model that can be suitable for *semi-uniform* saturation behaviour. Hill model can be matched with Brie's law for precise e empirical constant between 1.5 and 2.5. However, defining accurate V_p response to CO_2 injection in a laboratory is quite difficult because of supercritical state of CO_2 . Therefore, defining the mixing model or e empirical constant considering all rock and fluid elastic properties are definitely one of the critical part in the success of CO_2 sequestration.

Results of this study cannot recommend a suitable mixing model particularly for Utsira sand because estimated V_p from fully CO_2 saturated sand is matching with the expected results for all mixing models. On the other hand, results also show that measuring the Bulk modulus of dry Utsira sand is a must to rely on the V_p responses. This study verifies the suggested 2.56 GPa as dry Utsira sand Bulk modulus.

5. Conclusion

In light of the challenges and expenses associated with geological methods, this study emphasizes the widespread utilization of geophysical techniques, including seismic 3D/4D and AVO analysis, for monitoring CO_2 sequestration. Employing various fluid mixing scenarios and Gassmann fluid substitution theory for V_p estimation, we underscore the pivotal role of dry rock bulk modulus in modeling CO_2 sequestration processes, with a focus on the Utsira sand as a representative rock sample.

Multiple approaches, including Gassmann-Wood, Gassmann-Brie, Voigt Averaging and Gassmann-Hill are employed to calculate the bulk modulus of fluid mixtures. The results prominently highlight the unacceptably wide range of dry rock bulk modulus for sensitive CO_2 injection monitoring. Consequently, we strongly recommend (1) laboratory measurements of dry rock bulk modulus for reliable and sensitive V_p estimation. Additionally, our findings indicate the

indirect dependence of the V_p - SCO_2 curve on the bulk modulus of the fluid mixture, emphasizing the critical importance of understanding the saturation behavior or relaxation state of the formation for accurate CO_2 saturation estimation during injection.

This study specifically applies the Brie mixing law, revealing that (3) it can be suitable for both patchy and uniform saturation models with a precisely defined empirical constant. To determine the correct saturation model for Utsira sand, we propose (2) injecting a pre-defined fluid mixture into the pore spaces of Utsira sand in a laboratory study, plotting V_p versus fluid saturation graphs to identify a suitable mixing law or accurate empirical constant for various CO_2 injection scenarios.

Moreover, we raise a question regarding AVO responses of fluid mixing laws, suggesting that analyzing AVO responses of Utsira sand considering fluid mixing laws could be a valuable supplemental study. This comprehensive exploration provides valuable insights for refining methodologies in CO_2 sequestration monitoring, advocating for laboratory studies and precise empirical constants for improved accuracy in saturation estimation.

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Effect of degree of saturation on consolidation parameters of fine-grained soils

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Research Article

Keywords:

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Compression and
Recompression Index.

ABSTRACT

Terzaghi (1943)'s theory of consolidation phenomenon has some limitations for unsaturated geological environments in nature. Additionally, soils are subjected to consolidation tests under partially saturation conditions. The scope of this study is to illustrate the effect of degree of saturation (S_r) on the consolidation parameters using remolded soil samples. For this purpose, swelling pressure, compression index (c_c), re-compression index (c_r), coefficient of consolidation (c_v), and pre-consolidation stress (σ_p) were determined for the samples having different degrees of saturation and plasticity (PI). The consolidation parameters obtained from "undersaturated" remolded samples with varying degrees of saturation were compared with the consolidation parameters obtained from "fully saturated" remolded samples to illustrate which parameters of consolidation are affected most by the degree of saturation. The effect of swelling of partially saturated samples was eliminated prior to conducting oedometer tests. Results indicated a meaningful relationship between the compression index and the degree of saturation, though the relationship with the recompression index was less certain. Pre-consolidation stress generally decreased as saturation increased, while no definitive trend was observed between the degree of saturation and the consolidation coefficient. Overall, most consolidation parameters were found to be somewhat affected by the degree of saturation.

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1. Introduction

Terzaghi (1943)'s one dimensional consolidation theory the following assumptions are made: the soil is homogeneous and fully saturated, soil particles and pore water are incompressible, and water flow during pore water dissipation is only in the vertical direction, the consolidation is one-dimensional (1D). Additionally, the change in soil volume due to consolidation is negligible compared to the initial soil volume, flow is laminar and follows Darcy's law, and permeability remains uniform throughout the compressible soil layer. The coefficient of volume compressibility remains constant over time, and

compression occurs solely from pore water expulsion, so initial and secondary consolidation are neglected. Finally, the coefficient of consolidation for a specific soil and site conditions is assumed to be constant.

Terzaghi (1943)'s consolidation theory postulates that soil specimen subjected to oedometer tests are fully saturated, a postulation is valid only for samples collected from the capillary fringe or below the water table (ASTM 2435/D2435M-11, 2020). Thereby, the theory of consolidation has some limitations for unsaturated geological environments in nature, which are sited most of engineering projects and/

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or foundations above fully saturated zones where the soil is partially saturated. Additionally, soil samples taken from boreholes will be unsaturated due to volume expansion as overburden pressure is removed. And in practice, test specimens are directly subjected to consolidation test regardless of the zone where the soil sample collected from. That means no saturation procedure is applied prior to consolidation test. Although the soil specimen is inundated at the beginning of the test, this procedure is only for preventing of the specimen against drying during the test (ASTM 2435/D2435M-11, 2020). In other words, inundation of the test specimen prior to the consolidation test does not promote saturation of soil specimen at all. Eventually, it turns out that all soils are subjected to consolidation tests under partially saturation conditions.

No earlier study was found investigating the effect of saturation degree on consolidation parameters for remolded soils by performing conventional consolidation experiments. Some researchers investigated the effects of soil index properties on certain consolidation-related parameters such as the pre-consolidation pressure, compression index. Some previous studies investigating consolidation properties in natural and/or remolded soils are summarized below. Several studies have been carried out to date on the investigation of the consolidation properties of soils., recompression index and swelling pressure by employing methods such as fuzzy logic, finite elements, certain empirical relationships as well as computer simulations (e.g., Dipova and Cangir, 2010; Naser, 2013; Kayabalı and Yıldız, 2014; Sünnetçi and Ersoy, 2016; Yurtçu and Özocak, 2016). These researchers revealed in general that the index properties of soils and the consolidation parameters can be calculated. Kayabalı and Özdemir (2010), Asadi (2017) and Balcı et al. (2018) tried to find the consolidation parameters in natural and remolded soils by using centrifugal consolidation technique, which can reduce the duration of experimental studies that take more than a week in the traditional consolidation method to a few hours. For this purpose, the consolidation parameters obtained from the centrifugal consolidation method and the consolidation parameters obtained from the

one-dimensional consolidation experiments were compared. Gibson et al. (1967), Yong et al. (1983) and Fox and Berles (1997) explain high strains in soils. Biot (1941) developed Terzaghi (1925)'s one-dimensional consolidation theory in three dimensions and used this theory (Biot, 1956) in solving dynamic problems. Working on one-dimensional consolidation theory, Davis and Raymond (1965), Xie et al. (2002), Geng et al. (2006) and Cai et al. (2007) argued that as a result of loads applied on the sample during consolidation test, the compressibility and permeability decrease with the shrinking of the pores in the soil. Hansbo (1960), Davis and Raymond (1965) and Geng (2008) developed nonlinear solutions of consolidation problems by using compression and permeability coefficients that decrease as a result of loading. Working on remolded clays prepared by considering the degree of saturation, Barden (1965) explained the stress state on unsaturated soils using Bishop (1959)'s relationship. Fredlund (1977), Fredlund and Morgenstern (1976) developed mathematical relations for the solution of consolidation problems in unsaturated soils. Casagrande and Fadum (1940) and Taylor (1942) developed methods for finding the consolidation coefficient, and Cortellazzo (2002), Muntohar (2009) compared the methods developed by different researchers to determine the consolidation coefficient. Boone (2010), who worked on the calculation of the pre-consolidation stress, also presented the work of many researchers in summary form. Phanikumar and Amrutha (2014) aim that the roles of overburden pressure and the degree of saturation in changing the void ratio and compressibility of clays. For this purpose, conducted a series of consolidation tests on soil samples with a wide range of saturation degrees (25, 50, 75 and 100%) to ensure pre-excavation, post-excavation and post-construction conditions in a laboratory environment. However, the main purpose of their study is not to compare the effect of saturated and unsaturated soils on consolidation parameters. To realize the pre-excavation and post-excavation conditions in the laboratory environment, they applied and removed 25, 50 and 100 kPa stress to the samples with different degrees of saturation and applied a stress up to 400 kPa to achieve the post-construction condition.

2. Materials

A total of 8 soil samples with different plasticities were used in this investigation. Samples were collected from the vicinity of Ankara, Türkiye. They were dried and sieved through the #40 sieve prior to the compaction and consolidation tests. Phase relationships of solid-water-air system (e.g., Holtz and Kovacs, 1981) were used to prepare identical samples. For this, the initial void ratios and dry unit weights

of the samples were kept constant. The index and physical properties of the samples were determined, and the samples were compacted using static methods. The permeability-consolidation cell (Figure 1) was used to saturate the partially saturated samples and to perform consolidation experiments on these samples. Swelling pressure value of partially saturated samples was measured in the setup developed by Kayabali and Yıldız (2014) (Figure 2).



Figure 1- Permeability-consolidation test apparatus and its accessories (from Asadi, 2017).



Figure 2- Swelling pressure test apparatus (from Kayabali and Yıldız, 2014).

3. Methods

To determine the physical and index properties of samples, sieve analysis, hydrometer test, Atterberg limits tests and pycnometer test were performed, and soil classes were determined according to Unified Soil Classification System (USCS). For the plastic limit and liquid limit tests, the part of the samples that passed #40 sieve was used. Plastic limit tests were carried out using the hand-rolling method according to ASTM D 4318-05 (American Society of Testing Materials, 2005) with 5 replicates of each sample and 50 g of the sample amount in the replicate. Liquid limit experiments were performed using the falling cone method according to BSI 1377 (British Standards Institution, 1990) at 5 points corresponding to the penetration amounts between 10-30 mm for each sample. The solid particle densities of the samples were calculated by applying the pycnometer test according to ASTM D 854-06 (American Society of Testing Materials, 2006a), using 25 g dry sample in 50 ml pycnometers.

The nature of the investigation requires preparation of soil samples with certain physical properties. To investigate the effect of various degrees of saturation on specific consolidation parameters the void ratio, the dry mass of soil sample and the total volume of the compacted soil sample need to be kept constant. The only variable is the amount of water for the preparation of test specimens. From each soil sample, 4 identical specimens were prepared which are homogeneously mixed at 70, 80 and 90 percent degrees of saturation. During the sample preparation stage, 2 samples at 70% saturation and the other 2 samples at 80% and 90% degrees of saturation, respectively, were prepared using the static compaction method. One of the samples compacted at 70% degree of saturation was used to obtain a fully saturated sample in the permeability-consolidation setup.

Initial trial tests showed that respectively at void ratios of 0.7 and 0.9 to CL and CH types of soils are suitable to prepare the consolidation test specimens to result in total wet densities recorded in most fine-grained soils. All necessary computations were made to find the proper amounts of dry soil mass and water

for the initial degree of saturation of first 0.7 to fill a consolidation ring with the diameter of 50 mm and a height of 20 mm. The soil and water were mixed and poured into a hollow cylinder with 50 mm diameter. Compressive load was incrementally applied up to 1 kN onto the wet mixture using a piston and load frame. After this, the specimen was turned upside down and the same loading procedure was applied from the reverse side and the resulting specimen height was measured. This procedure was repeated until the specimen height is reduced to 20 mm. At the end, a test specimen with diameter of 50 mm, a height of 20 mm, a void ratio of 0.7 and a degree of saturation of 0.7 was obtained. The statically compacted soil specimen was then transferred to a consolidation ring. The same specimen preparation procedure was adopted to obtain specimens with degrees of saturation of 0.8 and 0.9 keeping the void ratio of 0.7, the amount of dry soil mass and the total volume constant. All procedures outlined above both for CL and CH type of soils were repeated for using the void ratio of 0.7.

It is well known that a compaction procedure never yields a final product with the “zero air void”. Therefore, to prepare a fully saturated specimen ($S_r=1.0$) a different scheme was employed. For this, the spare specimen with the degree of saturation of 0.7 was prepared and placed into a specific cell shown in Figure 1. This is a two-stage procedure to obtain a fully saturated test specimen. The specimen in this cell first is allowed to saturate under a hydraulic head of about 50. After the water appears at the bottom outlet the specimen is left under hydraulic pressure for an additional period so that at least one pore-volume of water passes through the specimen. During the saturation stage, a load equaling to swell force determined from the procedure outlined below is kept on top of the test specimen to prevent the swelling of specimen during saturation. The second stage involves the routine one-dimensional consolidation procedure.

Swell pressure of soils is known to be controlled by dry density and water content. Therefore, artificially prepared soil specimens are expected to exhibit some degree of expansion when subjected to inundation prior to consolidation test. To obtain reliable data free

of swelling effect from the consolidation tests, certain precautions need to be taken to eliminate such effects. For this, a methodology was adopted in which the swell pressure of the compacted soil sample under certain void ratio and degree of saturation was measured on another identical sample. The consolidation specimen was placed in a consolidation cell. The loading piston attached to a load cell was lowered gradually on top of the specimen (Kayabalı and Yıldız, 2014, Figure 2). An initial seating load of 10N was applied to ensure that there is no gap between the top of the loading piston and the top cap laying on top of the specimen. The specimen was inundated and left so for a period of 24 hours. At the end of 1 day the total swell force was read from the digital display from which the initial seating load was deducted and then the remaining value was divided by the surface area of the test specimen to obtain the swell pressure.

Consolidation tests was carried out starting by eliminating the swell pressure recorded earlier (American Society of Testing Materials, 2006b). For this, the specimen was loaded to the measured swell force and then it was inundated so that the additional height coming from the swelling process is cancelled out. Vertical loads applied such that the resulting effective stresses are 25, 50, 100, 200, and 400 kPa. Unloading of the specimens was rendered as the loading sequence was reversed; namely from 400 kPa to 200 kPa, from 200 kPa to 100 kPa and so on. Vertical displacements were recorded immediately after placing the consolidation load. The readings were taken at times of 5 sec., 10 sec., 30 sec., 60 sec.,

2 min., 4 min., 10 min., 30 min., 1 hr., 2 hrs., 4 hrs., 8 hrs., and 24 hrs. Vertical displacement versus square root of times (in minutes) curve were constructed to determine t_{90} values of each stage of consolidation test per specimen. t_{90} values were used in computing the coefficient of consolidation, c_v .

4. Experimental Results

4.1. Physical and Index Properties of Soil Samples

The physical and index properties of 8 clay samples were determined initially. After that, swelling pressures, compression indices, re-compression indices, pre-consolidation stresses and consolidation coefficients for 32 homogeneous specimens (8 samples multiplied by 4 different levels of saturation) obtained from these samples were determined through the swelling and consolidation tests. Particle size distribution, liquid limit, plastic limit, solid particle density and soil classes according to the USCS for the investigated soil samples are given in Table 1 and Figure 3.

The liquid limit of the samples varies between 24.4 and 98%, the plastic limits are between 14.8 and 31.4% and thus the plasticity indices range from 9.6 to 66.6%. N1, N2 and N3 samples were classified as low plasticity clay (CL) and N4, N5, N6, N7 and N8 samples as high plasticity clay (CH). The specific gravity of the samples ranges from 2.68 to 2.78. As a result of sieve and hydrometer analyzes the percentages of sand, silt and clay of the samples vary between 16-80%, 18-64% and 3-37%, respectively.

Table 1- Index properties of soil samples employed in the investigation.

Sample No.	N1	N2	N3	N4	N5	N6	N7	N8
LL (%)	24.4	35.7	43.1	57.0	70.5	77	84.5	98
PL (%)	14.8	18.9	22.9	24.3	25.9	21.5	28.3	31.4
PI (%)	9.6	16.8	20.2	32.7	44.6	55.5	56.2	66.6
Soil class (USGS)	CL	CL	CL	CH	CH	CH	CH	CH
Specific Gravity (G_s)	2.70	2.74	2.76	2.76	2.75	2.68	2.74	2.78
Sand (%)	70	23	23	20	19	51	18	16
Silt (%)	18	64	63	60	56	30	57	47
Clay (%)	3	13	14	20	25	19	25	37

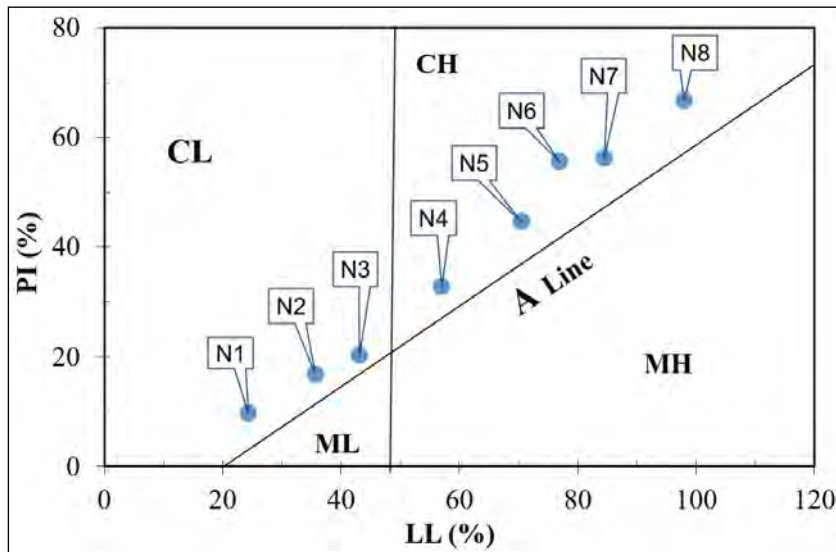


Figure 3- Distribution of soil samples according to Unified Soil Classification System (USCS).

4.2. Swelling Pressure

Swell tests were performed on partially saturated remolded soil specimens prior to the consolidation tests. The amount of swelling measured on those samples was used as the pre-load for the samples subjected to oedometer tests prior to inundation. Swelling pressures measured for all samples at different degrees of saturation are presented in Table 2.

The measured swelling pressures varied between 3 and 100 kPa. For the partially saturated samples, the relationships between swelling pressure and degree of saturation and plastic limit were also evaluated. It was observed that the swelling pressure values decreased

with increasing saturation degrees. This result is due to the high-water content of soils with high saturation degree and the low tendency of these soils to absorb water (Villar and Lloret, 2008, Elsharief et al., 2014, Keskin et al., 2023).

It was observed that the swelling pressure values increased with increasing plasticity. In addition, swelling behavior was not observed in the low plasticity samples (N1 and N2), while a small amount of swelling occurred in the low plasticity N3 sample.

4.3. Void Ratio - Effective Stress Relationships

As a result of the consolidation experiments, the void ratio-effective stress graphs were plotted, and the consolidation parameters of all the samples were determined using the Casagrande method (the details of this method may be found in Holtz and Kovacs, 1981). To compare the changes in consolidation parameters like that compression stress, re-compression stress and pre-consolidation stress, the void ratio-effective stress plots which show 4 different degrees of saturation (70%, 80%, 90%, and 100%) for each sample are presented in Figure 4. Detailed evaluations of these parameters are presented in the following sections.

4.4. Compression Index, c_c

Compression index values vary between 0.15-0.39 (Table 3). The degree of saturation versus the

Table 2- Swelling pressures for all soil samples (kPa).

No.	Degrees of Saturation, S_r			
	70%	80%	90%	100%
N1	-	-	-	-
N2	-	-	-	-
N3	10	3	-	-
N4	10	8	3	-
N5	25	20	10	-
N6	28	25	18	-
N7	65	35	30	-
N8	100	80	65	-

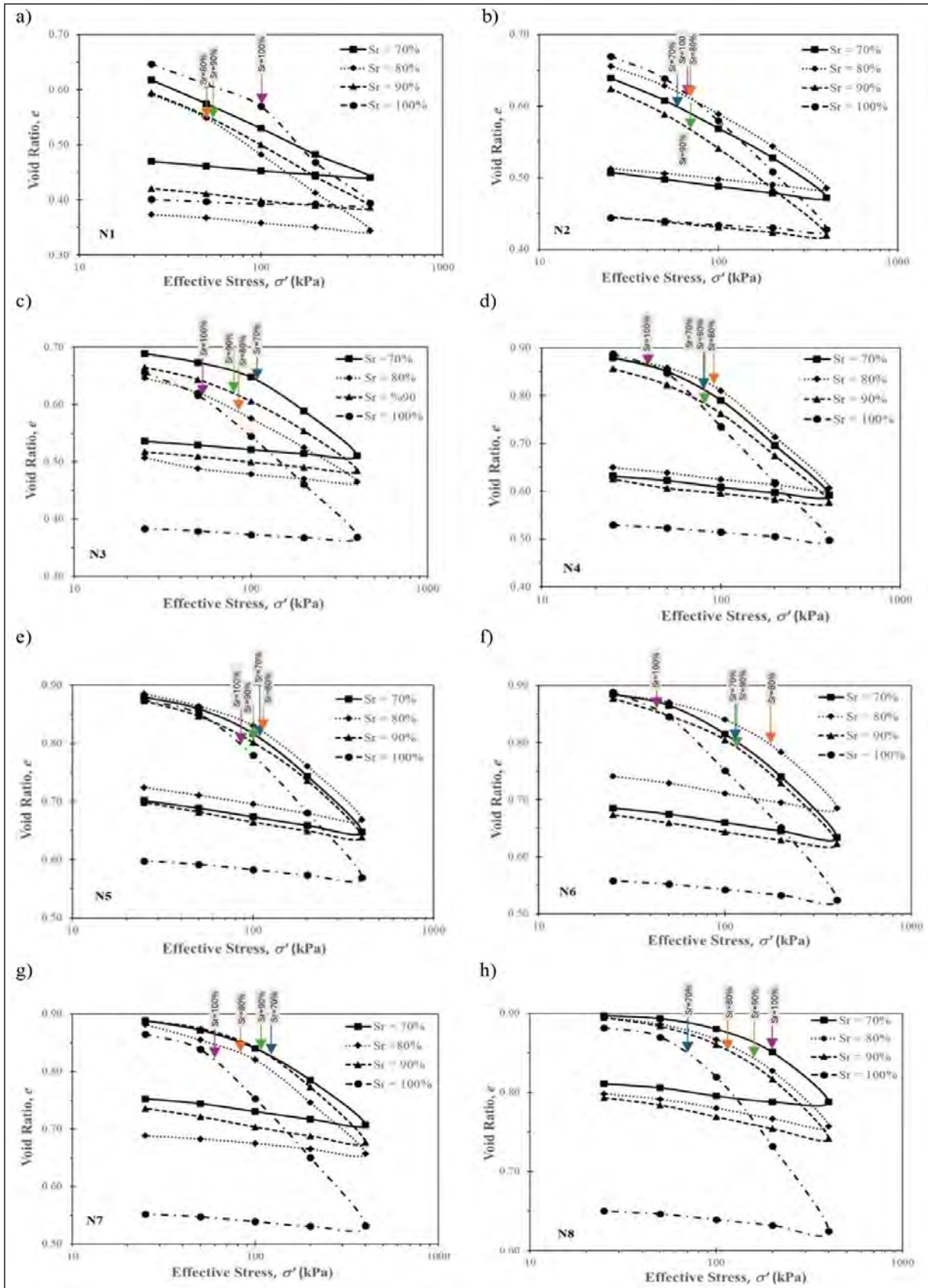


Figure 4- Oedometer test results for 8 soil samples conducted at 4 different saturation ratios for a) Sample N1, b) Sample N2, c) Sample N3, d) Sample N4, e) Sample N5, f) Sample N6, g) Sample N7 and h) Sample N8.

Table 3- The list of compression indices (c_c), recompression indices (c_r) and preconsolidation pressures (σ'_p) obtained from oedometer tests conducted on specimens of varying degrees of saturation (S_r).

No.	LL (%)	S_r	c_c	c_r	σ'_p (kPa)
N1	24	0.7	0.15	0.023	-
N1	24	0.8	0.23	0.024	50
N1	24	0.9	0.19	0.028	55
N1	24	1.0	0.29	0.006	100
N2	36	0.7	0.16	0.029	60
N2	36	0.8	0.17	0.022	70
N2	36	0.9	0.20	0.021	68
N2	36	1.0	0.25	0.013	70
N3	43	0.7	0.23	0.021	110
N3	43	0.8	0.18	0.035	85
N3	43	0.9	0.20	0.027	80
N3	43	1.0	0.29	0.012	56
N4	57	0.7	0.33	0.033	81
N4	57	0.8	0.34	0.036	90
N4	57	0.9	0.31	0.040	82
N4	57	1.0	0.39	0.026	44
N5	71	0.7	0.28	0.045	110
N5	71	0.8	0.27	0.046	115
N5	71	0.9	0.27	0.049	100
N5	71	1.0	0.35	0.024	82
N6	77	0.7	0.30	0.043	122
N6	77	0.8	0.26	0.047	180
N6	77	0.9	0.30	0.042	120
N6	77	1.0	0.38	0.028	57
N7	85	0.7	0.22	0.038	130
N7	85	0.8	0.27	0.026	82
N7	85	0.9	0.27	0.049	120
N7	85	1.0	0.37	0.017	58
N8	98	0.7	0.15	0.019	200
N8	98	0.8	0.18	0.034	170
N8	98	0.9	0.20	0.043	140
N8	98	1.0	0.32	0.021	98

compression index graphs of all the samples are presented in Figure 5.

It was determined that there were positive correlations between increasing degrees of saturation and compression index, with coefficients of determination ranging from $R^2=0.32$ to $R^2=0.94$. While evaluating the compression index versus degree of saturation ($S_r - c_c$) relationships of the samples,

plasticity was also considered. It was observed that the coefficients of determination of the relationships between S_r and c_c were different in each sample. The reason for this difference is due to the different PI values of the samples. Since the density values are varying in a very narrow range, the relation between the cc and the density has not been evaluated. Azzouz et al. (1976), Herrero (1983) and Kalantary and Kordnaeij (2012) revealed that LL and e_0 are the most effective parameters on c_c . Wroth and Wood (1978) and Mesri et al. (1975) revealed that the compressibility and compression index of clays are mainly affected by type of clay minerals, clay content, plastic limit and liquid limit. When the $S_r - c_c$ relationships are evaluated by considering the PI the determination coefficients of $c_c - S_r$ relationships for N1 and N2 samples with PI values of 9.6% and 16.8% are quite high, and there are positive linear relationships between c_c and S_r . However, it is seen that the $c_c - S_r$ relationship for the sample N3 with a PI value of 20.2% is still positive, but the coefficient of determination is quite low ($R^2 = 0.32$). Thus, although the PI for these three samples increased, the correlation coefficients of the $S_r - c_c$ relationships first increased and then decreased. When the $S_r - c_c$ relationships of other five samples with higher PI values were examined, it was observed that the determination coefficients of the $S_r - c_c$ relationships increased from 0.34 to 0.81, as the PI values increased from 32.7% to 66.6%. It was thought that the soil group (CL and CH) should also be considered while evaluating the samples with these different PI values. Accordingly, while there was inconsistency in the $S_r - c_c$ relationships for the first three samples in the CL soil group, the significance of the relations increased as the PI increased for the five samples in the other CH soil group. In addition to considering the PI effect when examining the $S_r - c_c$ relationships, differences between the partially saturated samples ($S_r = 70\%$, 80% and 90%) and the fully saturated samples were also observed. When the graphs in Figure 5 are examined, it is observed that the c_c values for all PI values also increased as the saturation increased until it reached 90%. However, c_c increased quite significantly in 100% saturated samples. These results clearly show that the c_c values of fully saturated and partially saturated samples can be quite different from each other.

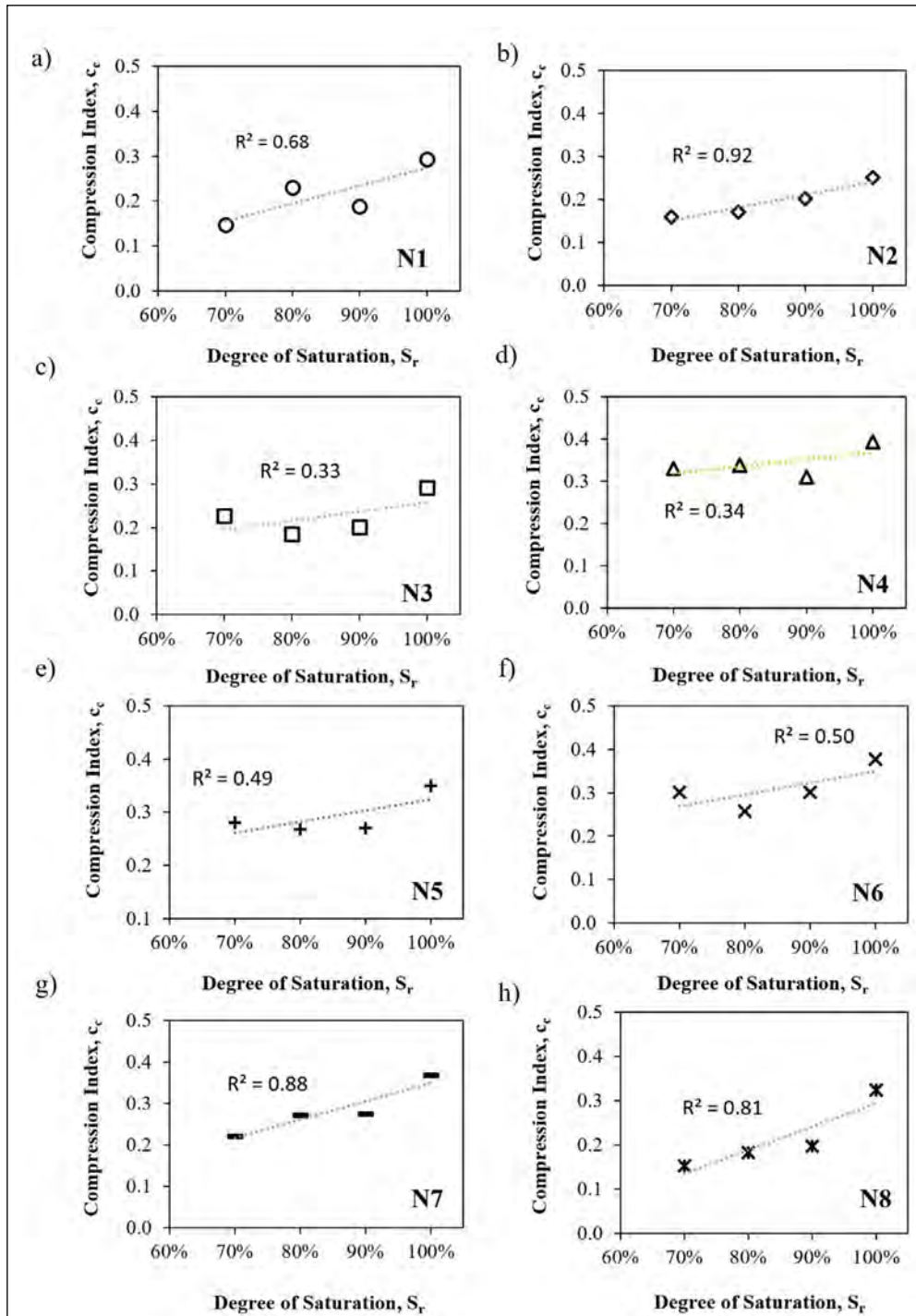


Figure 5- Graphs illustrating the relationship between the compression index and the degree of saturation for a) Sample N1, b) Sample N2, c) Sample N3, d) Sample N4, e) Sample N5, f) Sample N6, g) Sample N7 and h) Sample N8.

4.5. Re-compression Index, c_r

Recompression index values vary between 0.01 and 0.05 (Table 3). The saturation degree-recompression index graphs of all samples are presented in Figure 6.

It can be clearly seen from the graphs in Figure 6 that fully saturated ($S_r = 100\%$) and partially saturated ($S_r = 70\%$, 80% and 90%) samples significantly affect the c_r values in the recompression index-degree of saturation relationships ($c_r - S_r$). Accordingly, when

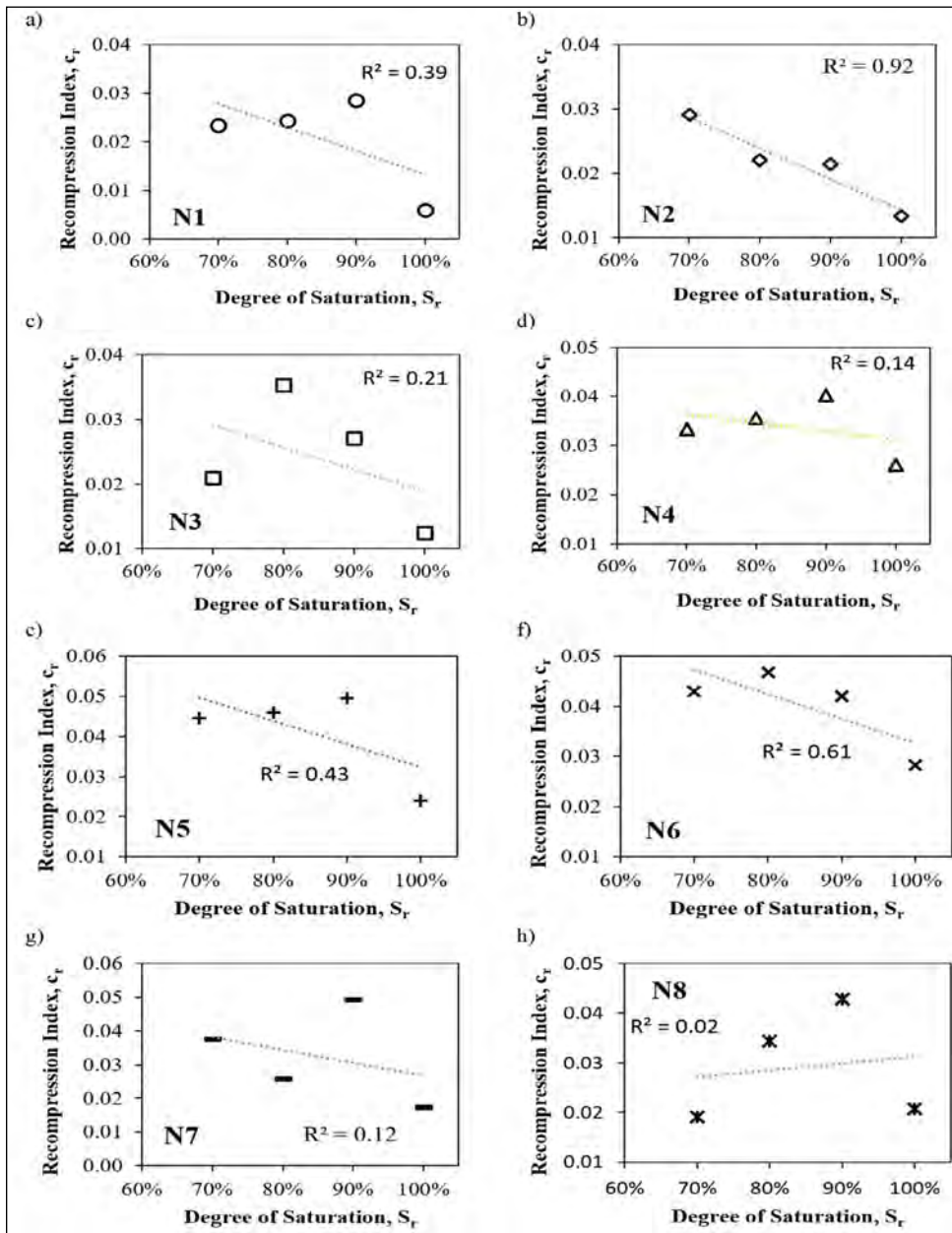


Figure 6- Graphs illustrate the relationship between the recompression index and the degree of saturation for a) Sample N1, b) Sample N2, c) Sample N3, d) Sample N4, e) Sample N5, f) Sample N6, g) Sample N7 and h) Sample N8.

the relationships of N1, N3, N4, N5 and N8 samples were examined, it was observed that the c_r values of the fully saturated sample were inconsistent with the c_r values of partially saturated samples. It was determined that the c_r value of the fully saturated sample decreased abruptly in comparison with the c_r value of the partially saturated samples in almost all cases. Thus, it has been understood that the c_r value of the saturated sample deteriorates the general trend

of the $c_r - S_r$ relationships and affects the R^2 values by significantly decreasing them. When $c_r - S_r$ relations were evaluated according to increasing PI values and soil group, it was determined that these two parameters did not have a significant effect on the relationships.

4.6. Pre-consolidation Stress, σ_p'

The pre-consolidation stress values of the samples vary between 44 and 200 kPa. The saturation degree

- pre-consolidation stress graphs of all samples are presented in Figure 7. Since the graph of $e\text{-}\log\sigma_p'$ at 70% saturation was linear in the N1 sample, the pre-consolidation stress could not be calculated. It was observed that the pre-consolidation stress of the saturated samples, especially those with high plasticity, was lower than the values of the partially saturated samples.

Except for samples N1, N2 and N6, the increasing degree of saturation causes the pre-consolidation stress to decrease. While the pre-consolidation stress values of partially saturated high plasticity samples (N5, N7 and N8) were very close to each other, a significant decrease was observed in the pre-consolidation stress of the fully saturated high plasticity samples (Figure 7).

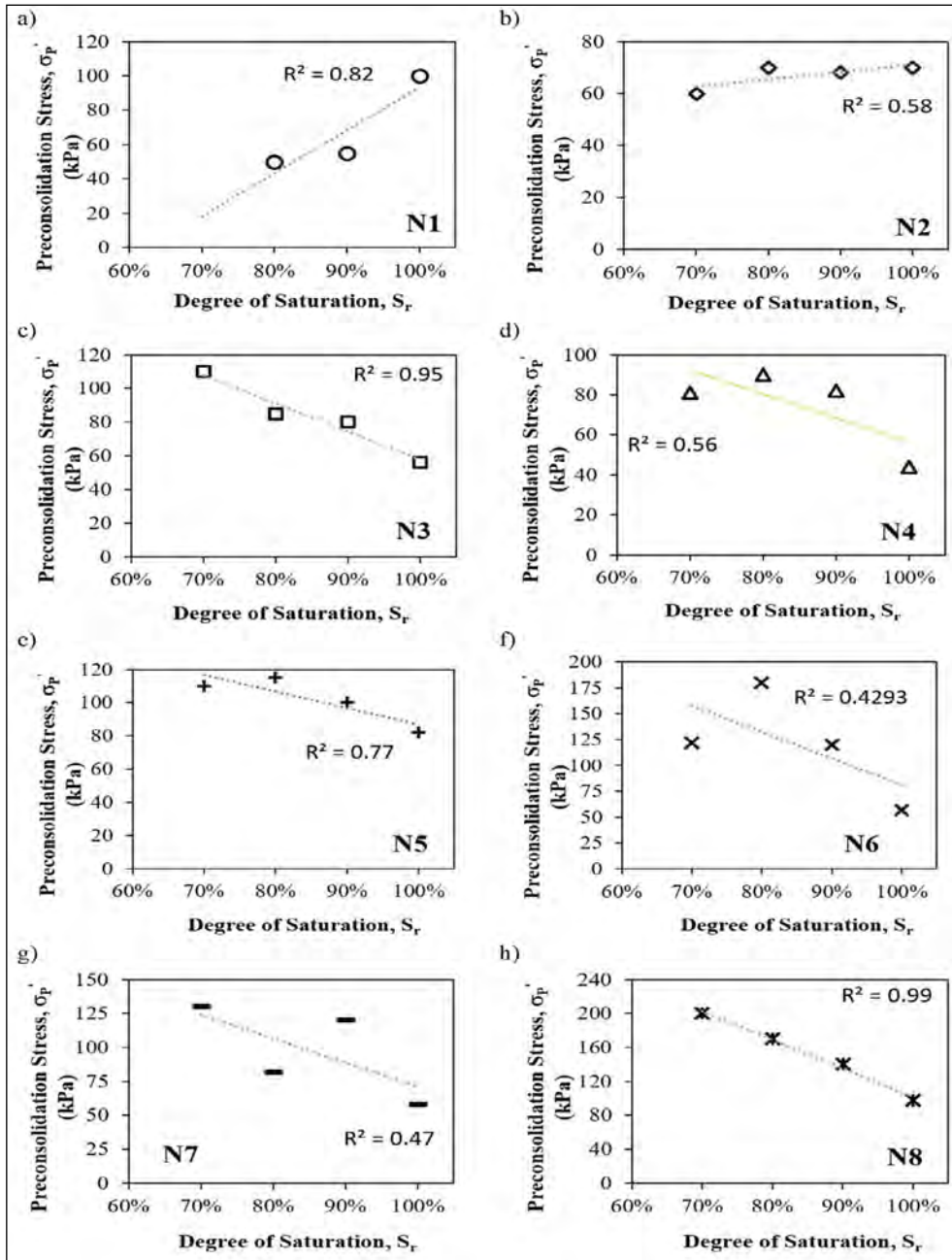


Figure 7- Graphs illustrating the relationship between the pre-consolidation pressure and the degree of saturation for a) Sample N1, b) Sample N2, c) Sample N3, d) Sample N4, e) Sample N5, f) Sample N6, g) Sample N7 and h) Sample N8.

4.7. Coefficient of Consolidation, c_v

The consolidation coefficient values of the samples vary between 1×10^{-4} – 14×10^{-4} cm²/s (Table 4). Saturation degree-consolidation coefficient graphs for 25, 50, 100, 200 and 400 kPa stresses applied at 70%, 80%, 90% and 100% saturation degrees of all the samples are presented in Figure 8.

When the relationship between the consolidation coefficient values and the effective stresses was evaluated it was observed that the coefficients of determination of this relationship are quite low, and it is concluded that there is no significant relationship between the saturation degree and the coefficient of consolidation. In addition, the relationship between the consolidation coefficient, the degree of saturation and the plastic limit were also evaluated. It was observed that the consolidation coefficient was not affected by the degree of saturation and the plasticity value of the samples. The relationships between the coefficient of consolidation and the degree of saturation, the effective stress and the plasticity index appear to be rather random.

5. Conclusions

The following conclusions were reached at the end of this investigation. The swelling pressures of all remolded samples at statically compacted at 70, 80, 90 and 100 percent degrees of saturation range from 3-100 kPa. It was observed that the swelling pressure decreases as the degree of saturation increases, as expected. The effect of soil plasticity on the swelling pressure was prominent as well. Swelling behavior was not observed in the partially saturated low plasticity samples in general. The aim of determining the swelling pressures for partially saturated soils was to ensure that swelling did not affect the results of oedometer tests.

The general tendency between the compression index (c_c) and the degree of saturation is that the compression index increases with the increasing degree of saturation. This conclusion is valid for both the low- and high-plasticity samples. The practical meaning of this conclusion is that the total settlement calculated for fine grained soils may differ depending on the degree of saturation.

Table 4- Coefficients of consolidation (x1000) determined through the oedometer tests conducted at different degrees of saturation.

No.	σ' (kPa)	70%	80%	90%	100%
N1	25	9.0	8.7	9.5	0.0
N1	50	8.7	8.4	9.2	0.0
N1	100	9.5	9.1	9.9	13.8
N1	200	10.1	9.7	10.8	11.3
N1	400	10.4	10.2	11.2	11.3
N2	25	9.1	6.5	4.1	0.0
N2	50	7.8	5.5	1.0	11.1
N2	100	7.5	6.0	5.3	11.7
N2	200	8.1	6.6	4.6	10.2
N2	400	8.3	7.6	7.3	9.0
N3	25	5.7	9.7	8.5	0.0
N3	50	9.4	6.9	7.7	5.0
N3	100	7.6	6.7	5.2	8.3
N3	200	9.3	8.1	6.8	6.7
N3	400	7.4	6.8	5.7	6.2
N4	25	4.2	10.3	6.0	0.0
N4	50	1.2	1.7	4.8	5.5
N4	100	6.6	7.8	5.3	11.3
N4	200	7.1	5.3	6.0	5.2
N4	400	3.7	1.8	4.6	3.4
N5	25	4.7	7.3	7.7	0.0
N5	50	5.4	1.1	3.6	1.7
N5	100	5.0	7.5	4.9	8.3
N5	200	6.8	7.3	4.6	6.7
N5	400	6.4	6.2	3.5	5.8
N6	25	0.0	6.4	5.0	0.0
N6	50	0.0		7.8	0.0
N6	100	6.4	6.8	4.2	8.3
N6	200	8.0	7.8	7.0	5.0
N6	400	6.6	7.1	6.2	3.7
N7	25	7.2	7.9	0.0	0.0
N7	50	0.0	7.6	0.0	7.1
N7	100	5.1	4.2	1.1	8.1
N7	200	5.1	5.4	7.5	4.9
N7	400	5.3	4.9	5.5	2.5
N8	25	0.0	3.6	6.0	0.0
N8	50	1.9	7.7	2.2	0.0
N8	100	2.2	0.0	2.6	0.0
N8	200	2.1	1.0	2.5	3.7
N8	400	4.4	7.4	5.3	1.6

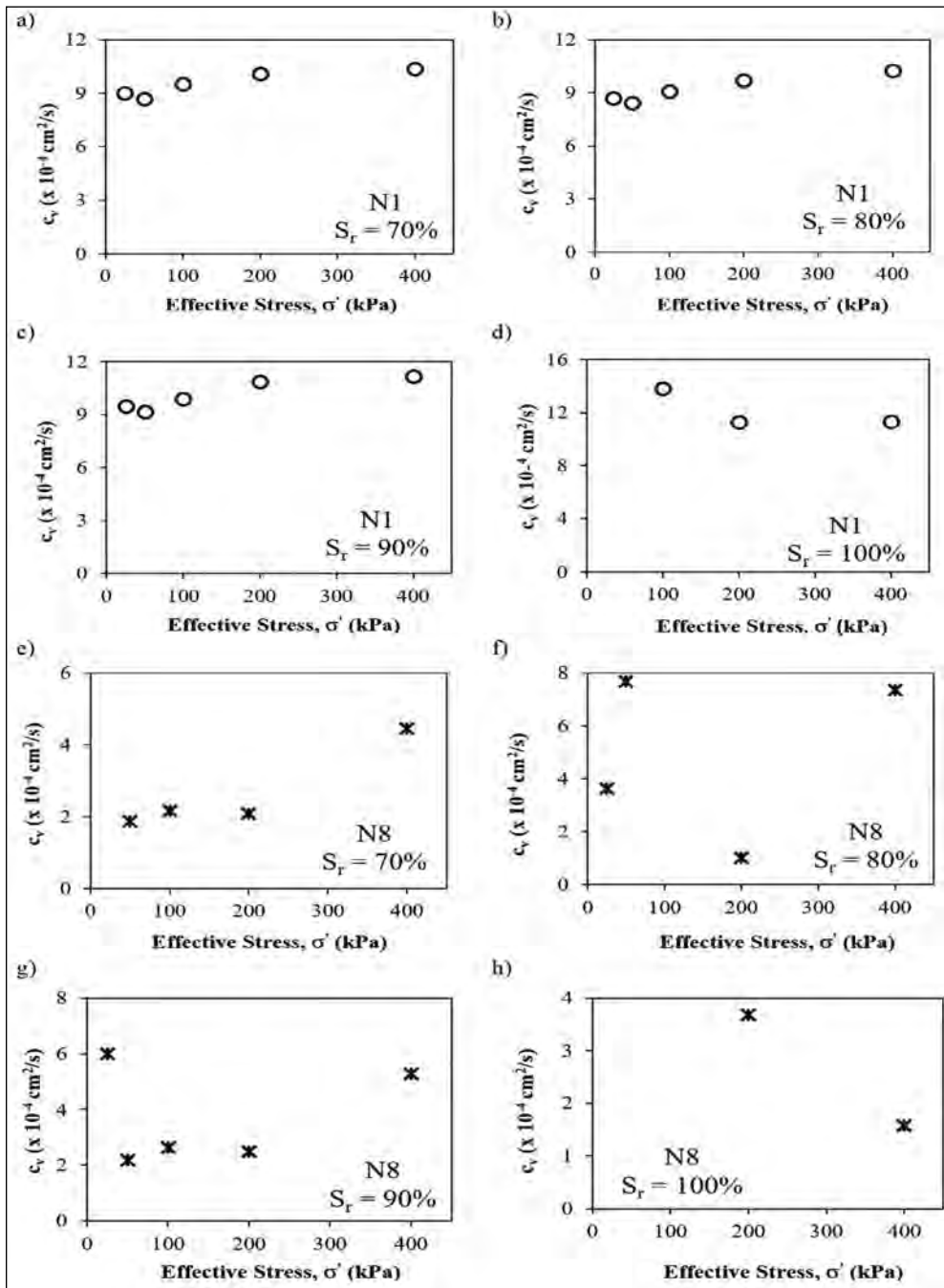


Figure 8- Consolidation coefficient versus effective stress plots for the selected two samples (i.e., N1 and N8).

Regarding the dependence of the re-compression index (c_r) on the degree of saturation, no conclusive statement can be made because of the inconsistent pattern between those two parameters while the general picture portrays that c_r tend to decrease with the increasing degree of saturation in contrary to the case of the compression index. The plasticity index did not appear to affect the c_r in general. Increasing

the number of soil samples may help asserting a more conclusive statement for this correlation.

Excluding the two samples with the lowest plasticity (i.e., N1, and N2), the pre-consolidation pressure (σ'_p) appears to decrease as a function of the increasing degree of saturation. In general, high-plasticity samples exhibit higher pre-consolidation

pressures than do the low-plasticity soils at similar degrees of saturation.

There was no systematical pattern between the coefficient of consolidation at varying effective stresses and the degree of saturation. And alike, no conclusive statement can be asserted for the dependence of the coefficient of consolidation on soil plasticity.

The overall conclusion is that all the consolidation parameters considered herein are affected somehow by the degree of saturation of remolded soil samples. This investigation was performed on laboratory prepared identical samples. Further investigation using natural soil deposits would help better handling the settlements calculations on fine-grained soils.

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Soil remediation at coastal plains with the help of DC Resistivity Method

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Research Article

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ABSTRACT

The coastline of the Dardanos, a district of Çanakkale, Türkiye, suffers from saltwater intrusion due to excessive extraction of groundwater for domestic usage and also agricultural activities. Thus, the salinity level increased, and much of the land became unusable. The electrokinetic remediation method was employed to reduce the salinity level in the soil samples in laboratory conditions. The sample used in remediation is silty agricultural soil, with pH value and electrical conductivity (EC) of which are 8.33 and 1282 µS/cm respectively. In the lab-scale experiments, three different types of electrodes, aluminum, copper, and galvanized steel, were used in the tests. For each type of metal, electrode pairs were placed in the soil that was filled in a plastic container. Current variation was monitored while 1 VDC/cm was applied to electrodes. Average electrical conductivity reduces to 13.5%. As a side effect, all electrodes suffered from heavy corrosion which may be prevented by using anti-corrosion additives to reduce damage for future applications.

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1. Introduction

The coastal areas accommodate the majority (~70%) of the world's population for residential and agricultural activities (Cohen, 2006; Bear and Cheng, 2010). Currently, shoreline settlements are facing a scarcity of water supply due to seawater intrusion (Pulido-Leboeuf, 2004; Abarca Cameo, 2006; Abd-Elaty et al., 2022). Excessive pumping of water from wells due to water needs in industrial activities and residential usage reduces the slope of the water table towards the sea or the water level falls below sea level. In either case, seawater rushes into freshwater aquifers. This invasion reaches a drastic level if the freshwater flow toward the shoreline is interrupted even for a relatively short period. In addition to the replacement of the saltwater and freshwater interface, pumping out of groundwater in coastal

areas excessively together with the usage of chemicals (fertilizers, etc.) to increase productivity causes some of the environmental problems and agricultural lands to become redundant.

Various types of soil remediation processes aim to eliminate contaminants from the soil. Traditional and novel soil remediation techniques are subject to various research (Sheoran et al., 1989; Luo et al., 2016; Karaca et al., 2017; 2018; 2022). Electrokinetic soil remediation has attracted increased interest (Acar and Alshawabkeh, 1993; Zelina and Rusling, 1999; Karaca et al., 2019; Bessaim et al., 2020; Özentürk et al., 2021; Karaca et al., 2022; Özentürk, 2022; Karaca et al., 2023; Taneja et al., 2023a; Taneja et al., 2024; Özentürk et al., 2024). The electrokinetic remediation processes employ the direct current (DC) and create an electric field in the geological unit. This

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is done by placing metal electrodes into the soil to be decontaminated. Then, approximately 50 to 150 V is applied to electrodes to create an adequate current to mobilize the ions in the soil.

Electrokinetic remediation (EKR) works with several different processes. Depending on the specific properties of the pollutants, each process acts individually or in combination to remove the target chemicals from soil. Electromigration, electrophoresis, and electro-osmosis transport the target pollutant to a location for removal (Reddy and Cameselle, 2009; Brusseau and Maier, 2014).

In this study, a bench-scale electrokinetic treatment trial was conducted to reduce soil salinity in the shoreline of the Çanakkale strait. The soil sample was taken from the Çanakkale-Dardanos Campus (CDC), northwest Türkiye (Figure 1). Alluvium stretches along the shoreline and overlays sandstone and mudstone units. Excessive pumping of water from wells with various depths in a relatively small area resulted in saline water intrusion. For testing purposes, we conducted an inter-disciplinary approach; first, we mapped out geological units of the target area and located geological units prone to be polluted by seawater. Then, we conducted a geophysical survey to obtain the resistivity distribution of the area. Based on the result we chose the place to take soil samples, and then an electrokinetic remediation study was carried out.

The outline objectives of this study are as follows;

- Map out possible geological units susceptible to pollution
- Execute a geophysical survey to map out salinization levels in the study area.
- Evaluate the success of electrokinetics for the remediation of salinized soil, and
- Examine the effectiveness of electrode types and configurations.

We recommend the following steps;

- The Direct Current Resistivity (DCR) method shall be used to reveal the geo-electrical image that helps to map the saline-rich geological units.

- Soil samples shall be collected according to resistivity model.
- Multi-electrode system for anodes and cathodes shall be employed for remediation.

Our primary findings indicate that upscaling the application from laboratory to field scale must be solved. Keeping the moisturizing at an adequate level and also removing the pollutants from the system immediately are vital for protecting the electrodes. We will present our results in due course.

In the following sections, we outlined the DCR Method to map out the resistivity distribution of subsurface geology and salty area. This map is used as a reference to locate a sampling point for soil. Then we executed lab experiments. Our findings and discussion follow the experiment section.

2. Methodology

Following our objectives, the local geological map indicates that alluvium stretches along the shoreline and also dominates the CDC area. The unit presents inhomogeneous features. To locate the sampling points, we utilize DCR method that is well known geophysical methods. Note that the electrical conductivity meters are in use for agricultural lands (e.g. McNeill, 1992; Corwin and Lesch, 2005; Kühn et al., 2009; Lech et al., 2016) and target the top-most layer of soil (depth < 1 meter). We employed DCR method to obtain pseudo three-dimensional (3D) resistivity distribution of survey area. In this approach, we are able to map the extension of the saline-rich units in x,y and z (depth) directions, in turn, we can also calculate approximate volume that needs remediation.

2.1. Direct Current Resistivity Method

DCR method is a geophysical method that was first suggested by Schlumberger (1920). Initially, the DCR method was used to delineate the saline and freshwater boundaries and has been subject to research since 1930. Swartz (1937; 1939) pioneered groundwater exploration and used the DCR on the Hawaiian Islands (Fretwell and Stewart, 1981; Ulugergerli, 2017). In simple terms, the DCR method requires injecting electrical current from two electrodes and measuring

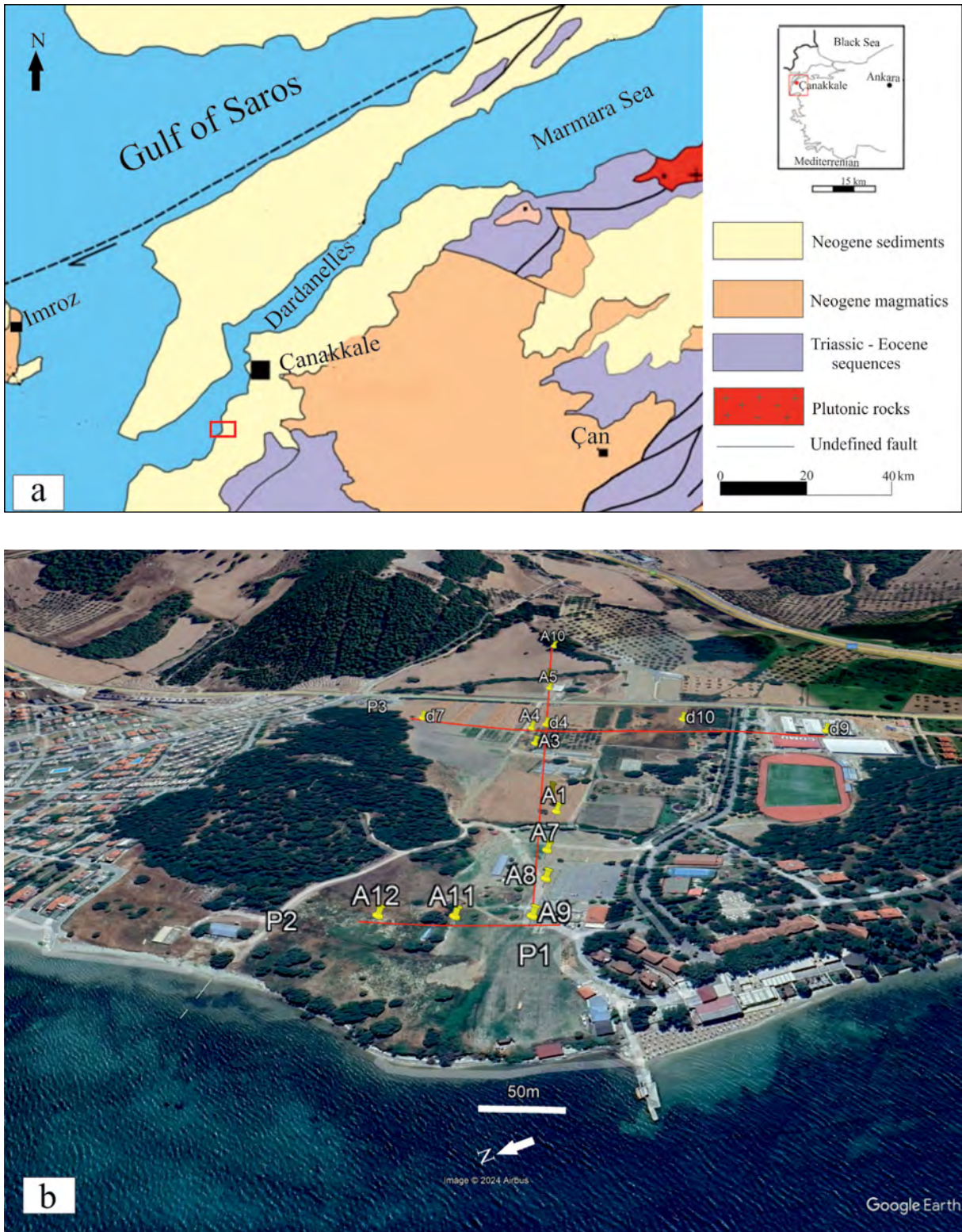


Figure 1- a) Geological setting of Biga peninsula (Okay and Satur, 2000) b) The study area and measurement stations for the DCR method (yellow pins).

voltage difference from the other two electrodes over the ground surface. The difference from EKR is that the duration of injecting current is relatively short, i.e., 10-20 sec.

The injected current and the measured potential difference are used to calculate apparent resistivities for each measurement point along the profiles (Telford et al., 1990). Apparent resistivities are not suitable for image resistivity distribution of geological stratum. Thus, a two-dimensional (2D) inversion scheme was chosen for the current study (Sasaki, 1992). This approach requires all stations to align along a profile and to be spatially dense enough (e.g. Ulugergerli, 2017; Ertekin and Ulugergerli, 2022). Then, an efficient and accurate 2D image of geo-electrical structures beneath the profile could be recovered employing 2D inversion. Note that Menke (1989), Meju (1994), Zhdanov (2002), and many other researchers provided details for both inversion and modeling for geophysical methods. Following the technological development, the DCR data can be gathered by using multi-electrode instruments, then this approach is called electrical resistivity tomography (Chambers et al., 2006; Candansayar, 2008; Wilkinson et al., 2012).

2.2. Soil Sampling

We collected the soil sample away from actively used roads. Removed the top dirt and got ~30 kg soil 10-20 cm below the surface. Sieved the soil using a 2 mm sieve and removed pebbles, organic materials, etc. before further steps.

In order to determine physico-chemical parameters, first, natural water content (ASTM D2216) was determined as 19.28%. Then the ratio of 1:5 (Soil: DI water) was used to determine salinity, pH and EC values. The samples were mixed for 6 hours at 180 rpm in the shaking device. At the end of 24 hours, the values measured with the Wtv multi 340i device are given in Table 1. As a result of the sieve analysis, the soil corresponded to the low plasticity clay (CL) class according to the Unified Soil Classification System (USCS).

Table 1- Chemical and physical properties of the sample taken from the study area.

Ground Class (USCS)	Water Content %	pH	Electrical conductivity (EC) $\mu\text{S}/\text{cm}$	Salinity (NaCl equivalent) %
CL	19.28	8.33	1282	0.45

2.3. Electrokinetic Method

During our experiments, a plastic container with dimensions of 40x58x15 cm was used to create a scaled sample for field application. Figure 2 shows the electrode configuration which we used for this study.

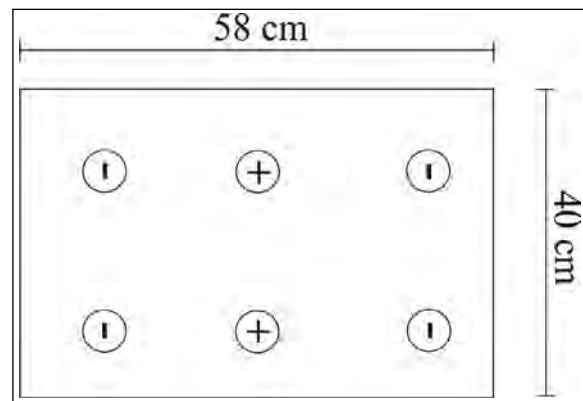


Figure 2- Electrode configuration; (+) anode (-) cathode electrodes.

The mud-cake containing soil and distilled water was placed into a plastic container (Figure 3). A wooden mallet was used to remove the air bubbles, if any, and make it tight. Note that the container must have a lid to prevent drying out during the remediation study.

Parameters such as the type of electrodes, content of electrolytes, and electrical current density affect the electrokinetic enhancement. Voltage set 1 VDC/cm considering the separation of the anode and cathode electrodes. Specifically, 11.6 V was applied because the distance between the electrodes was 11.6 cm. The current values measured during the experiment decreased over time. The experiments were terminated when the current values approached zero due to the displacement of ions in the soil and accumulation around the electrodes. At the end of the tests to determine the physicochemical parameters of the soil, soil samples were taken approximately 1 cm in front of the electrodes.

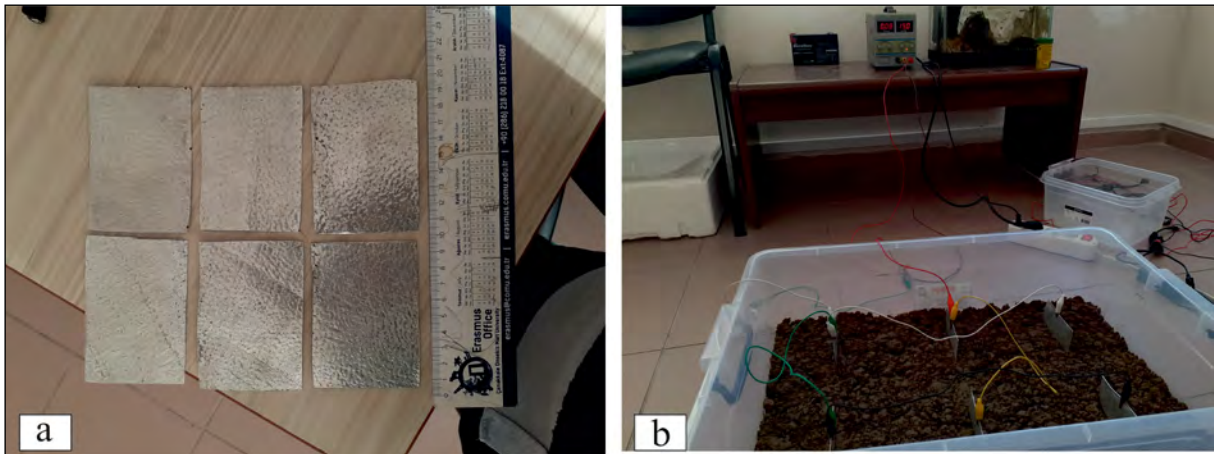


Figure 3- Electrokinetic experimental setup; a) preparation of electrodes, b) placement of electrodes.

3. Result and Discussion

3.1. DCR Survey

Considering the cost of the instruments, we used classical four electrode DCR equipment and we collected data using a vertical electrical sounding technique along lines and we combined and inverted those data sets using a 2D inversion algorithm.

As an example, the 2D resistivity-depth section of Profile 1, obtained from 2D inversion, is given in Figure 4. The agricultural soil (~ 10 ohm.m) covers the surface along the profile. Then, the existence of a low resistivity (< 5 ohm.m) unit points out seawater intrusion. The main geological unit presents discontinuities along the profile (dark blue colored unit).

3.2. DCR Survey Results

In order to define soil sampling location, the 2D resistivity models are combined and the pseudo-3D resistivity model is presented as a fence diagram (Figure 5). While deeper parts let us evaluate the inland limits of seawater intrusion. The shallow part of the fence diagram pointed out the contaminated soils. Considering the low resistivity (< 5 ohm.m) zone near the shoreline, we sample the soil between A9 and A11 (see also Figure 1).

A multi-dimensional evaluation of the DCR method pointed out the location requires remediation. Note that, if available, the multi-electrode DCR

systems can speed up the survey (Osinowo et al., 2018). Unfortunately, the exploration depth and may become insufficient if the land is surrounded by inaccessible lands. If it is the case electromagnetic method can be employed to map out the saline zones (e.g. Lesch et al., 1995; Çoban et al., 2017).

3.3. EKR Application and Findings

In the soil, the anions Cl^- , NaNO_3^- , NaO_3^- , SO_4^{2-} and cations consisting of Ca^{2+} , K^+ , Na^+ and Mg^+ form compounds and cause salinity (Kim et al., 2011). Depending on their charge, the contaminants and mobile ions present carry the electrical current from the anode to the cathode at the beginning of the test, causing a large current density peak after some time. The decrease following this peak is explained by the increase in electrical resistance of precipitates formed as a result of oxidation-reduction, electrolysis, dissolution and precipitation reactions during purification. Calcium ion production around the anode causes the salinity to increase and this value decreases due to calcium hydroxide precipitation at the cathode (Jayasekera and Hall, 2007).

The experiment conducted with copper electrodes was completed at the end of the 6th day when the current decreased to unmeasurable values. The experiment with the galvanized steel electrode was terminated on the 8th day under a 10 V constant current, and the one with the aluminum plate by applying 19 V current on the 15th day (Figure 6).

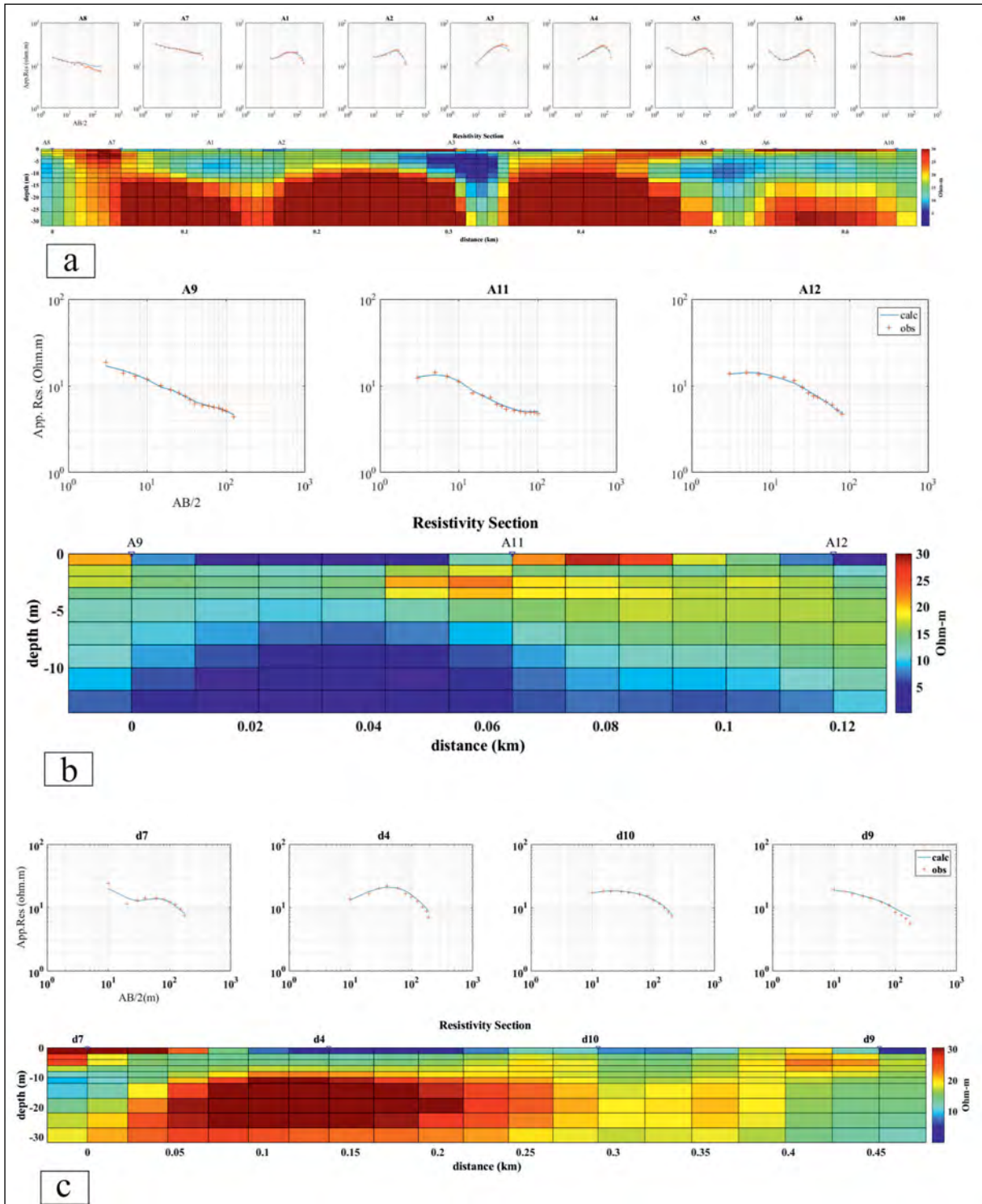


Figure 4- 2D resistivity models obtained from 2D inversion of DCR data. a) Profile 1, b) Profile 2, and c) Profile 3. The top graphs on each panel are apparent resistivity curves of observed (+) and calculated (solid lines) data.

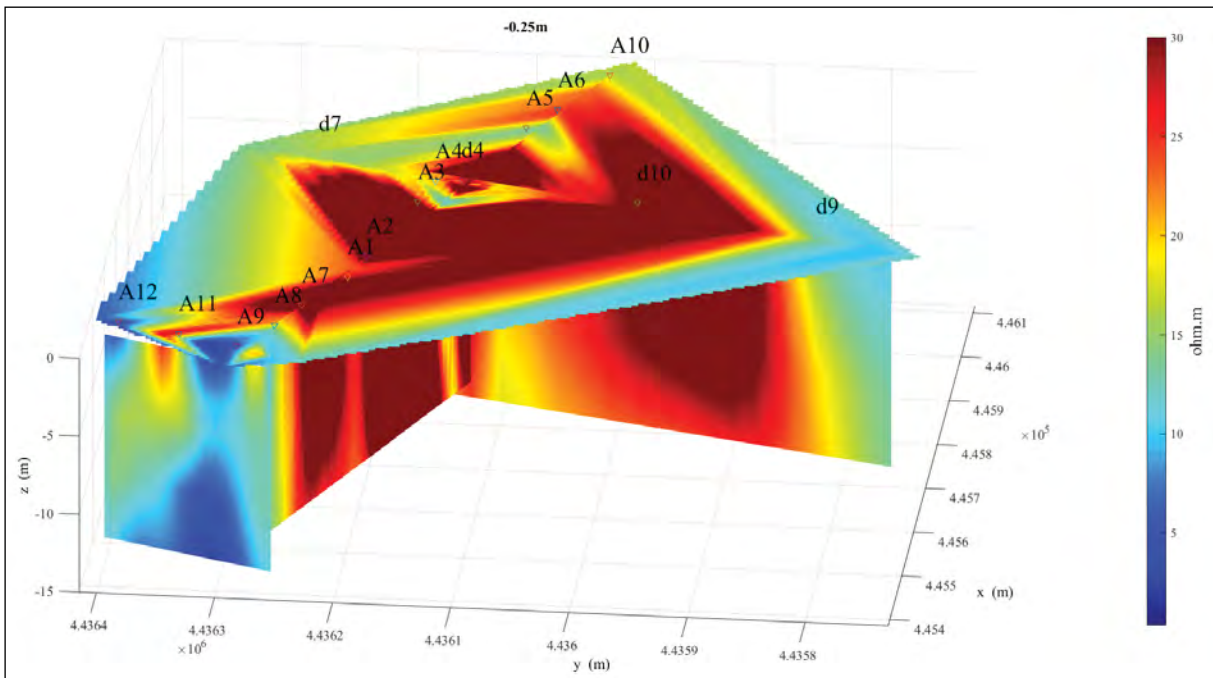


Figure 5- Depth - resistivity distribution at the surface and along the profiles. Resistivity values were extracted from all 2D inversion results.

Table 2 shows the parameters of the soil at the end of the electrokinetic experiments. pH differences at the electrodes are related to the electrolysis of water. By electrolysis of water, it produces O^2 and H^+ ions at the anode and H^2 and OH^- ions at the cathode. Therefore, the pH of the soil near the anode decreases and the pH around the cathode increases. Therefore, acidic pH measurements at the anode and alkaline pH measurements at the cathode are typical. At the same time, electrical conductivity (EC) decreased at the cathode, associated with sodium ion production at the anode. Consequently, it was observed that salinity

(NaCl equivalent) was intense, and the EC value was high in the anode region Table 2.

pH differences at the electrodes are related to the electrolysis of water. The oxygen and hydrogen ions produced at the anode create an acidic environment in this region, and the hydrogen and hydroxide ions produced at the cathode create a basic environment. In other words, the pH value is low in the anode region and high in the cathode region. At the same time, the difference in soil moisture content related to the electrolysis of water can be explained. A similar situation can be attributed to the corrosion of anodes

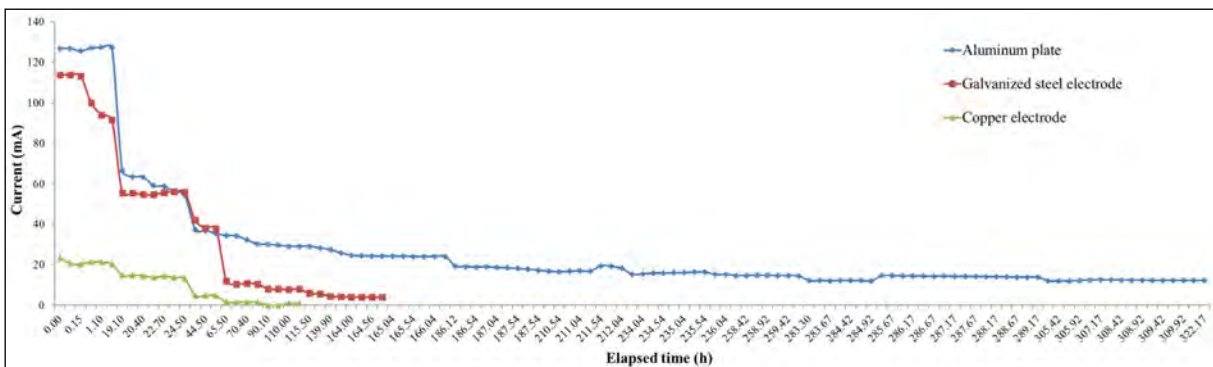


Figure 6- Variation of current intensity during the experiments.

Table 2- Moisture content, pH, EC, and salinity values of the soil after treatment.

End of treatment (average)	Copper electrode		Galvanized steel electrode		Aluminum plate	
	Anode	Cathode	Anode	Cathode	Anode	Cathode
Moisture content (%)	13.62	17.21	14.28	16.69	12.97	24.58
pH	6.21	9.50	6.53	8.59	6.40	9.78
EC ($\mu\text{S}/\text{cm}$)	328	146	320	178	4050	540
Salinity (%)	0.5	0.2	0.5	0.3	2.8	0.1

by oxidation of water (Barker et al., 2004). In addition, it can be said that other electrochemical reactions during treatment may also be effective. Although salinity removal was achieved in the study, corrosion occurred on the electrodes (Figure 7). In some studies, conducted for the remediation of heavy metals, it has been stated that since graphite oxidizes more easily at higher voltages, stainless steel may be used to conduct electric current at higher voltage with minimum corrosion effect and lower cost (Taneja et al., 2023b). Interruptions in the power supply and dryness of the soil affected the efficiency of the method. Thus, the added external liquid (pure water) restores humidity in the soil.

To reduce the electrode damage, pulsed current, instead of constant direct current may be applied. In this approach, the electric current is interrupted at regular intervals during the experiment. Thus, energy

savings will also be achieved as an important factor to consider for practical applications of EKR.

4. Conclusion

In this study, the 2D inversion results of DCR data were the keys to locating the polluted area. 2D resistivity models provided information down to 30 m of the study area. Also, the treatment efficiency of the electrodes used in the remediation of polluted soils by the EKR method and the corrosion, which occurred during the experiment, and is one of the important problems of this method, were investigated.

The experiments were conducted on saline soils and 1 V/cm DC voltage was applied to the soil during the experiments. The experiments conducted with copper, galvanized steel electrodes, and aluminum plates were completed after 6, 8, and 15 days, respectively.



Figure 7- a) Corroded copper electrodes, b) Corroded galvanized steel electrodes, c) Corroded aluminum plate electrodes.

The presence of water controls electrokinetic progress. The electrolysis of water and the ion release in the electrodes, which begins with the application of electric current, continues with the movement of existing and mobile ions in the soil to the electrodes. In the study conducted, this situation, which initially caused the current values to reach the upper values, ended with a decrease in the current values due to the transfer, accumulation and decrease in the concentration of existing ions in the process.

In EKR works, current is transferred to the soil through electrodes. Factors such as pH changes, high heat values due to current and types of contaminants cause corrosion on the electrodes. This causes interruptions in the current and disruptions in the treatment work.

At the end of the EKR, the pH values of the soils decreased slightly at the anode and increased at the cathode in all three experiments. Again, in all experiments, a clear decrease in EC was observed due to the removal of ions by the electrokinetic method, but the decrease at the cathode region was more remarkable due to the alkaline pH recorded.

Electric current applied directly to moist soil results in the electrolysis of water. O_2 and H^+ ions released at the anode result in a decrease in the pH value in this region, and the presence of H_2 and OH^- ions in the cathode results in an increase in the pH value in the region. The change in pH values causes this ion production in the anode region to react with soil components that dissolve carbonates and other basic salts and release metals. Similarly, on the cathode side, hydroxide, which is associated with high pH, provides the environment for the fixation of ionic metals. Therefore, this change in pH values affected the electrical conductivity of the soil, the development of electric current density during the experiment, and the distribution of ionic species in the soil after the electrokinetic treatment.

Since the corrosion of the metallic electrodes used in the study will lead to secondary metallic pollution in the soil due to the damage caused, thus, metal-based electrodes should not be used for treatment. Considering pH values and damage to the electrodes,

interrupting the electric current at regular intervals is one of the methods used to reduce high temperature and corrosion intensity in the soil. Using anti-corrosion additives can reduce damage for future applications.

Overall, the outcome indicates that EKR is well-suited to reduce the salinity level in soil. The success of the applicability is site-dependent, thus the electrode material and the current to be applied should be chosen accordingly. In addition, as a further study, before and after the remediation of the area DC resistivity data can be collected to evaluate how successful the remediation is.

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Mineralogical examination of biomass ashes: a preliminary study for potassium enrichment and phosphoric acid (H₃PO₄) production

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Research Article

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Biomass Ash,
Langbeinite, Phosphoric
Acid, Potassium Sulphate.

ABSTRACT

In this study, biomass fly ash was investigated and determined by XRD (XRay Diffractometer) to be predominantly langbeinite, apthitalite, sylvine, apatite and hydroxyapatite. An alkaline leaching study was carried out on the material in a glass reactor at l/s ratio 4 temperature 60°C at pH 12.85 and after drying was analyzed by ICP (Inducted couple plasma). The crystal structure composition of K₂SO₄ 72.28% and potassium content of 33.47% (w/w) was recovered. The elemental transformations in this leaching process were: K 74.16%, Mg 0.55% was found to be 0.8%. It was found that a minimum of 4.84 g H₂SO₄ per 100 g fly ash was required to precipitate the Ca(OH)₂ migrated into the liquid solution and the overall leaching efficiency was found to be 29.85%. In the alkaline leaching process, potassium was found to be easily recovered from each biomass ash. By adding 40 g H₂SO₄/100 g biomass waste remaining after leaching, a phosphorus recovery of 74.9% was observed. In this preliminary study, unsatisfactory results were obtained in obtaining the industry standard H₃PO₄. The reason for this is that the Al, Mg and K in the fly ash are subject to selective dissolution as a result of working at high pH in the alkaline leaching process.

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1. Introduction

Biomass ashes contain large differences in classification and structure compared to coal-based thermal power plant ashes. Currently, the ASTM C618-19 (2019) standard is used for this purpose. In order to classify the origin and structure of biomass ashes, the use of a triangular diagram based on the K₂O, CaO and SiO₂ w/w contents as a 3-phase diagram has been recommended (Zhai et al., 2021).

Biomass ashes can be accepted as secondary phosphorus and potassium sources due to their high phosphorus and potassium contents. In recent years,

the number of thermal power plants with different designs suitable for biomass combustion has increased rapidly. A new alternative material has emerged to meet the limited phosphate and potassium requirements due to the structure of these ashes. In the characterization study of biomass ashes by Çötelî and Karahan (2023), chemical analyses were carried out and it was found that the potassium in the ash was completely soluble in water and had a highly alkaline character.

Potassium is found in many igneous rocks such as feldspar (potassium aluminum silicate), KAlSi₃O₈ (leucite) and mica, KH₂Al₃ (SiO₄) three. As these

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rocks break down and dissolve, potassium mixes with the soil and water. Potassium chloride deposits are found in almost all salt deposits associated with sodium chloride. Some important potassium minerals are leucite, KAlSi_2O_6 ; glauconite (a complex silicoaluminate structure of varying composition); sylvine (KCl), carnallite ($\text{KCl}\cdot\text{MgCl}_2\cdot 6\text{H}_2\text{O}$), langbeinite ($\text{K}_2\text{SO}_4\cdot 2\text{MgSO}_4$) and polyhalite ($\text{K}_2\text{Ca}_2\text{Mg}(\text{SO}_4)_4\cdot 2(\text{H}_2\text{O})$) (Patnaik, 2002).

Although Krishnamurty et al. (2008) stated that potassium carbonate (K_2CO_3) is predominantly found in wood ash, the study of the calcination temperature in thermal power plants and the almost complete absence of carbon content in the ash suggest that it is present in different forms. In order to recover potassium, its mineralogical structure must be clearly identified. Currently, the biomass ash waste potential in our country is around 600 tonnes/day, which corresponds to 72 tonnes of K_2O /day. An alternative way to solve our country's potassium problem has been opened, mainly through good national management planning of existing material resources. Although some small private companies are still working on it, industrial adaptation and economic production cannot be achieved.

Although there is no sodium salt problem in our country in terms of reserves and production, there is no discovery and production of potassium ore and salt due to the geological structure of our country. A long time ago, especially in the 1980s, studies were carried out on the production of potassium chloride from İzmir Çamaltı Brine Salt Works, and although there were studies aimed at obtaining potassium from some clay minerals (such as potassium feldspar), studies were carried out to meet satisfactory consumption. ALKİM (2023) stated in its annual report that it uses imported potassium chloride and domestically produced crystalline sodium sulphate in the production of potassium sulphate. Russia and Belarus supply approximately 40% of the global potassium chloride market and are the countries closest to us logistically. The potassium sulphate capacity of the Alkim (Alkim Alkali Chemistry Company) - Dazkırı, Koralkim plants is 50,000 tonnes/year and K_2SO_4 production is done with H_2SO_4 via imported KCl . Until now, it has not been possible to produce enough different

potassium salts to meet the import requirements in terms of potassium mining. The country's industry meets all its potassium needs through imports.

Furthermore, if we look at the import data of TÜİK (Turkish statistical institution, 2022), it can be seen that the foreign exchange from the fertilizer sector has reached \$2.7 billion. Two of the main inputs to the fertilizer industry are the output of potash and phosphate mining.

In Burke (2021), ash management is important due to the increased use of biomass for energy production; burning unprocessed biomass in modern furnaces can produce small amounts of ash containing negligible persistent organic pollutants, and land application is possible due to low concentrations of contaminant metals. He noted that agricultural residue ashes contain high levels of potassium and useful phosphate, so could potentially be used as fertilizer. This highlighted the importance of recovering potassium from ash.

Although biomass has many uses in conventional agriculture, such as direct green manuring, composting and use for organic matter needs, small-scale thermal power plants for electricity generation have begun to grow rapidly as a result of the rapid increase in per capita energy consumption. The resulting fly ash has created an environmental and bio-waste and ash management challenge. Fly ash contents are never similar to coal based thermal power plant ashes and as an alternative secondary source of phosphorus and potassium for industrial waste recovery, studies on its use, recovery and product development have accelerated within the scientific framework.

In this study, the recovery of potassium compounds from biomass ashes from thermal power plants by dissolving them in water, their technological economisation, the outline of the process, their partial purification from impurities (such as Ca , Na) and their valorization in terms of mining as a 2nd stage industrial product (a mixture of K_2SO_4 , KCl , NaCl and Na_2SO_4) are planned. The remaining solid valorized waste was studied to obtain H_2SO_4 and H_3PO_4 . The aim was to develop a suitable process for economic production by determining potassium recovery and yield. The main objective is to obtain high value added products from

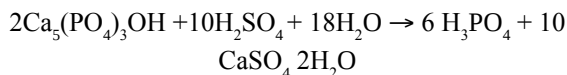
the biomass ashes consisting of these agricultural waste fertilizers with special status. A feasibility study for industrial economic production was carried out.

2. Literature Review

Weast and Astle (1980) stated that potassium is difficult to dissolve due to its crystal structures being bound to other minerals and that it occurs in nature mainly as sylvine, carnalite, langbeinite and polyhalite minerals and is found in small areas in Germany, New Mexico, California, Utah etc. Particularly large potash resources are found in Saskatchewan at a depth of 3000 ft. It is also found in sea water and in salt deposits.

Civelekoğlu et al. (1987) stated that the main minerals of potash salt production are K and Al silicates which are insoluble in water and that potash salts are refined from water soluble and potassium containing solutions by methods such as dissolving and cooling in hot (KCl, from sylvine) or decomposition (KCl, from carnallite) with NaCl and MgCl₂ salts and that the basis of this process is understandable 2-3 phase diagrams.

Krupa et al. (2008) also carried out a study on the production of phosphoric acid on bone meal ash, which has the mineral structure of hydroxyapatite.



Bone meal was subjected to calcination at 600°C. 43% H₃PO₄ solution was formed after reacting with the ash and adding unreacted jibs. They were reacted at 90-95°C and H₂SO₄ was added, resulting in a product that contained 45% H₃PO₄. The solid part was separated, and by adding steam to remove some water vapor, commercial phosphoric acid containing approximately 75% and 54% P₂O₅ was obtained (Figure 1).

Zhai (2022) stated that the common mineral phases are calcite (CaCO₃), fair calcocite [K₂Ca(CO₃)₂] and arcanite (K₂SO₄) based on the XRD raw spectra of different biomass ashes. All of the wood ash burnt in the laboratory is dominated by the calcite phase, with small amounts of Arcanite and hydroxyl apatite [Ca₅(PO₄)₃(OH)]. This ash contains some calcite, arcanite, larnite (Ca₂SiO₄) and polyhalite

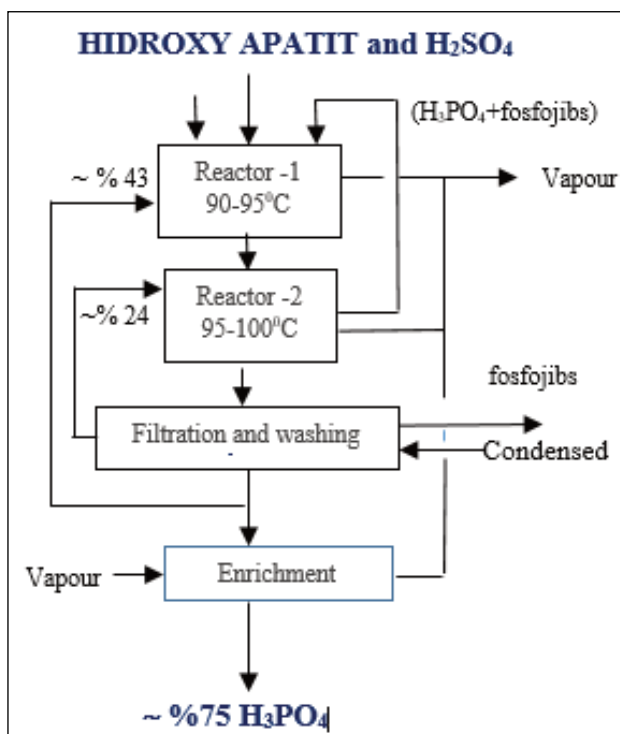
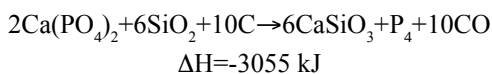


Figure 1- A brief representative description of the industrial production of phosphoric acid, from calcinated bones (Krupa et al., 2008).

[K₂Ca₂Mg(SO₄)₄·2H₂O]. In the example of industrial wood ash, quartz (SiO₂) and calcite are present. Lime (CaO) also contains some kalsilite (KAl₂SiO₄), gypsum (CaSO₄·2H₂O) and some periclase (MgO) and sodium, calcium silicate (Na₂Ca₃Si₂O₈). Archonite and sylvine (KCl) have also been identified. Although the rice husk ash sample also consists mainly of SiO₂, the main mineral phase of cristobalite contains less quartz, while in another sample it consists mostly of quartz, and in both samples, there is some amorphous content of sylvine, arcanite and less kalsilite (KHCO₃). Minerals detected in XRD analyses in biomass ash samples, depending on the ash source; arcanite (K₂SO₄), calcite (CaCO₃), cristobalite (SiO₂), fairchildite [K₂Ca(CO₃)₂], hydroxyapatite [Ca₅(PO₄)₃(OH)], calcilite (KAl₂SiO₄), kaolinite [Al₂Si₂O₅(OH)₄], larnite (Ca₂SiO₄), lime (CaO), periclase (MgO), polyhalite [K₂Ca₂Mg(SO₄)₄·2H₂O], silica (SiO₂), sodium calcium silicate (Na₂Ca₃Si₂O₈) and sylvine (KCl).

Regarding phosphorus recovery, Becker (1989) explained the fundamentals of phosphorus recovery economics, the differences between processes, how wet and dry phosphoric acid production differs, and how thermal processes vary based on the material structure. Although there are project studies called RecoPhos using a carbon-lime mixture at 1300-1900°C thermally, it is not thought to be as economical as the wet process. It is difficult to work due to the difficulty in reaching the temperature. Trial production is still ongoing, and large-scale production seems only possible by obtaining H₃PO₄ through the wet process. It seems to be an alternative way to obtain high purity phosphoric acid with low heavy metal contamination. This can be especially useful in food applications. Additionally, biomass ashes are a cleaner material than natural waste sludge and rock phosphate deposits. In this case, the mineralogical structure of the material is key. The process called Recophos is not actually a new process, but is a modified version of the electric furnace process (Austin, 1985), which was developed as an alternative for use on rock phosphates in the 1920's in case of cheap electricity supply.



Donatello and Cheeseman (2010) gave an empirical formula for P extraction from waste sludge ashes, as in Franz (2008).

Amount of g H₂SO₄ per 100 g material:

$$(1.479 \cdot \text{CaO} + 0.962 \cdot \text{Al}_2\text{O}_3 + 0.614 \cdot \text{Fe}_2\text{O}_3 + 2.433 \cdot \text{MgO} + 1.582 \cdot \text{Na}_2\text{O} + 1.041 \cdot \text{K}_2\text{O}) - (0.691 \cdot \text{P}_2\text{O}_5 + 1.225 \cdot \text{SO}_3 \%)$$

The empirical approach is actually similar to Nunn and Dee (1954). Provided that the liquid/solid ratio was 20, it was dissolved with H₂SO₄ solution at a concentration of 0.19 mol/l for 120 minutes, and then the liquid part of the filtered mixture was passed through cation exchange resin and analyzed for Zn, Fe, Mg, Ca, Al, P and SO₄²⁺. The final product had a concentration of approximately 1% H₃PO₄ and could reach 85% H₃PO₄ concentration by vacuum distillation.

Raupenstrauch (2015) explained the RecoPhos process for obtaining H₃PO₄ from industrial city biomass waste ashes. With 80% waste sludge ash (SSA) (8% P content) material at a feeding rate of 10kg/h, 10% C and 10% lime mixture, the P recovery in the melting furnace operating at 1900°C increased to 90%, and the heavy metals in the ash were eliminated. He explained that H₃PO₄ could be produced by extracting the gas phase in water.

Lijian et al. (2019) stated that biological wastes such as meat and bone meal and poultry manures are used as energy sources in industrial facilities, but the phosphorus in the biomass ashes remaining after combustion is thrown away without being used, and to encourage their use, biomass ashes from industries are highly alkaline with pH values as high as 13. The content is high in phosphorus and calcium. X-ray powder diffraction (XRD) reveals hydroxyapatite and potassium sodium calcium phosphate. Nearly 90% of phosphorus can be removed by acid extraction (H₂SO₄); optimized acid consumption is 3 (they determined that it was 2–5.3 mol H⁺/mol P). Nearly 90% of phosphorus can be removed by acid extraction (H₂SO₄); optimized acid consumption is 3 (they determined that it was 2–5.3 mol H⁺/mol P). Nearly 90% of phosphorus can be removed by acid extraction

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Gowariker et al. (2009) explained that the ash produced by burning wood is rich in potassium (in the form of K₂CO₃) and is useful for correcting the pH of acidic soils, is also explained the Mannheim process, which is the production of K₂SO₄ from KCl. This process is a 50% K₂O process in which KCl reacts with H₂SO₄, yielding excess KHSO₄, KCl and HCl as a gas.

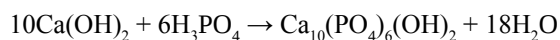
Schultz et al. (2000) described the production of potassium fertilizer from the natural complex salts kainite (KCl.MgSO₄.3H₂O) and langbeinite (K₂SO₄.2MgSO₄) and Carpathian poly-minerals. In the process, kainite is leached to form schonite (K₂SO₄.MgSO₄.6H₂O) and MgCl₂ is separated. He stated that schnite is separated into K₂SO₄ and MgSO₄ by leaching in the same way.

Wang et al. (2016), potassium recovery by leaching method on an ash sample containing 39.44 mg/g volatile matter, 158.92 mg/g fixed carbon, 51.64 mg/g K and 28.56 mg/g Mg. In the leaching process with H₂SO₄, the pH was adjusted to 6.1, the liquid/solid ratio was 3 and the temperature was 75°C. At the end of 30 minutes, the pH increased to nine and a K recovery efficiency of 86.4% was achieved. In his study, he observed that the solubility of potassium is independent of pH and that low pH leads to the formation of CaSO₄, which causes pH collapse. In addition, the world's potash resources are limited and potassium dolomite deposits (8.21%) could be an alternative. This process involves calcining the material ground below 74 microns with Na₂CO₃ at 840°C, leaching with water and CO₂ for 2 hours at a l/k ratio of 8-10, evaporation and crystallization, and K₂CO₃.

Akgül and Yoncacı (2021) gave different classifications on the classification of the thermal power plant producing fly ash and its classification is siliceous (S), silicocalcic (CS), ferrosilicic (FS) and ferrocalcic (FCS). In this classification, he also stated that they are categorized into three dominant tendencies such as high acid (HA), medium acid (MA) and low acid (LA) according to the sum of Si, Al, K, Ti and P oxides.

Li et al. (2015) found that aluminum and iron phosphates, which are difficult to use at high temperatures, react with lime (CaO) in the environment at different temperatures to the apatite form Ca₃(PO₄)₂, from which phosphorus recovery is easier. He explained that aluminum and iron are oxidized as Al₂O₃ and Fe₂O₃ and phosphorus is bound to Ca in the form of apatite.

Çağan (2014) stated that the general formula of apatite is X₁₀(TO₄)₆Z₂, and instead of X, Ca, Sr, Pb, Cd and Ba; P and As instead of T; OH, F, Cl can be entered instead of Z.



According to its reaction, it is possible to produce it in pure form. Hydroxyapatite has high biocompatibility; he stated that not only can the body easily accept it, but it can also create a suitable environment for the growth of bacteria, which are other biological structures, and can be used especially in dental implants.

Karabacak (2021) revealed some commercial processes for phosphorus recovery from waste sludge ash by listing the principles and effects/products. The material he works on is BUSKİ samples and the ash content is P₂O₅ 17.36-11.49% in two different samples; K₂O content is between 3.70-3.16 and used H₂SO₄ solution at different concentrations to transfer the phosphorus in the ash to the solution.

In KTÜ (2017) and Tor (1991) by adding liquid ammonia (NH₄OH or (NH₄)₂S) to a solution containing Fe²⁺, Fe³⁺, Al³⁺, Cr³⁺, Mn²⁺, Ni²⁺, Co²⁺, Ba²⁺, Ca²⁺, Mg²⁺, Na⁺, K⁺, It has been found that Al(OH)₃, Cr(OH)₃, Fe(OH)₃, MnS, NiS, CoS, FeS, ZnS precipitate while Ba²⁺, Ca²⁺, Mg²⁺, Na⁺ and K⁺ remain in solution.

Millsaps et al. (2021) determined the required H₃PO₄ ratio in a collaborative study involving acidification with H₃PO₄ for the production of phosphorus fertilizer from poultry manure, the total and water-soluble P₂O₅ content of the final product, and the granulation process. In the ideal study, 0.6 kmol H₃PO₄ per 100 kg ash was found. However, the reaction time is very short and it is stated that the granulation process of the paste material should be started immediately within a maximum of 4 minutes.

As stated by Bogusha et al. (2018), this is an increasing problem due to the rising number of biomass power plants in numerous countries. It is therefore essential to be comprehensively characterized biomass ashes, with particular focus on the elemental composition and solubility of nutrients (e.g., K and P) and contaminants. Furthermore, these residues can be utilised for soil nutrition. All biomass ashes studied were alkaline and exhibited high concentrations of P, K and Ca, which are essential for their potential utilisation. He stated that it contained the following: Phosphorus is present in: 1) Bottom ash contains apatite and other phosphates, including potassium hydrogen phosphate and potassium iron phosphate, which are produced when poultry litter is incinerated. 2) It is reported that this material originates from poultry litter incineration and contains potassium, sodium, calcium phosphate. Potassium is mainly present as sylvine, arcanite and some phosphates. Phosphorus, calcium and magnesium are difficult to leach with water, and recovery of potassium and phosphorus from these materials must be done carefully. It has been documented that this is a viable method.

Sof et al. (2021) and Çöteli and Karahan (2023) also studied the method of acidifying the material with H_2SO_4 in the production of PK and NPK organic and inorganic fertilizers in the fertilizer sector. The basis of the developed production method is the complete acidification of the phosphorus in the ash to make it soluble in water. The main purpose of the study is to use the existing material in the inorganic and organic fertilizer sector.

Çöteli and Karahan (2023) investigated the chemical composition of the ashes of the biomass thermal power plants installed in Turkey as a function of time and found that the ashes of the biomass thermal power plants had a chemical composition as shown in Table 1.

Puspita and Susanto (2021) also worked on the development of a new technique for the recovery of potassium salt from biomass ash extraction solution containing a mixture of KCl, NaCl, $MgCl_2$ and $CaCl_2$. The process involves partial evaporation of the solution, followed by the addition of ethanol

and further evaporation. In their study, they showed that KCl can be recovered in a different way than in traditional studies.

Table 1- A chemical characterization of Turkish biomass thermal power plant fly ashes (Çöteli and Karahan, 2023).

	Average%	Max %	Min %
Al_2O_3	4.75	16.97	2.88
CaO	16.05	19.24	10.57
Fe_2O_3	1.94	7.03	0.32
K_2O	12.75	27.0	2.94
MgO	6.56	11.50	1.19
Na_2O	6.47	27.38	4.79
P_2O_5	12.38	19.53	4.98
	mg/kg		
As	15	27	3
Cd	1	3	0
Co	8	25	3
Cr	121	995	16
Cu	432	1024	141
Pb	232	691	14
Zn	950	3997	100
Note:			
1-Ash samples have a highly alkaline character and the pH (1/10) is in the range of 11-13 depending on the sample.			
2-1 Valent cations and anions are completely soluble.			
3- P_2O_5 , it is 0.5-1.5% water-soluble, depending on the cation bounds to.			

3. Technological Studies

3.1. Material Used

The sample (fly ash) (Figure 2) obtained from the 20 MW installed capacity fluidised bed thermal



Figure 2- Biomass fly ash studied.

power plant located in Sakarya-Akyazı district has a particle size completely below 50 microns and the sample, which is believed to have been calcined (containing CaO, MgO, etc. material) due to the high temperature at the outlet of the plant, was placed in a closed package. The plant burns more than 50% of agricultural waste, in particular poultry waste and poultry manure.

3.2. Chemical, XRD and SEM (Scanning Electron Microscope) Analyses

The results of the ICP analysis are given in Table 2 after dissolution in aqua regia. It is a material with the status of the second source of potash and phosphate.

The XRD analyses were carried out on the Bruker-D-8 Advance, the values obtained from the XRD diffractogram were evaluated in the “HighScore Plus” software and the mineral structures contained therein were determined.

The powder sample to be examined on the FEI Inspect F50, FEG-SEM instrument was subjected to SEM-EDS (Elemental Analysis in scanning electron microscope) analysis after gold-palladium coating. The results of these analyses were SEM photographs and EDS graphs showing the elements contained.

The analyses of fly ash using SEM+EDS at the 1st point range, at the other 2 points and as an average are given in Table 3 as percentages. The ICP analyses were compared.

Table 2- Chemical composition of the studied fly ash material.

ELEMENT	VALUE	ELEMENT	VALUE	ELEMENT	VALUE
Al %	2.00	As ppm	15.49	Mn ppm	3069
Ca %	15.81	Ba ppm	258.61	Ni ppm	60
Fe%	1.09	Cd ppm	1.60	Pb ppm	19
K%	13.47	Ce ppm	10.11	Sb ppm	4
Mg%	4.33	Co ppm	7.19	Sr ppm	382
Na%	2.06	Cr ppm	39.24	V ppm	45
P%	4.93	Cu ppm	475.35	W ppm	39
S%	6.50	La ppm	6.99	Zn ppm	2880
				Zr ppm	32

Table 3- Chemical analysis of fly ash at the marked point by SEM-EDS.

ELEMENT	Point 1	Point 2	Point 3	Average	ICP
	% w/w				
Al	1.10	1.07	1.13	1.10	2.00
Ca	14.12	14.97	13.89	14.32	15.81
Cl	6.48	0.60	6.53	4.53	-
Fe	0.90	0.73	1.30	0.97	1.09
K	21.33	14.51	21.63	19.16	13.47
Mg	3.14	3.43	3.26	3.27	4.33
Mn	0.10	8.22	0.11	2.81	0.3
Na	4.31	8.31	3.94	5.52	2.08
O	34.19	36.31	34.02	34.84	-
P	4.70	12.21	4.82	7.24	4.93
S	7.61	6.33	7.41	7.12	6.50
Si	2.02	1.52	1.95	1.83	-

3.3. XRD Analysis on the Material and Pointwise (at the Marked Point) Mineralogical Composition Determination with SEM-EDS

As a continuation of the study, the crystal structure of Ca, K, Mg, P, compounds was determined by XRD to determine the mineral structure of the material.

When biomass ash (Figure 3) is subjected to XRD analysis, the main structure contains potassium minerals; as, in ascending order, sylvine (KCl), apthilatate ($\text{Ca}_3\text{NaO}_8\text{S}_2$), ($\text{K, Na}_3\text{Na}(\text{SO}_4)_2$, langbeinite [$\text{K}_2\text{Mg}_2(\text{SO}_4)_3$] and a very small amount of $\text{K}_4[\text{Fe}(\text{CN})_6]\cdot 3\text{H}_2\text{O}$ in its aqueous form were observed. In addition, $\text{Ca}(\text{OH})_2$, CaO and SiO_2 contents are also present and the ash structure has indeed reached a new mineralogical structure in the form of calcination. In terms of phosphorus, the dominant structure is apatite $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ and hydroxyapatite.

After the determination of the main minerals in the biomass ash by XRD, an elemental mass balance was carried out for the elements Ca, Fe, Al, Mg, P, K, Na, S, with an acceptable margin of error (oxygen (O), hydrogen (H) and carbon (C) analysis was not carried out, it was considered as an equation with several unknowns (matrix solution) and the ratio of components and minerals in the material was determined as (w/w) using an Excel spreadsheet (Table 4).

Patnaik (2002) gave these compounds of Ca, Mg, Na and K collectively with their mineral names and CAS numbers (Table 5).

XRD analysis of the material indicated that potassium and magnesium compounds were predominantly in the form of langbeinite and sylvine whereas phosphorus was in the form of apatite and hydroxyapatite.

4. Experimental Studies of Phosphorus and Potassium Recovery and Enrichment

In terms of potassium recovery in the study, the principle that II-valued cations (except Al and Si) are insoluble in alkali, but K, Na compounds and I-valued anions are soluble in water (Weast and Astle, 1980; Green and Southard, 2019). The purpose of this is to obtain a cleaner solution as Ca, Fe, Cu, Zn, P and Mg do not enter the solution.

Firstly, the alkaline leaching under normal conditions; (120 min) l/s ratio of 5 and 10 was observed and visual difficulties were observed, pH control and then the elemental balance, which could be adapted to the industry by increasing the temperature, and the possible yield and final product composition were tested.

In the production of phosphoric acid, it was determined how much phosphorus content PO_4^{3+} could be recovered by reacting the purified waste product with K, adding the necessary $\text{H}_2\text{O} + \text{H}_2\text{SO}_4$ and filtering out impurities such as phosphoric gypsum.

4.1. Potassium Recovery Studies

Using the elemental solubility tables given in Green and Southard, (2019) it was investigated what

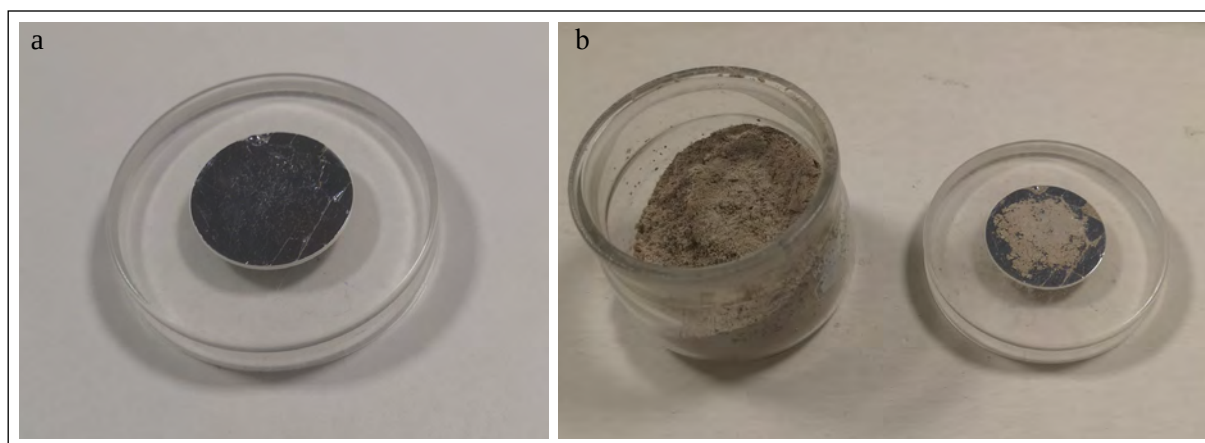


Figure 3- Sample preparation before XRD analysis of biomass ash, a) sample prepared for XRD, b) together.

Table 4- Determination of approximately chemical composition in fly ash according to XRD and ICP analysis results³.

COMPONENT	MW	QTY	%	Al	Ca	Fe	K	Mg	Na	P	S	Ref
Al ₂ O ₃	101.96	4.00	4.00	2.00								Krupa et al., 2008
Ca(OH) ₂	74	1.35	1.35		0.73							
Ca(PO ₄) ₂	230	1.62	1.62		0.28					0,44		
Ca ₁₀ (PO ₄) ₆ (OH) ₂	1004	22.20	22.20	0.00	8.84	0.00	0.00	0.00	0.00	4,11	0.00	
Ca ₅ (PO ₄) ₃ (OH)	502	2.00	2.00		0.80					0.37		
CaO	56	7.20	7.20		5.14							
Fe ₂ O ₃	160	1.56	1.56			1.09						Krupa et al., 2008
K ₂ Mg ₂ (SO ₄) ₃	414	9.00	9.00				1.70	1.04			2.09	
K ₂ SO ₄	174	10.00	10.00				4.48				1.84	
K ₄ [Fe(CN) ₆].3H ₂ O	368.35	~	~									
KCl	74.5	12.44	12.44				6.51					
MgCl ₂	95	8.00	8.00					2.02				
MgSO ₄	120	1.50	1.50					0.30			0.30	
Na ₂ SO ₄	142	1.00	1.00						0.32		0.32	
NaCl	59	4.45	4.45						1.73			
NaKMg ₂ (SO ₄) ₃	398	8.00	8.00				0.78	0.96			1.93	
SiO ₂	60	5.68	5.68									Çöteli and Karahan, 2023
Σ		100.00	100.00	2.00	15.80	1.09	13.47	4.33	2.06	4.92	6.48	
ICP Analyses results % (w/w)				2.00	15.81	1.09	13.47	4.33	2.06	4.93	6.50	

Note: ¹Krupa et al. (2008) stated that Fe and Al phosphates decompose into Fe₂O₃ and Al₂O₃ at high temperatures.

²Silicon analysis was not performed in ICP, only the % SiO₂ amount from XRF analysis was taken for the 5.68% for 100 balances in Çöteli and Karahan (2023).

³Possible compounds of 3Al and Fe in the silicate structure and potassium ferrocyanide were not taken into account, assuming that they were below 1%.

Table 5- Important minerals of potassium (Patnaik, 2002).

MINERAL NAME	FORMULA	CAS NO
Carnallite	KCl•MgCl ₂ •6H ₂ O	1318-27-0
Feldspar	KAlSi ₃ O ₈	68476-25-5
Kainite	4KCl•4MgSO ₄ •11H ₂ O	67145-93-1
Langbeinite	K ₂ SO ₄ •2MgSO ₄	13826-56-7
Mica	KH ₂ Al ₃ (SiO ₄) ₃	12001-26-2
Leucite	KAlSi ₂ O ₆	1302-34-7
Leunite	K ₂ SO ₄ •MgSO ₄ •4H ₂ O	15226-80-9
Polyhalite	K ₂ SO ₄ •MgSO ₄ •2CaSO ₄ •2H ₂ O	15278-29-2
Sylvine	KCl	7447-40-7

type of chemical structure was formed as a result of the dissolution of the material in an alkaline environment, as II-valued cations do not dissolve in alkali.

Experiment performed; primarily for observational purposes, in a glass reactor; liquid volume kept constant at 2000 ml at a speed of 500 rpm (number of recycle per minute for mixing unit) at a temperature of 20°C with turbine mixing measurements (Da/Dt=1/3, E/Dt=1/3, Da/Dt, Da=Blade width of turbine mixer blade, Dt=Diameter of stirrer reactor, E=Height of the mixer from the bottom) in (McCabe et al., 2005). Two experiments were performed with l/s [Ratio of liquid (weight) per solid (weight)]= 5 (2000 ml/400 g sample) and 10 (200 g sample per 2000 ml). The retention time was as long as possible, but not longer than 2 hours. After 1 hour the solid phase settled to the bottom. Although the settling time is short due to the small particle size, the use of a hydrocyclone, which is suitable for separating solid particles between 5-10 microns (Sinnot, 1993), should be preferred in the pilot process stage (Figure 4).

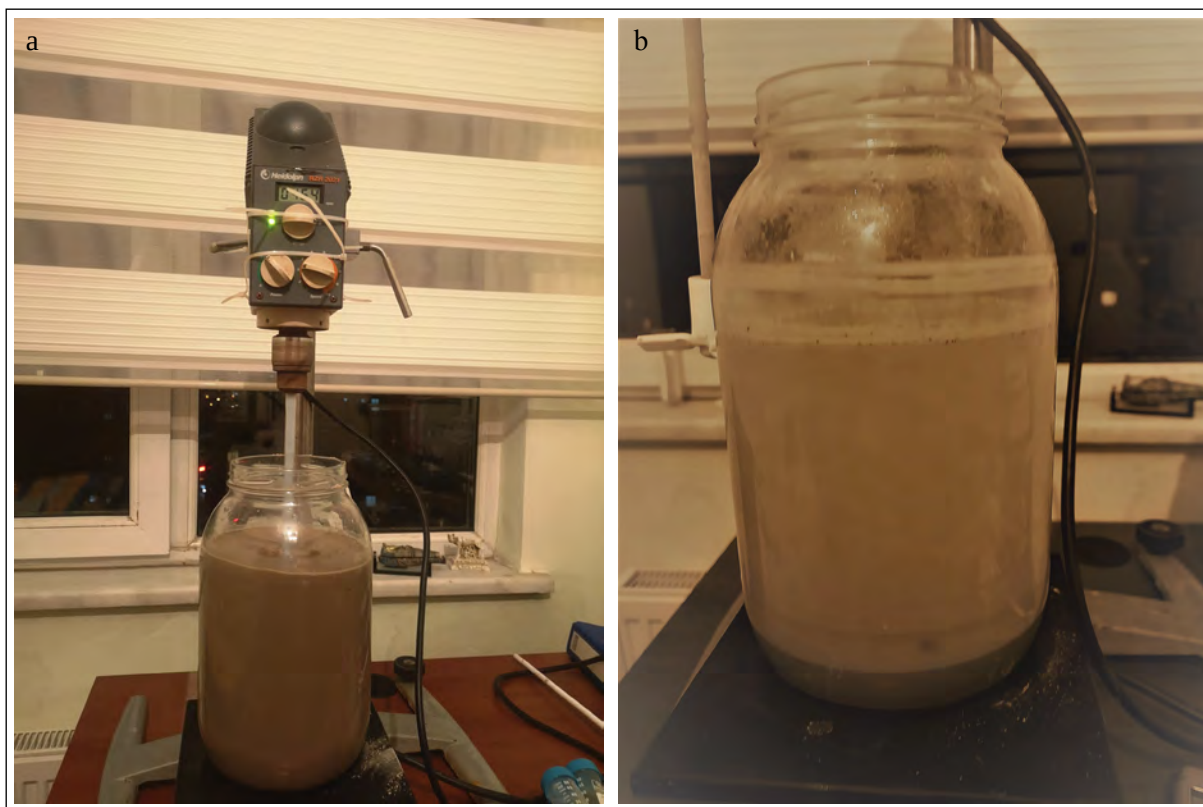


Figure 4- Laboratory studies, a) alkaline leaching studies on the material, b) resting and sedimentation phases.

In such a study, when the l/s ratio is 4, good mixing cannot be achieved below 500 rpm. It was found that 500-650 rpm is suitable.

In addition, as a final experiment, the liquid part was taken with a syringe as a result of the study carried out at 60°C with a l/s ratio of 4 (500 ml pure water/125 g material) and the solid phase (insoluble) accumulated at the bottom was washed 5 times with pure water and rested. It was dried at 105°C to observe the change in weight (Figure 5). The colour of the compounds that pass into the liquid on drying is light pink, close to the colour of natural langbeinite.

$$\eta\% = 100 \frac{\text{amount passing into liquid (g)}}{\text{total ash (g)}} 29.85$$

29.85% was found to be obtained from volatile biomass ash.

An almost pure K_2SO_4 crystal was observed at a random point marked in SEM-EDS (Figures 6a, b and 7) according to the results of the leaching study.

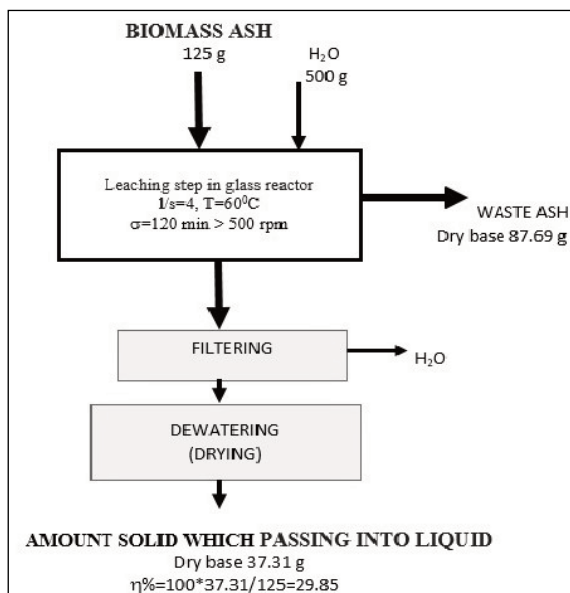


Figure 5- The leaching process.

The % gain of K: $\eta\% = 100 \times 12.49 / 16.84 = \% 74.16$ for Mg, $\eta\% = 100 \times 0.03 / 5.41 = \% 0.55$ has been found. The chemical composition of this structure was determined from Table 6 with acceptable elemental balance.

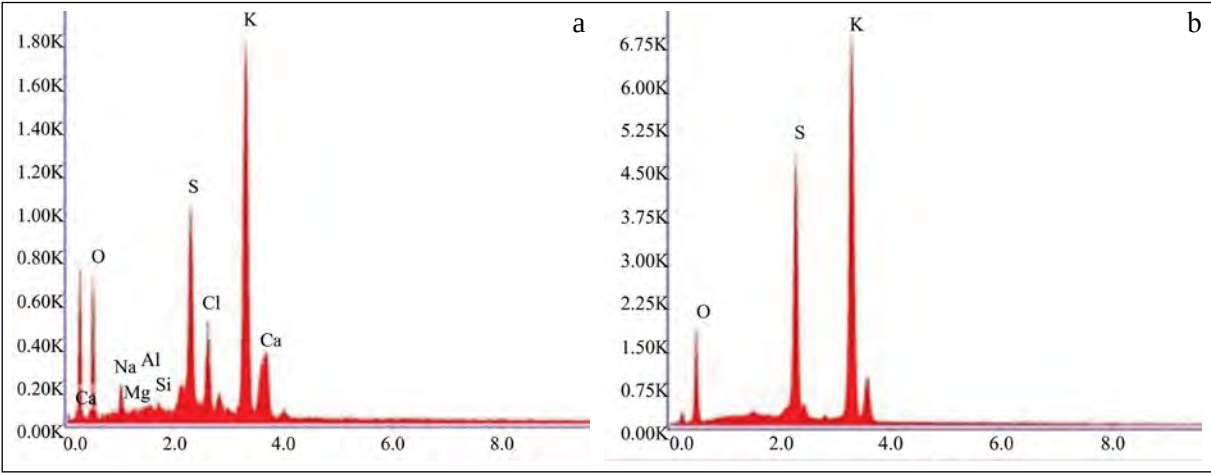


Figure 6- Peaks obtained by examination at 2 points marked in SEM (SEM-EDS). a) full area, b) clear K_2SO_4 crystal in selected or punctured full area.

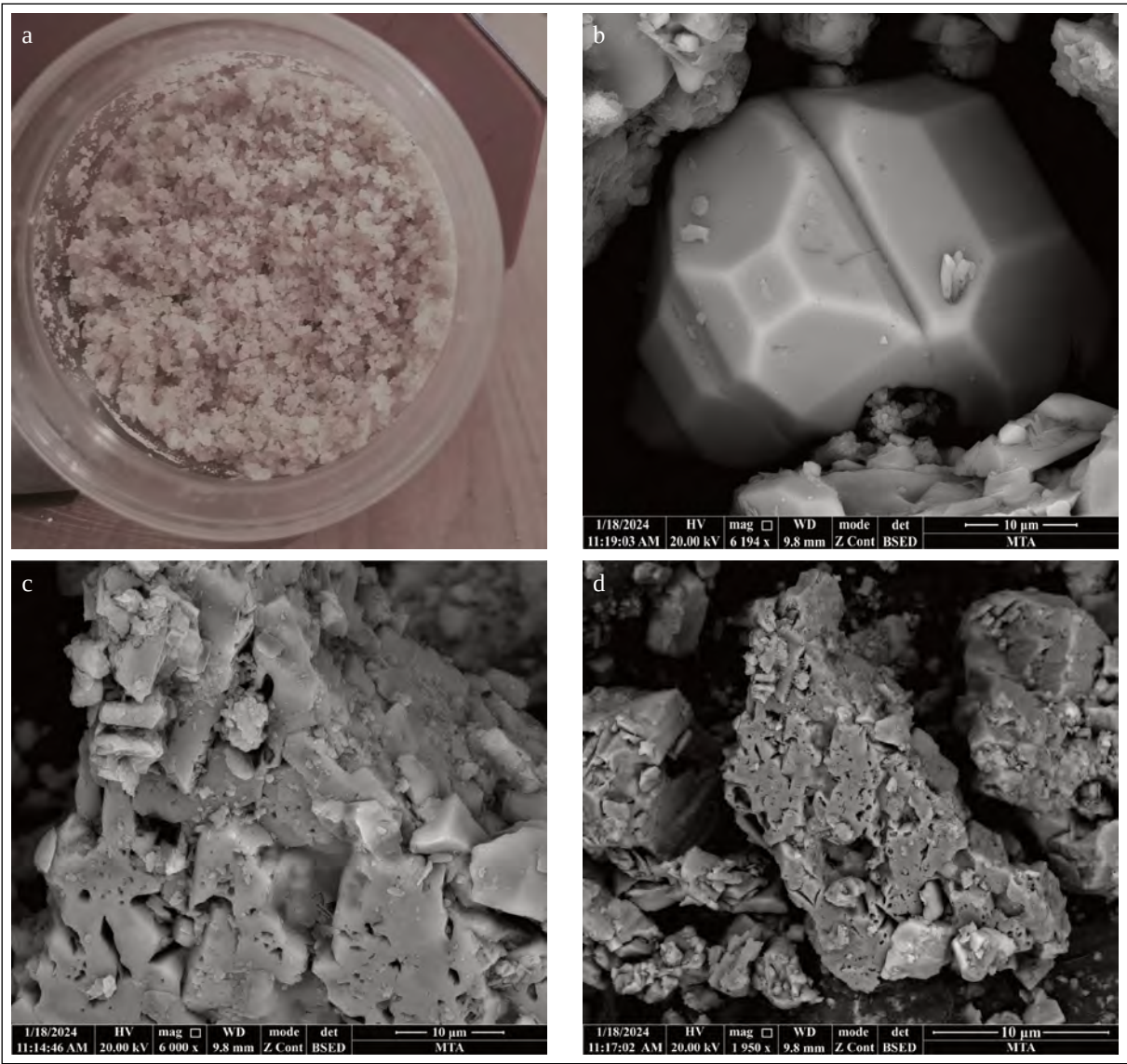


Figure 7- a) Arcanite crystals of normal size passing into the liquid part, b, c, d) Image of K_2SO_4 crystals in SEM analysis.

Table 6- ICP results and elemental balance according to gravimetric inflow and outflow in the leaching environment.

	INPUT FLY ASH (1)		OUTPUT WASTE (2)		PRODUCT (3)		(1)-(2+3) = ~0 (g)
	% w/w <i>icp</i>	In 125 (g)	%w/w <i>icp</i>	In 87.69 (g)	%w/w <i>icp</i>	In 37.31 (g)	
Al	2.00	2.50	2.80	2.46	0.05	0.02	0.03
Ca	15.81	19.76	22.10	19.38	6.79	2.53	-2.15
Fe	1.09	1.36	1.51	1.32	0.02	0.01	0.03
K	13.47	16.84	4.95	4.34	33.47	12.49	0.01
Mg	4.33	5.41	6.15	5.39	0.09	0.03	-0.01
Mn	0.31	0.38	0.42	0.37	0.00	0.00	0.02
Na	2.06	2.58	2.35	2.06	1.14	0.43	0.09
P	4.93	6.16	7.12	6.24	0.02	0.01	-0.09
S	6.50	8.13	2.80	2.46	15.24	5.69	-0.02
Zn	0.29	0.36	0.41	0.36	0.01	0.01	0.00

According to ICP analyses and XRF results, the main components are Ca, K, Mg, Na, Al and S, and with material balance solution, the product composition is thought to be 72.28% K₂SO₄ and also 1.23% KCl as a projection in future studies.

When the main mineral structure was examined, it was found to be aphtilide (K₃NaO₈S₂), sylvite (ClK_{0.7}Na_{0.3}), sylvine (KCl), dolomite (CaMgO₆-CaMgCO₃), Ca(OH)₂ and mainly arcanite (K₂SO₄). Chemical analysis was carried out at the point marked by the SEM, taken from 2 points, and a very large amount of K₂SO₄ and the visual structure (Figure 6b) are given in Table 8.

The post-leaching waste material, defined as Waste-1, was subjected to XRD analysis (Figure 8).

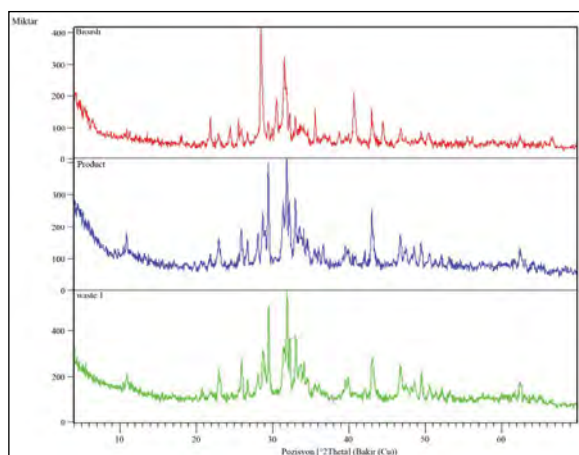


Figure 8- Detection of major minerals in the XRD analysis of the dewatered and dry solid product after leaching.

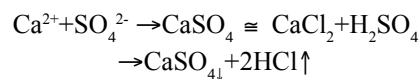
Comparative XRD diffractograms of the biomass ash (bioash), the waste and products in Figure 8. Arcanite crystals can be distinguished in the XRD diffractogram in Figure 9.

Preliminary experimental observations:

-During the study period, the pH of the liquid part was found to be 12.85. It is a highly alkaline work. In addition, a thin layer of lime cream forms on the liquid part. This is probably due to the reaction of CaO with water and Ca(OH)₂ in the sweat on Table 4.

-In particular, the sample with a l/s ratio of 4 was prepared at 60°C with a leaching time of 120 minutes, and when the liquid solution cools to 4-5°C, white crystals form at the bottom. As a result of this observation, it was concluded that it was necessary to clean this calcium-based contaminant structure.

-In the study, it was observed that when the pH was lowered to the 8.5-9 range, the solution became clear and the calcium content precipitated as black, grey crystals as



were observed as possible reactions (Bayram, 2018).

When the resolution table is examined; the solubility ratio between 100°C and 20°C is; K₂SO₄=(24.10/7.40)=2.17, KCl=1.64, MgSO₄=1.79, Na₂SO₄=2.17, NaCl=1.091, MgCl₂=1.34 was concluded that is necessary to use the temperature

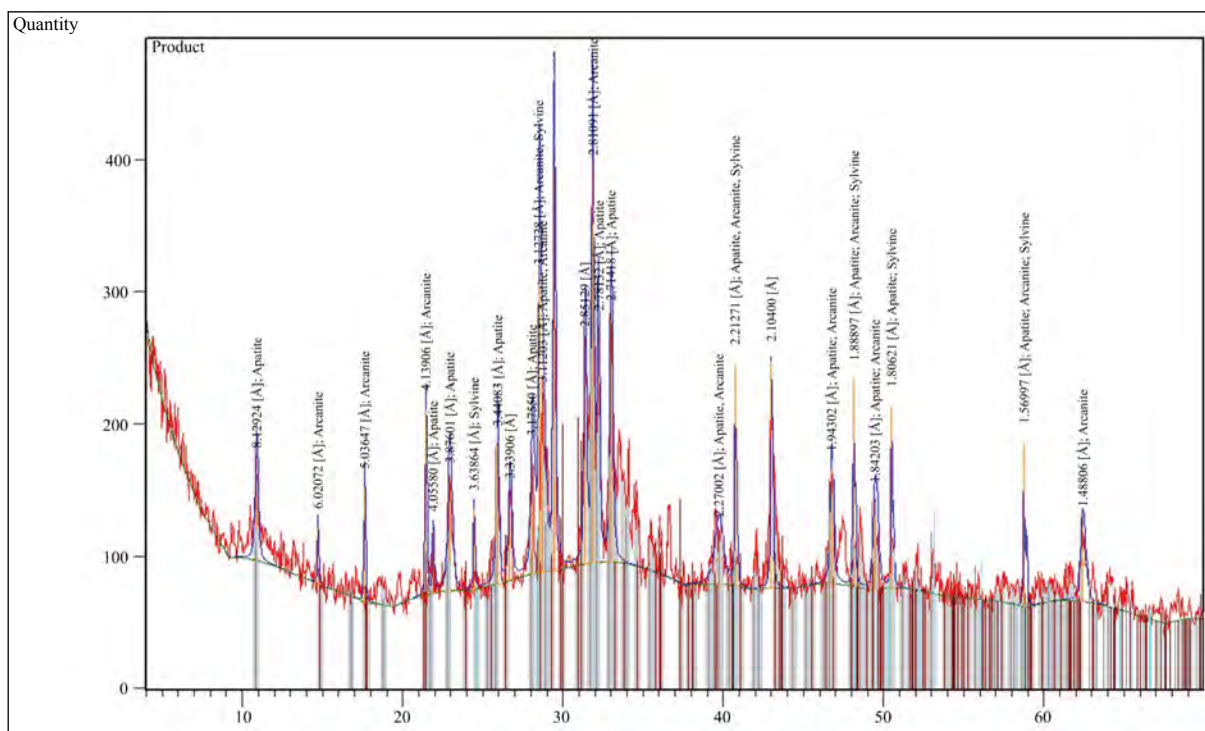
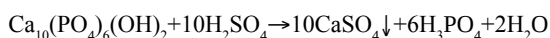
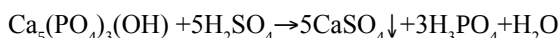
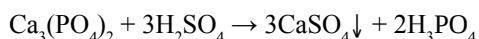


Figure 9- Detection of major minerals in the XRD analysis of the dewatered and dry solid product after leaching.

factor to reduce the l/s ratio for economic purposes. For higher solubility with less liquids, it has been found proper to operate the leaching temperature at about or close to $>60^{\circ}\text{C}$.

4.2. Phosphorus Recovery Studies

The main chemical reactions of wet process H_3PO_4 production are given in (Becker, 1989) and are similar to the reactions according to the apatite structure.



Stoichiometric acid requirement per 100 grams of ash; according to the study of Krupa et al. (2008), the required amount of H_2SO_4 is given on the Table 10.

The study carried out; 40 g of 98% H_2SO_4 per 100 g was added to the material (remaining after leaching). It was well mixed with 35 g of water. The temperature of the material was rapidly increased to 100°C and the addition of 1000 ml of pure water was completed without solidification (with l/s ratio of 10) (Figure 10).

The suspended material was then mixed at a speed of 500 rpm for one hour. As a result of the one-hour rest, solid particles (CaSO_4 + silicates and insoluble) settled to the bottom and a direct sample was taken from the clean solution before ICP. It is the amount corresponding to H_3PO_4 over the phosphorus (P) read in the liquid.

$$\text{H}_3\text{PO}_4 \text{ (mg/l)} = \text{reading (mgP/l)} * (98/31)$$

The maximum that can be increased is H_3PO_4 step for H_3PO_4 production from biomass ash in the light of the studies carried out, it seems possible to produce concentrated dirty phosphoric acid in a continuous system as in Krupa et al. (2008). Only the produced samples of H_3PO_4 are taken from the system. The circulation system is H_3PO_4 solution. It seems necessary to separate $\text{Al}_2(\text{SO}_4)_3$ and other Mg, Na, K compounds for a cleaner phosphoric acid production.

5. Results and Discussion

-Three different processes are used in the world to produce potassium sulphate. These are the Mannheim process, the salt lake potassium sulphate process and the polyhalide (potassium chloride-magnesium sulphate)

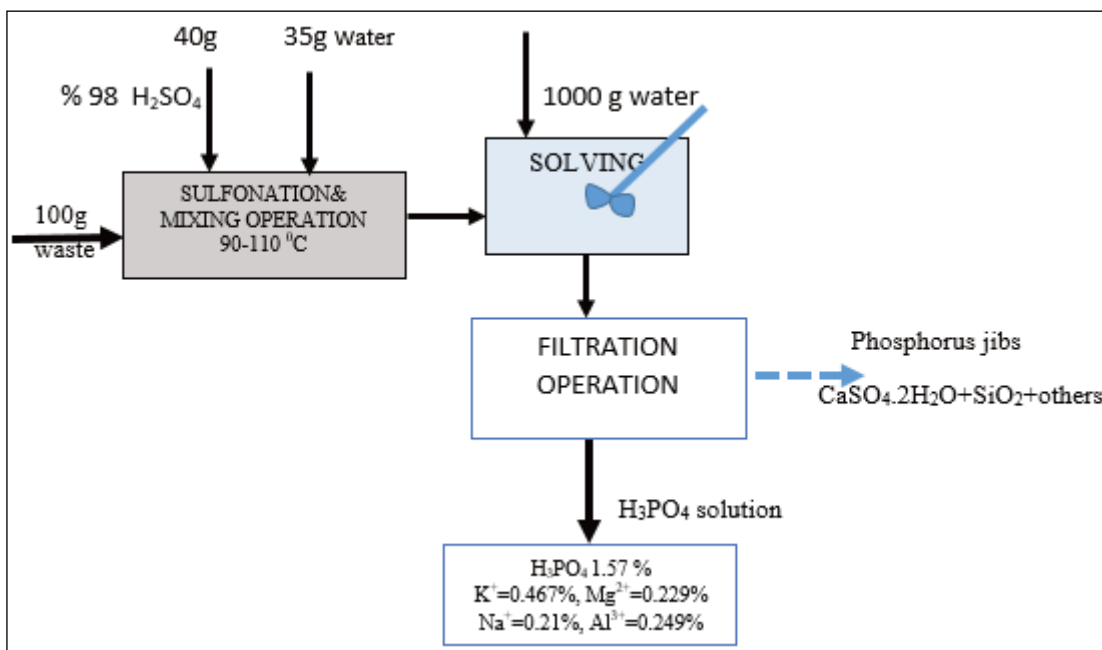


Figure 10- Suggested box-box continuation process.

potassium sulphate production process. The costs of each process are different. The process that produces the highest and best quality potassium sulphate is the Mannheim process. According to Aslan et al. (2020), depending on the process, global potassium sulphate production from saline lakes is approximately \$320/tonne, \$410/tonne for polyhalide and \$520/tonne for KCl and H_2SO_4 using the Mannheim process.

-The largest potash producers in the world are China, Canada and Russia. Potassium sulphate production in China will increase by 8.6% between 2013 and 2018, making it the largest potassium sulphate producer. Russia already has a significant share of the potassium sulphate market. Canada has decided to invest in potassium sulphate production in recent years. The world's largest importers of potassium sulphate are Germany, Belgium and the Netherlands. These three countries account for approximately 60% of the world's total potassium sulphate imports (Aslan et al., 2020).

How this distinction can be made is illustrated by two further studies of the process. As a result of the preliminary studies;

- When the XRD analyses of the materials were examined, it was found that the langbeinite content

on the fly ash was very high, but the dried material obtained at the end of the leaching process had no langbeinite content. There is a very small amount of langbeinite in the waste product.

- It has been observed that economically enriched concentrated potassium salts, which can have a market share, can be produced from biomass thermal power plant ashes by the alkaline leaching method. It is a concentrated potassium ore whose predominant structure is arcanite (K_2SO_4) with K_2O = 40.33% (pure industrial chemical K_2O is 54.02%, S=18.39%, O=36.78%) among the low temperature (even at 60°C) material.

- The reason for choosing the alkaline leaching method is that compounds such as $\text{Mg}(\text{OH})_2$, CaCO_3 , MgSO_4 , CaCl_2 precipitate at high pH in salt water mixtures. The reason for the presence of $\text{Ca}(\text{OH})_2$ in the current product is its alkaline nature. It is possible that this can be precipitated by the addition of H_2SO_4 .

- Solid recovery: 29.85% and K recovery: 74.16%.

- The Mg recovery in the leaching process is 0.55% and this is entirely due to the high pH of the leaching process. The results are in line with our expectations.

- In addition, increasing the leaching temperature to 95-100°C and washing the remaining solids with clean solution prior to centrifugation will increase the recoveries.

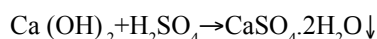
- The recovery of phosphoric acid from the material by the wet process did not appear to be possible within the method followed. An alternative route should be investigated by high temperature or other studies. For example, as in Krupa et al. (2008), mixing the resulting H_3PO_4 with higher concentration clean H_3PO_4 and placing it on the market.

-The tests carried out on the waste were only designed to detect the compounds that had leached into the liquid and did not investigate the new acidic structure that could be formed by dehydration. Since only the chemical structure and the percentage composition are known, as can be seen in Table 11, the percentage H_3PO_4 can reach up to 23.76, which means that this production is not possible. Therefore, purification of the liquid composition by various alternative methods should be considered. For example, the leaching process to be carried out on the waste at pH around 7 or pH 6-7 will largely remove the K_2SO_4 , $MgSO_4$, $FeSO_4$ and $Al_2(SO_4)_3$ content. However, as this process is carried out with water, it is far from being economically viable to produce due to the need for post-separation distillation etc. Nevertheless, the product is of industrial importance due to its high content.

-The resulting material is neutralized with $NH_4(OH)$ and converted into an N-P-K fertilizer.

-It has also been shown that the $MgSO_4$ and Na_2SO_4 in the material bind crystal water (Weast and Astle, 1980).

-In the light of the studies carried out, it has been seen that such a system has difficulties operating in the pH range of 11-13 in terms of corrosion and separation of materials, both theoretically and in the light of the studies carried out. Therefore, after the leaching process, the solution has to be neutralized with H_2SO_4 .



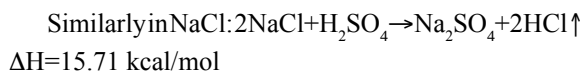
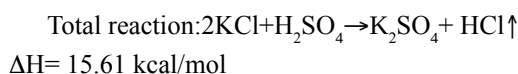
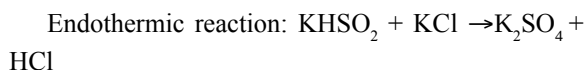
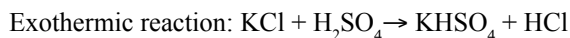
From Table 7, theoretically H_2SO_4 requirement per 100 g biomass ash;

$$\text{For } Ca(OH)_2: 29.85 * 12.25 * 98 / (74 * 100) = 4.84 \text{ g}$$

More than 50% of the world's sulphate production is based on the Mannheim process.

The Mannheim process was originally developed from the production of sodium sulphate by reacting NaCl with sulphuric acid. By replacing NaCl with KCl, potassium sulphate is produced (Felton et al., 2010).

The reaction is a two-step process:



$$\text{According to KCl: } 29.85 * 1.23 * 98 / (2 * 74.50 * 100) = 0.24 \text{ g}$$

$$\text{For NaCl: } 29.85 * 1.23 * 98 / (2 * 58.5 * 100) = 0.24 \text{ g}$$

A total (For $Ca(OH)_2 + KCl + NaCl$) of 5.32 g H_2SO_4 /100 g fly ash may be required.

$$\text{Maximum } K_2SO_4 \text{ that can be occur: } 29.85 * 33.47 * 174 / (2 * 39 * 100) = 22.29 \text{ g}$$

$$\text{Maximum } Na_2SO_4 \text{ that can be occur: } 29.85 * 1.23 * 142 / (2 * 23 * 100) = 1.14 \text{ g}$$

$Na_2SO_4 + K_2SO_4$ and the recovery of K_2SO_4 seem much easier at high temperatures (Table 9). This also means obtaining high purity K_2SO_4 .

K_2SO_4 saturation at 100°C 24.10 g/100 ml water will collapse first in the tchiner-1

$$Na_2SO_4 \text{ saturation at } 100^\circ C \text{ } 42.50 \text{ g/100 ml water}$$

- Precipitation of some of the extracted material by neutralization with NH_4OH at a higher pH.

Alternative evaluation methods such as separation can be given as examples.

- As a result of the preliminary studies carried out

Table 7- Determination of the approximately chemical composition of the material recovered in the liquid (K_2SO_4).

COMPONENTS	MW	MIXED MATERIAL QUANTITY	%	In 100 g material (g)							
				Ca	K	Mg	Na	S	Al	Fe	P
Al_2O_3	101.96	0.10	0.12						0.06		
$Ca(OH)_2$	74.00	10.00	12.25	6.62							
$CaMgO_6$	160.00	0.50	0.61	0.15		0.092					
$K_{0.7}Na_{0.3}Cl$	69.85	1.00	1.23		0.48		0.19				
$K_2Mg_2O_{12}S_3$	414.00	1.00	1.23		0.23	0.006		0.28			
K_2SO_4	174.00	59.00	72.28		32.40			13.29			
$K_3NaO_8S_2$	159.55	1.00	1.23		0.90		0.18	0.49			
KCl	74.50	1.00	1.23		0.64						
*Crystal- H_2O	18	0.90	1.10								
Na_2SO_4	142.00	1.00	1.23				0.40	0.28			
NaCl	58.50	1.00	1.23				0.48				
SiO_2	60.08	5.00	6.13								
$Fe(OH)_2$	90.00	0.03	0.04							0.02	
$Ca_3(PO_4)_2$	230.00	0.10	0.12	0.02							0.01
	Σ (g)	81.63	100.00	6.81	34.65	0.10	1.24	14.34	0.06	0.02	0.01

*weight loss at 1 hour at 300°C

Note: The analyses are carried out separately with ICP and SEM-EDS and there are some differences between the analyses. This is because analyses such as Cl, O, Si cannot be carried out by ICP, SEM-EDS can accurately determine the exact result with a large number of marked points, S analyses cannot be carried out reliably by ICP and also because this study is a preliminary study prior to the project. Verification was carried out using modelling of up to 100 oxide forms to determine the structure within engineering limits of error (+/-2.5%). According to the balance of 100 oxide forms Al_2O_3 0.12%, K_2O 40.34%, CaO 9.51%, SO_3 35.86%, MgO 0.13%, Na_2O 1.67%, Cl 5.06%, Fe_2O_3 0.03%, PO_4 0.04 %, volatiles 0.1%, crystals H_2O 1.1 % acceptable.

Table 8- Chemical analysis (%w/w) at the points marked with SEM-EDS.

Element	Point 1	Point 2	Average	ICP %w/w
Al		0.39	0.39	0.05
Ca		6.77	6.77	1.45
Cl		5.06	5.06	-
K	41.63	30.34	35.98	33.47
Mg		0.43	0.43	0.09
Na		4.04	4.04	1.14
O	40.09	40.52	40.31	-
S	18.28	12.04	30.32	15.24
Si		0.43	0.43	-

Table 10- The amount of H_2SO_4 required per 100 g obtaining H_3PO_4 in the material.

	%	Multiplier	Σ	Total= 47.13 g H_2SO_4 /100 g fly ash
Al_2O_3	3.85	0.614	2.36	
CaO	22.13	1.479	32.73	
Fe_2O_3	1.56	0.614	0.96	
K_2O	16.23	1.041	16.90	
MgO	7.21	2.433	17.54	
Na_2O	2.77	1.582	4.38	
P_2O_5	11.29	-0.691	-7.80	
SO_3	16.25	-1.225	-19.90	

Table 9- Solubility of Na, K, Mg salts in water (% w/w) at heat [from Green and Southard (2019)].

Chemicals		0°C	10°C	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
K_2SO_4	g/100 ml	7.40	9.30	11.10	13.00	14.80	16.50	18.20	19.80	21.40	22.90	24.10
KCl	g/100 ml	28.00	31.20	34.20	37.20	40.10	42.95	45.80	48.55	51.30	53.90	56.30
$MgCl_2$	g/100 ml	52.90	53.60	54.60	55.80	57.50	59.25	61.00	63.55	66.10	69.50	73.30
$MgSO_4$	g/100 ml	22.00	28.20	33.70	38.90	44.50	49.55	54.60	55.20	55.80	52.90	50.40
NaCl	g/100 ml	35.70	35.80	35.90	36.10	36.40	36.75	37.10	37.55	38.00	38.50	39.20
Na_2SO_4	g/100 ml	4.90	9.10	19.50	40.80	48.80	47.05	45.30	44.50	43.70	42.70	42.50

Table 11- The determination of the approximate liquid H_2PO_4 composition.

Element	WASTE % w/w	Expected mg/l	%	Read in ICP %w/w	liquefiable efficiency η %	Components	May occurs (g)	in liquid %	*Dry base %w/w
Al	2.8	2800	0.28	0.249	88.93	Al ₂ (SO ₄) ₃	22.41	2.09	31.61
Ca	22.1	22100	2.21	-	0.000	CaSO ₄	-	-	-
Fe	1.51	1510	0.151	0.115	76.16	FeSO ₄	3.12	0.29	4.0
K	4.95	4950	0.495	0.465	93.94	K ₂ SO ₄	10.37	0.97	14.63
Mg	2.8	2800	0.28	0.229	81.79	MgSO ₄	11.45	1.07	16.15
Na	2.35	2350	0.235	0.217	92.34	Na ₂ SO ₄	6.70	0.63	9.45
P	7.03	7030	0.703	0.533	75.82	H ₃ PO ₄	16.85	1.57	23.76
						H ₂ O	1000	93.38	0.00
						Σ	1070.90	100.00	100.00

*This calculation is for only projection purposes only and <1% Mn, Zn, Cu sulfate contents are also possible in the dry base composition.

in this study, the design process shown in Figure 11 is presented as an economic process.

- Further project work will continue in the form of key studies such as process optimisation, economisation, equipment specifications, detailed mass energy balances and determination of equipment retention times.

- This study was carried out with the resources of the MTA-Department of Mineral Analysis and Technology

(MAT) department as a preliminary study for the purpose of utilising biomass thermal power plant ashes, which have increased rapidly in our country in recent years due to the energy demand. It was carried

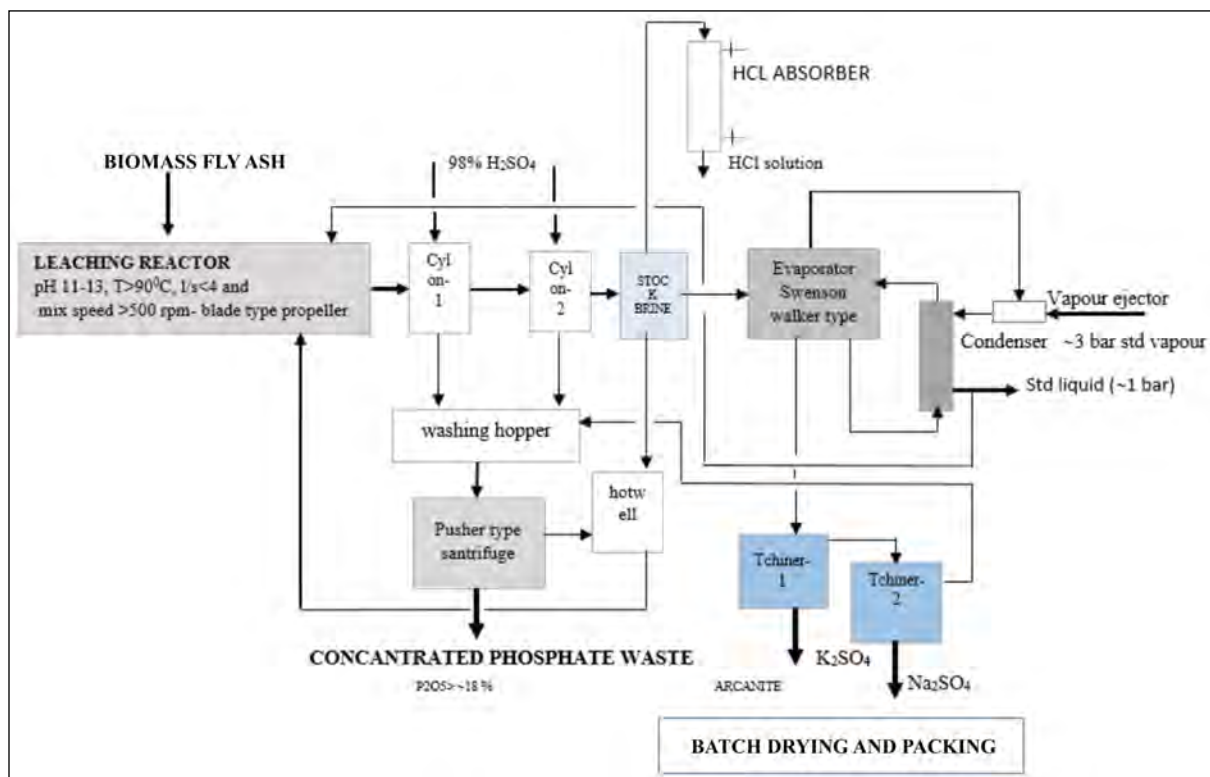


Figure 11- Suggestion draft box-box continue process step for concentrated potassium sulphate production from the biomass ash.

out only for the purpose of understanding the subject and developing a suitable method for the preliminary study of the project. Our country aims to reduce the use of potash. As far as the future waste management is concerned, the continuity of biomass ash at certain standards, the overcoming of official-bureaucratic problems in the field of the environment and the renewal of the waste management and waste disposal codes appear as an important issue for economic gain.

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Establishing of soil loss tolerance limit and sediment yield in Zagros fold-thrust belt: a case study in the Kanarwe river basin, Iraq-Iran

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Research Article

Keywords:

RUSLE, Sediment Delivery, Zagros Thrust Zone, CN-SCS Curve, Dam Lifespan.

ABSTRACT

Soil degradation is a significant cause of topsoil loss; in most mountainous region watersheds, it leads to decreased agriculture productivity and reservoir storage. This study targeted calculating and mapping soil loss and sediment yield in the Lesser Zab watershed located between Iraq and Iran. The Revised Universal Soil Loss Equation (RUSLE) model was used to calculate the soil loss. A Digital Elevation Model of 30 m, a Digital Soil map (1:500000), rainfall, and land cover were used to derive parameters. The soil loss rates are 58.1 and 0.1 $\text{tha}^{-1}\text{yr}^{-1}$. The total annual soil loss is 1037289 tonnes, of these 404512, covering 75% of Kanarwe river basin land. Most of these affected lands are in the eastern and middle part, which is below the FAOs standard allowable for tolerable soil erosion. Still, the rest covers 25 % of the basin in the west with a total annual soil loss equal to 632777 tonnes, above FAOs standard. The maximum and minimum sediment yield is 29 and 0.1 $\text{tha}^{-1}\text{yr}^{-1}$, respectively. Based on the statistical correlation coefficient, the most effective RUSLE parameters on sediment yield from high to low are topography 0.48, soil erodibility 0.38, and crop management 0.38.

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1. Introduction

Soil erosion is a geomorphic process that occurs naturally; however, humans' soil utilization and climate change lead to increased land degradation (Pennock, 2019; Yang et al., 2003). Soil loss particles are turned into sediments and delivered to the river by an erosion agent (Chuenchum et al., 2020), and erosion processes pose severe threats to agricultural production, dam lifespan, and the earth's landform (Balabathina et al., 2020; Lee et al., 2014). Man-made activities cause ten times faster soil loss than natural

agents such as rainfall or coastal waves (Pimentel and Burgess, 2013). Sometimes, trapped sediment yield by a natural or artificial agent leads to bank erosion and decreased riparian vegetation (Anthony et al., 2015).

The RUSLE equations were first developed on a small scale in the United States of America but later were used and calibrated in many countries (Alewell et al., 2019; Ezzaouini et al., 2020). New studies suggest that integrating the Revised Universal Soil Loss Equation (RUSLE) with satellite image data is more effective for calculating amounts of soil loss

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and mapping its distribution (Kebede et al., 2021; Somasiri et al., 2021).

The soil loss and sediment yield recently threatened water quality and quantities in Iraq and Iran due to poor management of soil loss (Zare et al., 2017; Band et al., 2022; Ostovari et al., 2022; Othman et al., 2022; Salar, 2022). The unmanaged soil loss from the upstream country (Iran) and downstream sediment delivery decreased dams' storage capacity and water resource life structures in the lesser Zab watershed within Iraq (Bhattarai and Dutta, 2007; Omeed H. Al-Kakey et al., 2023).

Many studies try calculating Iraq's sediment yield and soil loss, but the results are inaccurate. Hassan et al. (2016) studied sediment deposited types with the Dokan dam reservoir (DDR), located downstream of the Lesser Zab watershed, without considering the amount of Soil erosion risk and its effect on ecosystem management. Ezz-Aldeen et al. (2018) studied watershed sediment of DDR using the soil and water assessment model (SWAT) to estimate sediment delivery ratio-based Empirical Equation (Lumped model). However, this model is only applicable to small watersheds, not a watershed like DDR. Othman et al. (2022) conclude that the sediment yield is more than the soil loss, which is scientifically wrong. The soil erosion risk in Iran is also the same as in Iraq and many studies conclude that the risk of soil erosion has increased in the last decade (Sadeghi, 2017; Zare et al., 2017; Hosseinalizadeh et al., 2020).

Therefore, this study aims to answer four key research issues; How much soil eroded annually from the Lesser Zab watershed located between Iraq and Iran? Are the soil erosion processes uniformly over the basin or abnormally distributed over the Lesser Zab watershed? Is the potential for soil loss greater than the FAO's soil erosion tolerance limit? Which parameters of RUSLE are more significant on soil loss? This study's findings will serve as a benchmark for future dam site selection and ecosystem management in Iraq.

2. Research Methods

2.1. Study Area and Data Source

The Kanarwe River basin is part of the lesser Zab transboundary watershed within the Zagros

Mountain Range (Figure 1). The basin outlet is 20 km away from northeast Sulaymaniyah city with a catchment area of 1541 km² and extended about 71 km in length (Mohammad, 2023). The Mediterranean climate's hot summers and wet winters deliver more than 800 mm of rain and have an average temperature of 20°C per year (Abbas et al., 2017; Shubbar et al., 2017; Mohammed et al., 2021;). Several data sources to estimate soil erosion based on the RUSLE. Field survey and ground truth collection by a Global Position System (GPS) include soil sampling and lithology description. A (30*30 m) DEM from the Advanced Spaceborne Thermal Emission and Reflection Radiometer (ASTER; GDEM) dataset is used to derive slope length- steepness factor value. The geological map of study area (Figure 2a) was correlated with soil maps to measure accurate soil depth about geological units (Al-Qayim et al., 2018; Ma'ala, 2007). Soil map derived from the Harmonized Soil World Database (HWSD) (Figure 2b), with a scale of 1:500000 (Buringh, 1960; Nachtergaele et al., 2008) Mean annual rainfall data (1990–2015) from the four-gauge stations (Dokan, Chwarta, Mawat, and Penjween) obtained from Sulaymaniyah metrological Directorate was used to estimate the annual mean rainfall at Kanarwe river basin (Mohammed et al., 2023) (Figure 3).

The Global Land Use and Landcover (LULC) map was obtained from the Global Land Cover Facility (www.landinfo.com) for determining soil erodibility, cover management, and SCS-CN number.

3. Data Analysis

3.1. Soil Loss Estimation and Sediment Yield

The soil loss was calculated using the RUSLE, an empirical model developed by Renard in (1997) to estimate soil loss. The RUSLE model Equation 1 is expressed as:

$$S_{loss} = R_E * K_s * LS_T * C_V * P_E \quad (1)$$

Where S_{loss} is the mean annual soil loss due to water erosion [in tons per hectare per year (t ha⁻¹ year⁻¹)], R_E is the rainfall and runoff erosivity factor (in MJ-mm ha⁻¹ h⁻¹ year⁻¹), K_s is the soil erodibility factor (in tone ha⁻¹ MJ⁻¹ mm⁻¹); L is the slope length

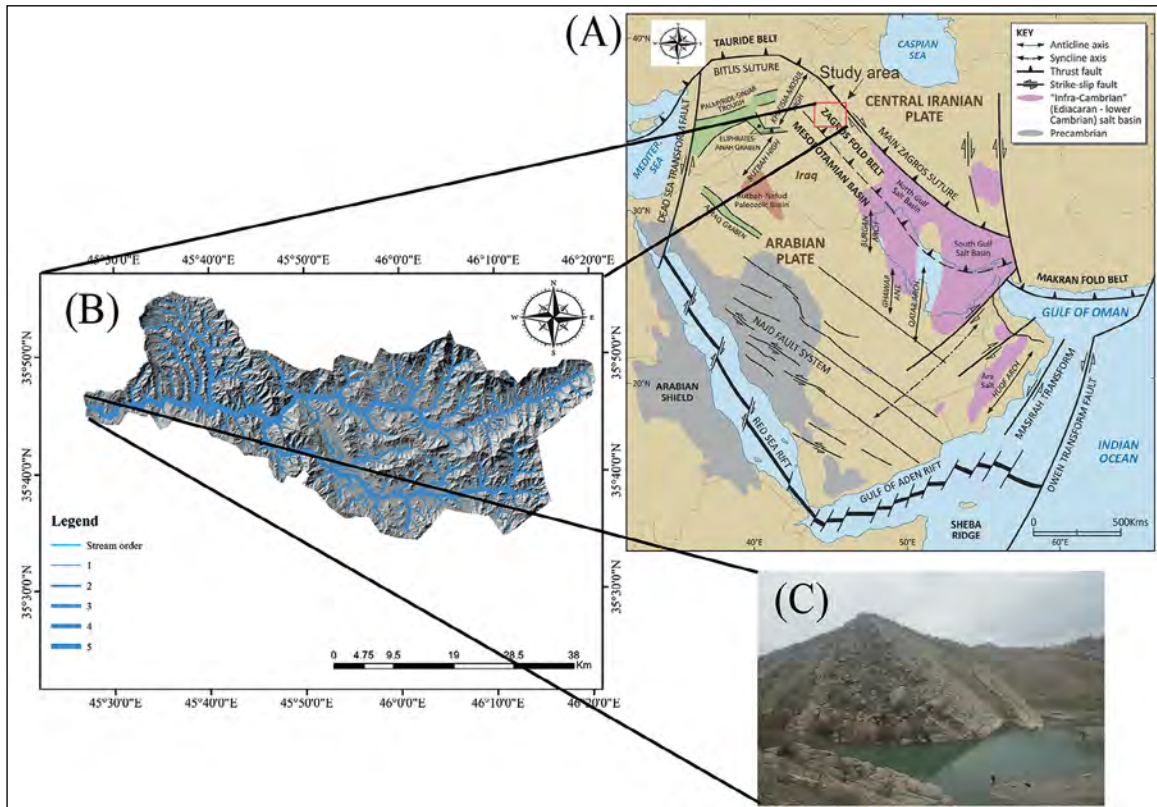


Figure 1- a) Regional tectonic setting Zagros mountains (English et al., 2015), b) Stream network of Siwail River that flows toward basin outlet (Dam site, East west), c) Proposed dam site.

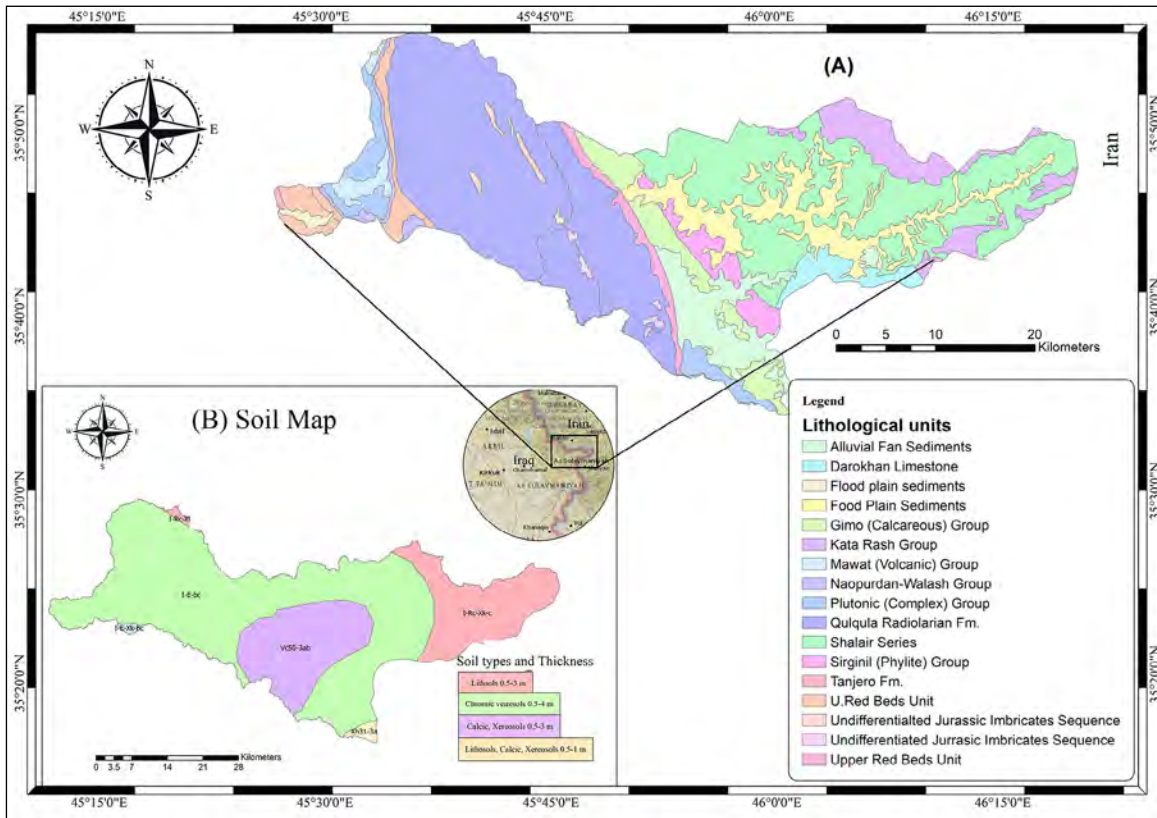


Figure 2- a) Show the geological map after (Ma'ala, 2007; Al-Qayim et al., 2018), b) Soil map of the study area after (Buringh, 1960).

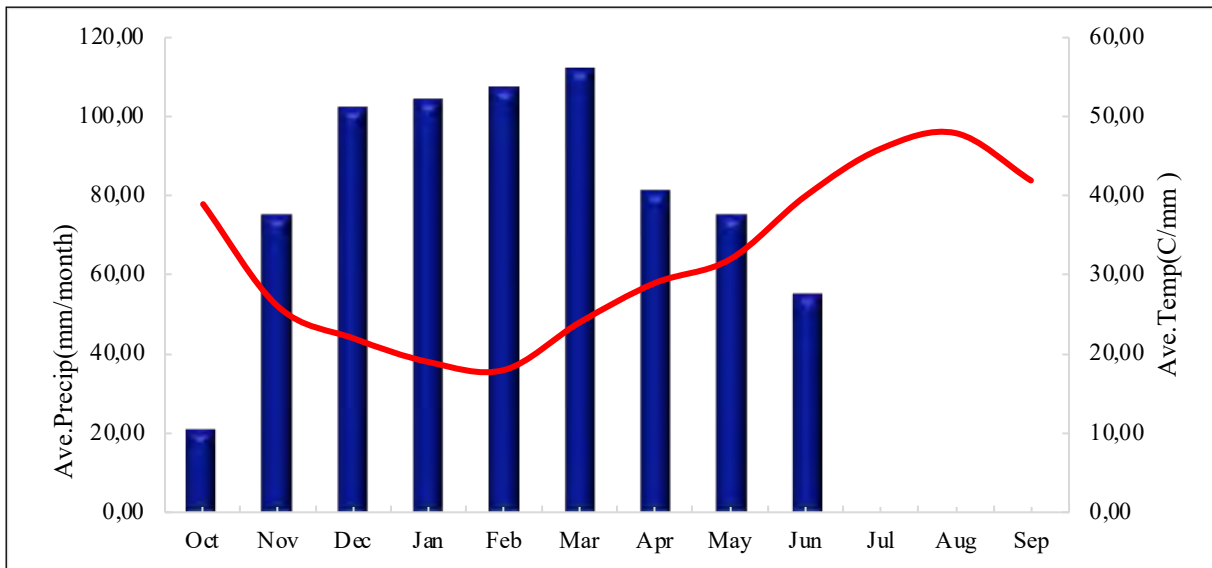


Figure 3- Mean monthly rainfall (blue bars) and temperature (red line).

factor (dimensionless), S is the slope steepness factor (dimensionless); C is the cover and management practice factor (dimensionless), and P_e is the erosion support practice (dimensionless). The sediment delivery ratio (SDR; $t \cdot ha^{-1} \cdot yr^{-1}$) was calculated based on Equation 2.

$$SDR = 1.366 \times 10^{-11} \cdot (DA) - 0.0998 \cdot (RL) + 0.3629 \cdot (SCS-CN) + 5.444 \quad (2)$$

Where DA is the drainage area in km^2 , RL is a relief-length ratio in $m \cdot km^{-1}$, and $SCS-CN$ is the soil conservation service curve number. Finally, the sediment delivery ratio was used with Soil loss rate Equation 3 to calculate the sediment yield (Sy ; $t \cdot ha^{-1} \cdot yr^{-1}$).

$$Sy = SDR \cdot Ri \quad (3)$$

Where Ri is soil erosion for each grid cell.

Spatial analysis techniques and satellite images were used to simulate the RUSLE model, sediment delivery ratio, and sediment production., as shown in Figure 4.

3.2. Rainfall Erosivity (R_e)

Rainfall erosivity is the erosive power of participation kinetic energy: the greater the intensity and rainstorm duration, the higher the erosion potential (Gholami et al., 2021). The R_e factor is generally

calculated from mean annual rainfall data using storm energy over a 30-min duration expressed by $R = EI_{30}$ Equation 4.

$$R = \frac{1}{n} \sum_{j=1}^n \left[\sum_{k=1}^m (E)(I_{30})^k \right] \quad (4)$$

Where n is the total number of years where data was collected, K is the total number of rainfall storms up to m , j is several years up to n , I_{30} is the maximum 30 min intensity ($mm \cdot hr^{-1}$), E_j is the total kinetic energy ($MJ \cdot ha^{-1}$) of a k storm in m year (Renard and Freimund, 1994) Developed (Equation 5).

$$R_e = 0.04830 \cdot P^{1.61} \quad P \text{ is less than } 850 \text{ mm} \quad (5)$$

where R = Rainfall Erosivity, P = Mean annual rainfall in mm.

For calculation (R_e), four stations' long-term mean annual rainfall (1990 to 2015) was obtained from the Sulaymaniyah Meteorological Directorate Table 1. For creating an erosivity map, the inverse distance weighted (IDW) approach is used to estimate the unknown cell value of erosivity for the whole watershed.

3.3. Soil Erodibility (K_s)

The soil erodibility factor (K_s) assesses how vulnerable soil types are to particles being separated and carried away by rainfall and runoff. (Wischmeier

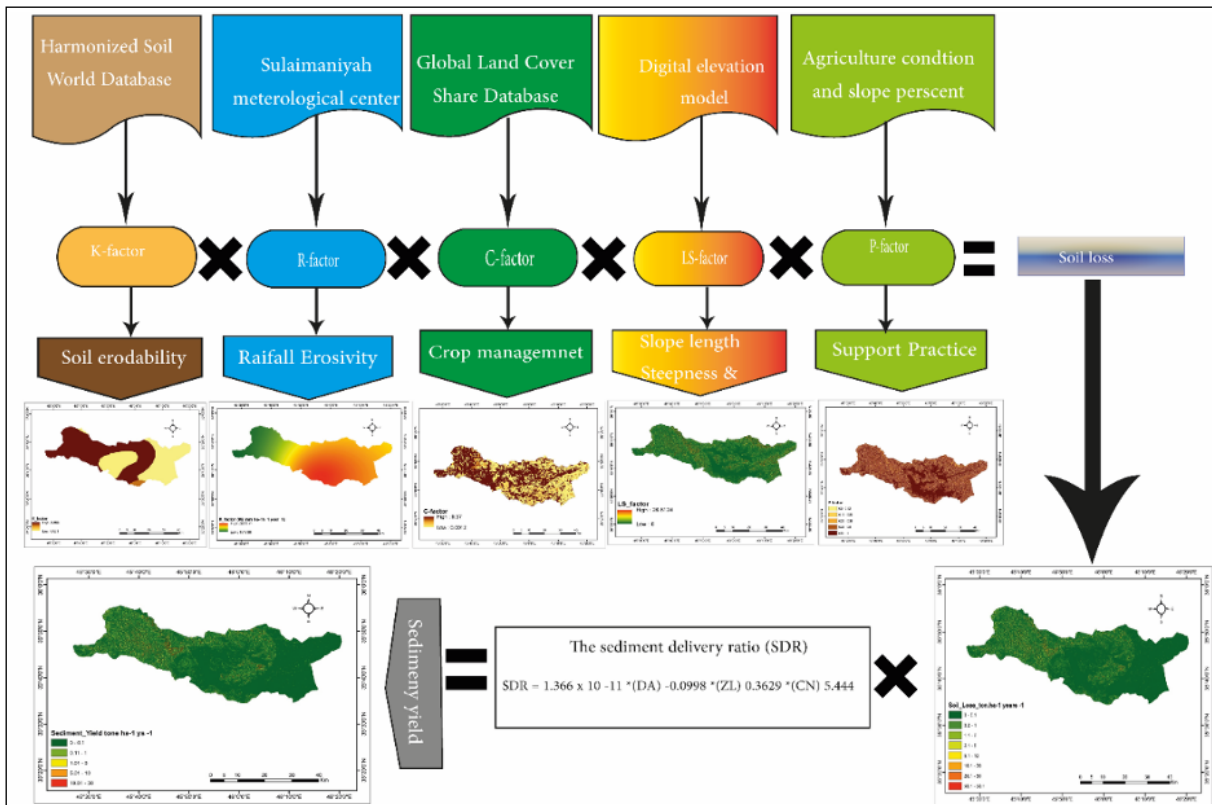


Figure 4- Conceptual modelling of estimation of RUSLE parameters.

Table 1- Rain gauge stations, source (Sulaymaniyah Directorate of Meteorology and Seismology).

No.	Gauging station	Location		Altitude(m)	Mean annual rainfall
		Longitude Latitude			
1	Penjween	45.945478	35.627854	1280	950
2	Chwarta-Mawat	45.409533	35.902678	1160	700
3	Dokan	44.958625	35.952052	540	640
4	Sulaymaniyah	45.435350	35.566579	880	650

and Smith, 1978). The soil types in the study area are classified into four classes-based FAO soil maps. The K_s value for each soil class is calculated based on soil types, composition, and texture Table 2.

3.4. Topography (LS_T)

The topographic factors, such as slope length (L), represent the length between the starting and end of

the inter-rill erosion process, and slope steepness (S) depends on steepness only. The LS factor is crucial in water movement (volume and velocity) in streams, considerably inducing soil erosion and Sediment transporting capacity (Gashaw et al., 2017). The LS factor in the RUSLE was estimated using spatial analysis techniques based on Equation 6 and Equation 7 (Simms et al., 2003).

Table 2- KS value for Soil types based on (Buringh, 1960).

Soil classification	Soil Texture	Ks
Lithosols	Clay and Loam.	0.001
Chromic vertosols	Loam to clay with variable gravel	0.065
Calcic xerosols	Clay marl gravel	0.065
Lithosols, Calcic, xerosols	Silty clay gravel	0.0041

$$LST = [A/22.13]^{0.6} * [\sin B/0.0869]^{1.3} \quad (6)$$

where A is the upslope contributing area factor, and B is the slope angle. Incorporating the DEM into GIS accurately determined the slope gradient S and length L. This study computed the LS_T -value based on a modified Equation 7 suggested by (Simms et al., 2003; Gelagay and Minale, 2016; Chuenchum et al., 2020) because upslope contributing and basin geometry is more influenced than slope length and steepness topsoil loss.

$$LS_T = \text{Pow} \left(\frac{\text{Flow accumulation} * (\text{DEM resolution})}{22.13} \right)^{0.6} * \text{Pow} \left(\frac{\sin(\text{DEM slope}) * 0.01745}{0.0869} \right)^{1.3} \quad (7)$$

Where Pow (power) is a function in the ArcGIS Spatial Analyst (Ashiagbor et al. 2013), the flow accumulation number of cells contributes to downward flow.

3.5. Cropping Types (C_v)

The cover management factor is the soil loss ratio soil loss rate and land cover types (Wischmeier and Smith, 1978). Benavidez et al. (2018) prefer using previous research papers on land use/land cover to estimate the C_v factor. This strategy was performed for the estimation C_v factor Table 3.

3.6. Support Soil Resistance Method (P_s)

P_s values range from zero to one, with zero indicating an ideal human-made structure for preventing erosion and one indicating no erosion resistance (Renard and Freimund, 1994). Such

resistance structures include contour plowing, strip cropping, and terracing, controlled slope. (Shin, 1999) assigned P_s -value for five slope percentage categories based on slope percentage Table 4.

Table 4- The P factor value (Shin, 1999).

Slope (%)	Land use	P factor
9–12	Non-Agriculture	0.6
13–16	Non-Agriculture	0.7
17–20	Non-Agriculture	0.8
21–25	Non-Agriculture	0.9
>25	Non-Agriculture	0.95

4. Results and Discussion

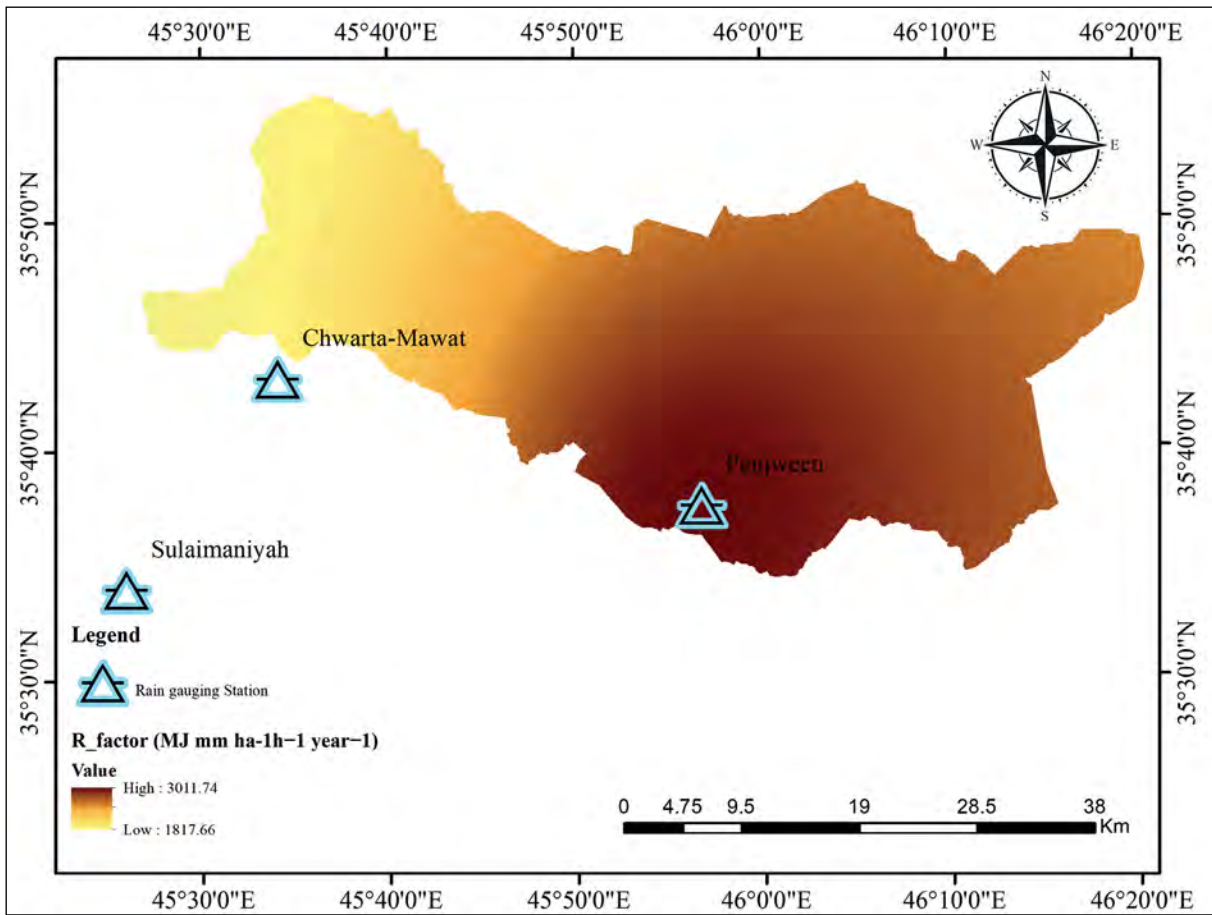
Three different software, including ArcGIS, WMS11.1 (watershed modelling surface), and RStudio Pro V1.1717-3 software, were used to compute the average yearly (soil loss, Sediment yield, and Tolerable Soil loss erosion) as follows.

4.1. R_E Factor

The rainfall in the Kanarwe River basin ranges from 700 mm to 950 m Table 5. Penjween is located within the watershed, while Chwarta-Mawat station is close to the basin's outlet; thus, these two stations significantly weigh R_E factor estimation. A high R_E -value (3011) is presented east, characterized by stormy weather and heavy intense rainfall. The western parts have a low R_E -value (1817), characterized by low terrain with less rainfall Figure 5.

Table 3- C_v Factor Values for LUC types.

Land cover	C	Source
Rainfed croplands	0.07	(Yang et al., 2003)
Mosaic cropland	0.37	(Tiwarly et al., 2014)
Mosaic vegetation	0.25	(Yang et al., 2003)
Closed (>40%) needle-leaved evergreen	0.0015	(Chadli, 2016)
Closed to open (>15%) mixed broadleaved and needle	0.1	(Chen et al., 2019)
Mosaic forest or shrubland	0.01	(Tiwarly et al., 2014)
Mosaic grassland	0.2	(Tiwarly et al., 2014)
Closed to open (>15%) (broadleaved or needle-leaved, evergreen, or deciduous)	0.0012	(Chadli, 2016)
Sparse (<15%) vegetation	0.01	(Yang et al., 2003)
Bare areas	0.35	(Yang et al., 2003)

Figure 5- R_e factor map.Table 5- R_e Factor Value.

Stations	Rainfall (mm)	RE [$MJ\ mm\ h^{-1}ha^{-1}year^{-1}$]
Penjween	950	3011.74
Chwarta-Mawat	700	1838.88

4.2. K_s Factor

The K_s factor was computed based on four soil types: Lithosols, Chromic Vertosols, Calcic xerosols, and Calcic Xerosols (Figure 5). The K values are estimated and compared to previous research on soil types in the northeastern part of Iraq (David, 1988; Hussein et al., 2007; Othman et al., 2021; Zakerinejad and Maerker, 2015). Accordingly, high K_s values (0.065) are presented for chromic and clastic xerosols in the west part and low values of K_s (0.01, 0.04) in the east region Figure 6a and Figure 6b. This substantial variation in K_s value is due to cropping out different rock types in the Kanarwe River basin.

4.3. LS_r Factor

The maximum value of LS_r is (26.6) when slope length and flow accumulation are high due to high altitude mountain and dense stream order in the watershed. The LS_r values become lower closer to the river (flat areas) of the Siwil River in the lower regions Figure 7.

4.4. P_s Factor

The Kanarwe river basin was classified into five classes of Slope percentage (slope %) Figure 8a, and then the P_s -value was assigned for each type of slope percent Figure 8b based on Table 4. Most of the lower P_s -value is given to the central part, which is flatter and contains flood plains. The West of the Kanarwe River basin shows a high P_s -value due to the topographic condition.

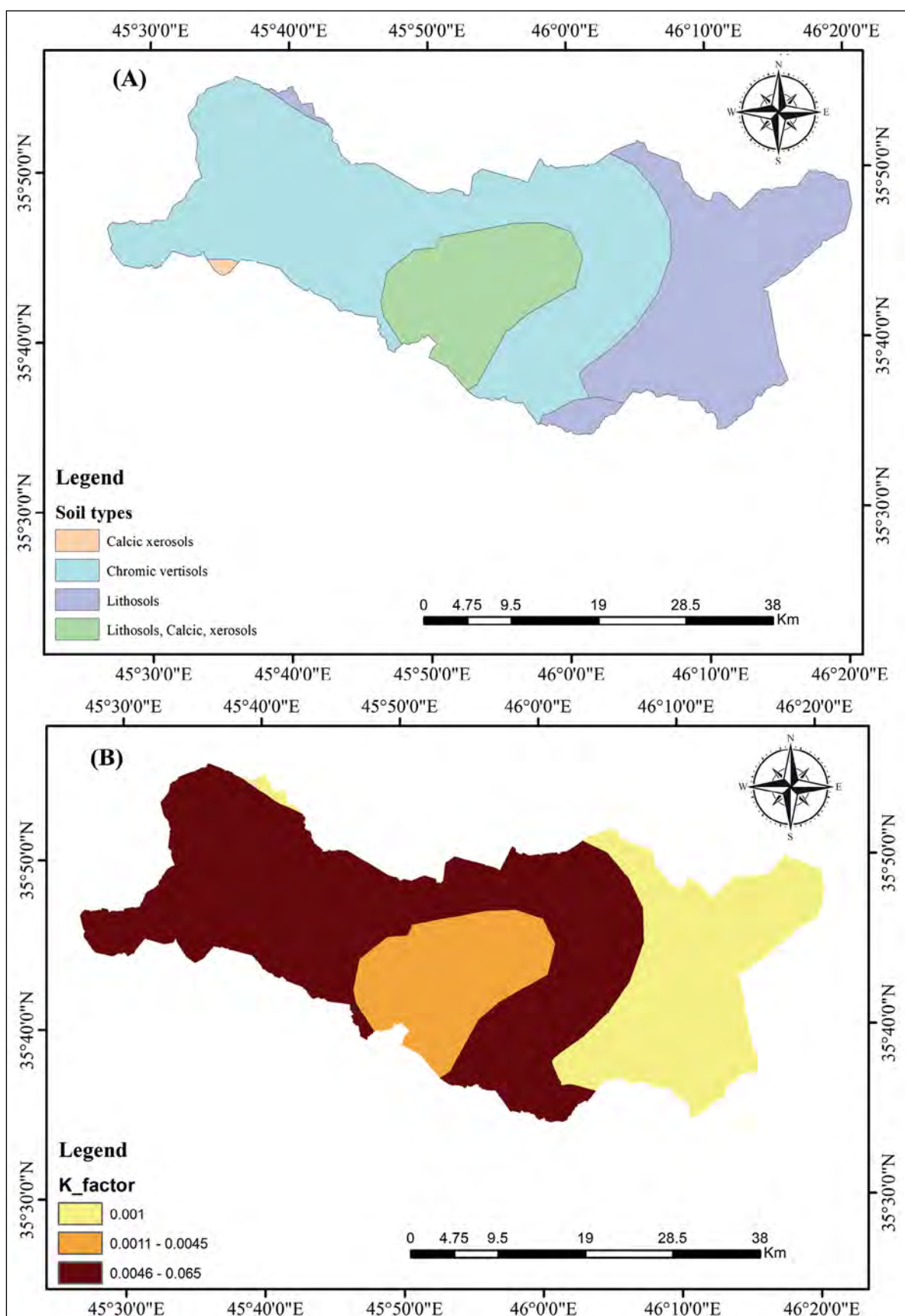
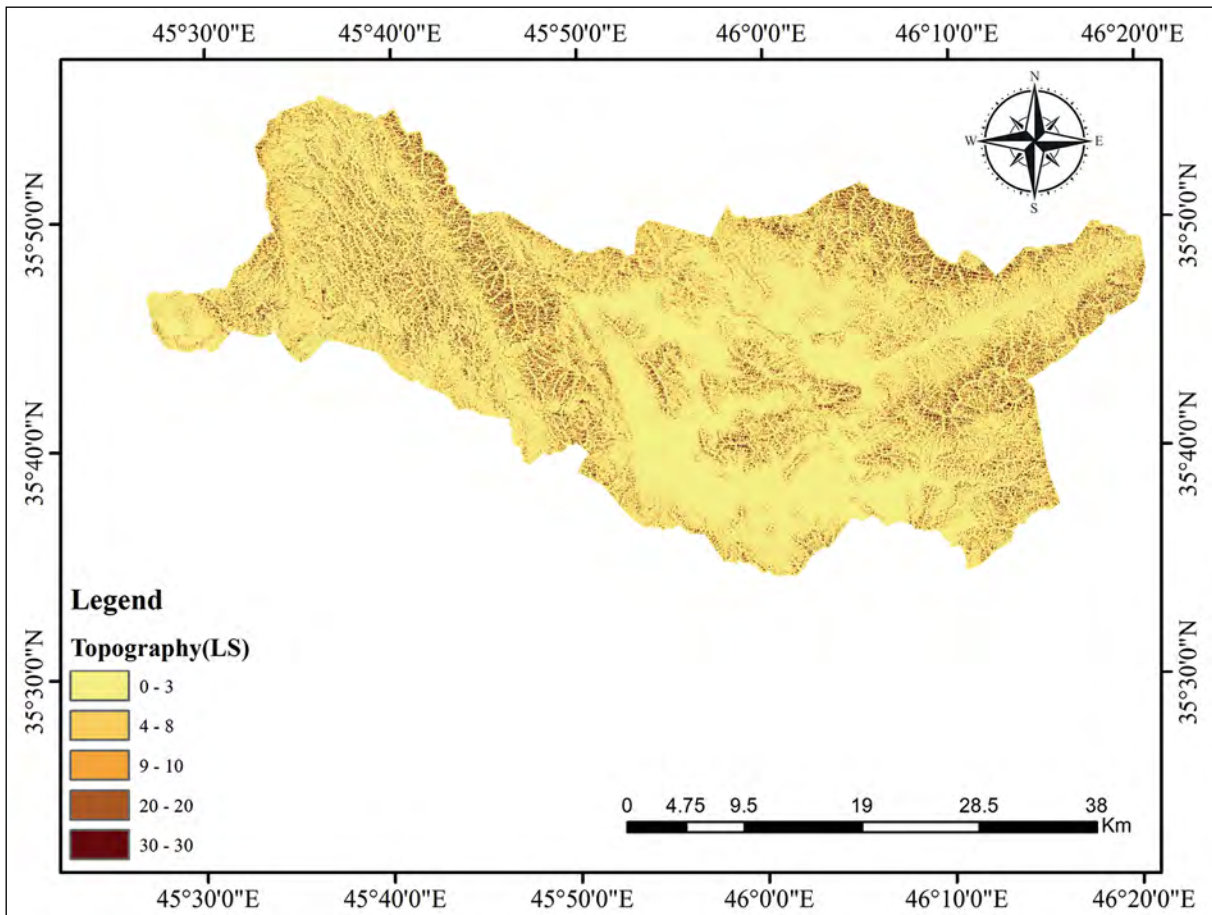


Figure 6- a) Soil map, b) K_s factor map.

Figure 7- LS_r factor map.

The mainland cover/use was (cropland, bare soil, and forests) Figure 9a. The C_v value ranges from (0.0012 to 0.37) and is assigned to land cover types of the Kanarwe river basin Figure 9b. Lower C_v factor values (0.0012) were observed in the eastern and south-eastern parts due to the present forests, especially oak trees. The higher C_v factor values (0.37) can be seen in the western parts, this area covers mostly weathered soils and recent deposits; hence it is more prone to water erosion.

4.5. Soil Loss and Tolerance

The final map of the soil loss was calculated using Equation 2 by integrating RUSLE layers (Parameters) to produce a grid map Figure 10. The soil loss rate expands from 0 in the east part (unweathered hard igneous) to $58.1 \text{ t ha}^{-1} \text{ yr}^{-1}$ in the central and west parts of the high weathered, sedimentary rock. The soil loss map was reclassified into five soil-lowering

risk zones Table 6, using Gelagay and Minale, 2016 classification.

The calculation means annual soil loss is useless scientifically without the calculation of soil erosion risk classes (Pennock, 2019), so soil erosion risk was calculated and reclassified. The total annual soil loss is $1037289 \text{ t ha}^{-1} \text{ yr}^{-1}$; 404512, which covers 75% of the Kanarwe river basin, is below soil erosion risk (3.9 mm yr^{-1}) (Pennock, 2019). The FAO's (2019) tolerable soil loss rate is $4.2\text{-}7.2 \text{ t ha}^{-1} \text{ yr}^{-1}$ (Montgomery, 2007; Verheijen et al., 2009).

Most of this low tolerable soil risk erosion is in the eastern and central parts due to the crop out of the un-weathered, hard rock of igneous rock and thin soil depth. The moderate to severe soil risk classes, which cover 25 % of the Kanarwe river basin, have an annual loss equal to 632777, above FAOs tolerable soil rate. This moderate to severe tolerable soil rate of

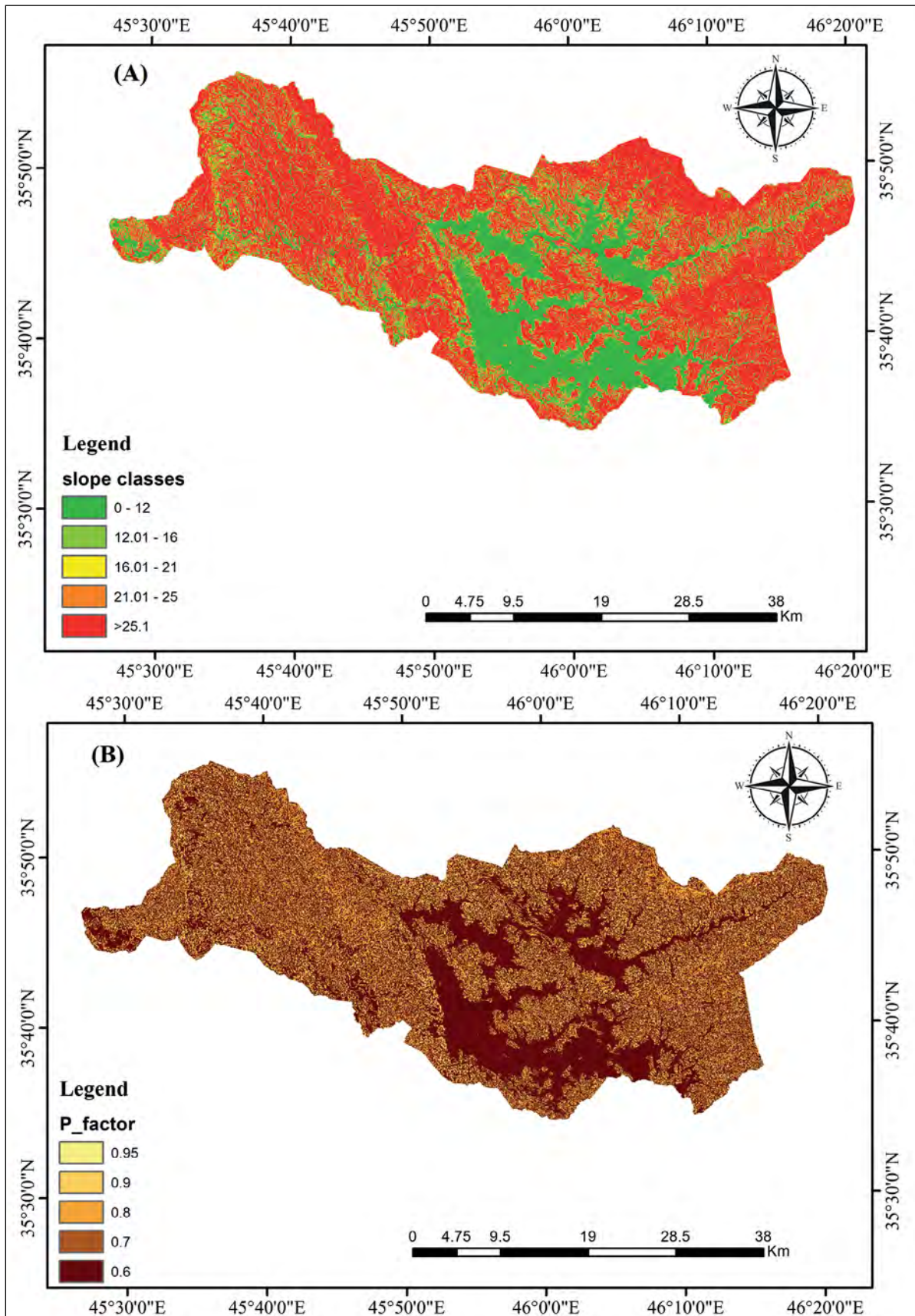


Figure 8- a) Slope class map, b) P factor map.

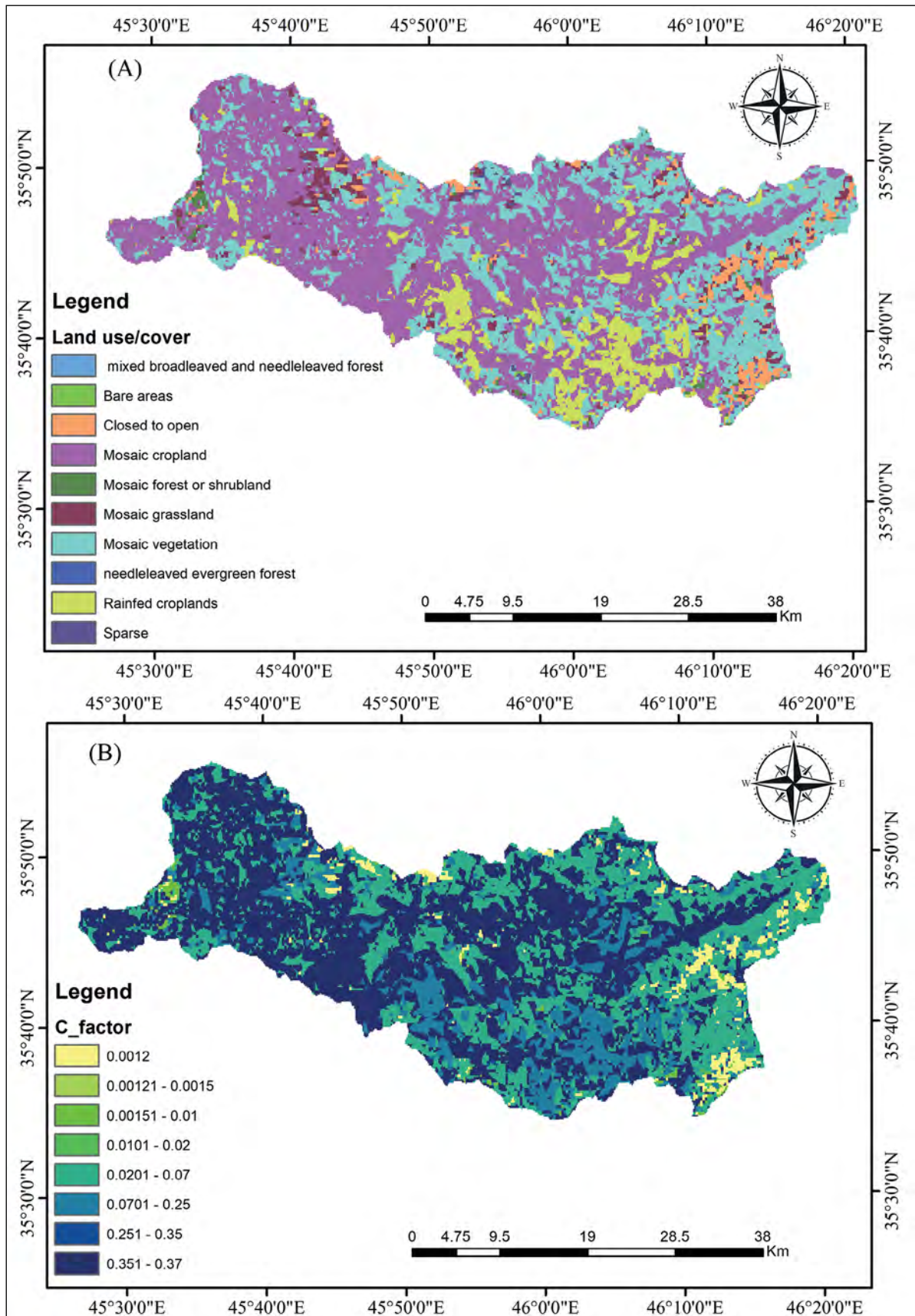


Figure 9- a) Land cover map, b) C_v factor map.

Table 6- Soil Loss class, covered area, and soil risk class.

Soil loses	Soil erosion risk	Area (ha)	Percent of total area	Annual soil Loss(ton)	Percent of total soil loss
0-7.0	Low	115575	74.4	404512	38.99
7.1-15	Moderate	22498.6	14.6	235200	22.67
15.1-25	High	10170.6	6.6	203412	19.6
25.1-45	Very high	4160.7	2.7	145624	14.03
45-60	Sever	924.6	0.6	48541	4.67

the western and central part is spatially situated in the narrower, steep slope close to the outlet Figure 10.

4.6. The Sediment Delivery Ratio (SDR)

The SDR 0.5022 tons is calculated based on Equation 2 and Table 7. The drainage area and relief-

length ratio are related to the time and rate of peak flow and the runoff. The SCS-CN parameter is more sensitive to the basin's soil types and land cover and calculates daily surface runoff (USDA, 1986; Mishra et al., 2006; Kayet et al., 2018). the CN values for each land-use class are calculated and then listed in the supplementary material Table 8 and converted in Grid format Figure 11.

Table 7- Drainage Basin parameters.

Drainage area (DA)	1543 Km ²	ArcGIS 18.1
Relief-length ratio (RL)	3.122	WMS 11.1

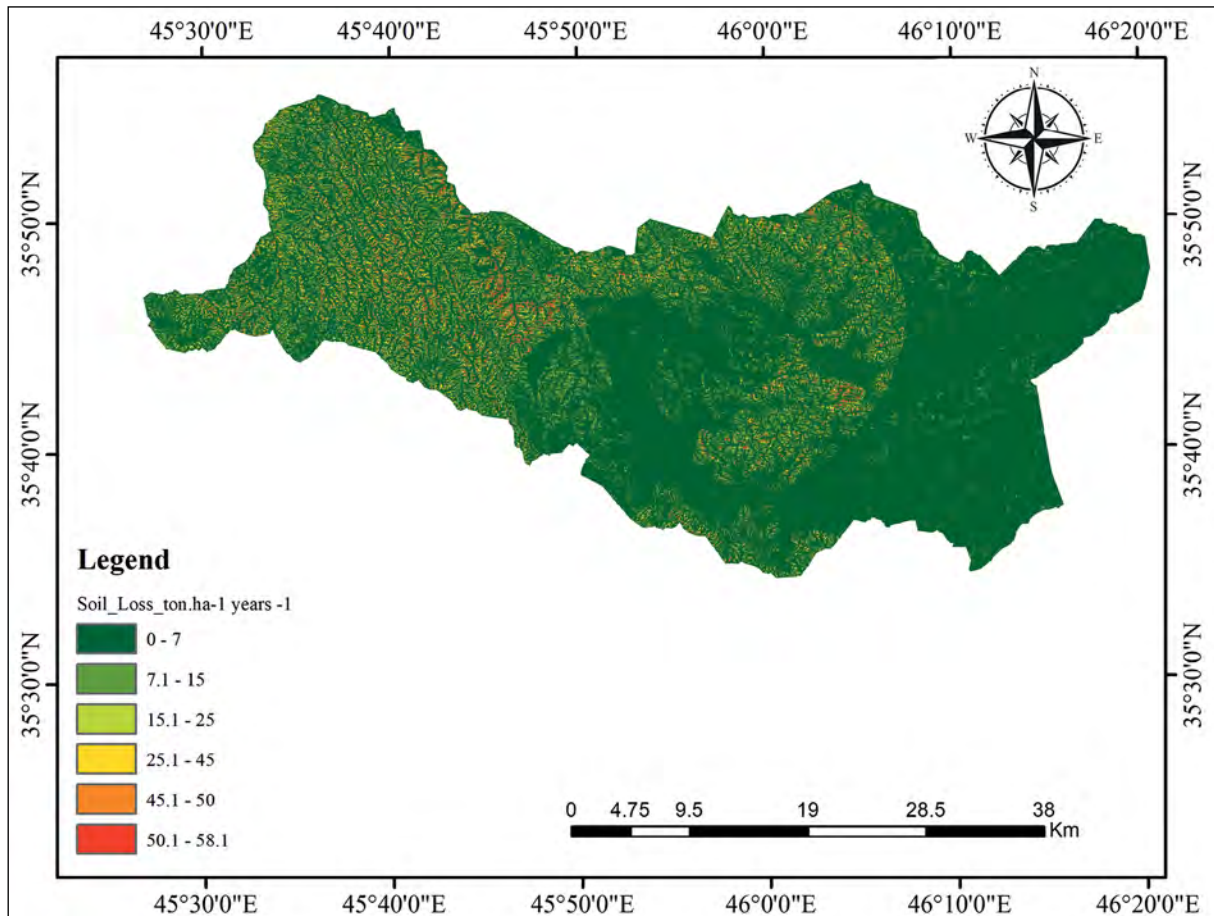
Figure 10- S_{loss} map.

Table 8- Runoff Curve Number values for each class of landcover types.

HSG (Harmonized Soil Group)	Land Use Description	CN code	Area (km ²)	Product (CN x A)
D	Sparse (<15%) vegetation	94	481.359	45247.74
D	Closed to open (>15%) (broadleaved or needleless)	77	21.111	1625.541
D	Rainfed croplands	84	72.466	6087.131
D	Mosaic vegetation (grassland/shrubland/forest)	79	114.837	9072.155
D	Mosaic forest or shrubland (50-70%) / grassland	77	22.009	1694.713
D	Mosaic cropland (50-70%) / vegetation (grassland	84	123.072	10338.06
D	Bare areas	98	13.176	1291.21
D	Mosaic grassland (50-70%) / forest or shrubland	77	16.919	1302.738
D	Closed (>40%) broadleaved deciduous forest (>5)	79	1.946	153.765
D	Closed (>40%) needle-leaved evergreen forest (>)	79	1.797	141.937
B	Sparse (<15%) vegetation	86	181.614	15618.79
B	Mosaic vegetation (grassland/shrubland/forest)	60	120.227	7213.646
B	Mosaic grassland (50-70%) / forest or shrubland	56	39.826	2230.271
B	Mosaic cropland (50-70%) / vegetation (grassland)	69	105.105	7252.274
B	Rainfed croplands	69	155.562	10733.78
B	Closed to open (>15%) (broadleaved or needleless)	56	22.758	1274.441
B	Mosaic forest or shrubland (50-70%) / grassland	56	37.131	2079.351
B	Bare areas	98	12.277	1203.173
D	Water bodies	0	0.15	0
B	Closed (>40%) broadleaved deciduous forest (>5)	60	5.689	341.368
CN	(Weighted) = Total Product \ Total Area	80.6		

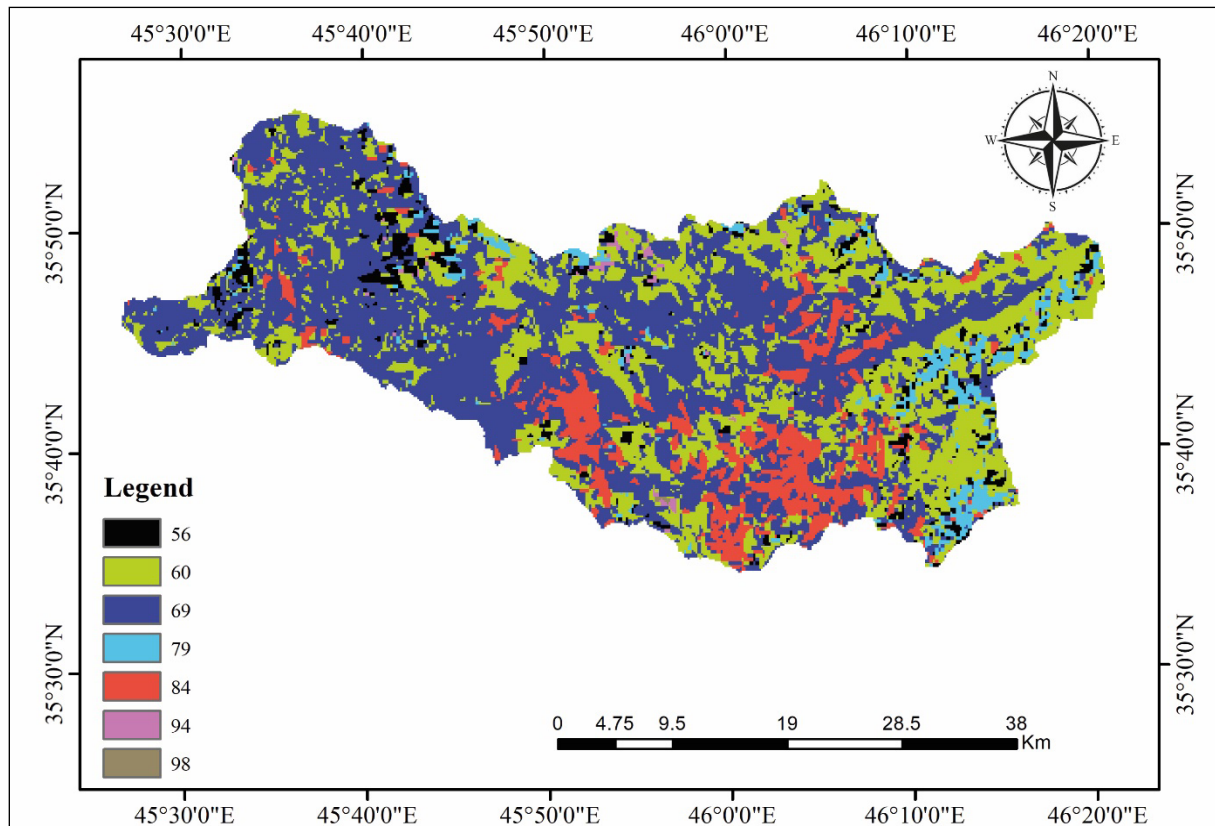


Figure 11- Curve number map.

4.7. The Sediment Yield (SY)

The sediment yield rate is calculated based on Equation 3. It is between 0 to 29.06 $1 \text{ t. ha}^{-1} \text{ yr}^{-1}$ and the mean value is 0.321 Figure 12. The (Sy) becomes higher in the western part compared to the central areas and becomes lower in the east due to geological conditions dominated by hard un-weathered igneous rocks, the obtained results were well matched with the international soil erosion map of the world Global soil degradation map (Source: UNEP, International Soil Reference and Information Centre) and reveals soil degradation states in Iraq and Iran.

5. Correlation Matrix Between RUSLE Variables and Soil Loss

The correlation coefficient analysis among RUSLE parameters shows that the LS_r , K_s , C_v , and

P_s factors showed a linear relationship with soil loss and sediment yield. In contrast, the R-factor showed an inverse relationship between sediment yield and soil loss Figure 13. The topography (LS_r) was more robust, influencing soil loss and sediment yield. A negative R_E value reveals nature and climate conditions in the Kanarwe river basin. (Mohamadi and Kavian, 2015) Conclude that during periods of intense rainfall, the relation between the soil loss-rainfall fitted with nonlinear functions, because it's the beginning rainstorm until 30 minutes there no soil erosion later with prolonged rainfall the soil saturation crosses the limit of plasticity so as a result the weather layers start eroded the erosion decreases when this weathered layer is gone.

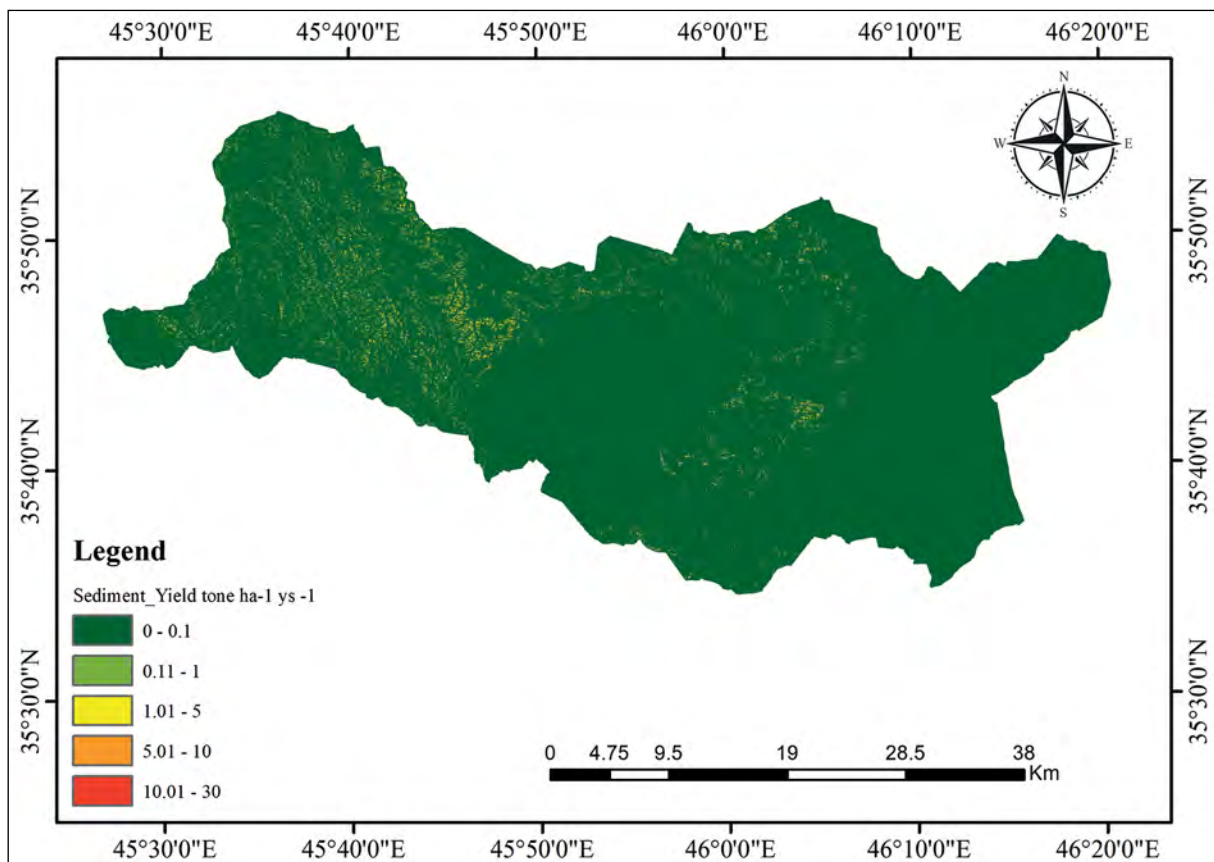


Figure 12- Sediment yield map.

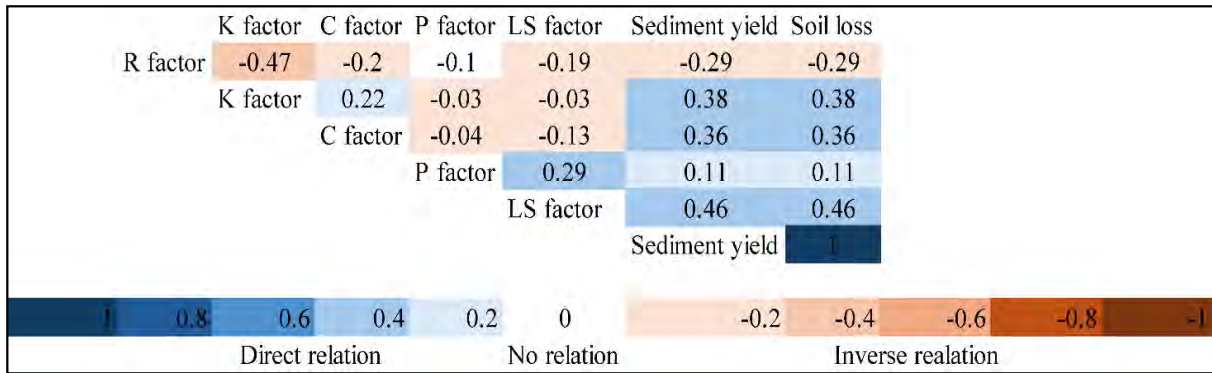


Figure 13- The correlation coefficient between RUSLE parameters.

6. Conclusion

Calculating soil loss, sediment yield, and soil erosion risk in a significant watershed like the Kanarwe river basin in Iraq by the RUSLE equation is very important. It guarantees the construction of any infrastructure, such as dams, railroads, urbanization, food production, and agriculture in the present and future. This study's findings include a spatially distributed mean annual soil loss, sediment yield, and tolerable soil loss in the Kanarwe river basin. The annual soil loss rate is extended from 0 to 58 t ha⁻¹ yr⁻¹, while the sediment yield is between sediment 0 to 30 t ha⁻¹ yr⁻¹. About 25 % of the watershed is moderate to severe, above FAOs tolerable soil loss rate, and 75% is below FAOs tolerable soil loss, this result reveals that the soil condition is degradation condition in Iraq and Iran. However, the main factors controlling the soil loss are topography, soil erodibility, and crop factors instead of rainfall erosivity R factor due to the geological condition Kanarwe river basin. The statistical analysis reveals that topography flysch rocks denudation makes the soil in ZSZ more vulnerable to weathering and erosion but less by anthropogenic factors. Finally, this study recommends the construction of sand dams in narrow gorges and artificial plantations at steep slopes upstream to control soil erosion risk effectively. The estimation of gully erosion is recommended because the RUSLE does not consider it.

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Tectono-stratigraphy, formation and emplacement ages of the Beyşehir-Hoyran Nappes in the south of the Sultan Dağları (Isparta, SW Türkiye)

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Research Article

Keywords:

Beyşehir-Hoyran Nappes,
Sultan Dağları, Gülbahar
Nappe, Metamorphic
Sole.

ABSTRACT

The Beyşehir-Hoyran Nappes (BHN), in the south of Sultan Dağları, consist of ophiolite, mélange and nappe slices of different lithologies and ages belonging to the oceanic crust and related rocks, which originated from the northern Neotethys and were thrust southwards over the Taurus Platform. These nappes, namely the Marmaris Ophiolite Nappe, the Gülbahar Nappe and the Domuzdağ Nappe, are represented by three tectonically related Nappe-slices that extend as a narrow NW-SE trending belt to the south of the Sultan Dağları. The Marmaris Ophiolite nappe (Upper Cretaceous) is formed by three subunits namely the Marmaris Ophiolite, the Kızılcadağ Mélange and the Yenice kale metamorphics. Hornblende minerals from amphibolites of the Yenice kale metamorphic rock unit yielded a ⁴⁰Ar-³⁹Ar age of 93.9±0.34 Ma (Cenomanian-Turonian boundary). In addition, the Middle Triassic-Early Jurassic Orluca Formation of the Gülbahar nappe is mapped for the first time in the Sultan Dağları region. Paleontological and radiometric ages obtained from this study show that the formation of the BHN should began in the Turonian and ended in the Late Maastrichtian. The nappes, on the other hand, were emplaced over southern Sultan Dağları in the early-middle Paleocene and reached their present position as a result of late Eocene movements.

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1. Introduction

Türkiye, which is situated on the Alpine-Himalayan orogenic belt, has been significantly shaped by the closure of the Neotethys Ocean during the Late Cretaceous-Early Cenozoic and the subsequent collision between the Anatolian and Arabian plates by the end of the Middle Miocene (Şengör and Yılmaz, 1981). During this process, several suture zones developed in Türkiye. The İzmir-Ankara-Erzincan suture zone (Şengör and Yılmaz, 1981; Görür et al., 1983; Okay and Tüysüz, 1999) and the Inner-Tauride suture zone (Görür et al., 1984; Robertson and Dixon,

1984; Okay and Tüysüz, 1999), formed by the closure of the northern branch of the Neotethys Ocean, constitutes the northern boundary of the Anatolide-Tauride Block (Okay and Tüysüz, 1999), while the Bitlis-Zagros suture zone (Şengör and Yılmaz, 1981; Yılmaz, 1993; Parlak et al., 2009), formed by the closure of the southern branch, defines the southern boundary.

The Anatolides (*sensu* Ketin, 1966), located in the Anatolide-Tauride Block, represent those parts of the Taurides that underwent regional metamorphism during the Late Cretaceous-Early Cenozoic due to

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the Alpine orogeny (e.g., Pourteau et al., 2013). The Taurides consist mainly of parautochthonous and allochthonous, imbricated, folded, non-metamorphic or low-grade metamorphic units (Geyikdağ, Aladağ, Bolkardağ, Bozkır, Antalya and Alanya units) (Özgül, 1976). The Sultan Dağları and its surroundings is one of the regions where the relationships between Geyikdağ, Aladağ and Bozkır units can be best observed.

The Sultan Dağları is a mountain range consisting of Paleozoic to Mesozoic low-grade metamorphic rocks and a Cenozoic cover that extends between the Afyon Zone (Okay, 1984; Okay and Tüysüz, 1999; Candan et al., 2005) and the Anamas-Akseki Autochthon (Şenel et al., 1992) with a NW-SE direction and forms the NE flank of the Isparta Angle (*sensu* Poisson et al., 1984). The NW-SE trending allochthonous units extending south of the Sultan Dağları are referred to as the BHN (Gutnic et al., 1968; Monod, 1977) (Figure 1). The BHN is composed of

ophiolites, mélanges and rocks of different ages and lithologies, derived from the northern branch of the Neotethys Ocean (İzmir-Ankara-Erzincan and/or Inner Tauride Oceans) and emplaced on the Taurus platform (Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Elitok and Drüppel, 2008). The BHN can be regarded as the eastern continuation of the Lycian Nappes (*sensu* Şenel et al., 1989), which lies west of the Isparta Angle.

Units of the BHN are genetically represented in the northern branch of the Neotethys by rift-related rocks in the Triassic, by carbonate and clastic sediments of the passive continental margin in the Jurassic-Cretaceous, and by supra-subduction ophiolites and mélanges associated with northward subduction in the Late Cretaceous (Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Elitok and Drüppel, 2008; Mackintosh and Robertson, 2009; Parlak et al., 2019). The formation and emplacement processes of the BHN are key to understanding the geodynamic evolution of

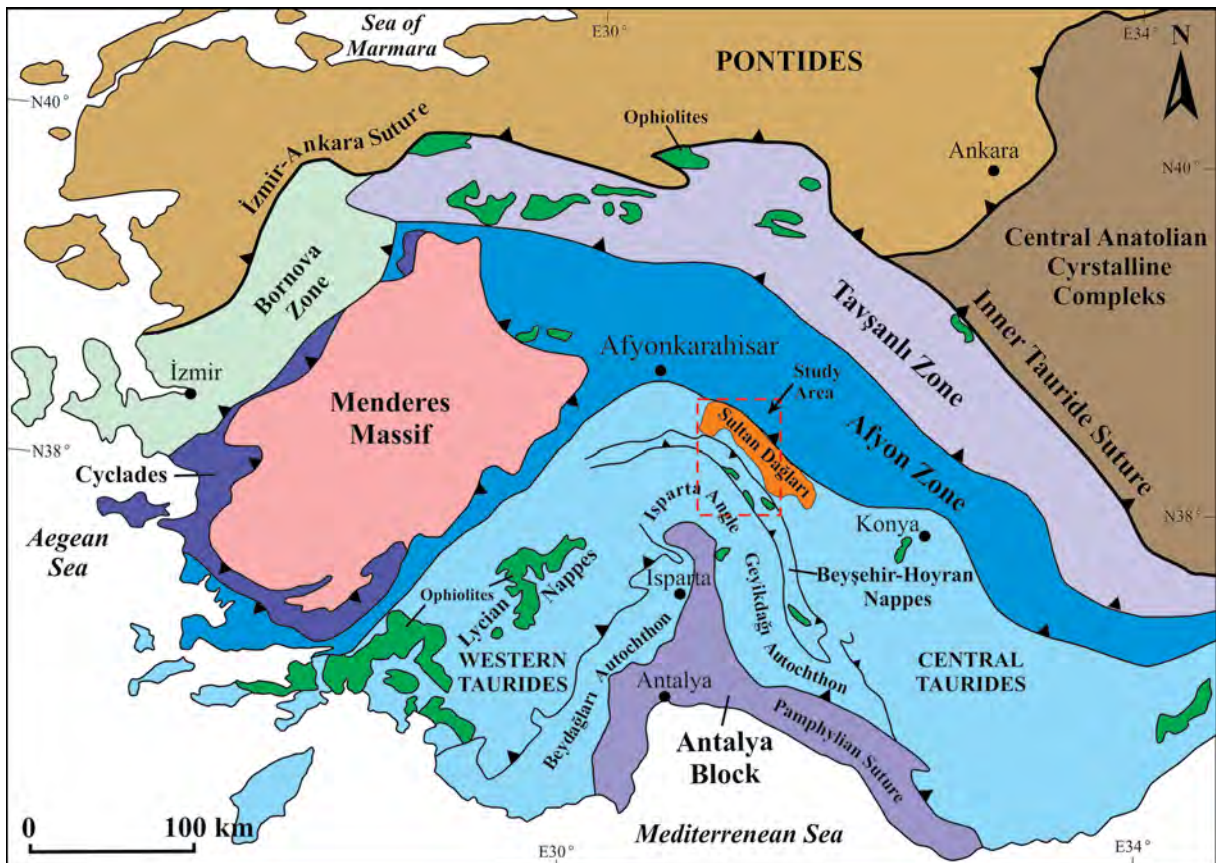


Figure 1- Tectonic map of the western Anatolia (modified after Pourteau et al., 2010, 2013).

the region. The general assumption about these nappes is that they were formed in the Late Cretaceous due to the closure of the northern branch of the Neotethys and emplaced on the Taurus platform during the Late Cretaceous-Eocene interval (Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Elitok and Drüppel, 2008; Güngör, 2013). Constraining these too-long intervals by paleontological and radiometric dating provides more accurate data on the formation and emplacement processes of the BHN. In addition, the majority of present studies of the BHN are concerned with the age and geochemical properties of the ophiolites and metamorphic sole rocks (e.g. Elitok and Drüppel, 2008; Çelik and Delaloye, 2006; Parlak et al., 2019). Thus, the tectono-stratigraphy of the nappes and their relationship to the cover rocks has not been sufficiently investigated.

This study aims to provide a detailed tectono-stratigraphy of the BHN and to constrain the formation and emplacement ages of these nappes by integrating the biostratigraphic, radiometric and structural data.

1.1. Material and Method

Basic studies to identify tectono-stratigraphic features and solve geological problems are based on the production of geological maps at a scale of 1:25,000 in the field. The paleontological and petrographic samples studied in the MTA laboratories were collected in the field from appropriate levels of the formations. Radiometric dating studies were carried out at the Nevada Isotope Geochronology Laboratory (USA) using the ^{40}Ar - ^{39}Ar method on hornblende minerals from amphibolites of the Yenicekale Metamorphics.

2. Tectono-stratigraphy

Early Cambrian to Quaternary rock units of different origins and lithologies outcrop in the study area (Figure 2). Formations of the Geyikdağı, Bolkardağı/Aladağ and Bozkır units form the basement of the region (Özgül, 1976).

The widespread parautochthonous rocks of the Geyikdağı Unit (Özgül, 1976) are studied under the names of Sultandağı Unit (*sensu* Özgül et al., 1991) and Anamas-Akseki Autochthon (*sensu* Şenel et al., 1992) in the study area, while the allochthonous rock assemblages of the Bolkardağı/Aladağ Unit and the

Bozkır Unit are studied under the names of Çay Unit (*sensu* Özgül et al., 1991) and the BHN (*sensu* Monod, 1977) respectively (Figures 2 and 3).

The late Paleocene-Lutetian Celeptaş Formation and deposits of the Miocene-Pliocene Yalvaç and Ilgın basins form the Cenozoic cover. Quaternary deposits are the youngest cover in the region. As the units of the BHN observed in the study area are a continuation of the Lycian Nappes and show strong stratigraphic similarities, the nomenclature of the Lycian Nappes is followed to ensure regional correlation.

2.1. Sultandağı Unit

The Sultandağı Unit forms the dominant rock mass of the Sultan Dağları. The unit consists of lower Cambrian to Upper Cretaceous metasedimentary and metavolcanic rocks (Özgül et al., 1991; Ergen et al., 2021). The Sultandağı Unit, which represents a transgressive sequence associated with back-arc basin development in the early Paleozoic (Linnemann et al., 2008; Nance et al., 2010; Dedeoğlu et al., 2021), is composed of quartzite, marble and turbiditic clastic rocks. An upper Paleozoic succession of quartzite, phyllite, recrystallized limestone and dolomite overlies the early Paleozoic units after the Late Devonian unconformity. The Mesozoic succession begins in the Middle Triassic with terrestrial metaconglomerates and metasandstones, overlain by Jurassic-Cretaceous metacarbonate rocks. The Sultandağı Unit underwent lower greenschist metamorphism (Güngör, 2013) in the early-middle Paleocene based on the emplacement of the Çay Unit and the BHN (Ergen et al., 2021; Ergen, 2023). The late Paleocene-Lutetian Celeptaş Formation and the Neogene deposits of the Yalvaç and Ilgın basins unconformably overlie the Sultandağı Unit.

2.2. Anamas-Akseki Autochthon

The unit consists of non-metamorphic Jurassic-Cretaceous aged (Şenel et al., 1992) neritic carbonates belonging to the Geyikdağı Unit in the south of the Sultan Dağları. The Anamas-Akseki Autochthon (*sensu* Şenel et al., 1992), observed in the SW parts of the study area, is represented by Jurassic-Cenomanian grey-colored, medium to thick-bedded limestones and dolomites. The lower contact of the unit is not seen in

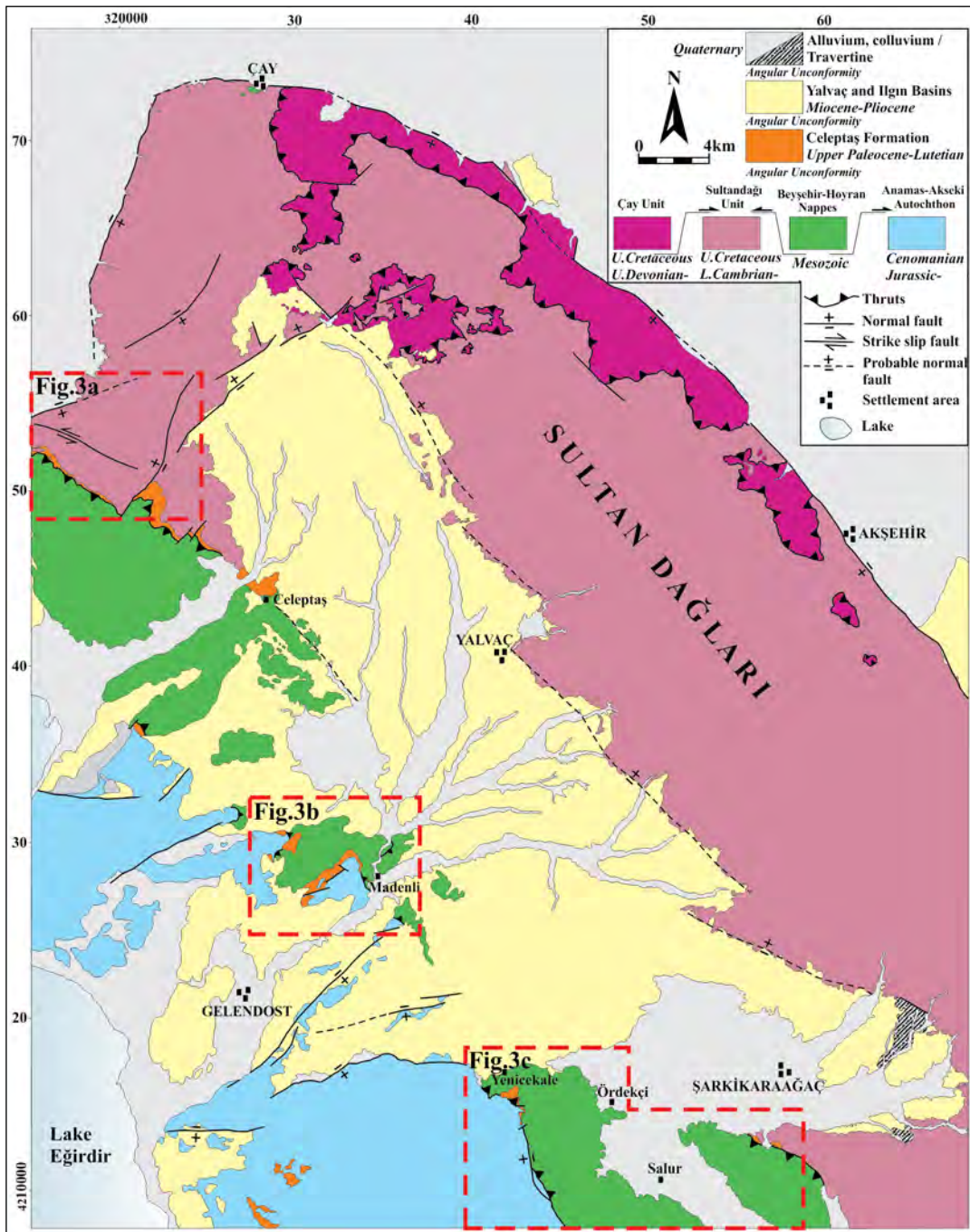


Figure 2- Simplified geological map of the Sultan Dağları and its surroundings (simplified after Ergen et al., 2021).

the study area, being unconformably overlain by the Celeptaş Formation and tectonically overlain in places by ophiolites of the BHN.

2.3. Çay Unit

The Late Devonian to Cretaceous lower greenschist-facies rocks of the Bolkardağı/Aladağ Unit,

outcropping in a NW-SE trending narrow strip along the northeastern edge of the Sultan Dağları, are called the Çay Unit (Özgül et al., 1991; Ergen et al., 2021). The Çay Unit, which consists of metasedimentary rocks intercalated with metavolcanics, tectonically overlies the Sultandağı Unit and is also tectonically overlain by the BHN (Figure 2).

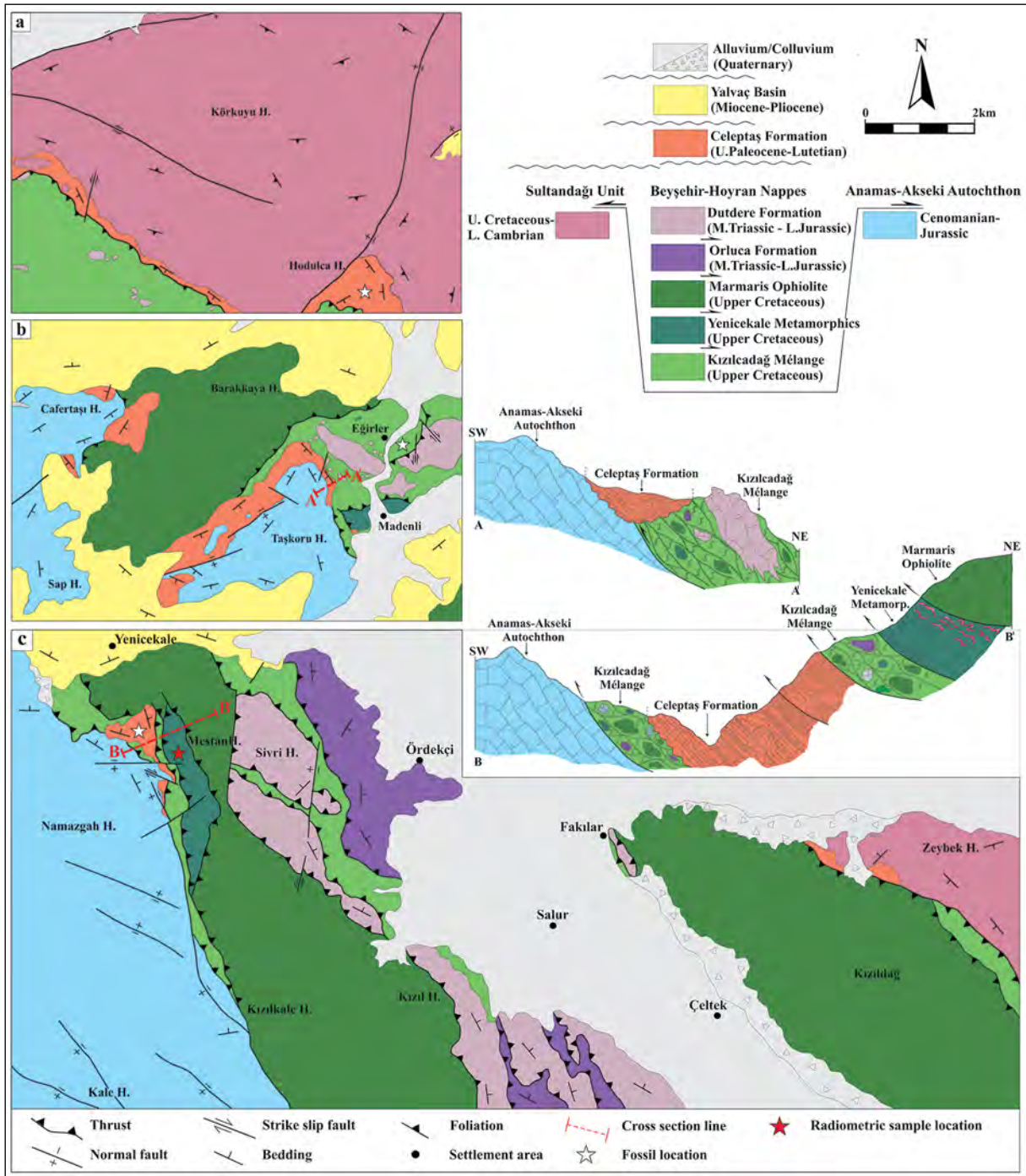


Figure 3- Geological map and cross-sections showing the relationship of the units of the BHN to the parautochthonous basement and cover rocks (modified after Ergen et al., 2021).

2.4. Beyşehir-Hoyran Nappes

The BHN are the allochthonous masses composed of ophiolite, mélangé and associated blocks and slices of different lithologies and ages, formed as a result of the closure of the northern branch of the

Neotethys Ocean and were emplaced on the Taurus Platform during the Late Cretaceous-early Cenozoic (Monod, 1977; Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Ergen et al., 2021). These nappes, which are exposed in a NW-SE trending narrow belt and are tectonically interrelated with each other, are the

Marmaris Ophiolite Nappe (the Kızılcaadağ Mélange, the Yenice kale Metamorphics and the Marmaris Ophiolite), the Gülbahar Nappe and the Domuzdağ Nappe from bottom to top (Ergen et al., 2021; Ergen, 2023) (Figure 4).

The Marmaris Ophiolite Nappe (Upper Cretaceous) is formed by three subunits as the Marmaris ophiolite, the Kızılcaadağ Mélange and the Yenice kale

Metamorphics. The Marmaris Ophiolite is composed of dunite, harzburgite, serpentinite and diabase dykes, while the Kızılcaadağ Mélange is composed by blocks of different ages and lithologies contained in an ophiolitic/sedimentary matrix. The Yenice kale Metamorphics consist mainly of amphibolites that characterize a sub-ophiolitic metamorphic sole (Elitok and Drüppel, 2008). The Orluca Formation (Şenel et al., 1989), which is composed of micritic limestone,

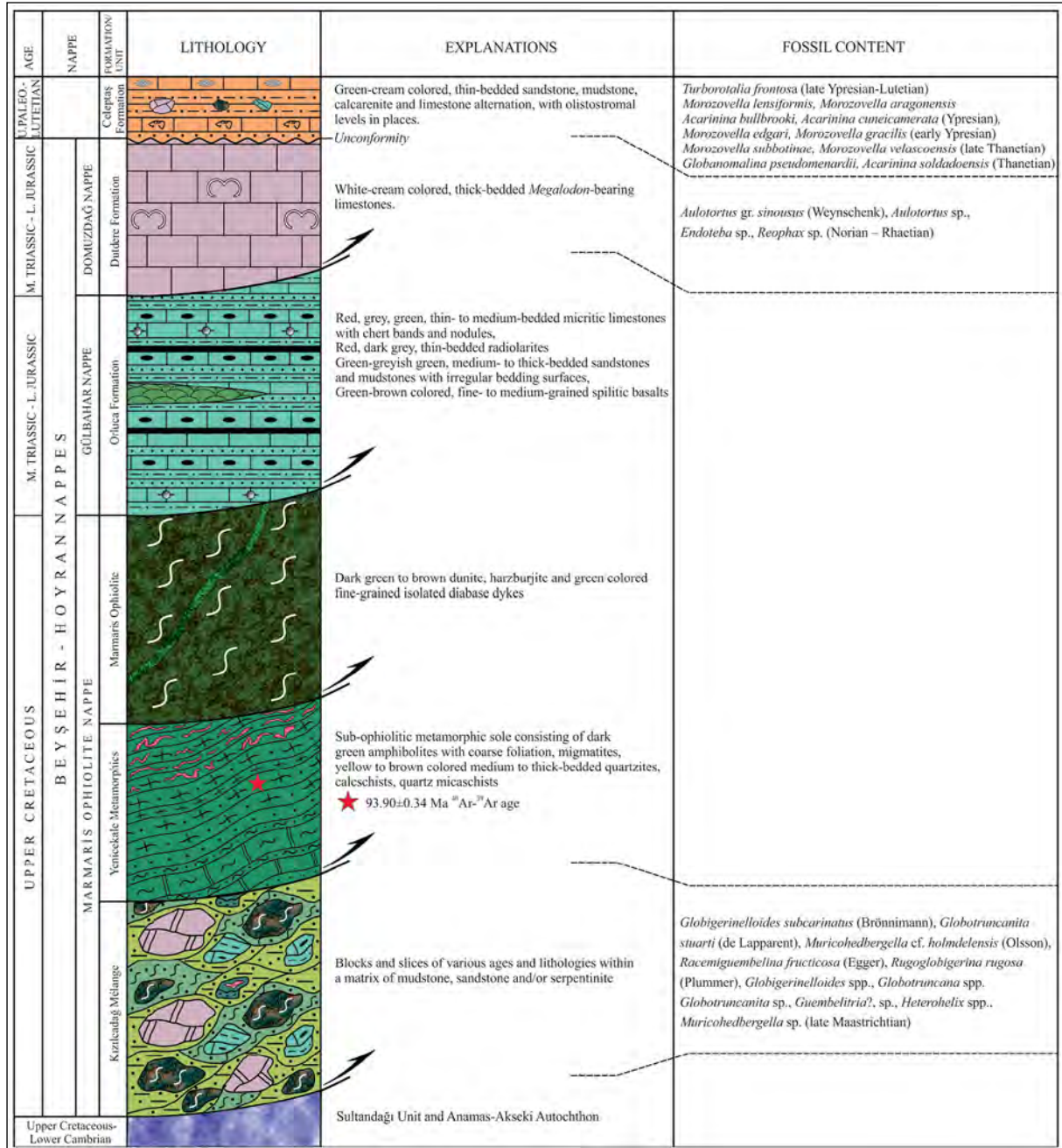


Figure 4- Tectono-stratigraphic column of the BHN in the study area (not to scale).

radiolarite, chert, mudstone and basalt intercalations, represents the Gülbahar Nappe. The Domuzdağ Nappe at the top is represented by the Dutdere Formation (Ersoy, 1989), which consists of *Megalodon*-bearing limestones.

The Celeptaş Formation unconformably overlies the BHN, which tectonically rest on the parautochthonous Sultandağı Unit and the Anamas-Akseki Autochthon.

2.4.1. Marmaris Ophiolite Nappe

Kızıladağ Mélange (Kkm): It is composed of blocks and slices of different lithologies contained in an ophiolitic and sedimentary matrix (Poisson, 1977; Şenel et al., 1989; Ergen et al., 2021). The majority of the matrix consists of green, intensely sheared serpentinites and, to a lesser extent, mudstones. Blocks and slices belonging to the Domuzdağ and Gülbahar Nappes and the Marmaris Ophiolite are observed in the matrix. Blocks and slices of the Domuzdağ and Gülbahar Nappes and the Marmaris Ophiolite, ranging from a few centimetres to tens of metres, are observed in the matrix, and they are composed of such rocks as marble, micritic limestone, chert, radiolarite, dunite, harzburgite, gabbro, diabase and amphibolite (Figures 5a and 5b).

The lower contact of the Kızıladağ Mélange is tectonic. It tectonically overlies the Jurassic-Cretaceous carbonates of the Sultandağı Unit to the

south of Şarkikaraağaç, while tectonically overlying the Jurassic-Cenomanian limestones of the Anamas-Akseki Autochthon to the south of Yenice kale (Figures 5a and 6a-d). The Celeptaş Formation covers the Kızıladağ Mélange with an unconformable contact. In addition, the Marmaris Ophiolite, Yenice kale Metamorphics, Domuzdağ and Gülbahar Nappes tectonically overlie the Kızıladağ Mélange. It is also unconformably overlain by deposits of the Miocene-Pliocene Yalvaç Basin to the south of Yenice kale village (Figure 3).

The age of the mélange in the Lycian Nappes is accepted as Late Cretaceous based on the planktonic foraminiferal assemblages (Poisson, 1977; Şenel et al., 1989). It was also reported that the age of the mélange near Bozkır district (Konya) is late Maastrichtian (Özgül, 1997; Andrew and Robertson, 2002).

In this current study, planktonic foraminiferal assemblage including *Globigerinelloides subcarinatus* (Brönnimann), *Globotruncanella stuarti* (de Lapparent), *Muricohedbergella* cf. *holmensis* (Olsson), *Racemiguembelina fruticosa* (Egger), *Rugoglobigerina rugosa* (Plummer), *Globigerinelloides* spp., *Globotruncana* spp., *Globotruncanella* sp., *Guembelitra*?, sp., *Heterohelix* spp., *Muricohedbergella* sp. species, indicating a late Maastrichtian age, has been identified from the mudstones that forms the sedimentary matrix of the Kızıladağ Mélange around the Eğirler village.

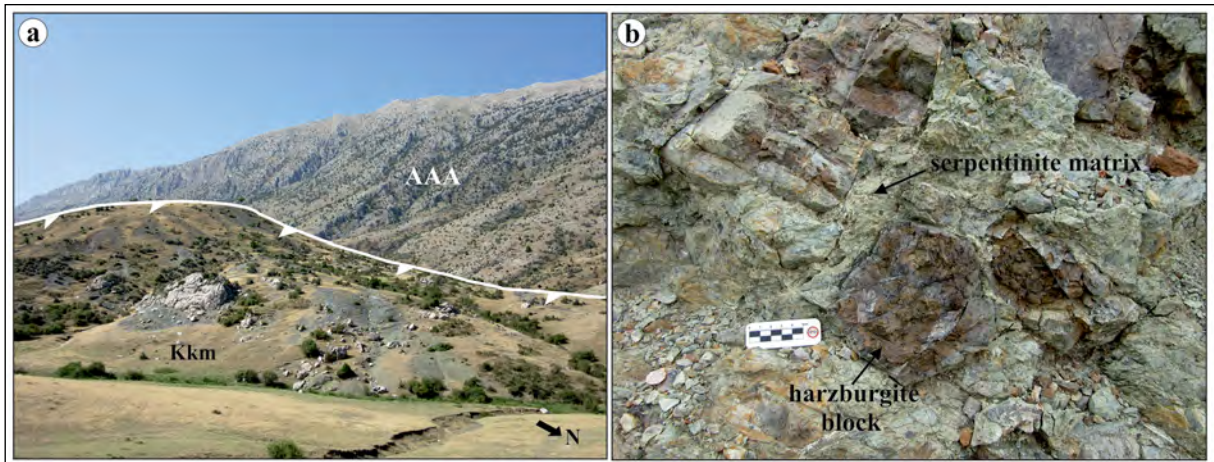


Figure 5- a) General view of the Kızıladağ Mélange (Kkm) and its tectonic contact with the Anamas-Akseki Autochthon (AAA), b) harzburgite blocks in the serpentinite matrix of the mélange, south of Yenice kale village, Şarkikaraağaç.

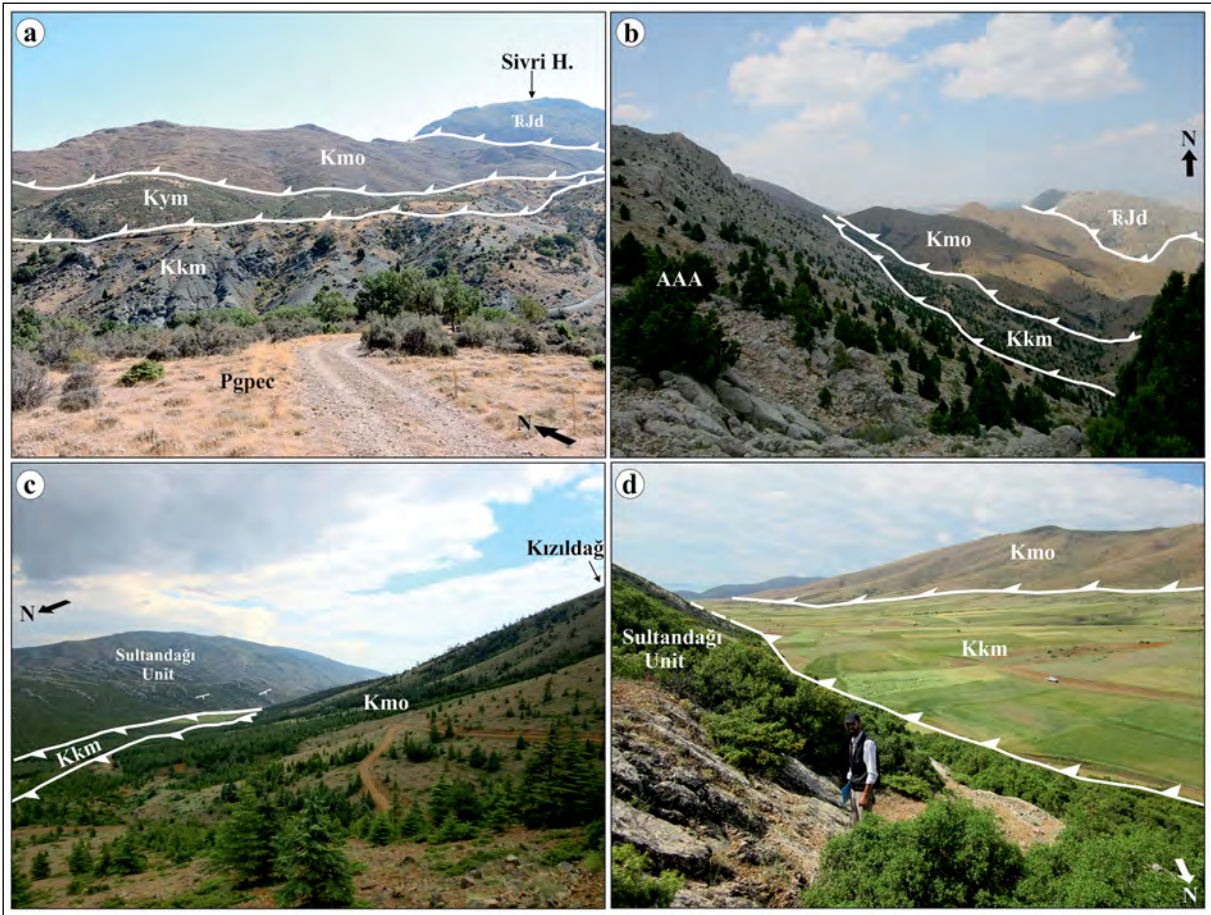


Figure 6- Tectonic relationships of units of the BHN. a, b) SW-vergent thrust contacts, SE of Yenicekale village, Şarkikaraağaç, c, d) NE-vergent back-thrusts, S of Şarkikaraağaç. Kmo: Marmaris Ophiolite, Kkm: Kızıladağ Mélange, Kym: Yenicekale metamorphics, T_RJd: Dutdere Formation, AAA: Anamas-Akseki Autochthon.

The Kızıladağ Mélange was formed by the emplacement of blocks and slices of different lithologies and ages in an ophiolitic/sedimentary matrix during the Late Cretaceous northward subduction of the northern branch of the Neotethys Ocean, and thus gained a chaotic structure depending on intense tectonics.

Yenicekale Metamorphics (Kym): The rock assemblage containing amphibolite, amphibole-biotite schist, calcschist and quartz schist, which are observed as sub-ophiolitic metamorphic sole in the BHN (Elitok and Drüppel, 2008), are referred to as the Yenicekale Metamorphics (Ergen et al., 2020, 2021).

The Yenicekale Metamorphics are composed of dark green, medium to coarsely foliated amphibolites, with pygmatic folds in places (Figures 7a and 7b),

and grey-blue, thin to medium-bedded calcschists and yellow-brown, thin to medium foliated quartzites (Figure 7d) and quartz schists. Amphibolites, which are the dominant lithologies of the unit, are composed of plagioclase, hornblende, chlorite and titanite minerals, with a granonematoblastic to nematoblastic texture (Figure 7f). Sericitization and argillization are common in plagioclase minerals. Quartzites are composed of quartz, feldspar, mica and apatite minerals, with a granoblastic texture. These rocks show a metamorphic grade from amphibolite to greenschist facies (Elitok and Drüppel, 2008). Inverted metamorphic grade, one of the characteristic features of the sub-ophiolitic metamorphic rocks, can be observed within the Yenicekale Metamorphics. The amphibolites are at the top of the sequence, while the greenschist facies rocks such as quartzite, quartz schist and calcschist are at the bottom of the sequence. These

metamorphic rocks are intruded by non-metamorphic isolated diabase dykes, as is in the Marmaris Ophiolite (Figure 7e). These dykes, which yield U-Pb zircon ages ranging from 90.8 ± 1.6 Ma to 87.6 ± 2.1 Ma (Parlak et al., 2019), are geochemically tholeiitic and,

to a lesser extent, alkaline in composition (Elitok and Drüppel, 2008; Parlak et al., 2019).

The Yenicekale Metamorphics are generally observed between the ophiolites and the ophiolitic



Figure 7- a, b) General view of the amphibolites of the Yenicekale Metamorphics, c) tectonic relationships between the Yenicekale Metamorphics (Kym) and the Marmaris Ophiolite (Kmo) W of Mestan Hill, d) general view of the quartzite, quartz schist and calcschist, SE of Yenicekale village, e) an isolated diabase dyke intruding the amphibolites of the Yenicekale Metamorphics (Kym), SE of Yenicekale village, f) thin section view of an amphibolite sample with a nematoblastic texture, under cross polarized light (Amp: amphibole, Plg: plagioclase).

mélange, representing various thicknesses (Figure 6a-b and 7c). It has a thickness of up to 140 metres around Yenicekale village (Elitok and Drüppel, 2008) and has a lenticular geometry.

Hornblende minerals from amphibolites of this unit in the south of Yenicekale village (coordinates: 36342124N/4214144E) yielded a ^{40}Ar - ^{39}Ar age of 93.90 ± 0.34 Ma (Figure 8). Parlak et al. (2019) obtained ages of 90-94 Ma and 91-93 Ma from amphibolites in the same area using U-Pb and ^{40}Ar - ^{39}Ar methods, respectively. These are in agreement with the 91-93 Ma age of Çelik et al. (2006) determined from amphibole and mica minerals of the Lycian Nappes, Antalya Nappes and metamorphic basement rocks of the BHN.

Based on the analysis of the amphibolites, P-T conditions of 630-770°C and 6 ± 1.5 kbar, corresponding to a burial depth of 18-20 km, are calculated for the metamorphism of these sub-ophiolitic metamorphic

sole rocks (Elitok and Drüppel, 2008). The amphibolites show two different geochemical characters, alkaline and tholeiitic. According to this, the protoliths for alkaline amphibolites are within-plate type alkaline basalts, while the tholeiitic amphibolites are ocean island basalts (Elitok and Drüppel, 2008; Parlak et al., 2019).

Marmaris Ophiolite (Kmo): The ophiolitic rocks outcropping in the Lycian Nappes and the BHN, which are continuations of each other in southwestern Anatolia, have been defined under different names by various researchers. Various names have been given to the rock association, which consists mainly of dunite, harzburgite and serpentized peridotite were referred to various names such as Beyşehir Ophiolite (Ricou et al., 1975; Çelik and Delaloye, 2006) and Hoyran Ophiolite (Demirkol and Yetiş (1983-1984) within the BHN; ophiolites (Andrew and Robertson, 2002) and peridotites (Elitok and Drüppel, 2008) within the Hoyran Nappes; Peridotite Nappe (Graciansky, 1972),

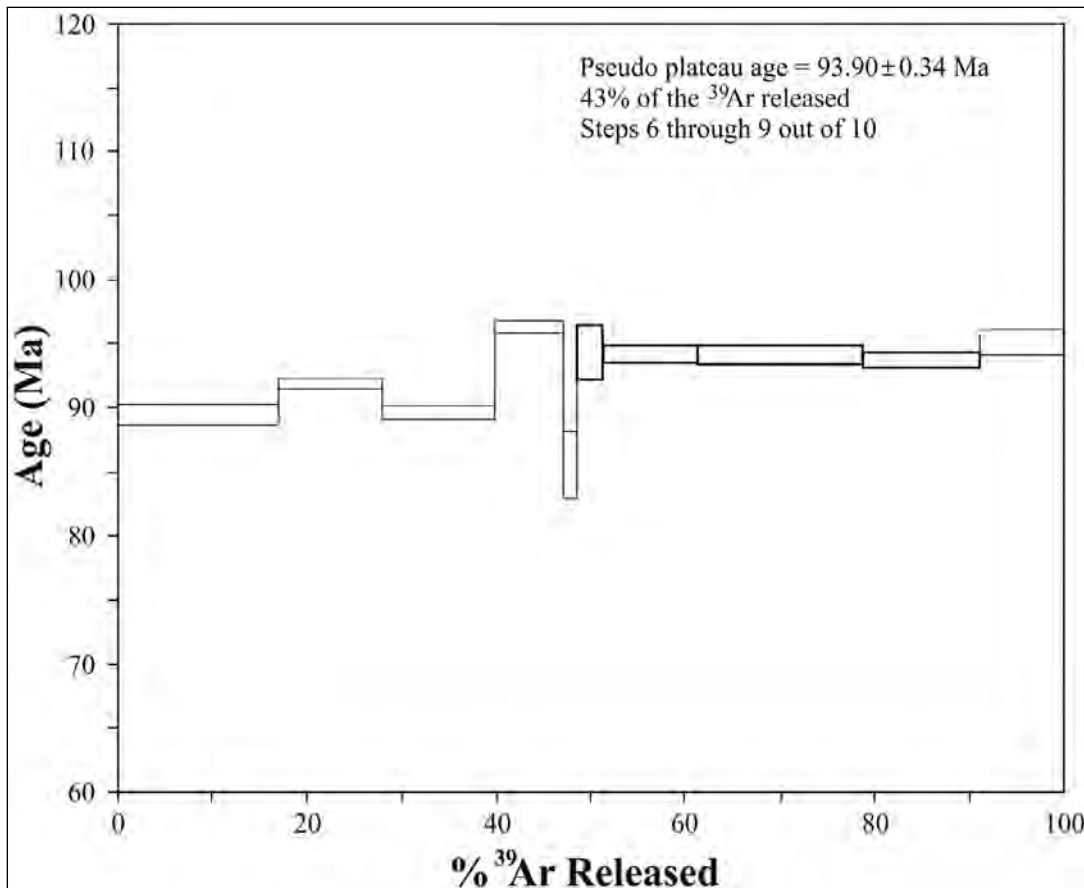


Figure 8- Age spectrum for the amphibolite of the Yenicekale Metamorphics.

Yeşilova-Tefenni Ophiolites (Sarıkaya and Seyrek, 1976), Yeşilova Ophiolites (Sarp, 1976), Marmaris Peridotite (Çapan, 1981) and Marmaris ophiolite (Ergen et al., 2021) within the Lycian Nappes.

The Marmaris Ophiolite, whose lower and upper contacts are tectonic, is up to 1000 m thick. (Şenel, 1997). In the study area, it tectonically overlies the Sultandağı Unit to the south of Şarkikaraağaç and the Anamas-Akseki Autochthon, the Kızılcadağ Mélange and the Yenicekale Metamorphics to the south of Yenicekale (Figure 9c). On the other hand, the units belonging to the Gülbahar and Domuzdağ Nappes rest on the ophiolites with a tectonic contact.

The Marmaris Ophiolite consists of serpentinized dunite and harzburgite and isolated diabase dykes (Figure 9a). The most common rocks observed in this

ophiolitic units are harzburgites, which are medium to coarse grained, with green olivine and grey to black pyroxene minerals, brown on altered surfaces and blackish green to green on fresh surfaces. Dunites, apart from olivine, also include orthopyroxene and chromite minerals, with a mesh texture, light green to greenish grey in color, are less common than harzburgites. Serpentinization is common, especially along fractures. Dunites and harzburgites are intruded by 0.5-2 m thick isolated diabase dykes composed of plagioclase, clinopyroxene and opaque minerals with an ophitic texture. Zircon and titanite minerals from these dykes, which characterize geochemically subduction-related island arc tholeiites, yielded U-Pb ages of 87.5-102 Ma, while hornblende minerals yielded a ^{40}Ar - ^{39}Ar age of 91-93 Ma (Çelik et al., 2006). Stockwork magnesite veins are widely observed in

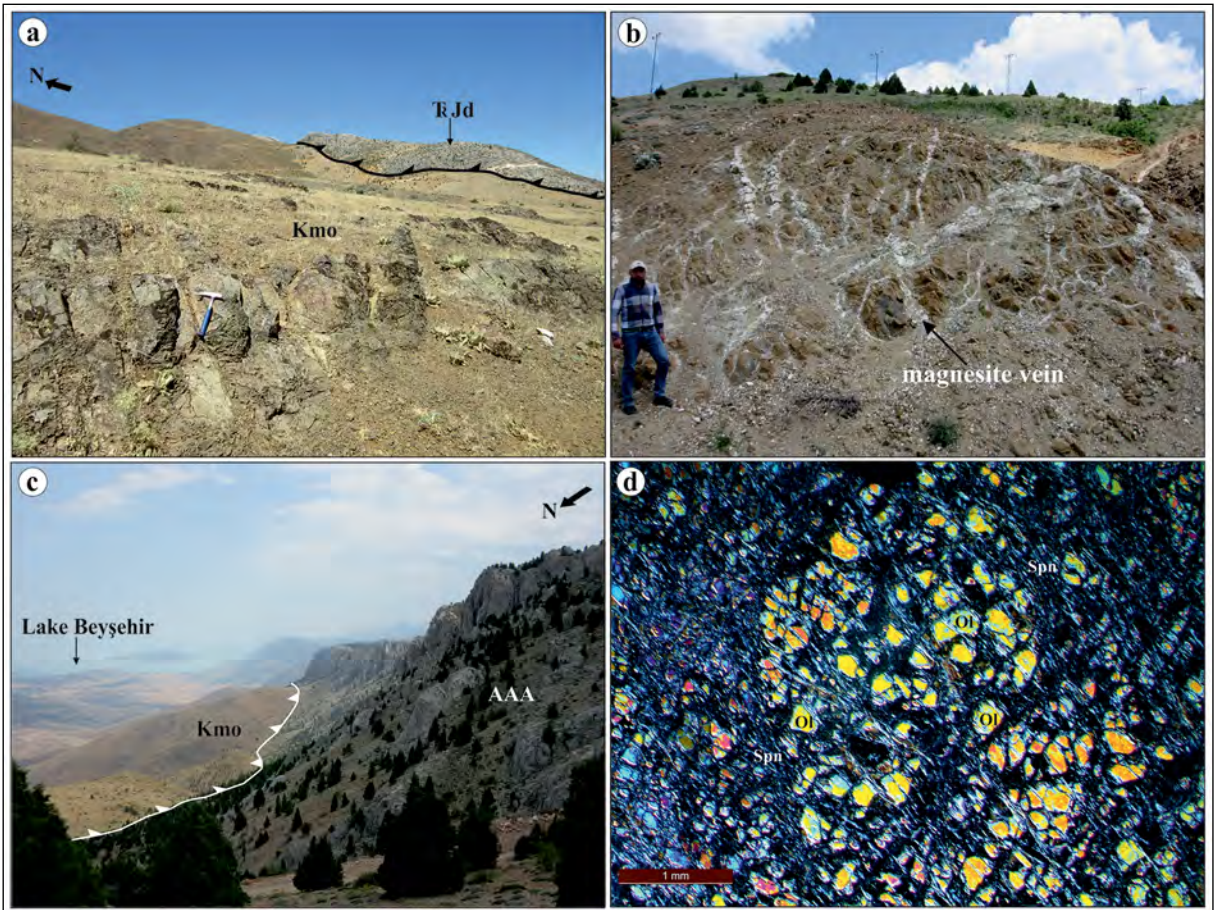


Figure 9- a) Tectonic relationship between the Marmaris Ophiolite (Kmo) and the Dutdere Formation (T_rJd) of the Domuzdağ Nappe, NW of Sivri Hill, Yenicekale, b) stockwork magnesite veins in the ophiolites, NW of Eğirler, c) tectonic contact between the Marmaris Ophiolite (Kmo) and the Anamas-Akseki Autochthon (AAA), NW of Lake Beyşehir, d) thin section view of a serpentinized dunite sample under cross polarized light (Ol: Olivine, Spn: Serpentine).

the ophiolites outcropping around Madenli village (Figure 9b).

The unit, which characterizes supra-subduction ophiolites, is accepted as Late Cretaceous in age based on radiometric data and geochemical analysis obtained from its isolated diabase dykes and metamorphic sole (Andrew and Robertson, 2002; Çelik et al., 2006; Elitok and Drüppel, 2008; Parlak et al., 2019; Ergen et al., 2021, Ergen, 2023).

2.4.2. Gülbahar Nappe

The Mesozoic allochthonous sequence of deep marine limestone, chert, radiolarite and mudstone and spilitic basalt is called the Gülbahar Nappe (Graciansky, 1972; Şenel et al., 1994). The Gülbahar Nappe and the Orluca Formation from this nappe have been identified and mapped in this study for the first time in the south of Sultan Dağları.

The Gülbahar Nappe is generally observed as blocks and slices of different sizes in the Kızılcaadağ mélangé. Outcropping west of Ördekçi village and south of Salur (Şarkikaraağaç), the Gülbahar Nappe is represented in the study area by the Orluca Formation. The aforementioned locations where the Orluca Formation is exposed have been misinterpreted by previous researchers (e.g. Elitok and Drüppel, 2008; Parlak et al., 2019) as Upper Cretaceous slope and basin deposits.

Orluca Formation (T_RJo): The formation, which consists of volcanics, radiolarites, cherty micritic limestones, mudstones and sandstones (Şenel, 1989), is exposed west of Ördekçi and south of Salur (Şarkikaraağaç).

The formation is composed of thin to medium-bedded, red, grey, green, intensely folded, deformed micritic limestones with chert bands and nodules, and red, dark grey thin-bedded radiolarites, and green-greyish green, medium- to thick-bedded sandstones and mudstones (Figures 10a-c). They are occasionally accompanied by green, brown, fine- to medium-grained spilitic basalts (Figure 10d), which consist of plagioclase, clinopyroxene, kaersutite, biotite, calcite and opaque minerals with an intersertal texture (Figure

10e). Sericitization and argillization are common in plagioclase minerals, while chloritization is common in pyroxene minerals. Radiolarites consist entirely of fossil radiolarian tests filled with very fine-grained siliceous minerals. The fact that the formation consists of sandstone, mudstone, radiolarite, cherty micritic limestone with basic volcanic intercalations indicates deposition in a slope and basin environment where both volcanism and turbidity currents are occasionally active.

Tectonically overlying the Kızılcaadağ Mélangé (Figures 10 c and 10f), the Orluca Formation is also observed as blocks of various sizes in the mélangé.

A Middle Triassic-Early Jurassic age was assigned to the Orluca Formation by Şenel et al. (1989) on the basis of the fossil assemblages. This age is also accepted in this study.

2.4.3. Domuzdağ Nappe

The unit, which is observed as blocks and slices at various sizes within the Marmaris Ophiolite Nappe and characterized by *Megalodon*-bearing neritic limestones, is recognized as the Domuzdağ Nappe (Poisson, 1977; Ersoy, 1989; Şenel et al., 1994). The “Middle” Triassic-Early Jurassic Dutdere Formation represents the Domuzdağ Nappe in the study area.

Dutdere Formation (T_RJd): The Dutdere Formation (*sensu* Ersoy, 1989) consists mainly of grey, light grey, beyaz and beige-colored, medium- to thick-bedded, *Megalodon* and algae-bearing and occasionally recrystallized limestones, which are very fine-grained biomicrite, microsparite and sparry micrite. The limestones, composed of calcite minerals and poorly preserved fossils in a micritic carbonate cement, are sometimes recrystallized due to the intense tectonics caused by nappe movements. It contains cracks and fractures developed in different directions and is observed as cataclastic and brecciated, especially at the nappe contacts. Şenel et al. (1994) reported that pink to red, thin- to medium-bedded, ammonite-bearing cherty limestones occur outside the study area above the *Megalodon*-bearing lower levels of the formation.

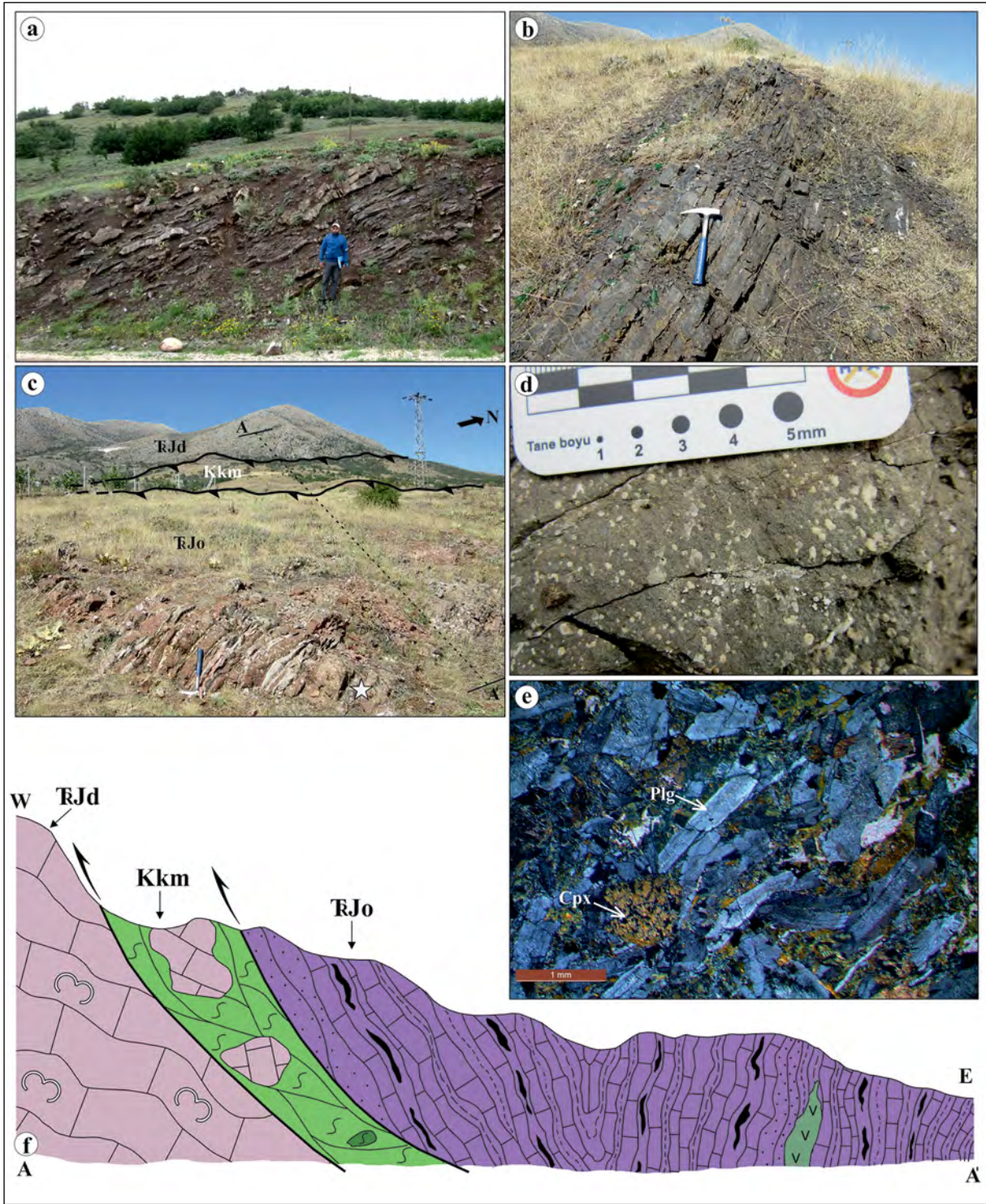


Figure 10- a) Alternation of micritic limestone and mudstone in the Orluca Formation, S of Salur, b) dark grey radiolites, W of Ördekçi, c) tectonic relationships of the Orluca Formation with the Kızılcaadağ mélangé and the Dutdere Formation, NW of Ördekçi, d) a close-up view of the spilitic basalts, e) thin section view of a basalt sample under cross polarized light (Cpx: Clinopyroxene, Plg: Plagioclase), f) a geological cross-section showing relations between the Dutdere Formation (T_RJd), Kızılcaadağ Mélangé (Kkm) and Orluca Formation (T_{Jo}).

In the study area, the formation tectonically overlies the Marmaris Ophiolite, Kızılcaadağ mélangé, and Orluca Formation. The Kızılcaadağ Mélangé is also observed over the Dutdere Formation, depending on the tectonic movements after its primary emplacement. One such contact relationship can be seen at the SE of Yenicekale.

Within the formation, which is easily recognized in the field by bearing *Megalodon* fossils, foraminifera such as *Aulotortus* gr. *sinousus* (Weynschenk), *Aulotortus* sp., *Endoteba* sp., *Reophax* sp. have been determined in this study at the south of Sultan Dağları, indicating a Late Triassic (Norian-Rhaetian) age. Şenel et al. (1989, 1994) assigned a Middle Triassic-Early Jurassic age to the formation, which is also accepted in this study, based on the foraminiferal fauna obtained from the same formation at different locations in the Western Taurides. Based on the lithological characteristics and fossil content, it can be inferred that the formation was deposited in a shallow carbonate shelf environment.

2.5. Cover rocks

2.5.1. Celeptaş Formation (Pgpec)

The Celeptaş Formation (sensu Demirkol, 1977) is mainly composed of thin-bedded limestones, calcarenites, siltstones, mudstones, sandstones and subordinate conglomerates that form different facies and facies associations (Figure 11). The thin-bedded, cream-coloured pelagic limestones and claret-coloured calcarenite and biomicrite with abundant planktonic foraminifera are exposed in the north of the basin (Figures 11a, b). The fine-grained calcarenites are well-sorted and show planar parallel stratification and wave-ripple cross-lamination (Figure 11b). They pass southwards into the interbedded mudstones, siltstones and sandstones, forming mudstone- and sandstone-dominated sequences (Figures 11c, d, e).

Greenish-grey mudstones are laterally extensive and generally massive in the mudstone-dominated sequences (Figure 11c). Very thin-bedded siltstones and very fine to fine sandstones interbedded with mudstones are generally 0.5-5 cm thick, and in lesser amounts up to 15 cm. Sedimentary structures include

planar parallel stratification, normal grading and current ripple cross-lamination.

The sandstone-dominated sequence consists mainly of grey to light brown sandstones and siltstones interbedded with grey mudstones (Figure 11d). The tabular sandstones, which are generally 10-30 cm thick, contain mainly Bouma-type Tbc and lesser amounts of Tabc turbidites. Other sedimentary structures include flute and groove marks, load casts, convolute laminations and some trace fossils. The interbedded sandstones, siltstones and mudstones, form coarsening- and thickening-upward bed packages a few metres thick (Figure 11e). These are erosionally overlain by lenticular channel-fill deposits in the axial part (Figure 11e). The fining upward bedset of the channel-fill deposits consist of coarse sandstones to granule conglomerates, rich in pebble gravels at the base.

The *Nummulites*-bearing pelagic limestones and biomicrites, rich in planktonic foraminifers, indicate a neritic carbonate platform formed on the basin-margin narrow shelf. The calcarenites are interpreted to represent wave-worked, lower shoreface to offshore transition deposits (cf. Clifton, 1981; Dott and Bourgeois, 1982). The mudstones are interpreted to be basin-plain hemipelagic deposits interlayered with thin siltstone and sandstone turbidites in the mudstone-dominated bed packages. The graded sandstone beds with planar stratification are tractional deposits of low-density turbidity currents (Bouma, 1962; Lowe, 1982; Kneller, 1995). The sandstone-dominated coarsening-upwards bed packages are interpreted as turbiditic depositional lobes and the overlying fining-upwards bedsets as channel-fill deposits. The sedimentary facies of these deposits indicate high- and low-density turbidity currents. The composition of the sandstone and the pebbles indicates that the sand grains and clasts are of ophiolitic origin.

The Celeptaş Formation unconformably covers the Sultandağı Unit, the Anamas-Akseki Autochthon and the BHN (Figure 12), while it is tectonically overlain in places by the BHN (Figure 13).

The results of the planktonic foraminiferal analyses performed on the marine mudstone

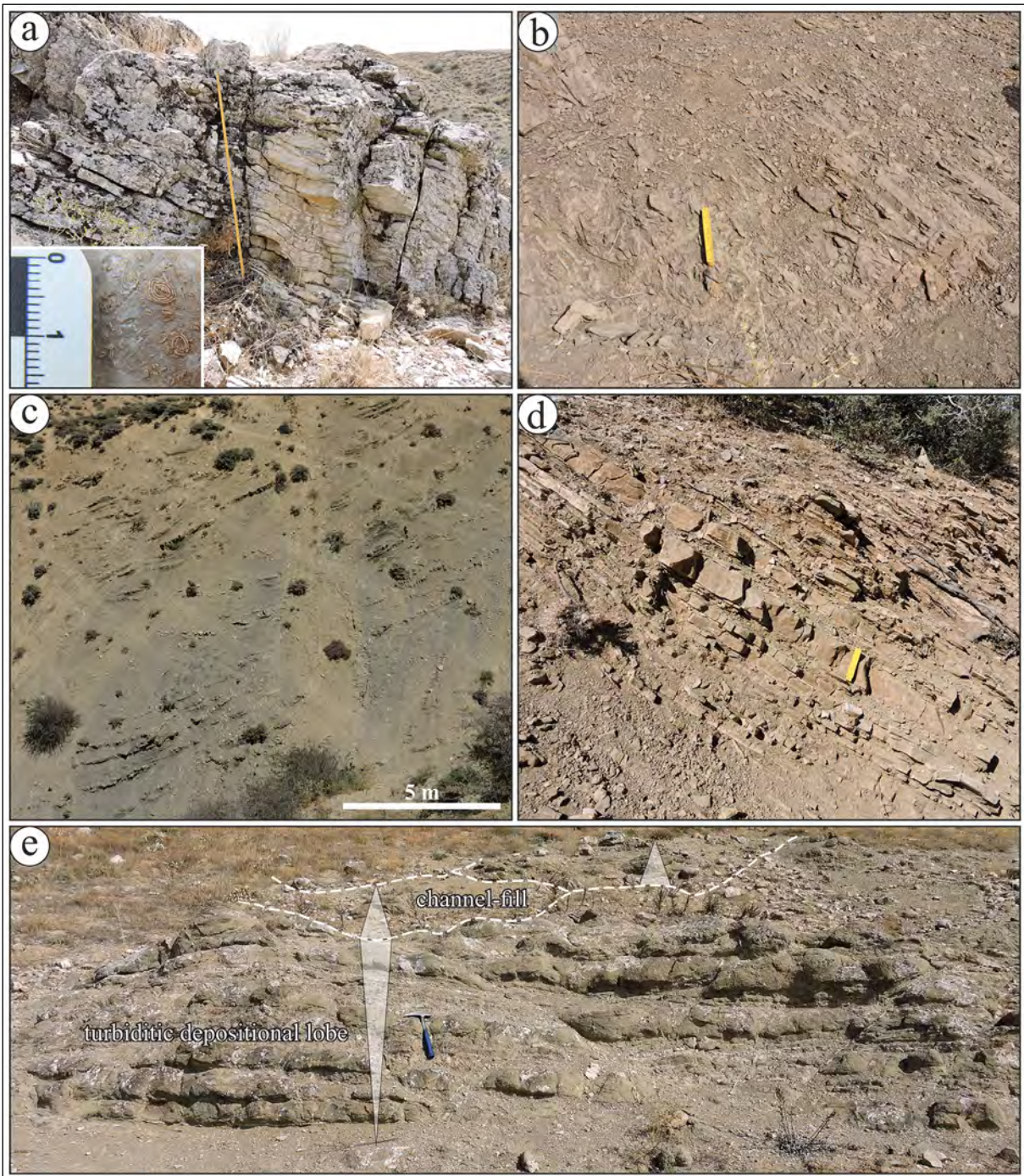


Figure 11- Facies details of Dursunlu Formation, a) Thin-bedded pelagic limestones with *Nummulites* fossils and b) claret-coloured calcarenite and biomicrite, c) Massive mudstones of the basin-plain hemipelagic deposits interbedded with siltstone and sandstone turbidites, d) The sandstone-dominated sequence consists mainly of Bouma-type Tbc and lesser amounts of Tabc turbidites, e) The coarsening- and thickening-upward bed packages of the turbiditic depositional lobes are erosionally overlain by the fining-upwards bedsets of the channel-fill deposits. The ruler is 1 m in figure a and 10 cm in figures b and d, the hammer is 33 cm.

samples from Hodulca Hill northwest of Yalvaç (327382E/4244438N), north of Celeptaş village (327382E/4244438N) and south of Yenicekale (341350E/4214700N) (Figures 2, 3) are given below.

Among the abundant planktonic foraminiferal assemblages of the Celeptaş Formation, the species such as *Globanomalina pseudomenardii* and *Acarinina soldadoensis* in the Thanetian, *Morozovella*

subbotinae and *Morozovella velascoensis* in the late Thanetian, *Morozovella edgari* and *Morozovella gracilis* in the early Ypresian, *Morozovella lensiformis* and *Morozovella aragonensis* in the Ypresian, *Acarinina bullbrooki*, *Acarinina cuneicamerata* and *Turborotalia frontosa* in the late Ypresian to Lutetian levels have been identified in a stratigraphical order as markers. The age of the Celeptaş Formation has been assigned as Late Paleocene-Lutetian based on this planktonic foraminiferal content.

2.5.2. Yalvaç Basin

The Yalvaç Basin, which is formed of terrestrial and lacustrine deposits, is a triangular molasse basin that extends in a NW-SE direction between the Sultan Dağları and the Anamas Mountains (Yağmurlu, 1991;

Ilgar et al., 2021). Deposits of the basin are alluvial fan, lacustrine clastic and carbonate sediments. The formation of the Yalvaç Basin, which began to open after nappe emplacement as a result of orogenic collapse, is known to have been influenced by the break-up or roll-back of the Southern Neotethys slab during the Early Miocene (Koçyiğit and Deveci, 2007; Koçyiğit et al., 2013; Koç et al., 2016; Ilgar et al., 2021). The sediments of the Yalvaç Basin unconformably overlie all pre-Miocene units in the region.

3. Discussions

The formation and emplacement processes of the BHN are important in revealing the geodynamic evolution of the region. The generally accepted aspect

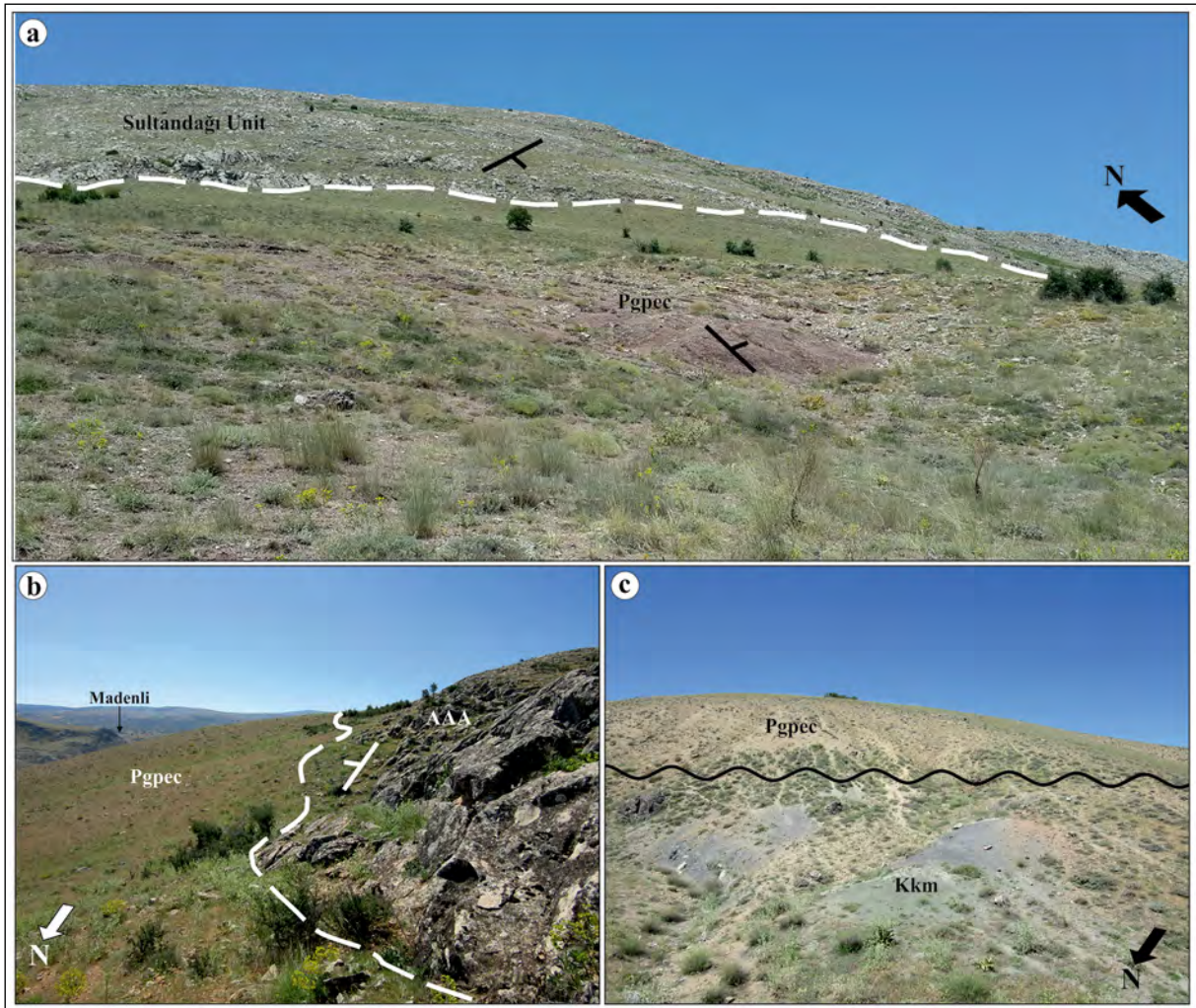


Figure 12- a) The unconformable contact between the Celeptaş Formation and the Middle Jurassic-Upper Cretaceous recrystallized limestones of the Sultandağı Unit, near Hodulca Hill, Yalvaç, b) unconformable contacts between the Celeptaş Formation and Jurassic-Cenomanian limestones of the Anamas-Akseki Autochthon (AAA) and c) Kızılcaadağ Mélange (Kkm) of the BHN, north of Madenli.



Figure 13- a) Thrust contact between the Kızılcadağ Mélange of the BHN and the Celeptaş Formation, SE of Yenice kale, b) close-up of the first figure showing folds.

is that the BHN was formed as a result of horizontal movements due to the closure of the northern branch of the Neotethys Ocean (İzmir-Ankara-Erzincan and/or Inner Tauride Ocean) and settled in the region by thrusting over the Sultan Dağları from north to south during the Late Cretaceous-Eocene (Özgül, 1984; Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Elitok and Drüppel, 2008).

There are several critical issues in dating nappes formed by oceanic subduction processes such as the BHN, the most important of which are the age of the sub-ophiolitic metamorphic rocks, the age of the matrix and blocks of the mélangé and the age of the cover of the nappes. The formation of nappes begins with the development of the sub-ophiolitic metamorphic rocks, which represent the onset of subduction, while it ends with the youngest age obtained from the matrix in the mélangé. The emplacement time, on the other hand, is constrained by the youngest unit the nappes thrust over and the overlying oldest unit.

The sub-ophiolitic metamorphic rocks, which formed during the onset of subduction, are widely accepted as definitive indicators of subduction initiation ages, with metamorphic ages derived from their amphibolites (Çelik and Delaloye, 2006). In this context, the 93.90 ± 0.34 Ma (Cenomanian-Turonian boundary) age data obtained from the Yenice kale Metamorphics is accepted as the onset of the formation of the BHN (Figure 14a).

The ages obtained from the blocks and matrix of the mélangé are another significant parameter related to the formation of the mélangé. Accordingly, the occurrence of Middle Triassic Domuzdağ Nappe, Middle Triassic-Lower Jurassic Gülbahar Nappe and Upper Cretaceous Marmaris Ophiolite Nappe blocks and slices within the Kızılcadağ Mélange and the determination of late Maastrichtian planktonic foraminifera from the matrix of the mélangé indicate that the formation of the BHN lasts possibly from the Turonian to the late Maastrichtian.

The basin type in which the Celeptaş Formation was deposited has been inferred from the sedimentary characteristics of the formation and its tectonostratigraphic relationship with the surrounding units. The rapid transition of the neritic carbonate and offshore transition deposits of the Celeptaş Formation into the deep marine facies indicates a narrow shelf and increased supply of terrigenous sediments to the basin. In addition, intense compressional tectonic deformation during and after deposition suggests that the sediments were deposited in a tectonically controlled basin. Therefore, taken together, we have interpreted the Celeptaş Formation as being deposited in a foreland basin that was fed by and developed in front of the BHN. The flexural subsidence of the basin floor, driven by the crustal load of the BHN, led to the development of sediment accommodation

space. Tectonic loading from the north resulted in the development of a deep-marine environment. Thus, the turbidite deposits of the Celeptas Formation both transgressively overlie the BHN and, in the ongoing

process, the BHN tectonically overlies the Celeptas Formation from north to south (Figure 14d).

The first emplacement age of the BHN must be younger than the tectonically underlying early

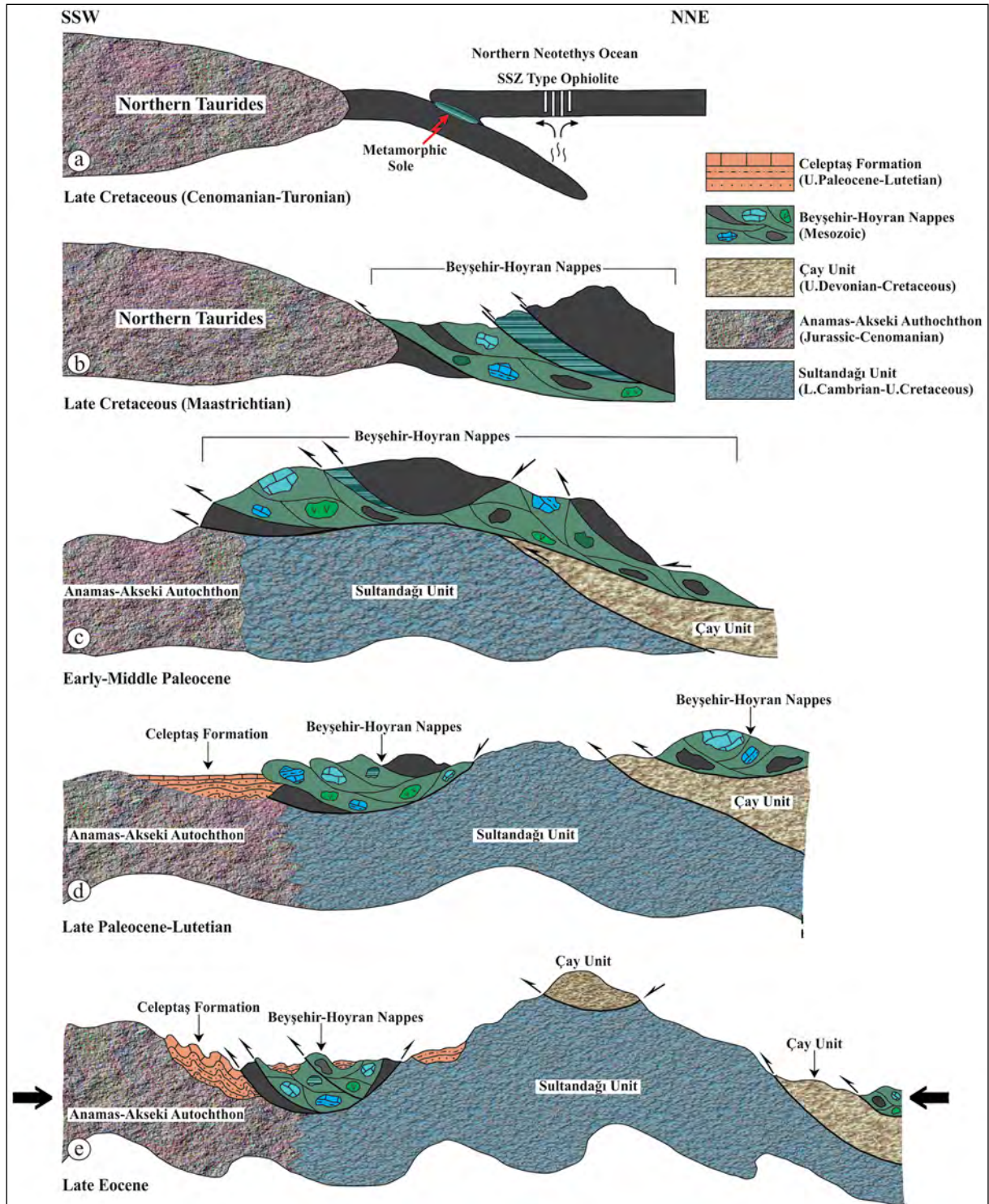


Figure 14- Geological sections illustrating the evolution of the BHN during the Late Cretaceous to late Eocene period.

Cambrian-Late Cretaceous Sultandağı Unit and the Jurassic-Cenomanian Anamas-Akseki Autochthon, while it must be older than the late Paleocene-Lutetian Celeptaş Formation, the common cover of the parautochthonous and allochthonous units in the region, thus pointing to an early-middle Paleocene emplacement age (Figure 14c). The ongoing movements of the BHN and its settlement in the basin caused the syndimentary deformation of the Celeptaş Formation.

Şenel (1991) argued for the presence of a Paleocene-Lutetian common marine basin extending from the Lycian Nappes towards Seydişehir along the Isparta Angle. These data also support the idea that the BHN was not emplaced in the region during late Eocene, contrarily, but already existed there before the late Paleocene.

The stratigraphic studies and geological mapping carried out in the region during this study, as well as the age data obtained from both the BHN and the Celeptaş Formation, helped to reinterpret the Late Cretaceous-Eocene geological evolution of the region. The initiation of the northward subduction of the northern branch of the Neotethys Ocean led to the formation of SSZ-type ophiolites (Marmaris Ophiolite) and consequently to the development of sub-ophiolitic metamorphic sole (Yenicekale Metamorphics) at the Cenomanian-Turonian boundary (Figure 14a). During the Late Cretaceous-early Paleocene, from north to south, the BHN thrust over the Çay Unit and the Çay Unit thrust over the Sultandağı Unit (Figures 14b-c). In the early Paleocene, the BHN crossed the Sultan Dağları and reached the Anamas-Akseki Autochthon (Figure 14c). During this period, the rocks of the Sultandağı and Çay units underwent both deformation and low-grade metamorphism depending on the emplacement of the BHN (Figure 14c). Following this process, the Celeptaş Formation was deposited in the foreland basin, which developed in the south of Sultan Dağları, in the late Paleocene-Lutetian period. Following this process, the Celeptaş Formation was deposited in the foreland basin that developed in the south of Sultan Dağları in the late Paleocene-Lutetian period (Figure 14d).

The Celeptaş Formation was folded due to the tectonic movements, probably occurred in the late Eocene, and subsequently was overthrust in places by units of the BHN. These can be considered as secondary thrusts after the primary emplacement (Figure 14e).

4. Conclusions

The BHN, which extends as a NW-SE belt to the south of Sultan Dağları, consists of allochthonous masses such as the Marmaris Ophiolite Nappe, Gülbahar Nappe and Domuzdağ Nappe. The Marmaris Ophiolite Nappe (Upper Cretaceous) is formed by three subunits as the Marmaris Ophiolite, the Kızılcadağ Mélange and the Yenicekale Metamorphics. The Marmaris Ophiolite is composed of dunite, harzburgite, serpentinite and isolated diabase dykes, while the Kızılcadağ Mélange is composed by blocks of different ages and lithologies contained in an ophiolitic/sedimentary matrix. The Yenicekale Metamorphics are characterized by sub-ophiolitic metamorphic sole rocks such as amphibolites, quartzites and calcschists.

The Gülbahar Nappe is represented by the Middle Triassic-Early Jurassic Orluca Formation, which is composed of deep marine mudstones, cherts, radiolarites, micritic limestone and basic volcanics. The Orluca Formation, which has been identified and mapped for the first time at the south of Sultan Dağları.

In addition, we obtained a ^{40}Ar - ^{39}Ar age of 93.90 ± 0.34 Ma (Cenomanian-Turonian boundary) from the hornblende minerals of amphibolites from the Yenicekale metamorphics. Tectonic processes, which were triggered by and occurred immediately following the formation of the metamorphic sole during the aforementioned time started the formation of the nappes. We therefore accepted that the Turonian time should be the onset of the formation of the BHN.

On the other hand, the late Maastrichtian age, which is obtained from the mudstones in the matrix of the Kızılcadağ mélange, is considered to mark the end of the formation of the BHN.

The evaluation of the stratigraphic, palaeontological and radiometric data together reveals that the formation of the BHN began in the Turonian and

ended in the Late Maastrichtian. The Late Paleocene-Lutetian Celeptaş Formation unconformably overlies the Sultandağı Unit, Anamas-Akseki Autochthon and BHN as common cover in the study area. This clearly indicates that the BHN was emplaced in the region during the early-middle Paleocene. The BHN reached its final position as a result of late Eocene movements.

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Magnetic anomalies and geological signatures in the Black Sea: a comprehensive study of the east and west basins

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Research Article

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Curie Point Depth, Heat Flow, Tilt Derivative, Local Wavenumber, Correlation Plot.

ABSTRACT

This study investigates the origin of the Black Sea, focusing on the East and West Black Sea basins. Data such as Curie Point Depth values, heat flow, bathymetry, Moho depth, sediment thickness, and magnetic depth estimation were analyzed to explore the region's geological features. Correlations between these parameters were assessed for each basin, shedding light on their tectonic history and the influence of volcanic rocks. Thinner sedimentary covers enhanced the detectability of rocks with higher magnetic properties, while the interiors showed lower magnetic anomalies due to younger sedimentary rocks. Edge detection techniques, like tilt angle and Local Wavenumber methods, improved magnetic data interpretation. The study computed Curie Point Depth and heat flow using 65 blocks, revealing greater depths in the Western Black Sea (up to 32 km) compared to the Eastern Black Sea (24-28 km). Two profiles (A-A' and B-B') were examined for further insights. Overall, this study provides valuable insights into the geological evolution of the Black Sea basins.

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1. Introduction

To limit the thermal structure and lithospheric rheology, it is significant to assess the spatial discrepancies in temperature across the Earth (Audet and Gosselin, 2019; Sobh et al., 2021). In addition, analysing the variances in magnetic data and the depth of the Moho can provide insight into the tectonic evolution of the region. Calculating the depth of the Curie point, at which crustal rocks reach their Curie temperature [approximately 580° for magnetite (Dunlop et al., 1998)], provides temperature constraints over a region. When rocks are heated above the Curie temperature, they lose their ferromagnetic magnetism and become paramagnetic. Thus, the depth of the magnetised crust can be determined by identifying the

Curie depth, which is the point at which rocks lose their ability to preserve ferromagnetic magnetism (Haggerty, 1978).

Although estimating the Curie depth from magnetic data is significant, uncertainties remain as to whether spectral techniques can produce dependable depth estimates and whether these estimations portray the Curie isotherms or a structural boundary. To clarify this issue, optimum wavenumber ranges for estimating Curie depth was limited by comparing theoretical approximation and linear estimation (Núñez Demarco et al., 2021; Pamuk and Özsöz, 2022). Furthermore, additional constraints such as uncertainties in Curie depth estimation, Moho depth, sediment thickness, and magnetic depth estimation techniques can be

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employed to verify the estimations of Curie depth (Özsöz, 2021).

Numerous researchers have conducted studies on the thermal composition of the Black Sea's crust, specifically within the West and East Black Sea Basins (WBS and EBS). Maden (2013) investigated the EBS and determined that the elevated values of heat flow observed may be attributed to the melting of the lithospheric mantle, triggered by the upwelling of the asthenosphere. Starostenko et al. (2014) noted that the WBS and EBS exhibit a greater depth for the Curie point (CPD) as compared to the Mid-Black Sea Ridge (MBSR) and coastal regions, which are characterised by thinner sediment layers. This finding suggests that areas with thicker sediment deposits correspond to deeper CPD values. In their study, Tütünsatar et al. (2018) presented findings on the fluctuating heat flow values in the Black Sea, ranging from 22 mW/m² to 41 mW/m². They posited that regions with higher heat flow values than the average are indicative of potential deep fault zones and areas where gas migration occurs. Entezar-Saadat et al. (2020) presented a comprehensive lithospheric model of the Black Sea, incorporating thermal structure, sediment thickness, and the depths to the Moho and lithosphere-asthenosphere boundary (LAB). The study utilized a combination of 3D nonlinear inversion of satellite gravity and gravity gradiometry data with 2D iterative forward modelling of potential field data and heat flow measurements. The findings revealed that the average basement depths are 10–11 km in the East Black Sea (EBS) and 11–12 km in the West Black Sea (WBS), with shallower depths of 2–5 km beneath ridges. The mean crustal thickness was determined to be 20 km in the EBS and 21 km in the WBS. Additionally, the surface heat flow was found to be highest on the ridges and lowest over the thick central sedimentary basins. This model offers a detailed and integrated understanding of the Black Sea's lithospheric structure. Bilim et al. (2021) shed light on crustal thickness beneath the Black Sea and surrounding region by gravity data. Their findings indicate that the Shatsky Ridge displays the thinnest crustal thickness at 10 km, while the Sea of Azov boasts the greatest crustal thickness, measuring at 28 km. Akar (2024) conducted a study in the Çameli Basin and its surrounding region in southwestern

Anatolia, Türkiye, utilizing aeromagnetic data and a fractal-based centroid method to estimate the Curie Point Depth, which is essential for understanding regional tectonic and geothermal characteristics. The estimated depths of magnetic sources range between 6.9 and 14.05 km, while the thermal gradient varies from 41.28°C/km to 89.23°C/km, with an average value of 58.59°C/km. The results correspond with NW-SE earthquake distributions and indicate that the Curie isotherm is significantly shallower than the Moho boundary.

The primary objective of this study is to establish a comprehensive theoretical framework elucidating the origin and evolution of the Black Sea, with a specific focus on both the EBS and WBS basins. To achieve this, a wide array of data is employed, encompassing parameters such as CPD (Curie Point Depth) values, heat flow values derived from CPD data, bathymetry, Moho depth, sediment thickness, and magnetic depth estimation techniques (Tilt depth and Local Wavenumber methods). The investigation also examines how the distribution of volcanic rocks in the Black Sea region impacts magnetic anomalies. Furthermore, a meticulous analysis of the correlation between CPD values, bathymetry, Moho depth, sediment thickness, tilt depth, and Local Wavenumber depths is conducted separately for the EBS and WBS basins. By closely examining these correlations and considering the tectonic history and presence of volcanic rocks, the notable discrepancies between the EBS and WBS are thoroughly interpreted. This research aims to shed light on the distinct geological processes and factors that have contributed to the development and divergence of the EBS and WBS basins.

2. Geological Setting

The Black Sea, a large semi-enclosed basin, is surrounded by orogenic belts in the south (Pontides), northeast (Crimea Mountains and Greater Caucasus), and west (Balkanides). Beneath the Neogene to present-day sedimentary succession of the basin fill, representing a unified depocenter, lie two sub-basins, namely the WBS and the EBS, along with a structural high, the Mid Black Sea Ridge (MBSR), comprising Andrusov Ridge and Arkhangelsky Ridge in an

en-echelon arrangement. The WBS and EBS display distinct differences in structural complexity, with the latter featuring a prominent ridge (Shatsky) and a pair of troughs (Tuapse and Sorokin) near its northeastern margin.

The Black Sea Basin is categorized as a back-arc basin, resulting from the northward subduction of the Neo-Tethys Ocean under the southern margin of Laurasia (Letouzey et al., 1977; Zonenshain and Le Pichon, 1986; Finetti et al., 1988). The process of rifting in the extensional back-arc led to the separation of a continental fragment (İstanbul Zone) from the Moesian Platform, giving rise to the asymmetric WBS, where the southern margin exhibits a steeper inclination (Okay et al., 1994; Nikishin et al., 2015). On the other hand, the EBS took shape through the separation and rotation (Okay et al., 1994; Robinson et al., 1996) of the Mid MBSR from the Shatsky Ridge, forming a more symmetrical basin (Shillington et al., 2009). The timing of sub-basin formation is generally believed to be late Cretaceous to early Paleogene, with the EBS considered to be the younger of the two, a subject of ongoing debate (Görür, 1988; Görür et al., 1993; Robinson et al., 1995; Okay and Şahintürk, 1997; Hippolyte et al., 2017; 2018).

The two sub-basins are situated on crusts with distinct thermal and mechanical properties (Spadini et al., 1996). After the initial rifting, the WBS evolved into a proper ocean with an inferred oceanic crust, likely during the late Santonian period, indicated by the age of corresponding pelagic sediments and volcanics in the Pontides region (Tüysüz et al., 2012; Tüysüz, 2018). On the other hand, the EBS has a thinned continental or an oceanic crust beneath it (Shillington et al., 2009), and its opening started in the late Paleocene and was completed in the middle Eocene (Robinson et al., 1995). The sedimentary fill thickness in the WBS ranges from 14 to 18 km, whereas in the EBS, it can reach up to 12 km.

Seismic surveys have revealed the presence of multiple buried volcanoes along the southern offshore margin of the Black Sea basin and on the Shatsky and Andrusov ridges (Nikishin et al., 2015). Due to the absence of sufficient well data, the age of these volcanoes is inferred to be likely Santonian-

Campanian based on the correlation with volcanic units in the Pontides and Crimea regions (Nikishin et al., 2003).

Based on seismic reflection studies (Letouzey et al., 1977; Nikishin et al., 2015), the undisturbed post-rift sedimentary layers within the basin indicate that the crust beneath the Black Sea basin possesses sufficient strength to withstand the tectonic forces responsible for the deformation of its margins since at least the Eocene period (Espurt et al., 2014). GPS measurements (Tari et al., 2000) and seismic activity (Neprochnov and Ross, 1978) indicate an active shortening on the EBS. Figure 1 illustrates simplified tectonic map of the study area.

3. Data

The magnetic data used in this study was obtained from the EMAG 2 model. The EMAG 2 model presents a detailed description of the Earth's magnetic field on a global scale. It was calculated using satellite magnetic data as well as land and sea measurements. The EMAG 2 model can identify magnetic anomalies larger than 1 nT with a coverage of 2 arc-minutes and a spatial resolution of approximately 4 km. (Maus et al., 2009).

A robust inversion approach that has been widely validated in multiple regional studies is used to extract the first global model of Curie-point depth (GCDM) from magnetic anomaly inversion based on fractal magnetisation (Li et al., 2017). According to the statistical analysis, the mean depth of the Curie point in the marine zone is lower than in the continental region. The continental Curie depths, on the other hand, have a virtually bimodal distribution, with some old cratons having shallow Curie points. Hydrothermal circulations in young oceanic lithosphere and thermal disturbances in old oceanic lithosphere affect oceanic Curie depths. Furthermore, the oceanic Curie depths are heavily dependent on the spreading rate along active spreading centres (Li et al., 2017). In this research, the estimated Curie-depth points were compared and validated with the global model of Curie-point depth within the same geographical region.

The bathymetry data used in this study, which focused on the Black Sea region, was obtained

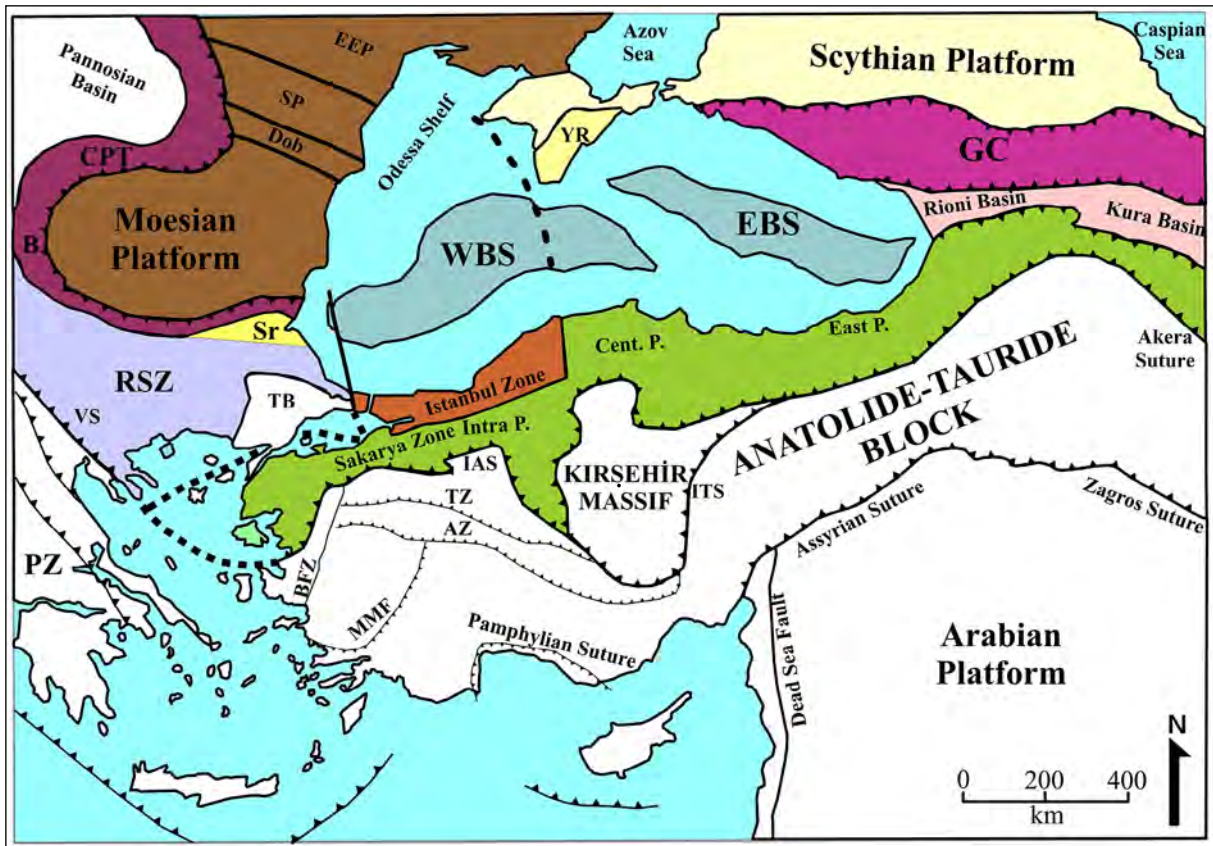


Figure 1- Simplified tectonic map of the Black Sea and its surrounding regions (modified after Şengör, 1984; Okay, 1989; Okay and Tüysüz, 1999; Özsöz and Tokar, 2022). PZ= Pelagonian Zone, VS= Vardar Suture, RSZ= Rhodope-Strandja Zone, B= Balkanides, CPT= Carpathians, Dob= Dobruđa, SP= Scythian Platform, EEP= East European Platform, YR= Yayla Range, WBS=West Black Sea Basin, EBS= East Black Sea Basin, GC= Greater Caucasus, Intra P.= Intra Pontide Suture, Cent. P.= Central Pontides, East P.= Eastern Pontides, IAS= İzmir - Ankara Suture, TZ= Tavşanlı Zone, AZ= Afyon Zone, MMF= Menderes Massif, BFZ= Bornove Flysch Zone, SR= Srednogorie).

from the ETOPO 1 model. ETOPO 1 model includes combination of land topography and ocean bathymetry into a global grid. The model was developed by The National Oceanic and Atmospheric Administration (NOAA). ETOPO1 grid cell spans an area of around 1.8 km² at a resolution of one arc-minute at the equator. The model is constructed with an integration of satellite and marine observations, as well as data from other sources. ETOPO1 is widely used in oceanography, geology, and climate research because it provides a precise depiction of the Earth's topography, including mountain ranges, valleys, and oceanic trenches (Amante and Eakins, 2009).

Moho depth and sediment thickness data were obtained from CRUST1.0 model. CRUST1.0, 1-by-1° global crustal model, which forms the initial framework for the comprehensive database to compile

a worldwide model of the Earth's crust and lithosphere, LITHO1.0 (Pasyanos et al., 2013). The Moho depth and sediment thickness featured in CRUST1.0 is based on 1-degree averages obtained from a recently updated database of crustal thickness data gathered from active-source seismic studies, as well as from receiver function studies (Laske et al., 2013).

4. Methods

4.1. CPD Estimation

The Curie temperature is the fundamental physical phenomenon that controls the exploration of magnetic depth method. This method's ability to detect magnetic bodies is restricted by the depth of the Curie isotherm. It is feasible to utilize magnetic data to deduce heat flow since the magnetic characteristics of rocks are dependent by temperature,

and rocks lose their magnetic properties at the Curie temperature. Nonetheless, this methodology is subject to certain limitations due to its dependence on several assumptions. The technique requires that the magnetic sources' depth be significantly greater than their height, which is vital for spectral separation (Bouguern et al., 2015).

The estimation of CPDs has been accomplished in numerous locations across the globe through the application of magnetic data. Two primary techniques have been employed to calculate CPDs: the spectral peak and the centroid depths. The spectral peak method was initially introduced by Spector and Grant (1970), while the centroid approach was first presented by Bhattacharyya and Leu (1977) and was later improved by Okubo et al. (1985). In this study, the latter approach was used.

Our assumption is that the lithosphere's magnetised layer is limited by two surfaces situated at depths Z_t (the top of the magnetised layer) and Z_b (the bottom of the magnetised layer). It should be noted that if $Z_t = 0$, this would imply that the magnetised layer's top coincides with the measurement plane (Sobh et al., 2021). The thickness of the magnetised layer is represented by ΔZ , which is equal to Z_b minus Z_t (Figure 2).

Rozimant et al. (2009) identified a correlation between heat flow and Curie Point Depth (CPD) values but noted that this relationship does not consistently align with crustal depth. They also emphasized that the Curie point isotherm marks the boundary between the magnetic and non-magnetic regions of the crust.

Spector and Grant (1970) first introduced the method of radially averaging power spectra, which provides information about the depth of the magnetic layer. Subsequently, this method has been improved upon by Bhattacharyya and Leu (1975), Connard et al. (1983), Okubo et al. (1985), Tanaka et al. (1999), Ross et al. (2006). These advancements enable the thermal structure of an area to be determined using magnetic anomaly values. In simple terms, the radially averaged spectrum can be defined as:

$$P(|k|) = Ae^{-2|k|Z_t}(1 - e^{-|k|(Z_b - Z_t)})^2 \quad (1)$$

where A is constant, wavenumber represented by k , and depth measurements for the top and bottom of a magnetic source, denoted as Z_b and Z_t . The next step in the equation 1 involves taking the natural logarithm of both sides.

$$\ln[P(|k|)] = \ln[A] - 2Z_t(k) + 2\ln[1 - e^{-|k|(Z_b - Z_t)}] \quad (2)$$

When the wavenumber components are of high or medium values, the exponential factor in equation

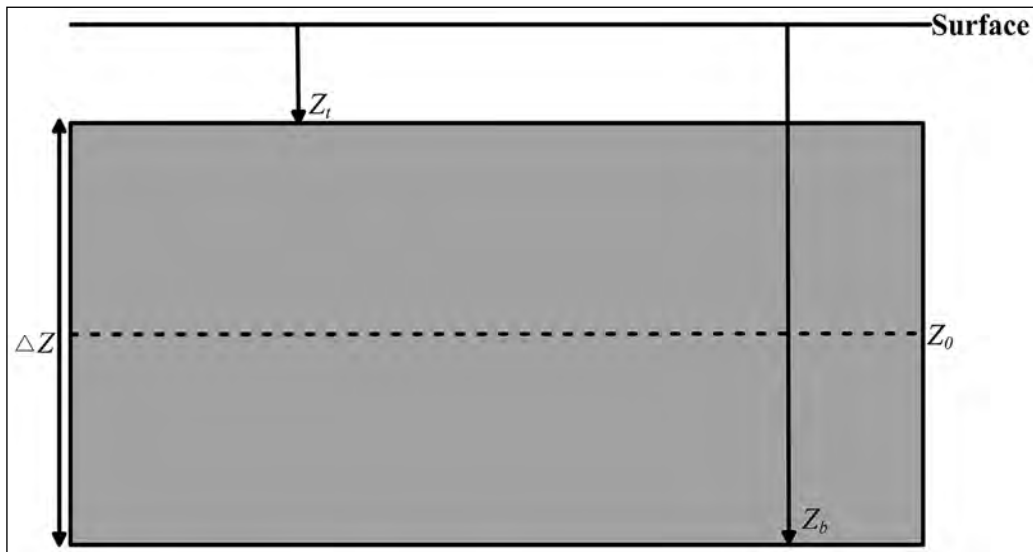


Figure 2- Schematic diagram of magnetic depths where Z_t = top of the magnetised layer, Z_b = the bottom of the magnetised layer, Z_0 = centroid depth that corresponds to $\frac{Z_b + Z_t}{2}$ and ΔZ = thickness of the magnetised layer.

2 becomes insignificant and can be disregarded. As a result, equation 2 can be expressed differently as shown in the following rewritten form:

$$\ln[P(|k|)] = \ln[A] - |k|2Z_t \quad (3)$$

Equation 3 indicates that the slope is equal to $2Z_t$. By dividing equation 3 by 2, a linear estimation model can be derived:

$$\ln[P(|k|)^{1/2}] = C - |k|Z_t \quad (4)$$

The equation 4 involves a constant value, denoted as C . The slope of equation 4 is indicative of the depth measurement to the top of the magnetic layer, represented as Z_t . To obtain an estimation for Z_0 , one can multiply equation 1 by $e^{-|k|(z_0-z_0)}$, and then divide the resulting equation by 2:

$$\ln[P(|k|)^{1/2}] = Ae^{-|k|Z_0}(e^{-|k|(Z_t-Z_0)} - e^{-|k|(Z_b-Z_0)}) \quad (5)$$

The depth to the centroid of the magnetic source is denoted by Z_0 . If we substitute the first terms of the Taylor series expansion for the exponential term in equation 5, an approximate equation for Z_0 can be obtained.

$$\ln[P(|k|)^{1/2}] \approx Ae^{-|k|Z_0}(Z_b - Z_t) \quad (6)$$

The thickness of the magnetic crust (ΔZ) is represented by $Z_b - Z_t$, as defined by Gasparini et al. (1979). By substituting and rearranging the thickness of the magnetic crust with Z_0 , we obtain:

$$\ln\left[\frac{P(|k|)^{1/2}}{|k|}\right] = \ln[C_2] - |k|Z_0 \quad (7)$$

Equation 7 involves a constant value for C_2 , while Z_0 is determined by calculating the slope for the low wavenumber components. The depth of the magnetic layer's centroid is related to the magnetic layer's bottom, as indicated by Okubo et al., (1985) and Tanaka et al., (1999):

$$Z_b = 2Z_0 - Z_t \quad (8)$$

Overall, equation 4 and 7 represent straight-line equations, and ultimately, the estimation of Z_t and Z_0 can be achieved by determining the slope of the linear fit. Once Z_t and Z_0 are estimated, it is possible to articulate the uncertainty (ΔZ_t and ΔZ_0) associated with these estimations in the following manner (Martos et al., 2017; Carrillo-de la Cruz et al., 2020):

$$\Delta Z_t = (RMS_{Z_t}/2\pi) \times [\max(k_{s_{Z_t}}) - \min(k_{s_{Z_t}})] \quad (9)$$

$$\Delta Z_0 = (RMS_{Z_0}/2\pi) \times [\max(k_{s_{Z_0}}) - \min(k_{s_{Z_0}})] \quad (10)$$

The expressions RMS_{Z_t} and RMS_{Z_0} refer to the Root Mean Square (RMS) error of the linear regression model divided by the number of data points used in the analysis. On the other hand, $k_{s_{Z_t}}$ and $k_{s_{Z_0}}$ represent the wavenumber range of the linear segment chosen to estimate Z_t and Z_0 , respectively. Ultimately, the uncertainty in the estimated value of Z_b denoted as ΔZ_b can be expressed as per the definition proposed by Martos et al. (2019):

$$\Delta Z_b = \sqrt{2\Delta Z_0^2 - \Delta Z_t^2} \quad (11)$$

4.1.1. Wavenumber Ranges for Curie Depth Estimations

To ensure the accuracy of the calculated Z_b and Z_t values, it is essential to validate them by comparing with both theoretical approximations and linear estimations. However, many authors tend to overlook this step and manually select the wavenumber ranges. In a recent study, Núñez Demarco et al. (2021) analyzed 72 articles and recommended that the mathematical validity of the computation can be evaluated by comparing the results of equations (2) and (3) for Z_t and equations (5) and (6) for Z_b . The comparison can be done by examining the difference in the slope between the linear estimation model and the theoretical curve.

$$\Delta m = \left[\frac{t(k_{i+1}) - t(k_i)}{k_{i+1} - k_i} - \frac{l(k_{i+1}) - l(k_i)}{k_{i+1} - k_i} \right] \quad (12)$$

The symbol Δm denotes the difference in slope, where $t(k)$ represents the theoretical curve and $l(k)$ denotes the linear estimation. When Δm approaches 0, it implies that the estimated values of Z_0 and Z_t are reliable. Specifically, wavenumber ranges that exhibit a difference of less than 5% between the linear approximation and the theoretical curve can be considered as valid regions for estimating Z_0 and Z_t . Generally, the valid regions for Z_t are larger than those for Z_0 . As the thickness of the magnetic layer (ΔZ) increases, the valid regions tend to be wider for Z_t and narrower for Z_0 . Additionally, when Z_t is deeper for a constant ΔZ , the confidence region becomes broader.

Figure 3 shows that the regions of confidence are estimated for the expected maximum, minimum, and average values of magnetic thickness (ΔZ). In order to achieve the expected maximum value of ΔZ (34 km),

the maximum expected value of Z_b is taken as 36 km, while the minimum expected value of Z_t is taken as 2 km. Similarly, to obtain the expected average value of ΔZ (23 km), the expected average values of Z_b and

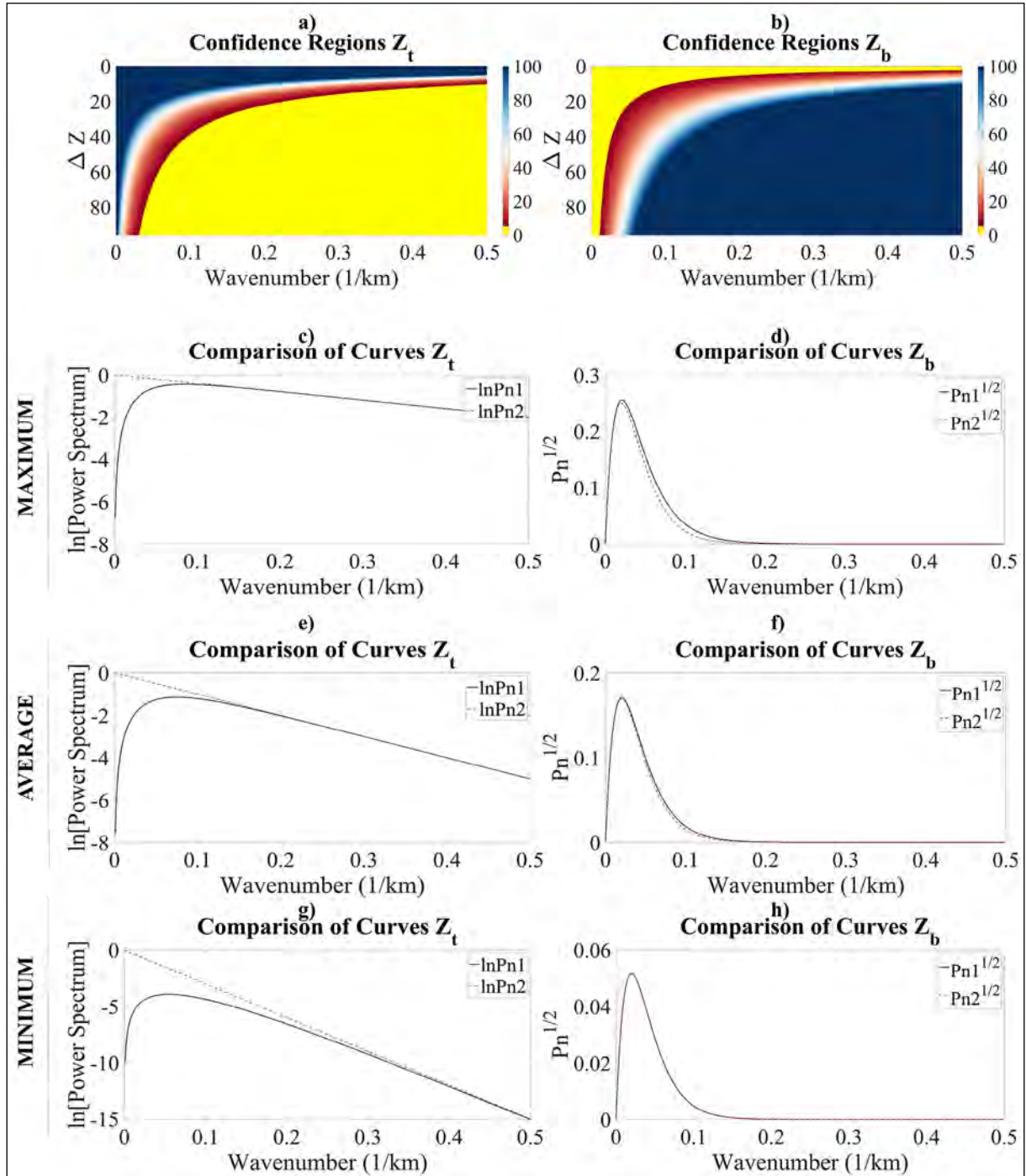


Figure 3- The confidence regions for CPD estimations are presented in the following manner: panels a) and b) display reliable ranges of wavenumbers, represented by the yellow area, where the slope difference (Δm) is less than 5%. Panels c) and d) illustrate the wavenumber ranges associated with the maximum value of ΔZ (34 km), where $Z_b = 36$ km, $Z_t = 2$ km. Panels g) and h) show the wavenumber ranges corresponding to the average value of ΔZ (23 km), where $Z_b = 28$ km, $Z_t = 5$ km. Panels e) and f) depict the wavenumber ranges for the minimum value of ΔZ (7 km), where $Z_b = 22$ km, $Z_t = 15$ km.

Z_t are assumed as 28 km and 5 km, respectively. To achieve the expected minimum value of ΔZ (7 km), the expected minimum values of Z_b and Z_t are taken as 22 km and 15 km, respectively. The assumptions for the expected values of Z_t and Z_b are based on previous studies conducted by Starostenko et al. (2014), Li et al. (2017), Tütünsatar et al. (2018).

4.1.2. Heat Flow Estimations from CPD Results

The estimation of heat flow and thermal gradient is carried out utilizing Fourier's Law (Fourier, 1878). As CPD is connected with magnetite's 580 °C temperature, heat flow can be determined based on the estimated Z_b values. The heat flow can be given as:

$$q(z) = \lambda \frac{dT(z)}{dz} \quad (13)$$

where $q(z)$ is heat flow as a function of depth (z), λ is the thermal conductivity with a value of 2.2 W/mK and $\frac{dT}{dz}$ is the geothermal gradient. To solve equation 13 for conductive heat transfer, which is outlined in (Martos et al., 2017), it is necessary to determine the boundary limits. Thus, the equation can be rearranged and expressed as:

$$q(z) = \lambda \frac{(T_c - T_0)}{Z_b} \quad (14)$$

Equation 10 considers T_c as the Curie temperature of magnetite (approximately 580°C), while T_0 represents the surface temperature, which has a value of 20°C. Notably, the equation 14 does not account for radiogenic heat production, mass advection, temperature dependence of thermal conductivity, and transient cooling, as described in (Ravat et al., 2016).

4.2. Edge Detection and Depth Estimation Methods

4.2.1. Tilt Depth Method

The magnetic tilt angle is a mathematical expression that is normalized by comparing the vertical and horizontal derivatives of the reduction to the pole (RTP) field. This approach is called the "tilt depth method" and enables a more intuitive comprehension of the changes in depth of magnetic source bodies (Salem et al., 2007). Tilt angle is initially introduced by Miller and Singh (1994). The most basic version of the tilt depth method assumes that the source formations have perpendicular interfaces, no remnant magnetism,

and that the magnetisation is oriented vertically. Then the method was subsequently refined by Verduzco et al. (2004), and subsequently defined as:

$$\theta = \arctan \left[\frac{\frac{\partial M}{\partial z}}{\frac{\partial M}{\partial H}} \right] \quad \text{where} \quad \frac{\partial M}{\partial H} = \sqrt{\left(\frac{\partial M}{\partial x} \right)^2 + \left(\frac{\partial M}{\partial y} \right)^2} \quad (15)$$

where $\partial M/\partial x$, $\partial M/\partial y$, and $\partial M/\partial z$ represent the first order differentials of the magnetic field with respect to the x, y, and z axes, correspondingly. Additionally, $\frac{\partial M}{\partial H}$ is total horizontal derivative of the magnetic field M. One can observe several interesting attributes of the tilt angle (θ). Specifically, due to the nature of the "tan⁻¹" trigonometric function, the magnitude of all tilts falls within the range of -90° to +90°, independent of the magnitude of the vertical or absolute value of the overall horizontal gradient. In addition to equalizing the amplitude of magnetic anomalies across a grid or profile, this approach preserves the spectral consistency of the signal, thereby enabling additional quantitative analysis such as the determination of local wavenumber (Salem et al., 2007).

Tilt angle over the edges of the contact is 0° ($h=0$), while it reaches 45° when h equals Z_{tilt} and -45° when h is $-Z_{tilt}$. As a result, it is plausible to use contours of the magnetic tilt angle to determine both the location ($\theta=0^\circ$) and top depth (half the physical distance between $\pm 45^\circ$ contours) of contact-like formations (Doğru et al., 2017). This phenomenon quantitatively identified as (Salem et al., 2007):

$$\theta = \arctan \left(\frac{h}{Z_{tilt}} \right) \quad (16)$$

where h is horizontal distance and Z_{tilt} refers to the depth of the top of contact-like structures that have been estimated through the use of the tilt-depth method.

4.2.2. Local Wavenumber Method

The method of determining the local wavenumber requires the calculation of both first and second derivatives in both horizontal and vertical directions. It is the most vulnerable to disturbances from noise and interference. However, if such disturbances are absent, this method can provide precise measurements of the horizontal and vertical positions of individual sources,

as well as the sources' structural characteristics (Phillips, 2000). According to Thurston and Smith (1997) work, they provide a formula for determining the local wavenumber along a 2-dimensional source by measuring a profile. Subsequently, Phillips (2000) derived an equivalent formula for the local wavenumber (LW) of the anomalous magnetic field in a gridded format:

$$LW = \frac{1}{\left(\frac{\partial M}{\partial x}\right)^2 + \left(\frac{\partial M}{\partial y}\right)^2 + \left(\frac{\partial M}{\partial z}\right)^2} \left[\frac{\partial M}{\partial x} \frac{\partial^2 M}{\partial z \partial x} + \frac{\partial M}{\partial y} \frac{\partial^2 M}{\partial z \partial y} + \frac{\partial M}{\partial z} \frac{\partial^2 M}{\partial z \partial z} \right] \quad (17)$$

The LW technique, which employs the second-order derivatives, is susceptible to noise and aliasing in the data, resulting in frequent occurrence of discontinuous contacts and inadequate management of strike direction.

Depth estimations can be conducted by LW with appropriate structural index (N). The numerical value assigned to the structural index differs depending on the geological feature being analysed. A value of zero is assigned to the uppermost portion of a contact or

fault that extends to a significant depth, while a value of one corresponds to the edge of a thin sheet. An extended pipe or cylinder is assigned a value of two, while the centroid of a sphere or dipole is assigned a value of three. The local wavenumber method can accurately determine depths for straightforward sources. However, it fails for thick dikes because of the interfering effects of the two corners, as do all other methods (Phillips, 2000). Depth is estimated using equation 18.

$$\text{At peak} \rightarrow Z_{LW} = \frac{(N + 1)}{LW} \quad (18)$$

where N is structural index which is taken as 0 for this study and Z_{LW} is estimated depth from local peak of LW grid.

5. Results and Discussion

The magnetic data obtained from the EMAG2 model was subjected to the RTP technique, employing a predetermined declination of $6.87^\circ \pm 0.35^\circ$ and an inclination of $61.43^\circ \pm 0.21^\circ$. In Figure 4, the RTP

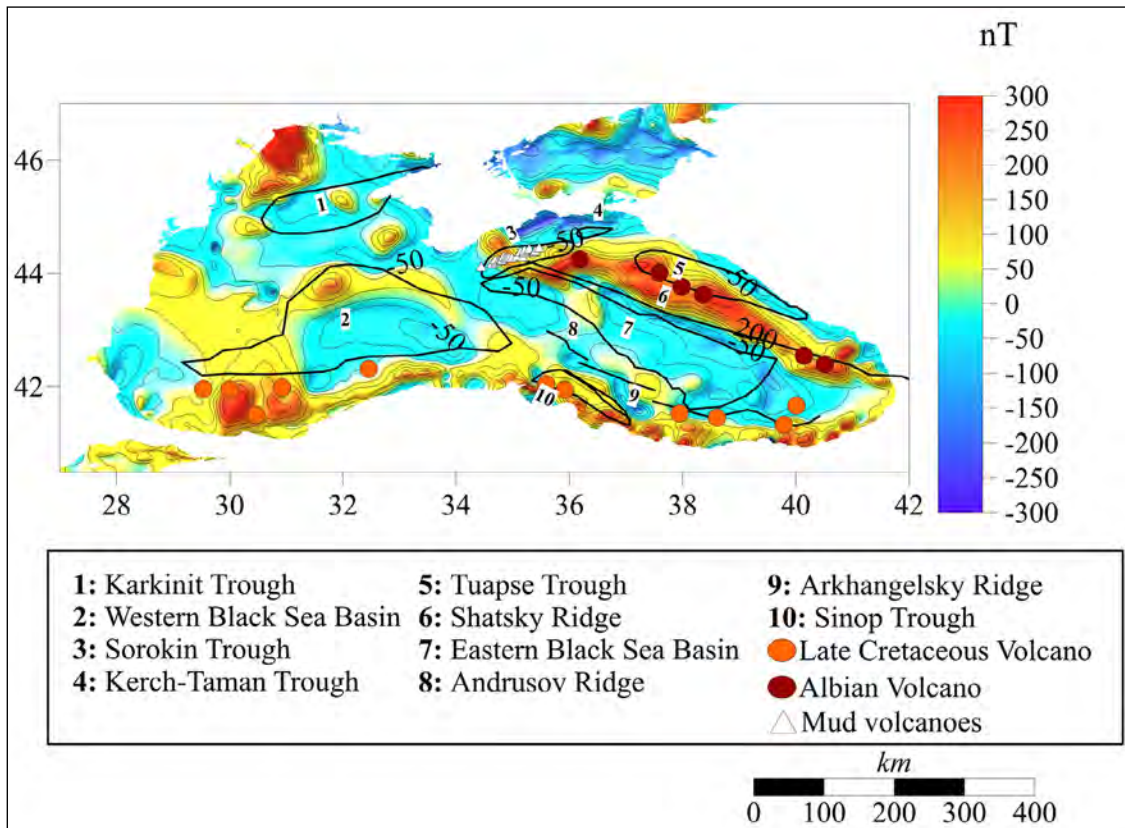


Figure 4- RTP magnetic anomaly with tectonic features in the study area.

magnetic anomaly is depicted, illustrating discernible tectonic features that contribute to the overall magnetic profile.

In each map, the locations of Cretaceous Volcanoes are derived from Nikishin et al. (2015) and Tari and Simmons (2018), while the locations of Albian Volcanoes are sourced from Nikishin et al. (2015). Additionally, the locations of mud volcanoes are digitized based on the data presented in Wagner-Friedrichs et al. (2008).

The range of RTP magnetic anomalies spans from -300 nT to 300 nT, with the most prominent magnetic anomalies localized along the Shatsky Ridge and basin boundaries. Conversely, the EBS and WBS exhibit relatively lower magnetic anomalies. The higher magnetic anomalies observed in the Shatsky Ridge and basin boundaries can be attributed to the prevalence of older magnetic-rich rocks. These regions are predominantly composed of rocks characterized by a high abundance of magnetic minerals, including magnetite, which contribute to the manifestation of larger magnetic anomalies. Additionally, the relatively thinner sedimentary layer in these areas facilitates the detection of the underlying magnetic rocks.

In contrast, the EBS and WBS present lower magnetic anomalies primarily due to their composition of younger sedimentary rocks with reduced magnetic susceptibility compared to the older rocks found in the basin boundaries and the Shatsky Ridge. Furthermore, the presence of a thick sedimentary layer covering the EBS and WBS masks the magnetic basement rocks, leading to a diminished magnetic anomaly.

The Shatsky Swell consists of the Gudanta and Ochamchiri domes, which are aligned with the structural features of the Dzirula Massif in Georgia and are composed of Cretaceous and Jurassic rock sequences. A significant positive magnetic anomaly of approximately 300 nT is depicted in Figure 4, possibly attributable to the presence of volcanic rocks. This observation implies the potential extension of Middle Jurassic volcanics, particularly Bajocian volcanics originating from the Dzirula Massif and the Gagro-Chjawa zone of the southern slope of the Greater Caucasus, into the Shatsky Swell. The Shatsky Swell

is viewed as a connecting region between the areas featuring Jurassic volcanics in the Caucasus and the Crimea Highland. In addition, the analysis of magnetic data reveals a correlation between the magnetic anomalies observed in the Pontides region and the occurrence of Cretaceous-Eocene volcanic rocks, which are extensively documented along the entire coastline. This distinctive feature is characterized by prominently positive magnetic anomalies in the northern part of Türkiye's shoreline.

Edge detection techniques, namely the tilt angle and local wavenumber (LW), were utilized to enhance the interpretation of the magnetic data, as illustrated in Figure 5. The adoption of the tilt angle method was deemed appropriate due to its effectiveness in improving the interpretation of the signal field, which spans a range of ± 1.57 radians. This particular technique proves valuable in accentuating weaker anomalies while equalizing stronger ones, enabling the identification of subtle magnetic anomalies. It is important to note that the sign of the tilt angle is determined by the vertical derivative, as the total horizontal derivative consistently maintains a positive value. Consequently, a direct comparison between the vertical derivative and the total horizontal derivative can be made, further aiding the interpretation process.

The LW technique is used for a variety of reasons. The first reason is that because of it uses second derivatives of the magnetic potential and its capacity to highlight magnetic anomalies with short wavelengths, it is a valuable tool in the analysis of shallower tectonic structures. Secondly, LW map presents higher resolution over the contacts especially narrower structures such as dykes. Finally, the depth of the contact can be estimated by inverse of the local maxima values of the LW (Fairhead, 2016).

The tilt angle map presented in Figure 5a reveals negative readings within the basin and positive values over the ridges and basin boundary. The sign of the tilt angle is influenced by the distribution and orientation of magnetic rocks. Horizontally stratified magnetic rocks within the EBS and WBS contribute to elevated values in the horizontal magnetic field and reduced values in the vertical derivative. Additionally, the presence of younger sedimentary rocks in the basin,

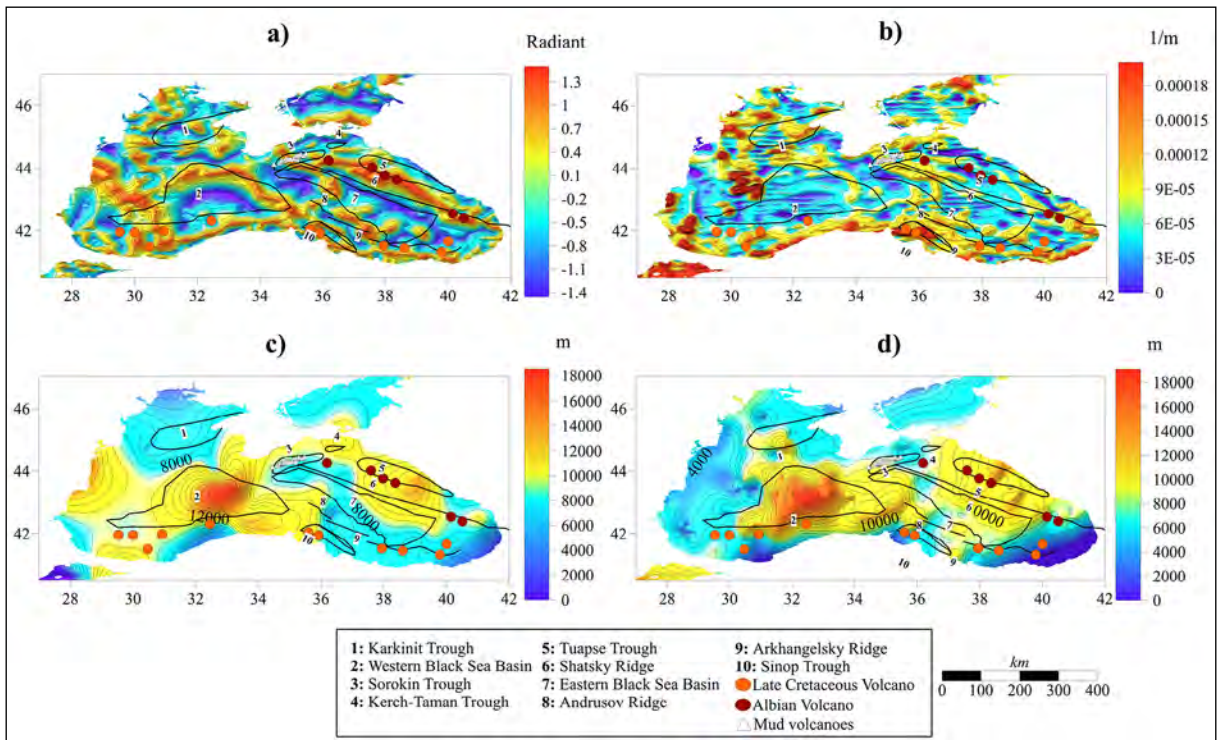


Figure 5- Results of the edge detection methods with tectonic features in the study area: a) Tilt angle map, b) LW map, c) tilt depth, d) LW depth.

exhibiting a negative magnetic susceptibility, results in notable negative anomalies within the tilt angle map. In contrast, ridges often consist of vertically-oriented magnetic rocks, which yield higher values in the vertical derivative and positive tilt angle values. These rocks exhibit a positive magnetic susceptibility. Thus, the determination of the tilt angle map's sign hinges upon the arrangement and quantity of magnetic rocks. At the interface between basement and sedimentary rocks, a sudden shift in magnetic characteristics occurs, leading to an immediate change in the magnetic field and a steep gradient along the edges. Nonetheless, this gradient approaches zero at two neighboring points along the contact boundary. Consequently, tilt angle values either become zero or converge to zero at contact points between distinct rock units.

The LW map, obtained by computing the first and second derivatives of the magnetic field, exhibits local maxima at the contacts between distinct rock types, as demonstrated in Figure 5b. These contacts represent locations where substantial variations in magnetic properties occur, resulting in prominent gradients

that can be detected through the first and second derivatives of the magnetic field. The first derivatives, both in the horizontal and vertical directions, enable the detection of the magnetic field's rate of change, while the second derivatives, also in the horizontal and vertical directions, are particularly responsive to abrupt changes within the LW map. Thus, the presence of local maxima within the LW map serves as a reliable indicator of the existence of notable magnetic gradients.

Figure 5c and 5d provide visual representations of the estimated depth of the uppermost portion of the contact, as inferred from the analysis of the tilt angle and LW maps. Notably, an overestimation of depth by approximately 18 km is observed in the WBS when compared to the EBS. This disparity can be attributed to a combination of factors, including tectonic structures, magmatic content, and sediment thickness.

One factor contributing to the exaggerated depth estimations in the WBS, as opposed to the EBS, is the presence of the Shatsky and Andrusov Ridges. Surrounding the EBS, these ridges generate shorter-

wavelength anomalies, resulting in shallower depth estimations derived from both the tilt depth and LW depth analyses.

Another influential factor is the magma content of the EBS and WBS. The WBS is characterized as magma-poor, while the EBS is known for its magma-rich nature (Kaymakçı et al., 2015). This discrepancy in magma composition directly affects the magnetic properties of the rocks and sediments within these basins, consequently influencing the resulting depth estimations.

Furthermore, sediment thickness plays a crucial role in the wavelength content of the tilt and LW maps. Typically, the WBS exhibits a higher sediment thickness compared to the EBS, leading to the observed overestimation of depth within the WBS.

In addition to employing edge detection methods, such as tilt and LW, the study area involved the calculation of Curie point depth (CPD) and heat flow. The CPD calculations were conducted by dividing

the study area into 65 blocks measuring 200×200 km each, with a 50% overlap between adjacent blocks. The size of these blocks played a crucial role in determining the vertical resolution of Z_b . Following the recommendation of Salem et al. (2014), a block size six times larger than the desired depth was utilized. In this particular study, a block size of 200×200 km resulted in an approximate vertical resolution of 33 km. The CPD values obtained were subsequently employed to infer the heat flow, disregarding the contribution of radiogenic heat production. Consequently, an inverse correlation is observed between the distribution of CPD and heat flow. The outcomes of the CPD calculations, top depth of the magnetic source derived from spectral analysis, and heat flow assessments are visually presented in Figure 6.

Figure 6a provides data indicating a greater Curie point depth (CPD) in the WBS as opposed to the EBS. Furthermore, the CPD values in the EBS exhibit abrupt changes, while the WBS demonstrates a more consistent pattern. These variations can be attributed

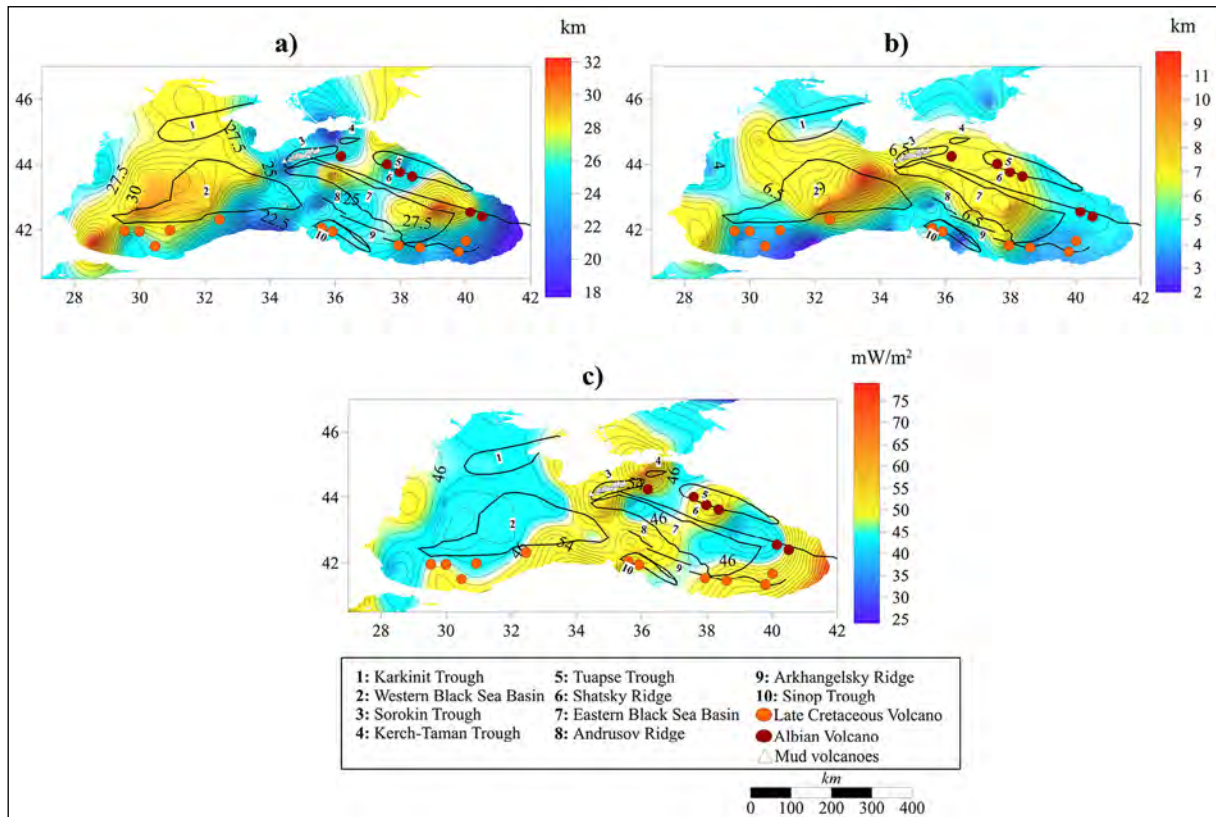


Figure 6- Results of the CPD, Z_t and heat flow with tectonic features in the study area: a) CPD map, b) Z_t map, c) heat flow map.

to various factors, including sediment thickness, the presence of volcanic rocks, and the tectonic history of the EBS and WBS.

To begin with, the thermal insulation provided by sediment thickness hinders heat transfer from the underlying mantle, resulting in deeper CPD values for both the EBS and WBS. Additionally, the WBS exhibits even deeper CPD readings in comparison to the EBS due to its thicker sediment cover.

Moreover, the presence of volcanic rocks enhances the amplitude of higher wavenumber sections, leading to shallower CPD values in the EBS. Furthermore, the abundance of volcanic rocks within the Shatsky Ridge, situated in the northeastern part of the Black Sea, influences the computation of CPD estimates in the EBS. The spectral characteristics of the short-wavelength magnetic anomaly originating from the Shatsky Ridge impact the CPD calculations in the adjacent blocks of the EBS, resulting in underestimated CPD values within the EBS.

Lastly, it is important to note the active tectonic processes occurring in the EBS, while the WBS exhibits a relatively more stable condition. The tectonic instability around the margins of EBS can trigger the upwelling of hot mantle materials. This explanation accounts for the shallower CPD values and higher heat flow observed in the EBS in comparison to the WBS. Letouzey et al. (1977) suggested that the higher heat flow values observed to the south of Crimea and west of the Caucasus might be associated with mud diapirism. Mud volcanoes are known to play a significant role in heat transfer, particularly in geothermal areas.

The Z_t map in Figure 6b provides insights into the top depth of the magnetic basement. Nevertheless, it is important to note that Z_t estimations are more subjective compared to the tilt and LW methods. Therefore, the Z_t estimations were solely utilized in correlation plots to facilitate a comparison of results with sediment thickness, LW maps, and tilt maps.

To facilitate additional investigation, two profiles were designated in the EBS and the WBS as illustrated in Figure 7. Profile A-A' was selected along the WBS, while profile B-B' was chosen along the EBS. These profiles were assessed to evaluate various parameters,

including global CPD (Li et al., 2017), Moho depth (Laske et al., 2013), estimated CPD values (Z_b values), LW depth, tilt depth, sediment thickness (Laske et al., 2013), Z_t values, and bathymetry data (Amante and Eakins, 2009).

The correlation plot depicted in Figure 8 was thoroughly examined to assess the relationships between different parameters along the A-A' and B-B' profiles. Based on the analysis, the following expected relationships between the parameters can be outlined:

- Moho depth and bathymetry are projected to exhibit an inverse correlation. This can be attributed to the requirement for the lithospheric load to be balanced or compensated by adjustments at the Moho interface, resulting in a reciprocal relationship between bathymetry and Moho depth.
- A positive correlation is anticipated between the estimated CPD and global CPD.
- A positive correlation is expected among LW depth, tilt depth, and Z_t values. These parameters provide valuable insights into the uppermost depth of the magnetic basement. Consequently, it is anticipated that at least two of these parameters will exhibit a positive correlation.
- The positive correlation between sediment thickness, LW depth, tilt depth, and Z_t values suggests a potentially rapid sediment accumulation process. The LW depth, tilt depth, and Z_t values are indicators of the top depth of the magnetic basement. If an increase in sediment thickness coincides with an increase in the top depth of the magnetic basement, it can be inferred that sedimentation accumulation rate remains unaffected by deformation, folding, seismic activity or variations in riverbed conditions.

The correlation plots shown in Figure 8 provide a quick and simultaneous comparison of various physical and geometric parameters. These correlation plots are essentially lower triangular matrices, where the comparison of the same geometric and physical parameters results in a value of 1.

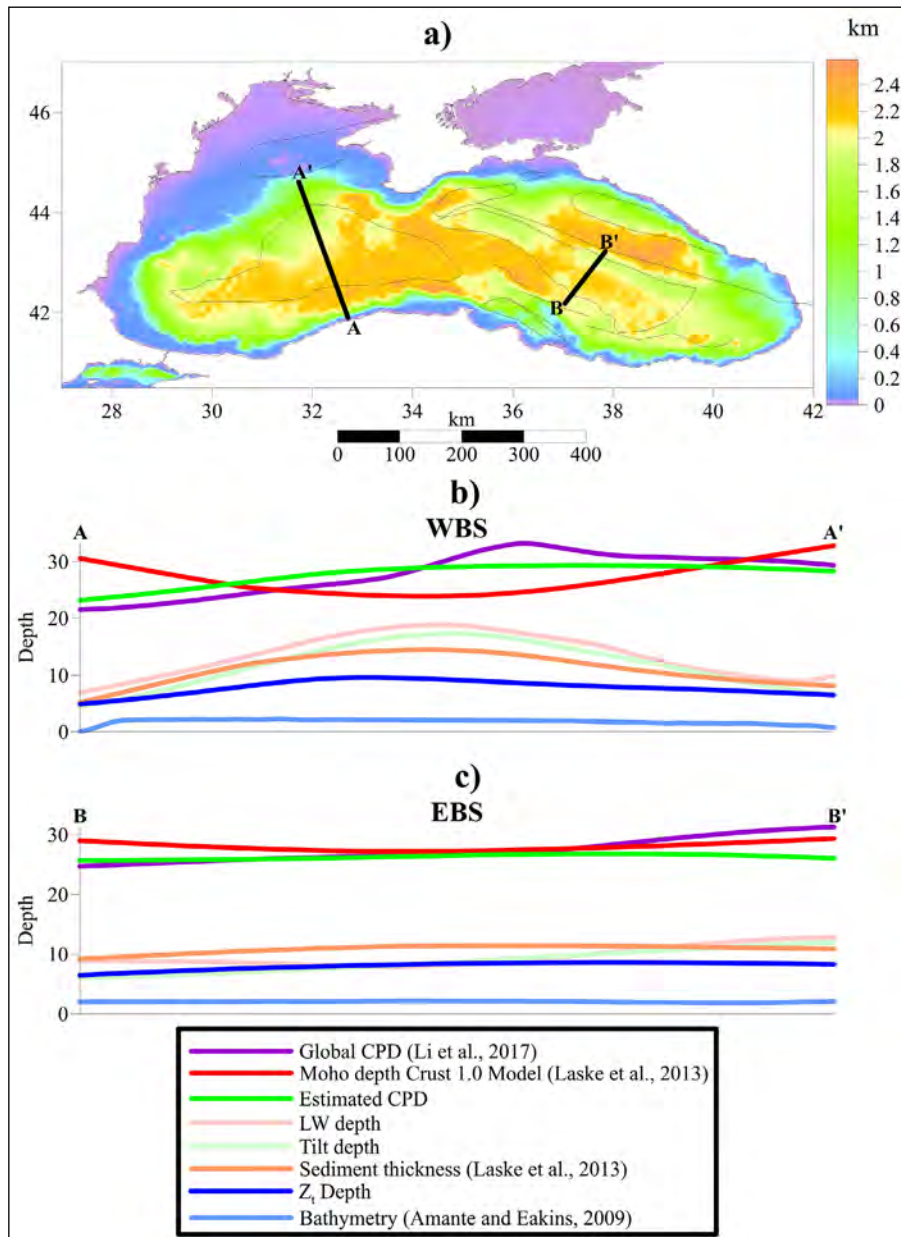


Figure 7- The selected profiles along the WBS and EBS: a) profile locations overlaid on the bathymetry, b) profile A-A' along the WBS, and c) profile B-B' along the EBS. Each profile includes the Global CPD, Moho depth, Estimated CPD, LW depth, Tilt Depth, sediment thickness, Z_1 , and bathymetry.

Figure 8 presents the correlation analysis conducted for each variable along the A-A' profile representing the Western Black Sea (WBS) and the B-B' profile representing the EBS. The correlation coefficients range from -1 to 1, with 1 indicating a perfect positive correlation, 0 denoting no association, and -1 representing a complete inverse correlation. Specifically, the focus was on examining the correlation between the estimated Curie point depth (E CPD) and

the global CPD (G CPD) to compare our estimations with the findings reported by Li et al. (2017). Along the A-A' profile (WBS), a strong correlation of 0.93 is observed between G CPD and E CPD, while along the B-B' profile (EBS), a moderate correlation of 0.58 is found between these variables. It is anticipated to observe a high correlation between E CPD and G CPD. However, the relatively lower correlation along the B-B' profile may be attributed to various factors,

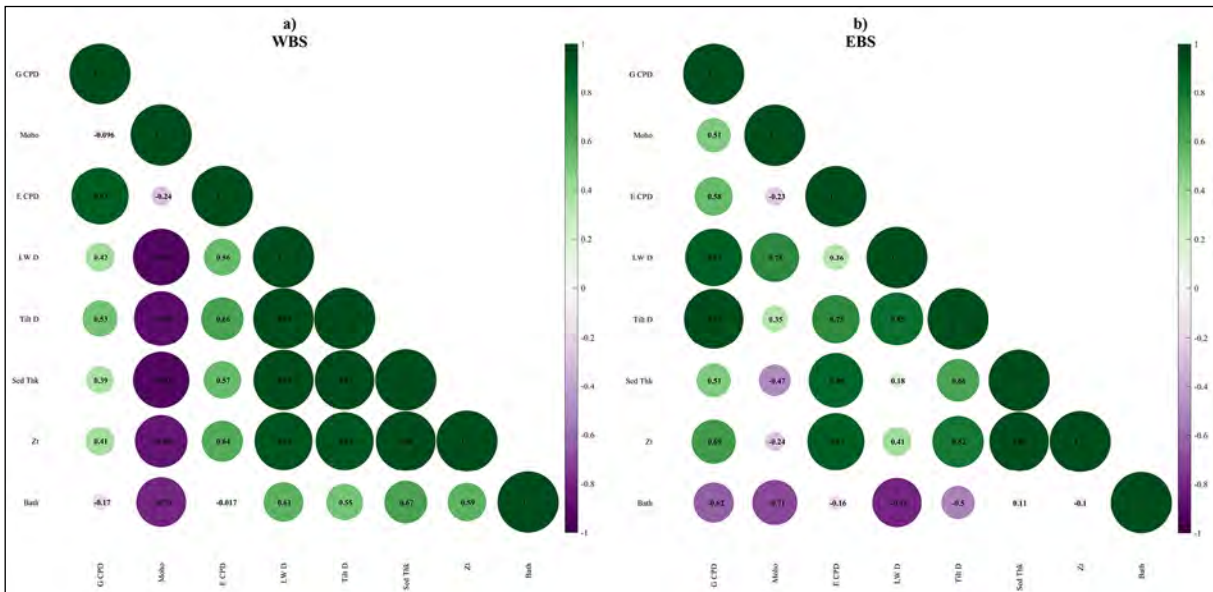


Figure 8- Correlation plots of WBS (A-A' profile) and EBS (B-B' profile). The radius of the circles is controlled by the correlation value. G CPD: global Curie point depth, Moho: Moho depth, E CPD: estimated Curie Point Depth, LW D: Local Wavenumber depth, Tilt D: Tilt depth, Sed Thk: sediment thickness, Z_t: top depth of the magnetic basement and Bath: bathymetry.

including the grid cell size, chosen block size, range of selected wavenumber components during CPD estimation, or the positioning of overlapping adjacent blocks.

As mentioned previously, Z_t, tilt depth (Tilt D), and LW depth (LW D) are indicators of the uppermost depth of the magnetic basement. Therefore, a strong correlation is expected among these variables, approaching a value of 1. Along the A-A' profile, the correlation between these variables exceeds 0.94, indicating a strong positive correlation. However, along the B-B' profile, while Tilt D - Z_t and Tilt D - LW D exhibit a high correlation, the correlation between LW D and Z_t is relatively low at 0.41. This lower correlation may arise from distinct characteristics of LW D and Z_t in the south-western part along the B-B' profile (Figure 7c).

The correlation between sediment thickness (Sed Thk), Tilt D, LW D, and Z_t plays a crucial role in understanding sediment accumulation rates. Along the A-A' profile, the correlation values between Sed Thk and LW D, Sed Thk and Tilt D, and Sed Thk and Z_t are above 0.97. These findings suggest a strong association between the top depth of the magnetic basement inferred by Tilt D, LW D, Z_t, and Sed Thk.

It is possible that the sediment deposition process remains uninterrupted by deformation, folding, seismic activity, or variations in riverbed conditions.

Furthermore, the higher sedimentation rate can be attributed to Balkanide Orogeny during Eocene. The Western Black Sea, being older, had more time for sediment deposition and provided a more suitable environment for sediment accumulation. Therefore, the expected outcome of a higher sediment accumulation rate in the WBS is justified by the correlation values of Sed Thk, Tilt D, LW D, and Z_t. Additionally, Nikishin et al. (2015) emphasized a higher sedimentation rate in the WBS from the Cretaceous to Eocene period, further supporting these findings.

The outcomes of Spadini et al. (1996) findings have yielded significant insights into the origins of the WBS and EBS regions, highlighting fundamental differences in their lithospheric properties. Notably, the WBS's formation can be attributed to the rifting of a thick lithosphere (approximately 200 km), characterised by lower temperatures and higher mechanical strength, thereby contributing to a substantial depth of necking (around 25 km). This thick lithosphere corresponds to the stable continental Moesian Platform. Conversely, the EBS's genesis is associated with the rifting of a

thinner lithosphere (approximately 80 km), resulting in elevated temperatures and weaker mechanical properties, leading to a shallower depth of necking (approximately 15 km). This scenario aligns well with the development of the EBS on the site of a prior (Mesozoic) back-arc basin (Spadini et al., 1996). To summarize, the geological history of the Black Sea region exhibits notable differences between its western and eastern basins. In the WBS, the rifting process was primarily responsible for the development of the stable Moesian Platform. On the other hand, in the Eastern Black Sea, a younger extensional basin was formed, overlaying a pre-existing area that had previously acted as a back-arc basin since the Early Jurassic period. This intriguing contrast in geological evolution sheds light on the diverse tectonic processes shaping the Black Sea basin's geology. These distinctive lithospheric properties and structural features offer plausible explanations for the observed variations in correlation plots between the Eastern and Western Black Sea regions.

6. Conclusion

This study presents a comprehensive theoretical framework aimed at unravelling the origin of the Black Sea, with a specific emphasis on both the East and West Black Sea basins. By leveraging a diverse range of data, including CPD values, heat flow values derived from CPD data, bathymetry, Moho depth, sediment thickness, and magnetic depth estimation techniques (Tilt angle and LW methods), a detailed examination of the region's geological characteristics has been conducted. Particularly, the impact of volcanic rock distribution on magnetic anomalies within the Black Sea area has been investigated. Moreover, an extensive analysis of the correlations between CPD values, bathymetry, Moho depth, sediment thickness, tilt depth, and LW depths was performed independently for the EBS and WBS basins. By scrutinizing these correlations in light of the tectonic history and presence of volcanic rocks, the significant disparities observed between the EBS and WBS have been thoroughly interpreted. This research has provided valuable insights into the distinct geological processes and contributing factors that have shaped the development and divergence of the EBS and WBS.

On the whole major findings in this study can be summarised as:

- Edge detection techniques, namely the tilt angle and LW methods, have played a crucial role in enhancing the interpretation of magnetic data. The analysis of tilt angle and LW maps reveals a significant difference in the estimated uppermost contact depth between the WBS and the EBS, with the WBS showing an approximately 18 km higher depth than the EBS.
- The WBS exhibits a higher Curie Point Depth (CPD), ranging up to 32 km, compared to the East Black Sea (EBS), where CPD values range between 24 to 28 km. Additionally, the CPD values in the EBS demonstrate abrupt changes, while the WBS displays a more consistent pattern, indicating the presence of thermal and magnetic property variations between these two regions.
- The correlation between Sed Thk, Tilt D, LW D, and Z_c is crucial for assessing sediment accumulation rates. Along the A-A' profile, the correlation values between these variables are all above 0.97, indicating a strong association between the top depth of the magnetic basement and sediment thickness. This suggests undisturbed sediment deposition, unaffected by deformation, folding, seismic activity, or variations in riverbed conditions.
- In aggregate, the findings derived from this empirical inquiry delineate that the two basins underwent divergent tectonic processes.

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Sediment thickness in the Thrace Basin estimated from the three-dimensional inversion of magnetotelluric data

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Research Article

In Memoriam

by Ahmet Tuğrul Başokur

The first author, Cemal Kaya, passed away in February, 2022. The Turkish geophysical community will always remember Cemal Kaya's friendship, honesty and sincerity. He graduated from İstanbul Technical University (ITU) Faculty of Mines, Department of Geophysical Engineering in 1981. He started his career as a geophysical engineer at the General Directorate of Mineral Research and Exploration. He completed his MS and PhD under my supervision at Ankara University. We completed many research projects in the 1990s, including the controlled-source audio-magnetotelluric (CSAMT) method for massive sulfide exploration and the magnetotelluric method in geothermal resource exploration, the first examples in Türkiye. We also initiated the systematic use of the MT method in crustal research as a joint TÜBİTAK project of Ankara University and the General Directorate of Mineral Research and Exploration in 1995. This article is the first profile of national crustal research project. Since only two-dimensional modelling could be applied in the 1990s, a conductive artefact zone appeared under the Hisarlıdağ uplift in the resistivity model since the Thrace Basin surrounded by the Aegean, Marmara and Black Seas on three sides. Now, we have constructed a more realistic model of the Thrace Basin using the three-dimensional modelling that permits including the seabed topography.

Keywords:

Three-Dimensional Magnetotelluric Inversion, Thrace Basin, Sediments Thicknesses.

ABSTRACT

The time-variation of the magnetotelluric (MT) fields was recorded at 37 measurement stations in the Thrace Basin (North-western Türkiye) by a remote-reference MT system that records the MT data in two frequency bands allowing a data acquisition between 320–7.5 Hz and 6–0.00055 Hz, respectively. The measurement stations were located from the Aegean Sea to the Black Sea seashores along a 207 km profile in the southeast-northwest direction. Three-dimensional inversion of MT data outlined the main geological elements of the region, the interface between sedimentary cover and basement rocks. The total thickness of the sedimentary layers in the basin is interpreted using the inflection and deflection points of the three-dimensional resistivity model in the vertical direction.

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1. Introduction

MT studies have been used to investigate oil, gas and geothermal reservoirs, deep sedimentary basins, the upper mantle and the crustal thickness for more than six decades (Swift, 1967; Vozoff, 1972; Jupp and Vozoff, 1977; Beblo et al., 1983; Wannamaker, 1983; Hersir and Björnsson, 1991; Lezaeta et al., 2000; Gokarn et al., 2008; Kaya, 2010; Hacıoğlu et al., 2020; Başokur et al., 2022; Hacıoğlu et al., 2023). We used the MT method to investigate the thickness of the sedimentary layer in the Thrace Basin of northwestern Türkiye because of its cost-effective implementation along a profile. The basin is one of the most important hydrocarbon production areas in Türkiye (mainly natural gas). The existing drill holes ended in the sedimentary cover around the MT line. An estimation for the total sediment thickness was published by Siyako and Huvaz (2007) using seismic and well data as a contour map. Three-dimensional inversion of MT data provides additional information about the

shape and depth of the basement along the 207 km line between the Aegean and Black Seas seashores.

2. Geology of the Thrace Basin

The Thrace Basin is considered to be an intramontane Tertiary basin surrounded by the Strandja in the north, the Rhodope in the west, the İstanbul Zone in the east, and the Sakarya Continent and Intra-Pontid Suture in the south, as shown in Figure 1. The basement of the surrounding massifs consists of metamorphics, İstanbul Paleozoic-Mesozoic sedimentary sequences, ophiolitic melanges and occasional igneous bodies of the pre-Upper Cretaceous age (Doust and Arıkan, 1974; Turgut et al., 1983; Siyako and Huvaz, 2007). Okay et al. (2023) partitioned the sedimentary layers into three sequences separated by major unconformities: Lower-middle Eocene, Middle/upper Eocene-Oligocene, and Miocene-Pliocene. The basin is elliptical, and its long axis extends in the southwest-northeast direction. A series of basement step faults limits the

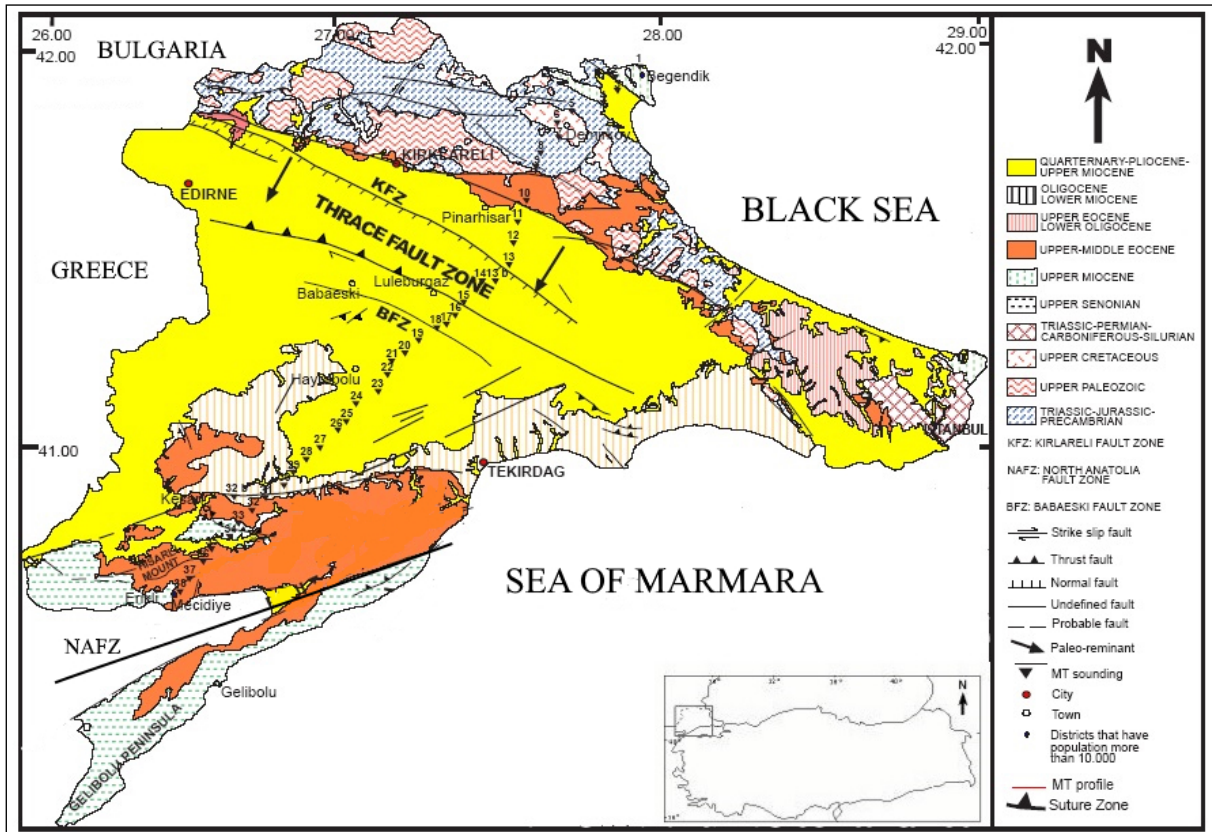


Figure 1- Geological map of Thrace Basin and MT stations (station numbers over triangles), modified from the Geological Map of Türkiye, General Directorate of Mineral Research and Exploration.

basin in NE, and a fault scarp controls the flanks of the basin in SE. Very thick flysch-type clastics were due to a very fast subsidence. Some clastic materials were deposited in the Lower Oligocene because of continuous transgression that caused the submerging of the southwestern parts of the basin. In contrast, the carbonates were deposited on the northern shelf simultaneously. An active volcanism caused dacitic and andesitic ashes. In the Middle Oligocene, the subsidence slowed, and the basin's depositional environment became lacustrine and fluvial. The deposition continued without interruption until the Upper Oligocene. Volcanic activities lost intensity in the Upper Oligocene (Turgut et al., 1983; Siyako, 2006).

Doust and Arıkan (1974) postulated that a major erosional phase caused the removal of much of the Lower and Upper Oligocene sediments into the centre and southern part of the basin. The Lower and Upper Oligocene sedimentary sequence demonstrates a transgressive-regressive depositional cycle that includes shallow marine sediments. A major tectonic event in the Middle Eocene gave rise to faulting and folding in the SW-NE direction. The late Pliocene tectonic activities shaped the present configurations of the basin (Turgut et al., 1983). On the other hand, according to Perinçek (1991) and Perinçek et al. (2015a), the final most important tectonic event in Thrace Basin occurred before the deposition of Ergene formation. Thrace fault system became active before the Ergene formation and continued during the sedimentation of it. Thrace fault systems cause uplift and erosion along the fault zones (Perinçek et al., 2015b).

Two tectonic trends are dominant in the study area. 1) The Ganos Mount trend is in the NE-SW direction parallel to the Ganos Mount and constitutes the southward limit of the basin. 2) The Strandja trend has an NW-SE strike parallel to the Strandja basement ridge, limiting the north basin (Doust and Arıkan 1974). Coşkun (1997) postulates that the older structures trending in the NE-SW direction were influenced by basement paleotopography, and the younger structures trending in the NW-SE direction were under the influence of the North Anatolian Fault (NAF). Perinçek (1991) interpret

the Thrace fault system as the oldest strand of the North Anatolian Fault, releasing-bend extensional structures. The Thrace strike-slip fault system consists of the west-northwest-striking Kirklareli, Lüleburgaz, and Babaeski fault zones that are inactive presently (Perinçek, 1991).

The collision between the Sakarya and Rhodope continents leads to lateral movements and normal basement faults, which become detachment faults during the subsequent opening of the Thrace Basin (Turgut et al., 1991; Tüysüz et al., 1998). The Bouguer gravity map clearly shows these trends and, the thick basin fills are represented by a low value of gravity anomalies (Figure 2). Sakıncı et al. (1999) suggested that the basin principally developed by the activities of the Thrace and Ganos strike-slip fault systems following the continental collision during the Late Oligocene-Early Miocene period. Thrace Basin was opened during the Middle Eocene on Strandja Massif as a post-collisional fault-controlled extensional basin and turned into an intramontane basin in the Middle Miocene (Tüysüz et al., 1998). However, a fore-arc origin for the Thrace Basin has been proposed, which is linked to the subduction of the Intra-Pontide Ocean between the Middle Eocene and Oligocene. The Rodope-Pontide magmatic arc was developed on the overriding Strandja zone and started to rift and extend, forming the Thrace Basin (Görür and Okay, 1996).

3. Data Acquisition and Processing

The MT profile lies in the southwest-northeastern direction from the Aegean Sea to the Black Sea and consists of 40 measurement stations (Figure 1). Station intervals ranged from 4 to 5 km, depending on the accessibility of the territory. The total length of the profile was 207 km. The first and last measurement stations were close to the Black Sea and Aegean coast.

The magnetotelluric (MT) method simultaneously measures orthogonal components of the natural horizontal electric and magnetic fields. The electromagnetic variations, lasting longer than 1 second, consist of micropulsations originating from the earth's magnetosphere's interaction with charged particles ejected from the sun and other sources. Lightning strokes almost entirely generate

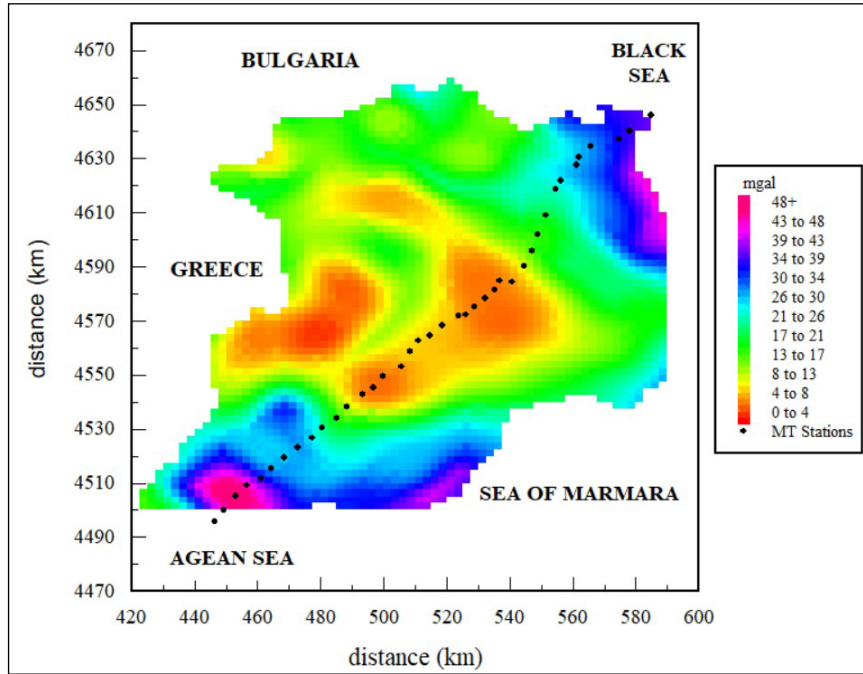


Figure 2- Location of MT stations used in this study over Bouguer gravity anomaly map (MTA, 1999).

electromagnetic variations below one second. Phoenix-V5 equipment was used to acquire MT data consisting of three orthogonal magnetic (H) and two orthogonal electric (E) field components. MT electrical dipoles of 100 m, equal to the length of one side of the TEM transmitter loop, were applied to measure E-fields in NS and EW directions. Nonpolarizable lead-lead chloride ($Pb-PbCl_2$) electrodes were used for E-field measurement because of their long-term stability of polarization (Petiau and Dupis, 1980). Induction coils and an air loop on the ground were used to measure the horizontal and vertical components of the magnetic fields, respectively. Additional two orthogonal magnetic fields were measured at a remote reference station.

The data were acquired in two steps by measuring high and low frequency bands in 1995. The high band consisted of 12 frequencies between 320 and 7.5 Hz. There were 28 frequencies in the low band in the range of $6-5.493 \times 10^{-4}$ Hz. MT records were acquired for more than 24 hours in the lower band. The impedance values in 40 frequencies were combined in the frequency domain after the Fourier transformation of time-domain data using cascade decimation (Wight and Bostick, 1980).

The orthogonal components of the horizontal electric and magnetic fields measured in the time-domain are linearly dependent. Consequently, their Fourier transform leads to a 2×2 tensor, and its elements are called impedances (Z). Impedance values are estimated from a complex electric field (E) ratio over the magnetic field (H) in the frequency domain. The tipper (T) connects the vertical and two horizontal magnetic fields. The impedance tensor and tipper vector are given in the frequency domain after the application of Fourier transformation as follows (Equations 1, 2) :

$$\begin{pmatrix} E_x \\ E_y \end{pmatrix} = \begin{pmatrix} Z_{xx} & Z_{xy} \\ Z_{yx} & Z_{yy} \end{pmatrix} \begin{pmatrix} H_x \\ H_y \end{pmatrix} \quad (1)$$

$$\begin{pmatrix} H_z \end{pmatrix} = \begin{pmatrix} T_x & T_y \end{pmatrix} \begin{pmatrix} H_x \\ H_y \end{pmatrix} \quad (2)$$

where x and y denote the orthogonal components in the north and east directions, and z indicates the vertical component. The impedances are always a decreasing function of the period, and the shape of MT curves is independent of subsurface resistivity distribution. Then, the general behaviour of impedance data may not give any idea about the resistivity variation

in the subsurface. The following normalization (Equation 3) improves the physical significance of the data (Başokur, 1994; 1999):

$$F = \frac{Z}{\sqrt{i\omega\mu}} = \frac{I}{\sqrt{i\omega\mu}} \frac{E_x}{H_y} \quad (3)$$

where $i = \sqrt{-1}$. F is named as the frequency normalized impedance or shortly FNI. The unit of the FNI function is the square root of ohm-m ($\sqrt{\text{ohm-m}}$). μ and ω denotes the magnetic permeability of free space and angular frequency, respectively. This normalization connects the measured data and the subsurface resistivity distribution and directly reflects resistivity variations versus depth as the function period (Başokur, 1999; Başokur, 2023). The validity of the measured MT data can be tested using the causality concept. Başokur (2023) proved that the real and imaginary parts of the FNI function can be calculated from each other via the Hilbert transform. This procedure help check if the data satisfies natural EM field conditions. As an example, Figure 3 shows the measured and Hilbert-transformed FNI data for the station 33 and proves the data validity for this station.

4. Three-dimensional Inversion

The purpose of geoelectrical data inversion is to estimate the subsurface resistivity distribution. A resistivity model is derived indirectly by searching for a fit between theoretical data computed for a conceptual model and the measured data. The most commonly used conceptual models are 1D, 2D or 3D subsurfaces in historical order. The reliability of the derived model is dependent on the consistency of the conceptual model with the actual surface conditions. The type of conceptual model should be decided before geophysical exploration so that the survey design can be planned according to the selected conceptual model. All constants defining a particular conceptual model are named ‘model parameters’. A 3-D model consisting of a huge number of cube-shaped cells is preferred for a better description of subsurface physical conditions. Usually, the inversion algorithm is initialized by a homogeneous earth model by assigning the same resistivity value to all model cells. The model parameters only comprise the resistivity values of each cell. Keeping the geometry of cells constant during iterations drastically reduces the number of model parameters to be estimated.

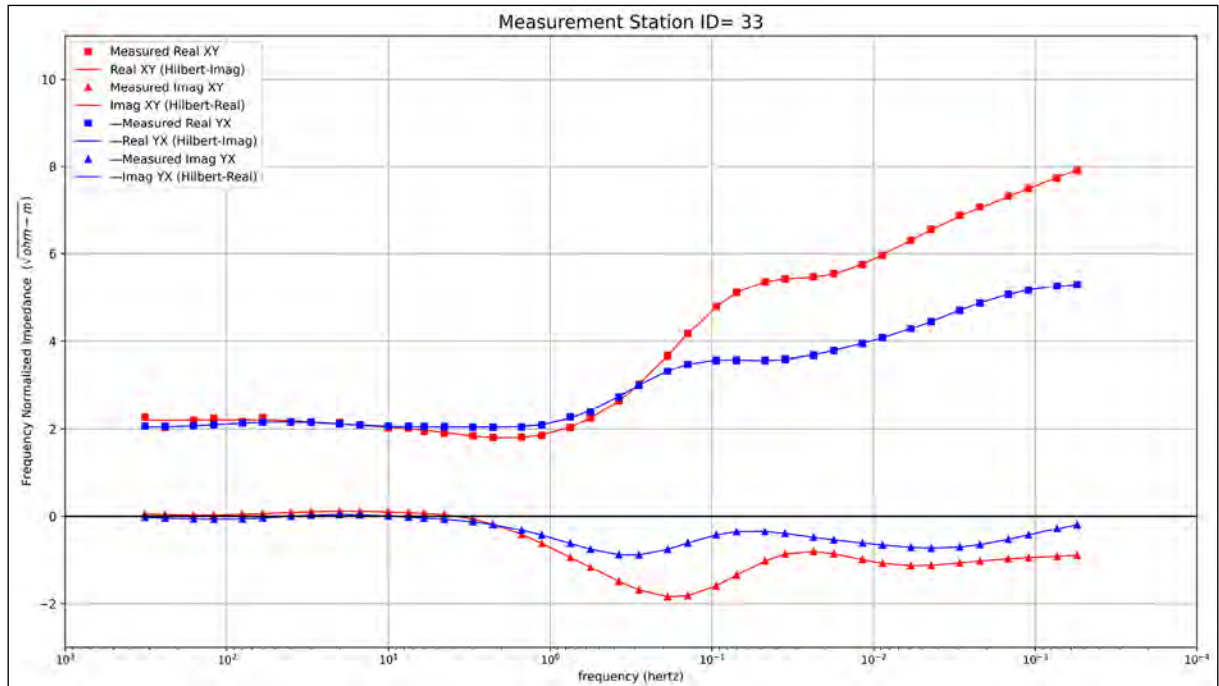


Figure 3- Frequency normalized impedance, real (squares) and imaginary (triangles) components, and their corresponding Hilbert transforms (continuous curves).

Then, the cell resistivity values are updated iteratively by computing a parameter update vector at each iteration from the differences between measured and theoretical data sets (Başokur and Akca, 2011). Models corresponding to misfit values $E(p)$ lower than a threshold are considered a possible solution to represent subsurface resistivity distribution. The reduction of misfits along successive iterations is not sufficient to obtain a smooth and geologically acceptable model. Also, preventing random resistivity changes among neighbouring cells is necessary. For this purpose, a stabilizer function $S(p)$ is added in the objective function $\phi(p)$ to control the model resistivity variations as follows (Equation 4):

$$\phi(p) = E(p) + \alpha S(p) \quad (4)$$

where α is the regularization coefficient. The misfit function is defined as the sum of the square of the differences between measured and calculated data values at each frequency (Equation 5) :

$$E(p) = \sum_{i=1}^n \frac{(d_i - f_i)^2}{\sigma_i^2} \quad (5)$$

d_i and f_i are the measured and theoretical data, respectively. σ_i is the standard deviation corresponding to i th frequency value. n is the number of measured data values. The $nmrs$ value quantifies the degree of misfit, and an $nmrs$ value which is lower than three is expected in the final step of inversion (Equation 6):

$$nmrs = \frac{1}{n} \left[\sum_{i=1}^n \frac{(d_i - f_i)^2}{\sigma_i^2} \right]^{1/2} \quad (6)$$

The 3D non-linear conjugate gradient (NLCG) software was used and named ModEM (Egbert and Kelbert, 2012; Kelbert et al., 2014). ModEM is a modular system of parallel computer codes for the inversion of electromagnetic (EM) geophysical data and uses the following stabilizer function (Equation 7):

$$S(p) = \sum_{j=1}^m \frac{(m_j - m_j^0)^2}{\sigma_j^2} \quad (7)$$

where m_j is the resistivity value of j th cell and m_j^0 corresponds to the initial resistivity value of a priori model. σ_j represents the model covariance value corresponding to the j th cell. m is the number of model parameters. A 3-D MT inversion requires a lot

of computing time despite using a multi-core cluster and parallelized calculations. The computation was performed using an HP ProLiant DL580 Gen10 computer with 80 cores.

Bayrak et al. (2006) applied a dimensional analysis to the current MT data. They concluded that the high-frequency part, corresponding to the younger sedimentary fills, shows a 1D behaviour, while the data at the low frequencies show a 3D behaviour. Consequently, we applied 3D inversion and the bathymetry of the Black Sea, Marmara and Aegean Seas were inserted into the model and a sea resistivity value of 0.33 ohm-m was assigned to represent their contribution to the data. The topography is modelled from SRTM90 data. An average elevation is used for the z coordinate of the top of the corresponding cell. Figure 4 shows the 3D modelling grid in the x-y plane comprising 85 and 80 cells, respectively. The model is also extended to 95 cells in the z -direction. The total number of parameters to be solved is 646.000. The ModEM software has been executed in a cluster so that one processor serves the calculation of the theoretical data for one frequency. The inverted data consists of 40 sample values in the range of frequencies between 320 and 5.493×10^{-4} Hz.

The Thrace Basin consists of a sediment layer with low resistivity, so the industrial noise was attenuated relatively short distances. Because of its higher resistivity values, the noise contribution over Strandja Massif was higher than that of the basin in intermediate and long periods. The two off-diagonal components of the impedance tensor have been used in the inversion. However, the diagonal components corresponding to the low frequencies were scattered while almost zero at the high-frequency band because of the thick 1D sedimentary cover. For these reasons, the diagonal components were not used in the inversion. The tipper values have been disregarded because of high noise contamination. The algorithm was initialized with homogeneous resistivity values of 100 ohm-m. Meanwhile, various error levels and smoothing factors for horizontal and vertical directions were used for each initial-guess model. Finally, the model produced the smallest misfit ($nmrs$) value of 1.8 after 55 NLCG iterations. The measured

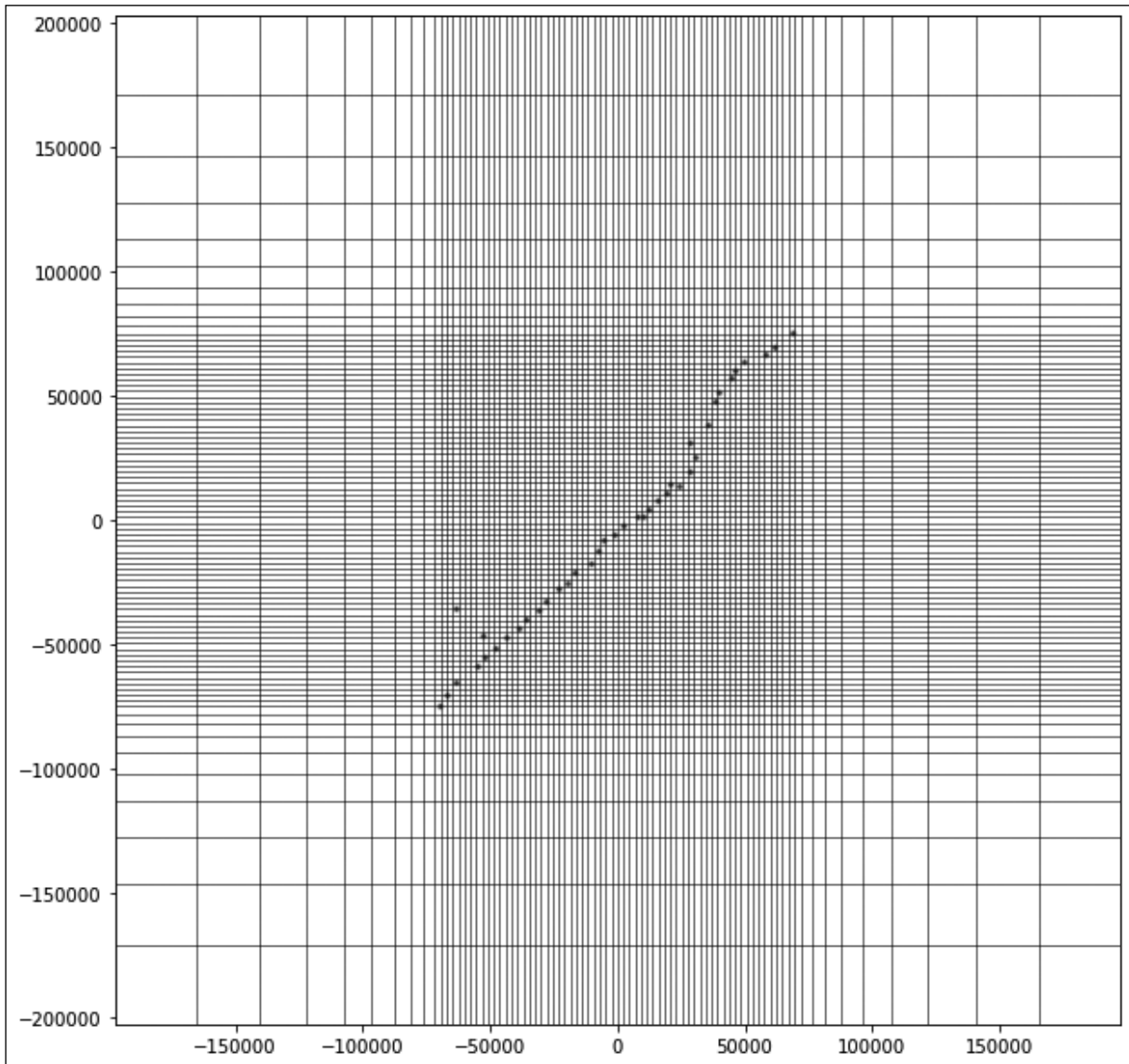


Figure 4- Plan view (x-y plane) of the modelling grid used in the 3-D inversion. The yellow dots show measurement stations. The survey area is subdivided into 646,000 cells. There are 85, 80 and 95 cells in the north, east and depth.

and calculated curves corresponding to station 33 are shown in Figure 5.

Figure 6 shows the FNI pseudo-sections of the measured and calculated data in a distance-frequency (x - f) plane for the YX component. It can be concluded that the final 3D model produced a satisfactory fit between the measured data and model response, and there are no artefacts in the model due to noise or surrounding seas. The pseudo-section derived from the measured data indicates the shape of the basement since the MT profile is almost perpendicular to the geological strike of the basin.

5. Interpretation

Figure 7a illustrates the final model obtained from the 3D inversion. A cell was colourized by a specific colour proportional to its intrinsic resistivity value. The resistivity limits were selected in the range $[0,3]$ $\log_{10}$ ohm-m corresponding to the resistivity values of 1 and 1000 ohm-m in a linear scale, respectively. The selected resistivity scale better reflects the shape of the basin due to the high resistivity contrast between relatively low resistivity young sediments and high resistivity basement. However, interpreting the depth of interfaces using a colour scale will

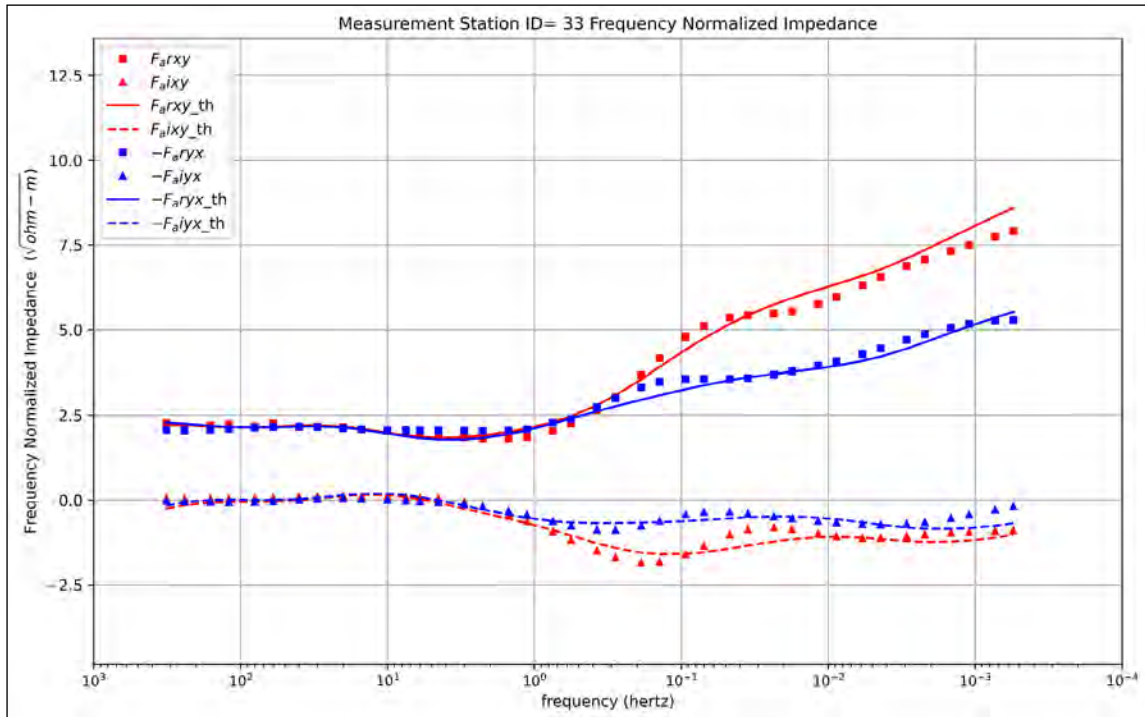


Figure 5- Data fit for the 3D inversion at station 33. The red and blue colours indicate the xy and yx components. Squares and triangles show the measured real and imaginary components, respectively. The continuous curves are the model responses.

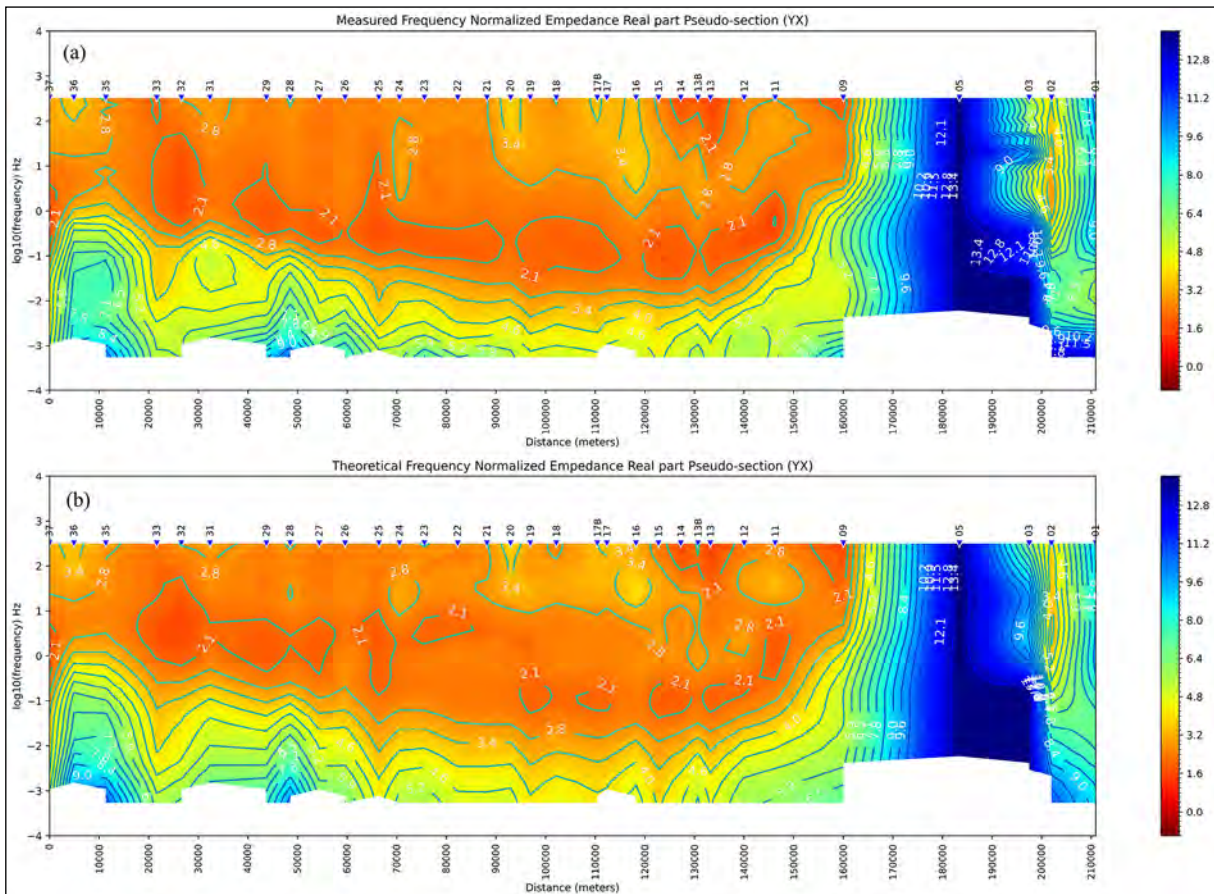


Figure 6- Comparison of the measured a) and calculated b) real parts of the FNI function for the YX component pseudo-sections.

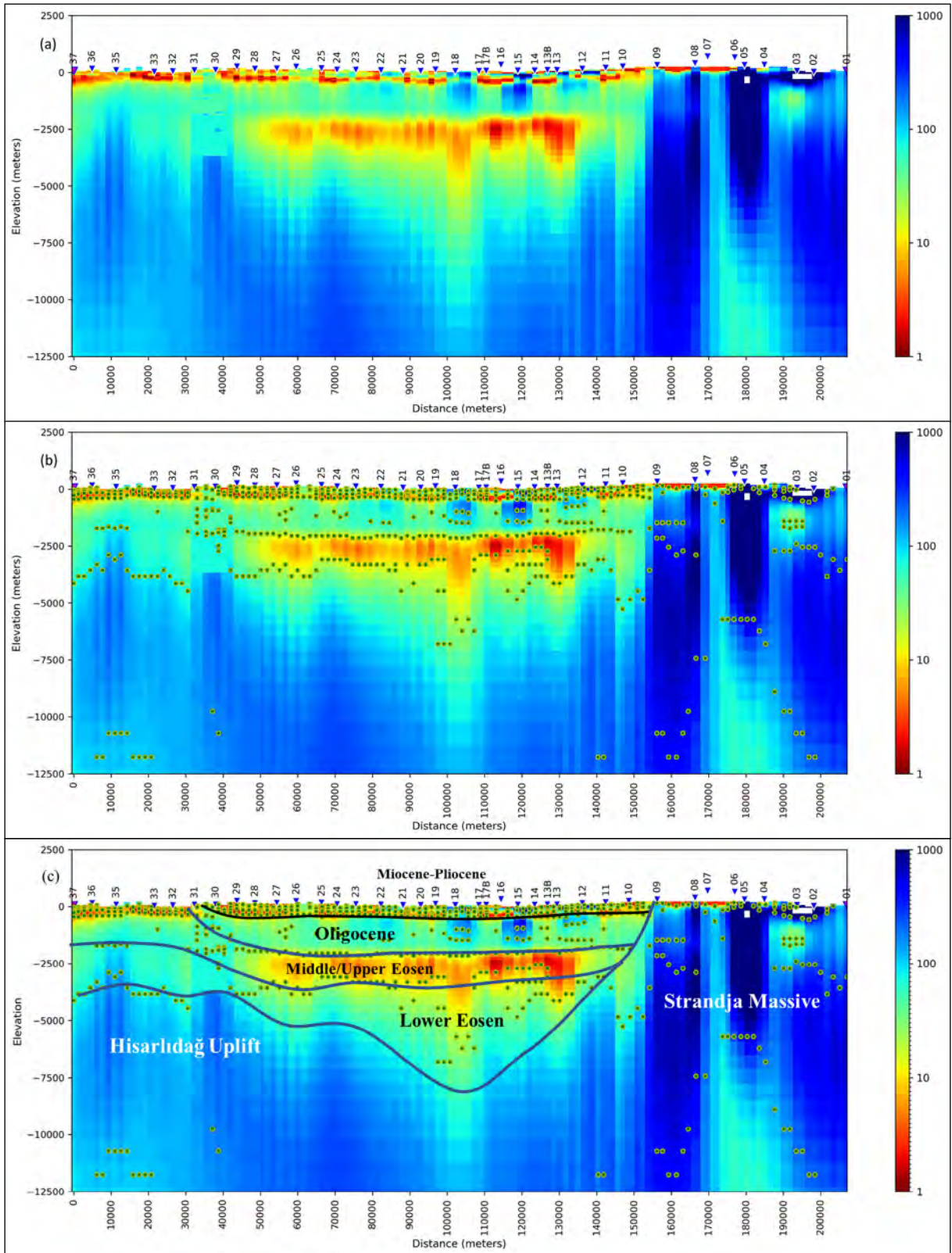


Figure 7- a) Resistivity section derived from the 3D inversion, b) the inflection and deflection points of the model in the vertical direction, c) possible interpreted interfaces.

remain subjective. Changing the resistivity scale of the colour map will produce images in which the probable location of interfaces will be perceived differently. This is the main difficulty in identifying the layer interfaces by visual inspection. Akca and Başokur (2010) developed the concept of the structure-based model in which the model parameters simulate undulating interfaces in 2D subsurface. However, the conventional derivative-based inversion methods could not be applied to the structure-based model parameterization. Searching for a model that produces minimum misfit can be performed by model verification algorithms that generate and verify many distinct and complicated models. For cases where many models should be verified, the genetic algorithm is one of the most suitable for verifying large model sets (Başokur et al., 2007). However, the computation of 3D models is expensive and time-consuming, so the next computer generation can tackle constructing structure-based 3D models.

ModEM software produces smooth models; consequently, the resistivity versus depth is a smooth curve. For this reason, we apply the scheme developed by Başokur (2022) to delineate possible interfaces. The variation of resistivity along a column in the vertical direction with the same central x and y coordinates is extracted as a function of z values from the smooth model. For example, Figure 8 shows the depth-resistivity curve derived from the 3D model cube for particular x-y coordinates corresponding to station 23 (black lines). The maxima, minima, (magenta dots in Figure 8) inflection and deflection points (red dots) can be derived using the Numpy library of Python (<https://numpy.org/>) from the first and second numerical derivatives, respectively. The inflection and deflection points of the depth-resistivity (z - ρ) curve are the most probable boundaries of the interfaces since they indicate sharp resistivity variations. The applied procedure is similar to edge detection algorithms. A subsurface model is constructed by connecting the points (green lines in Figure 8). The method can be repeated for all z - ρ curves along a profile, and possible layer interfaces can be plotted as dots over a resistivity section (Figure 7b). This numerical strategy helps the interpreter identify the layer interfaces from a cell-based smooth 3D model.

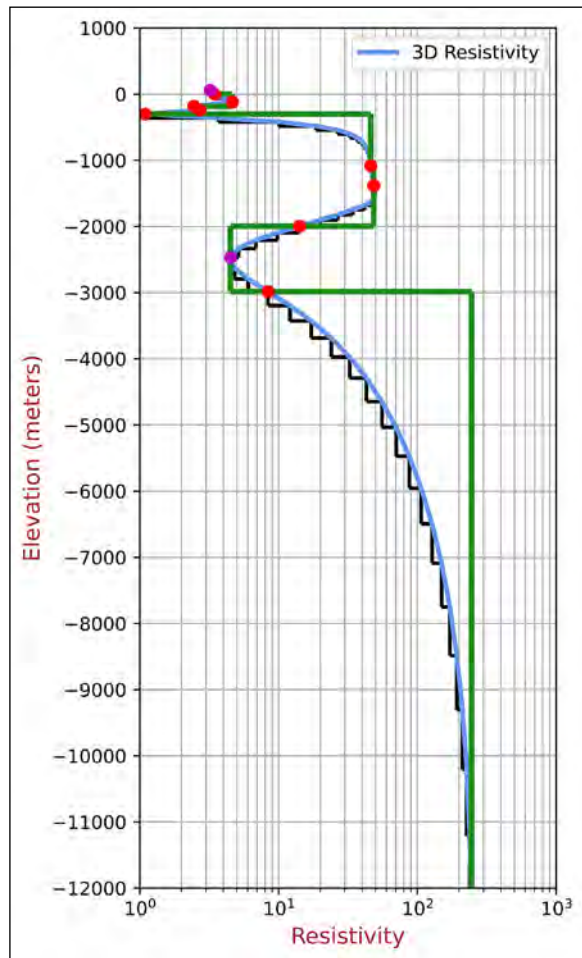


Figure 8- Resistivity variation with depth. Black lines are the resistivity values of each cell. Green lines indicate the probable depth of interfaces.

Figure 7c illustrates our interpreted model derived from the dotted inflection and deflection points. There is no significant sedimentary cover on the top of the Strandja massif. The slope variations in the basement floor were well delineated because of the high resistivity contrast between sedimentary units and crystalline basement. The overall interpretation could imply that major structural elements of the region could be outlined with the help of the MT method without much difficulty. Moreover, the main fault zones may be identified with the help of the information obtained from the surface geology. The depth to the basement along the MT profile reaches 8000-8500 meters. However, the total sediment thickness can reach 10-11 km at the east of the MT profile. A depth estimate using the power spectrum of the gravity data produces a similar result.

6. Discussion and Conclusion

There have been three different suggestions for the location of the Intra-Pontid suture zone representing the boundaries of former Sakarya and Rhodope continents: (1) It has been suggested that a suture of the Intra-Pontide Ocean existed between Strandja Massif and Sakarya continent as noted by Şengör and Yılmaz (1981). The ocean closed during the Cretaceous to Palaeocene period. However, there has been a controversy over the existence and location of this suture zone. It has been proposed that the suture was located at the south of the Thrace Basin, extending towards the west-east direction along the north coast of the Aegean Sea towards the Marmara Sea, as reported by Şengör and Yılmaz (1981). (2) The north of the Strandja massif has been suggested to be the location of the suture zone (Yılmaz et al., 1997). (3) Bayrak et al. (2004) reported that the suture was an N-S-oriented zone between İstanbul and Çatalca in the north. They have also proposed an alternative location close to the surface contact between Thrace Basin and the Strandja massive in the north based on the interpretation of their nine magnetotelluric stations. Their short profile extended approximately in the N-S direction from Strandja Massif towards the Sea of Marmara. It crossed our MT line around station 13 and contained no information about the south of station 13. A possible location may correspond to the low resistivity anomaly beneath stations 12 and 13 in Figure 7c. Additional MT profiles parallel to the existing line are required to resolve the exact location. Modelling over an MT profile data with distant stations would not allow a detailed geological interpretation. Still, we demonstrate that the MT method could map sedimentary cover and undulations in the basement. Also, the present study shows that the main differences in the depth of the conductive units of Thrace and other similar sedimentary basins can be inexpensively delineated over large areas using the MT method.

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Discussion: Determination of Atterberg Limits Using the Vane Shear Test Method [Bull. Min. Res. Exp. (2024) 174: 1–10]

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Discussion

Keywords:

Fine-Grained Soil, Liquid Limit, Plastic Limit, Vane Shear Strength, Regression Analysis.

ABSTRACT

This article presents a discussion of the original research reported in the paper by Kayabalı et al. (2024) (the Authors) that was recently published in the Bulletin of the Mineral Research and Exploration Vol. 174, pp. 1–10. Using liquid limit (LL), plastic limit (PL) and vane shear strength against water content [i.e., $s_{u(VST)}-w$] results obtained for 100 fine-grained soil samples, the Authors performed multiple regression analyses to produce four strength-based correlations for predicting the LL and PL values based on $s_{u(VST)}-w$ measurements. The Authors' dataset primarily consisted of residual soils formed through the weathering of igneous rocks, along with a few lacustrine soil samples, all sourced from the vicinity of Ankara, Türkiye. This discussion article examines the veracity of the Authors' claims regarding the predictive performance of their proposed correlations when applied to other fine-grained soils. Using a sizable independent database of dissimilar fine-grained soils compiled from the research literature, it is conclusively shown that, contrary to the Authors' assertions, their proposed correlations generally produce poor LL and PL predictions when employed beyond the calibration soil types. This outcome is not unexpected, since the Authors' data-driven correlations were deduced based on a specific dataset with limited diversity in terms of soil physico-chemical and mineralogical attributes. The article closes with a discussion of the plausible explanations for the poor applicability of the Authors' correlations when applied to dissimilar fine-grained soils.

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ABBREVIATIONS

AS	Australian Standard
ASTM	American Society for Testing and Materials
BS	British Standard
FC	Fall cone (LL testing method)
HR	Hand rolling (PL testing method)
IS	Indian Standard
LL	Liquid limit

PL	Plastic limit
RP	Rolling plate (PL testing method)
VST	Vane shear test

NOTATIONS

a	y -axis intercept of best-fitting exponential curve to $s_{u(VST)}$ against w data plot
b	Gradient of best-fitting exponential curve to $s_{u(VST)}$ against w data plot

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I_p	Plasticity index [= $LL_{(FC-BS)} - PL_{(RP)}$ or $LL_{(FC-BS)} - PL_{(HR)}$]
$LL_{(FC-BS)}$	British Standard fall-cone liquid limit
N	Number of investigated fine-grained soils
$PL_{(HR)}$	Standard hand-rolling plastic limit
$PL_{(RP)}$	ASTM rolling-plate plastic limit
$s_{u(VST)}$	Saturated remolded vane-shear strength
x, y	Exponent parameters
w	Water content
σ	Standard deviation

1. Introduction

This article presents a discussion of the original research reported in the paper by Kayabalı et al. (2024) (hereafter referred to as the Authors) that was recently published in the Bulletin of the Mineral Research and Exploration Vol. 174, pp. 1–10. Following standard testing procedures, the Authors determined the water content values corresponding to the liquid limit (LL) and plastic limit (PL) state transitions, as well as the variation in vane-shear strength $s_{u(VST)}$ with water content w , for 100 fine-grained soil samples (henceforth denoted as Dataset 1). This dataset primarily consisted of residual soils formed through the weathering of igneous rocks, along with a few lacustrine soil samples, all sourced from the vicinity of Ankara, Türkiye. Specifically, the Authors performed their LL [i.e., $LL_{(FC-BS)}$], PL [i.e., $PL_{(RP)}$] and vane shear test (VST) measurements following the British Standard (BS) fall cone (FC) method (BS 1377–2 1990), the ASTM rolling-plate (RP) device approach (ASTM D4318, 2005), and by using a miniature VST apparatus for determining the $s_{u(VST)}$ of the saturated soil specimens in accordance with ASTM D4648 (2000), respectively. Applying multiple regression analyses to their compiled database of $LL_{(FC-BS)}:PL_{(RP)}:s_{u(VST)}-w$ measurements, the Authors deduced four strength-based empirical correlations (presented as *Equations 4–7* in their paper, and reproduced herein as *Equations 1–4*) for predicting the $LL_{(FC-BS)}$ and $PL_{(RP)}$ parameters, based on either a single VST measurement (i.e., *Equations 1 and 2*), or by employing the fitting parameters (a and b) that define the $s_{u(VST)}-w$ regression relationship as $s_{u(VST)} = ae^{-bw}$ (i.e., *Equations 3 and 4*).

$$LL_{(FC-BS)} = 0.902 \times w^{0.997} \times s_{u(VST)}^{0.138} \text{ (Authors' Equation 4)} \quad (1)$$

$$PL_{(RP)} = 0.609 \times w^{0.959} \times s_{u(VST)}^{0.139} \text{ (Authors' Equation 5)} \quad (2)$$

$$LL_{(FC-BS)} = 3.62 \times a^{0.106} \times b^{-0.92} \text{ (Authors' Equation 6)} \quad (3)$$

$$PL_{(RP)} = 1.72 \times a^{0.129} \times b^{-0.91} \text{ (Authors' Equation 7)} \quad (4)$$

For the 100 soils of Dataset 1, the Authors demonstrated that their *Equations 4–7* produced good predictions of the measured $LL_{(FC-BS)}$ and $PL_{(RP)}$ values. This discussion article examines the veracity of the Authors' claims regarding the predictive performance of their proposed equations/correlations when applied to other fine-grained soils, i.e., different from the soils comprising Dataset 1. For this purpose, the Discussers employ a separate $LL_{(FC-BS)}:PL_{(RP)}:s_{u(VST)}$ dataset (hereafter referred to as Dataset 2) comprising 180 dissimilar fine-grained soils to re-examine the predictive performance/applicability of the Authors' *Equations 6 and 7*. Employing routine statistical techniques, it is demonstrated that, contrary to the Authors' assertions, their proposed correlations generally produce poor $LL_{(FC-BS)}$ and $PL_{(RP)}$ predictions when applied for fine-grained soils dissimilar to those comprising their calibration dataset (i.e., Dataset 1). This finding is not unexpected, since the Authors' data-driven correlations were deduced based on a specific dataset with limited diversity in terms of soil physico-chemical and mineralogical attributes. In other words, when considering a wide range of fine-grained soil types, the fitting parameters a and b are not unique. The article closes with a discussion of the plausible explanations for the poor/limited applicability of the Authors' *Equations 4–7* (to dissimilar fine-grained soils).

2. Critical Statistical Appraisal of the Authors' Proposed Correlations for $LL_{(FC-BS)}$ and $PL_{(RP)}$ Estimations

Table 1 summarizes Dataset 2, which comprises of $LL_{(FC-BS)}$ and $PL_{(RP)}$, and the a and b coefficients (obtained from exponential curve fitting of experimental $s_{u(VST)}-w$ results) for 180 fine-grained soils reported in two earlier investigations performed by the research group of the lead author for the paper under discussion [i.e., Kayabalı et al. (2015a, 2015b)]. Compared to the ranges of $LL_{(FC-BS)} = 29.3\text{--}117.0\%$

Table 1- Summary of Dataset 2 employed by the Discussers in assessing the predictive performance/applicability of the Authors' Equations 6 and 7.

Reference	Source in Reference	N	$LL_{(FC-BS)}$ (%)	$PL_{(RP)}$ (%)	I_p (%)	$\log a$	$b (\times 10^{-2})$	Remarks
Kayabali et al. (2015a)	Table 3 (pp. 128–129)	60	29.0–117.0	15.4–29.1	12.1–90.0	2.50–7.95	5.76–62.2	Prepared from natural soil mixed with fine sand and commercial bentonite at various dry mass ratios.
Kayabali et al. (2015b)	Table 1 (pp. 719)	120	46.0–91.0	22.0–44.0	20.0–57.0	2.41–3.12	3.50–8.50	Remolded soil samples from different regions of Türkiye.
Overall	—	180	29.3–117.0	15.4–44.0	12.1–90.0	2.41–7.95	3.50–62.2	—

Note: N = number of investigated fine-grained soils; $LL_{(FC-BS)}$ and $PL_{(RP)}$ determined in accordance with BS 1377–2 (1990) and ASTM D4318 (2000), respectively; I_p = plasticity index [= $LL_{(FC-BS)} - PL_{(RP)}$]; and $s_{u(VST)}-w$ data (used for a and b determination) obtained in accordance with ASTM D4648 (2000).

and $I_p = 12.1\text{--}90.0\%$ [where I_p denotes the plasticity index, defined as the difference of $LL_{(FC-BS)}$ and $PL_{(RP)}$] for these 180 soils, the Authors' 100 soils of Dataset 1 had comparable ranges of 23.3–106.0% and 7.5–49.8%, respectively.

Figure 1 illustrates the 180 soils (of Dataset 2) plotted in the standard plasticity chart, with 132 and 48 of them classifying as *clay* and *silt*, respectively (i.e., only 27% are of *silt* type). Whereas in the case of the Authors' 100 soils (of Dataset 1), 30 and 70 of them classified as *clay* and *silt*, respectively (i.e., 70% were *silt* type). In other words, the fine-grained soils comprising Datasets 1 and 2 are quite different.

Employing the Authors' Equations 6 and 7, the Discussers calculated the values of $LL_{(FC-BS)}$ and $PL_{(RP)}$ predicted for each of the 180 soils (of Dataset 2) by inputting their reported experimental values of

a and b . Note that Kayabali et al. (2015a) reported values of a and b based on the equation $\log_{10} s_{u(VST)} = \log_{10} a - bw$ while the required form for the current analysis is $s_{u(VST)} = ae^{-bw}$ (or $\log_{10} s_{u(VST)} = \log_{10} a - bw \log_{10} e$), necessitating a conversion of the reported b parameter [in Kayabali et al. (2015a)] to $b/\log_{10} e$. Figures 2a and 2b illustrate the predicted against measured values of the $LL_{(FC-BS)}$ and $PL_{(RP)}$ parameters, respectively. The Authors reported that for their 100 soils (of Dataset 1), the overall absolute percent errors for the $LL_{(FC-BS)}$ and $PL_{(RP)}$ predictions were 6.3% and 3.9%, respectively, for the Dataset 2 soils. However, when the Authors' Equations 6 and 7 are applied to the 180 soils of Dataset 2, it is clear from Figures 2a and 2b that they are poor $LL_{(FC-BS)}$ and $PL_{(RP)}$ predictors, generally overestimating, often seriously, the measured $LL_{(FC-BS)}$ and $PL_{(RP)}$ values, with the aforementioned absolute percent errors significantly increasing to 44.8% and 60.6%, respectively. To put this in context, ASTM D4318 (2005) deems acceptable PL measurement variations of up to $\pm 3.5\%$ and $\pm 7.0\%$ (both in terms of water content) for fine-grained soils of low ($LL < 50\%$) and high ($LL \geq 50\%$) plasticity, respectively. However, for the 180 soils of Dataset 2, 74% of the $PL_{(RP)}$ values predicted by the Authors' Equation 7 differed by more than 7% water content from their measured counterparts, which is deemed unacceptable according to the ASTM D4318 (2005) standard.

Additionally, Figure 3 presents the predicted $I_p:LL_{(FC-BS)}$ data pairs [with I_p defined as the difference of the predicted $LL_{(FC-BS)}$ and $PL_{(RP)}$ values] for the 180 soils of Dataset 2 plotted in the standard plasticity chart.

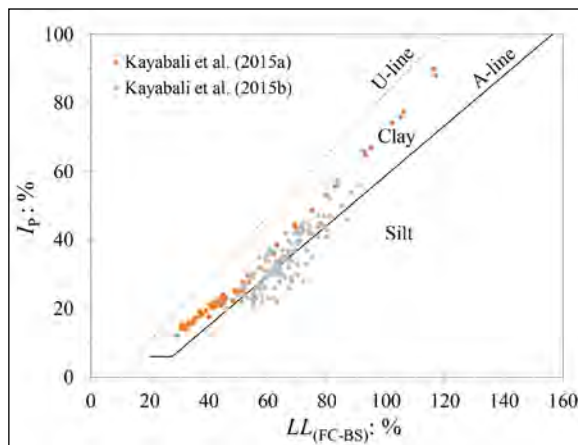


Figure 1- Dataset 2 of 180 fine-grained soils plotted in the standard plasticity chart. Note: $LL_{(FC-BS)}$ and I_p [= $LL_{(FC-BS)} - PL_{(RP)}$] are measured values.

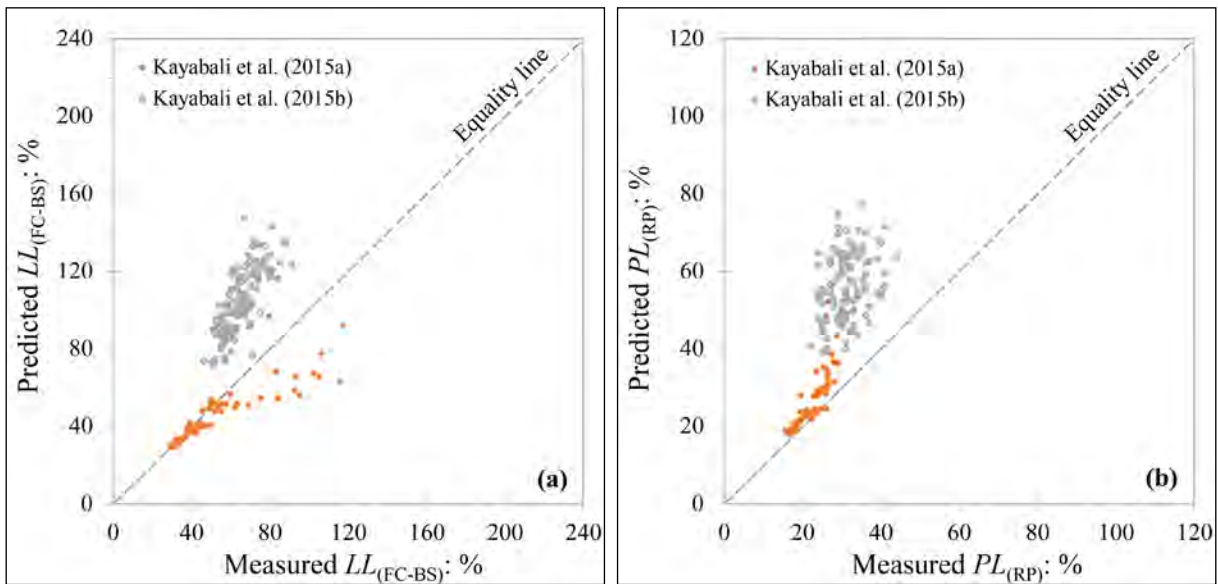


Figure 2- Predicted (by the Authors' Equations 6 and 7) against measured values of (a) $LL_{(FC-BS)}$ and (b) $PL_{(RP)}$ for the 180 fine-grained soils of Dataset 2.

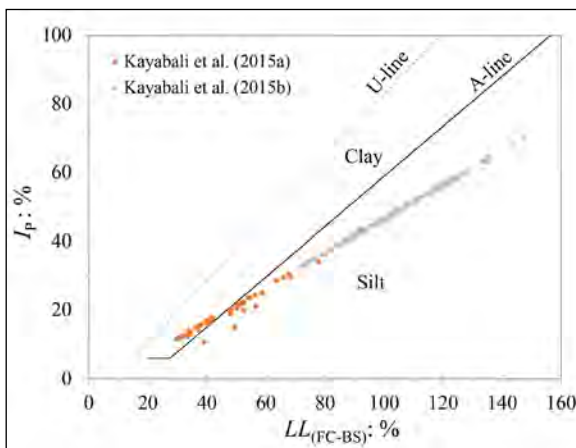


Figure 3- Predicted values of I_p against $LL_{(FC-BS)}$ (deduced using the Authors' Equations 6 and 7) for the 180 fine-grained soils of Dataset 2.

A comparison of Figures 1 and 3 clearly indicates that the predicted soil classes (based on the computed results from the Authors' Equations 6 and 7) are generally different to the measured soil classes, as determined using the standard plasticity-chart classification framework. For instance, whereas Figure 1 indicated that only 27% of the 180 soils actually classify as *silt* type (i.e., 73% being *clay*), their $I_p:LL_{(FC-BS)}$ data pairs predicted by the Authors' Equations 6 and 7 incorrectly suggest that 83% of the 180 soils should be classified as *silt* type (i.e.,

plotting below the A-Line demarcation boundary). Furthermore, as observed in Figure 3, the predicted $I_p:LL_{(FC-BS)}$ data exhibit a general linear relationship, on which the Discussers note the following. The measured data (in Figure 1) themselves exhibit a general linear $I_p:LL_{(FC-BS)}$ tendency, which naturally carries over in applying the Authors' Equation 6 and 7 to obtain the predicted $I_p:LL_{(FC-BS)}$ data. More importantly, however, the power exponents for a and b in the Authors' Equations 6 and 7 for $LL_{(FC-BS)}$ and $PL_{(RP)}$, respectively, are very close (i.e., 0.106 versus 0.129 for a , and -0.91 versus -0.92 for b). Consequently, the predicted $LL_{(FC-BS)}$ -to- I_p ratio can be estimated as follows. Given that $LL_{(FC-BS)} \approx 3.62 a^x b^y$, $PL_{(RP)} \approx 1.72 a^x b^y$ and $I_p = LL_{(FC-BS)} - PL_{(RP)} \approx 3.62 a^x b^y - 1.72 a^x b^y$, it follows that $LL_{(FC-BS)}/I_p \approx 3.62/(3.62 - 1.72) = 1.91$. This implies that the predicted $I_p:LL_{(FC-BS)}$ data approximately follow a linear trend with a slope close to $1/1.91$ (in practice, however, an intercept parameter would also exist, which would slightly adjust/modify this slope to a different value). In other words, because of the structure of the Authors' Equations 6 and 7, the predicted $LL_{(FC-BS)}$ -to- I_p ratio is expected to remain fairly constant (see Figure 3).

Considering all the above, the predictive performance of the Authors' Equations 6 and 7 when applied to other fine-grained soils (i.e., different from

the 100 soils of Dataset 1) is generally considered unreliable and therefore unacceptable for routine geotechnical engineering practice. Moreover, for all the same reasons discussed in this section, the predictive performance of the Authors' *Equations 4* and *5*, which are simplified forms of their *Equations 6* and *7*, is anticipated to be equally (if not more) unreliable.

3. Discussion on Poor Predictive Performance/ Applicability of the Authors' Proposed Correlations for $LL_{(FC-BS)}$ and $PL_{(RP)}$ Estimations

This section discusses various reasons for the poor predictive performance of the Authors' *Equations 4–7* when applied to the dissimilar fine-grained soils of Dataset 2 (compared to the soils in the Authors' Dataset 1). The first and most obvious reason is that these equations were deduced/calibrated using experimental results for residual soil samples formed through the weathering of igneous rocks, along with a few lacustrine soil samples, all sourced from the vicinity of Ankara, Türkiye. Whereas Dataset 2 concerned natural soil mixed with fine sand and commercial bentonite at various dry mass ratios, as well as remolded soils sampled from different regions of Türkiye. Accordingly, with the Authors' *Equations 4–7* deduced (optimally calibrated for) the particular soil types comprising Dataset 1, it is not unexpected that these equations produce sub-optimal predictions for the dissimilar soils (in terms of their particle-size and mineralogical compositions and hence plasticity characteristics) comprising Dataset 2. Notably, the Authors investigated mostly *silt* type soils (70%), whereas the fine-grained soils comprising Dataset 2 were mostly clay type (73%).

Additionally, as a fundamental overriding point, Atterberg's PL is defined as the water content corresponding to the plastic/ductile–brittle state transition, determined experimentally using the standard hand-rolling [i.e., $PL_{(HR)}$] or rolling-plate [i.e., $PL_{(RP)}$] methods, and deduced by a significant change in soil deformation behavior occurring for water contents either side of this state transition. It should be noted that comprehensive statistical investigations

by Soltani and O'Kelly (2021) for a database of 60 diverse fine-grained soils demonstrated that these two standard PL testing methods produce essentially similar results, with the likelihoods of $PL_{(RP)}$ underestimating and overestimating $PL_{(HR)}$ found to be 50% and 40%, respectively. Given that standard PL testing evaluates the soil's ductility properties, strength-based approaches (such as the Authors' *Equations 4–7*) are fundamentally inappropriate for making such assessments and in determining Atterberg's PL (Haigh et al., 2013; Sivakumar et al., 2016; O'Kelly et al., 2018; O'Kelly, 2021, 2024). For instance, when applied to Dataset 2, the Authors' *Equations 4* and *5* predict the values of $LL_{(FC-BS)}$ and $PL_{(RP)}$ for the 180 soils based on mean $s_{u(VST)}$ values of ~ 2.31 kPa (standard deviation of $\sigma = 0.01$ kPa) and 94.72 kPa ($\sigma = 5.96$ kPa), respectively (where the standard deviation arises from insufficient precision of the equations' fitting coefficient values). However, when considering diverse fine-grained soils, the strength mobilized at Atterberg's PL state transition has been shown to vary over a much wider range (Nagaraj et al., 2012; Haigh et al., 2013; O'Kelly, 2013). Regarding the $LL_{(FC-BS)}$ parameter, strength-based approaches are appropriate for its determination, with the BS 1377–2 (1990) FC test linking the $LL_{(FC-BS)}$ state transition to an $s_{u(VST)}$ value of ~ 1.7 kPa (O'Kelly et al., 2018; O'Kelly, 2024).

Finally, the Discussers would point out that *Equation 2* in the Author's paper (used for calculating the vane blade constant K) is not consistent with a cruciform vane used for $s_{u(VST)}$ determination. The correct form of the equation, as reported in the ASTM D4648 (2000) and BS 1377–7 (1990) standards, is given as follows:

$$K = \pi D^2 \left(\frac{H}{2} + \frac{D}{6} \right) \quad (5)$$

where H and D are the vane height and diameter (width), respectively.

For the 12.7×12.7 mm blade dimensions employed in the Authors' study (assuming a cruciform vane), the Authors' *Equation 2* would yield a K value of $3.38 \times 10^{-4} \text{ m}^3$, whereas *Equation 5* would give a value of $4.29 \times 10^{-6} \text{ m}^3$. It is not possible for the Discussers

to know whether there are typographical errors in the Authors' *Equation 2*, or whether this incorrect equation (as written) was used by them in performing their $s_{u(VST)}$ calculations.

4. Summary and Conclusions

The Authors developed their *Equations 4–7* from regression analyses of experimental results obtained for residual soil samples formed through the weathering of igneous rocks, along with a few lacustrine soil samples, all sourced from the vicinity of Ankara, Türkiye. Being calibrated for these particular soils, *Equations 4–7* gave good predictability of the investigated soils' measured $LL_{(FC-BS)}$ and $PL_{(RP)}$ parameters. However, using a sizable independent database compiled from the research literature, the Discussers have carefully demonstrated that the Authors' *Equations 4–7* generally have poor $LL_{(FC-BS)}$ and $PL_{(RP)}$ predictive performances when applied to dissimilar fine-grained soils, generally overestimating, often seriously, their measured values. Accordingly, the use of the Authors' *Equations 4–7* should be strictly limited to the calibration soils (i.e., they should not be employed for dissimilar soil types, as they generally produce unreliable predictions).

Finally, because repeatability and reproducibility remain significant challenges in PL determination using standard thread-rolling techniques [i.e., $PL_{(HR)}$ or $PL_{(RP)}$], the idea of exploring alternative, ductility-based PL measurement methods is certainly warranted. However, this need not be the case for LL determination, as standard FC testing methods offer reasonably high repeatability and reproducibility, with the added benefit of being able to establish the LL state transition by standard (e.g., IS 2720–5 1985; BS 1377–2 1990; AS 1289.3.9.2 2006) or non-standard (e.g., Kayabalı et al., 2023; O'Kelly and Soltani, 2024) single-point measurement methods. In other words, there appears to be little justification in attempting to establish the LL parameter by means of $s_{u(VST)}$ measurements.

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Reply to: “Discussion: Determination of Atterberg Limits Using the Vane Shear Test Method [Bull. Min. Res. Exp. (2024) 174: 1–10]”

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Reply

The main focus of the criticisms raised by O’Kelly and Soltani (2025) (hereafter referred to as discussers) to the authors’ article in Bulletin of Mineral Research and Exploration (174, 1-10) is to examine the veracity of the strength-based equations presented by Kayabali et al. (2024). The discussers applied the predictive equations proposed by the authors to dissimilar soils in the two articles (Kayabali et al., 2015a; Kayabali et al., 2015b) published earlier by the lead author with his research group. The discussers showed that the predictive equations 6 and 7 given by Kayabali et al. (2024) yield extremely high values of liquid limit (LL) and plastic limit (PL) when applied to dissimilar soils. Further, discussers argued that the approach proposed by Kayabali et al. (2024) led to erroneous results in determining soil classes.

First of all, authors need to emphasize that 500 sets of tests were conducted for the 100 soils examined in the article under discussion. It should be appreciated that such a voluminous data set cannot be presented in full detail in an article that is expected to be of a reasonable length. Therefore, the experimental results were presented as only a and b coefficients of vane

shear tests (VST) for space-saving considerations. The same approach was adopted by Kayabali et al. (2015a) and Kayabali et al. (2015b), where only a and b coefficients of VST were preferred for presenting test data in place of lengthy details. Among the predictive equations in article under discussion, authors’ priority of use is surely the first two ones (i.e. equations 4 and 5), since they estimate the two important Atterberg limits (namely LL and PL) with only one set of data comprising only water content and undrained shear strength from VST. Obtaining the a and b coefficients for equations 6 and 7 requires at least a few- to several sets of data for any fine-grained soil. Since the authors could not present the experimental data in full detail in the article under discussion, the discussers naturally had to use prediction equations 6 and 7 presented in the paper.

The detailed data sets produced by the lead author’s research group in the past and published in Kayabali et al. (2015a) and Kayabali et al. (2015b) were retrieved from the archive and the predictive performance of especially the equations 4 and 5 presented in Kayabali et al. (2024) were re-evaluated using those

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dissimilar soils. The data in Kayabali et al. (2015a) originally belonged to 60 soils. Approximately 300 sets of test data were produced by performing 3-7 sets of experiments for each soil comprising LL, PL, water content, and undrained shear strengths. The authors applied Equation 4 in Kayabali et al. (2024) to the data sets in Kayabali et al. (2015a). In this way, the LLs obtained for each soil by employing the predictive equation, varying in number between 3 and 7, were compared with the LLs of that soil determined through the falling cone method (Figure 1). This figure comprises only 60 pairs of LLs instead of some 300+ pairs of LLs for the sake of simplicity. That is to say, the mean value of the LLs for the 3-7 pairs of water content and undrained shear strength predicted by the strength-based equation of 4 was compared with the LL value determined by the fall-cone test. According to this figure, the prediction performance of our strength-based equation yields quite reasonable comparisons up to about LL = 60%. Liquid limits calculated by predictive equations are generally 15% higher than the experimentally found LLs. Similar steps were employed using equation 5 to PL as shown in Figure 2. A quick glimpse shows that while equation 5 in Kayabali et al. (2024) gave reasonable values when the plastic limits were low, it overpredicted PL for higher plastic limits. Plastic limits calculated by the prediction equation are generally 18% higher than those determined experimentally.

After retrieving some 600+ sets of experimental data employed by Kayabali et al. (2016b) from the archive for 120 dissimilar soils, the LLs again were predicted by Equation 4 by Kayabali et al. (2024) as shown in Figure 3. It is observed from Figure 3 that the proposed predictive equation 4 performs better for this data set. According to Figure 3, the prediction equation in our 2024 article overpredicted the liquid limit by 9% in 103 of 120 soils and underpredicted it by 4% in 17 of them. When looking at the performance of the prediction equations in predicting the plastic limit for the last data set (Figure 4), it is seen that the plastic limit is generally overpredicted by about 33%.

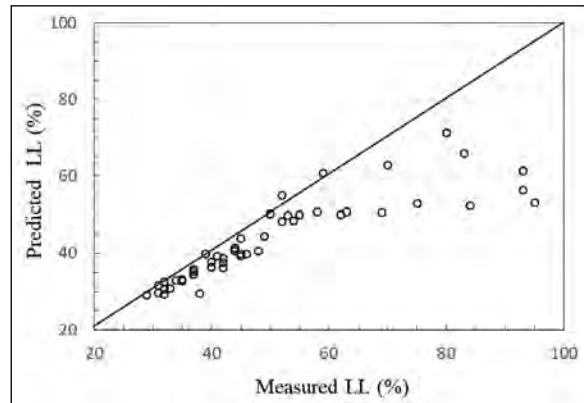


Figure 1- Comparison between the mean values of predicted LLs and experimental LLs for each soil by employing Kayabali et al. (2015a) data.

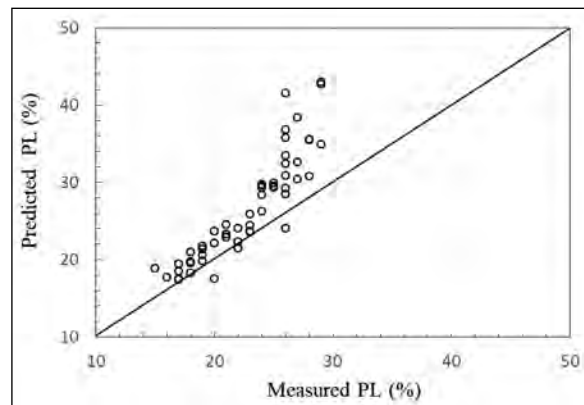


Figure 2- Comparison between the mean values of predicted PLs and experimental PLs for each soil by employing Kayabali et al. (2015a) data.

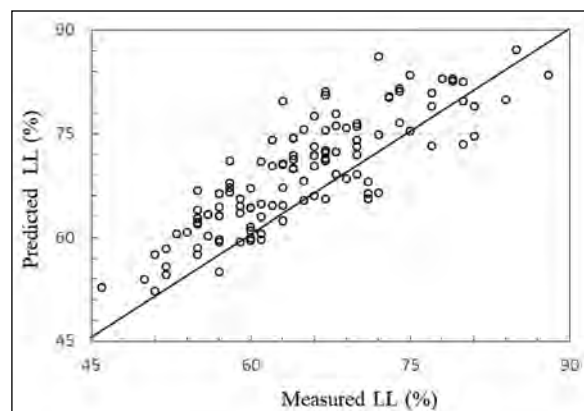


Figure 3- Comparison between the mean values of predicted LLs and experimental LLs for each soil by employing Kayabali et al. (2015b) data.

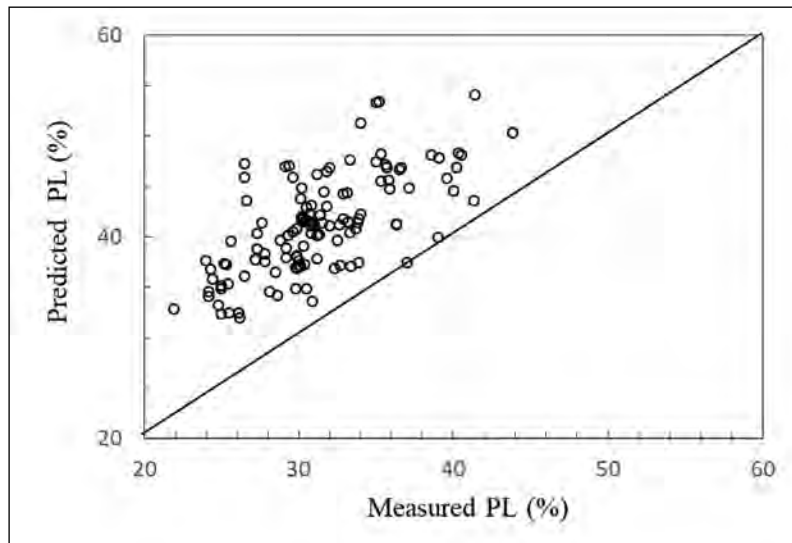


Figure 4- Comparison between the mean values of predicted PLs and experimental PLs for each soil by employing Kayabali et al. (2015b) data.

The authors acknowledge that there is a typo in Equation 2 as pointed out by the discussers. It should have been given as follows:

$$K = \frac{\pi D^2 H}{2 \times 10^6} \left[1 + \frac{D}{3H} \right] \quad (1)$$

This equation presented in the paper under discussion is given only as background information. It was not used directly in the computations. The software of the VST apparatus directly gave the undrained shear strength values.

The evaluations of the proposed predictive equations rendered within the context of this reply show that the predictive equations 4 and 5 yielded reasonably acceptable results for dissimilar soils of our research group's previous studies. It can be seen that the discrepancy in the scatter of predicted LLs and PLs in this reply gets significantly narrower compared to the scatter of the results obtained by the discussers. An important point the authors need to mention here is that the undrained shear strengths obtained by our research group in both 2015 investigations were carried out using the VST mechanism shown in Figure 5. In this device, calibrated springs are used to determine the undrained shear strength. The VST mechanism used in current article under discussion is servo-controlled and provides much more precise results. Therefore the authors are of the opinion that



Figure 5- The old-fashioned vane shear test apparatus used in the earlier investigations of the lead author and his research group.

the scatter obtained in the previous set of data that tested with our predictive equations mainly may be due to the mechanism involved in apparatus set being used. Other minor effects may be the use of unusual soils such as bentonite mixtures in experimental

studies and the fact that the operators performing the experiments are different for each of the study.

It was emphasized in a great number of earlier studies that the hand-rolling or the plate-rolling method used in determining the plastic limit is somehow irrational for being not quantitative. Because it is akin to the liquid limit, several research was performed in the past on determining the PL together with the LL by using the fall-cone method. The authors believe that research will continue for a longer time to establish the plastic limit on a more rational basis. Despite everything, the authors are of the opinion that the method used in the paper under discussion has a significant premise in determining both the liquid limit and plastic limit of fine-grained soils. So, the authors recommend prospective researchers employ a VST apparatus giving more precise undrained shear strength values and avoid the use of a spring-type VST machine for further investigations of Atterberg limits.

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1. Purposes

- To contribute to the establishment of scientific communication issues in earth sciences both in Türkiye and internationally.
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- To make the earth science scientific research and applications made by the MTA on publicly known,
- To use the bulletin as an effective tool in the international publication exchange by keeping it at a high level in terms of quality, scope and format,

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- Such articles cover original scientific research and its results that contribute to the fundamental issues of earth sciences, research and evaluation of underground resources, and examine the environmental problems in terms of earth sciences,
- It covers research that apply new approaches and methods in solving problems related to earth sciences.

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- Articles that criticize all or part of an article of the bulletin in the latest issue are published in the following first issue, if submitted within six months at the latest from the date of publication digitally.
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- The text of the manuscripts to be sent for the first review with the request to be published in the

Bulletin of the Mineral Research and Exploration should be written in A4 (29.7 x 21 cm) size, word format, Times New Roman 10 pt., normal with 2.0 line spacing.

- At the bottom, top, left and right of the page 2.5 cm indent must be left. Formulas that require the use of special letters and symbols should be presented in computer media.
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- In this section, the necessary information for preparatory and facilitative to understand the article such as the purpose of the study, its location, methods of study and previous reviews on the subject should be given.
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- The data and findings objectively conveyed in the “Main Text” section of the article should be discussed by the author in this section. Discussions should be separate from the “Results” section.

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- New data and findings obtained from the review that constitutes the subject of the article should be stated concisely and concretely in this section.
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- In this section, important contributions in the realization of the study, which is the subject of the article, are indicated. An attitude that will distract this section from its main purpose should not be taken in the Acknowledgements.

Contribution should be stated as short and concise as possible to the persons and/or organizations that provided assistance (reading, writing, language assistance, etc.) during the research, and should not take an attitude that would distract this section from its main purpose.

5.10. References

- In this section, only the documents mentioned in the article should be included in complete.
- Abbreviations should be avoided in naming the publications and journals.
- The mentioned documents should be written in Times New Roman and 9 pt.
- The first line of the references should be written as justified to the left margin of the page, and the other lines should be written by giving a hanging indent value of 1.25.

- The references should be listed in alphabetical order, taking into account the surnames of the authors.
- If one author has more than one work in the same year, lowercase alphabet letters should be used right after the year of publication and the letters should be italic (e.g. Saklar, 2011*a*, *b*).
- If more than one document of the same author is cited, first his / her single-name publications in chronological order, then double-names according to the chronological order, and then multi-names according to chronological order should be given.

For example:

- Corradini, C. 2007. The conodont genus *Pseudooneotodus* Drygant from the Silurian and Lower Devonian of Sardinia and the Carnic Alps (Italy). *Bollettino-Societa Paleontologica Italiana* 46 (2/3), 139-148.
- Corradini, C., Serpagli E. 1999. A Silurian conodont biozonation from late Llandovery to end Pridoli in Sardinia (Italy), In Serpagli (Ed.), *Studies on conodonts: Proceedings of the 7th European Conodont Symposium*. *Bollettino della Società Paleontologica Italiana* 37 (2-3) (1998), 255-273.
- Corradini, C., Corriga, M. G. 2010. Silurian and lowermost Devonian conodonts from the Passo Volaja area (Carnic Alps, Italy). *Bollettino della Società Paleontologica Italiana* 49 (3), 237-253.
- Corradini, C., Corriga, M. G. A. 2012. Pridoli – Lochkovian conodont zonation in Sardinia and the Carnic Alps: implications for a global zonation scheme. *Bulletin of Geosciences* 87 (4), 635-650.
- Corradini, C., Leone, F., Loi, A., Serpagli, E. 2001. Conodont Stratigraphy of A Highly Tectonised Silurian-Devonian Section in The San Basilio Area (Se Sardinia, Italy). *Bollettino Della Societa Paleontologica Italiana* 40 (3), 315-323, 1 Pl.
- Corradini, C., Pondrelli, M., Serventi, P., Simonetto, L. 2003. The Silurian cephalopod limestone in the Monte Cocco area (Carnic Alps, Italy): conodont biostratigraphy. *Revista Española de Micropaleontologia* 35 (3), 285-294.

Corradini, C., Corrigan, M. G., Männik, P., Schönlaub, H. P. 2015. Revised conodont stratigraphy of the Cellon section (Silurian, Carnic Alps). *Lethaia* 48 (1), 56-71.

- If documents of different authors with the same surname are mentioned, they should be written in alphabetical order, considering their first names.
- If documents of different author(s) with the same surname are mentioned in the same year, they should be cited as given below. Same publication rule should be applied for the single author.

For example:

“Usta, M., Yetiş, C., Nazik, A. 2018. Anamur (Mersin) dolayının stratigrafisi ve Kambriyen yaşlı kuvarsitler ile dolomitlerin endüstriyel hammadde potansiyeli. Çukurova Üniversitesi Fen ve Mühendislik Bilimleri Dergisi 35, 6, 11-22, Adana”

should be cited as (Usta, M. et. al., 2018)

“Usta, D., Ateş, Ş., Çoban, M., Deveci, Ö, Sağlam, F.M. 2014. Adıyaman-Sincik-Hilvan arasındaki bölgenin stratigrafisi ve kaya türü özellikleri. 67. Türkiye Jeoloji Kurultayı Bildiri Özleri, Ankara, 98-99”

should be cited as (Usta, D. et. al., 2018)

“Usta, M. 2018. Anamur (Mersin) dolayının stratigrafisi ve Kambriyen yaşlı kuvarsitler ile dolomitlerin endüstriyel hammadde potansiyeli. Çukurova Üniversitesi Fen ve Mühendislik Bilimleri Dergisi 35, 6, 11-22, Adana”

should be cited as (Usta, M., 2018)

“Usta, D. 2014. Adıyaman-Sincik-Hilvan arasındaki bölgenin stratigrafisi ve kaya türü özellikleri. 67. Türkiye Jeoloji Kurultayı Bildiri Özleri, Ankara, 98-99”

should be cited as (Usta, D., 2018)

- If the document is in a periodical publication (if it is an article), information about the document is given in the following order: Authors ‘surname, first letters of the authors’ first names. Year of publication. The name of the article. The name of the publication in which the article was published, volume number and / or issue number with the first letters in capital, the numbers of the first and last page of the document.

Punctuation marks like comma and etc. after journal names should not be used.

- In the examples below, the information about the mentioned documents is organized according to different document types, taking into account the punctuation marks.

For example:

Gürsoy, M. 2017. Munzur Dağları Alt Miyosen çökelleri mollusk topluluğu ve paleoekolojisi (Doğu Anadolu, Türkiye). *Maden Tetkik ve Arama Dergisi* 155, 75-99.

Pamir, H. N. 1953. Türkiye’de kurulacak bir Hidrojeoloji Enstitüsü hakkında rapor. *Türkiye Jeoloji Bülteni* 4 (1), 63-68.

Robertson, A. H. F. 2002. Overview of the genesis and emplacement of Mesozoic ophiolites in the Eastern Mediterranean Tethyan region. *Lithos* 65, 1-67.

- If the document is a book: authors’ surnames, authors’ first names. Year of publication. Title of the book with capital letters. The name of the publishing organization or the name of the publication in which the document was published, the volume and / or issue number, and the total number of pages of the book should be specified, respectively.

For example:

Einsele, G. 1992. *Sedimentary Basins*. Springer Verlag, 628.

Ketin, İ., Canitez, N. 1956. *Yapısal Jeoloji*. İTÜ, 308.

Meriç, E. 1983. *Foraminiferler*. Maden Tetkik ve Arama Genel Müdürlüğü Eğitim Serisi, 26, 280.

- If the document is published in a book containing the articles of various authors, the usual order for the document included in a periodical publication is followed until the end of the document title. Then the editors’ surnames and initials and the abbreviation of the editor word “Ed.” is written in parentheses. Then, the title of the book in which the document is located is written with the first letters in capital letters. Name of publishing organization. The place of publication, the volume number of the publication in which the document was published, and the numbers of the first and last pages of the document should be written.

For example:

Anderson, L. 1967. Latest information from seismic observations. Gaskell, T. F. (Ed.). The Earth's Mantle. Academic Press. London, 335-420.

Göncüoğlu, M. C., Turhan, N., Şentürk, K., Özcan, A., Uysal, S., Yalınız, K. 2000. A geotraverse across northwestern Türkiye. Bozkurt, E., Winchester, J. A., Piper, J. D. A. (Ed.). Tectonics and Magmatism in Türkiye and the Surrounding Area. Geological Society of London. Special Publication, 173, 139-162.

- If it is desired to specify the name of a book in which the writings of various authors are collected as a document; following the surnames and names of the book's editors, in parentheses the "Ed." statement is written. Year of publication. Title of the book with capital letters. The name of the publishing organization or the name of the publication in which the document was published, the volume and / or issue number and the total number of pages of the book should be specified.

For example:

Gaskel, T. F. (Ed.). 1967. The Earth's Mantle. Academic Press, 520.

- If the document is "published abstract", information about the document is given in the following order: Authors' surnames, authors' first names. Year of publication. Name of the document (paper). The name, date and place of the meeting where the paper is published, and the first and last page numbers in the book containing the abstract should be written.

For example:

Öztunalı, Ö., Yenyol, M. 1980. Yunak (Konya) yöresi kayaçlarının petrojenezi. Türkiye Jeoloji Kurumu 34. Bilim Teknik Kurultayı, Ankara, 36.

Yılmaz, Y. 2001. Some striking features of the Anatolian geology. 4. International Turkish Geology Symposium, 24-28 Eylül 2001, Adana, 13-14.

- If the mentioned document has not been published like report, lecture notes and etc., the word "unpublished" should be written at the end of the information about the document in parentheses after the information about the document is given

in the usual order for the document in a periodical publication.

For example:

Akyol, E. 1978. Palinoloji ders notları. EÜ Fen Fakültesi Yerbilimleri Bölümü, 45, İzmir (unpublished).

Özdemir, C., Biçen, C. 1971. Erzincan ili, İliç ilçesi ve civarı demir etütleri raporu. Maden Tetkik Arama Genel Müdürlüğü, Rapor No: 4461, 21, Ankara (unpublished).

- For unpublished courses, seminars and similar notes, the course organizer after document name. The place of the meeting. Title of the book and relevant page numbers should be given.

For example:

Walker, G.R., Mutti, E. 1973. Turbidity facies and facies associations. Society for Sedimentary Geology Pacific Section Short Course. Anaheim. Turbidities and Deep Water Sedimentation, 119-157.

- If the document is a thesis; author's surname, initial of the author's first name. Year of publication. Name of the thesis. The type of the thesis, the university where it was given, the total number of pages, its province and the word "unpublished" are written in parentheses.

For example:

Akıllı, H. 2019. Polatlı-Haymana (Ankara) civarı sıcak sularının izotop jeokimyası ($\delta^{18}\text{O}$, δD , 3H , $\delta^{13}\text{C}$, $\delta^{34}\text{S}$, $87\text{Sr}/86\text{Sr}$) ve ana iz element bileşimleri ile incelenmesi. PhD Thesis, Ankara University, 255, Ankara (unpublished).

Argun Aktan, Ö. 2019. Marmara Denizi Batı Kıta Sahaneliği Yüzeysel Çökellerinde Jeojenik ve Antropojenik Ağır Metal Zenginleşmesine Yönelik Araştırmalar (Şarköy Kanyonu, KB Türkiye). MSc Thesis, Ankara University, 179, Ankara.

- Anonymous works should be arranged according to the publishing institution.

For example:

MTA. 1964. 1/500.000 ölçekli Türkiye Jeoloji Haritası, İstanbul Paftası. Maden Tetkik ve Arama Genel Müdürlüğü, Ankara.

- For the documents that are in print, no date is put after the name of the author, the name of the article and the source to be published should be specified and the word “in print” and / or “in review” should be written at the end (in parentheses).

For example:

Ishihara, S. The granitoid and mineralization. Economic Geology 75th Anniversary (in press).

- Information downloaded from the Internet should be given in the form of the name of the institution, its web address, and the date on which the web address was accessed. Turkish references should be given directly in Turkish and should be written in Turkish characters.

For example:

ERD (Earthquake Research Department of Türkiye). <http://www.afad.gov.tr>. 3 March 2013.

- While citing the source, the original language should be adhered to, and the title of the article should not be translated.

6. Illustrations

- All of the drawings, photographs, plates and tables used in the article are referred to as “illustrating”.
- Illustrations should be used when their use is unavoidable or when they make the subject easier to understand.
- In the selection and arrangement of the format and size of the illustrations, an attitude should be made to prevent loss of space as much as possible considering the page length and layout of the bulletin.
- The number of illustrations used should be proportional to the size of the text.
- All illustrations should be submitted in separate files regardless of the text.
- Abbreviations should not be used in illustration explanations in the text and should be numbered in the order of mention within the text.
- Photographs and plates must be submitted as a computer file in which all details can be seen for the examination of the article, with EPS, TIFF or JPEG extension and at least 300 dpi resolution.

6.1. Figures

- Drawings and photographs other than the plate to be included in the article are evaluated together as “Figure” and numbered in the order of mention in the text.
- The figures should be prepared in computer considering the dimensions of a single column width as 7.4 cm or double column width as 15.8 cm. The figure area with its caption should not exceed 15.8x21 cm.
- While preparing the figures, unnecessary details should not be included and care should be taken not to use more space than necessary for the transfer of information.
- In figure descriptions, a space should be left after the word “Figure” is written, and the number is given in the usual sequence number, followed by a hyphen (-) and a space again, and a description of the relevant figure should be written. If the figure legend exceeds the bottom lines, the following lines should to be written after the “Figure 1-” statement alignment. Figure descriptions should be created as follows, without exceeding the edges of the figure and justified on both sides.

For example:

Figure 1- The district of Sandıklı (Afyon); a) geological map of the southwest, b) the general vertical section of the study area (Seymen, 1981), c) Türkiye’s most important neotectonic structures (modified from Koçyiğit, 1994).

- Drawings should be drawn in computer properly, clean and with care.
- The use of thin lines that may disappear when minimized in figures should be avoided.
- Symbols or letters used in all drawings should not be less than 2 mm (7 pt.) in Times New Roman.
- All standardized symbols used in the drawings should preferably be explained in the drawing, if they are too long then they should be explained in the figure below.
- Bar scale should be used in all drawings and the north direction should be indicated on all maps.
- The name of the author, description of the figure, figure number should not be included in the drawing.

- Photographs should reflect the aims of the subject and should be in adequate numbers.
- Figures should be framed.

6.2. Plates

- Plates should be used in cases where multiple photographs are required to be printed together on a special paper.
- Plate dimensions must be equal to the size of the bulletin's usable area of the page.
- Figure numbers should be written under each of the figures on the plate and bar scale should be used.
- Original plates must be attached to the final copy to be submitted in the case of acceptance of the manuscript.
- Figures and plates should be numbered among themselves and independently. Figures should be numbered with Latin numerals and plates with Roman numerals (eg Figure 1, Plate I).
- There should be no explanation text on the figures inside the plate.

6.3. Tables

- All tables should be arranged in word format and should be prepared in Times New Roman.
- Tables should not exceed the size of 15x8 cm together with the table caption.
- Table explanations should be created without exceeding the edges of the figure and justified as in the example below.

For example:

Table 1- Hydrogeochemical analysis results of geothermal waters in the study area.

7. Nomenclature and Abbreviation

- Abbreviations must be in the accepted international or national form. Unusual nomenclature and abbreviations that are not standardized in the article should be avoided. In cases where it is deemed necessary to use such nomenclature and abbreviations, the way and method followed should be explained.
- There should not be a dot between the words initials used in standard abbreviations (such as MTA, DSİ).
- Abbreviations of geography aspects should be made in English (N, S, E, W, NE and etc.).

The word group to be abbreviated should be written clearly where it is mentioned first time and the abbreviation should be given in parentheses, then only the abbreviated form should be written throughout the article.

- Systems with international validity (m, inch, etc.) should be used as the unit of measure. Decimals should be separated with commas in Turkish articles and with a period in English articles.
- The names of figures, plates and tables in the article should not be abbreviated. For example, "As seen in the generalized stratigraphic section of the region (Figure 1)".

7.1. Chronostratigraphic and Geochronologic Nomenclature

- "International Chronostratigraphic Chart" (<https://stratigraphy.org/chart>), which is updated annually by the International Stratigraphic Committee, should be taken into consideration in chronostratigraphic and geochronological nomenclature.
- Position within a chronostratigraphic unit can be expressed in adjectives indicating the position, for example: lower, middle, upper and etc. When using these adjectives, it should be decided whether the lower, middle and upper distinction is formal / informal in the International Chronostratigraphic Chart.

For example:

lower Miocene, Upper Holocene and etc.

- When stating the time where a geochronological unit is, temporal adjectives such as; early, middle, late and etc. are used. When using these adjectives, the International Chronostratigraphic Chart should be taken into consideration to decide whether the adjectives begin with capital or lowercase letters.

For example:

early Miocene, Late Holocene etc.

7.2. Paleontological Nomenclature and Spelling of Fossil Names

- Original names of fossils should be used.

For example:

Nummulites with limestone

- Fossil genus and species names are written in italics, cf., aff. and gr. etc. expressions are written as normal (perpendicular). When writing fossil names for the first time, the surnames of the people who identify them and the year in which they were first defined should be written. In later uses, the surnames and the year in which they are defined may not be written. The surnames and dates of identifiers coming at the end of the fossil names are not references, they should not be included in the mentioned documents.

For example:

Alveolina aragonensis Hottinger, 1960 not a reference.

Alveolina cf. *aragonensis* Hottinger, 1960 not a reference.

Alveolina aff. *aragonensis* Hottinger, 1960 not a reference.

Alveolina gr. *aragonensis* Hottinger, 1960 not a reference.

- After the first use of the same genus in the text is written clearly, it can be abbreviated as in the example so that it will not be confused with another genus in later use.

For example:

Alveolina aragonensis, *A. polathensis*, *A. ellipsoidalis* etc.

- If the date is in parentheses after the person describing it after the name of the fossil in the text, this is a reference and should be included in the mentioned documents.

For example:

Alveolina aragonensis Hottinger (1960) is a reference.

- The following rules should be taken into account when writing the systematic paleontologic section.
 - a. First of all, genus, species and subspecies to be identified should be written in hierarchical order like the order, upper family, family type species and so on. Later, the species to be described should be written together with the surnames and date of the people who defined the subspecies name. If there is a photograph of the described fossil, the plate or figure with the photograph should be added under the fossil name. The names of the authors listed here are not references, so they are not included in the reference.

For example:

Order: Foraminiferida Eichwald, 1830

Superfamily: Alveolinacea Ehrenberg, 1839

Family: Alveolinidae Ehrenberg, 1839

Type Genus: *Borelis* de Montfort, 1808

Type Species: *Borelis melenoides* de Montfort, 1808
= *Nautilus melo* Fichtel and Moll, 1798

Borelis vonderschmitti (Schweighauser, 1951)

(Plate II, Figure 3-5 or Figure 3A-H).

- b. Similar or synonyms (synonym) list should be left-aligned in chronological order. The page and figure number of the synonymous fossil in the relevant study should be included in the synonymous list. Authors in the synonymous list are references and must be included in the references.

For example:

1951 *Neoalveolina vonderschmitti* **Schweighauser**, page 468, Figures 1-4.

1974 *Borelis vonderschmitti* (Schweighauser), **Hottinger**, page 67, plate 98, Figures 1-7.

- c. After the synonymous list is given, the definition, explanations (similarities and differences), dimensions, material, stratigraphic distribution (according to the characteristics of the fossil) should be written.
- d. If the fossil is defined for the first time (new species) in the systematic paleontology section, the origin of the name, holotype, type locality, material, description, explanation (similarity and differences), age and geographical distribution, dimensions (according to the features that define the fossil) should be written. Photographs of the fossil identified for the first time by the authors must necessarily be placed in plates or figures.
- e. Bar scale indicating the size of fossils must be used definitely in plates / figures.

8. References

- In the references to be made in the Main Text, only the surnames of the authors and the publication year of the mentioned article should be specified. Referencing should be arranged according to one of the following examples:
- Referring to a publication with a single author (in chronological order):

-Altınlı (1972, 1976) defined the Bilecik sandstone in detail.

It is known that the fold axes of the Devonian and Carboniferous units around İstanbul are N-S trending (Ketin, 1953, 1956; Altınlı, 1999).

- Referring to a publication with two authors:
- The upper parts of the unit include Ilerdian fossils (Sirel and Gündüz, 1976; Keskin and Turhan, 1987, 1989).
- Referring to a publication with more than two authors:
- According to Caner et al. (1975), the Alıcı formation reflects the conditions of fluvial environment.
- Unit disappears by wedging towards E (Tokay et al., 1984).
- Referring to a reference in another publication:

It is known that Lebling mentioned about the existence of Liassic around Çakraz (Lebling, 1932; Charles, 1933).

- When referring to the works of the authors with the same surname in the same year, referring the authors' first names by writing their initials:
- Many studies have been done in the field of structural geology in the study area (Gutnic et al., 1979; Yılmaz A., 1983; Yılmaz, İ., 1983; Poisson et al., 1984 etc.).

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Assessment of active tectonics based on GIS and statistical model: a case study of upper Alaknanda catchment (western Himalaya, India)

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Research Article

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Clustering, Himalayan
Tectonics, Active
Tectonics.

ABSTRACT

Due to continuing deformation of the Earth's crust, degradation and aggradation processes, the upper part of the Alaknanda basin lying in the Garhwal Himalaya, India, is considered to be tectonically active. Active tectonics in this region made a considerable impact on the drainage system and the topographic expression. Using a digital elevation model (DEM) with a resolution of 30 m * 30 m and based on eight geomorphic indices [hypsometric integral (HI), drainage texture (Dt), asymmetry factor (AF), ruggedness number (Rd), valley floor width/height ratio (Vfw), lemniscate coefficient (k) and basin shape index (Bs)], we evaluated active tectonics. Application of Index of Active Tectonic (IAT) and clustering model disclose the sub-watersheds in the southern part covering watersheds 16, 17 and near Badrinath sub-watershed 4, 9 and 8 are tectonically highly active (Class-1, 1.500-1.781). Cluster analysis defines two prominent clusters of sub-watersheds: 2, 4, 14 and 15 in cluster 1; and 6, 7, 8 and 18 in cluster 2. Cluster 1 includes sub-watersheds 2, 4, 14, 15, and 16 and very high, high, and moderate IAT magnitudes. Sub-watersheds 6, 7, 8, and 18 are included in Cluster 2, which has very high and high tectonic activity.

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1. Introduction

In the Himalayan landscape, tectonics is the most important factor forming the present-day topography, which could have an additional role of erosion/denudation (Harkins et al., 2005; Andermann and Gloaguen, 2009; Perez-Pena et al., 2009). In the upper Alaknanda basin, crystallines of the Lesser and the Higher Himalaya exist at south and north, respectively (e.g., Metcalfe, 1993). This section of Alakanada catchment is composed of phyllites, slates and other metamorphic rocks (e.g., Celerier et al., 2009). It is possible to retrieve geomorphic evidence of active tectonics from topo-sheets, aerial photos, satellite

information and through morpho-tectonic parameters (Horton 1945; Keller and Pinter, 2002). Morphometric investigation presents quantitatively the geometry of basins to comprehend initial slant or differences in the drainage basin's topography, geomorphic history, structural control, recent tectonics as well as rock hardness (Strahler, 1964). The Alaknanda River Basin is a tectonically active area with a history of destructive landslides and earthquakes. Landslides and/or seismicity has been reported frequently from locations such as Chamoli, Birahi, Pipalkoti and Rudraprayag. This area is traversed by several significant tectonically active thrusts e.g., the Main

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Central Thrust (MCT)- I, also known as the Vaikrita Thrust; MCT II, which is also known as the Munsiri Thrust; MCT III, also known as the Ramgarh Thrust, and the North Almora Thrust. (Shukla et al., 2014). Therefore morphological study in this area is an important exercise.

Even though several geomorphologic works have been undertaken on the Alaknanda river and the

surroundings (review in Table 1), a morphometric study such as the preset work remained a due. This work involves hierarchical clustering of drainages and estimates Index of Active Tectonics (IAT) for different watersheds of the area taken into consideration. These are performed in order to understand the different degrees of tectonic activeness within the considered area.

Table 1- Geomorphologic research done on Alaknanda river valley that partially or completely covers the study area of this work.

Sl no.	Reference	Aim	Key approach	Software used	Salient result
1	Bhattacharya and Jugran (1982)	To evaluate land subsidence as a landslide cause.	Ground truth has been used in conjunction with aerial photo interpretation.	-	The important factor in soil subsidence is seepage.
2	Barnard et al. (2001)	To comprehend the causes of landslides, both natural and anthropogenic.	Major landslides were mapped and samples from boulders on the surface were taken for cosmogenic radionuclide dating.	-	Human activity was responsible for ~ 2/3 of the landslides, primarily by removing toes of the slope at road cuttings.
3	Joshi and Kumar (2006)	To determine the relationship between historical occurrences of natural disasters and very heavy rainfall.	Daily rainfall data analysed statistically.	-	Frequent extreme rainfall is one of the main causes of landslides.
4	Sarkar et al. (2006)	To identify unstable slopes along the National Highway in the Alaknanda valley of Garhwal Himalaya.	Through photo documentation, probable slope failure zones were demarcated.	-	Mitigation of landslide-prone zones recommended.
5	Sati et al. (2007)	To attribute the development of the morphologic evidence along the North Almora Thrust.	Comprehending the Late Quaternary seismicity in the Alaknanda Valley near Srinagar from the morphologic evidence of Uttarak Fluvial terraces, entrenched stream channels, landslide-induced ponding, and active and stabilised landslide deposits.	-	NAT and its related lineaments are active in the Late Quaternary Period.
6	Sundriyal et al. (2007)	To carry out observations on lake deposits that have been impacted by landslides at the south-dipping, E-W and NW-SE trending North Almora Thrust.	Application of lithofacies, geochemistry and optically stimulated luminescence chronology.	-	Second and third order tributaries of the Alaknanda river were temporarily dammed due to activation of crumpled and unstable phyllite-dominated slopes. Seasonal deposition is indicated by sedimentary patterns of the succession under a transient lacustrine environment.
7	Kundalia et al. (2009)	To set rainfall thresholds for landslide initiation.	To determine the connection between rainfall amounts and the onset of a slope failure, an analytical approach is used.		Precipitation is the primary mechanism for slope failure.
8	Tyagi et al. (2009)	To locate areas of substantial surface uplift in the Alaknanda valley .	Application of steepness index.	ERDAS IMAGINE	Convex river profiles and high steepness index values identify areas that are experiencing quicker surface uplift. The zone of high uplift (incision) are the Main Central Thrust (MCT). South of the MCT, at Chamoli, Nand Prayag, and Karn Prayag.

Table 1- Continued.

9	Juyal et al. (2010)	To understand geographic and temporal variability in stages of aggradation/incision in response to variations in climate and seismicity over the Late Quaternary in the Alaknanda valley.	Fill and strath terraces were mapped using total station survey. Paleoenvironmental reconstruction employed the fill sequences' sediment texture, structures and vertical lithostratigraphy. Infrared stimulated luminescence applied on feldspar grains.	-	Debris flow terraces formed during the early part of pluvial Marine Isotopic Stage 3 are the earliest fluvial landforms retained in the south of the Main Central Thrust.
10	Onagh et al. (2012)	To produce a landslide susceptibility map .	Multiple linear regression analysis using slope, aspect, lithology, land cover, rainfall, distances from fault, river, and road .	ILWIS 3.31 Academic, Arc GIS 9.3, Global Mapper 13.0 and Excel	The study area has been divided into Low, Moderate, High, and Very High relative landslide susceptibility classifications.
11	Parmar and Purohit (2013)	Mapping landslide hazard zones in Alaknanda Valley.	Lithology, structure, slope morphometry, relative relief, landuse and land cover, and hydrological conditions considered.		Shear zones, faults, heavily weathered and saturated sediments with unfavourable discontinuities characterize very high hazard zones.
12	Sarkar et al. (2013)	To evaluate the level of risk in the probable landslide zones, an effort has been made to quantify landslide intensity.	By assessing the severity of the present and possible landslides, landslide hazard assessment has been done to define the hazard levels. Individual landslides were initially subjected to hazard assessments in order to determine the possible risk of the research region.	ArcGIS (<i>version not referred</i>)	Most of the time, rock fall, rock slide and debris flow fall into the very high and high risk categories. To categorize the zones into different classes, the cumulative impact of landslide hazards was considered.
13	Shukla et al. (2014)	To assess the tectonic activeness using morphometric techniques and GIS .	Stream length ratio, bifurcation ratio, transverse topographic symmetry factor, asymmetry factor, drainage density, stream frequency, drainage texture form factor, circulatory ratio, and elongation ratio, valley width to valley height ratio, roughness number, the relief ratio, and the hypsometric integral have been computed on 8 sub basins.	Arc GIS 9.3, ERDAS 9.1 and MicroDEM software	The regions surrounding the MCT II (Munsiari Thrust), MCT III (Ramgarh Thrust), and North Almora Thrust, which are located in sub-basins 2, 3 and sections of 5 are tectonically quite active.
14	Chaturvedi et al. (2014)	Regional landslide susceptibility analysis using remote sensing.	A landslide susceptibility map was created involving lithology, fault, lineament, geomorphology, drainage, slope, aspect, land-use and land cover, soil texture, and soil depth.	-	After field validation, this method for susceptibility evaluation is fairly reliable and exact.
15	Devrani and Singh (2014)	To recognize the significance of location and regional elements in the valley filling process in an active mountain belt.	Hypsometry along with the lithofacies analysis were conducted .	ArcGIS 9.3	Sedimentation in the Pipalkoti valley stretch was mostly caused due to local reasons.
16	Gupta and Tandon (2014)	To identify the probable zones of instability, which causes frequent rock fall.	For each of the 23 locations where <i>in-situ</i> rocks are found, a kinematic rockfall hazard analysis was done.	-	The HH is mostly of low- to moderate-hazard, whereas the LH has a moderate- to high-hazard potential.
17	Chaudhary et al. (2015)	To understand the genesis of (paleo)valley.	The geomorphic configuration, sedimentological character, sediment source, flow velocity, and Optical Stimulated Luminescence (OSL) chronology have all been emphasised by authors.	ArcGIS 9.1	In the Alaknanda valley in the Central Himalaya, paleovalleys formed in two ways- (1) by significant valley aggradation, and (2) locally through landslides and lake breaching. Most of the Alaknanda valley's paleovalleys evolved between 15 to 8 ka, during significant valley aggradation.

Table 1- Continued.

18	Sajwan and Sushil (2016)	To examine the factors causing slope instability and landslides.	510 landslides identified in field. Landslides categorised as per rigidity and rockmass movements and debris that constitute the slid mass.		The atmospheric precipitation that causes landslides and flash floods can both be anticipated if real-time rainfall data is available.
19	Sundriyal et al. (2015)	To determine the type and reasons for the valley damage in June 2013.	Geochemical analysis of flood sediments.	-	Sediments came from two main sources: landslides from the Higher and Lesser Himalaya, and moraines and alluvial fans from the Trans and Higher Himalaya.
20	Chandak et al. (2016)	To highlight the locations at risk for landslides.	Information Value Method was adopted for landslide hazard mapping.	Integrated Land and Water Information System (ILWIS)	The unexpected dislodgment of small to large jointed blocks leads to rock avalanches / debris flows.
21	Rana et al. (2016 a,b)	To analyze the morphometric indices for neotectonic implications in a fluvial erosion-dominated regime.	Application of morphometric indices e.g., stream order, stream number, bifurcation ratio, drainage density, stream frequency, texture ratio, circularity ratio, basin hypsometry and elevation-relief ratio to assess neotectonic imprint upon the study area.	ArcGIS 9.3	The findings point to two high deformation/uplift zones in the valley: the first is in the Inner LH, close to the Main Central Thrust (MCT), and the second is in the Outer LH, south of the Tones Thrust.
22	Rana et al. (2016 a,b)	To categorize the seismic and aseismic categories for the fluvio-lacustrine sediment successions and valley-fill terraces.	Analyzing Soft sediment deformation structure and brittle deformation features in fluvial and fluvio-lacustrine deposits in the Alaknanda Valley.	-	Most of the deformation structures produced presumably in aseismic manner, although the terrain lies in the active seismic zone of the Central Himalaya.
23	Maurya et al. (2017)	To map probable landslide hazard zones in Badrinath, Garhwal Himalaya.	Application of GIS techniques .	ArcGIS 9.2	54% of the area has a high chance of landslides. Risk is moderate in 25% of the area.
24	Mehta et al. (2017)	To assess the change in terrain as an effect of 2013's disaster in Alaknanda and Mandakini basin.	The use of a field survey and a GIS technique allowed us to evaluate the changes in the topography.		Debris of 0.3×10^6 and 0.72×10^6 m ³ deposited at Govindghat and Pulna Village, respectively.
25	Prasad and Pandey (2017)	To evaluate the effects of LULC change, climate change, and their effects on hydrological hazard.	Both qualitative and quantitative methodologies. Peoples' perception about the origin of hazards, trend, frequency, magnitude, and mitigation measures were analyzed using a Likert-scale.		Households in the away district headquarter (ADH) village are more vulnerable than those in the near district headquarter (NDH) village. Due to their traditional and local knowledge of the responses to climate change, the NDH households had more potential for adjustment within the framing system.
26	Khanduri (2018)	To analyse and evaluate the effects of landslides in the Chamoli district.	Understanding the historical landslip episodes and resulting damages by overlying several thematic layers.	Arc View 9.3	These slides have been made possible in large part by the combination of heavy rainfall and the weak shear strength of the rocks.
27	Sharma et al. (2018)	To assess the relative active tectonics of the Alaknanda basin of Garhwal Himalaya.	Active tectonics in 8 sub-basins of the Alaknanda basin using DEMs based on seismicity and 8 geomorphic indices.	ArcGIS 10.2	The middle portion of the tectonically most active sub-basin I exhibits movement rate of 41.1 mm y^{-1} in accordance with GNSS data.

Table 1- Continued.

28	Chauniyal (2018)	To locate the areas that have recently experienced uplift and displacement along the NAT in the Lesser Garhwal Himalaya's Lower Alaknanda Valley.	To identify places of neotectonic activity along N-S transverse faults.	-	Neotectonic movement along the NAT were noticeable between 15 to 9 ka and demonstrated recurring tectonics. Minor structures characteristic of Quaternary sediments identified.
29	Pant et al. (2020)	To identify the soil erosion and flood hazard zones using morphometric techniques.	Six primary morphotectonic parameters were examined on the upper Alaknanda basin and its sub-basins.	ArcGIS and SPSS (v.22)	Future development in the region should consider soil erosion, tectonic activity and potential for flash floods.
30	Khanduri and Rautela (2021)	To recognise the primary causes of slope instability.	In a GIS environment, the distribution of landslides has been connected with numerous parameters affecting their occurrence.	Arc Info 9.3	Landslides are most strongly influenced by proximity to the road and drainage system.
31	Meghanadh et al. (2021)	To generate a of landslide susceptibility map between Rudraprayag and Srinagar.	Multi-temporal interferometric synthetic aperture radar (MT-InSAR) and AHP approaches.	Sentinel Application Platform (SNAP) and Stanford Method of Persistent Scatterer (StaMPS)	There have been found 44.5% new landslide susceptible zones (LSZs).
32	Kumar et al. (2022)	To assess the vulnerable sites of slope instability along the NH-58 from Dhari Devi to Rudraprayag.	Kinematic analysis, geologic strength index, uniaxial compressive strength, rock quality designation, rock mass rating, slope mass rating and continuous slope mass rating are considered to assess slope instability.	-	Active landslides are caused by the Kaliasuar Fault in Uttarakhand. The Kaliasaur landslide is unstable due to pre-existing tectonic fracture and a plunging fold.
33	Joshi and Biswakarma (2023)	To compare models regarding landslide susceptibility mapping in the Alaknanda catchment area.	Application of Frequency Ratio Model, Analytic Hierarchical Process and Information Value Method to evaluate landslide susceptibility.	ArcGIS, Earth Resource Data Analysis System (ERDAS) IMAGINE	The Information Value Method yields the most precise results.
34	Clubb et al. (2023)	To evaluate sediment-storage mechanisms and identify the main influences on valley-floor morphology.	Application of the valley-floor width ratio throughout the Himalaya.	LSDTopoTools software	Long-term tectonically induced exhumation, not water discharge or bedrock erodibility, controls valley-floor width.
35	Raha et al. (this work)	To assess the active tectonics of the Upper Alaknanda Catchment.	Morphometric parameters such as Hypsometric integral (HI), Drainage texture (Dt), Asymmetry Factor (AF), Ruggedness number (Rd), Circularity Ratio (Rc), Valley Floor Width/Height Ratio (Vfw), Lemniscate Coefficient (k) and Basin Shape Index (Bs) are taken into consideration for the calculation of IAT. Cluster analysis has been performed on the sub-watersheds using dendrogram.	ArcGIS 10.3 and SPSS software (v26)	Tectonic activity is particularly high in the southern region where the NW-SE tectonic lineaments intersect with MCT II, encompassing sub-watersheds 16 and 17, and where numerous lineaments are present in various directions.

2. Geology

The upper catchment of the Alaknanda watershed lies under the Tethys Himalaya (TH) and is composed of Badrinath Granite and mostly the Central Crystallines. Towards south it is characterized by the Tethys Himalaya, the Higher Himalaya (HH), and a portion of the Lesser Himalaya (LH). Trans-Himadri Fault (THF) or the South Tibetan Detachment System (STDS) separates THS from HH. Main Central Thrust (MCT) is the boundary between the HH and the LH (Figure 1). The southern boundary of the LH is the Main Boundary Thrust (MBT). To the north and south of the Dudatoli syncline, respectively, the North Almora Thrust (NAT) and South Almora Thrust (SAT) divide the LH (Yin, 2006; Mukherjee, 2013). NAT is an out-of-sequence thrust and is presently active (review in Mukherjee, 2015).

With a variety of lithologies, dissection and drainage developments, the region is intricate. The litho-units in the study area are the Ramgarh Formation (lower crystalline zone) between the Munsiyari and

Ramgarh Thrusts, the Munsiyari Formation (middle crystalline zone) between the Vaikrita and Munsiyari Thrusts, and the Vaikrita Group (upper crystalline portion lying between the STDS and the Vaikrita Thrust) (Srivastava and Mitra, 1994; Valdiya, 1995, 2001; Bhattacharya, 2008; Ray and Srivastava, 2010). In detail, the study area comprises of ten major geologic units- Badrinath Granite/Tourmaline Granite, Baijanath Formation, Central Crystallines, Garhwal Group (Pithoragarh), Garhwal Group (Bering), Martoli Formation, Rakcham Granite, Rawali Bagar Group, Sumna Group and an unamapped area (Figure 2).

3. Data acquisition and methods

From the Digital Elevation Model (DEM) of 30 m * 30 m spatial resolution of the SRTM the drainage lines were delineated. Based on the DEM (taken on 11-Feb-2000, released on 23-Sept-2014), watersheds were identified, evaluated and morphometric indices were generated (Table 2).

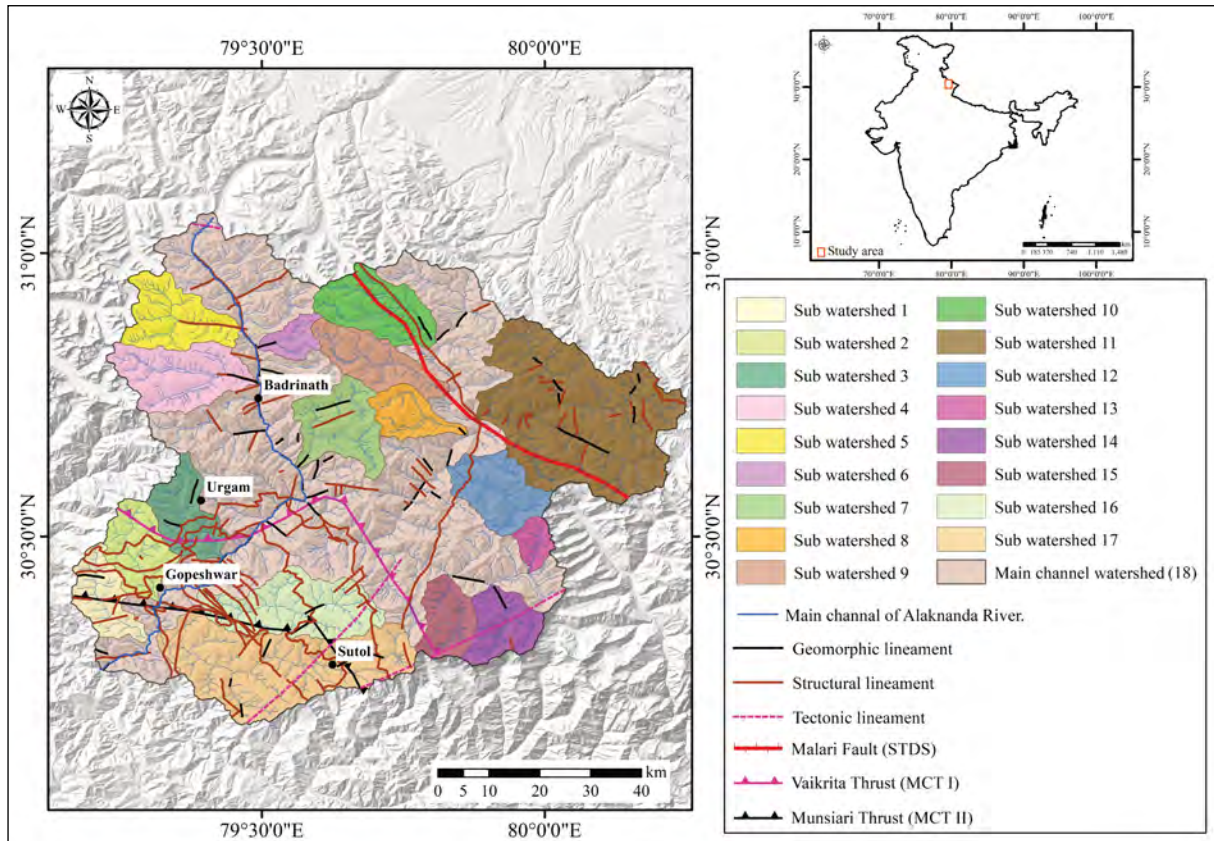


Figure 1- Location of the study area upper part of Alaknanda River with 17 delineated sub-watersheds and major tectonic features.

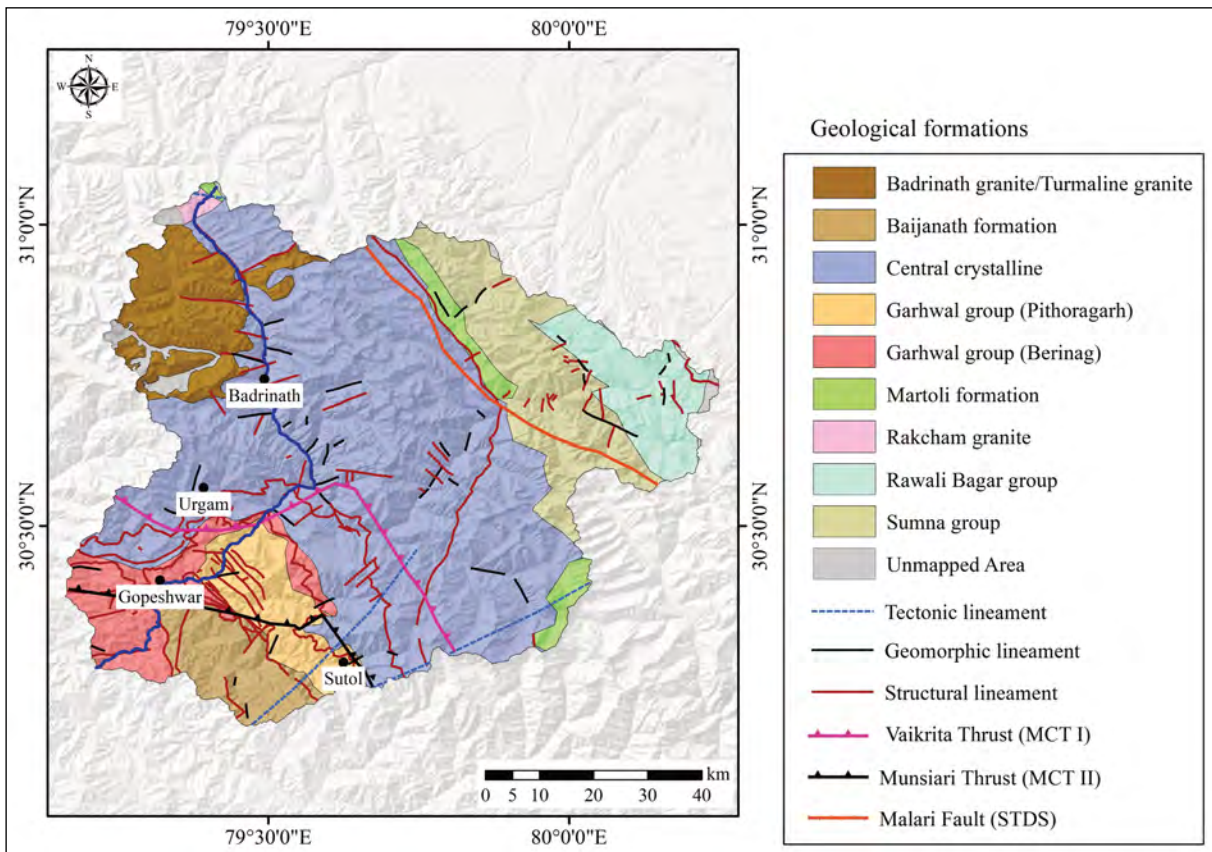


Figure 2- Map of general geology of the study area.

There were no clouds present when the photographs were taken. Therefore, no atmospheric correction was necessary. Several areal, linear and relief are assessed using the ArcGIS of the version of 10.3 (2014). The desired threshold value was utilized to create and extract the stream network using the D8 Python program incorporated in the ArcGIS itself. Slope directions have been deduced from each pixel to the eight-neighbour pixels. The watershed limits delineated by discrete flow angles and a single flow direction. The extracted drainage lines were mapped in the ArcGIS 10.3 (2014) platform. Seventeen sub-watersheds were delineated along the main channel of Alaknanda. The delineation of the sub-watersheds is based on the order of streams as per Strahler (1964). The streams of and above third order are considered for the delineation of the sub-watersheds. The lower order streams are not considered as the sub-watersheds do not have a considerable areal extent to understand the tectonic imprint upon them.

After careful review of the past literature, we have selected eight morphometric indices incorporating relief, areal aspects. The selected parameters are: Hypsometric integral (HI), Drainage texture (D_t), Asymmetry Factor (AF), Ruggedness number (R_d), Circularity Ratio (R_c), Valley Floor Width/Height Ratio (V_{fw}), Lemniscate Coefficient (k) and Basin Shape Index (B_s). The details of all the parameters along with its relevance in morpho-tectonic studies are listed in the Table 2. These are considered to calculate the IAT and to perform the clustering analysis using dendrogram for the considered watersheds. IAT ranks the watersheds as per their tectonic prioritization into four classes- very high, high, medium and low through mutual comparison. Magnitudes of these parameters have been grouped into four classes 1 to 4. The watersheds are categorized under low, moderate, high and very high classes in respect to tectonic activeness. The previously mentioned indices have been adopted for the selected seventeen sub watersheds as well as on the main channel watershed. The relation is:

Table 2- Details of equations and results of considered spatial and linear parameters for the study.

Sl. No.	Formula for basin scale indicators (unitless)	Equations & meaning of symbols	Explanations	References
1.	Hypsometric integral (HI)	$\frac{(Ele_{avg} - Ele_{min})}{(Ele_{max} - Ele_{min})}$ Ele_{avg}: Average of elevation Ele_{min}: Minimum elevation Ele_{max}: Maximum elevation	The hypsometric integral is a dimensionless number that allows different sub-basins to be compared regardless of the scale. Hypsometric Integral indicates both tectonic activeness and lithological control. The rate of relief uplift is positively correlated with the hypsometric integral. HI value varies between 0 and 1 and a value nearer to 1 indicate a tectonically very active basin, 0.5 HI value is an indicator of equilibrium (mature) state and a value nearer to 0 indicates tectonically less active basin. Pike and Wilson (1971) mathematically derived the simpler form of hypsometric integral and shown proved that it is a valid approximation of actual hypsometric integral.	Strahler (1952) e.g. Chen et al. (2003); e.g. Pike and Wilson (1971); (Hamdouni et al., 2008).
2.	Elongation ratio (R_e)	$Re = 1.128 \sqrt{\frac{A}{L_b}}$ A: signifies the area of the basin, L_b: denotes the length of the basin.	Elongation ratio is influenced by geology and climate. The values vary from 0 to 1 (e.g. Wołosiewicz, 2018). As the landscape evolves, the river basin becomes circular and the value tends to be 1.	Schumm (1956); e.g. Wołosiewicz (2018)
3.	Lemniscate coefficient (k)	$k = \frac{\pi L_b^2}{4A_B}$ Where, A_B is the area of basin and L_b is the length of the basin.	Lemniscate coefficient based on expression at basin with Lemniscate curve (i.e. ratio of basin area to its length) in order to determine the shape of the basin. Higher Lemniscate values indicate an elongated basin, which in term denote tectonically very active drainage basin, whereas the low Lemniscate values are associated with less tectonically active basin.	Chorley (1957)
4.	Asymmetry factor (AF)	$AF = \left(\frac{A_r}{A_t}\right) * 100$ A_r: area of the basin (km^2) to the right of the main channel facing downstream and A_t: total area (km^2) of the basin.	An indicator to measure how much a river basin is tilted due to tectonics. Tectonic activity causes the main stream to change course, sloping away from the basin's midline.	Hare and Gardner (1985); e.g. Anand and Pradhan (2019)
5.	Ruggedness number (Hd)	$Hd = R * D$ R = Basin relief D = Drainage density of the basin	Surface unevenness as topographic differentiation is measured.	Horton (1932)
6.	Valley floor width height ratio (V_{fw})	$V_{fw} = \frac{2V_{fw}}{[(E_{ld} - E_{rd}) + (E_{rd} - E_{sc})]}$ Here V_{fw}: width of the valley floor, E_{ld} and E_{rd}: elevations of the divide on the left and right side of the valley, respectively. E_{sc}: average elevation of the valley.	This index differentiates between valleys with a wide floor relative to the height of valley dividers with a "U" shape compared to narrow, steep valleys with a "V" shape. Valleys with a U shape generally have high values of V_p while V-shaped valleys with relatively low values. Since uplift is associated with incision, the index is thought to be a cause for dynamic tectonics where low values of V_f are associated with higher rates of uplift and incision. This index may be a degree of incision and not uplift; but in an equilibrium state, incision and uplift are nearly coordinated.	Bull and McFadden (1977)

Table 2- Continued.

7.	Drainage texture (D _t)	$D_t = N_u/P$ Nu: Number of streams of a given order. P: Perimeter (km) of the watershed.	The underlying lithology, infiltration potential, and relief characteristics of the terrain all affect drainage texture. D _t is the total number of stream segments in all orders along the watershed's edge (Horton, 1945). This is the relative channel spacing in fluidly prepared terrain, depending on many natural factors such as climate, precipitation, vegetation, rock / soil type, permeability, undulations, and watershed development stages. On the other hand, T is the product of D _d and F. It is calculated by multiplying the drainage density by the current frequency. If less than 4, T is classified as a coarse drainage texture. Intermediate drainage structure if it is between 4 and 10; fine drainage structure if it is between 10 and 15; ultrafine drainage structure if it exceeds 15 (Smith 1950).	Cox (1994)
8.	Basin shape index (Bs)	$B_s = \frac{Bl}{B_w}$ Bl: measured length from headwater to the point on the mouth of the basin, B _w : measured width at the widest point on the basin.	In the tectonically active area, the basin tends to be elongated.	E.g. Ramírez-Herrera (1998); Bull and McFadden (1977); Anand and Pradhan (2019)

$$IATc = \sum p_n(1r + 2r + 3r \dots + nr)/N \quad (\text{equation 1})$$

IATc: class of Index of Active Tectonics, p_n : parameter number, r: rank, N: number of parameters.

Clustering can discriminate signals (grouping signals) from noise (source signals). This technique works on unsupervised classification. Therefore, no prior assumptions are needed. This enables a better research outcome from the heterogeneous value ranges of the concerned indicators. In this study, hierarchical clustering is performed based on same indicators in sub-watershed scale. Using the Euclidean-based dissimilarity method, each segment was compared to its neighbours to assess how similar they were. The procedure was carried out using the following formula:

$$dR = (\sqrt{\sum_{i=0}^n (X_i - Y_i)})/n \quad (\text{equation 2; Clubb et al., 2014})$$

Here n: number of segments, X_i: distance between the segments, Y_i: difference in the steepness indices between the two profiles, i: component of the array, and dR: Euclidean-based dissimilarity. Using Ward's minimum variance method, hierarchical clustering was carried out using the dR values in the SPSS software (version 26, 2019). To show how each

segment mutually relates, a dendrogram is created. Information about stress patterns is gathered from the World Stress Map (Internet reference 2: <https://www.world-stress-map.org/> (Accessed on 16-Aug-2023).

4. Results

IAT was computed by taking the mean of the different classes of the areal and relief parameter. IAT values were sorted into four classes (Table 3) and mapped. As per the IAT analysis, sub-watersheds 4, 8, 9, 16 and 17 come under the tectonically very highly active region where IAT values under the range of 1.500-1.781. Sub-watersheds 6, 7, 10, 15 and 16 are under the magnitude of high tectonic activity (IAT = 1.782-2.063). The IAT range from 2.064-2.344 includes 1, 2, 3 and 14 sub watersheds, which are moderately active. Sub-watersheds at the NE portion as 11, 12 and 13 and at the NW part as 3 and 5 are under low range of active tectonic with IAT = 2.345-2.625 (Table 3). It is clear from the IAT map (Figure 3) that (i) tectonically active regions (watersheds 16 and 17) are characterized by the presence of Munsiyari Thrust (MCT II) that extends east-west. Besides there are NE-trending tectonic lineament and several multidirectional structural lineaments. (ii) Tectonically

Table 3- Class ranges and results of IAT analysis with magnitude.

Sub-watershed	Asymmetry Factor (AF)	Basin Shape (Bs)	Elongation Ratio(Re)	Lemniscate Coefficient (k)	Hypsometric Integral (HI)	Drainage Texture (Dt)	Ruggedness Number (Rd)	Valley Floor Width/ Height Ratio (Vfw)
1	6.955	1.836	0.709	1.989	0.383	1.963	21.462	0.030
2	4.619	2.029	0.683	2.143	0.426	2.135	29.896	0.016
3	1.666	2.089	0.656	2.323	0.465	1.795	47.841	0.164
4	8.518	2.046	0.672	2.214	0.469	4.621	52.415	0.070
5	1.324	1.248	0.819	1.488	0.509	3.476	32.595	0.420
6	13.396	1.422	0.679	2.167	0.474	1.760	18.309	0.576
7	19.142	1.096	0.780	1.643	0.511	4.146	49.232	0.125
8	18.105	1.618	0.629	2.522	0.524	3.401	45.456	0.098
9	0.695	2.645	0.549	3.319	0.460	2.361	58.393	0.209
10	16.025	1.856	0.729	1.878	0.398	3.996	53.190	0.402
11	4.388	1.219	0.923	1.172	0.461	5.704	24.294	0.173
12	0.127	1.434	0.790	1.601	0.523	4.443	31.643	0.230
13	6.122	1.432	0.697	2.057	0.426	1.454	36.302	0.704
14	8.260	1.436	0.806	1.537	0.367	2.641	58.522	0.026
15	10.702	1.937	0.745	1.799	0.513	2.741	54.558	0.272
16	4.579	2.216	0.604	2.740	0.367	2.342	46.025	0.044
17	2.736	2.348	0.608	2.701	0.313	5.037	45.065	0.023
Main channel watershed (18)	26.304	1.247	0.907	1.214	0.479	15.824	29.045	0.019

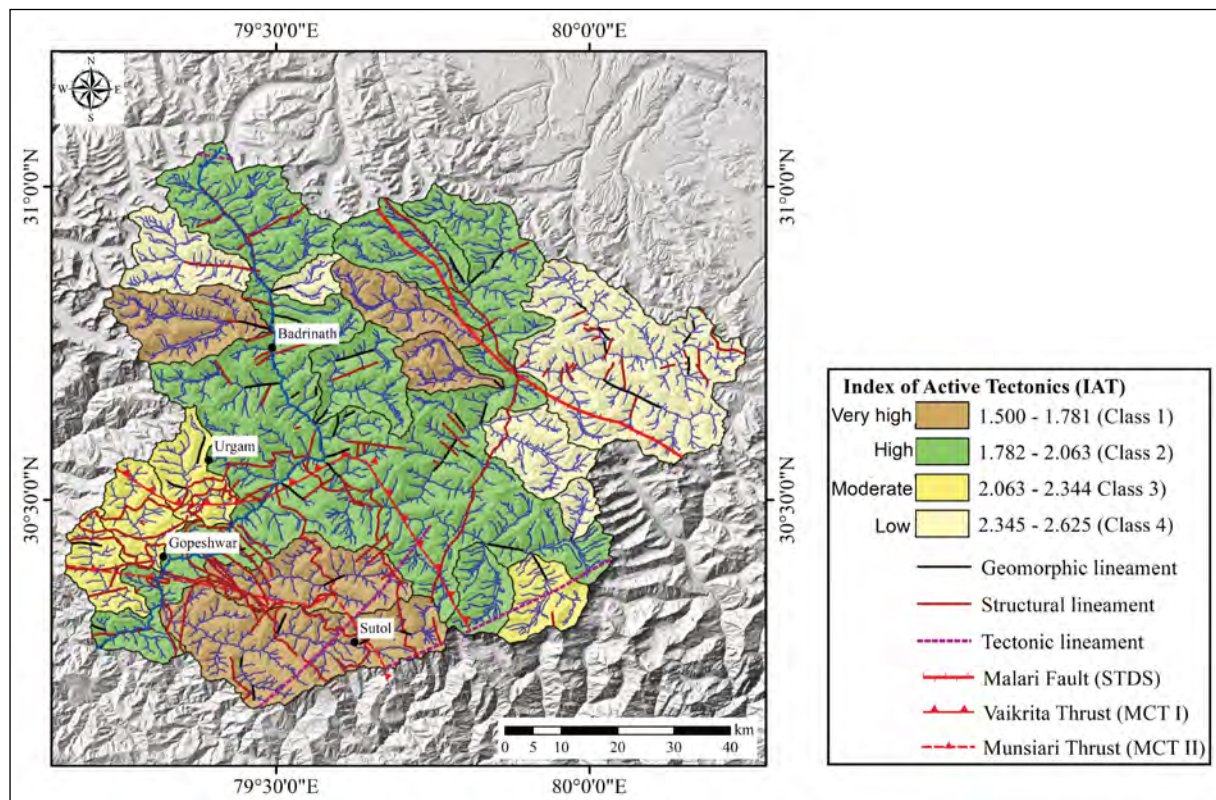


Figure 3- Map of Index of Active Tectonics (IAT) with 4 classes and 4 magnitudes as very high, high, moderate and low.

less active regions are distinguished by fewer presence or even absence of structural lineaments / faults.

The prepared dendrogram from the dR values enabled clustering of the calculated indicators of all watersheds (Figure 4). Cluster investigation of waterway profiles can compare the groups of similar waterway profiles inside heterogeneous scenes. The dR values are calculated to create a symmetric nearness lattice (Table 4). There are two major clusters as per the rescaled remove cluster combination. These are cluster 1 (sub-watersheds 2, 4, 14, 15 and 16) and cluster 2 (sub-watersheds 6-8 and 18) (Table 4).

5. Discussions

Comparison between two methods as IAT and clustering done in this work indicate a positive similarity in assessing active tectonics of Alaknanda sub-watersheds. Cluster 1 covers watersheds 2, 4, 14, 15 and 16 belong to very high (1.500-1.781), high

Table 4- Class ranges and results of IAT analysis with magnitude.

Class range	Magnitude	Sub-watersheds
1.500-1.781	Very High	4, 8,9, 16, 17
1.782-2.063	High	10,18,15
2.064-2.344	Moderate	1,2, 3, 14
2.35-2.625	Low	11,12,13

(1.782-2.063) and moderate (2.064-2.344) magnitudes of IAT. Cluster 2 covers sub-watersheds 6, 7, 8 and 18, which are under very high (IAT = 1.500-1.781) and high (IAT = 1.782-2.063) tectonic activeness (Table 5a,b).

Active thrust faults in the southern direction of the considered area covers sub watersheds 1, 2, 7, 16, and 18 (Figure 5). These watersheds belong to class 1 and 3 according to the IAT classes. Along the main channel of Alaknanda river near 20 km upstream of Gopeshwar, a hitherto unnamed strike-slip fault

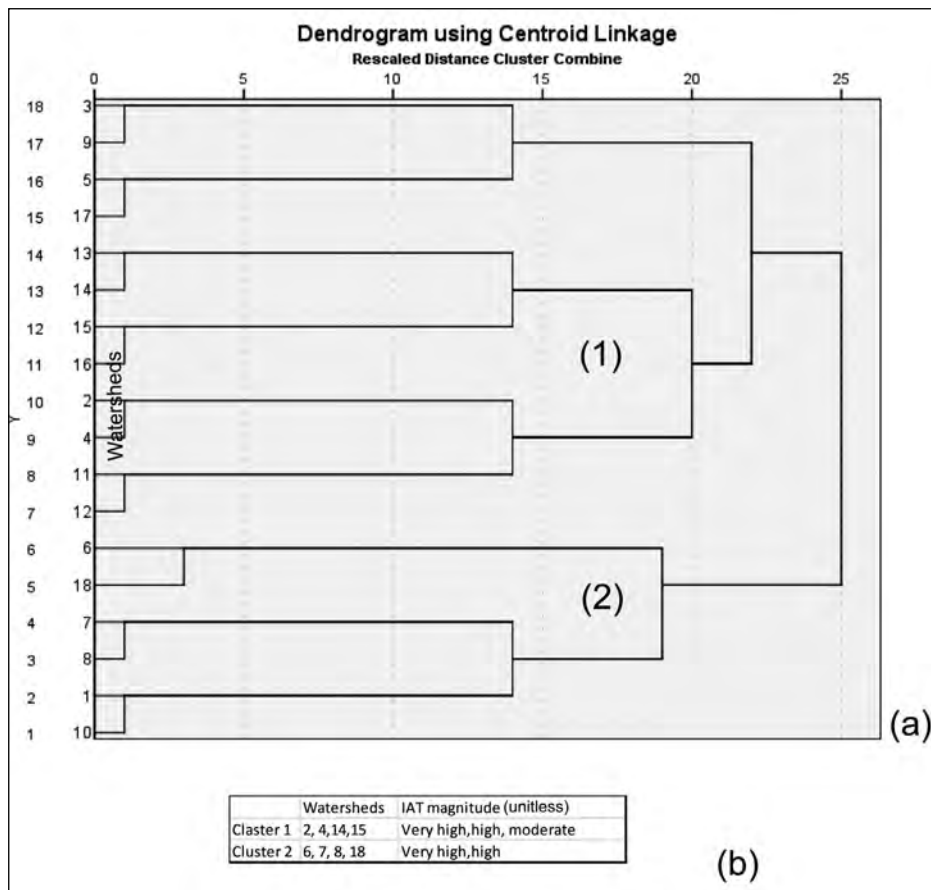


Figure 4- a) Dendrogram showing two specific clusters, b) Considered watersheds under class 1 and 2.

Table 5a- Results of agglomeration schedule for calculating cluster 1 and 2.

Agglomeration Schedule						
Stage	Cluster	Combined	Coefficients	Stage Cluster First Appears		Next Stage
	Cluster 1	Cluster 2		Cluster 1	Cluster 2	
1	5	12	3.358	0	0	6
2	16	17	11.599	0	0	4
3	4	15	13.126	0	0	7
4	3	16	14.503	0	2	13
5	7	8	16.956	0	0	10
6	2	5	17.929	0	1	9
7	4	14	25.790	3	0	11
8	1	11	29.720	0	0	12
9	2	13	34.458	6	0	14
10	7	10	36.056	5	0	15
11	4	9	60.159	7	0	13
12	1	6	71.818	8	0	14
13	3	4	97.740	4	11	15
14	1	2	129.028	12	9	16
15	3	7	149.008	13	10	17
16	1	18	262.847	14	0	17
17	1	3	468.580	16	15	0

Table 5b- Proximity Matrix (similarity) of 18 sub-watersheds.

Proximity Matrix																		
Case	Correlation between Vectors of Values																	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	1.000	0.986	0.960	0.987	0.957	0.929	0.994	0.995	0.954	0.998	0.972	0.943	0.987	0.985	0.992	0.976	0.964	0.813
2	0.986	1.000	0.993	0.999	0.991	0.854	0.967	0.966	0.991	0.986	0.984	0.983	0.999	0.999	0.997	0.999	0.994	0.725
3	0.960	0.993	1.000	0.991	0.998	0.792	0.934	0.933	1.000	0.962	0.974	0.994	0.991	0.993	0.986	0.998	0.997	0.650
4	0.987	0.999	0.991	1.000	0.991	0.862	0.973	0.971	0.987	0.989	0.990	0.983	0.998	0.999	0.998	0.997	0.993	0.747
5	0.957	0.991	0.998	0.991	1.000	0.786	0.933	0.930	0.998	0.961	0.984	0.998	0.987	0.991	0.983	0.996	0.999	0.670
6	0.929	0.854	0.792	0.862	0.786	1.000	0.955	0.958	0.777	0.926	0.843	0.756	0.866	0.856	0.882	0.829	0.799	0.914
7	0.994	0.967	0.934	0.973	0.933	0.955	1.000	1.000	0.925	0.996	0.960	0.915	0.973	0.969	0.981	0.954	0.939	0.855
8	0.995	0.966	0.933	0.971	0.930	0.958	1.000	1.000	0.924	0.995	0.956	0.911	0.972	0.968	0.980	0.953	0.937	0.851
9	0.954	0.991	1.000	0.987	0.998	0.777	0.925	0.924	1.000	0.955	0.971	0.995	0.987	0.990	0.981	0.996	0.997	0.635
10	0.998	0.986	0.962	0.989	0.961	0.926	0.996	0.995	0.955	1.000	0.976	0.946	0.989	0.987	0.994	0.977	0.966	0.815
11	0.972	0.984	0.974	0.990	0.984	0.843	0.960	0.956	0.971	0.976	1.000	0.982	0.980	0.984	0.982	0.981	0.987	0.784
12	0.943	0.983	0.994	0.983	0.998	0.756	0.915	0.911	0.995	0.946	0.982	1.000	0.977	0.982	0.972	0.989	0.997	0.652
13	0.987	0.999	0.991	0.998	0.987	0.866	0.973	0.972	0.987	0.989	0.980	0.977	1.000	0.999	0.999	0.997	0.989	0.728
14	0.985	0.999	0.993	0.999	0.991	0.856	0.969	0.968	0.990	0.987	0.984	0.982	0.999	1.000	0.999	0.998	0.992	0.726
15	0.992	0.997	0.986	0.998	0.983	0.882	0.981	0.980	0.981	0.994	0.982	0.972	0.999	0.999	1.000	0.994	0.986	0.755
16	0.976	0.999	0.998	0.997	0.996	0.829	0.954	0.953	0.996	0.977	0.981	0.989	0.997	0.998	0.994	1.000	0.997	0.693
17	0.964	0.994	0.997	0.993	0.999	0.799	0.939	0.937	0.997	0.966	0.987	0.997	0.989	0.992	0.986	0.997	1.000	0.685
18	0.813	0.725	0.650	0.747	0.670	0.914	0.855	0.851	0.635	0.815	0.784	0.652	0.728	0.726	0.755	0.693	0.685	1.000

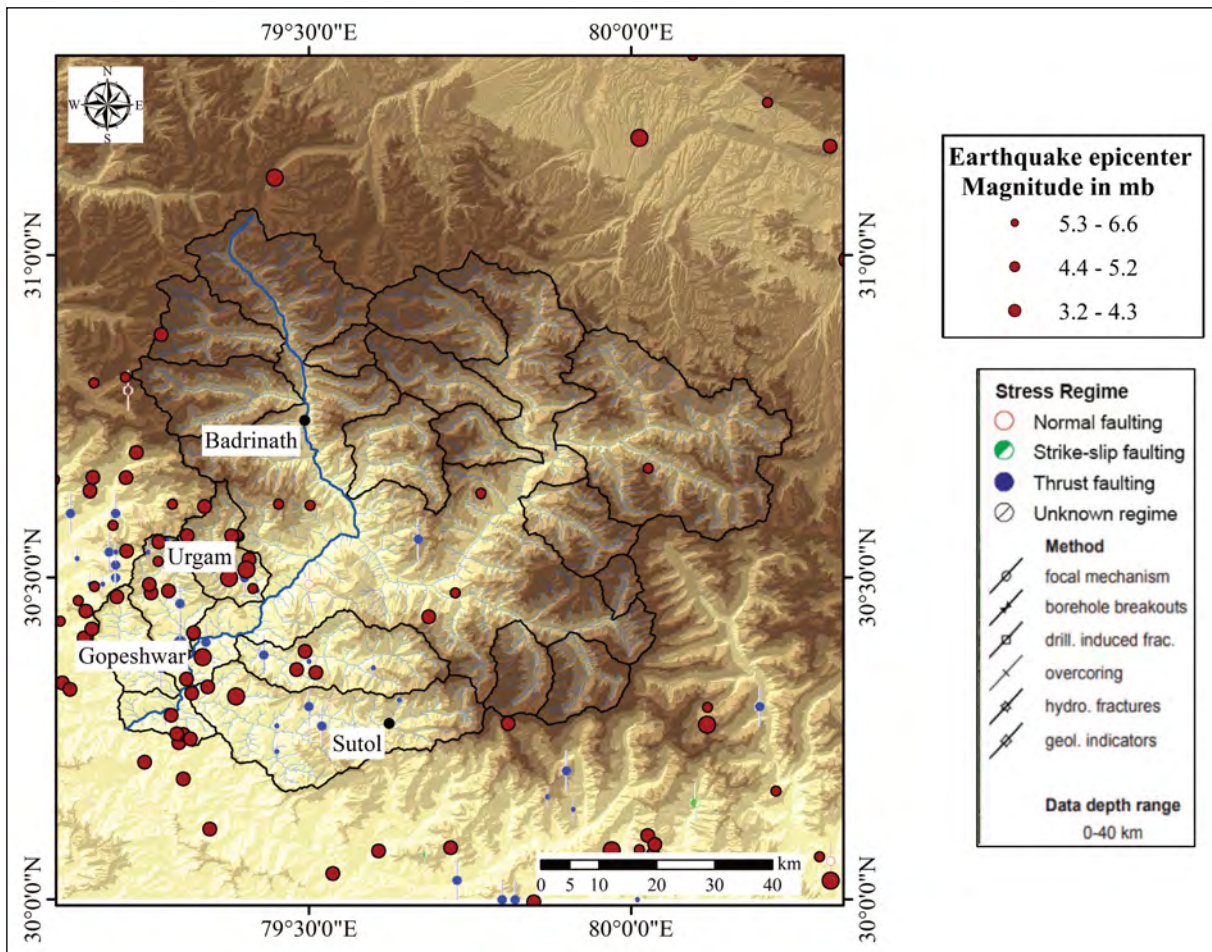


Figure 5- World stress map of the study area with 18 watersheds and IAT classes marked as C1-4.

exists (Figure 5). GPS time series data acquired near Gopeshwar disclose a high rate of displacement of 24.72 mm y^{-1} (Sharma et al., 2018). Major strike-slip and thrusts are located between the MCT I and MCT II (Figure 5).

Conclusions

For morphometric assessment of active tectonic, geomorphic indices that can be easily produced from the high resolution DEMs presently available through satellite observation are used. Such indices can be used to analyze data that covers a broader area quickly. We study 18 sub-watersheds of the upper Alaknanda basin in this work. Sub-watersheds 16 and 17 at the southern portion where NW-SE tectonic lineaments cross MCT II and where several lineaments exist in multi-direction, tectonic activity is very strong. High-to-moderate activeness was observed at the places on

the periphery of the very high activity zone. This is also confirmed from cluster analysis.

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Effect of calculated dry rock bulk modulus on uncertainty in compressional wave velocities derived from Gassmann fluid substitution theory and fluid mixing law in a CO₂ sequestration case

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Research Article

Keywords:

Fluid Mixing
Laws, Gassmann
Fluid Substitution,
Compressional
Wave Velocity, CO₂
Sequestration, Bulk
Modulus.

ABSTRACT

This study examines the effect of variations in dry rock bulk modulus (K_{dry}) on compressional wave velocity (V_p) using Gassmann fluid substitution theory and various fluid mixing laws in the context of CO₂ sequestration in the Utsira formation at the Sleipner Field. Significant changes in V_p , exceeding 600 m/s, were observed, particularly when applying the Gassmann-Wood law, which demonstrated heightened sensitivity to saturation variations. In contrast, the Gassmann-Brie approach, utilizing an empirically selected constant, produced more stable and reliable results. This study emphasizes that relying solely on the Gassmann-Wood equation for K_{dry} calculations may result in inaccurate predictions, particularly for CO₂-saturated formations. To mitigate this, laboratory measurements of K_{dry} are recommended to enhance the reliability of predictions. Furthermore, initial saturation conditions critically affect the V_p calculations, underlining the importance of selecting an appropriate mixing law to ensure accurate assessments during CO₂ injection and storage.

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1. Introduction

Since the industrial revolution, the effect of greenhouse gases in the atmosphere has dramatically increased. Taking its effect on the environment and human health into account, CO₂ is obviously the most harmful greenhouse gas with a high proportion (64%) in the atmosphere (Bachu, 2000). The main reason for this situation is the consumption of fossil fuels as a primary energy resource all around the world (Omeregbe et al., 2020). Only in 2019, the total emission of CO₂ by the U.S. refining industry is nearly 166×10^7 tonnes (EPA, 2020). In order to reduce the concentration of CO₂ gases in the atmosphere, carbon capture and storage (CCS) projects have been widely implemented (Yao et al., 2018; Metz et al., 2005).

Sleipner project is the first industrial scale carbon capture and storage project that has been approved and successfully operated in the North Sea since 1996 (Chadwick et al., 2014; Eliasson et al., 2014; Ghosh et al., 2015; Eliasson and Romdhane et al., 2017). Understanding the structural model is critical to increase success rate of CO₂ sequestration. Overall, 16 Mt CO₂ has been injected and stored into the Utsira Sand successfully by 2017 (Park et al., 2013; Kvamme and Aromada, 2018) and 17.8 Mt by 2019 (Ahmadinia and Shariatipour, 2020). Project target of 20 Mt CO₂ injection (Chadwick et al., 2004).

Geophysical techniques, including AVO (Amplitude versus offset) analysis, are powerful tools to estimate CO₂ saturations in the target formation

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(Roverud, 2017). After CO₂ injection, initial condition and final condition can exhibit different elastic properties. So, the injection zone is selected to include highly porous and permeable sand units which are sealed by impermeable cap rocks, like shale or limestone (Wisman, 2012). Porous media includes chemical substance based pore fluids. The security of a CO₂ injection process is indirectly influenced by the estimation of brine and CO₂ saturations in the reservoir while in-situ fluid mixture properties are changing during the injection (Mavko et al., 2009). Therefore, for monitoring the leakage risks, modelling the fluid mixture properties and the rock elastic properties at initial condition are two critical stages. However, modelling the fluid mixture is almost impossible if one of the constituents is CO₂ (Yam and Schmitt, 2011).

The temperature and pressure of the target formation are critical because the compressibility and density characteristics of CO₂ are totally changed if the pressure and temperature of the formation are higher than the critical pressure (~7.4 MPa) and critical temperature (~31 C) (Yam and Schmitt, 2011). In this situation, the phase of CO₂ transforms from gas to liquid or supercritical state. Yam and Schmitt (2011) also showed that the phase of CO₂ changes from gas to liquid instantly around the critical values. Seismic signatures of the target rock are widely varied corresponding to the different phases of injected CO₂ (Njiekak et al., 2013). Thus, identifying the phase of CO₂ has a significant effect on the results.

For a CO₂ injection case, the properties of the final fluid mixture are completely different from those of each constituent. (Vera, 2012; Khalid and Ghazi, 2013; Maurya and Singh, 2019). Recommended mixing models in the literature can be listed as Gassmann-Wood (Gassmann, 1951), Gassmann-Brie (Brie et al., 1995), Gassmann-Hill (Hill, 1963) and Voigt averaging (Voight, 1910). These models provide a theoretical way to estimate the bulk density and Bulk modulus of CO₂-fluid mixture, which is essential to reveal the seismic signature of saturated rock (Wisman, 2012; Njiekak et al., 2013; Ivandic et al., 2018; Vasco et al., 2019). In fact, mixing laws are not suitable for every formation. Defining an appropriate mixing law for target formation is quite significant for reliable

V_p responses on final (Mavko et al., 2009; Roverud, 2017). Gassmann-Wood mixing law is commonly used in the literature to model CO₂ and brine mixture. This approach has been applied in several studies including those by Si et al. (2016), Zhang et al. (2017), Papageorgiou and Chapman (2017). Falcon-Suarez et al. (2018) specifically applied the Wood's law, which is the case of Gassmann equation, to Utsira formation.

Gassmann-Hill approximation is another traditional model used for fluid mixing scenarios. This approach is suggested in the literature if patchy saturation is assumed. Similar to Voigt averaging, mass and heat transfer between the fluids are assumed. Gassmann-Hill approximation is used by many authors and to some cases (Pabst et al., 2015; Eid et al., 2015).

The perspective of Gassmann Brie techniques is a bit different from the other methods because of the 'e' empirical constant in the equation. 'e' constant is a variable that can be defined considering geological details of the porous formation. (Brie et al., 1995; Papageorgiou et al., 2016; Si et al., 2016).

1.1. Geological Settings of Sleipner Field

To increase the reliability of the results, rock and fluid properties are applied to the models in Utsira formation of Sleipner field where is almost the best-known injection site in the world.

The injection location within Utsira Formation is approximately 6 km away from Sleipner Vest field as indicated in Figure 1 (Karstens et al., 2017). Utsira sand is lying between Hordland (bottom) and Nordland (top). The Utsira sand deposition occurred during the Mio-Pliocene period, spanning a significant distance of nearly 400 km in a south-north trending manner. Formation surface is generally smooth, but it exhibits some small undulations, valleys and domes within the depth range of 550 m to 1500 m depths. Formation is dipping mostly towards the south and thicknesses are varying along 200 km from the north (200 m) to the south (300 m). The formation contains about 12 m height domes, which are suitable for CO₂ injection. Within the Utsira Formation, there are mud diapirs accompanied by thrust faults that dip towards the base of the formation (Chadwick et al., 2004; Dupuy et al., 2016).

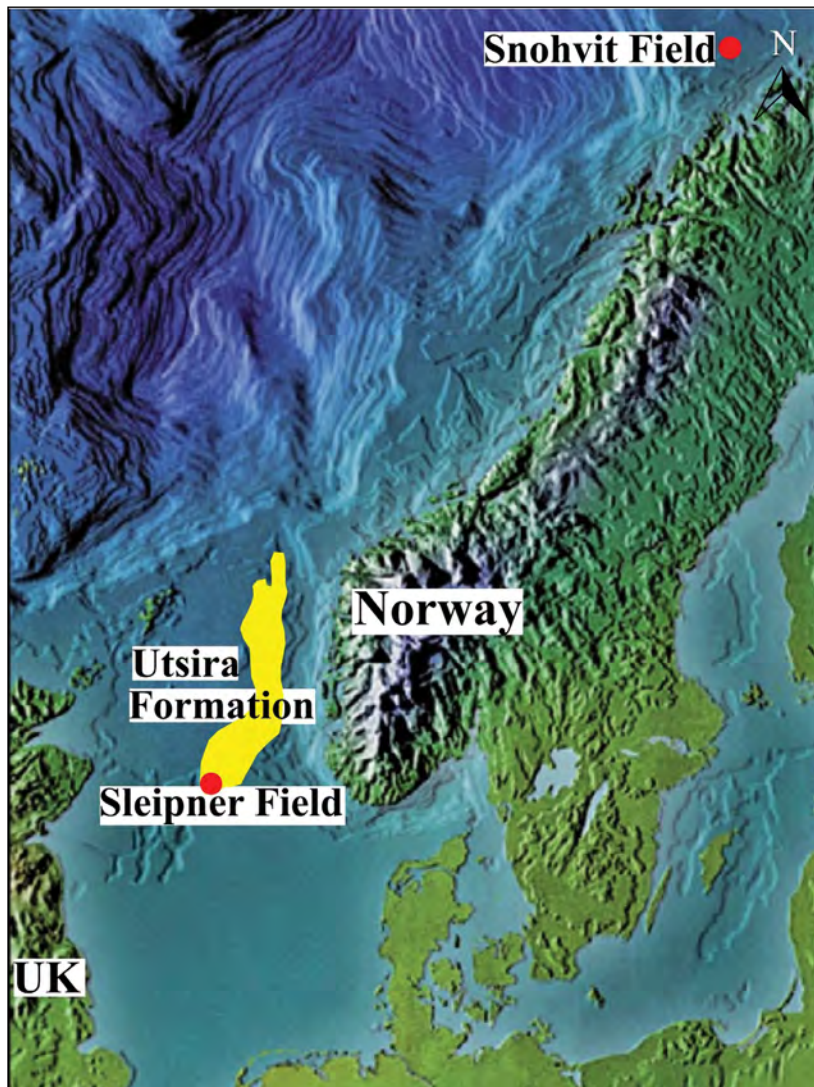


Figure 1- Location of Sleipner field around Utsira formation (Modified from Labat et al., 2012).

Regarding to the caprock properties, thicknesses are around a few hundred meters and capillary pressure is ranging from 2 to 5.5 MPa that is capable to seal a few hundreds of meters thick CO₂ column. The estimated porosity of the caprock is approximately 0.11% which means that it is managed to store 16 billion tons CO₂ considering less than 12 km migration way (Chadwick et al., 2004; Karstens et al., 2017).

Utsira formation has mostly homogenous sand with a few meters' thick shale layers (Dupuy et al., 2016). The Utsira Sand reflector is typically observed at depths ranging between 800 and 1000 meters. Porosity refers to the fraction of pore spaces within the rock, and these values indicate a significant volume

of available pore space for fluid storage, including CO₂. The estimated porosity is between 30% and 40%. Permeability of the sand is ranging from 1 to 5 Darcy. These values clearly indicate the great storage potential of Utsira sand (Dupuy et al., 2016; Karstens et al., 2017).

Utsira formation also includes poorly cemented, fine-to-medium grain size, mostly moderate sorted 1-6 m thick mudstone layers with 10 to 40 meters overall thickness. Laboratory studies show that Utsira sand consist of multiple constituents including quartz grains, K-feldspars, calcite, plagioclase, shell fragments, calcite, albite, aragonite, muscovite and biotite (Dupuy et al., 2016; Karstens et al., 2017).

1.2. Gassmann Fluid Substitution

Gassmann's fluid substitution theory is applied to estimate P (V_p) and S (V_s) wave velocities of a saturated rock using elastic properties of fluid and rock matrix (Gassmann, 1951). Gassmann's equations use bulk modulus of dry rock that are derived from initial case parameters or sometimes from laboratory measurements.

To estimate dry rock Bulk modulus, the equation given in the Gassmann's theory is expressed as;

$$K_{dry} = \frac{K_{sat\_init} \left(\frac{\phi K_m}{K_{fl\_init}} + 1 - \phi \right) - K_m}{\frac{\phi K_m}{K_{fl\_init}} + \frac{K_{sat\_init}}{K_m} - 1 - \phi} \quad (1)$$

In the equation (1), K_m is Bulk modulus of rock matrix, ρ_{init} is initial bulk density, ϕ is porosity, K_{fl_init} initial Bulk modulus of fluid mixture and V_{p_init} is P wave velocity in initial state (Russell et al., 2001; Bacon et al., 2003).

One of the critical parameter is Bulk modulus of the dry rock (K_{dry}) here because this is derived from the is initially predicted values. In the next step, calculated K_{dry} is used to estimate final saturated rock. Bulk modulus which is essential for traditional compressional wave velocity theory (Telford et al., 1990).

Bulk modulus of the saturated rock in final case can be calculated using equation (2)

$$K_{sat\_fin} = K_{dry} + \frac{(1 - \frac{K_{dry}}{K_m})^2}{\frac{\phi}{K_{fl\_fin}} + \frac{1 - \phi}{K_m} - \frac{K_{dry}}{K_m^2}} \quad (2)$$

One can see in the equations above (Bacon et al., 2003), Bulk modulus of the initial and final fluid mixture are the deterministic inputs that can be estimated from a range of mixing laws.

1.3. Fluid Mixing Laws

Each mixing laws are not suitable for every formation. Therefore, assuming saturation behaviour of the formation is the key parameter to prefer appropriate mixing law. Some mixing laws are based a consideration that the fluid is flowing inside the pore spaces homogenously (uniform saturation), while some of them assume heterogeneous distribution

which is also called patchy saturation (Gassmann, 1951; Mavko et al., 2009; Si et al., 2016). Uniform saturation is expressed if formation reaches equilibrium (relaxed state). In other word, formation fluids are homogenously distributed into the pore spaces of the rock due to a long geological time. This situation is seen mostly in hydrocarbon cases. On the other hand, most of the CO₂ sequestration cases have a different perspective. If any external process, such as CO₂ injection, is applied to the formation, saturation model is frequently assumed as heterogeneous (unrelaxed state formation).

1.3.1. Gassmann-Wood

First essential assumption to consider that Wood's law is formation is saturated homogenously and constituents must remain unrelaxed or frozen state. In other word, any mass or heat transfer between fluids must be neglected (Khalid and Ghazi, 2013). Dry rock Bulk modulus of the fluid mixture are calculated using

$$\frac{1}{K_f} = \sum_i^N \frac{S_i}{K_i} \quad (3)$$

In the equation (3), S_i and K_i are volume fraction, elastic modulus and density of i^{th} fluid respectively (Wood, 1955; Mavko et al., 2009).

1.3.2. Voigt Averaging

This method can also be called Hill's equation or arithmetic average in Si et al. (2016). On the other hand, this equation was called Voigt average in Hill (1963). Bulk modulus of fluid mixture can be calculated using equation (4)

$$\frac{1}{K_f} = \sum_i^N S_i K_i \quad (4)$$

Voigt average has been suggested for heterogeneously saturated formations. In contrast to the Wood's law, due to the patchy saturation model, it is assumed that heat and mass manage to be transferred between fluids. Voigt averaging are applied to CO₂ sequestration cases in Si et al. (2016). Voigt average is also easily applicable to mix three or more fluid constituents.

1.3.3. Gassmann-Brie

Success of this approach directly depends on e empirical constant which is generally between 2 and

5 (Papageorgiou et al., 2016). In Gassmann-Brie approach (Brie et al., 1995), Bulk modulus of fluid mixture (K_{fl}) is derived from equation (5)

$$K_{fl} = (K_w - K_{CO2})S_w^e + K_{CO2} \quad (5)$$

Here, K_w and K_{CO2} are the Bulk modulus of brine and supercritical state CO_2 . S_w and S_{CO2} are the saturation of water and supercritical state CO_2 respectively. Recommended e empirical constants associated with porosity and permeability are given in Si et al. (2016) (Table 3).

However, Papageorgiou et al. (2016) suggested series of equations to calculate e constant in terms of only saturations and capillary pressure (P_{cap}) which is given in equation (6)

$$e = \frac{\log\left(\frac{S_w}{S_w + \alpha(1-S_w)}\right)}{\log(S_w)} \quad (6)$$

And α constant can be calculated using equation (7)

$$\alpha = \frac{K_w - S_w(1-S_w)P_{cap}(S_w)}{K_g - S_w(1-S_w)P_{cap}(S_w)} \quad (7)$$

Here, P_{cap} is the capillary pressure. Capillary pressure between the brine filled formation and injected CO_2 is defined by Chadwick et al. (2005)

$$P_{cap} = 810.35(1 - S_g)^{-0.948} \quad (8)$$

Equation (8) is suggested only if gas in the fluid mixture is CO_2 instead of hydrocarbon

1.3.4. Gassmann-Hill

Gassmann-Hill mixing law (Hill, 1963) takes shear modulus of the patchy saturated rock (μ) into account. So, bulk modulus of fluid mixture can be calculated using equation (9)

$$K_{fl} = \left(\frac{S_w}{K_w + \frac{4}{3}\mu} + \frac{S_{CO2}}{K_{CO2} + \frac{4}{3}\mu} \right)^{-1} - \frac{4}{3}\mu \quad (9)$$

Here, K_{CO2} and S_{CO2} are bulk modulus and saturation of CO_2 respectively.

2. Workflow

General workflow is illustrated in a flowchart given in Figure 2 that shows 2 main stages calculating K_{fl} and approximating V_p . In the first calculations, Fluid mixing laws are applied with regards to the range of scenarios from fully brine to fully CO_2 saturated Utsira sand. In order to estimate V_p by Gassmann fluid substitution equation, needed K_{dry} parameter is both calculated from XRD results

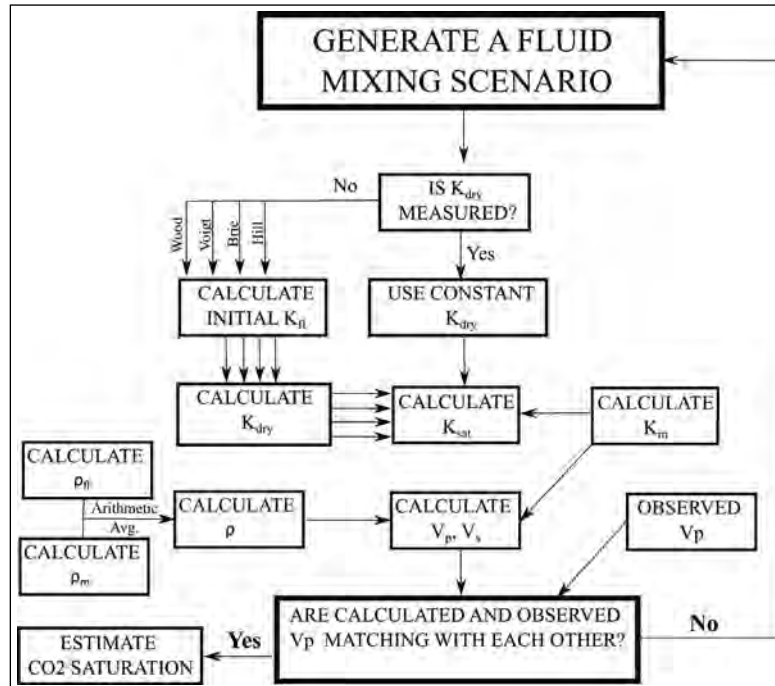


Figure 2- Flowchart of the study. K_m , K_{dry} and K_{sat} represent Bulk modulus of matrix, dry and saturated rock respectively. Also, ρ_f , ρ_m and ρ are densities of fluid mixture, matrix and saturated rock respectively. V_p and V_s are the seismic velocities.

(Chadwick et al., 2005), given in the Table 1, and directly taken from measurements (Dupuy et al., 2017). Calculations are done in MATLAB (Matrix Laboratory) software of Mathworks®. It helps to interpret the results comparatively. Calculated V_p values are checked if it matches with each other and the V_p suggested by Chadwick et al. (2005) which is called ‘observed’ data hereinafter. It helps to analyse the suitability of the mixing laws and observing sensitivity of K_{dry} calculation on final V_p .

3. Field Data and Model Parameters

In this study, observed rock elastic properties and seismic characteristics of Utsira sand within Sleipner CO₂ injection site is used as a field data (Table 2 and Table 3).

As it was expressed, temperature and pressure effecting on the formation are determinant factors to estimate phase of injected CO₂. Critical temperature and pressure of CO₂ are given as 31.1°C and 7.39 MPa respectively in Ravazzoli and Gomez (2014). Temperature and pressure of Utsira sand is measured as nearly 116 °C and 26.2 MPa respectively (Ahmed et al., 2016). Thus, injected CO₂ in this formation condition is turned into liquid phase (Ravazzoli and Gomez, 2014).

On the other hand, sand and shale matrix are measured using XRD analyse from core samples

(Chadwick et al., 2004) and given in Table 2. Using matrix constituents and its volumes, Bulk modulus and density of rock matrix are also derived from Voigt-Reuss-Hill averaging method in this study (Table 3). Elastic properties of the minerals are taken from Bacon et al. (2003) and Pabst et al. (2015).

Relaxation state and saturation model of a formation are dependant to the time needed for equilibrium. In hydrocarbon case, gas or oil inside the formation is distributed homogenously (uniformly) during a long geological time. This is called relaxed state. Contrarily, injected CO₂ cannot be distributed homogenously due to the limited time (Eid et al., 2015; Zhang et al., 2017). This is called unrelaxed state. In this situation, patchy saturation is considered as saturation model of the formation. Defining the saturation model is essential to choose the suitable mixing law. In this study, relaxed and unrelaxed state is regarded to illustrate the differences between results. Observed values from Chadwick et al. (2004) and Dupuy et al. (2017) indicate that full of brine water saturated Utsira sand patch has 2050 m/sec and 636 m/sec P and S wave velocities respectively. Moreover, Bulk density of sand patch is measured as 2.078 g/cc (Table 3). Also, Chadwick et al. (2004) suggested that P wave velocity reduces to 1420 m/sec in the case of whole CO₂ saturated sand that is consistent with

Table 1- Mineral constituents of Utsira sand and Utsira shale (Chadwick et al., 2004).

	Quartz	Calcite	K-feldspar	Albite	Aragonite	Smectite	Kaolinite	Mica	Other
Utsira Sand	75%	3%	13%	3%	3%	-	-	3%	
Utsira Shale	30%	3%	5%	2%	-	3%	14%	30%	11%

Table 2- Utsira formation rock and fluid properties. Porosity (ϕ), Shear modulus (μ), Bulk modulus of rock matrix (K_m), P wave velocity (V_p), S wave velocity (V_s), bulk density of rock (ρ), Bulk modulus of fluids (K_f), density of fluids (ρ_f) and saturation of fluids (S) (Chadwick et al., 2005; Dupuy et al., 2017; Roverud, 2017; Falcon-Suarez, 2018).

		ϕ [%]	μ [GPa]	K_m [GPa]	V_p [m/sec]	V_s [m/sec]	ρ [g/cc]
Formation	Utsira Sand	37.5	0.84	39.29	2050	636	2.078
	Utsira Shale	13	2.4	22.6	2450	1050	2.2
		K_f [GPa]		ρ_f [g/cc]		S [%]	
Fluids	Water	2.3		1.03		100	
	CO₂	0.075		0.7		0	

Table 3- Calculated parameters used to calculate final P wave velocities. While final saturations are stayed the same, initial saturations of fluids are varied to observe the effect of mixing laws on final P wave velocities.

S_{w_init}	S_{g_init}	S_{w_fin}	S_{g_fin}	Methd	K_{fl_init}	K_{fl_fin}	K_{sat_init}	K_{dry}	K_{sat_fin}	r_m	r_{fin}	V_{p_fin}
Initial water saturation	Initial CO ₂ saturation	Final water saturation	Final gas saturation	Mixing Law	Bulk modulus of fluid mixture in initial case	Bulk modulus of fluid mixture in final case	Bulk modulus of saturated rock in initial case	Bulk modulus of dry rock	Bulk modulus of saturated rock in final case	Density of matrix	Density of the rock in final case	P wave velocity in final case
[]	[]	[]	[]	[]	[Gpa]	[Gpa]	[Gpa]	[Gpa]	[Gpa]	[g/cc]	[g/cc]	[km/sec]
1	0	0.00	1.00	Wood	2.3	0.075	7.61	2.72	2.89	2.71	1.95	1.43
				Voigt	2.3			2.72	2.89			1.43
				Brie	2.3			2.72	2.89			1.43
				Hill	2.3			2.72	2.89			1.43
0.998	0.002	0.00	1.00	Wood	2.1712	0.075	7.61	3.05	3.22	2.71	1.95	1.49
				Voigt	2.2955			2.73	2.91			1.44
				Brie	2.2956			2.73	2.91			1.44
				Hill	2.2873			2.75	2.93			1.44
0.99	0.01	0.00	1.00	Wood	1.7738	0.075	7.61	4.03	4.19	2.71	1.96	1.65
				Voigt	2.2777			2.78	2.95			1.44
				Brie	2.2782			2.78	2.95			1.44
				Hill	2.2375			2.88	3.06			1.46
0.95	0.05	0.00	1.00	Wood	0.9262	0.075	7.61	5.88	6.02	2.72	1.96	1.91
				Voigt	2.1887			3.01	3.18			1.48
				Brie	2.1909			3.00	3.17			1.48
				Hill	2.0087			3.46	3.63			1.56
0.9	0.1	0.00	1.00	Wood	0.5798	0.075	7.61	6.56	6.70	2.73	1.97	1.99
				Voigt	2.0775			3.29	3.46			1.53
				Brie	2.0817			3.28	3.45			1.52
				Hill	1.7632			4.05	4.21			1.65
0.8	0.2	0.00	1.00	Wood	0.3317	0.075	7.61	7.02	7.15	2.75	1.98	2.05
				Voigt	1.855			3.83	4.00			1.61
				Brie	1.8635			3.81	3.98			1.60
				Hill	1.372			4.94	5.09			1.77

Dupuy et al. (2017). This expression is considered to define ‘expected values’ that must be matched with the model results with an acceptable uncertainty range. However, formation saturation associated with these velocities has some uncertainties. Measuring or estimating exact P wave velocities of fully CO₂ saturated sand is not possible in practice. In any case, these published V_p values are defined as the closest values to the V_p of fully CO₂ and water saturated sand case (Figure 3). Thus, these values are used to test the theoretical approaches in this study.

4. Result and Discussion

The main objective in the study is to understand the effect of Bulk modulus of dry rock on V_p . Therefore, initial results focussed on the Bulk modulus of fluid mixture behaviour depending on the fluid saturation variations. As it can be seen in the Figure 4, Bulk modulus of fluid mixture within the Utsira sand is decreasing exponentially from nearly 2.2 GPa to 0.1 GPa with ascending the CO₂ saturations. This module forms the Bulk modulus of the saturated Utsira sand proportionally and linearly between 2.7 and 7.6 GPa in Figure 5. Relevant graph shows also V_p is slowing

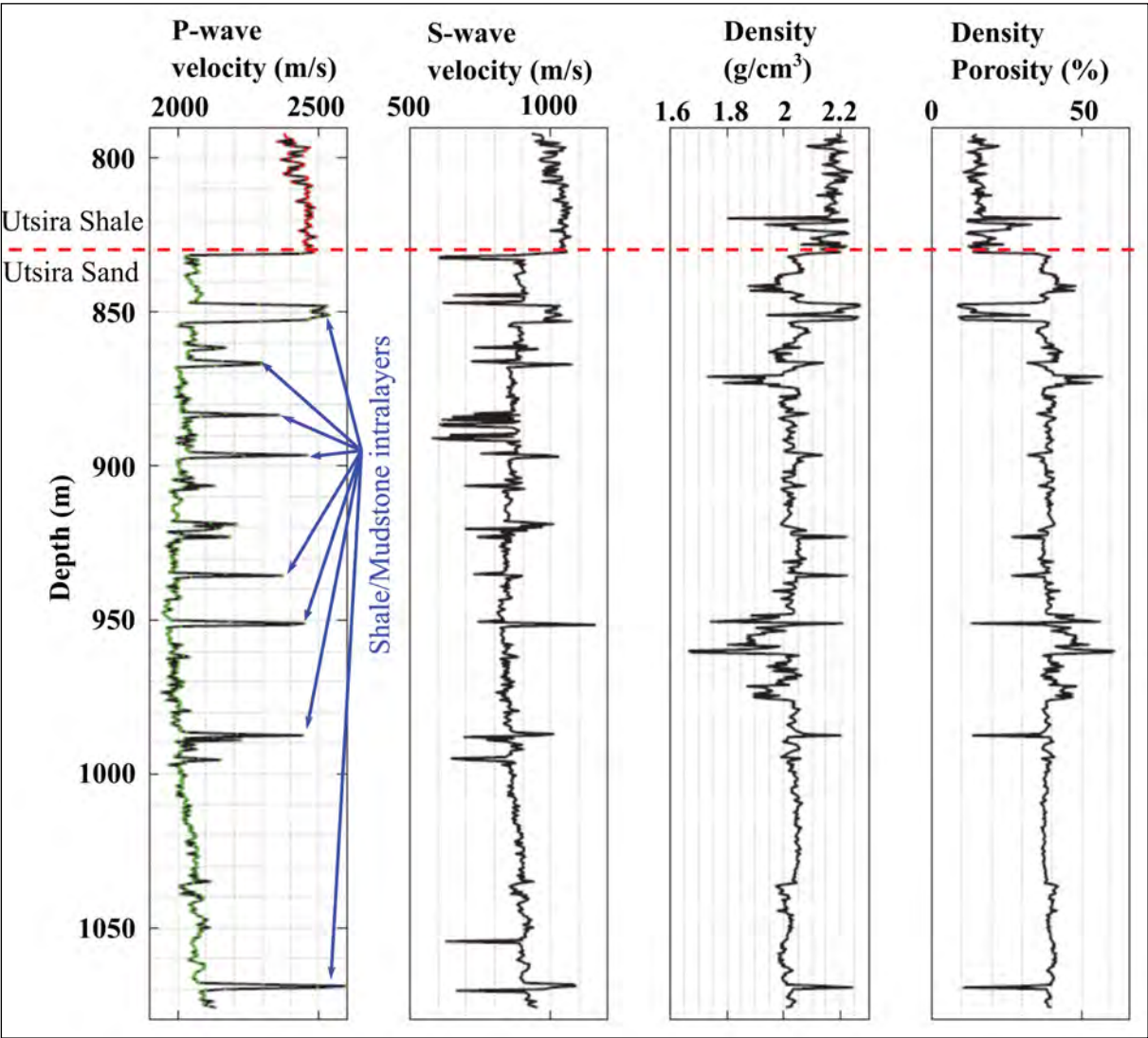


Figure 3- Seismic velocities, density and porosity logs of Utsira formation between 790 m and 1080 m depth (Modified from Dupuy et al., 2017).

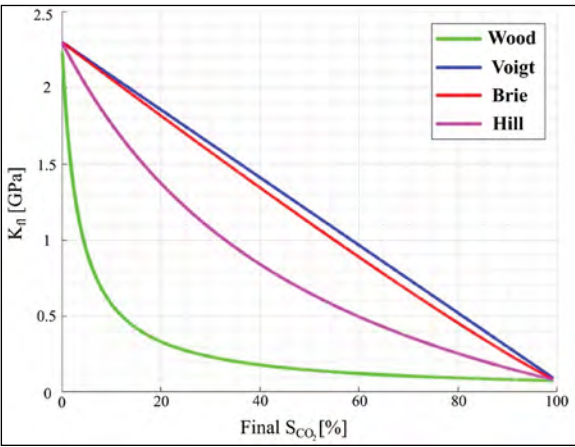


Figure 4- Changing of Bulk modulus of fluid mixture within the pore spaces of the rock as CO₂ saturation increases.

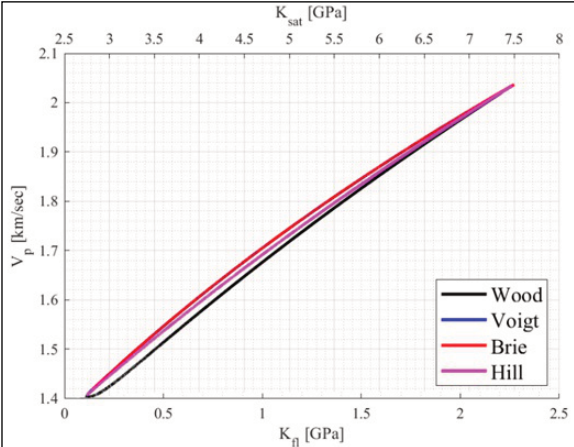


Figure 5- Bulk modulus of the fluid within the rock (K_f) versus P wave velocity (V_p) of the rock graph.

down from nearly 2.05 km/sec to 1.4 km/sec if Utsira sand CO_2 saturation increases during the injection.

In order to analyse the behaviour of V_p in detail during a CO_2 injection scenario, each mixing law is applied separately for calculating Bulk modulus of fluid mixture (Figure 6). Particularly, Brie mixing law is applied multiple time because its results are being changed dramatically by e empirical constant. As it is illustrated in Figure 7, 30% CO_2 saturated Utsira sand gives a range of Bulk modulus of fluid mixture between 0.1 GPa and 1.65 GPa regarding to e empirical constant changing from 10 to 1. These range is relevant to 1.4 km/sec and 1.85 km/sec in the same formation respectively (Figure 5). However, suggested maximum value of e empirical constant is lesser than 5 (Papageorgiou and Chapman, 2017) which point out maximum 1 GPa fluid mixture Bulk modulus around 50% CO_2 saturation scenario (Figure 7). Therefore, 3 different scenarios for Brie mixing law are taken into account in Figure 6 to analyse the results of the mixing

laws comparatively. Plots in Figure 6 is also derived from different initial CO_2 saturation estimation (0%, 1%, 5% and 20% respectively). Results clearly show that initial fluid saturation estimations dominate the estimated V_p for a CO_2 sequestration scenario. While fully brine and fully CO_2 saturated Utsira sand indicates the same V_p (2.05 km/sec and 1.45 km/sec respectively) in Figure 6a, finally full of CO_2 saturated Utsira sand scenario shows a wide range of V_p (between 2.05 km/sec and 1.6 km/sec) for an initially 20% CO_2 saturated Utsira sand in Figure 6d. Similarly, the same range is seen in finally full of brine saturated Utsira sand case. This indicates a question mark on reliability of the results derived from different fluid mixing laws. In Figure 6d, for dully CO_2 saturated Utsira sand, while almost 2.03 km/sec V_p is read from Wood's curve, 1.6 km/sec is seen on Voigt average and Brie's law ($e=1$). Practically, the mixing models showing exponential behaviour in the graph plot response CO_2 saturation changes more sensitively than the ones

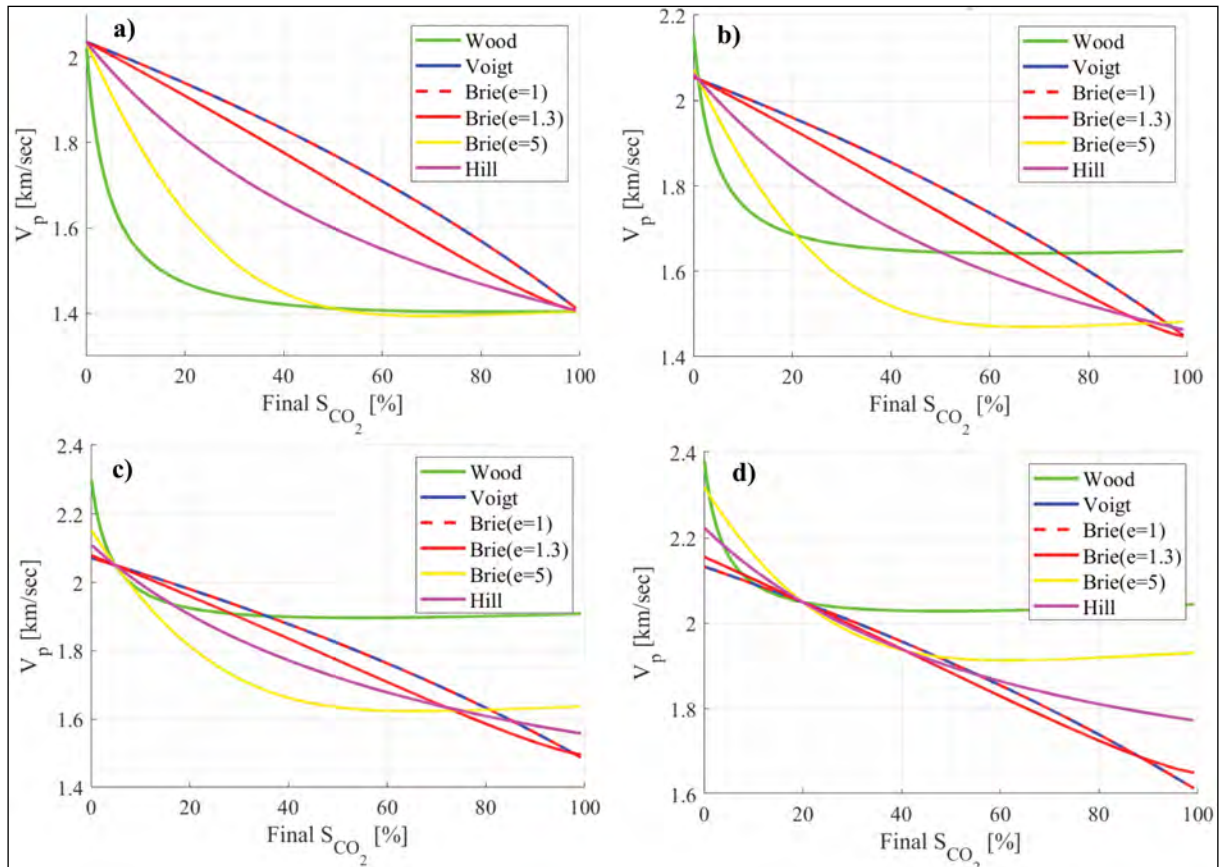


Figure 6- P wave velocity versus final CO_2 saturation graphs, a) Bulk modulus of dry rock is calculated using different mixing laws. Initial CO_2 saturations are entered 0%, b) 1%, c) 5%, d) 20%.

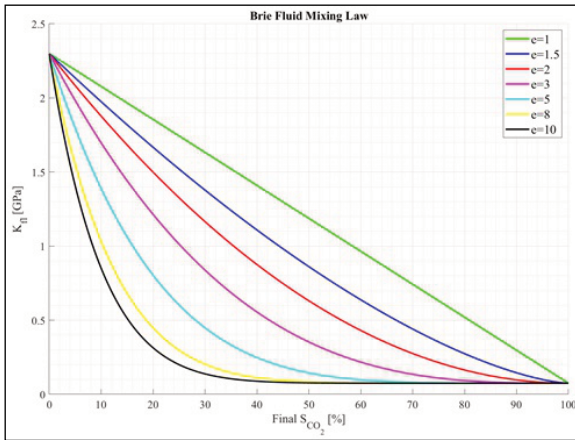


Figure 7- Change of bulk modulus of fluid mixture with increasing final CO₂ saturation of the rock. Bulk modulus of fluid mixture is derived from Brie mixing law with a range of e empirical constants.

that show linear behaviour. Similar V_p range is also seen in the Brie mixing laws derived from $e=1$ and $e=5$. Curve behaviours are obviously coming from the mathematical equation of Bulk modulus of fluid mixture of each mixing law. Overall results reveal the significance of defining accurate initial saturations if dry rock Bulk modulus is not measured in laboratory tests.

Initial CO₂ saturation dependency of dry rock Bulk modulus is also modelled in Figure 9. Final CO₂ saturation is set as 100% for an Utsira sand sample while initial saturations are varied. It is clearly seen that Wood's law is quite sensitive to the CO₂ saturation changes if its saturations assumed to be less than 20%. Considering initial CO₂ saturation between 0% and

20%, dry Utsira sand Bulk modulus is ranged from 2.7 GPa to 7 GPa. On the other hand, if initial CO₂ saturation is higher than these critical values (around 20%), dry Utsira sand Bulk modulus is not changing remarkably. Consequently, attention must be paid if Wood's law is preferred for a scenario CO₂ injected into initially brine saturated formation.

Sensitivity of each mixing law is also seen in Table 3 that includes Bulk modulus of fluid mixture, Bulk modulus of dry Utsira sand, Bulk modulus of saturated Utsira sand and P wave velocity respectively. Only initial CO₂ saturation assumption is ranged to observe the sensitivity of mixing laws. While initial CO₂ saturation is increasing from 0% to 20%, difference between Wood's response and the other mixing law response are increasing exponentially. Differences in Wood's law from initially Bulk modulus of fluid mixture to V_p on final is folding with each variable derivation. On the other hand, linear behaviour mixing laws response similarly ($V_p \approx 1.6$ km/sec), such as Voigt average and Brie's law ($e=1$).

It is also possible to measure K_{dry} in the laboratory. In this point, in order to dig in the K_{dry} dependency of the mixing laws, constant and measured K_{dry} are used in the equations to analyse the estimated V_p velocities comparatively. Regarding to the observed K_{dry} (2,56 GPa) (Dupuy et al., 2017), V_p versus CO₂ saturation graphs plotted in Figure 8a and 8b that clearly show the limits and behaviour of the V_p are not dependant to the initial saturation assumptions. However,

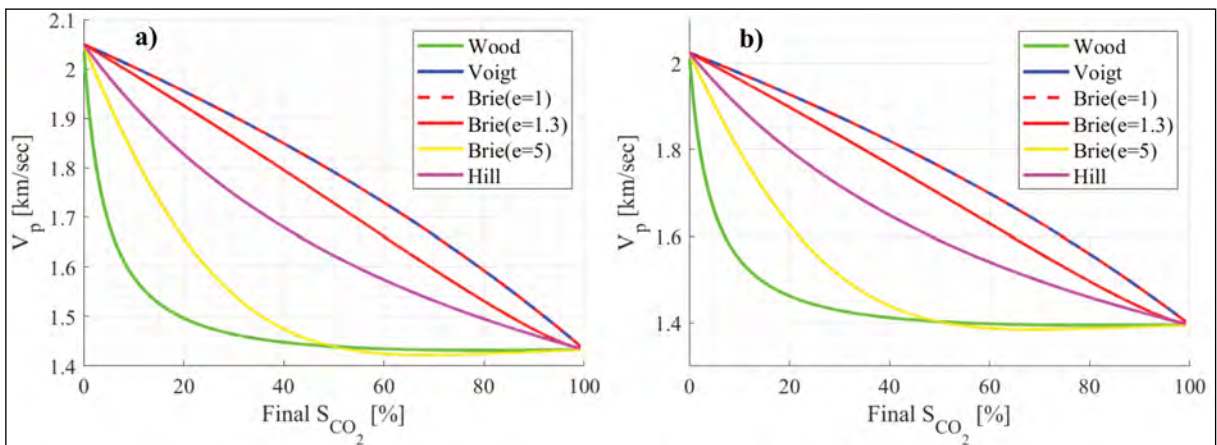


Figure 8- P wave velocity versus final CO₂ saturation graphs, a) Measured Bulk modulus of dry rock (2.56 GPa) (Dupuy et al., 2017) is used for all mixing laws. Initial water saturations are 100%, b) 80%.

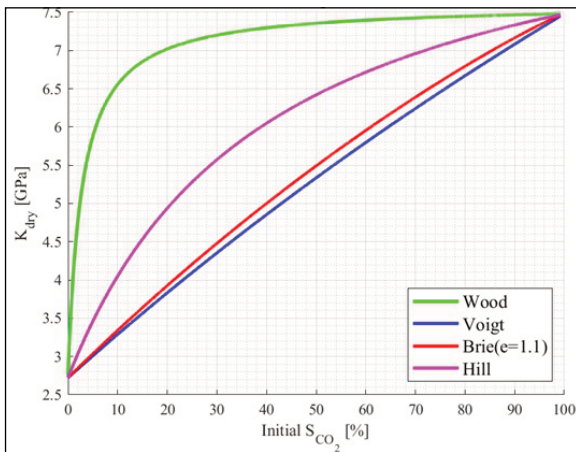


Figure 9- Initial saturation dependency of the K_{dry} values derived from finally full of CO_2 saturated Utsira sand sample.

unrecognizable descend can be seen in Figure 8b because of diminished average density of the fluid mixture. Furthermore, V_p of the fully brine and CO_2 saturated sand is almost 2.05 km/sec and 1.4 km/sec respectively in every mixing model. These values are matching not only with each other but also with the suggested results for Utsira sand case in Chadwick et al. (2004) and Dupuy et al. (2017) (Figure 3).

V_p curve characteristics differences between the results derived from observed K_{dry} (Figure 8) and calculated K_{dry} (Figure 6) are dramatically increasing with increasing CO_2 saturation of the sand. For a scenario of fully CO_2 saturated Utsira sand which is initially 20% CO_2 saturated, approximately 2.05 km/sec V_p is estimated from Wood's law if dry Utsira sand Bulk modulus is calculated using Gassmann fluid substitution theory. 2.05 km/sec indicates sand is still fully brine saturated, considering the suggested V_p for a fully CO_2 saturated Utsira sand (Figure 6d). Consequently, these misleading results show that using calculated dry rock bulk modulus in a CO_2 leakage monitoring study is risky and unreliable. Not only Wood's law but also even Voigt average and Brie's law ($e=1$) cannot give the reliable results ($V_p \approx 1.6$ km/sec). On the other hand, V_p is estimated 1.4 km/sec (Figure 8b) from the observed and constant dry Utsira sand bulk modulus that is matching with the literature (Chadwick et al., 2004; Dupuy et al., 2017). Saturation behaviour of a formation can be uniform or patchy which mean the seismic signature of the formation is changing suddenly or in time. In other

words, V_p - S_{CO_2} graph gives a clue about the unrelaxed state (suitable for patchy saturation) or relaxed state (suitable for uniform saturation) of the formation.

Exponential response of V_p indicates Gassmann Wood or Brie mixing law ($e>3$) as a suitable model. Otherwise, Voigt or Brie mixing law ($e=1$) suggest more reliable V_p . Hill's law is particular model that can be suitable for *semi-uniform* saturation behaviour. Hill model can be matched with Brie's law for precise e empirical constant between 1.5 and 2.5. However, defining accurate V_p response to CO_2 injection in a laboratory is quite difficult because of supercritical state of CO_2 . Therefore, defining the mixing model or e empirical constant considering all rock and fluid elastic properties are definitely one of the critical part in the success of CO_2 sequestration.

Results of this study cannot recommend a suitable mixing model particularly for Utsira sand because estimated V_p from fully CO_2 saturated sand is matching with the expected results for all mixing models. On the other hand, results also show that measuring the Bulk modulus of dry Utsira sand is a must to rely on the V_p responses. This study verifies the suggested 2.56 GPa as dry Utsira sand Bulk modulus.

5. Conclusion

In light of the challenges and expenses associated with geological methods, this study emphasizes the widespread utilization of geophysical techniques, including seismic 3D/4D and AVO analysis, for monitoring CO_2 sequestration. Employing various fluid mixing scenarios and Gassmann fluid substitution theory for V_p estimation, we underscore the pivotal role of dry rock bulk modulus in modeling CO_2 sequestration processes, with a focus on the Utsira sand as a representative rock sample.

Multiple approaches, including Gassmann-Wood, Gassmann-Brie, Voigt Averaging and Gassmann-Hill are employed to calculate the bulk modulus of fluid mixtures. The results prominently highlight the unacceptably wide range of dry rock bulk modulus for sensitive CO_2 injection monitoring. Consequently, we strongly recommend (1) laboratory measurements of dry rock bulk modulus for reliable and sensitive V_p estimation. Additionally, our findings indicate the

indirect dependence of the V_p - SCO_2 curve on the bulk modulus of the fluid mixture, emphasizing the critical importance of understanding the saturation behavior or relaxation state of the formation for accurate CO_2 saturation estimation during injection.

This study specifically applies the Brie mixing law, revealing that (3) it can be suitable for both patchy and uniform saturation models with a precisely defined empirical constant. To determine the correct saturation model for Utsira sand, we propose (2) injecting a pre-defined fluid mixture into the pore spaces of Utsira sand in a laboratory study, plotting V_p versus fluid saturation graphs to identify a suitable mixing law or accurate empirical constant for various CO_2 injection scenarios.

Moreover, we raise a question regarding AVO responses of fluid mixing laws, suggesting that analyzing AVO responses of Utsira sand considering fluid mixing laws could be a valuable supplemental study. This comprehensive exploration provides valuable insights for refining methodologies in CO_2 sequestration monitoring, advocating for laboratory studies and precise empirical constants for improved accuracy in saturation estimation.

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Effect of degree of saturation on consolidation parameters of fine-grained soils

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ABSTRACT

Terzaghi (1943)'s theory of consolidation phenomenon has some limitations for unsaturated geological environments in nature. Additionally, soils are subjected to consolidation tests under partially saturation conditions. The scope of this study is to illustrate the effect of degree of saturation (S_r) on the consolidation parameters using remolded soil samples. For this purpose, swelling pressure, compression index (c_c), re-compression index (c_r), coefficient of consolidation (c_v), and pre-consolidation stress (σ_p) were determined for the samples having different degrees of saturation and plasticity (PI). The consolidation parameters obtained from "undersaturated" remolded samples with varying degrees of saturation were compared with the consolidation parameters obtained from "fully saturated" remolded samples to illustrate which parameters of consolidation are affected most by the degree of saturation. The effect of swelling of partially saturated samples was eliminated prior to conducting oedometer tests. Results indicated a meaningful relationship between the compression index and the degree of saturation, though the relationship with the recompression index was less certain. Pre-consolidation stress generally decreased as saturation increased, while no definitive trend was observed between the degree of saturation and the consolidation coefficient. Overall, most consolidation parameters were found to be somewhat affected by the degree of saturation.

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1. Introduction

Terzaghi (1943)'s one dimensional consolidation theory the following assumptions are made: the soil is homogeneous and fully saturated, soil particles and pore water are incompressible, and water flow during pore water dissipation is only in the vertical direction, the consolidation is one-dimensional (1D). Additionally, the change in soil volume due to consolidation is negligible compared to the initial soil volume, flow is laminar and follows Darcy's law, and permeability remains uniform throughout the compressible soil layer. The coefficient of volume compressibility remains constant over time, and

compression occurs solely from pore water expulsion, so initial and secondary consolidation are neglected. Finally, the coefficient of consolidation for a specific soil and site conditions is assumed to be constant.

Terzaghi (1943)'s consolidation theory postulates that soil specimen subjected to oedometer tests are fully saturated, a postulation is valid only for samples collected from the capillary fringe or below the water table (ASTM 2435/D2435M-11, 2020). Thereby, the theory of consolidation has some limitations for unsaturated geological environments in nature, which are sited most of engineering projects and/

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or foundations above fully saturated zones where the soil is partially saturated. Additionally, soil samples taken from boreholes will be unsaturated due to volume expansion as overburden pressure is removed. And in practice, test specimens are directly subjected to consolidation test regardless of the zone where the soil sample collected from. That means no saturation procedure is applied prior to consolidation test. Although the soil specimen is inundated at the beginning of the test, this procedure is only for preventing of the specimen against drying during the test (ASTM 2435/D2435M-11, 2020). In other words, inundation of the test specimen prior to the consolidation test does not promote saturation of soil specimen at all. Eventually, it turns out that all soils are subjected to consolidation tests under partially saturation conditions.

No earlier study was found investigating the effect of saturation degree on consolidation parameters for remolded soils by performing conventional consolidation experiments. Some researchers investigated the effects of soil index properties on certain consolidation-related parameters such as the pre-consolidation pressure, compression index. Some previous studies investigating consolidation properties in natural and/or remolded soils are summarized below. Several studies have been carried out to date on the investigation of the consolidation properties of soils., recompression index and swelling pressure by employing methods such as fuzzy logic, finite elements, certain empirical relationships as well as computer simulations (e.g., Dipova and Cangir, 2010; Naser, 2013; Kayabalı and Yıldız, 2014; Sünnetçi and Ersoy, 2016; Yurtçu and Özocak, 2016). These researchers revealed in general that the index properties of soils and the consolidation parameters can be calculated. Kayabalı and Özdemir (2010), Asadi (2017) and Balcı et al. (2018) tried to find the consolidation parameters in natural and remolded soils by using centrifugal consolidation technique, which can reduce the duration of experimental studies that take more than a week in the traditional consolidation method to a few hours. For this purpose, the consolidation parameters obtained from the centrifugal consolidation method and the consolidation parameters obtained from the

one-dimensional consolidation experiments were compared. Gibson et al. (1967), Yong et al. (1983) and Fox and Berles (1997) explain high strains in soils. Biot (1941) developed Terzaghi (1925)'s one-dimensional consolidation theory in three dimensions and used this theory (Biot, 1956) in solving dynamic problems. Working on one-dimensional consolidation theory, Davis and Raymond (1965), Xie et al. (2002), Geng et al. (2006) and Cai et al. (2007) argued that as a result of loads applied on the sample during consolidation test, the compressibility and permeability decrease with the shrinking of the pores in the soil. Hansbo (1960), Davis and Raymond (1965) and Geng (2008) developed nonlinear solutions of consolidation problems by using compression and permeability coefficients that decrease as a result of loading. Working on remolded clays prepared by considering the degree of saturation, Barden (1965) explained the stress state on unsaturated soils using Bishop (1959)'s relationship. Fredlund (1977), Fredlund and Morgenstern (1976) developed mathematical relations for the solution of consolidation problems in unsaturated soils. Casagrande and Fadum (1940) and Taylor (1942) developed methods for finding the consolidation coefficient, and Cortellazzo (2002), Muntohar (2009) compared the methods developed by different researchers to determine the consolidation coefficient. Boone (2010), who worked on the calculation of the pre-consolidation stress, also presented the work of many researchers in summary form. Phanikumar and Amrutha (2014) aim that the roles of overburden pressure and the degree of saturation in changing the void ratio and compressibility of clays. For this purpose, conducted a series of consolidation tests on soil samples with a wide range of saturation degrees (25, 50, 75 and 100%) to ensure pre-excavation, post-excavation and post-construction conditions in a laboratory environment. However, the main purpose of their study is not to compare the effect of saturated and unsaturated soils on consolidation parameters. To realize the pre-excavation and post-excavation conditions in the laboratory environment, they applied and removed 25, 50 and 100 kPa stress to the samples with different degrees of saturation and applied a stress up to 400 kPa to achieve the post-construction condition.

2. Materials

A total of 8 soil samples with different plasticities were used in this investigation. Samples were collected from the vicinity of Ankara, Türkiye. They were dried and sieved through the #40 sieve prior to the compaction and consolidation tests. Phase relationships of solid-water-air system (e.g., Holtz and Kovacs, 1981) were used to prepare identical samples. For this, the initial void ratios and dry unit weights

of the samples were kept constant. The index and physical properties of the samples were determined, and the samples were compacted using static methods. The permeability-consolidation cell (Figure 1) was used to saturate the partially saturated samples and to perform consolidation experiments on these samples. Swelling pressure value of partially saturated samples was measured in the setup developed by Kayabali and Yıldız (2014) (Figure 2).



Figure 1- Permeability-consolidation test apparatus and its accessories (from Asadi, 2017).



Figure 2- Swelling pressure test apparatus (from Kayabali and Yıldız, 2014).

3. Methods

To determine the physical and index properties of samples, sieve analysis, hydrometer test, Atterberg limits tests and pycnometer test were performed, and soil classes were determined according to Unified Soil Classification System (USCS). For the plastic limit and liquid limit tests, the part of the samples that passed #40 sieve was used. Plastic limit tests were carried out using the hand-rolling method according to ASTM D 4318-05 (American Society of Testing Materials, 2005) with 5 replicates of each sample and 50 g of the sample amount in the replicate. Liquid limit experiments were performed using the falling cone method according to BSI 1377 (British Standards Institution, 1990) at 5 points corresponding to the penetration amounts between 10-30 mm for each sample. The solid particle densities of the samples were calculated by applying the pycnometer test according to ASTM D 854-06 (American Society of Testing Materials, 2006a), using 25 g dry sample in 50 ml pycnometers.

The nature of the investigation requires preparation of soil samples with certain physical properties. To investigate the effect of various degrees of saturation on specific consolidation parameters the void ratio, the dry mass of soil sample and the total volume of the compacted soil sample need to be kept constant. The only variable is the amount of water for the preparation of test specimens. From each soil sample, 4 identical specimens were prepared which are homogeneously mixed at 70, 80 and 90 percent degrees of saturation. During the sample preparation stage, 2 samples at 70% saturation and the other 2 samples at 80% and 90% degrees of saturation, respectively, were prepared using the static compaction method. One of the samples compacted at 70% degree of saturation was used to obtain a fully saturated sample in the permeability-consolidation setup.

Initial trial tests showed that respectively at void ratios of 0.7 and 0.9 to CL and CH types of soils are suitable to prepare the consolidation test specimens to result in total wet densities recorded in most fine-grained soils. All necessary computations were made to find the proper amounts of dry soil mass and water

for the initial degree of saturation of first 0.7 to fill a consolidation ring with the diameter of 50 mm and a height of 20 mm. The soil and water were mixed and poured into a hollow cylinder with 50 mm diameter. Compressive load was incrementally applied up to 1 kN onto the wet mixture using a piston and load frame. After this, the specimen was turned upside down and the same loading procedure was applied from the reverse side and the resulting specimen height was measured. This procedure was repeated until the specimen height is reduced to 20 mm. At the end, a test specimen with diameter of 50 mm, a height of 20 mm, a void ratio of 0.7 and a degree of saturation of 0.7 was obtained. The statically compacted soil specimen was then transferred to a consolidation ring. The same specimen preparation procedure was adopted to obtain specimens with degrees of saturation of 0.8 and 0.9 keeping the void ratio of 0.7, the amount of dry soil mass and the total volume constant. All procedures outlined above both for CL and CH type of soils were repeated for using the void ratio of 0.7.

It is well known that a compaction procedure never yields a final product with the “zero air void”. Therefore, to prepare a fully saturated specimen ($S_r=1.0$) a different scheme was employed. For this, the spare specimen with the degree of saturation of 0.7 was prepared and placed into a specific cell shown in Figure 1. This is a two-stage procedure to obtain a fully saturated test specimen. The specimen in this cell first is allowed to saturate under a hydraulic head of about 50. After the water appears at the bottom outlet the specimen is left under hydraulic pressure for an additional period so that at least one pore-volume of water passes through the specimen. During the saturation stage, a load equaling to swell force determined from the procedure outlined below is kept on top of the test specimen to prevent the swelling of specimen during saturation. The second stage involves the routine one-dimensional consolidation procedure.

Swell pressure of soils is known to be controlled by dry density and water content. Therefore, artificially prepared soil specimens are expected to exhibit some degree of expansion when subjected to inundation prior to consolidation test. To obtain reliable data free

of swelling effect from the consolidation tests, certain precautions need to be taken to eliminate such effects. For this, a methodology was adopted in which the swell pressure of the compacted soil sample under certain void ratio and degree of saturation was measured on another identical sample. The consolidation specimen was placed in a consolidation cell. The loading piston attached to a load cell was lowered gradually on top of the specimen (Kayabalı and Yaldız, 2014, Figure 2). An initial seating load of 10N was applied to ensure that there is no gap between the top of the loading piston and the top cap laying on top of the specimen. The specimen was inundated and left so for a period of 24 hours. At the end of 1 day the total swell force was read from the digital display from which the initial seating load was deducted and then the remaining value was divided by the surface area of the test specimen to obtain the swell pressure.

Consolidation tests was carried out starting by eliminating the swell pressure recorded earlier (American Society of Testing Materials, 2006b). For this, the specimen was loaded to the measured swell force and then it was inundated so that the additional height coming from the swelling process is cancelled out. Vertical loads applied such that the resulting effective stresses are 25, 50, 100, 200, and 400 kPa. Unloading of the specimens was rendered as the loading sequence was reversed; namely from 400 kPa to 200 kPa, from 200 kPa to 100 kPa and so on. Vertical displacements were recorded immediately after placing the consolidation load. The readings were taken at times of 5 sec., 10 sec., 30 sec., 60 sec.,

2 min., 4 min., 10 min., 30 min., 1 hr., 2 hrs., 4 hrs., 8 hrs., and 24 hrs. Vertical displacement versus square root of times (in minutes) curve were constructed to determine t_{90} values of each stage of consolidation test per specimen. t_{90} values were used in computing the coefficient of consolidation, c_v .

4. Experimental Results

4.1. Physical and Index Properties of Soil Samples

The physical and index properties of 8 clay samples were determined initially. After that, swelling pressures, compression indices, re-compression indices, pre-consolidation stresses and consolidation coefficients for 32 homogeneous specimens (8 samples multiplied by 4 different levels of saturation) obtained from these samples were determined through the swelling and consolidation tests. Particle size distribution, liquid limit, plastic limit, solid particle density and soil classes according to the USCS for the investigated soil samples are given in Table 1 and Figure 3.

The liquid limit of the samples varies between 24.4 and 98%, the plastic limits are between 14.8 and 31.4% and thus the plasticity indices range from 9.6 to 66.6%. N1, N2 and N3 samples were classified as low plasticity clay (CL) and N4, N5, N6, N7 and N8 samples as high plasticity clay (CH). The specific gravity of the samples ranges from 2.68 to 2.78. As a result of sieve and hydrometer analyzes the percentages of sand, silt and clay of the samples vary between 16-80%, 18-64% and 3-37%, respectively.

Table 1- Index properties of soil samples employed in the investigation.

Sample No.	N1	N2	N3	N4	N5	N6	N7	N8
LL (%)	24.4	35.7	43.1	57.0	70.5	77	84.5	98
PL (%)	14.8	18.9	22.9	24.3	25.9	21.5	28.3	31.4
PI (%)	9.6	16.8	20.2	32.7	44.6	55.5	56.2	66.6
Soil class (USGS)	CL	CL	CL	CH	CH	CH	CH	CH
Specific Gravity (G_s)	2.70	2.74	2.76	2.76	2.75	2.68	2.74	2.78
Sand (%)	70	23	23	20	19	51	18	16
Silt (%)	18	64	63	60	56	30	57	47
Clay (%)	3	13	14	20	25	19	25	37

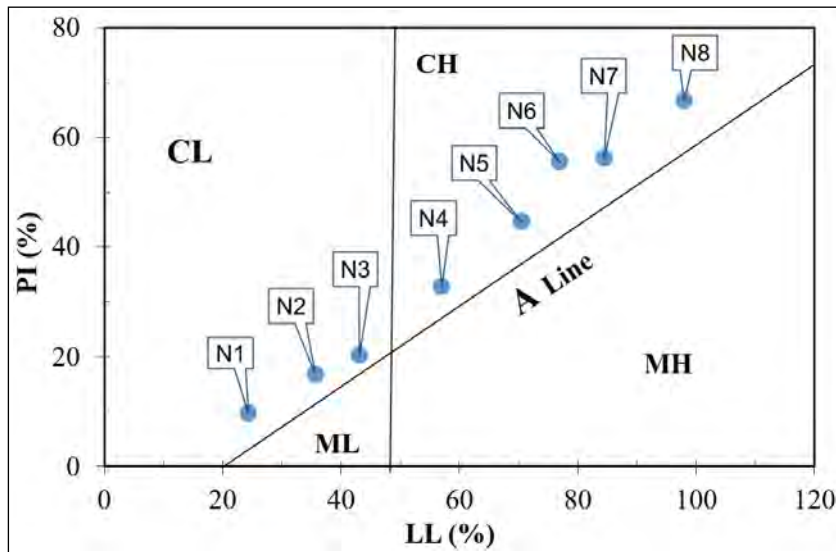


Figure 3- Distribution of soil samples according to Unified Soil Classification System (USCS).

4.2. Swelling Pressure

Swell tests were performed on partially saturated remolded soil specimens prior to the consolidation tests. The amount of swelling measured on those samples was used as the pre-load for the samples subjected to oedometer tests prior to inundation. Swelling pressures measured for all samples at different degrees of saturation are presented in Table 2.

The measured swelling pressures varied between 3 and 100 kPa. For the partially saturated samples, the relationships between swelling pressure and degree of saturation and plastic limit were also evaluated. It was observed that the swelling pressure values decreased

with increasing saturation degrees. This result is due to the high-water content of soils with high saturation degree and the low tendency of these soils to absorb water (Villar and Lloret, 2008, Elsharief et al., 2014, Keskin et al., 2023).

It was observed that the swelling pressure values increased with increasing plasticity. In addition, swelling behavior was not observed in the low plasticity samples (N1 and N2), while a small amount of swelling occurred in the low plasticity N3 sample.

4.3. Void Ratio - Effective Stress Relationships

As a result of the consolidation experiments, the void ratio-effective stress graphs were plotted, and the consolidation parameters of all the samples were determined using the Casagrande method (the details of this method may be found in Holtz and Kovacs, 1981). To compare the changes in consolidation parameters like that compression stress, re-compression stress and pre-consolidation stress, the void ratio-effective stress plots which show 4 different degrees of saturation (70%, 80%, 90%, and 100%) for each sample are presented in Figure 4. Detailed evaluations of these parameters are presented in the following sections.

4.4. Compression Index, c_c

Compression index values vary between 0.15-0.39 (Table 3). The degree of saturation versus the

Table 2- Swelling pressures for all soil samples (kPa).

No.	Degrees of Saturation, S_r			
	70%	80%	90%	100%
N1	-	-	-	-
N2	-	-	-	-
N3	10	3	-	-
N4	10	8	3	-
N5	25	20	10	-
N6	28	25	18	-
N7	65	35	30	-
N8	100	80	65	-

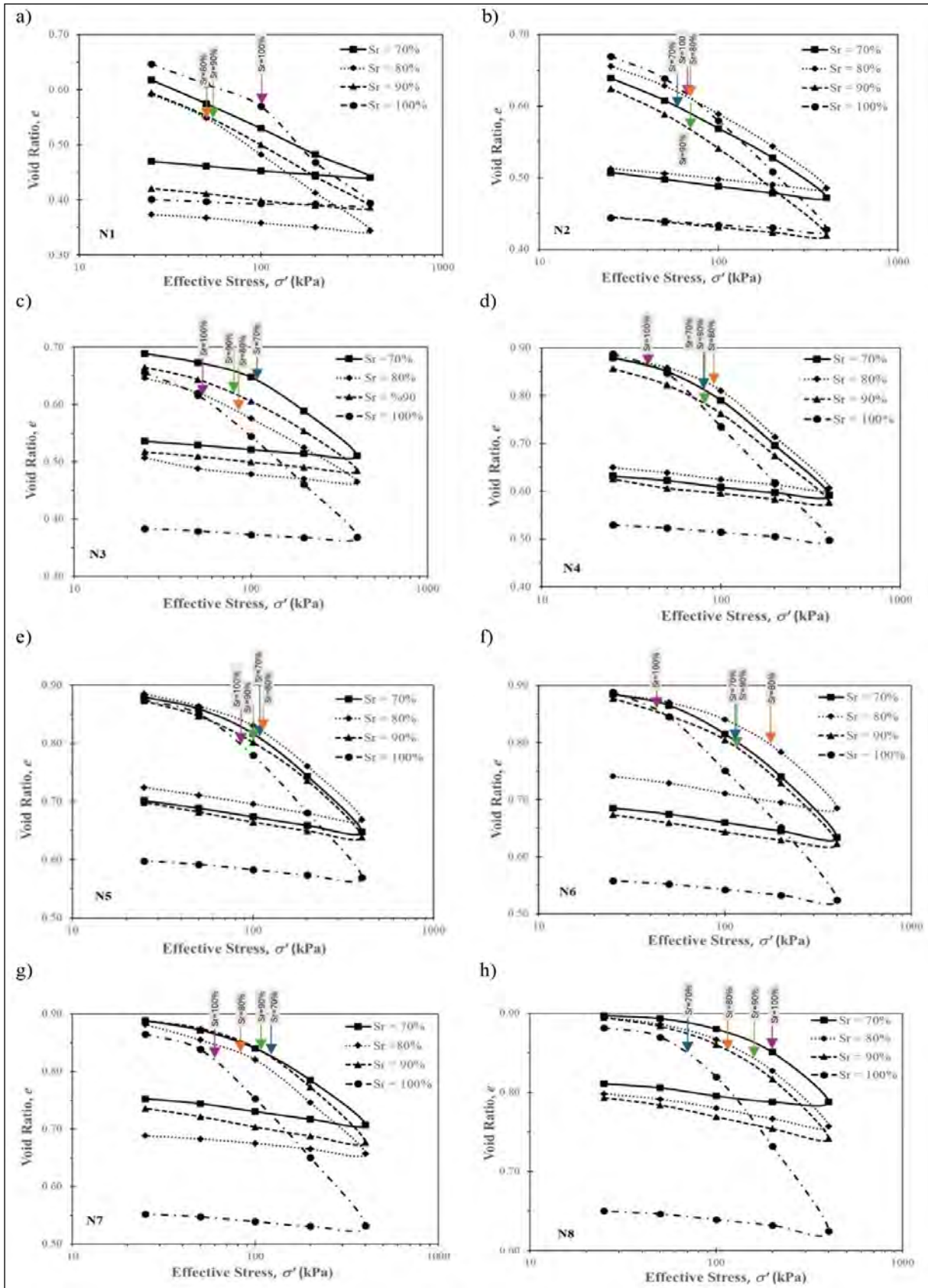


Figure 4- Oedometer test results for 8 soil samples conducted at 4 different saturation ratios for a) Sample N1, b) Sample N2, c) Sample N3, d) Sample N4, e) Sample N5, f) Sample N6, g) Sample N7 and h) Sample N8.

Table 3- The list of compression indices (c_c), recompression indices (c_r) and preconsolidation pressures (σ'_p) obtained from oedometer tests conducted on specimens of varying degrees of saturation (S_r).

No.	LL (%)	S_r	c_c	c_r	σ'_p (kPa)
N1	24	0.7	0.15	0.023	-
N1	24	0.8	0.23	0.024	50
N1	24	0.9	0.19	0.028	55
N1	24	1.0	0.29	0.006	100
N2	36	0.7	0.16	0.029	60
N2	36	0.8	0.17	0.022	70
N2	36	0.9	0.20	0.021	68
N2	36	1.0	0.25	0.013	70
N3	43	0.7	0.23	0.021	110
N3	43	0.8	0.18	0.035	85
N3	43	0.9	0.20	0.027	80
N3	43	1.0	0.29	0.012	56
N4	57	0.7	0.33	0.033	81
N4	57	0.8	0.34	0.036	90
N4	57	0.9	0.31	0.040	82
N4	57	1.0	0.39	0.026	44
N5	71	0.7	0.28	0.045	110
N5	71	0.8	0.27	0.046	115
N5	71	0.9	0.27	0.049	100
N5	71	1.0	0.35	0.024	82
N6	77	0.7	0.30	0.043	122
N6	77	0.8	0.26	0.047	180
N6	77	0.9	0.30	0.042	120
N6	77	1.0	0.38	0.028	57
N7	85	0.7	0.22	0.038	130
N7	85	0.8	0.27	0.026	82
N7	85	0.9	0.27	0.049	120
N7	85	1.0	0.37	0.017	58
N8	98	0.7	0.15	0.019	200
N8	98	0.8	0.18	0.034	170
N8	98	0.9	0.20	0.043	140
N8	98	1.0	0.32	0.021	98

compression index graphs of all the samples are presented in Figure 5.

It was determined that there were positive correlations between increasing degrees of saturation and compression index, with coefficients of determination ranging from $R^2=0.32$ to $R^2=0.94$. While evaluating the compression index versus degree of saturation ($S_r - c_c$) relationships of the samples,

plasticity was also considered. It was observed that the coefficients of determination of the relationships between S_r and c_c were different in each sample. The reason for this difference is due to the different PI values of the samples. Since the density values are varying in a very narrow range, the relation between the cc and the density has not been evaluated. Azzouz et al. (1976), Herrero (1983) and Kalantary and Kordnaeij (2012) revealed that LL and e_0 are the most effective parameters on c_c . Wroth and Wood (1978) and Mesri et al. (1975) revealed that the compressibility and compression index of clays are mainly affected by type of clay minerals, clay content, plastic limit and liquid limit. When the $S_r - c_c$ relationships are evaluated by considering the PI the determination coefficients of $c_c - S_r$ relationships for N1 and N2 samples with PI values of 9.6% and 16.8% are quite high, and there are positive linear relationships between c_c and S_r . However, it is seen that the $c_c - S_r$ relationship for the sample N3 with a PI value of 20.2% is still positive, but the coefficient of determination is quite low ($R^2 = 0.32$). Thus, although the PI for these three samples increased, the correlation coefficients of the $S_r - c_c$ relationships first increased and then decreased. When the $S_r - c_c$ relationships of other five samples with higher PI values were examined, it was observed that the determination coefficients of the $S_r - c_c$ relationships increased from 0.34 to 0.81, as the PI values increased from 32.7% to 66.6%. It was thought that the soil group (CL and CH) should also be considered while evaluating the samples with these different PI values. Accordingly, while there was inconsistency in the $S_r - c_c$ relationships for the first three samples in the CL soil group, the significance of the relations increased as the PI increased for the five samples in the other CH soil group. In addition to considering the PI effect when examining the $S_r - c_c$ relationships, differences between the partially saturated samples ($S_r = 70\%$, 80% and 90%) and the fully saturated samples were also observed. When the graphs in Figure 5 are examined, it is observed that the c_c values for all PI values also increased as the saturation increased until it reached 90%. However, c_c increased quite significantly in 100% saturated samples. These results clearly show that the c_c values of fully saturated and partially saturated samples can be quite different from each other.

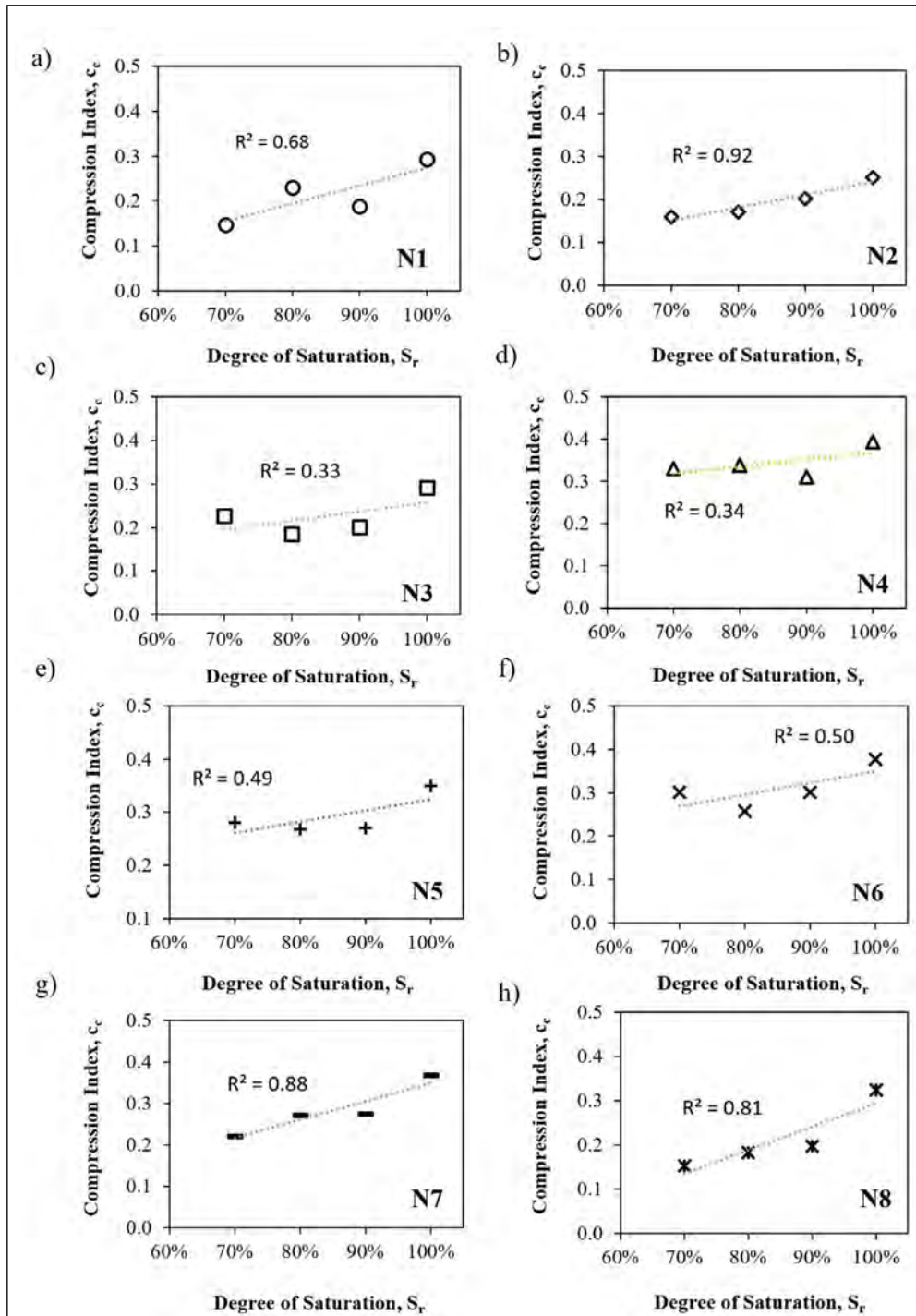


Figure 5- Graphs illustrating the relationship between the compression index and the degree of saturation for a) Sample N1, b) Sample N2, c) Sample N3, d) Sample N4, e) Sample N5, f) Sample N6, g) Sample N7 and h) Sample N8.

4.5. Re-compression Index, c_r

Recompression index values vary between 0.01 and 0.05 (Table 3). The saturation degree-recompression index graphs of all samples are presented in Figure 6.

It can be clearly seen from the graphs in Figure 6 that fully saturated ($S_r = 100\%$) and partially saturated ($S_r = 70\%$, 80% and 90%) samples significantly affect the c_r values in the recompression index-degree of saturation relationships ($c_r - S_r$). Accordingly, when

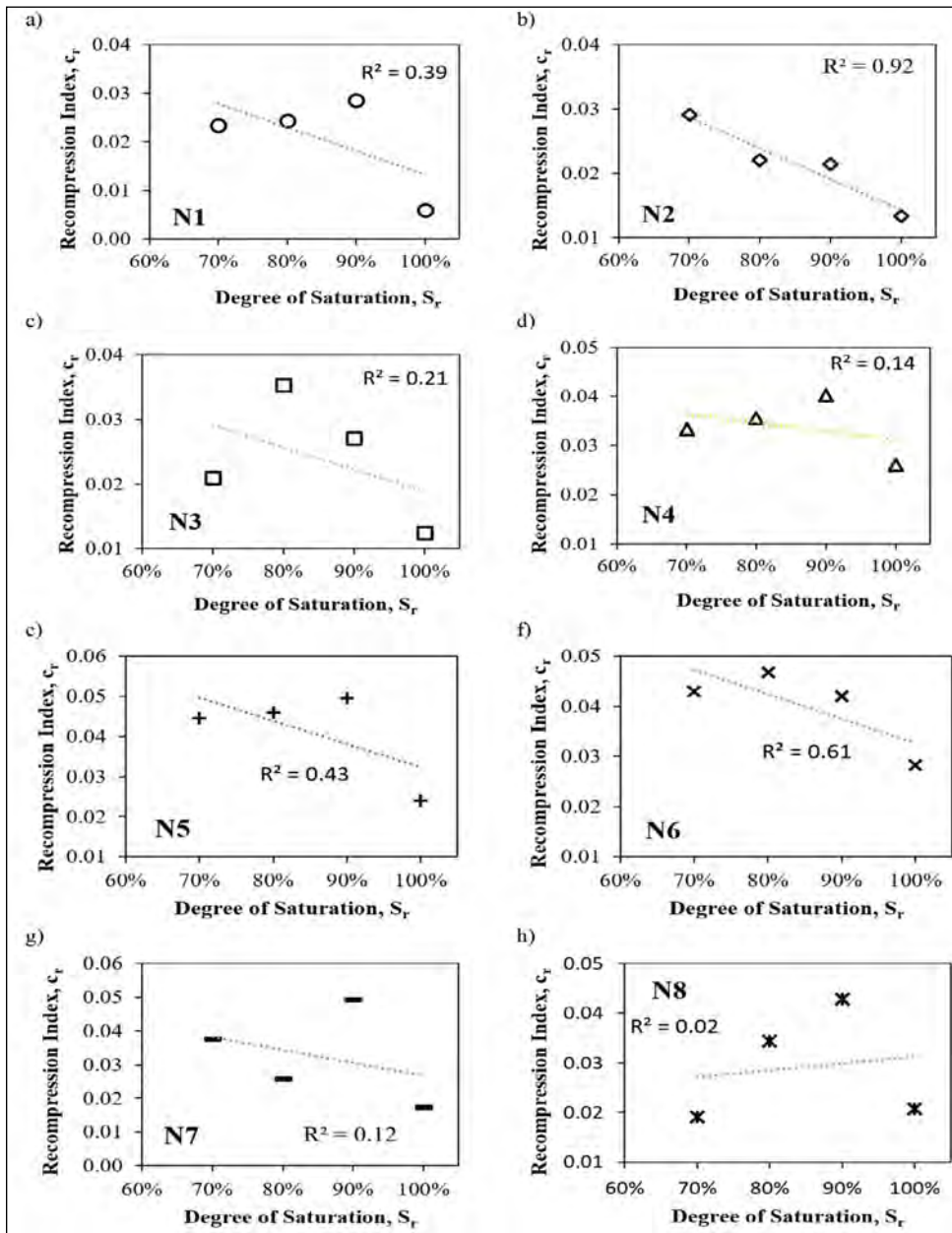


Figure 6- Graphs illustrate the relationship between the recompression index and the degree of saturation for a) Sample N1, b) Sample N2, c) Sample N3, d) Sample N4, e) Sample N5, f) Sample N6, g) Sample N7 and h) Sample N8.

the relationships of N1, N3, N4, N5 and N8 samples were examined, it was observed that the c_r values of the fully saturated sample were inconsistent with the c_r values of partially saturated samples. It was determined that the c_r value of the fully saturated sample decreased abruptly in comparison with the c_r value of the partially saturated samples in almost all cases. Thus, it has been understood that the c_r value of the saturated sample deteriorates the general trend

of the $c_r - S_r$ relationships and affects the R^2 values by significantly decreasing them. When $c_r - S_r$ relations were evaluated according to increasing PI values and soil group, it was determined that these two parameters did not have a significant effect on the relationships.

4.6. Pre-consolidation Stress, σ_p'

The pre-consolidation stress values of the samples vary between 44 and 200 kPa. The saturation degree

- pre-consolidation stress graphs of all samples are presented in Figure 7. Since the graph of $e\text{-}\log\sigma_p'$ at 70% saturation was linear in the N1 sample, the pre-consolidation stress could not be calculated. It was observed that the pre-consolidation stress of the saturated samples, especially those with high plasticity, was lower than the values of the partially saturated samples.

Except for samples N1, N2 and N6, the increasing degree of saturation causes the pre-consolidation stress to decrease. While the pre-consolidation stress values of partially saturated high plasticity samples (N5, N7 and N8) were very close to each other, a significant decrease was observed in the pre-consolidation stress of the fully saturated high plasticity samples (Figure 7).

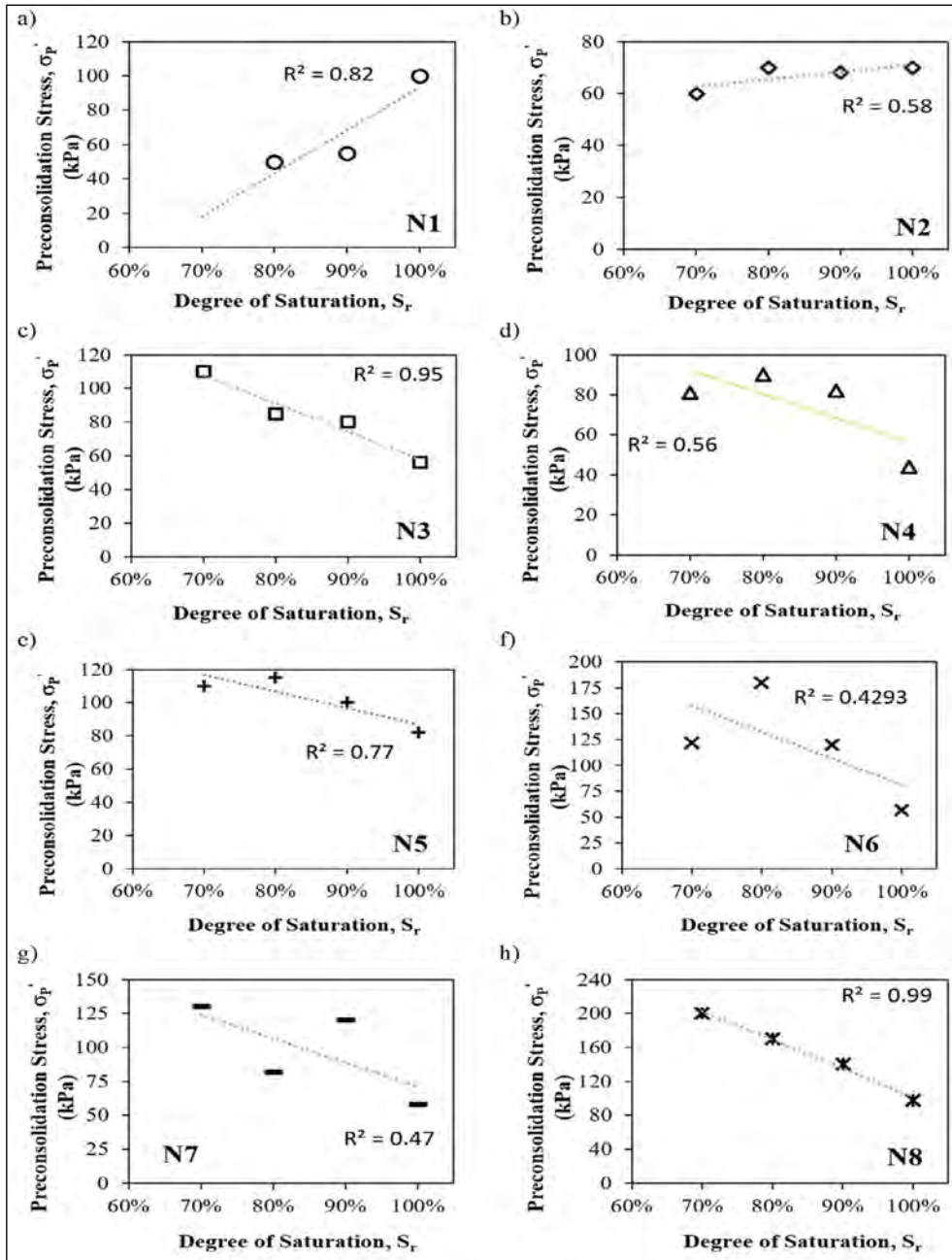


Figure 7- Graphs illustrating the relationship between the pre-consolidation pressure and the degree of saturation for a) Sample N1, b) Sample N2, c) Sample N3, d) Sample N4, e) Sample N5, f) Sample N6, g) Sample N7 and h) Sample N8.

4.7. Coefficient of Consolidation, c_v

The consolidation coefficient values of the samples vary between 1×10^{-4} – 14×10^{-4} cm²/s (Table 4). Saturation degree-consolidation coefficient graphs for 25, 50, 100, 200 and 400 kPa stresses applied at 70%, 80%, 90% and 100% saturation degrees of all the samples are presented in Figure 8.

When the relationship between the consolidation coefficient values and the effective stresses was evaluated it was observed that the coefficients of determination of this relationship are quite low, and it is concluded that there is no significant relationship between the saturation degree and the coefficient of consolidation. In addition, the relationship between the consolidation coefficient, the degree of saturation and the plastic limit were also evaluated. It was observed that the consolidation coefficient was not affected by the degree of saturation and the plasticity value of the samples. The relationships between the coefficient of consolidation and the degree of saturation, the effective stress and the plasticity index appear to be rather random.

5. Conclusions

The following conclusions were reached at the end of this investigation. The swelling pressures of all remolded samples at statically compacted at 70, 80, 90 and 100 percent degrees of saturation range from 3-100 kPa. It was observed that the swelling pressure decreases as the degree of saturation increases, as expected. The effect of soil plasticity on the swelling pressure was prominent as well. Swelling behavior was not observed in the partially saturated low plasticity samples in general. The aim of determining the swelling pressures for partially saturated soils was to ensure that swelling did not affect the results of oedometer tests.

The general tendency between the compression index (c_c) and the degree of saturation is that the compression index increases with the increasing degree of saturation. This conclusion is valid for both the low- and high-plasticity samples. The practical meaning of this conclusion is that the total settlement calculated for fine grained soils may differ depending on the degree of saturation.

Table 4- Coefficients of consolidation (x1000) determined through the oedometer tests conducted at different degrees of saturation.

No.	σ' (kPa)	70%	80%	90%	100%
N1	25	9.0	8.7	9.5	0.0
N1	50	8.7	8.4	9.2	0.0
N1	100	9.5	9.1	9.9	13.8
N1	200	10.1	9.7	10.8	11.3
N1	400	10.4	10.2	11.2	11.3
N2	25	9.1	6.5	4.1	0.0
N2	50	7.8	5.5	1.0	11.1
N2	100	7.5	6.0	5.3	11.7
N2	200	8.1	6.6	4.6	10.2
N2	400	8.3	7.6	7.3	9.0
N3	25	5.7	9.7	8.5	0.0
N3	50	9.4	6.9	7.7	5.0
N3	100	7.6	6.7	5.2	8.3
N3	200	9.3	8.1	6.8	6.7
N3	400	7.4	6.8	5.7	6.2
N4	25	4.2	10.3	6.0	0.0
N4	50	1.2	1.7	4.8	5.5
N4	100	6.6	7.8	5.3	11.3
N4	200	7.1	5.3	6.0	5.2
N4	400	3.7	1.8	4.6	3.4
N5	25	4.7	7.3	7.7	0.0
N5	50	5.4	1.1	3.6	1.7
N5	100	5.0	7.5	4.9	8.3
N5	200	6.8	7.3	4.6	6.7
N5	400	6.4	6.2	3.5	5.8
N6	25	0.0	6.4	5.0	0.0
N6	50	0.0		7.8	0.0
N6	100	6.4	6.8	4.2	8.3
N6	200	8.0	7.8	7.0	5.0
N6	400	6.6	7.1	6.2	3.7
N7	25	7.2	7.9	0.0	0.0
N7	50	0.0	7.6	0.0	7.1
N7	100	5.1	4.2	1.1	8.1
N7	200	5.1	5.4	7.5	4.9
N7	400	5.3	4.9	5.5	2.5
N8	25	0.0	3.6	6.0	0.0
N8	50	1.9	7.7	2.2	0.0
N8	100	2.2	0.0	2.6	0.0
N8	200	2.1	1.0	2.5	3.7
N8	400	4.4	7.4	5.3	1.6

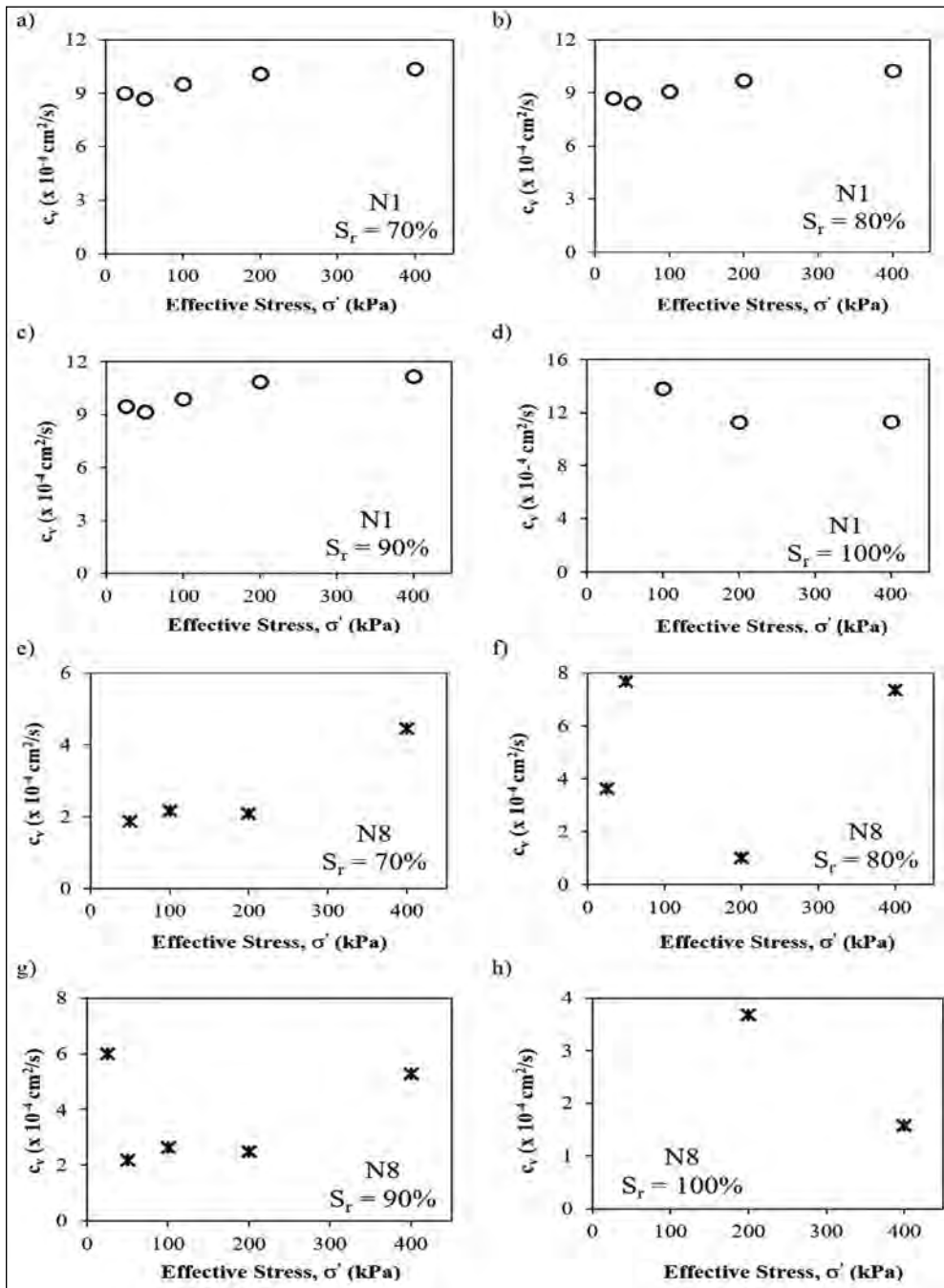


Figure 8- Consolidation coefficient versus effective stress plots for the selected two samples (i.e., N1 and N8).

Regarding the dependence of the re-compression index (c_r) on the degree of saturation, no conclusive statement can be made because of the inconsistent pattern between those two parameters while the general picture portrays that c_r tend to decrease with the increasing degree of saturation in contrary to the case of the compression index. The plasticity index did not appear to affect the c_r in general. Increasing

the number of soil samples may help asserting a more conclusive statement for this correlation.

Excluding the two samples with the lowest plasticity (i.e., N1, and N2), the pre-consolidation pressure (σ'_p) appears to decrease as a function of the increasing degree of saturation. In general, high-plasticity samples exhibit higher pre-consolidation

pressures than do the low-plasticity soils at similar degrees of saturation.

There was no systematical pattern between the coefficient of consolidation at varying effective stresses and the degree of saturation. And alike, no conclusive statement can be asserted for the dependence of the coefficient of consolidation on soil plasticity.

The overall conclusion is that all the consolidation parameters considered herein are affected somehow by the degree of saturation of remolded soil samples. This investigation was performed on laboratory prepared identical samples. Further investigation using natural soil deposits would help better handling the settlements calculations on fine-grained soils.

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Soil remediation at coastal plains with the help of DC Resistivity Method

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Research Article

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Saline Soil, Pollution,
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Corrosion.

ABSTRACT

The coastline of the Dardanos, a district of Çanakkale, Türkiye, suffers from saltwater intrusion due to excessive extraction of groundwater for domestic usage and also agricultural activities. Thus, the salinity level increased, and much of the land became unusable. The electrokinetic remediation method was employed to reduce the salinity level in the soil samples in laboratory conditions. The sample used in remediation is silty agricultural soil, with pH value and electrical conductivity (EC) of which are 8.33 and 1282 µS/cm respectively. In the lab-scale experiments, three different types of electrodes, aluminum, copper, and galvanized steel, were used in the tests. For each type of metal, electrode pairs were placed in the soil that was filled in a plastic container. Current variation was monitored while 1 VDC/cm was applied to electrodes. Average electrical conductivity reduces to 13.5%. As a side effect, all electrodes suffered from heavy corrosion which may be prevented by using anti-corrosion additives to reduce damage for future applications.

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1. Introduction

The coastal areas accommodate the majority (~70%) of the world's population for residential and agricultural activities (Cohen, 2006; Bear and Cheng, 2010). Currently, shoreline settlements are facing a scarcity of water supply due to seawater intrusion (Pulido-Leboeuf, 2004; Abarca Cameo, 2006; Abd-Elaty et al., 2022). Excessive pumping of water from wells due to water needs in industrial activities and residential usage reduces the slope of the water table towards the sea or the water level falls below sea level. In either case, seawater rushes into freshwater aquifers. This invasion reaches a drastic level if the freshwater flow toward the shoreline is interrupted even for a relatively short period. In addition to the replacement of the saltwater and freshwater interface, pumping out of groundwater in coastal

areas excessively together with the usage of chemicals (fertilizers, etc.) to increase productivity causes some of the environmental problems and agricultural lands to become redundant.

Various types of soil remediation processes aim to eliminate contaminants from the soil. Traditional and novel soil remediation techniques are subject to various research (Sheoran et al., 1989; Luo et al., 2016; Karaca et al., 2017; 2018; 2022). Electrokinetic soil remediation has attracted increased interest (Acar and Alshawabkeh, 1993; Zelina and Rusling, 1999; Karaca et al., 2019; Bessaim et al., 2020; Özentürk et al., 2021; Karaca et al., 2022; Özentürk, 2022; Karaca et al., 2023; Taneja et al., 2023a; Taneja et al., 2024; Özentürk et al., 2024). The electrokinetic remediation processes employ the direct current (DC) and create an electric field in the geological unit. This

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is done by placing metal electrodes into the soil to be decontaminated. Then, approximately 50 to 150 V is applied to electrodes to create an adequate current to mobilize the ions in the soil.

Electrokinetic remediation (EKR) works with several different processes. Depending on the specific properties of the pollutants, each process acts individually or in combination to remove the target chemicals from soil. Electromigration, electrophoresis, and electro-osmosis transport the target pollutant to a location for removal (Reddy and Cameselle, 2009; Brusseau and Maier, 2014).

In this study, a bench-scale electrokinetic treatment trial was conducted to reduce soil salinity in the shoreline of the Çanakkale strait. The soil sample was taken from the Çanakkale-Dardanos Campus (CDC), northwest Türkiye (Figure 1). Alluvium stretches along the shoreline and overlays sandstone and mudstone units. Excessive pumping of water from wells with various depths in a relatively small area resulted in saline water intrusion. For testing purposes, we conducted an inter-disciplinary approach; first, we mapped out geological units of the target area and located geological units prone to be polluted by seawater. Then, we conducted a geophysical survey to obtain the resistivity distribution of the area. Based on the result we chose the place to take soil samples, and then an electrokinetic remediation study was carried out.

The outline objectives of this study are as follows;

- Map out possible geological units susceptible to pollution
- Execute a geophysical survey to map out salinization levels in the study area.
- Evaluate the success of electrokinetics for the remediation of salinized soil, and
- Examine the effectiveness of electrode types and configurations.

We recommend the following steps;

- The Direct Current Resistivity (DCR) method shall be used to reveal the geo-electrical image that helps to map the saline-rich geological units.

- Soil samples shall be collected according to resistivity model.
- Multi-electrode system for anodes and cathodes shall be employed for remediation.

Our primary findings indicate that upscaling the application from laboratory to field scale must be solved. Keeping the moisturizing at an adequate level and also removing the pollutants from the system immediately are vital for protecting the electrodes. We will present our results in due course.

In the following sections, we outlined the DCR Method to map out the resistivity distribution of subsurface geology and salty area. This map is used as a reference to locate a sampling point for soil. Then we executed lab experiments. Our findings and discussion follow the experiment section.

2. Methodology

Following our objectives, the local geological map indicates that alluvium stretches along the shoreline and also dominates the CDC area. The unit presents inhomogeneous features. To locate the sampling points, we utilize DCR method that is well known geophysical methods. Note that the electrical conductivity meters are in use for agricultural lands (e.g. McNeill, 1992; Corwin and Lesch, 2005; Kühn et al., 2009; Lech et al., 2016) and target the top-most layer of soil (depth < 1 meter). We employed DCR method to obtain pseudo three-dimensional (3D) resistivity distribution of survey area. In this approach, we are able to map the extension of the saline-rich units in x,y and z (depth) directions, in turn, we can also calculate approximate volume that needs remediation.

2.1. Direct Current Resistivity Method

DCR method is a geophysical method that was first suggested by Schlumberger (1920). Initially, the DCR method was used to delineate the saline and freshwater boundaries and has been subject to research since 1930. Swartz (1937; 1939) pioneered groundwater exploration and used the DCR on the Hawaiian Islands (Fretwell and Stewart, 1981; Ulugergerli, 2017). In simple terms, the DCR method requires injecting electrical current from two electrodes and measuring

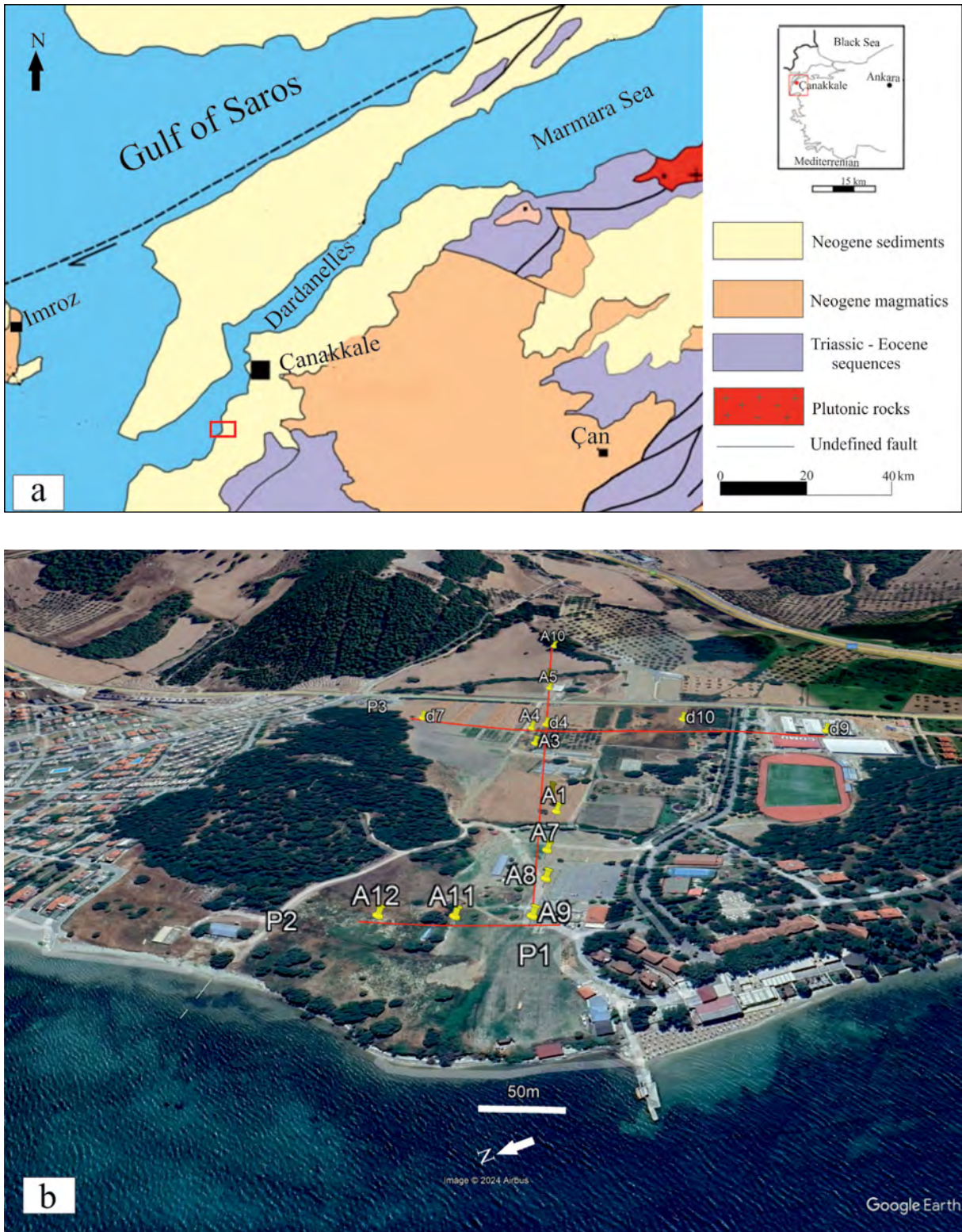


Figure 1- a) Geological setting of Biga peninsula (Okay and Satur, 2000) b) The study area and measurement stations for the DCR method (yellow pins).

voltage difference from the other two electrodes over the ground surface. The difference from EKR is that the duration of injecting current is relatively short, i.e., 10-20 sec.

The injected current and the measured potential difference are used to calculate apparent resistivities for each measurement point along the profiles (Telford et al., 1990). Apparent resistivities are not suitable for image resistivity distribution of geological stratum. Thus, a two-dimensional (2D) inversion scheme was chosen for the current study (Sasaki, 1992). This approach requires all stations to align along a profile and to be spatially dense enough (e.g. Ulugergerli, 2017; Ertekin and Ulugergerli, 2022). Then, an efficient and accurate 2D image of geo-electrical structures beneath the profile could be recovered employing 2D inversion. Note that Menke (1989), Meju (1994), Zhdanov (2002), and many other researchers provided details for both inversion and modeling for geophysical methods. Following the technological development, the DCR data can be gathered by using multi-electrode instruments, then this approach is called electrical resistivity tomography (Chambers et al., 2006; Candansayar, 2008; Wilkinson et al., 2012).

2.2. Soil Sampling

We collected the soil sample away from actively used roads. Removed the top dirt and got ~30 kg soil 10-20 cm below the surface. Sieved the soil using a 2 mm sieve and removed pebbles, organic materials, etc. before further steps.

In order to determine physico-chemical parameters, first, natural water content (ASTM D2216) was determined as 19.28%. Then the ratio of 1:5 (Soil: DI water) was used to determine salinity, pH and EC values. The samples were mixed for 6 hours at 180 rpm in the shaking device. At the end of 24 hours, the values measured with the Wtv multi 340i device are given in Table 1. As a result of the sieve analysis, the soil corresponded to the low plasticity clay (CL) class according to the Unified Soil Classification System (USCS).

Table 1- Chemical and physical properties of the sample taken from the study area.

Ground Class (USCS)	Water Content %	pH	Electrical conductivity (EC) $\mu\text{S}/\text{cm}$	Salinity (NaCl equivalent) %
CL	19.28	8.33	1282	0.45

2.3. Electrokinetic Method

During our experiments, a plastic container with dimensions of 40x58x15 cm was used to create a scaled sample for field application. Figure 2 shows the electrode configuration which we used for this study.

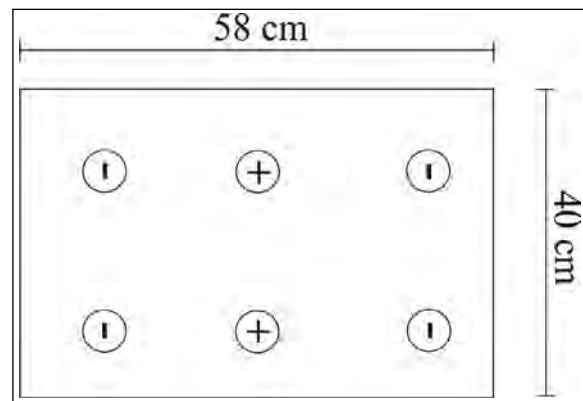


Figure 2- Electrode configuration; (+) anode (-) cathode electrodes.

The mud-cake containing soil and distilled water was placed into a plastic container (Figure 3). A wooden mallet was used to remove the air bubbles, if any, and make it tight. Note that the container must have a lid to prevent drying out during the remediation study.

Parameters such as the type of electrodes, content of electrolytes, and electrical current density affect the electrokinetic enhancement. Voltage set 1 VDC/cm considering the separation of the anode and cathode electrodes. Specifically, 11.6 V was applied because the distance between the electrodes was 11.6 cm. The current values measured during the experiment decreased over time. The experiments were terminated when the current values approached zero due to the displacement of ions in the soil and accumulation around the electrodes. At the end of the tests to determine the physicochemical parameters of the soil, soil samples were taken approximately 1 cm in front of the electrodes.

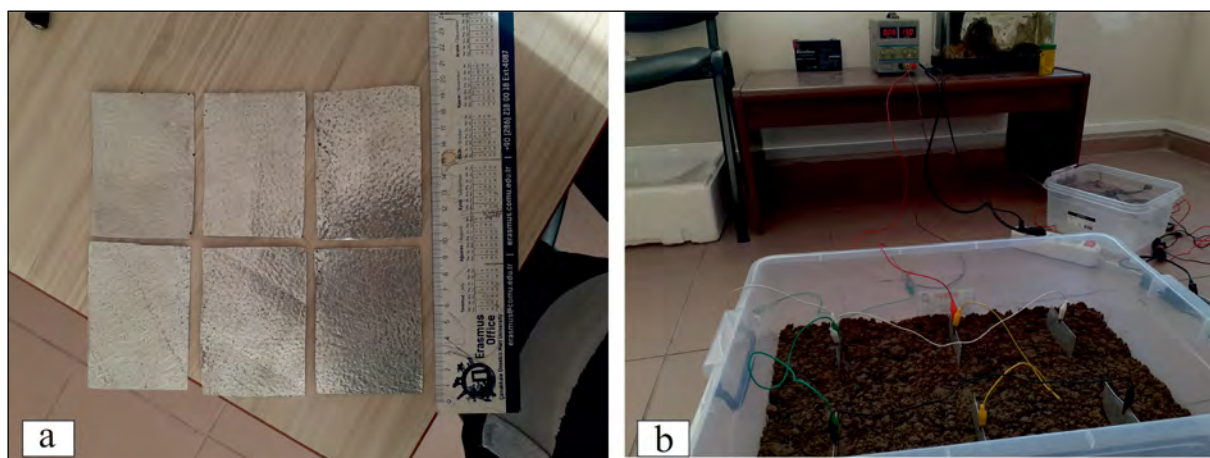


Figure 3- Electrokinetic experimental setup; a) preparation of electrodes, b) placement of electrodes.

3. Result and Discussion

3.1. DCR Survey

Considering the cost of the instruments, we used classical four electrode DCR equipment and we collected data using a vertical electrical sounding technique along lines and we combined and inverted those data sets using a 2D inversion algorithm.

As an example, the 2D resistivity-depth section of Profile 1, obtained from 2D inversion, is given in Figure 4. The agricultural soil (~ 10 ohm.m) covers the surface along the profile. Then, the existence of a low resistivity (<5 ohm.m) unit points out seawater intrusion. The main geological unit presents discontinuities along the profile (dark blue colored unit).

3.2. DCR Survey Results

In order to define soil sampling location, the 2D resistivity models are combined and the pseudo-3D resistivity model is presented as a fence diagram (Figure 5). While deeper parts let us evaluate the inland limits of seawater intrusion. The shallow part of the fence diagram pointed out the contaminated soils. Considering the low resistivity (<5 ohm.m) zone near the shoreline, we sample the soil between A9 and A11 (see also Figure 1).

A multi-dimensional evaluation of the DCR method pointed out the location requires remediation. Note that, if available, the multi-electrode DCR

systems can speed up the survey (Osinowo et al., 2018). Unfortunately, the exploration depth and may become insufficient if the land is surrounded by inaccessible lands. If it is the case electromagnetic method can be employed to map out the saline zones (e.g. Lesch et al., 1995; Çoban et al., 2017).

3.3. EKR Application and Findings

In the soil, the anions Cl^- , NaNO_3^- , NaO_3^- , SO_4^{2-} and cations consisting of Ca^{2+} , K^+ , Na^+ and Mg^+ form compounds and cause salinity (Kim et al., 2011). Depending on their charge, the contaminants and mobile ions present carry the electrical current from the anode to the cathode at the beginning of the test, causing a large current density peak after some time. The decrease following this peak is explained by the increase in electrical resistance of precipitates formed as a result of oxidation-reduction, electrolysis, dissolution and precipitation reactions during purification. Calcium ion production around the anode causes the salinity to increase and this value decreases due to calcium hydroxide precipitation at the cathode (Jayasekera and Hall, 2007).

The experiment conducted with copper electrodes was completed at the end of the 6th day when the current decreased to unmeasurable values. The experiment with the galvanized steel electrode was terminated on the 8th day under a 10 V constant current, and the one with the aluminum plate by applying 19 V current on the 15th day (Figure 6).

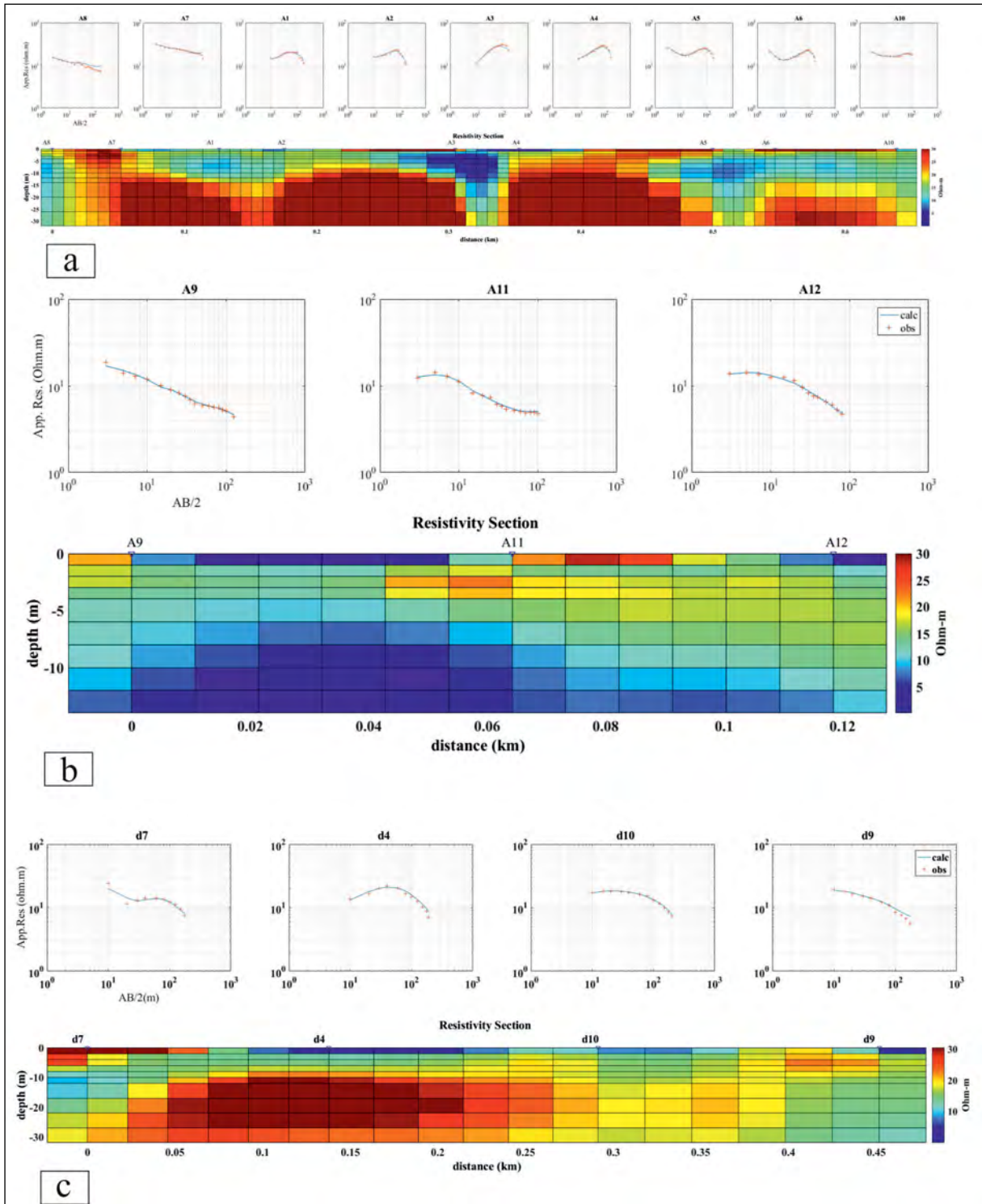


Figure 4- 2D resistivity models obtained from 2D inversion of DCR data. a) Profile 1, b) Profile 2, and c) Profile 3. The top graphs on each panel are apparent resistivity curves of observed (+) and calculated (solid lines) data.

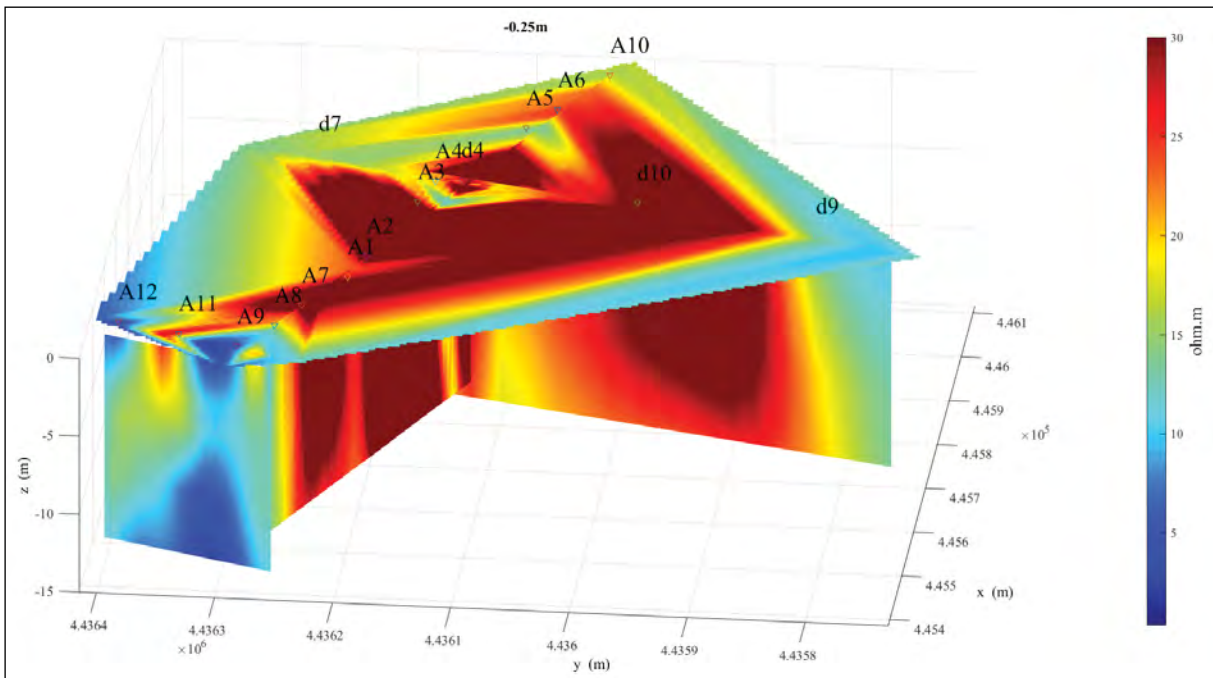


Figure 5- Depth - resistivity distribution at the surface and along the profiles. Resistivity values were extracted from all 2D inversion results.

Table 2 shows the parameters of the soil at the end of the electrokinetic experiments. pH differences at the electrodes are related to the electrolysis of water. By electrolysis of water, it produces O^2 and H^+ ions at the anode and H^2 and OH^- ions at the cathode. Therefore, the pH of the soil near the anode decreases and the pH around the cathode increases. Therefore, acidic pH measurements at the anode and alkaline pH measurements at the cathode are typical. At the same time, electrical conductivity (EC) decreased at the cathode, associated with sodium ion production at the anode. Consequently, it was observed that salinity

(NaCl equivalent) was intense, and the EC value was high in the anode region Table 2.

pH differences at the electrodes are related to the electrolysis of water. The oxygen and hydrogen ions produced at the anode create an acidic environment in this region, and the hydrogen and hydroxide ions produced at the cathode create a basic environment. In other words, the pH value is low in the anode region and high in the cathode region. At the same time, the difference in soil moisture content related to the electrolysis of water can be explained. A similar situation can be attributed to the corrosion of anodes

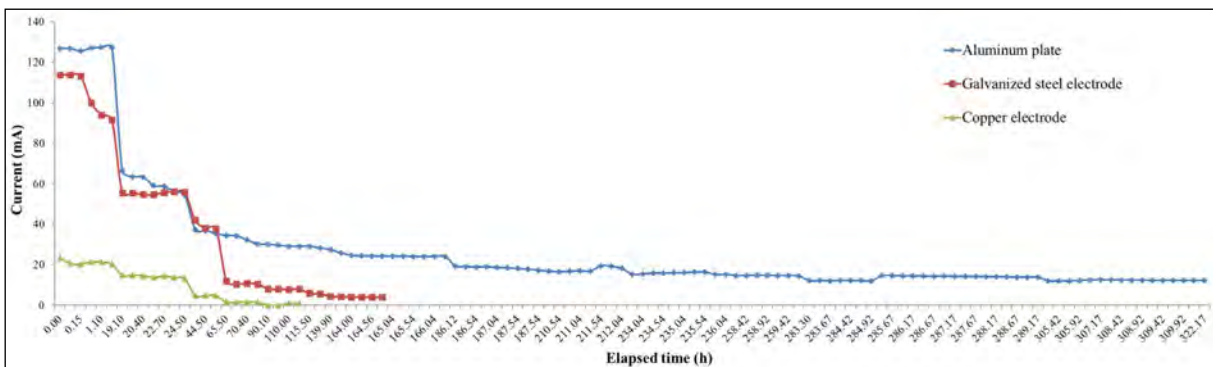


Figure 6- Variation of current intensity during the experiments.

Table 2- Moisture content, pH, EC, and salinity values of the soil after treatment.

End of treatment (average)	Copper electrode		Galvanized steel electrode		Aluminum plate	
	Anode	Cathode	Anode	Cathode	Anode	Cathode
Moisture content (%)	13.62	17.21	14.28	16.69	12.97	24.58
pH	6.21	9.50	6.53	8.59	6.40	9.78
EC ($\mu\text{S}/\text{cm}$)	328	146	320	178	4050	540
Salinity (%)	0.5	0.2	0.5	0.3	2.8	0.1

by oxidation of water (Barker et al., 2004). In addition, it can be said that other electrochemical reactions during treatment may also be effective. Although salinity removal was achieved in the study, corrosion occurred on the electrodes (Figure 7). In some studies, conducted for the remediation of heavy metals, it has been stated that since graphite oxidizes more easily at higher voltages, stainless steel may be used to conduct electric current at higher voltage with minimum corrosion effect and lower cost (Taneja et al., 2023b). Interruptions in the power supply and dryness of the soil affected the efficiency of the method. Thus, the added external liquid (pure water) restores humidity in the soil.

To reduce the electrode damage, pulsed current, instead of constant direct current may be applied. In this approach, the electric current is interrupted at regular intervals during the experiment. Thus, energy

savings will also be achieved as an important factor to consider for practical applications of EKR.

4. Conclusion

In this study, the 2D inversion results of DCR data were the keys to locating the polluted area. 2D resistivity models provided information down to 30 m of the study area. Also, the treatment efficiency of the electrodes used in the remediation of polluted soils by the EKR method and the corrosion, which occurred during the experiment, and is one of the important problems of this method, were investigated.

The experiments were conducted on saline soils and 1 V/cm DC voltage was applied to the soil during the experiments. The experiments conducted with copper, galvanized steel electrodes, and aluminum plates were completed after 6, 8, and 15 days, respectively.



Figure 7- a) Corroded copper electrodes, b) Corroded galvanized steel electrodes, c) Corroded aluminum plate electrodes.

The presence of water controls electrokinetic progress. The electrolysis of water and the ion release in the electrodes, which begins with the application of electric current, continues with the movement of existing and mobile ions in the soil to the electrodes. In the study conducted, this situation, which initially caused the current values to reach the upper values, ended with a decrease in the current values due to the transfer, accumulation and decrease in the concentration of existing ions in the process.

In EKR works, current is transferred to the soil through electrodes. Factors such as pH changes, high heat values due to current and types of contaminants cause corrosion on the electrodes. This causes interruptions in the current and disruptions in the treatment work.

At the end of the EKR, the pH values of the soils decreased slightly at the anode and increased at the cathode in all three experiments. Again, in all experiments, a clear decrease in EC was observed due to the removal of ions by the electrokinetic method, but the decrease at the cathode region was more remarkable due to the alkaline pH recorded.

Electric current applied directly to moist soil results in the electrolysis of water. O_2 and H^+ ions released at the anode result in a decrease in the pH value in this region, and the presence of H_2 and OH^- ions in the cathode results in an increase in the pH value in the region. The change in pH values causes this ion production in the anode region to react with soil components that dissolve carbonates and other basic salts and release metals. Similarly, on the cathode side, hydroxide, which is associated with high pH, provides the environment for the fixation of ionic metals. Therefore, this change in pH values affected the electrical conductivity of the soil, the development of electric current density during the experiment, and the distribution of ionic species in the soil after the electrokinetic treatment.

Since the corrosion of the metallic electrodes used in the study will lead to secondary metallic pollution in the soil due to the damage caused, thus, metal-based electrodes should not be used for treatment. Considering pH values and damage to the electrodes,

interrupting the electric current at regular intervals is one of the methods used to reduce high temperature and corrosion intensity in the soil. Using anti-corrosion additives can reduce damage for future applications.

Overall, the outcome indicates that EKR is well-suited to reduce the salinity level in soil. The success of the applicability is site-dependent, thus the electrode material and the current to be applied should be chosen accordingly. In addition, as a further study, before and after the remediation of the area DC resistivity data can be collected to evaluate how successful the remediation is.

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Mineralogical examination of biomass ashes: a preliminary study for potassium enrichment and phosphoric acid (H₃PO₄) production

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Research Article

Keywords:

Alkaline Leaching,
Biomass Ash,
Langbeinite, Phosphoric
Acid, Potassium Sulphate.

ABSTRACT

In this study, biomass fly ash was investigated and determined by XRD (XRay Diffractometer) to be predominantly langbeinite, apthitalite, sylvine, apatite and hydroxyapatite. An alkaline leaching study was carried out on the material in a glass reactor at l/s ratio 4 temperature 60°C at pH 12.85 and after drying was analyzed by ICP (Inducted couple plasma). The crystal structure composition of K₂SO₄ 72.28% and potassium content of 33.47% (w/w) was recovered. The elemental transformations in this leaching process were: K 74.16%, Mg 0.55% was found to be 0.8%. It was found that a minimum of 4.84 g H₂SO₄ per 100 g fly ash was required to precipitate the Ca(OH)₂ migrated into the liquid solution and the overall leaching efficiency was found to be 29.85%. In the alkaline leaching process, potassium was found to be easily recovered from each biomass ash. By adding 40 g H₂SO₄/100 g biomass waste remaining after leaching, a phosphorus recovery of 74.9% was observed. In this preliminary study, unsatisfactory results were obtained in obtaining the industry standard H₃PO₄. The reason for this is that the Al, Mg and K in the fly ash are subject to selective dissolution as a result of working at high pH in the alkaline leaching process.

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1. Introduction

Biomass ashes contain large differences in classification and structure compared to coal-based thermal power plant ashes. Currently, the ASTM C618-19 (2019) standard is used for this purpose. In order to classify the origin and structure of biomass ashes, the use of a triangular diagram based on the K₂O, CaO and SiO₂ w/w contents as a 3-phase diagram has been recommended (Zhai et al., 2021).

Biomass ashes can be accepted as secondary phosphorus and potassium sources due to their high phosphorus and potassium contents. In recent years,

the number of thermal power plants with different designs suitable for biomass combustion has increased rapidly. A new alternative material has emerged to meet the limited phosphate and potassium requirements due to the structure of these ashes. In the characterization study of biomass ashes by Çötelİ and Karahan (2023), chemical analyses were carried out and it was found that the potassium in the ash was completely soluble in water and had a highly alkaline character.

Potassium is found in many igneous rocks such as feldspar (potassium aluminum silicate), KAlSi₃O₈ (leucite) and mica, KH₂Al₃ (SiO₄) three. As these

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rocks break down and dissolve, potassium mixes with the soil and water. Potassium chloride deposits are found in almost all salt deposits associated with sodium chloride. Some important potassium minerals are leucite, KAlSi_2O_6 ; glauconite (a complex silicoaluminate structure of varying composition); sylvine (KCl), carnallite ($\text{KCl}\cdot\text{MgCl}_2\cdot 6\text{H}_2\text{O}$), langbeinite ($\text{K}_2\text{SO}_4\cdot 2\text{MgSO}_4$) and polyhalite ($\text{K}_2\text{Ca}_2\text{Mg}(\text{SO}_4)_4\cdot 2(\text{H}_2\text{O})$) (Patnaik, 2002).

Although Krishnamurty et al. (2008) stated that potassium carbonate (K_2CO_3) is predominantly found in wood ash, the study of the calcination temperature in thermal power plants and the almost complete absence of carbon content in the ash suggest that it is present in different forms. In order to recover potassium, its mineralogical structure must be clearly identified. Currently, the biomass ash waste potential in our country is around 600 tonnes/day, which corresponds to 72 tonnes of K_2O /day. An alternative way to solve our country's potassium problem has been opened, mainly through good national management planning of existing material resources. Although some small private companies are still working on it, industrial adaptation and economic production cannot be achieved.

Although there is no sodium salt problem in our country in terms of reserves and production, there is no discovery and production of potassium ore and salt due to the geological structure of our country. A long time ago, especially in the 1980s, studies were carried out on the production of potassium chloride from İzmir Çamaltı Brine Salt Works, and although there were studies aimed at obtaining potassium from some clay minerals (such as potassium feldspar), studies were carried out to meet satisfactory consumption. ALKİM (2023) stated in its annual report that it uses imported potassium chloride and domestically produced crystalline sodium sulphate in the production of potassium sulphate. Russia and Belarus supply approximately 40% of the global potassium chloride market and are the countries closest to us logistically. The potassium sulphate capacity of the Alkim (Alkim Alkali Chemistry Company) - Dazkırı, Koralkim plants is 50,000 tonnes/year and K_2SO_4 production is done with H_2SO_4 via imported KCl . Until now, it has not been possible to produce enough different

potassium salts to meet the import requirements in terms of potassium mining. The country's industry meets all its potassium needs through imports.

Furthermore, if we look at the import data of TÜİK (Turkish statistical institution, 2022), it can be seen that the foreign exchange from the fertilizer sector has reached \$2.7 billion. Two of the main inputs to the fertilizer industry are the output of potash and phosphate mining.

In Burke (2021), ash management is important due to the increased use of biomass for energy production; burning unprocessed biomass in modern furnaces can produce small amounts of ash containing negligible persistent organic pollutants, and land application is possible due to low concentrations of contaminant metals. He noted that agricultural residue ashes contain high levels of potassium and useful phosphate, so could potentially be used as fertilizer. This highlighted the importance of recovering potassium from ash.

Although biomass has many uses in conventional agriculture, such as direct green manuring, composting and use for organic matter needs, small-scale thermal power plants for electricity generation have begun to grow rapidly as a result of the rapid increase in per capita energy consumption. The resulting fly ash has created an environmental and bio-waste and ash management challenge. Fly ash contents are never similar to coal based thermal power plant ashes and as an alternative secondary source of phosphorus and potassium for industrial waste recovery, studies on its use, recovery and product development have accelerated within the scientific framework.

In this study, the recovery of potassium compounds from biomass ashes from thermal power plants by dissolving them in water, their technological economisation, the outline of the process, their partial purification from impurities (such as Ca , Na) and their valorization in terms of mining as a 2nd stage industrial product (a mixture of K_2SO_4 , KCl , NaCl and Na_2SO_4) are planned. The remaining solid valorized waste was studied to obtain H_2SO_4 and H_3PO_4 . The aim was to develop a suitable process for economic production by determining potassium recovery and yield. The main objective is to obtain high value added products from

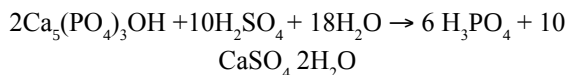
the biomass ashes consisting of these agricultural waste fertilizers with special status. A feasibility study for industrial economic production was carried out.

2. Literature Review

Weast and Astle (1980) stated that potassium is difficult to dissolve due to its crystal structures being bound to other minerals and that it occurs in nature mainly as sylvine, carnalite, langbeinite and polyhalite minerals and is found in small areas in Germany, New Mexico, California, Utah etc. Particularly large potash resources are found in Saskatchewan at a depth of 3000 ft. It is also found in sea water and in salt deposits.

Civelekoğlu et al. (1987) stated that the main minerals of potash salt production are K and Al silicates which are insoluble in water and that potash salts are refined from water soluble and potassium containing solutions by methods such as dissolving and cooling in hot (KCl, from sylvine) or decomposition (KCl, from carnallite) with NaCl and MgCl₂ salts and that the basis of this process is understandable 2-3 phase diagrams.

Krupa et al. (2008) also carried out a study on the production of phosphoric acid on bone meal ash, which has the mineral structure of hydroxyapatite.



Bone meal was subjected to calcination at 600°C. 43% H₃PO₄ solution was formed after reacting with the ash and adding unreacted jibs. They were reacted at 90-95°C and H₂SO₄ was added, resulting in a product that contained 45% H₃PO₄. The solid part was separated, and by adding steam to remove some water vapor, commercial phosphoric acid containing approximately 75% and 54% P₂O₅ was obtained (Figure 1).

Zhai (2022) stated that the common mineral phases are calcite (CaCO₃), fair calcocite [K₂Ca (CO₃)₂] and arcanite (K₂SO₄) based on the XRD raw spectra of different biomass ashes. All of the wood ash burnt in the laboratory is dominated by the calcite phase, with small amounts of Arcanite and hydroxyl apatite [Ca₅ (PO₄)₃ (OH)]. This ash contains some calcite, arcanite, larnite (Ca₂SiO₄) and polyhalite

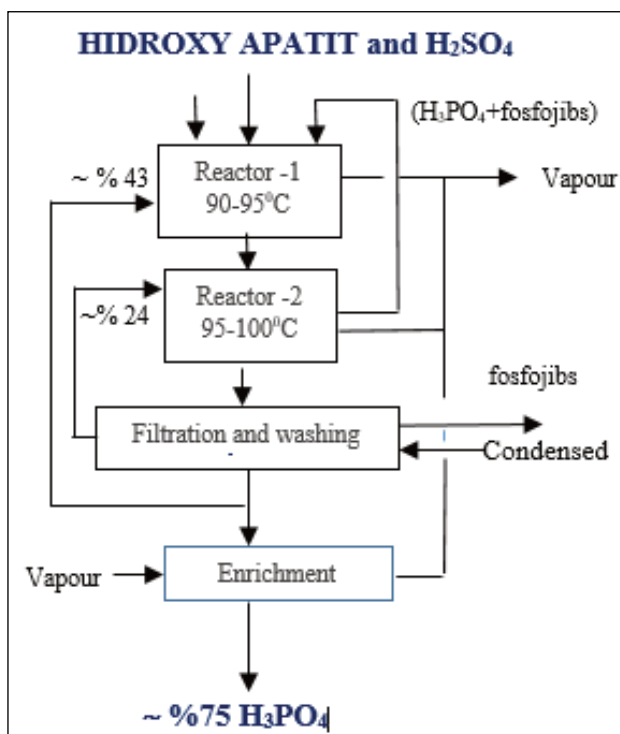
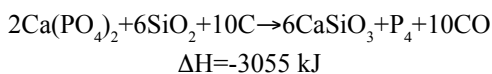


Figure 1- A brief representative description of the industrial production of phosphoric acid, from calcinated bones (Krupa et al., 2008).

[K₂Ca₂Mg(SO₄)₄·2H₂O]. In the example of industrial wood ash, quartz (SiO₂) and calcite are present. Lime (CaO) also contains some kalsilite (KAl₂SiO₄), gypsum (CaSO₄·2H₂O) and some periclase (MgO) and sodium, calcium silicate (Na₂Ca₃Si₂O₈). Archonite and sylvine (KCl) have also been identified. Although the rice husk ash sample also consists mainly of SiO₂, the main mineral phase of cristobalite contains less quartz, while in another sample it consists mostly of quartz, and in both samples, there is some amorphous content of sylvine, arcanite and less kalsilite (KHCO₃). Minerals detected in XRD analyses in biomass ash samples, depending on the ash source; arcanite (K₂SO₄), calcite (CaCO₃), cristobalite (SiO₂), fairchildite [K₂Ca(CO₃)₂], hydroxyapatite [Ca₅(PO₄)₃(OH)], calcilite (KAl₂SiO₄), kaolinite [Al₂Si₂O₅(OH)₄], larnite (Ca₂SiO₄), lime (CaO), periclase (MgO), polyhalite [K₂Ca₂Mg(SO₄)₄·2H₂O], silica (SiO₂), sodium calcium silicate (Na₂Ca₃Si₂O₈) and sylvine (KCl).

Regarding phosphorus recovery, Becker (1989) explained the fundamentals of phosphorus recovery economics, the differences between processes, how wet and dry phosphoric acid production differs, and how thermal processes vary based on the material structure. Although there are project studies called RecoPhos using a carbon-lime mixture at 1300-1900°C thermally, it is not thought to be as economical as the wet process. It is difficult to work due to the difficulty in reaching the temperature. Trial production is still ongoing, and large-scale production seems only possible by obtaining H₃PO₄ through the wet process. It seems to be an alternative way to obtain high purity phosphoric acid with low heavy metal contamination. This can be especially useful in food applications. Additionally, biomass ashes are a cleaner material than natural waste sludge and rock phosphate deposits. In this case, the mineralogical structure of the material is key. The process called Recophos is not actually a new process, but is a modified version of the electric furnace process (Austin, 1985), which was developed as an alternative for use on rock phosphates in the 1920's in case of cheap electricity supply.



Donatello and Cheeseman (2010) gave an empirical formula for P extraction from waste sludge ashes, as in Franz (2008).

Amount of g H₂SO₄ per 100 g material:

$$(1.479 \cdot \text{CaO} + 0.962 \cdot \text{Al}_2\text{O}_3 + 0.614 \cdot \text{Fe}_2\text{O}_3 + 2.433 \cdot \text{MgO} + 1.582 \cdot \text{Na}_2\text{O} + 1.041 \cdot \text{K}_2\text{O}) - (0.691 \cdot \text{P}_2\text{O}_5 + 1.225 \cdot \text{SO}_3 \%)$$

The empirical approach is actually similar to Nunn and Dee (1954). Provided that the liquid/solid ratio was 20, it was dissolved with H₂SO₄ solution at a concentration of 0.19 mol/l for 120 minutes, and then the liquid part of the filtered mixture was passed through cation exchange resin and analyzed for Zn, Fe, Mg, Ca, Al, P and SO₄²⁺. The final product had a concentration of approximately 1% H₃PO₄ and could reach 85% H₃PO₄ concentration by vacuum distillation.

Raupenstrauch (2015) explained the RecoPhos process for obtaining H₃PO₄ from industrial city biomass waste ashes. With 80% waste sludge ash (SSA) (8% P content) material at a feeding rate of 10kg/h, 10% C and 10% lime mixture, the P recovery in the melting furnace operating at 1900°C increased to 90%, and the heavy metals in the ash were eliminated. He explained that H₃PO₄ could be produced by extracting the gas phase in water.

Lijian et al. (2019) stated that biological wastes such as meat and bone meal and poultry manures are used as energy sources in industrial facilities, but the phosphorus in the biomass ashes remaining after combustion is thrown away without being used, and to encourage their use, biomass ashes from industries are highly alkaline with pH values as high as 13. The content is high in phosphorus and calcium. X-ray powder diffraction (XRD) reveals hydroxyapatite and potassium sodium calcium phosphate. Nearly 90% of phosphorus can be removed by acid extraction (H₂SO₄); optimized acid consumption is 3 (they determined that it was 2–5.3 mol H⁺/mol P). Nearly 90% of phosphorus can be removed by acid extraction (H₂SO₄); optimized acid consumption is 3 (they determined that it was 2–5.3 mol H⁺/mol P). Nearly 90% of phosphorus can be removed by acid extraction

(H_2SO_4); optimized acid consumption is 3 (they determined that it was 2–5.3 mol H^+ /mol P).

Gowariker et al. (2009) explained that the ash produced by burning wood is rich in potassium (in the form of K_2CO_3) and is useful for correcting the pH of acidic soils, is also explained the Mannheim process, which is the production of K_2SO_4 from KCl. This process is a 50% K_2O process in which KCl reacts with H_2SO_4 , yielding excess KHSO_4 , KCl and HCl as a gas.

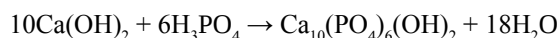
Schultz et al. (2000) described the production of potassium fertilizer from the natural complex salts kainite ($\text{KCl} \cdot \text{MgSO}_4 \cdot 3\text{H}_2\text{O}$) and langbeinite ($\text{K}_2\text{SO}_4 \cdot 2\text{MgSO}_4$) and Carpathian poly-minerals. In the process, kainite is leached to form schonite ($\text{K}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 6\text{H}_2\text{O}$) and MgCl_2 is separated. He stated that schnite is separated into K_2SO_4 and MgSO_4 by leaching in the same way.

Wang et al. (2016), potassium recovery by leaching method on an ash sample containing 39.44 mg/g volatile matter, 158.92 mg/g fixed carbon, 51.64 mg/g K and 28.56 mg/g Mg. In the leaching process with H_2SO_4 , the pH was adjusted to 6.1, the liquid/solid ratio was 3 and the temperature was 75°C . At the end of 30 minutes, the pH increased to nine and a K recovery efficiency of 86.4% was achieved. In his study, he observed that the solubility of potassium is independent of pH and that low pH leads to the formation of CaSO_4 , which causes pH collapse. In addition, the world's potash resources are limited and potassium dolomite deposits (8.21%) could be an alternative. This process involves calcining the material ground below 74 microns with Na_2CO_3 at 840°C , leaching with water and CO_2 for 2 hours at a l/k ratio of 8-10, evaporation and crystallization, and K_2CO_3 .

Akgül and Yoncacı (2021) gave different classifications on the classification of the thermal power plant producing fly ash and its classification is siliceous (S), silicocalcic (CS), ferrosilicic (FS) and ferrocalcic (FCS). In this classification, he also stated that they are categorized into three dominant tendencies such as high acid (HA), medium acid (MA) and low acid (LA) according to the sum of Si, Al, K, Ti and P oxides.

Li et al. (2015) found that aluminum and iron phosphates, which are difficult to use at high temperatures, react with lime (CaO) in the environment at different temperatures to the apatite form $\text{Ca}_3(\text{PO}_4)_2$, from which phosphorus recovery is easier. He explained that aluminum and iron are oxidized as Al_2O_3 and Fe_2O_3 and phosphorus is bound to Ca in the form of apatite.

Çağan (2014) stated that the general formula of apatite is $\text{X}_{10}(\text{TO}_4)_6\text{Z}_2$, and instead of X, Ca, Sr, Pb, Cd and Ba; P and As instead of T; OH, F, Cl can be entered instead of Z.



According to its reaction, it is possible to produce it in pure form. Hydroxyapatite has high biocompatibility; he stated that not only can the body easily accept it, but it can also create a suitable environment for the growth of bacteria, which are other biological structures, and can be used especially in dental implants.

Karabacak (2021) revealed some commercial processes for phosphorus recovery from waste sludge ash by listing the principles and effects/products. The material he works on is BUSKİ samples and the ash content is P_2O_5 17.36-11.49% in two different samples; K_2O content is between 3.70-3.16 and used H_2SO_4 solution at different concentrations to transfer the phosphorus in the ash to the solution.

In KTÜ (2017) and Tor (1991) by adding liquid ammonia (NH_4OH or $(\text{NH}_4)_2\text{S}$) to a solution containing Fe^{2+} , Fe^{3+} , Al^{3+} , Cr^{3+} , Mn^{2+} , Ni^{2+} , Co^{2+} , Ba^{2+} , Ca^{2+} , Mg^{2+} , Na^+ , K^+ , It has been found that $\text{Al}(\text{OH})_3$, $\text{Cr}(\text{OH})_3$, $\text{Fe}(\text{OH})_3$, MnS , NiS , CoS , FeS , ZnS precipitate while Ba^{2+} , Ca^{2+} , Mg^{2+} , Na^+ and K^+ remain in solution.

Millsaps et al. (2021) determined the required H_3PO_4 ratio in a collaborative study involving acidification with H_3PO_4 for the production of phosphorus fertilizer from poultry manure, the total and water-soluble P_2O_5 content of the final product, and the granulation process. In the ideal study, 0.6 kmol H_3PO_4 per 100 kg ash was found. However, the reaction time is very short and it is stated that the granulation process of the paste material should be started immediately within a maximum of 4 minutes.

As stated by Bogusha et al. (2018), this is an increasing problem due to the rising number of biomass power plants in numerous countries. It is therefore essential to be comprehensively characterized biomass ashes, with particular focus on the elemental composition and solubility of nutrients (e.g., K and P) and contaminants. Furthermore, these residues can be utilised for soil nutrition. All biomass ashes studied were alkaline and exhibited high concentrations of P, K and Ca, which are essential for their potential utilisation. He stated that it contained the following: Phosphorus is present in: 1) Bottom ash contains apatite and other phosphates, including potassium hydrogen phosphate and potassium iron phosphate, which are produced when poultry litter is incinerated. 2) It is reported that this material originates from poultry litter incineration and contains potassium, sodium, calcium phosphate. Potassium is mainly present as sylvine, arcanite and some phosphates. Phosphorus, calcium and magnesium are difficult to leach with water, and recovery of potassium and phosphorus from these materials must be done carefully. It has been documented that this is a viable method.

Sof et al. (2021) and Çöteli and Karahan (2023) also studied the method of acidifying the material with H_2SO_4 in the production of PK and NPK organic and inorganic fertilizers in the fertilizer sector. The basis of the developed production method is the complete acidification of the phosphorus in the ash to make it soluble in water. The main purpose of the study is to use the existing material in the inorganic and organic fertilizer sector.

Çöteli and Karahan (2023) investigated the chemical composition of the ashes of the biomass thermal power plants installed in Turkey as a function of time and found that the ashes of the biomass thermal power plants had a chemical composition as shown in Table 1.

Puspita and Susanto (2021) also worked on the development of a new technique for the recovery of potassium salt from biomass ash extraction solution containing a mixture of KCl, NaCl, $MgCl_2$ and $CaCl_2$. The process involves partial evaporation of the solution, followed by the addition of ethanol

and further evaporation. In their study, they showed that KCl can be recovered in a different way than in traditional studies.

Table 1- A chemical characterization of Turkish biomass thermal power plant fly ashes (Çöteli and Karahan, 2023).

	Average%	Max %	Min %
Al_2O_3	4.75	16.97	2.88
CaO	16.05	19.24	10.57
Fe_2O_3	1.94	7.03	0.32
K_2O	12.75	27.0	2.94
MgO	6.56	11.50	1.19
Na_2O	6.47	27.38	4.79
P_2O_5	12.38	19.53	4.98
	mg/kg		
As	15	27	3
Cd	1	3	0
Co	8	25	3
Cr	121	995	16
Cu	432	1024	141
Pb	232	691	14
Zn	950	3997	100
Note:			
1-Ash samples have a highly alkaline character and the pH (1/10) is in the range of 11-13 depending on the sample.			
2-1 Valent cations and anions are completely soluble.			
3- P_2O_5 , it is 0.5-1.5% water-soluble, depending on the cation bounds to.			

3. Technological Studies

3.1. Material Used

The sample (fly ash) (Figure 2) obtained from the 20 MW installed capacity fluidised bed thermal



Figure 2- Biomass fly ash studied.

power plant located in Sakarya-Akyazı district has a particle size completely below 50 microns and the sample, which is believed to have been calcined (containing CaO, MgO, etc. material) due to the high temperature at the outlet of the plant, was placed in a closed package. The plant burns more than 50% of agricultural waste, in particular poultry waste and poultry manure.

3.2. Chemical, XRD and SEM (Scanning Electron Microscope) Analyses

The results of the ICP analysis are given in Table 2 after dissolution in aqua regia. It is a material with the status of the second source of potash and phosphate.

The XRD analyses were carried out on the Bruker-D-8 Advance, the values obtained from the XRD diffractogram were evaluated in the “HighScore Plus” software and the mineral structures contained therein were determined.

The powder sample to be examined on the FEI Inspect F50, FEG-SEM instrument was subjected to SEM-EDS (Elemental Analysis in scanning electron microscope) analysis after gold-palladium coating. The results of these analyses were SEM photographs and EDS graphs showing the elements contained.

The analyses of fly ash using SEM+EDS at the 1st point range, at the other 2 points and as an average are given in Table 3 as percentages. The ICP analyses were compared.

Table 2- Chemical composition of the studied fly ash material.

ELEMENT	VALUE	ELEMENT	VALUE	ELEMENT	VALUE
Al %	2.00	As ppm	15.49	Mn ppm	3069
Ca %	15.81	Ba ppm	258.61	Ni ppm	60
Fe%	1.09	Cd ppm	1.60	Pb ppm	19
K%	13.47	Ce ppm	10.11	Sb ppm	4
Mg%	4.33	Co ppm	7.19	Sr ppm	382
Na%	2.06	Cr ppm	39.24	V ppm	45
P%	4.93	Cu ppm	475.35	W ppm	39
S%	6.50	La ppm	6.99	Zn ppm	2880
				Zr ppm	32

Table 3- Chemical analysis of fly ash at the marked point by SEM-EDS.

ELEMENT	Point 1	Point 2	Point 3	Average	ICP
	% w/w				
Al	1.10	1.07	1.13	1.10	2.00
Ca	14.12	14.97	13.89	14.32	15.81
Cl	6.48	0.60	6.53	4.53	-
Fe	0.90	0.73	1.30	0.97	1.09
K	21.33	14.51	21.63	19.16	13.47
Mg	3.14	3.43	3.26	3.27	4.33
Mn	0.10	8.22	0.11	2.81	0.3
Na	4.31	8.31	3.94	5.52	2.08
O	34.19	36.31	34.02	34.84	-
P	4.70	12.21	4.82	7.24	4.93
S	7.61	6.33	7.41	7.12	6.50
Si	2.02	1.52	1.95	1.83	-

3.3. XRD Analysis on the Material and Pointwise (at the Marked Point) Mineralogical Composition Determination with SEM-EDS

As a continuation of the study, the crystal structure of Ca, K, Mg, P, compounds was determined by XRD to determine the mineral structure of the material.

When biomass ash (Figure 3) is subjected to XRD analysis, the main structure contains potassium minerals; as, in ascending order, sylvine (KCl), apthilatate ($\text{Ca}_3\text{NaO}_8\text{S}_2$), ($\text{K, Na}_3\text{Na (SO}_4)_2$, langbeinite [$\text{K}_2\text{Mg}_2(\text{SO}_4)_3$] and a very small amount of $\text{K}_4[\text{Fe (CN)}_6]\cdot 3\text{H}_2\text{O}$ in its aqueous form were observed. In addition, Ca(OH)_2 , CaO and SiO_2 contents are also present and the ash structure has indeed reached a new mineralogical structure in the form of calcination. In terms of phosphorus, the dominant structure is apatite $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ and hydroxyapatite.

After the determination of the main minerals in the biomass ash by XRD, an elemental mass balance was carried out for the elements Ca, Fe, Al, Mg, P, K, Na, S, with an acceptable margin of error (oxygen (O), hydrogen (H) and carbon (C) analysis was not carried out, it was considered as an equation with several unknowns (matrix solution) and the ratio of components and minerals in the material was determined as (w/w) using an Excel spreadsheet (Table 4).

Patnaik (2002) gave these compounds of Ca, Mg, Na and K collectively with their mineral names and CAS numbers (Table 5).

XRD analysis of the material indicated that potassium and magnesium compounds were predominantly in the form of langbeinite and sylvine whereas phosphorus was in the form of apatite and hydroxyapatite.

4. Experimental Studies of Phosphorus and Potassium Recovery and Enrichment

In terms of potassium recovery in the study, the principle that II-valued cations (except Al and Si) are insoluble in alkali, but K, Na compounds and I-valued anions are soluble in water (Weast and Astle, 1980; Green and Southard, 2019). The purpose of this is to obtain a cleaner solution as Ca, Fe, Cu, Zn, P and Mg do not enter the solution.

Firstly, the alkaline leaching under normal conditions; (120 min) l/s ratio of 5 and 10 was observed and visual difficulties were observed, pH control and then the elemental balance, which could be adapted to the industry by increasing the temperature, and the possible yield and final product composition were tested.

In the production of phosphoric acid, it was determined how much phosphorus content PO_4^{3+} could be recovered by reacting the purified waste product with K, adding the necessary $\text{H}_2\text{O} + \text{H}_2\text{SO}_4$ and filtering out impurities such as phosphoric gypsum.

4.1. Potassium Recovery Studies

Using the elemental solubility tables given in Green and Southard, (2019) it was investigated what

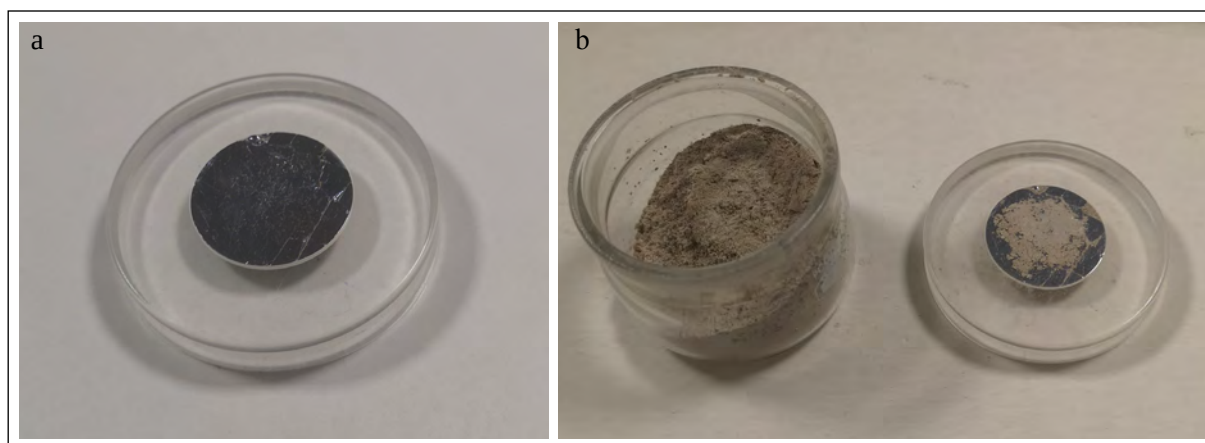


Figure 3- Sample preparation before XRD analysis of biomass ash, a) sample prepared for XRD, b) together.

Table 4- Determination of approximately chemical composition in fly ash according to XRD and ICP analysis results³.

COMPONENT	MW	QTY	%	Al	Ca	Fe	K	Mg	Na	P	S	Ref
Al ₂ O ₃	101.96	4.00	4.00	2.00								Krupa et al., 2008
Ca(OH) ₂	74	1.35	1.35		0.73							
Ca(PO ₄) ₂	230	1.62	1.62		0.28					0,44		
Ca ₁₀ (PO ₄) ₆ (OH) ₂	1004	22.20	22.20	0.00	8.84	0.00	0.00	0.00	0.00	4,11	0.00	
Ca ₅ (PO ₄) ₃ (OH)	502	2.00	2.00		0.80					0.37		
CaO	56	7.20	7.20		5.14							
Fe ₂ O ₃	160	1.56	1.56			1.09						Krupa et al., 2008
K ₂ Mg ₂ (SO ₄) ₃	414	9.00	9.00				1.70	1.04			2.09	
K ₂ SO ₄	174	10.00	10.00				4.48				1.84	
K ₄ [Fe(CN) ₆].3H ₂ O	368.35	~	~									
KCl	74.5	12.44	12.44				6.51					
MgCl ₂	95	8.00	8.00					2.02				
MgSO ₄	120	1.50	1.50					0.30			0.30	
Na ₂ SO ₄	142	1.00	1.00						0.32		0.32	
NaCl	59	4.45	4.45						1.73			
NaKMg ₂ (SO ₄) ₃	398	8.00	8.00				0.78	0.96			1.93	
SiO ₂	60	5.68	5.68									Çöteli and Karahan, 2023
Σ		100.00	100.00	2.00	15.80	1.09	13.47	4.33	2.06	4.92	6.48	
ICP Analyses results % (w/w)				2.00	15.81	1.09	13.47	4.33	2.06	4.93	6.50	

Note: ¹Krupa et al. (2008) stated that Fe and Al phosphates decompose into Fe₂O₃ and Al₂O₃ at high temperatures.

²Silicon analysis was not performed in ICP, only the % SiO₂ amount from XRF analysis was taken for the 5.68% for 100 balances in Çöteli and Karahan (2023).

³Possible compounds of 3Al and Fe in the silicate structure and potassium ferrocyanide were not taken into account, assuming that they were below 1%.

Table 5- Important minerals of potassium (Patnaik, 2002).

MINERAL NAME	FORMULA	CAS NO
Carnallite	KCl•MgCl ₂ •6H ₂ O	1318-27-0
Feldspar	KAlSi ₃ O ₈	68476-25-5
Kainite	4KCl•4MgSO ₄ •11H ₂ O	67145-93-1
Langbeinite	K ₂ SO ₄ •2MgSO ₄	13826-56-7
Mica	KH ₂ Al ₃ (SiO ₄) ₃	12001-26-2
Leucite	KAlSi ₂ O ₆	1302-34-7
Leunite	K ₂ SO ₄ •MgSO ₄ •4H ₂ O	15226-80-9
Polyhalite	K ₂ SO ₄ •MgSO ₄ •2CaSO ₄ •2H ₂ O	15278-29-2
Sylvine	KCl	7447-40-7

type of chemical structure was formed as a result of the dissolution of the material in an alkaline environment, as II-valued cations do not dissolve in alkali.

Experiment performed; primarily for observational purposes, in a glass reactor; liquid volume kept constant at 2000 ml at a speed of 500 rpm (number of recycle per minute for mixing unit) at a temperature of 20°C with turbine mixing measurements (Da/Dt=1/3, E/Dt=1/3, Da/Dt, Da=Blade width of turbine mixer blade, Dt=Diameter of stirrer reactor, E=Height of the mixer from the bottom) in (McCabe et al., 2005). Two experiments were performed with l/s [Ratio of liquid (weight) per solid (weight)]= 5 (2000 ml/400 g sample) and 10 (200 g sample per 2000 ml). The retention time was as long as possible, but not longer than 2 hours. After 1 hour the solid phase settled to the bottom. Although the settling time is short due to the small particle size, the use of a hydrocyclone, which is suitable for separating solid particles between 5-10 microns (Sinnot, 1993), should be preferred in the pilot process stage (Figure 4).

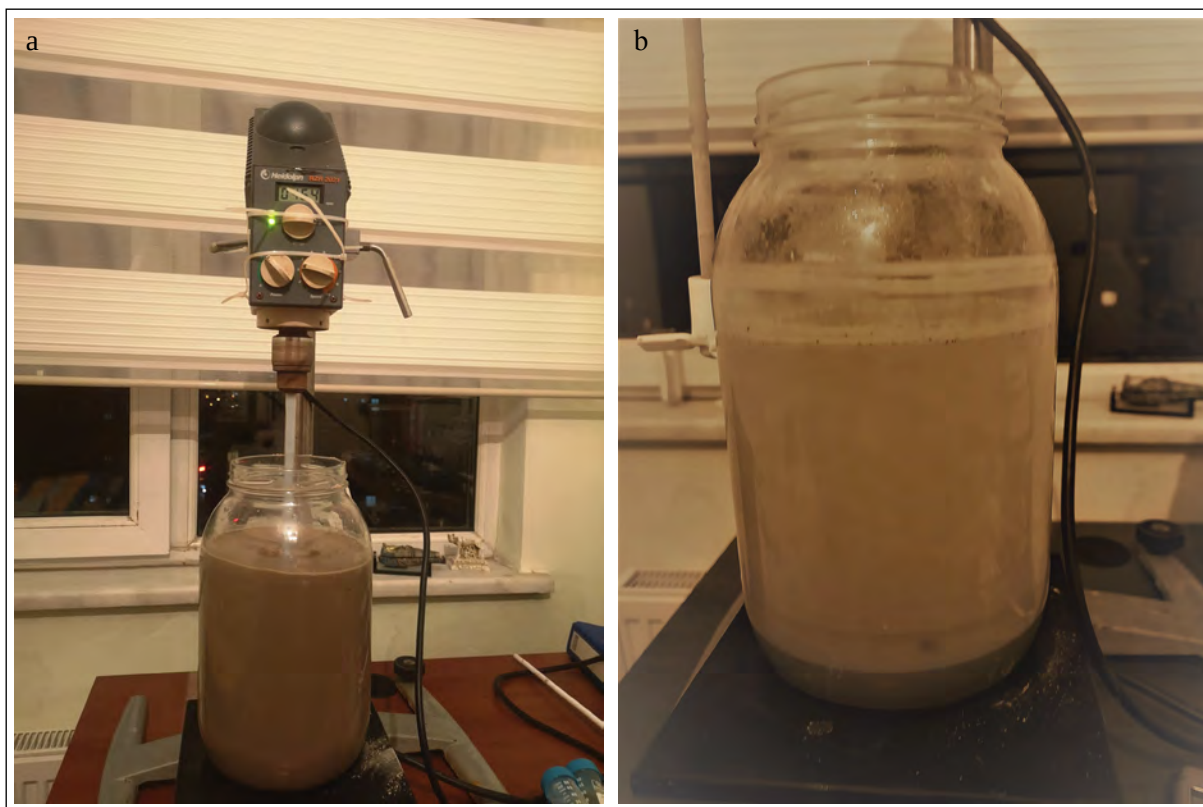


Figure 4- Laboratory studies, a) alkaline leaching studies on the material, b) resting and sedimentation phases.

In such a study, when the l/s ratio is 4, good mixing cannot be achieved below 500 rpm. It was found that 500-650 rpm is suitable.

In addition, as a final experiment, the liquid part was taken with a syringe as a result of the study carried out at 60°C with a l/s ratio of 4 (500 ml pure water/125 g material) and the solid phase (insoluble) accumulated at the bottom was washed 5 times with pure water and rested. It was dried at 105°C to observe the change in weight (Figure 5). The colour of the compounds that pass into the liquid on drying is light pink, close to the colour of natural langbeinite.

$$\eta\% = 100 \frac{\text{amount passing into liquid (g)}}{\text{total ash (g)}} 29.85$$

29.85% was found to be obtained from volatile biomass ash.

An almost pure K_2SO_4 crystal was observed at a random point marked in SEM-EDS (Figures 6a, b and 7) according to the results of the leaching study.

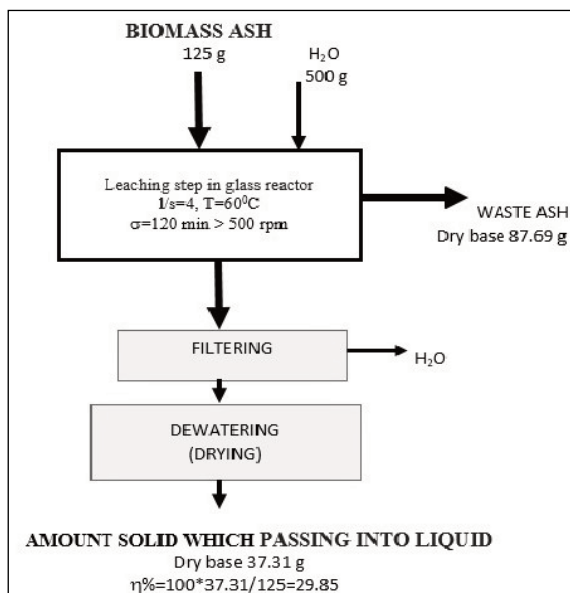


Figure 5- The leaching process.

The % gain of K: $\eta\% = 100 \times 12.49 / 16.84 = \% 74.16$ for Mg, $\eta\% = 100 \times 0.03 / 5.41 = \% 0.55$ has been found. The chemical composition of this structure was determined from Table 6 with acceptable elemental balance.

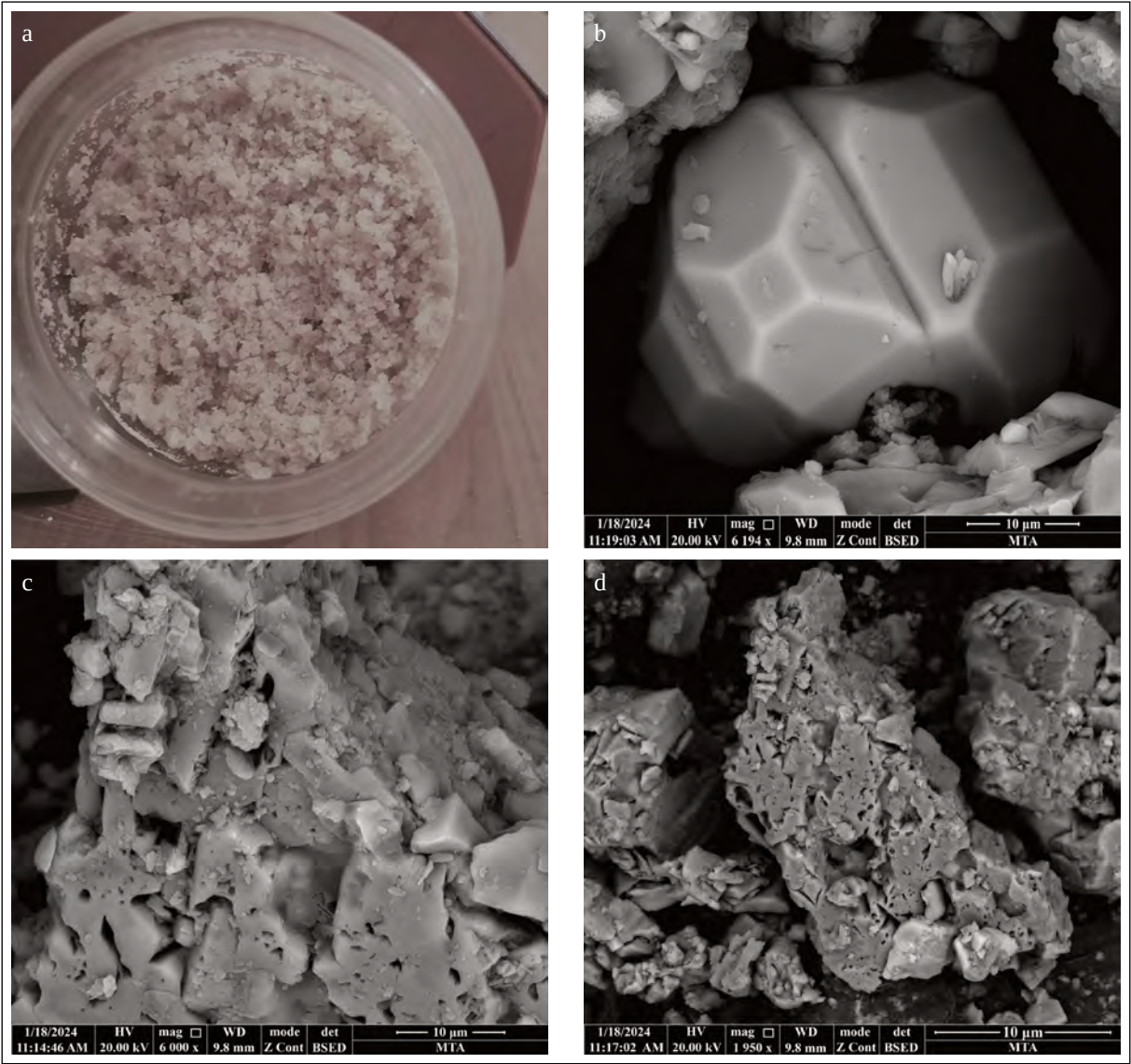
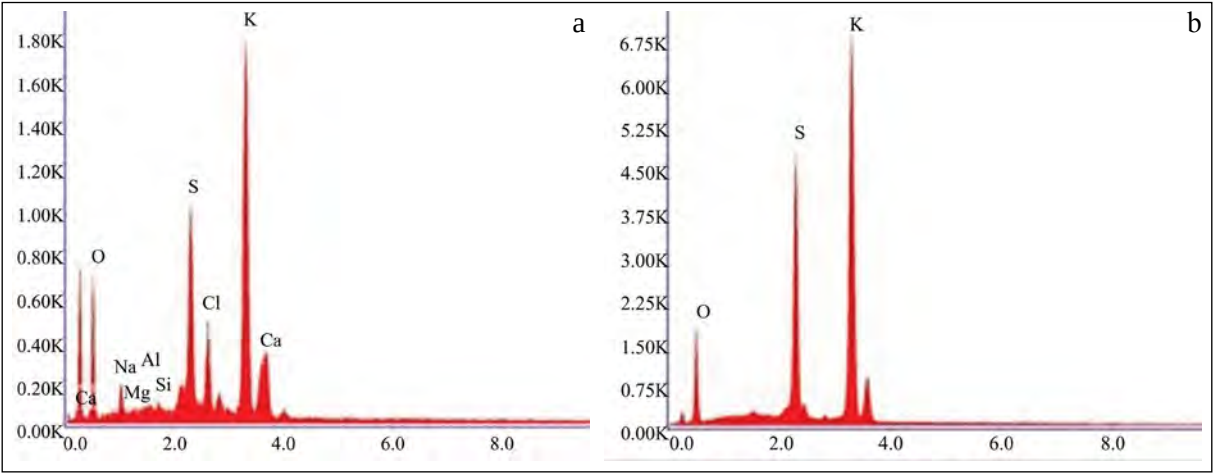


Table 6- ICP results and elemental balance according to gravimetric inflow and outflow in the leaching environment.

	INPUT FLY ASH (1)		OUTPUT WASTE (2)		PRODUCT (3)		(1)-(2+3) = ~0 (g)
	% w/w <i>icp</i>	In 125 (g)	%w/w <i>icp</i>	In 87.69 (g)	%w/w <i>icp</i>	In 37.31 (g)	
Al	2.00	2.50	2.80	2.46	0.05	0.02	0.03
Ca	15.81	19.76	22.10	19.38	6.79	2.53	-2.15
Fe	1.09	1.36	1.51	1.32	0.02	0.01	0.03
K	13.47	16.84	4.95	4.34	33.47	12.49	0.01
Mg	4.33	5.41	6.15	5.39	0.09	0.03	-0.01
Mn	0.31	0.38	0.42	0.37	0.00	0.00	0.02
Na	2.06	2.58	2.35	2.06	1.14	0.43	0.09
P	4.93	6.16	7.12	6.24	0.02	0.01	-0.09
S	6.50	8.13	2.80	2.46	15.24	5.69	-0.02
Zn	0.29	0.36	0.41	0.36	0.01	0.01	0.00

According to ICP analyses and XRF results, the main components are Ca, K, Mg, Na, Al and S, and with material balance solution, the product composition is thought to be 72.28% K₂SO₄ and also 1.23% KCl as a projection in future studies.

When the main mineral structure was examined, it was found to be aphtilide (K₃NaO₈S₂), sylvite (ClK_{0.7}Na_{0.3}), sylvine (KCl), dolomite (CaMgO₆-CaMgCO₃), Ca(OH)₂ and mainly arcanite (K₂SO₄). Chemical analysis was carried out at the point marked by the SEM, taken from 2 points, and a very large amount of K₂SO₄ and the visual structure (Figure 6b) are given in Table 8.

The post-leaching waste material, defined as Waste-1, was subjected to XRD analysis (Figure 8).

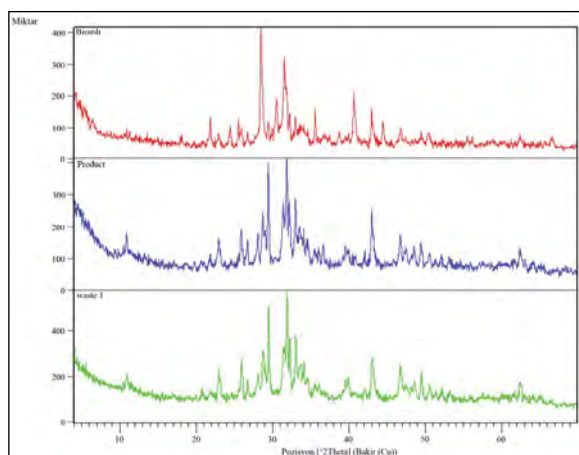


Figure 8- Detection of major minerals in the XRD analysis of the dewatered and dry solid product after leaching.

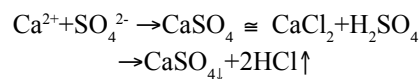
Comparative XRD diffractograms of the biomass ash (bioash), the waste and products in Figure 8. Arcanite crystals can be distinguished in the XRD diffractogram in Figure 9.

Preliminary experimental observations:

-During the study period, the pH of the liquid part was found to be 12.85. It is a highly alkaline work. In addition, a thin layer of lime cream forms on the liquid part. This is probably due to the reaction of CaO with water and Ca(OH)₂ in the sweat on Table 4.

-In particular, the sample with a l/s ratio of 4 was prepared at 60°C with a leaching time of 120 minutes, and when the liquid solution cools to 4-5°C, white crystals form at the bottom. As a result of this observation, it was concluded that it was necessary to clean this calcium-based contaminant structure.

-In the study, it was observed that when the pH was lowered to the 8.5-9 range, the solution became clear and the calcium content precipitated as black, grey crystals as



were observed as possible reactions (Bayram, 2018).

When the resolution table is examined; the solubility ratio between 100°C and 20°C is; K₂SO₄=(24.10/7.40)=2.17, KCl=1.64, MgSO₄=1.79, Na₂SO₄=2.17, NaCl=1.091, MgCl₂=1.34 was concluded that is necessary to use the temperature

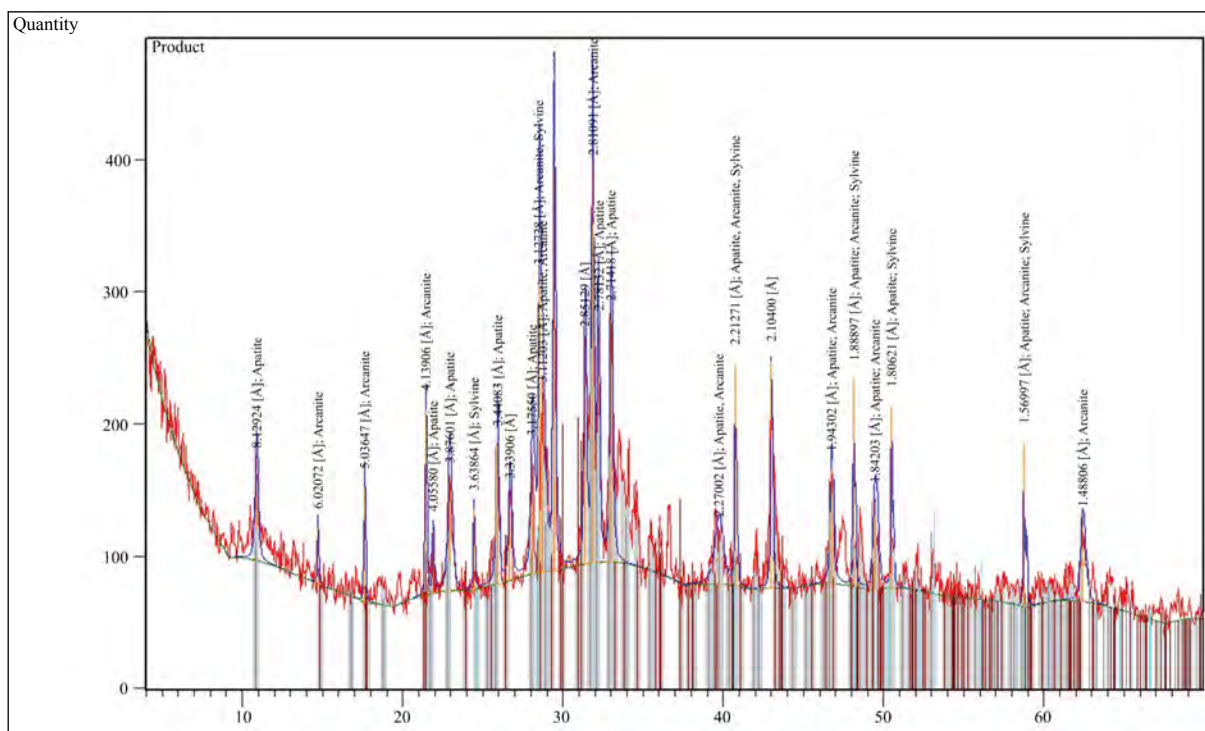
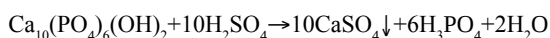
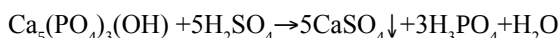
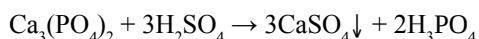


Figure 9- Detection of major minerals in the XRD analysis of the dewatered and dry solid product after leaching.

factor to reduce the l/s ratio for economic purposes. For higher solubility with less liquids, it has been found proper to operate the leaching temperature at about or close to $>60^{\circ}\text{C}$.

4.2. Phosphorus Recovery Studies

The main chemical reactions of wet process H_3PO_4 production are given in (Becker, 1989) and are similar to the reactions according to the apatite structure.



Stoichiometric acid requirement per 100 grams of ash; according to the study of Krupa et al. (2008), the required amount of H_2SO_4 is given on the Table 10.

The study carried out; 40 g of 98% H_2SO_4 per 100 g was added to the material (remaining after leaching). It was well mixed with 35 g of water. The temperature of the material was rapidly increased to 100°C and the addition of 1000 ml of pure water was completed without solidification (with l/s ratio of 10) (Figure 10).

The suspended material was then mixed at a speed of 500 rpm for one hour. As a result of the one-hour rest, solid particles (CaSO_4 + silicates and insoluble) settled to the bottom and a direct sample was taken from the clean solution before ICP. It is the amount corresponding to H_3PO_4 over the phosphorus (P) read in the liquid.

$$\text{H}_3\text{PO}_4 \text{ (mg/l)} = \text{reading (mgP/l)} * (98/31)$$

The maximum that can be increased is H_3PO_4 step for H_3PO_4 production from biomass ash in the light of the studies carried out, it seems possible to produce concentrated dirty phosphoric acid in a continuous system as in Krupa et al. (2008). Only the produced samples of H_3PO_4 are taken from the system. The circulation system is H_3PO_4 solution. It seems necessary to separate $\text{Al}_2(\text{SO}_4)_3$ and other Mg, Na, K compounds for a cleaner phosphoric acid production.

5. Results and Discussion

-Three different processes are used in the world to produce potassium sulphate. These are the Mannheim process, the salt lake potassium sulphate process and the polyhalide (potassium chloride-magnesium sulphate)

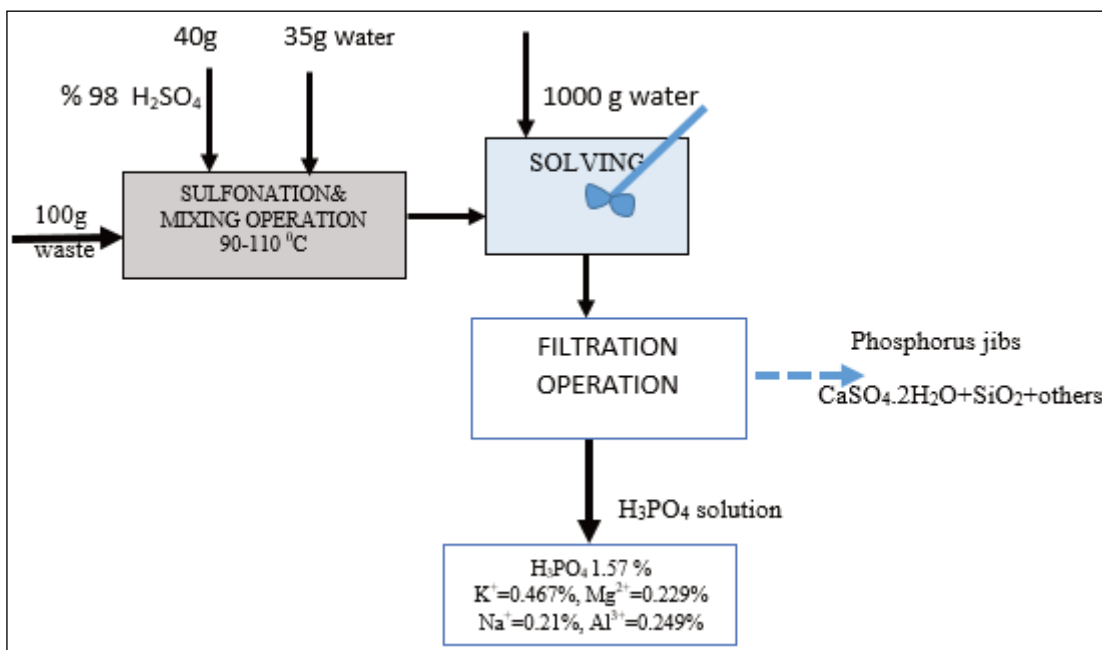


Figure 10- Suggested box-box continuation process.

potassium sulphate production process. The costs of each process are different. The process that produces the highest and best quality potassium sulphate is the Mannheim process. According to Aslan et al. (2020), depending on the process, global potassium sulphate production from saline lakes is approximately \$320/tonne, \$410/tonne for polyhalide and \$520/tonne for KCl and H_2SO_4 using the Mannheim process.

-The largest potash producers in the world are China, Canada and Russia. Potassium sulphate production in China will increase by 8.6% between 2013 and 2018, making it the largest potassium sulphate producer. Russia already has a significant share of the potassium sulphate market. Canada has decided to invest in potassium sulphate production in recent years. The world's largest importers of potassium sulphate are Germany, Belgium and the Netherlands. These three countries account for approximately 60% of the world's total potassium sulphate imports (Aslan et al., 2020).

How this distinction can be made is illustrated by two further studies of the process. As a result of the preliminary studies;

- When the XRD analyses of the materials were examined, it was found that the langbeinite content

on the fly ash was very high, but the dried material obtained at the end of the leaching process had no langbeinite content. There is a very small amount of langbeinite in the waste product.

- It has been observed that economically enriched concentrated potassium salts, which can have a market share, can be produced from biomass thermal power plant ashes by the alkaline leaching method. It is a concentrated potassium ore whose predominant structure is arcanite (K_2SO_4) with K_2O = 40.33% (pure industrial chemical K_2O is 54.02%, S=18.39%, O=36.78%) among the low temperature (even at 60°C) material.

- The reason for choosing the alkaline leaching method is that compounds such as $\text{Mg}(\text{OH})_2$, CaCO_3 , MgSO_4 , CaCl_2 precipitate at high pH in salt water mixtures. The reason for the presence of $\text{Ca}(\text{OH})_2$ in the current product is its alkaline nature. It is possible that this can be precipitated by the addition of H_2SO_4 .

- Solid recovery: 29.85% and K recovery: 74.16%.

- The Mg recovery in the leaching process is 0.55% and this is entirely due to the high pH of the leaching process. The results are in line with our expectations.

- In addition, increasing the leaching temperature to 95-100°C and washing the remaining solids with clean solution prior to centrifugation will increase the recoveries.

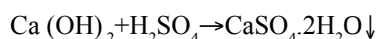
- The recovery of phosphoric acid from the material by the wet process did not appear to be possible within the method followed. An alternative route should be investigated by high temperature or other studies. For example, as in Krupa et al. (2008), mixing the resulting H_3PO_4 with higher concentration clean H_3PO_4 and placing it on the market.

-The tests carried out on the waste were only designed to detect the compounds that had leached into the liquid and did not investigate the new acidic structure that could be formed by dehydration. Since only the chemical structure and the percentage composition are known, as can be seen in Table 11, the percentage H_3PO_4 can reach up to 23.76, which means that this production is not possible. Therefore, purification of the liquid composition by various alternative methods should be considered. For example, the leaching process to be carried out on the waste at pH around 7 or pH 6-7 will largely remove the K_2SO_4 , $MgSO_4$, $FeSO_4$ and $Al_2(SO_4)_3$ content. However, as this process is carried out with water, it is far from being economically viable to produce due to the need for post-separation distillation etc. Nevertheless, the product is of industrial importance due to its high content.

-The resulting material is neutralized with $NH_4(OH)$ and converted into an N-P-K fertilizer.

-It has also been shown that the $MgSO_4$ and Na_2SO_4 in the material bind crystal water (Weast and Astle, 1980).

-In the light of the studies carried out, it has been seen that such a system has difficulties operating in the pH range of 11-13 in terms of corrosion and separation of materials, both theoretically and in the light of the studies carried out. Therefore, after the leaching process, the solution has to be neutralized with H_2SO_4 .



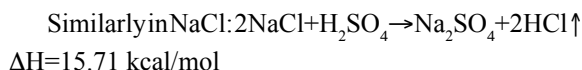
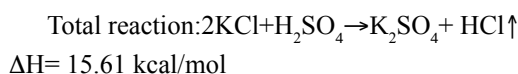
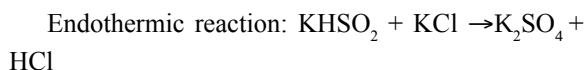
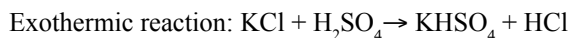
From Table 7, theoretically H_2SO_4 requirement per 100 g biomass ash;

$$\text{For } Ca(OH)_2: 29.85 * 12.25 * 98 / (74 * 100) = 4.84 \text{ g}$$

More than 50% of the world's sulphate production is based on the Mannheim process.

The Mannheim process was originally developed from the production of sodium sulphate by reacting NaCl with sulphuric acid. By replacing NaCl with KCl, potassium sulphate is produced (Felton et al., 2010).

The reaction is a two-step process:



$$\text{According to KCl: } 29.85 * 1.23 * 98 / (2 * 74.50 * 100) = 0.24 \text{ g}$$

$$\text{For NaCl: } 29.85 * 1.23 * 98 / (2 * 58.5 * 100) = 0.24 \text{ g}$$

A total (For $Ca(OH)_2 + KCl + NaCl$) of 5.32 g H_2SO_4 /100 g fly ash may be required.

$$\text{Maximum } K_2SO_4 \text{ that can be occur: } 29.85 * 33.47 * 174 / (2 * 39 * 100) = 22.29 \text{ g}$$

$$\text{Maximum } Na_2SO_4 \text{ that can be occur: } 29.85 * 1.23 * 142 / (2 * 23 * 100) = 1.14 \text{ g}$$

$Na_2SO_4 + K_2SO_4$ and the recovery of K_2SO_4 seem much easier at high temperatures (Table 9). This also means obtaining high purity K_2SO_4 .

K_2SO_4 saturation at 100°C 24.10 g/100 ml water will collapse first in the tchiner-1

$$Na_2SO_4 \text{ saturation at } 100^\circ C \text{ } 42.50 \text{ g/100 ml water}$$

- Precipitation of some of the extracted material by neutralization with NH_4OH at a higher pH.

Alternative evaluation methods such as separation can be given as examples.

- As a result of the preliminary studies carried out

Table 7- Determination of the approximately chemical composition of the material recovered in the liquid (K_2SO_4).

COMPONENTS	MW	MIXED MATERIAL QUANTITY	%	In 100 g material (g)							
				Ca	K	Mg	Na	S	Al	Fe	P
Al_2O_3	101.96	0.10	0.12						0.06		
$Ca(OH)_2$	74.00	10.00	12.25	6.62							
$CaMgO_6$	160.00	0.50	0.61	0.15		0.092					
$K_{0.7}Na_{0.3}Cl$	69.85	1.00	1.23		0.48		0.19				
$K_2Mg_2O_{12}S_3$	414.00	1.00	1.23		0.23	0.006		0.28			
K_2SO_4	174.00	59.00	72.28		32.40			13.29			
$K_3NaO_8S_2$	159.55	1.00	1.23		0.90		0.18	0.49			
KCl	74.50	1.00	1.23		0.64						
*Crystal- H_2O	18	0.90	1.10								
Na_2SO_4	142.00	1.00	1.23				0.40	0.28			
NaCl	58.50	1.00	1.23				0.48				
SiO_2	60.08	5.00	6.13								
$Fe(OH)_2$	90.00	0.03	0.04							0.02	
$Ca_3(PO_4)_2$	230.00	0.10	0.12	0.02							0.01
	Σ (g)	81.63	100.00	6.81	34.65	0.10	1.24	14.34	0.06	0.02	0.01

*weight loss at 1 hour at 300°C

Note: The analyses are carried out separately with ICP and SEM-EDS and there are some differences between the analyses. This is because analyses such as Cl, O, Si cannot be carried out by ICP, SEM-EDS can accurately determine the exact result with a large number of marked points, S analyses cannot be carried out reliably by ICP and also because this study is a preliminary study prior to the project. Verification was carried out using modelling of up to 100 oxide forms to determine the structure within engineering limits of error (+/-2.5%). According to the balance of 100 oxide forms Al_2O_3 0.12%, K_2O 40.34%, CaO 9.51%, SO_3 35.86%, MgO 0.13%, Na_2O 1.67%, Cl 5.06%, Fe_2O_3 0.03%, PO_4 0.04 %, volatiles 0.1%, crystals H_2O 1.1 % acceptable.

Table 8- Chemical analysis (%w/w) at the points marked with SEM-EDS.

Element	Point 1	Point 2	Average	ICP %w/w
Al		0.39	0.39	0.05
Ca		6.77	6.77	1.45
Cl		5.06	5.06	-
K	41.63	30.34	35.98	33.47
Mg		0.43	0.43	0.09
Na		4.04	4.04	1.14
O	40.09	40.52	40.31	-
S	18.28	12.04	30.32	15.24
Si		0.43	0.43	-

Table 10- The amount of H_2SO_4 required per 100 g obtaining H_3PO_4 in the material.

	%	Multiplier	Σ	Total= 47.13 g H_2SO_4 /100 g fly ash
Al_2O_3	3.85	0.614	2.36	
CaO	22.13	1.479	32.73	
Fe_2O_3	1.56	0.614	0.96	
K_2O	16.23	1.041	16.90	
MgO	7.21	2.433	17.54	
Na_2O	2.77	1.582	4.38	
P_2O_5	11.29	-0.691	-7.80	
SO_3	16.25	-1.225	-19.90	

Table 9- Solubility of Na, K, Mg salts in water (% w/w) at heat [from Green and Southard (2019)].

Chemicals		0°C	10°C	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
K_2SO_4	g/100 ml	7.40	9.30	11.10	13.00	14.80	16.50	18.20	19.80	21.40	22.90	24.10
KCl	g/100 ml	28.00	31.20	34.20	37.20	40.10	42.95	45.80	48.55	51.30	53.90	56.30
$MgCl_2$	g/100 ml	52.90	53.60	54.60	55.80	57.50	59.25	61.00	63.55	66.10	69.50	73.30
$MgSO_4$	g/100 ml	22.00	28.20	33.70	38.90	44.50	49.55	54.60	55.20	55.80	52.90	50.40
NaCl	g/100 ml	35.70	35.80	35.90	36.10	36.40	36.75	37.10	37.55	38.00	38.50	39.20
Na_2SO_4	g/100 ml	4.90	9.10	19.50	40.80	48.80	47.05	45.30	44.50	43.70	42.70	42.50

Table 11- The determination of the approximate liquid H_2PO_4 composition.

Element	WASTE % w/w	Expected mg/l	%	Read in ICP %w/w	liquefiable efficiency η %	Components	May occurs (g)	in liquid %	*Dry base %w/w
Al	2.8	2800	0.28	0.249	88.93	Al ₂ (SO ₄) ₃	22.41	2.09	31.61
Ca	22.1	22100	2.21	-	0.000	CaSO ₄	-	-	-
Fe	1.51	1510	0.151	0.115	76.16	FeSO ₄	3.12	0.29	4.0
K	4.95	4950	0.495	0.465	93.94	K ₂ SO ₄	10.37	0.97	14.63
Mg	2.8	2800	0.28	0.229	81.79	MgSO ₄	11.45	1.07	16.15
Na	2.35	2350	0.235	0.217	92.34	Na ₂ SO ₄	6.70	0.63	9.45
P	7.03	7030	0.703	0.533	75.82	H ₃ PO ₄	16.85	1.57	23.76
						H ₂ O	1000	93.38	0.00
						Σ	1070.90	100.00	100.00

*This calculation is for only projection purposes only and <1% Mn, Zn, Cu sulfate contents are also possible in the dry base composition.

in this study, the design process shown in Figure 11 is presented as an economic process.

- Further project work will continue in the form of key studies such as process optimisation, economisation, equipment specifications, detailed mass energy balances and determination of equipment retention times.

- This study was carried out with the resources of the MTA-Department of Mineral Analysis and Technology

(MAT) department as a preliminary study for the purpose of utilising biomass thermal power plant ashes, which have increased rapidly in our country in recent years due to the energy demand. It was carried

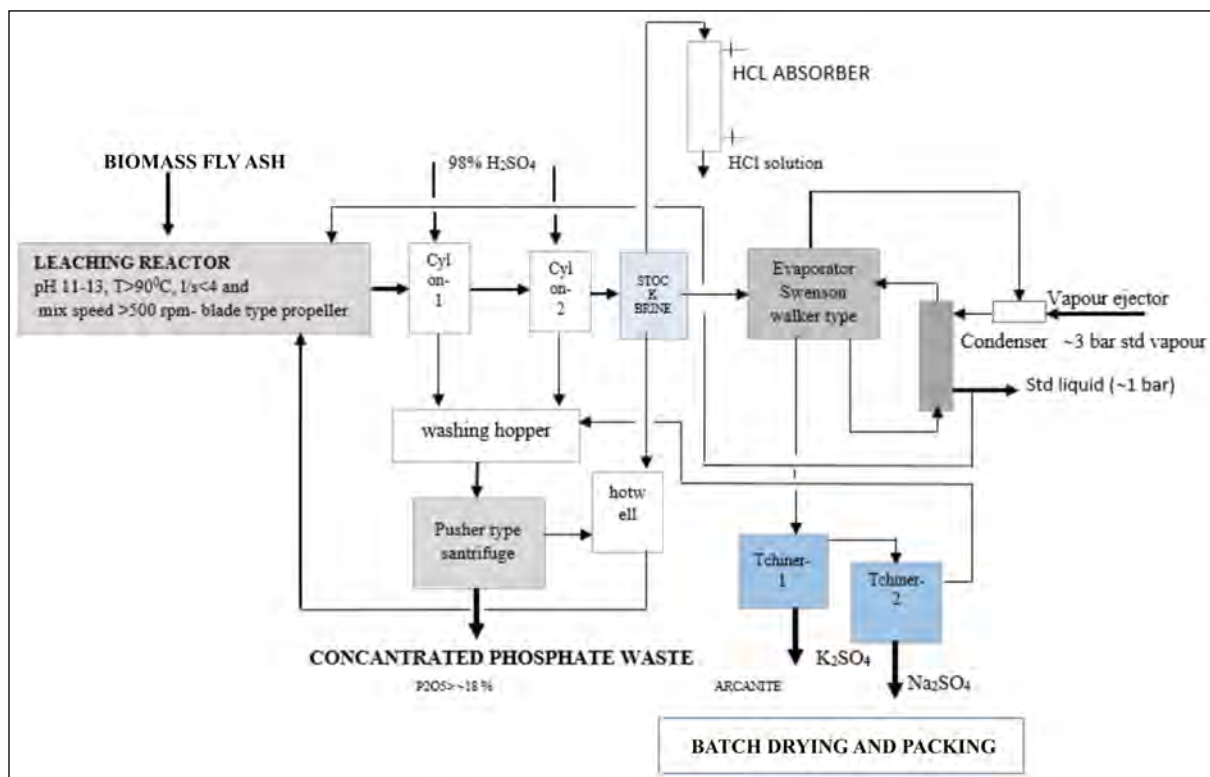


Figure 11- Suggestion draft box-box continue process step for concentrated potassium sulphate production from the biomass ash.

out only for the purpose of understanding the subject and developing a suitable method for the preliminary study of the project. Our country aims to reduce the use of potash. As far as the future waste management is concerned, the continuity of biomass ash at certain standards, the overcoming of official-bureaucratic problems in the field of the environment and the renewal of the waste management and waste disposal codes appear as an important issue for economic gain.

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Establishing of soil loss tolerance limit and sediment yield in Zagros fold-thrust belt: a case study in the Kanarwe river basin, Iraq-Iran

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Research Article

Keywords:

RUSLE, Sediment Delivery, Zagros Thrust Zone, CN-SCS Curve, Dam Lifespan.

ABSTRACT

Soil degradation is a significant cause of topsoil loss; in most mountainous region watersheds, it leads to decreased agriculture productivity and reservoir storage. This study targeted calculating and mapping soil loss and sediment yield in the Lesser Zab watershed located between Iraq and Iran. The Revised Universal Soil Loss Equation (RUSLE) model was used to calculate the soil loss. A Digital Elevation Model of 30 m, a Digital Soil map (1:500000), rainfall, and land cover were used to derive parameters. The soil loss rates are 58.1 and 0.1 $\text{tha}^{-1}\text{yr}^{-1}$. The total annual soil loss is 1037289 tonnes, of these 404512, covering 75% of Kanarwe river basin land. Most of these affected lands are in the eastern and middle part, which is below the FAOs standard allowable for tolerable soil erosion. Still, the rest covers 25 % of the basin in the west with a total annual soil loss equal to 632777 tonnes, above FAOs standard. The maximum and minimum sediment yield is 29 and 0.1 $\text{tha}^{-1}\text{yr}^{-1}$, respectively. Based on the statistical correlation coefficient, the most effective RUSLE parameters on sediment yield from high to low are topography 0.48, soil erodibility 0.38, and crop management 0.38.

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1. Introduction

Soil erosion is a geomorphic process that occurs naturally; however, humans' soil utilization and climate change lead to increased land degradation (Pennock, 2019; Yang et al., 2003). Soil loss particles are turned into sediments and delivered to the river by an erosion agent (Chuenchum et al., 2020), and erosion processes pose severe threats to agricultural production, dam lifespan, and the earth's landform (Balabathina et al., 2020; Lee et al., 2014). Man-made activities cause ten times faster soil loss than natural

agents such as rainfall or coastal waves (Pimentel and Burgess, 2013). Sometimes, trapped sediment yield by a natural or artificial agent leads to bank erosion and decreased riparian vegetation (Anthony et al., 2015).

The RUSLE equations were first developed on a small scale in the United States of America but later were used and calibrated in many countries (Alewell et al., 2019; Ezzaouini et al., 2020). New studies suggest that integrating the Revised Universal Soil Loss Equation (RUSLE) with satellite image data is more effective for calculating amounts of soil loss

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and mapping its distribution (Kebede et al., 2021; Somasiri et al., 2021).

The soil loss and sediment yield recently threatened water quality and quantities in Iraq and Iran due to poor management of soil loss (Zare et al., 2017; Band et al., 2022; Ostovari et al., 2022; Othman et al., 2022; Salar, 2022). The unmanaged soil loss from the upstream country (Iran) and downstream sediment delivery decreased dams' storage capacity and water resource life structures in the lesser Zab watershed within Iraq (Bhattarai and Dutta, 2007; Omeed H. Al-Kakey et al., 2023).

Many studies try calculating Iraq's sediment yield and soil loss, but the results are inaccurate. Hassan et al. (2016) studied sediment deposited types with the Dokan dam reservoir (DDR), located downstream of the Lesser Zab watershed, without considering the amount of Soil erosion risk and its effect on ecosystem management. Ezz-Aldeen et al. (2018) studied watershed sediment of DDR using the soil and water assessment model (SWAT) to estimate sediment delivery ratio-based Empirical Equation (Lumped model). However, this model is only applicable to small watersheds, not a watershed like DDR. Othman et al. (2022) conclude that the sediment yield is more than the soil loss, which is scientifically wrong. The soil erosion risk in Iran is also the same as in Iraq and many studies conclude that the risk of soil erosion has increased in the last decade (Sadeghi, 2017; Zare et al., 2017; Hosseinalizadeh et al., 2020).

Therefore, this study aims to answer four key research issues; How much soil eroded annually from the Lesser Zab watershed located between Iraq and Iran? Are the soil erosion processes uniformly over the basin or abnormally distributed over the Lesser Zab watershed? Is the potential for soil loss greater than the FAO's soil erosion tolerance limit? Which parameters of RUSLE are more significant on soil loss? This study's findings will serve as a benchmark for future dam site selection and ecosystem management in Iraq.

2. Research Methods

2.1. Study Area and Data Source

The Kanarwe River basin is part of the lesser Zab transboundary watershed within the Zagros

Mountain Range (Figure 1). The basin outlet is 20 km away from northeast Sulaymaniyah city with a catchment area of 1541 km² and extended about 71 km in length (Mohammad, 2023). The Mediterranean climate's hot summers and wet winters deliver more than 800 mm of rain and have an average temperature of 20°C per year (Abbas et al., 2017; Shubbar et al., 2017; Mohammed et al., 2021;). Several data sources to estimate soil erosion based on the RUSLE. Field survey and ground truth collection by a Global Position System (GPS) include soil sampling and lithology description. A (30*30 m) DEM from the Advanced Spaceborne Thermal Emission and Reflection Radiometer (ASTER; GDEM) dataset is used to derive slope length- steepness factor value. The geological map of study area (Figure 2a) was correlated with soil maps to measure accurate soil depth about geological units (Al-Qayim et al., 2018; Ma'ala, 2007). Soil map derived from the Harmonized Soil World Database (HWSD) (Figure 2b), with a scale of 1:500000 (Buringh, 1960; Nachtergaele et al., 2008) Mean annual rainfall data (1990–2015) from the four-gauge stations (Dokan, Chwarta, Mawat, and Penjween) obtained from Sulaymaniyah metrological Directorate was used to estimate the annual mean rainfall at Kanarwe river basin (Mohammed et al., 2023) (Figure 3).

The Global Land Use and Landcover (LULC) map was obtained from the Global Land Cover Facility (www.landinfo.com) for determining soil erodibility, cover management, and SCS-CN number.

3. Data Analysis

3.1. Soil Loss Estimation and Sediment Yield

The soil loss was calculated using the RUSLE, an empirical model developed by Renard in (1997) to estimate soil loss. The RUSLE model Equation 1 is expressed as:

$$S_{loss} = R_E * K_s * LS_T * C_V * P_E \quad (1)$$

Where S_{loss} is the mean annual soil loss due to water erosion [in tons per hectare per year (t ha⁻¹ year⁻¹)], R_E is the rainfall and runoff erosivity factor (in MJ-mm ha⁻¹ h⁻¹ year⁻¹), K_s is the soil erodibility factor (in tone ha⁻¹ MJ⁻¹ mm⁻¹); L is the slope length

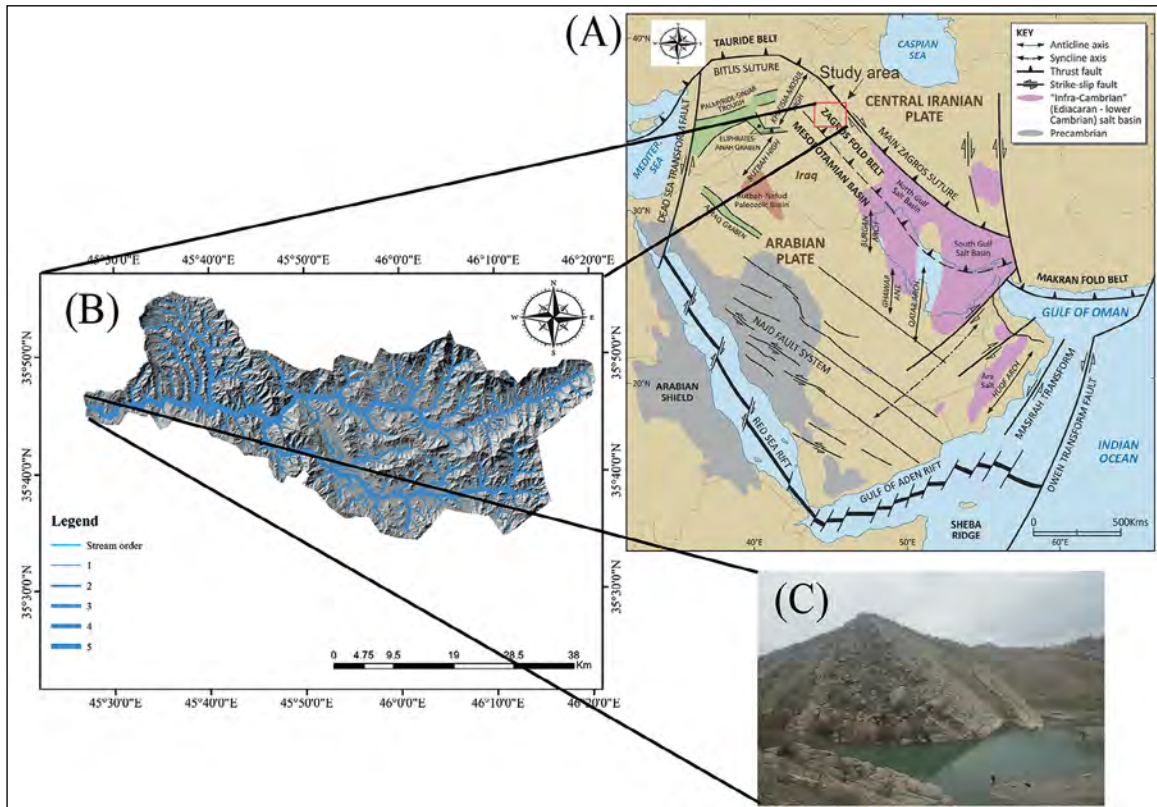


Figure 1- a) Regional tectonic setting Zagros mountains (English et al., 2015), b) Stream network of Siwail River that flows toward basin outlet (Dam site, East west), c) Proposed dam site.

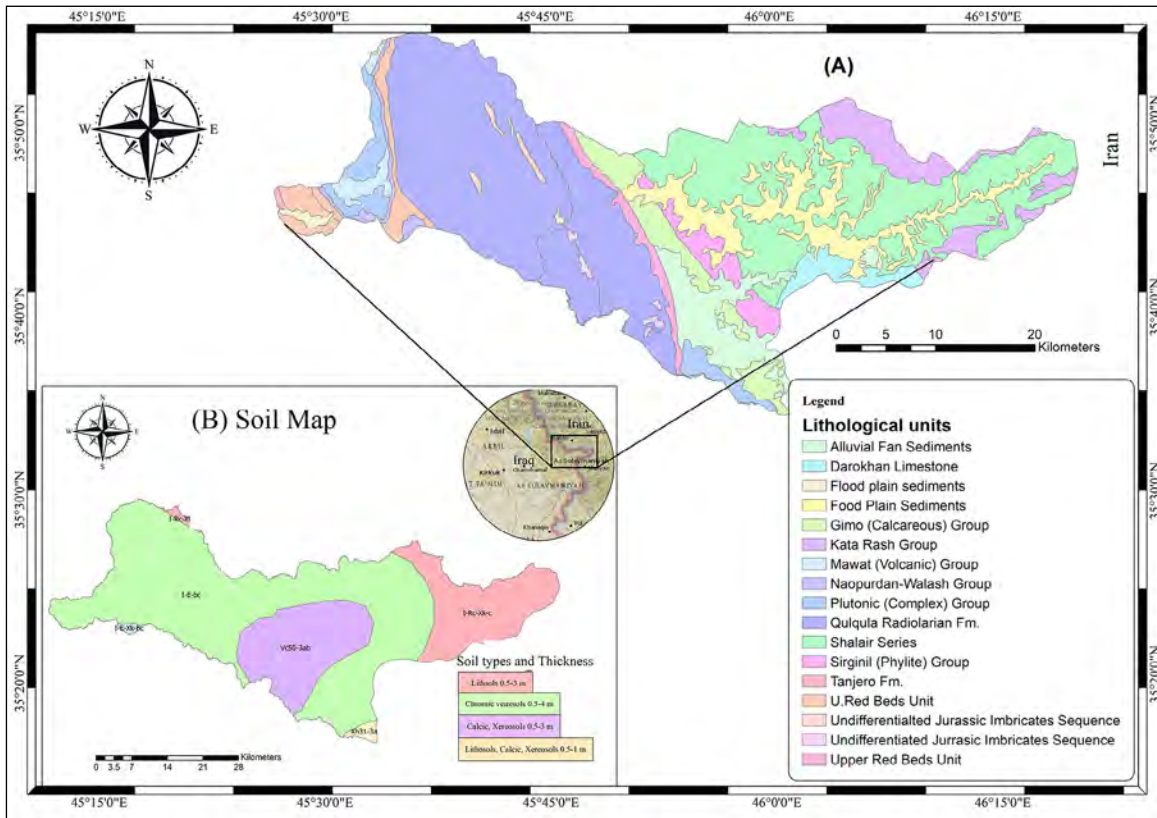


Figure 2- a) Show the geological map after (Ma'ala, 2007; Al-Qayim et al., 2018), b) Soil map of the study area after (Buringh, 1960).

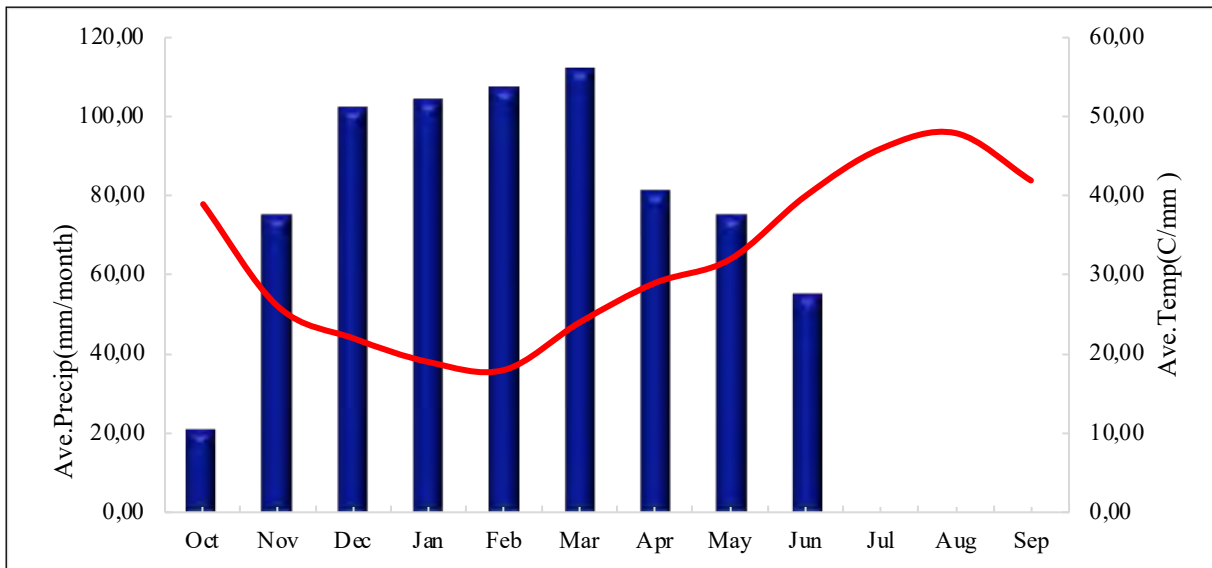


Figure 3- Mean monthly rainfall (blue bars) and temperature (red line).

factor (dimensionless), S is the slope steepness factor (dimensionless); C is the cover and management practice factor (dimensionless), and P_E is the erosion support practice (dimensionless). The sediment delivery ratio (SDR; $t \cdot ha^{-1} \cdot yr^{-1}$) was calculated based on Equation 2.

$$SDR = 1.366 \times 10^{-11} * (DA) - 0.0998 * (RL) + 0.3629 * (SCS-CN) + 5.444 \quad (2)$$

Where DA is the drainage area in km^2 , RL is a relief-length ratio in $m \cdot km^{-1}$, and $SCS-CN$ is the soil conservation service curve number. Finally, the sediment delivery ratio was used with Soil loss rate Equation 3 to calculate the sediment yield (Sy ; $t \cdot ha^{-1} \cdot yr^{-1}$).

$$Sy = SDR * Ri \quad (3)$$

Where Ri is soil erosion for each grid cell.

Spatial analysis techniques and satellite images were used to simulate the RUSLE model, sediment delivery ratio, and sediment production., as shown in Figure 4.

3.2. Rainfall Erosivity (R_E)

Rainfall erosivity is the erosive power of participation kinetic energy: the greater the intensity and rainstorm duration, the higher the erosion potential (Gholami et al., 2021). The R_E factor is generally

calculated from mean annual rainfall data using storm energy over a 30-min duration expressed by $R = EI_{30}$ Equation 4.

$$R = \frac{1}{n} \sum_{j=1}^n \left[\sum_{k=1}^m (E)(I_{30})^k \right] \quad (4)$$

Where n is the total number of years where data was collected, K is the total number of rainfall storms up to m , j is several years up to n , I_{30} is the maximum 30 min intensity ($mm \cdot hr^{-1}$), E_j is the total kinetic energy ($MJ \cdot ha^{-1}$) of a k storm in m year (Renard and Freimund, 1994) Developed (Equation 5).

$$R_E = 0.04830 * P^{1.61} \quad P \text{ is less than } 850 \text{ mm} \quad (5)$$

where R = Rainfall Erosivity, P = Mean annual rainfall in mm.

For calculation (R_E), four stations' long-term mean annual rainfall (1990 to 2015) was obtained from the Sulaymaniyah Meteorological Directorate Table 1. For creating an erosivity map, the inverse distance weighted (IDW) approach is used to estimate the unknown cell value of erosivity for the whole watershed.

3.3. Soil Erodibility (K_s)

The soil erodibility factor (K_s) assesses how vulnerable soil types are to particles being separated and carried away by rainfall and runoff. (Wischmeier

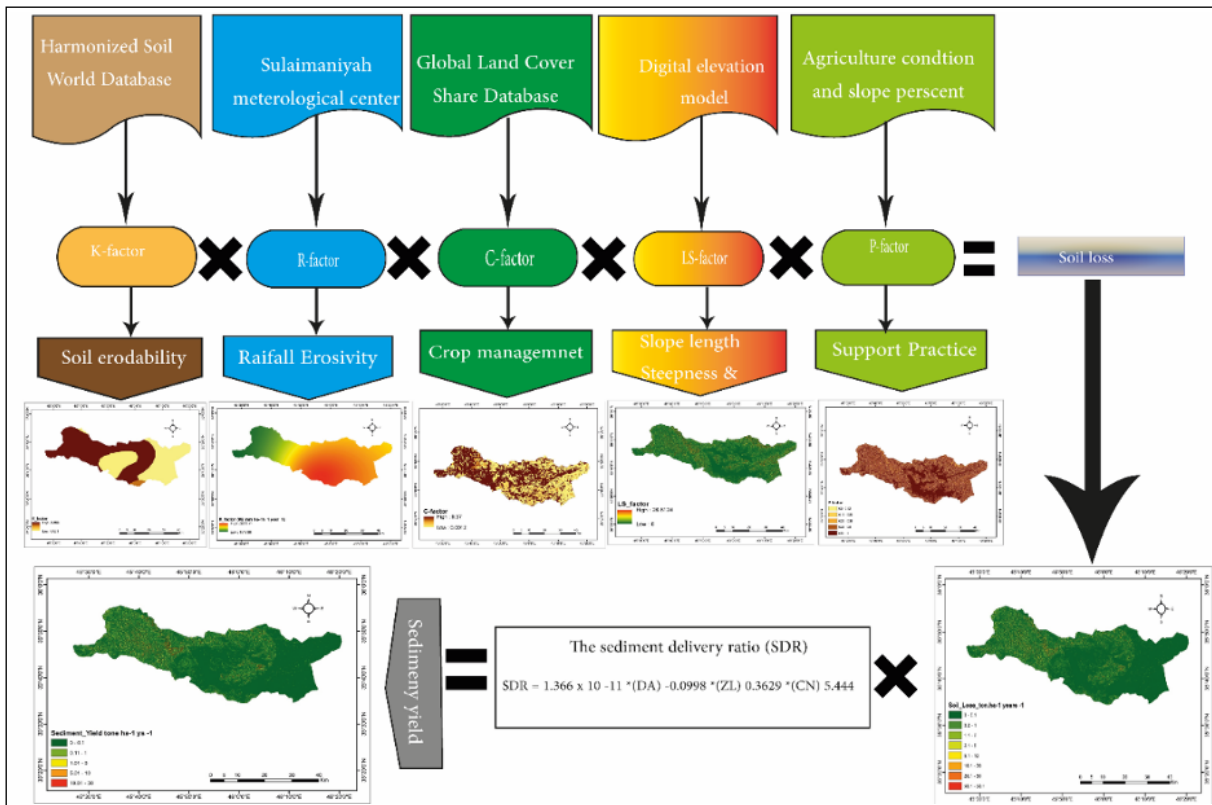


Figure 4- Conceptual modelling of estimation of RUSLE parameters.

Table 1- Rain gauge stations, source (Sulaymaniyah Directorate of Meteorology and Seismology).

No.	Gauging station	Location		Altitude(m)	Mean annual rainfall
		Longitude Latitude			
1	Penjween	45.945478	35.627854	1280	950
2	Chwarta-Mawat	45.409533	35.902678	1160	700
3	Dokan	44.958625	35.952052	540	640
4	Sulaymaniyah	45.435350	35.566579	880	650

and Smith, 1978). The soil types in the study area are classified into four classes-based FAO soil maps. The K_s value for each soil class is calculated based on soil types, composition, and texture Table 2.

3.4. Topography (LS_T)

The topographic factors, such as slope length (L), represent the length between the starting and end of

the inter-rill erosion process, and slope steepness (S) depends on steepness only. The LS factor is crucial in water movement (volume and velocity) in streams, considerably inducing soil erosion and Sediment transporting capacity (Gashaw et al., 2017). The LS factor in the RUSLE was estimated using spatial analysis techniques based on Equation 6 and Equation 7 (Simms et al., 2003).

Table 2- KS value for Soil types based on (Buringh, 1960).

Soil classification	Soil Texture	Ks
Lithosols	Clay and Loam.	0.001
Chromic vertosols	Loam to clay with variable gravel	0.065
Calcic xerosols	Clay marl gravel	0.065
Lithosols, Calcic, xerosols	Silty clay gravel	0.0041

$$LST = [A/22.13]^{0.6} * [\sin B/0.0869]^{1.3} \quad (6)$$

where A is the upslope contributing area factor, and B is the slope angle. Incorporating the DEM into GIS accurately determined the slope gradient S and length L. This study computed the LS_T -value based on a modified Equation 7 suggested by (Simms et al., 2003; Gelagay and Minale, 2016; Chuenchum et al., 2020) because upslope contributing and basin geometry is more influenced than slope length and steepness topsoil loss.

$$LS_T = \text{Pow} \left(\frac{\text{Flow accumulation} * (\text{DEM resolution})}{22.13} \right)^{0.6} * \text{Pow} \left(\frac{\sin(\text{DEM slope}) * 0.01745}{0.0869} \right)^{1.3} \quad (7)$$

Where Pow (power) is a function in the ArcGIS Spatial Analyst (Ashiagbor et al. 2013), the flow accumulation number of cells contributes to downward flow.

3.5. Cropping Types (C_v)

The cover management factor is the soil loss ratio soil loss rate and land cover types (Wischmeier and Smith, 1978). Benavidez et al. (2018) prefer using previous research papers on land use/land cover to estimate the C_v factor. This strategy was performed for the estimation C_v factor Table 3.

3.6. Support Soil Resistance Method (P_s)

P_s values range from zero to one, with zero indicating an ideal human-made structure for preventing erosion and one indicating no erosion resistance (Renard and Freimund, 1994). Such

resistance structures include contour plowing, strip cropping, and terracing, controlled slope. (Shin, 1999) assigned P_s -value for five slope percentage categories based on slope percentage Table 4.

Table 4- The P factor value (Shin, 1999).

Slope (%)	Land use	P factor
9–12	Non-Agriculture	0.6
13–16	Non-Agriculture	0.7
17–20	Non-Agriculture	0.8
21–25	Non-Agriculture	0.9
>25	Non-Agriculture	0.95

4. Results and Discussion

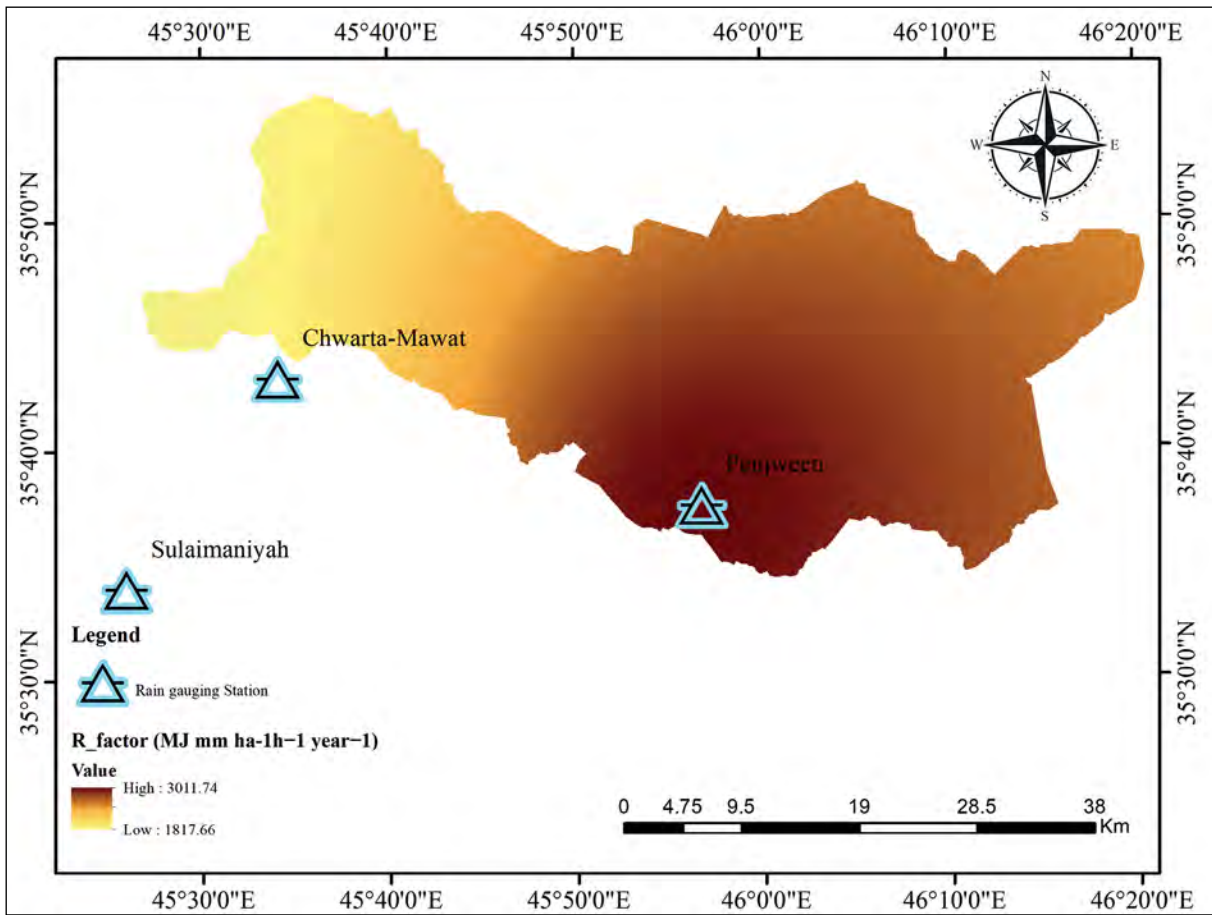
Three different software, including ArcGIS, WMS11.1 (watershed modelling surface), and RStudio Pro V1.1717-3 software, were used to compute the average yearly (soil loss, Sediment yield, and Tolerable Soil loss erosion) as follows.

4.1. R_E Factor

The rainfall in the Kanarwe River basin ranges from 700 mm to 950 m Table 5. Penjween is located within the watershed, while Chwarta-Mawat station is close to the basin's outlet; thus, these two stations significantly weigh R_E factor estimation. A high R_E -value (3011) is presented east, characterized by stormy weather and heavy intense rainfall. The western parts have a low R_E -value (1817), characterized by low terrain with less rainfall Figure 5.

Table 3- C_v Factor Values for LUC types.

Land cover	C	Source
Rainfed croplands	0.07	(Yang et al., 2003)
Mosaic cropland	0.37	(Tiwarly et al., 2014)
Mosaic vegetation	0.25	(Yang et al., 2003)
Closed (>40%) needle-leaved evergreen	0.0015	(Chadli, 2016)
Closed to open (>15%) mixed broadleaved and needle	0.1	(Chen et al., 2019)
Mosaic forest or shrubland	0.01	(Tiwarly et al., 2014)
Mosaic grassland	0.2	(Tiwarly et al., 2014)
Closed to open (>15%) (broadleaved or needle-leaved, evergreen, or deciduous)	0.0012	(Chadli, 2016)
Sparse (<15%) vegetation	0.01	(Yang et al., 2003)
Bare areas	0.35	(Yang et al., 2003)

Figure 5- R_e factor map.Table 5- R_e Factor Value.

Stations	Rainfall (mm)	RE [$MJ\ mm\ h^{-1}ha^{-1}year^{-1}$]
Penjween	950	3011.74
Chwarta-Mawat	700	1838.88

4.2. K_s Factor

The K_s factor was computed based on four soil types: Lithosols, Chromic Vertosols, Calcic xerosols, and Calcic Xerosols (Figure 5). The K values are estimated and compared to previous research on soil types in the northeastern part of Iraq (David, 1988; Hussein et al., 2007; Othman et al., 2021; Zakerinejad and Maerker, 2015). Accordingly, high K_s values (0.065) are presented for chromic and clastic xerosols in the west part and low values of K_s (0.01, 0.04) in the east region Figure 6a and Figure 6b. This substantial variation in K_s value is due to cropping out different rock types in the Kanarwe River basin.

4.3. LS_r Factor

The maximum value of LS_r is (26.6) when slope length and flow accumulation are high due to high altitude mountain and dense stream order in the watershed. The LS_r values become lower closer to the river (flat areas) of the Siwil River in the lower regions Figure 7.

4.4. P_s Factor

The Kanarwe river basin was classified into five classes of Slope percentage (slope %) Figure 8a, and then the P_s -value was assigned for each type of slope percent Figure 8b based on Table 4. Most of the lower P_s -value is given to the central part, which is flatter and contains flood plains. The West of the Kanarwe River basin shows a high P_s -value due to the topographic condition.

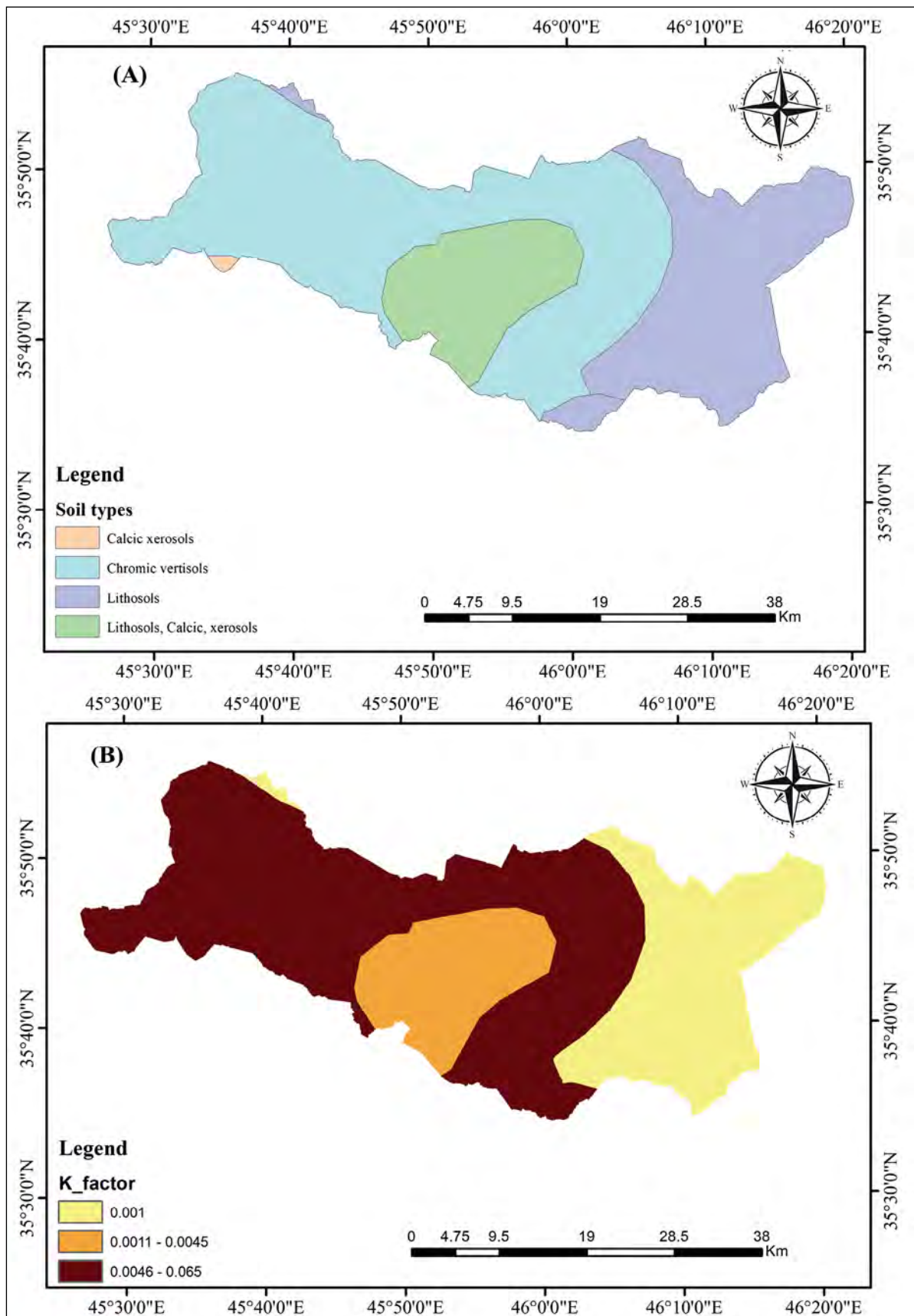
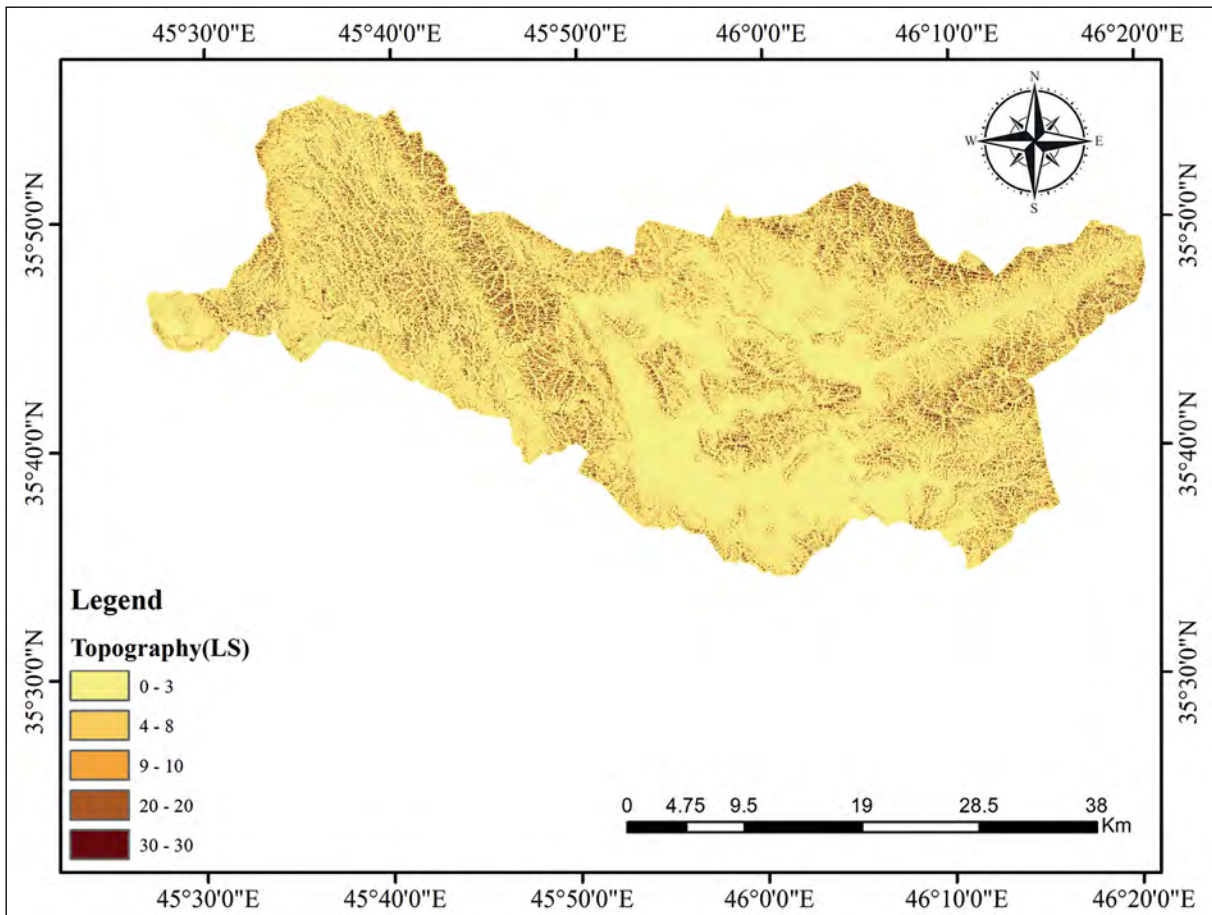


Figure 6- a) Soil map, b) K_s factor map.

Figure 7- LS_r factor map.

The mainland cover/use was (cropland, bare soil, and forests) Figure 9a. The C_v value ranges from (0.0012 to 0.37) and is assigned to land cover types of the Kanarwe river basin Figure 9b. Lower C_v factor values (0.0012) were observed in the eastern and south-eastern parts due to the present forests, especially oak trees. The higher C_v factor values (0.37) can be seen in the western parts, this area covers mostly weathered soils and recent deposits; hence it is more prone to water erosion.

4.5. Soil Loss and Tolerance

The final map of the soil loss was calculated using Equation 2 by integrating RUSLE layers (Parameters) to produce a grid map Figure 10. The soil loss rate expands from 0 in the east part (unweathered hard igneous) to $58.1 \text{ t ha}^{-1} \text{ yr}^{-1}$ in the central and west parts of the high weathered, sedimentary rock. The soil loss map was reclassified into five soil-lowering

risk zones Table 6, using Gelagay and Minale, 2016 classification.

The calculation means annual soil loss is useless scientifically without the calculation of soil erosion risk classes (Pennock, 2019), so soil erosion risk was calculated and reclassified. The total annual soil loss is $1037289 \text{ t ha}^{-1} \text{ yr}^{-1}$; 404512, which covers 75% of the Kanarwe river basin, is below soil erosion risk (3.9 mm yr^{-1}) (Pennock, 2019). The FAO's (2019) tolerable soil loss rate is $4.2\text{-}7.2 \text{ t ha}^{-1} \text{ yr}^{-1}$ (Montgomery, 2007; Verheijen et al., 2009).

Most of this low tolerable soil risk erosion is in the eastern and central parts due to the crop out of the un-weathered, hard rock of igneous rock and thin soil depth. The moderate to severe soil risk classes, which cover 25 % of the Kanarwe river basin, have an annual loss equal to 632777, above FAOs tolerable soil rate. This moderate to severe tolerable soil rate of

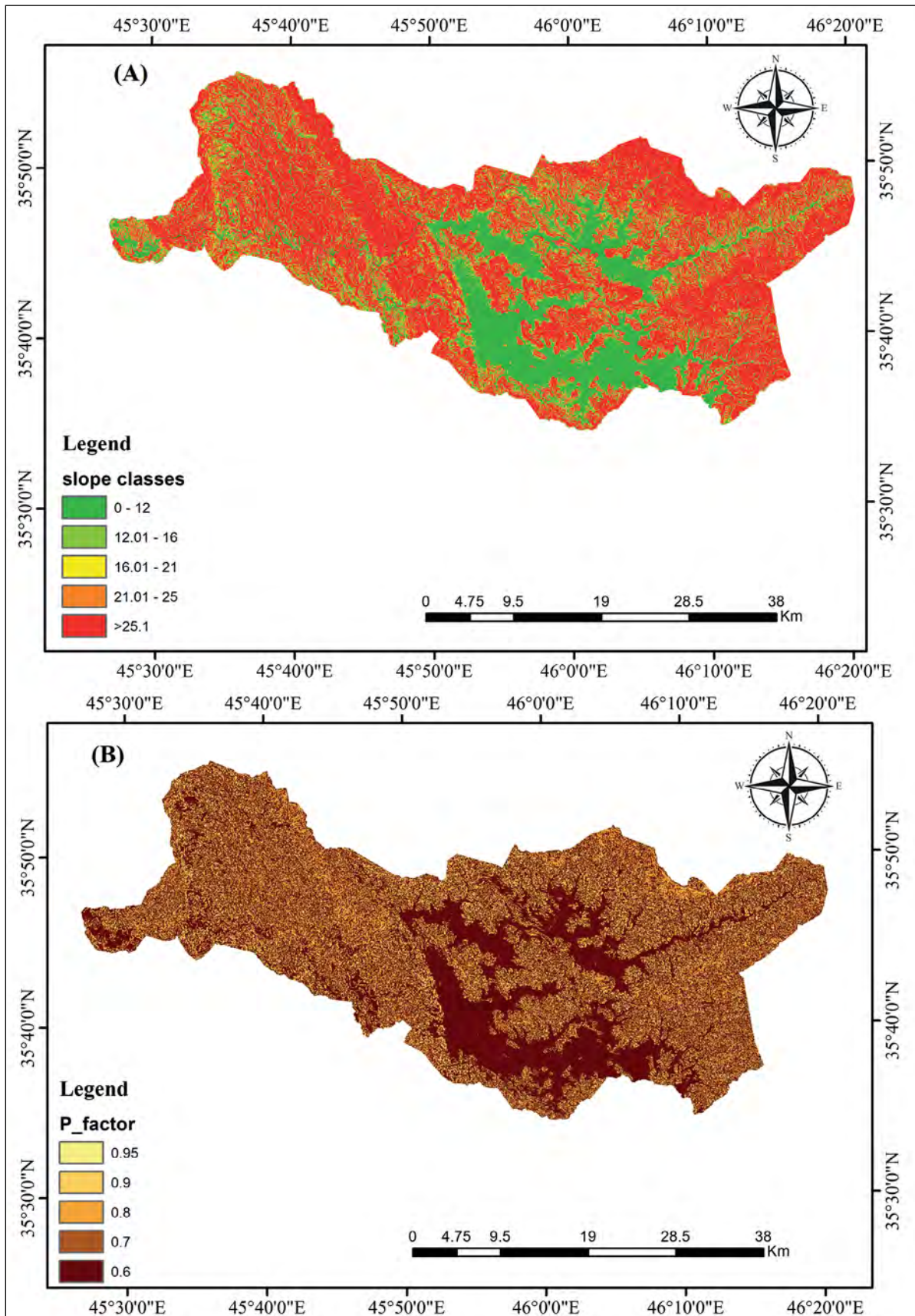


Figure 8- a) Slope class map, b) P factor map.

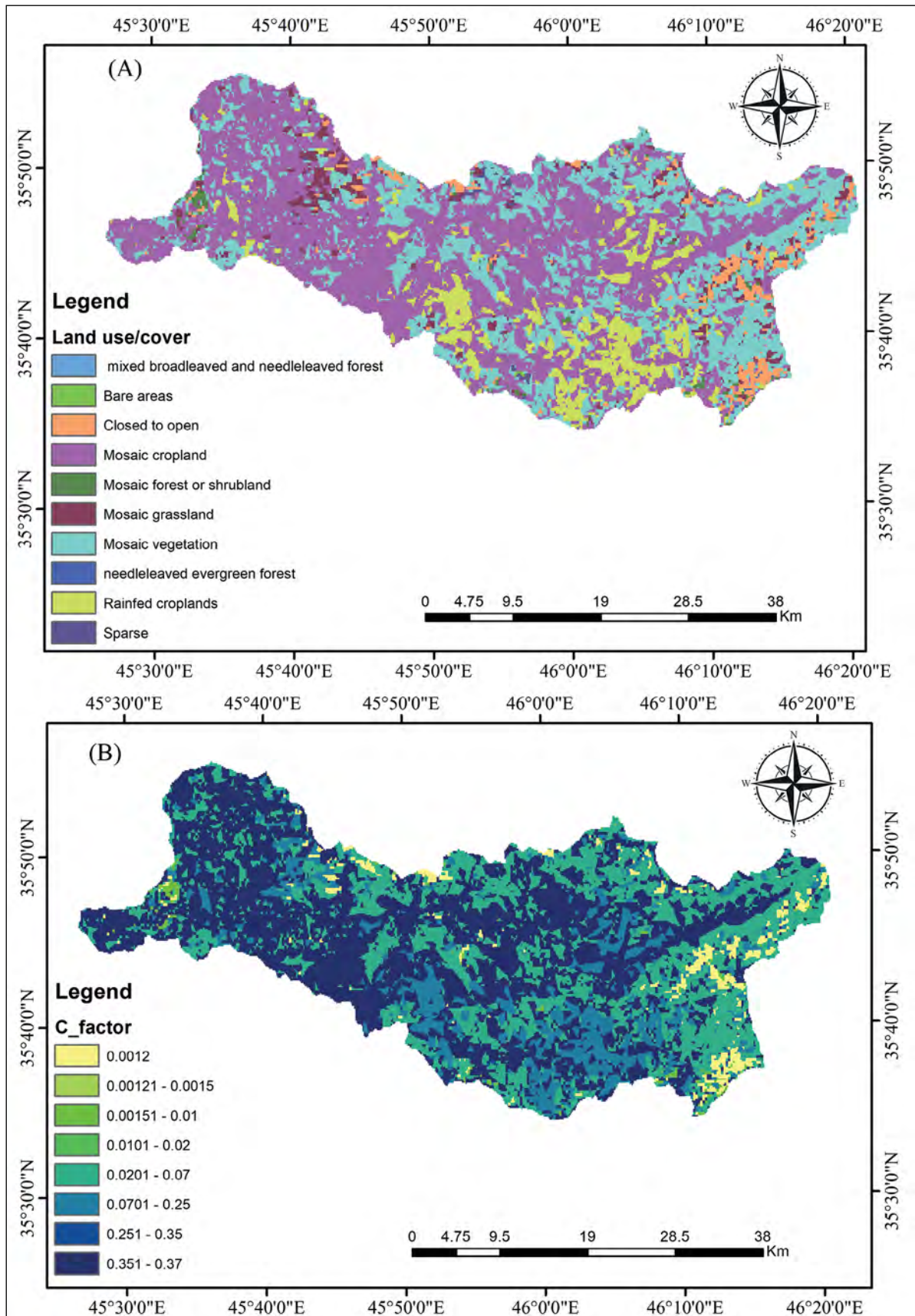


Figure 9- a) Land cover map, b) C_v factor map.

Table 6- Soil Loss class, covered area, and soil risk class.

Soil loses	Soil erosion risk	Area (ha)	Percent of total area	Annual soil Loss(ton)	Percent of total soil loss
0-7.0	Low	115575	74.4	404512	38.99
7.1-15	Moderate	22498.6	14.6	235200	22.67
15.1-25	High	10170.6	6.6	203412	19.6
25.1-45	Very high	4160.7	2.7	145624	14.03
45-60	Sever	924.6	0.6	48541	4.67

the western and central part is spatially situated in the narrower, steep slope close to the outlet Figure 10.

4.6. The Sediment Delivery Ratio (SDR)

The SDR 0.5022 tons is calculated based on Equation 2 and Table 7. The drainage area and relief-

length ratio are related to the time and rate of peak flow and the runoff. The SCS-CN parameter is more sensitive to the basin's soil types and land cover and calculates daily surface runoff (USDA, 1986; Mishra et al., 2006; Kayet et al., 2018). the CN values for each land-use class are calculated and then listed in the supplementary material Table 8 and converted in Grid format Figure 11.

Table 7- Drainage Basin parameters.

Drainage area (DA)	1543 Km ²	ArcGIS 18.1
Relief-length ratio (RL)	3.122	WMS 11.1

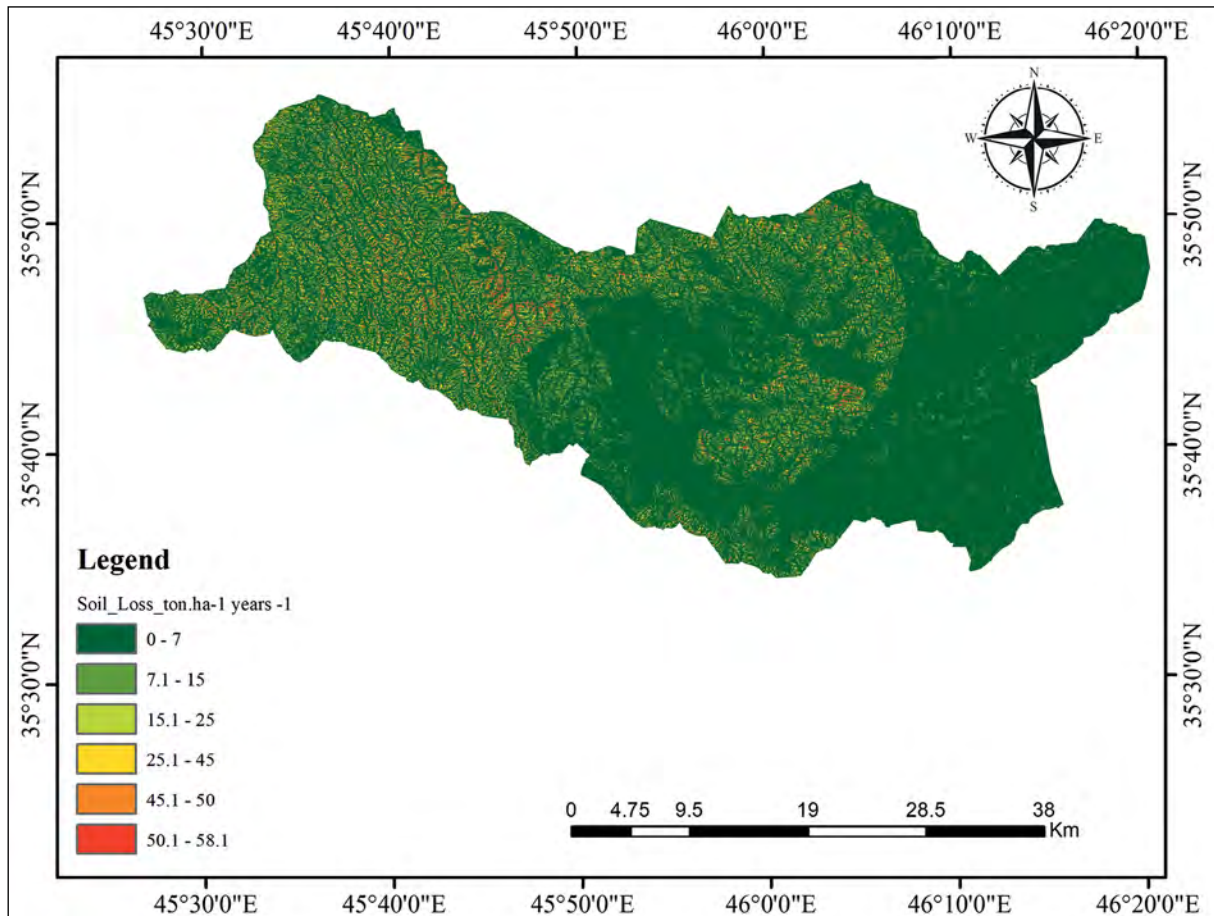
Figure 10- S_{loss} map.

Table 8- Runoff Curve Number values for each class of landcover types.

HSG (Harmonized Soil Group)	Land Use Description	CN code	Area (km ²)	Product (CN x A)
D	Sparse (<15%) vegetation	94	481.359	45247.74
D	Closed to open (>15%) (broadleaved or needleless)	77	21.111	1625.541
D	Rainfed croplands	84	72.466	6087.131
D	Mosaic vegetation (grassland/shrubland/forest)	79	114.837	9072.155
D	Mosaic forest or shrubland (50-70%) / grassland	77	22.009	1694.713
D	Mosaic cropland (50-70%) / vegetation (grassland	84	123.072	10338.06
D	Bare areas	98	13.176	1291.21
D	Mosaic grassland (50-70%) / forest or shrubland	77	16.919	1302.738
D	Closed (>40%) broadleaved deciduous forest (>5)	79	1.946	153.765
D	Closed (>40%) needle-leaved evergreen forest (>)	79	1.797	141.937
B	Sparse (<15%) vegetation	86	181.614	15618.79
B	Mosaic vegetation (grassland/shrubland/forest)	60	120.227	7213.646
B	Mosaic grassland (50-70%) / forest or shrubland	56	39.826	2230.271
B	Mosaic cropland (50-70%) / vegetation (grassland)	69	105.105	7252.274
B	Rainfed croplands	69	155.562	10733.78
B	Closed to open (>15%) (broadleaved or needleless)	56	22.758	1274.441
B	Mosaic forest or shrubland (50-70%) / grassland	56	37.131	2079.351
B	Bare areas	98	12.277	1203.173
D	Water bodies	0	0.15	0
B	Closed (>40%) broadleaved deciduous forest (>5)	60	5.689	341.368
CN	(Weighted) = Total Product \ Total Area	80.6		

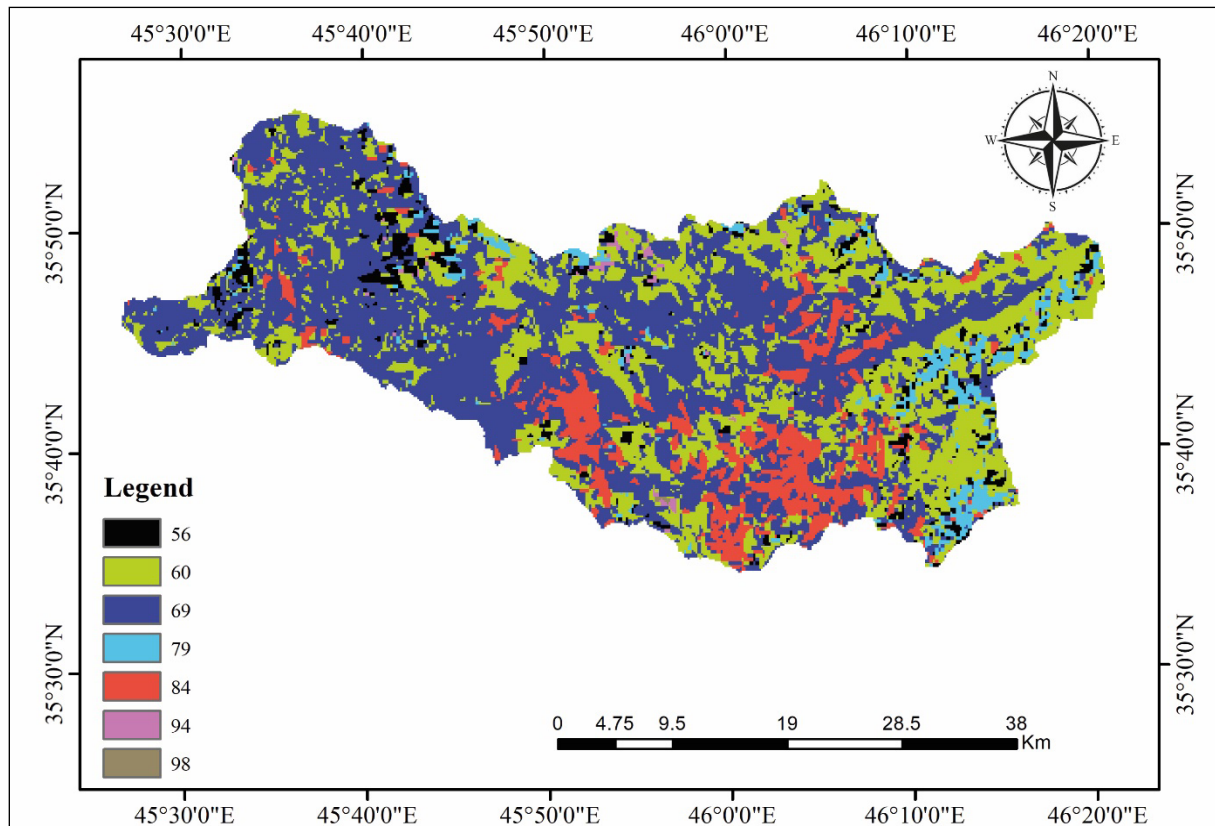


Figure 11- Curve number map.

4.7. The Sediment Yield (SY)

The sediment yield rate is calculated based on Equation 3. It is between 0 to 29.06 $1 \text{ t. ha}^{-1} \text{ yr}^{-1}$ and the mean value is 0.321 Figure 12. The (Sy) becomes higher in the western part compared to the central areas and becomes lower in the east due to geological conditions dominated by hard un-weathered igneous rocks, the obtained results were well matched with the international soil erosion map of the world Global soil degradation map (Source: UNEP, International Soil Reference and Information Centre) and reveals soil degradation states in Iraq and Iran.

5. Correlation Matrix Between RUSLE Variables and Soil Loss

The correlation coefficient analysis among RUSLE parameters shows that the LS_r , K_s , C_v , and

P_s factors showed a linear relationship with soil loss and sediment yield. In contrast, the R-factor showed an inverse relationship between sediment yield and soil loss Figure 13. The topography (LS_r) was more robust, influencing soil loss and sediment yield. A negative R_E value reveals nature and climate conditions in the Kanarwe river basin. (Mohamadi and Kavian, 2015) Conclude that during periods of intense rainfall, the relation between the soil loss-rainfall fitted with nonlinear functions, because it's the beginning rainstorm until 30 minutes there no soil erosion later with prolonged rainfall the soil saturation crosses the limit of plasticity so as a result the weather layers start eroded the erosion decreases when this weathered layer is gone.

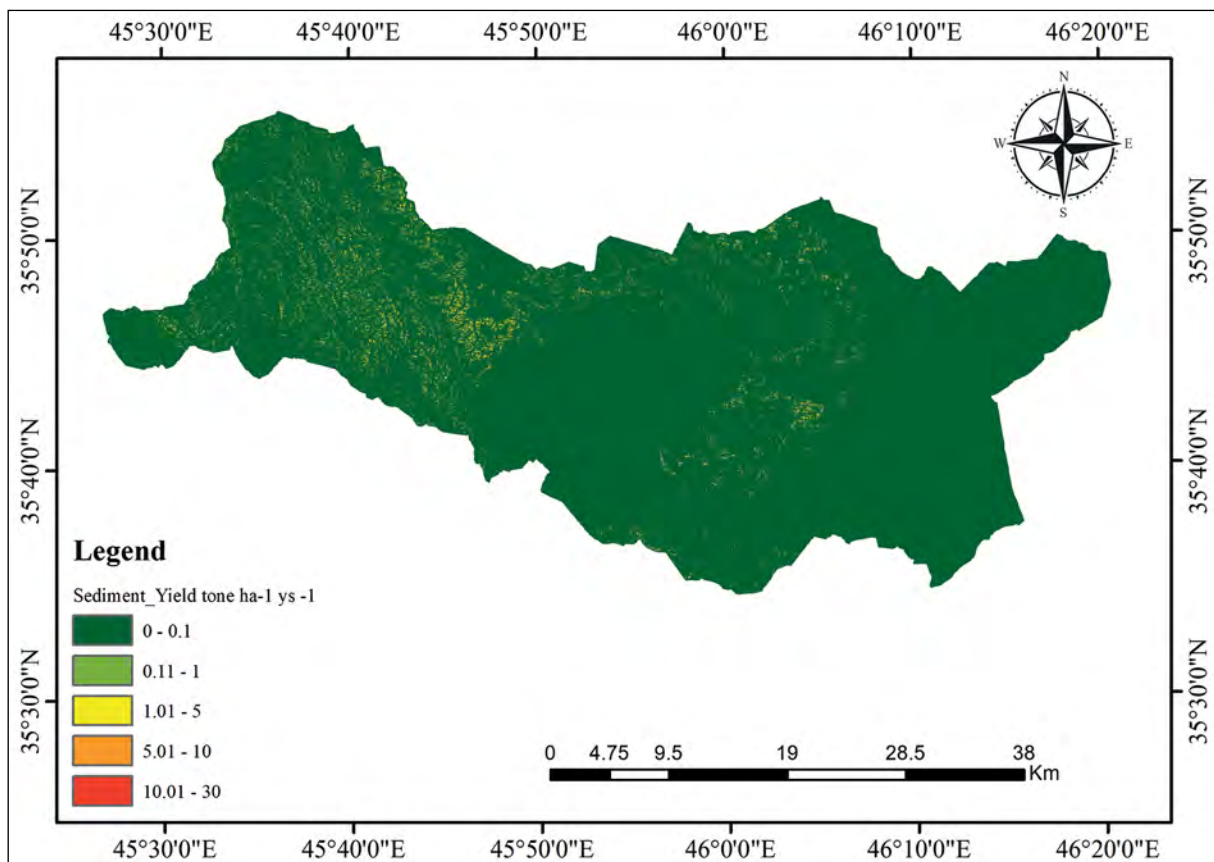


Figure 12- Sediment yield map.

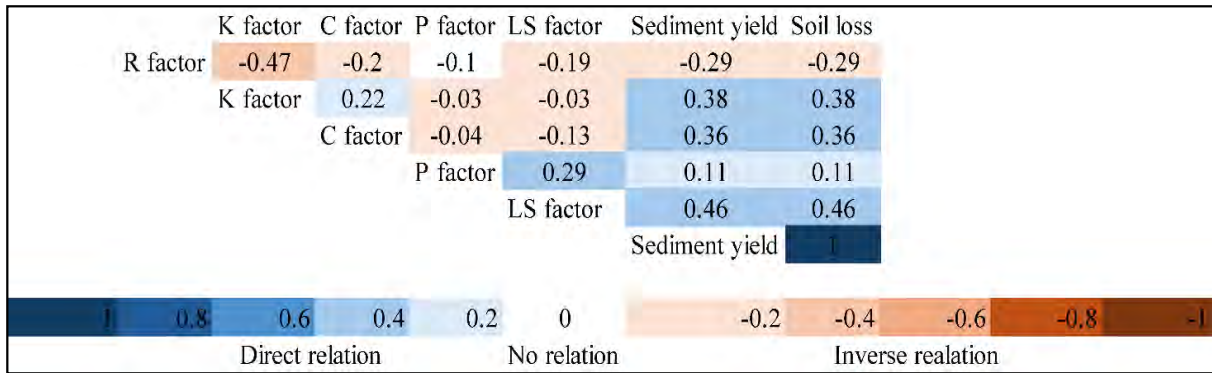


Figure 13- The correlation coefficient between RUSLE parameters.

6. Conclusion

Calculating soil loss, sediment yield, and soil erosion risk in a significant watershed like the Kanarwe river basin in Iraq by the RUSLE equation is very important. It guarantees the construction of any infrastructure, such as dams, railroads, urbanization, food production, and agriculture in the present and future. This study's findings include a spatially distributed mean annual soil loss, sediment yield, and tolerable soil loss in the Kanarwe river basin. The annual soil loss rate is extended from 0 to 58 t ha⁻¹ yr⁻¹, while the sediment yield is between sediment 0 to 30 t ha⁻¹ yr⁻¹. About 25 % of the watershed is moderate to severe, above FAOs tolerable soil loss rate, and 75% is below FAOs tolerable soil loss, this result reveals that the soil condition is degradation condition in Iraq and Iran. However, the main factors controlling the soil loss are topography, soil erodibility, and crop factors instead of rainfall erosivity R factor due to the geological condition Kanarwe river basin. The statistical analysis reveals that topography flysch rocks denudation makes the soil in ZSZ more vulnerable to weathering and erosion but less by anthropogenic factors. Finally, this study recommends the construction of sand dams in narrow gorges and artificial plantations at steep slopes upstream to control soil erosion risk effectively. The estimation of gully erosion is recommended because the RUSLE does not consider it.

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Tectono-stratigraphy, formation and emplacement ages of the Beyşehir-Hoyran Nappes in the south of the Sultan Dağları (Isparta, SW Türkiye)

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ABSTRACT

The Beyşehir-Hoyran Nappes (BHN), in the south of Sultan Dağları, consist of ophiolite, mélange and nappe slices of different lithologies and ages belonging to the oceanic crust and related rocks, which originated from the northern Neotethys and were thrust southwards over the Taurus Platform. These nappes, namely the Marmaris Ophiolite Nappe, the Gülbahar Nappe and the Domuzdağ Nappe, are represented by three tectonically related Nappe-slices that extend as a narrow NW-SE trending belt to the south of the Sultan Dağları. The Marmaris Ophiolite nappe (Upper Cretaceous) is formed by three subunits namely the Marmaris Ophiolite, the Kızılcadağ Mélange and the Yenice kale metamorphics. Hornblende minerals from amphibolites of the Yenice kale metamorphic rock unit yielded a ⁴⁰Ar-³⁹Ar age of 93.9±0.34 Ma (Cenomanian-Turonian boundary). In addition, the Middle Triassic-Early Jurassic Orluca Formation of the Gülbahar nappe is mapped for the first time in the Sultan Dağları region. Paleontological and radiometric ages obtained from this study show that the formation of the BHN should began in the Turonian and ended in the Late Maastrichtian. The nappes, on the other hand, were emplaced over southern Sultan Dağları in the early-middle Paleocene and reached their present position as a result of late Eocene movements.

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1. Introduction

Türkiye, which is situated on the Alpine-Himalayan orogenic belt, has been significantly shaped by the closure of the Neotethys Ocean during the Late Cretaceous-Early Cenozoic and the subsequent collision between the Anatolian and Arabian plates by the end of the Middle Miocene (Şengör and Yılmaz, 1981). During this process, several suture zones developed in Türkiye. The İzmir-Ankara-Erzincan suture zone (Şengör and Yılmaz, 1981; Görür et al., 1983; Okay and Tüysüz, 1999) and the Inner-Tauride suture zone (Görür et al., 1984; Robertson and Dixon,

1984; Okay and Tüysüz, 1999), formed by the closure of the northern branch of the Neotethys Ocean, constitutes the northern boundary of the Anatolide-Tauride Block (Okay and Tüysüz, 1999), while the Bitlis-Zagros suture zone (Şengör and Yılmaz, 1981; Yılmaz, 1993; Parlak et al., 2009), formed by the closure of the southern branch, defines the southern boundary.

The Anatolides (*sensu* Ketin, 1966), located in the Anatolide-Tauride Block, represent those parts of the Taurides that underwent regional metamorphism during the Late Cretaceous-Early Cenozoic due to

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the Alpine orogeny (e.g., Pourteau et al., 2013). The Taurides consist mainly of parautochthonous and allochthonous, imbricated, folded, non-metamorphic or low-grade metamorphic units (Geyikdağ, Aladağ, Bolkardağ, Bozkır, Antalya and Alanya units) (Özgül, 1976). The Sultan Dağları and its surroundings is one of the regions where the relationships between Geyikdağ, Aladağ and Bozkır units can be best observed.

The Sultan Dağları is a mountain range consisting of Paleozoic to Mesozoic low-grade metamorphic rocks and a Cenozoic cover that extends between the Afyon Zone (Okay, 1984; Okay and Tüysüz, 1999; Candan et al., 2005) and the Anamas-Akseki Autochthon (Şenel et al., 1992) with a NW-SE direction and forms the NE flank of the Isparta Angle (*sensu* Poisson et al., 1984). The NW-SE trending allochthonous units extending south of the Sultan Dağları are referred to as the BHN (Gutnic et al., 1968; Monod, 1977) (Figure 1). The BHN is composed of

ophiolites, mélanges and rocks of different ages and lithologies, derived from the northern branch of the Neotethys Ocean (İzmir-Ankara-Erzincan and/or Inner Tauride Oceans) and emplaced on the Taurus platform (Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Elitok and Drüppel, 2008). The BHN can be regarded as the eastern continuation of the Lycian Nappes (*sensu* Şenel et al., 1989), which lies west of the Isparta Angle.

Units of the BHN are genetically represented in the northern branch of the Neotethys by rift-related rocks in the Triassic, by carbonate and clastic sediments of the passive continental margin in the Jurassic-Cretaceous, and by supra-subduction ophiolites and mélanges associated with northward subduction in the Late Cretaceous (Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Elitok and Drüppel, 2008; Mackintosh and Robertson, 2009; Parlak et al., 2019). The formation and emplacement processes of the BHN are key to understanding the geodynamic evolution of

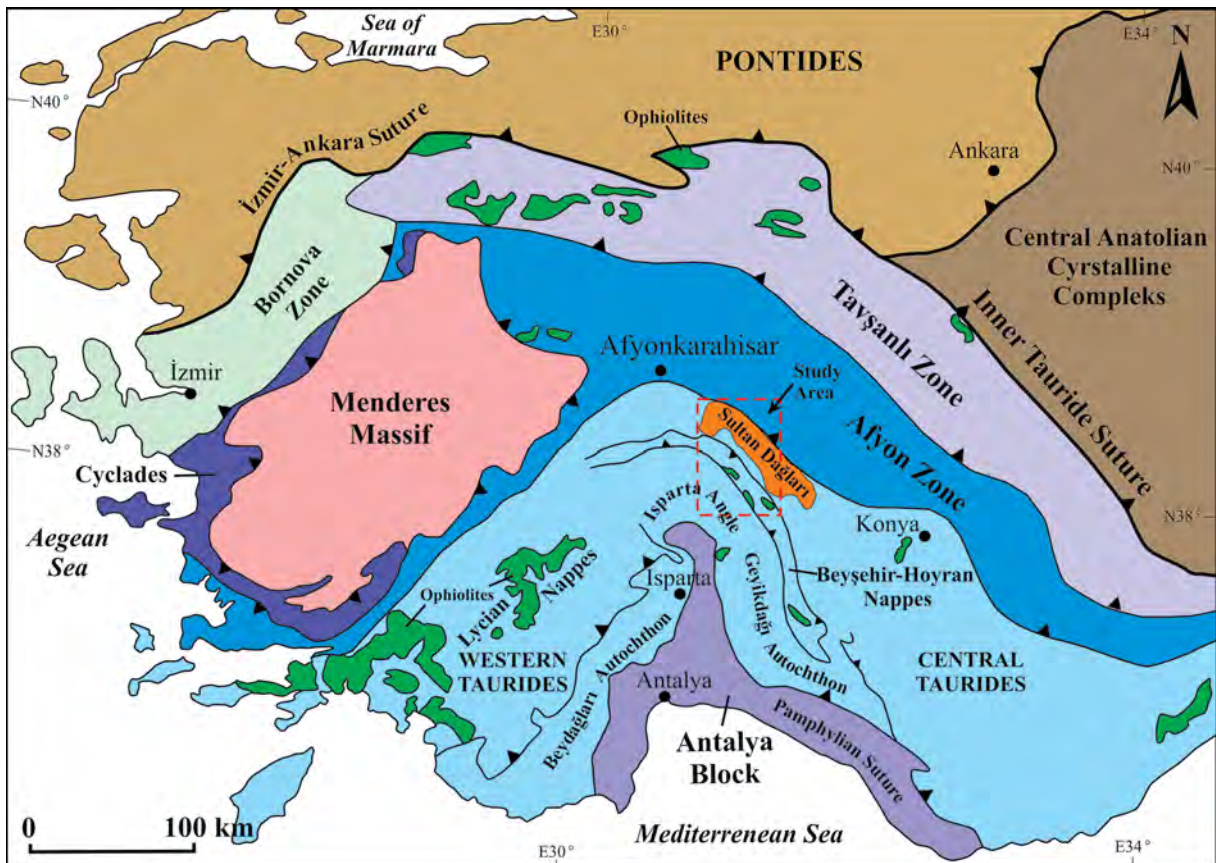


Figure 1- Tectonic map of the western Anatolia (modified after Pourteau et al., 2010, 2013).

the region. The general assumption about these nappes is that they were formed in the Late Cretaceous due to the closure of the northern branch of the Neotethys and emplaced on the Taurus platform during the Late Cretaceous-Eocene interval (Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Elitok and Drüppel, 2008; Güngör, 2013). Constraining these too-long intervals by paleontological and radiometric dating provides more accurate data on the formation and emplacement processes of the BHN. In addition, the majority of present studies of the BHN are concerned with the age and geochemical properties of the ophiolites and metamorphic sole rocks (e.g. Elitok and Drüppel, 2008; Çelik and Delaloye, 2006; Parlak et al., 2019). Thus, the tectono-stratigraphy of the nappes and their relationship to the cover rocks has not been sufficiently investigated.

This study aims to provide a detailed tectono-stratigraphy of the BHN and to constrain the formation and emplacement ages of these nappes by integrating the biostratigraphic, radiometric and structural data.

1.1. Material and Method

Basic studies to identify tectono-stratigraphic features and solve geological problems are based on the production of geological maps at a scale of 1:25,000 in the field. The paleontological and petrographic samples studied in the MTA laboratories were collected in the field from appropriate levels of the formations. Radiometric dating studies were carried out at the Nevada Isotope Geochronology Laboratory (USA) using the ^{40}Ar - ^{39}Ar method on hornblende minerals from amphibolites of the Yenicekale Metamorphics.

2. Tectono-stratigraphy

Early Cambrian to Quaternary rock units of different origins and lithologies outcrop in the study area (Figure 2). Formations of the Geyikdağı, Bolkardağı/Aladağ and Bozkır units form the basement of the region (Özgül, 1976).

The widespread parautochthonous rocks of the Geyikdağı Unit (Özgül, 1976) are studied under the names of Sultandağı Unit (*sensu* Özgül et al., 1991) and Anamas-Akseki Autochthon (*sensu* Şenel et al., 1992) in the study area, while the allochthonous rock assemblages of the Bolkardağı/Aladağ Unit and the

Bozkır Unit are studied under the names of Çay Unit (*sensu* Özgül et al., 1991) and the BHN (*sensu* Monod, 1977) respectively (Figures 2 and 3).

The late Paleocene-Lutetian Celeptaş Formation and deposits of the Miocene-Pliocene Yalvaç and Ilgın basins form the Cenozoic cover. Quaternary deposits are the youngest cover in the region. As the units of the BHN observed in the study area are a continuation of the Lycian Nappes and show strong stratigraphic similarities, the nomenclature of the Lycian Nappes is followed to ensure regional correlation.

2.1. Sultandağı Unit

The Sultandağı Unit forms the dominant rock mass of the Sultan Dağları. The unit consists of lower Cambrian to Upper Cretaceous metasedimentary and metavolcanic rocks (Özgül et al., 1991; Ergen et al., 2021). The Sultandağı Unit, which represents a transgressive sequence associated with back-arc basin development in the early Paleozoic (Linnemann et al., 2008; Nance et al., 2010; Dedeoğlu et al., 2021), is composed of quartzite, marble and turbiditic clastic rocks. An upper Paleozoic succession of quartzite, phyllite, recrystallized limestone and dolomite overlies the early Paleozoic units after the Late Devonian unconformity. The Mesozoic succession begins in the Middle Triassic with terrestrial metaconglomerates and metasandstones, overlain by Jurassic-Cretaceous metacarbonate rocks. The Sultandağı Unit underwent lower greenschist metamorphism (Güngör, 2013) in the early-middle Paleocene based on the emplacement of the Çay Unit and the BHN (Ergen et al., 2021; Ergen, 2023). The late Paleocene-Lutetian Celeptaş Formation and the Neogene deposits of the Yalvaç and Ilgın basins unconformably overlie the Sultandağı Unit.

2.2. Anamas-Akseki Autochthon

The unit consists of non-metamorphic Jurassic-Cretaceous aged (Şenel et al., 1992) neritic carbonates belonging to the Geyikdağı Unit in the south of the Sultan Dağları. The Anamas-Akseki Autochthon (*sensu* Şenel et al., 1992), observed in the SW parts of the study area, is represented by Jurassic-Cenomanian grey-colored, medium to thick-bedded limestones and dolomites. The lower contact of the unit is not seen in

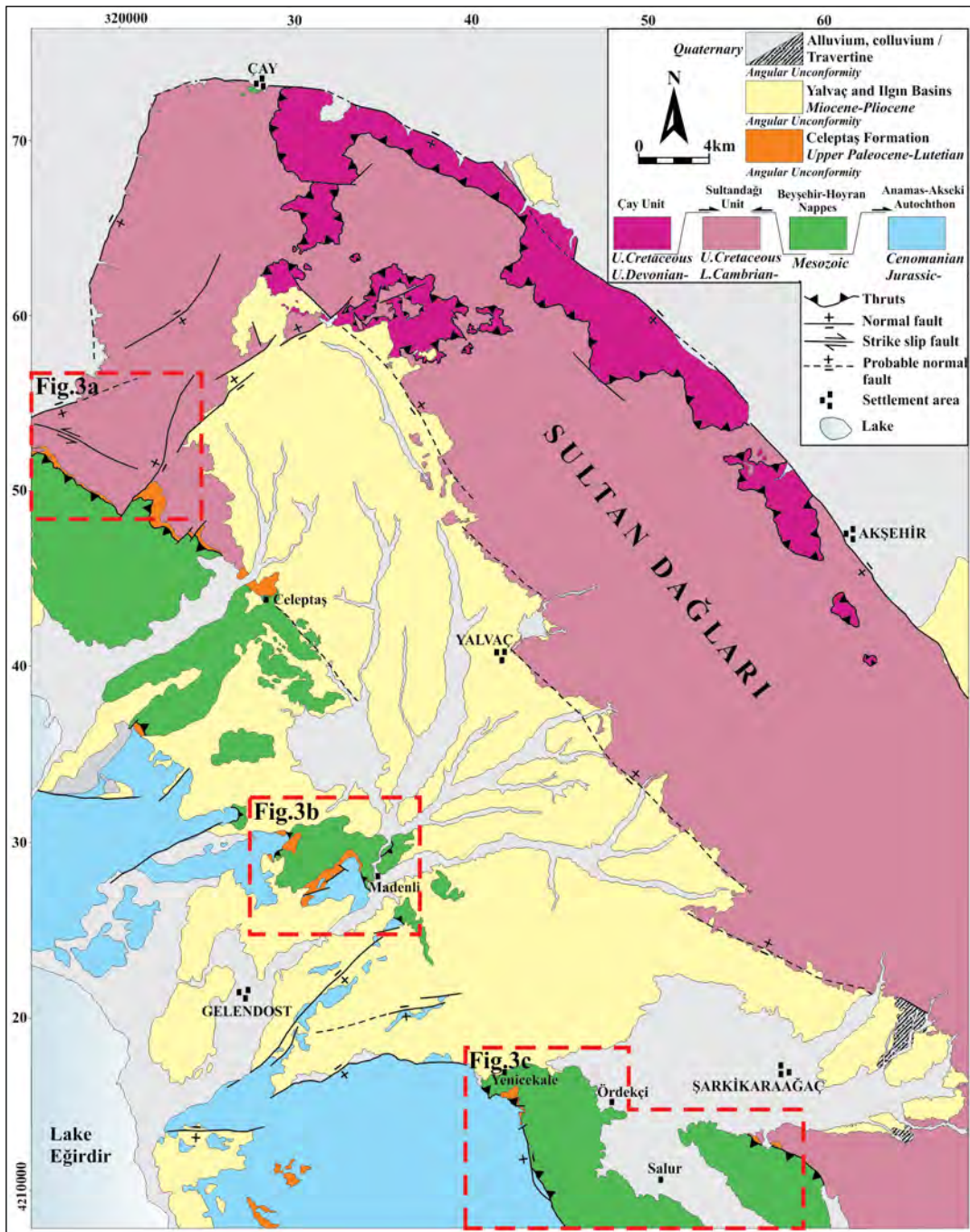


Figure 2- Simplified geological map of the Sultan Dağları and its surroundings (simplified after Ergen et al., 2021).

the study area, being unconformably overlain by the Celeptaş Formation and tectonically overlain in places by ophiolites of the BHN.

2.3. Çay Unit

The Late Devonian to Cretaceous lower greenschist-facies rocks of the Bolkardağı/Aladağ Unit,

outcropping in a NW-SE trending narrow strip along the northeastern edge of the Sultan Dağları, are called the Çay Unit (Özgül et al., 1991; Ergen et al., 2021). The Çay Unit, which consists of metasedimentary rocks intercalated with metavolcanics, tectonically overlies the Sultandağı Unit and is also tectonically overlain by the BHN (Figure 2).

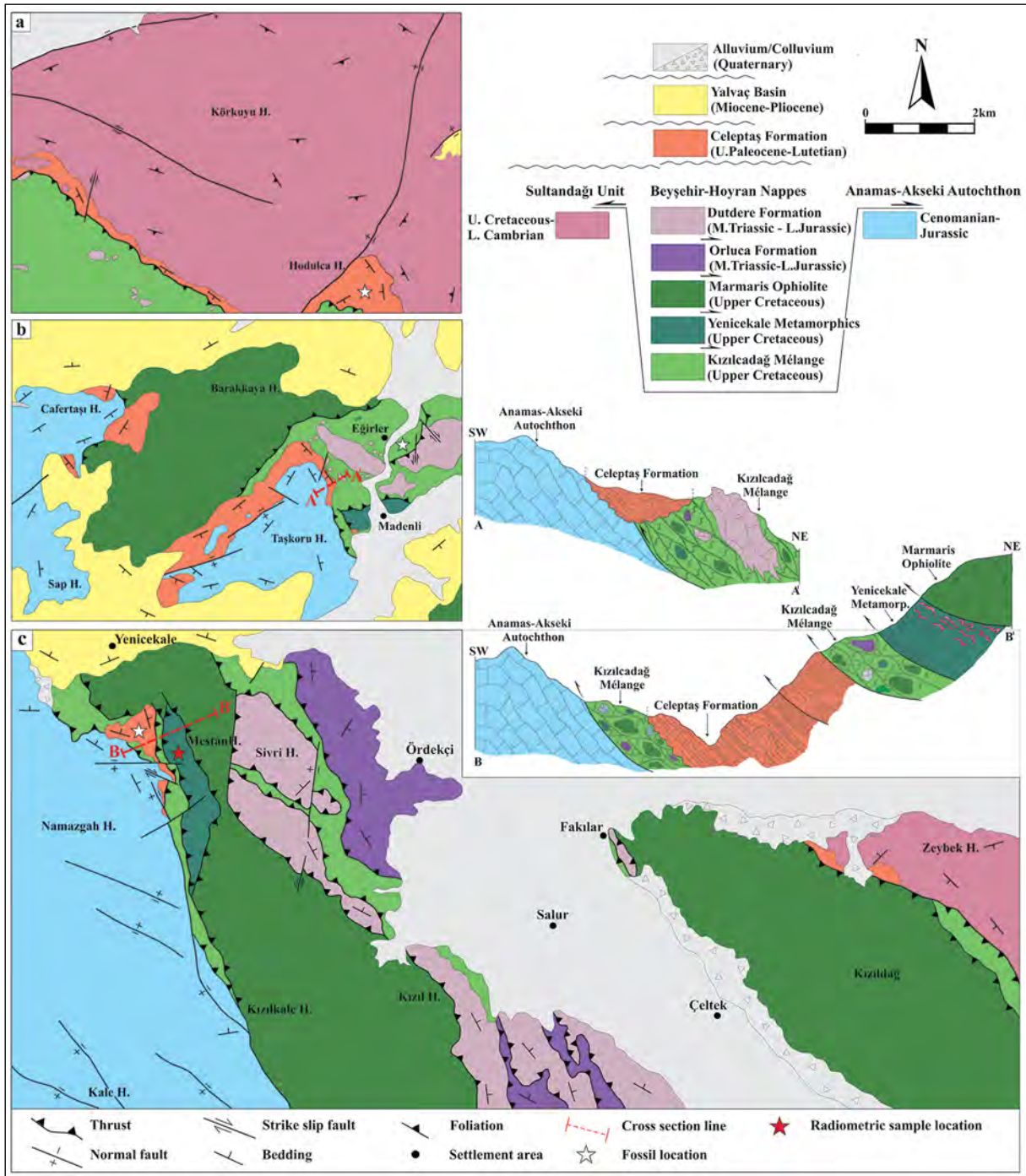


Figure 3- Geological map and cross-sections showing the relationship of the units of the BHN to the parautochthonous basement and cover rocks (modified after Ergen et al., 2021).

2.4. Beyşehir-Hoyran Nappes

The BHN are the allochthonous masses composed of ophiolite, mélangé and associated blocks and slices of different lithologies and ages, formed as a result of the closure of the northern branch of the

Neotethys Ocean and were emplaced on the Taurus Platform during the Late Cretaceous-early Cenozoic (Monod, 1977; Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Ergen et al., 2021). These nappes, which are exposed in a NW-SE trending narrow belt and are tectonically interrelated with each other, are the

Marmaris Ophiolite Nappe (the Kızılcaadağ Mélange, the Yenicekale Metamorphics and the Marmaris Ophiolite), the Gülbahar Nappe and the Domuzdağ Nappe from bottom to top (Ergen et al., 2021; Ergen, 2023) (Figure 4).

The Marmaris Ophiolite Nappe (Upper Cretaceous) is formed by three subunits as the Marmaris ophiolite, the Kızılcaadağ Mélange and the Yenicekale

Metamorphics. The Marmaris Ophiolite is composed of dunite, harzburgite, serpentinite and diabase dykes, while the Kızılcaadağ Mélange is composed by blocks of different ages and lithologies contained in an ophiolitic/sedimentary matrix. The Yenicekale Metamorphics consist mainly of amphibolites that characterize a sub-ophiolitic metamorphic sole (Elitok and Drüppel, 2008). The Orluca Formation (Şenel et al., 1989), which is composed of micritic limestone,

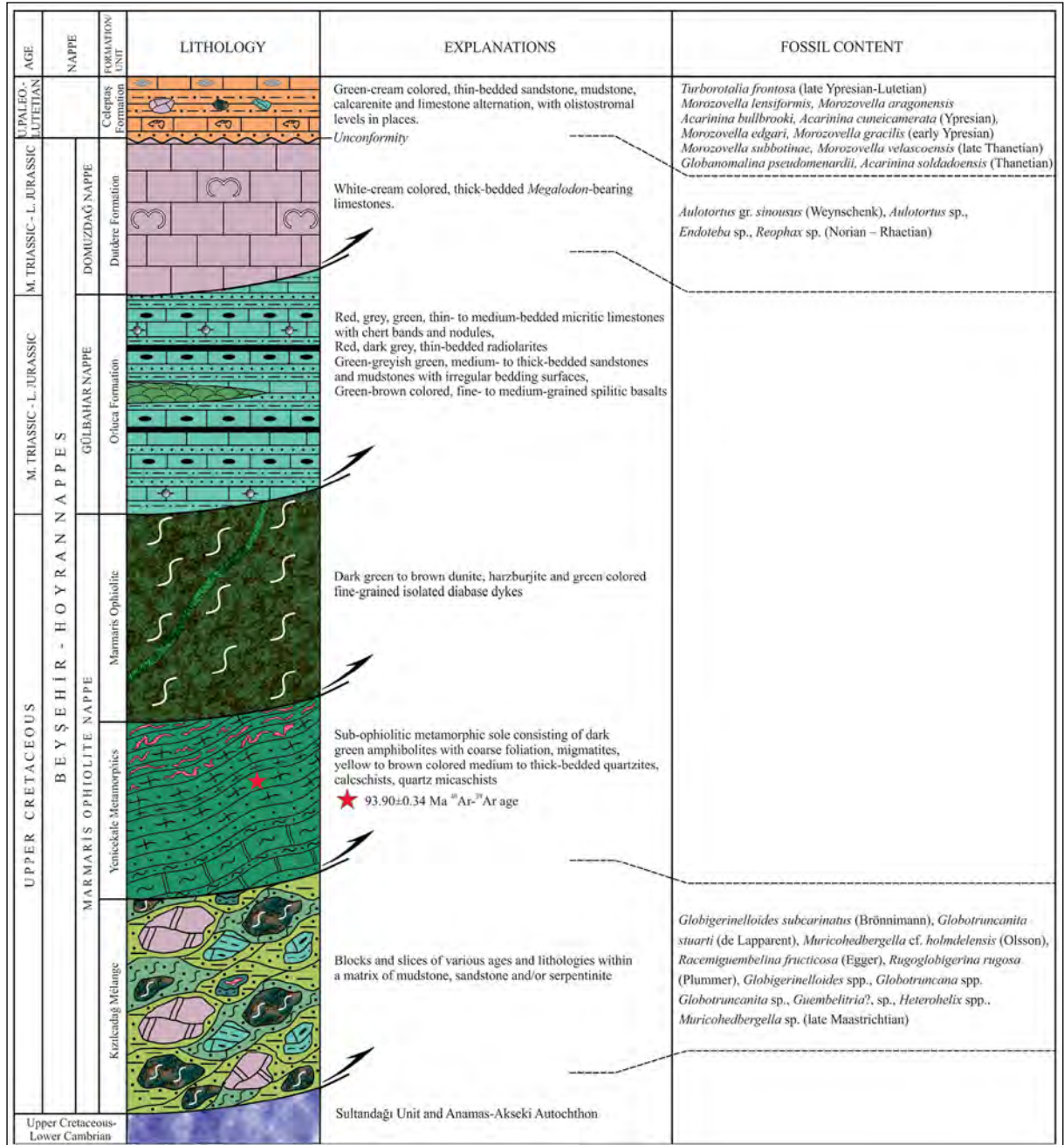


Figure 4- Tectono-stratigraphic column of the BHN in the study area (not to scale).

radiolarite, chert, mudstone and basalt intercalations, represents the Gülbahar Nappe. The Domuzdağ Nappe at the top is represented by the Dutdere Formation (Ersoy, 1989), which consists of *Megalodon*-bearing limestones.

The Celeptaş Formation unconformably overlies the BHN, which tectonically rest on the parautochthonous Sultandağı Unit and the Anamas-Akseki Autochthon.

2.4.1. Marmaris Ophiolite Nappe

Kızıladağ Mélange (Kkm): It is composed of blocks and slices of different lithologies contained in an ophiolitic and sedimentary matrix (Poisson, 1977; Şenel et al., 1989; Ergen et al., 2021). The majority of the matrix consists of green, intensely sheared serpentinites and, to a lesser extent, mudstones. Blocks and slices belonging to the Domuzdağ and Gülbahar Nappes and the Marmaris Ophiolite are observed in the matrix. Blocks and slices of the Domuzdağ and Gülbahar Nappes and the Marmaris Ophiolite, ranging from a few centimetres to tens of metres, are observed in the matrix, and they are composed of such rocks as marble, micritic limestone, chert, radiolarite, dunite, harzburgite, gabbro, diabase and amphibolite (Figures 5a and 5b).

The lower contact of the Kızıladağ Mélange is tectonic. It tectonically overlies the Jurassic-Cretaceous carbonates of the Sultandağı Unit to the

south of Şarkikaraağaç, while tectonically overlying the Jurassic-Cenomanian limestones of the Anamas-Akseki Autochthon to the south of Yenicekale (Figures 5a and 6a-d). The Celeptaş Formation covers the Kızıladağ Mélange with an unconformable contact. In addition, the Marmaris Ophiolite, Yenicekale Metamorphics, Domuzdağ and Gülbahar Nappes tectonically overlie the Kızıladağ Mélange. It is also unconformably overlain by deposits of the Miocene-Pliocene Yalvaç Basin to the south of Yenicekale village (Figure 3).

The age of the mélange in the Lycian Nappes is accepted as Late Cretaceous based on the planktonic foraminiferal assemblages (Poisson, 1977; Şenel et al., 1989). It was also reported that the age of the mélange near Bozkır district (Konya) is late Maastrichtian (Özgül, 1997; Andrew and Robertson, 2002).

In this current study, planktonic foraminiferal assemblage including *Globigerinelloides subcarinatus* (Brönnimann), *Globotruncanella stuarti* (de Lapparent), *Muricohedbergella* cf. *holmensis* (Olsson), *Racemiguembelina fruticosa* (Egger), *Rugoglobigerina rugosa* (Plummer), *Globigerinelloides* spp., *Globotruncana* spp., *Globotruncanella* sp., *Guembelitra*?, sp., *Heterohelix* spp., *Muricohedbergella* sp. species, indicating a late Maastrichtian age, has been identified from the mudstones that forms the sedimentary matrix of the Kızıladağ Mélange around the Eğirler village.

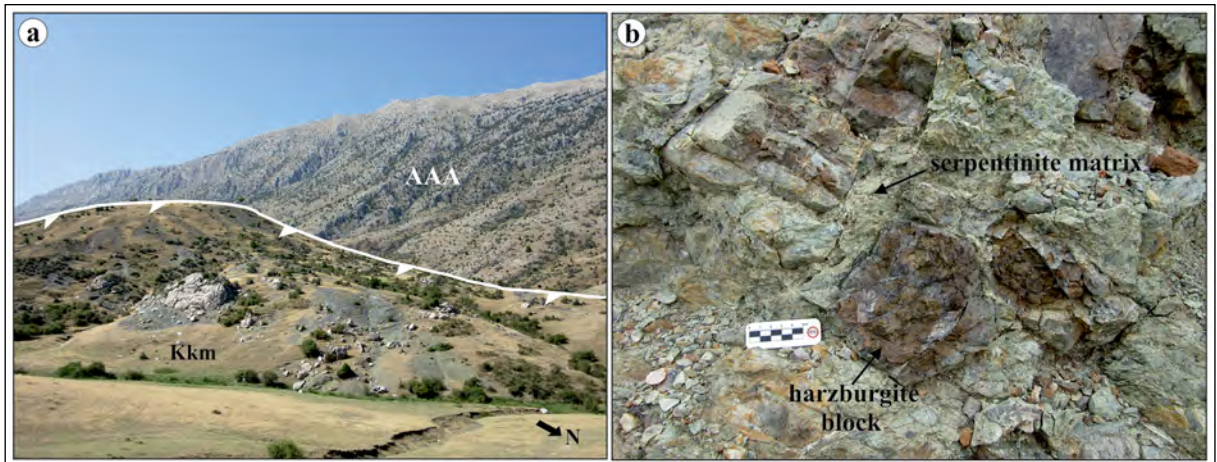


Figure 5- a) General view of the Kızıladağ Mélange (Kkm) and its tectonic contact with the Anamas-Akseki Autochthon (AAA), b) harzburgite blocks in the serpentinite matrix of the mélange, south of Yenicekale village, Şarkikaraağaç.

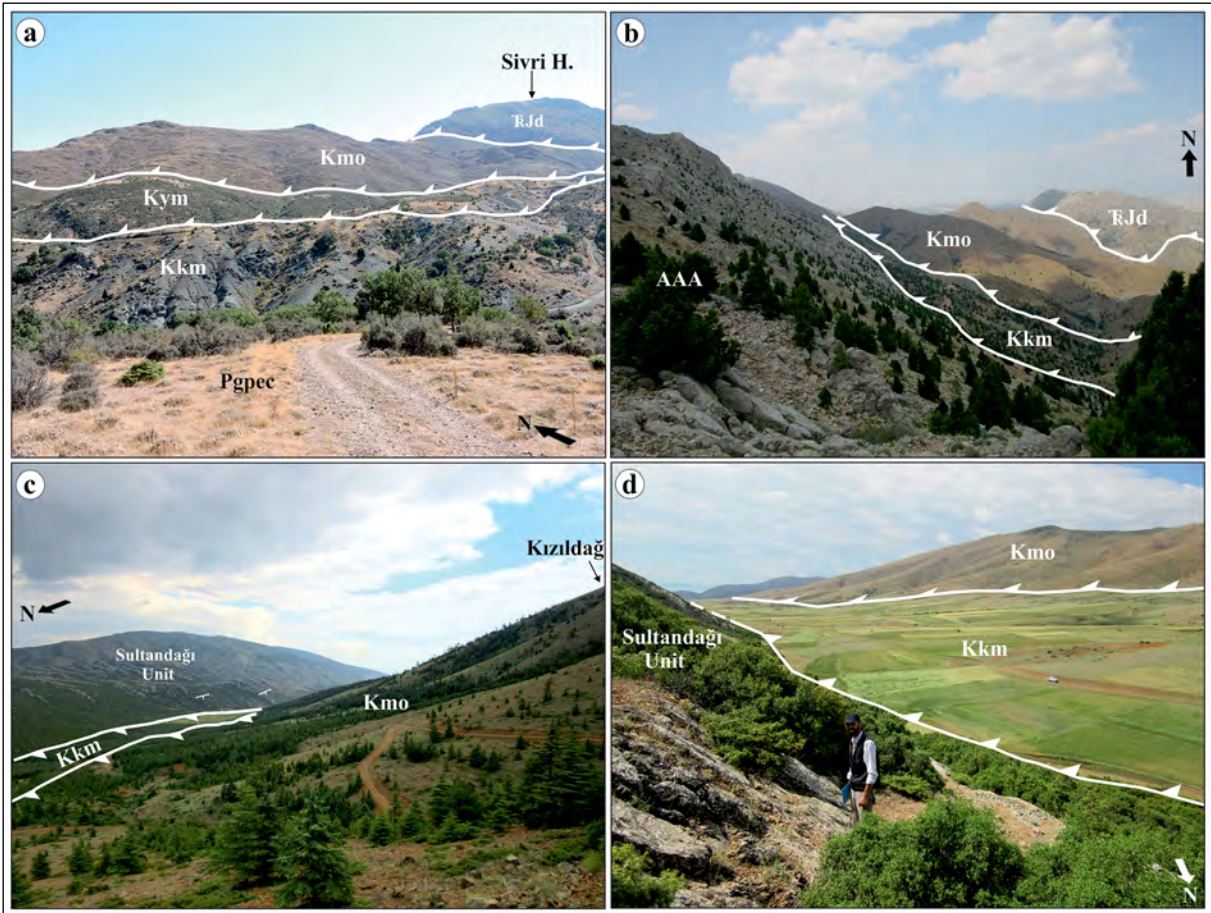


Figure 6- Tectonic relationships of units of the BHN. a, b) SW-vergent thrust contacts, SE of Yenicekale village, Şarkikaraağaç, c, d) NE-vergent back-thrusts, S of Şarkikaraağaç. Kmo: Marmaris Ophiolite, Kkm: Kızıladağ Mélange, Kym: Yenicekale metamorphics, T_RJd: Dutdere Formation, AAA: Anamas-Akseki Autochthon.

The Kızıladağ Mélange was formed by the emplacement of blocks and slices of different lithologies and ages in an ophiolitic/sedimentary matrix during the Late Cretaceous northward subduction of the northern branch of the Neotethys Ocean, and thus gained a chaotic structure depending on intense tectonics.

Yenicekale Metamorphics (Kym): The rock assemblage containing amphibolite, amphibole-biotite schist, calcschist and quartz schist, which are observed as sub-ophiolitic metamorphic sole in the BHN (Elitok and Drüppel, 2008), are referred to as the Yenicekale Metamorphics (Ergen et al., 2020, 2021).

The Yenicekale Metamorphics are composed of dark green, medium to coarsely foliated amphibolites, with ptygmatic folds in places (Figures 7a and 7b),

and grey-blue, thin to medium-bedded calcschists and yellow-brown, thin to medium foliated quartzites (Figure 7d) and quartz schists. Amphibolites, which are the dominant lithologies of the unit, are composed of plagioclase, hornblende, chlorite and titanite minerals, with a granonematoblastic to nematoblastic texture (Figure 7f). Sericitization and argillization are common in plagioclase minerals. Quartzites are composed of quartz, feldspar, mica and apatite minerals, with a granoblastic texture. These rocks show a metamorphic grade from amphibolite to greenschist facies (Elitok and Drüppel, 2008). Inverted metamorphic grade, one of the characteristic features of the sub-ophiolitic metamorphic rocks, can be observed within the Yenicekale Metamorphics. The amphibolites are at the top of the sequence, while the greenschist facies rocks such as quartzite, quartz schist and calcschist are at the bottom of the sequence. These

metamorphic rocks are intruded by non-metamorphic isolated diabase dykes, as is in the Marmaris Ophiolite (Figure 7e). These dykes, which yield U-Pb zircon ages ranging from 90.8 ± 1.6 Ma to 87.6 ± 2.1 Ma (Parlak et al., 2019), are geochemically tholeiitic and,

to a lesser extent, alkaline in composition (Elitok and Drüppel, 2008; Parlak et al., 2019).

The Yenicekale Metamorphics are generally observed between the ophiolites and the ophiolitic



Figure 7- a, b) General view of the amphibolites of the Yenicekale Metamorphics, c) tectonic relationships between the Yenicekale Metamorphics (Kym) and the Marmaris Ophiolite (Kmo) W of Mestan Hill, d) general view of the quartzite, quartz schist and calcschist, SE of Yenicekale village, e) an isolated diabase dyke intruding the amphibolites of the Yenicekale Metamorphics (Kym), SE of Yenicekale village, f) thin section view of an amphibolite sample with a nematoblastic texture, under cross polarized light (Amp: amphibole, Plg: plagioclase).

mélange, representing various thicknesses (Figure 6a-b and 7c). It has a thickness of up to 140 metres around Yenicekale village (Elitok and Drüppel, 2008) and has a lenticular geometry.

Hornblende minerals from amphibolites of this unit in the south of Yenicekale village (coordinates: 36342124N/4214144E) yielded a ^{40}Ar - ^{39}Ar age of 93.90 ± 0.34 Ma (Figure 8). Parlak et al. (2019) obtained ages of 90-94 Ma and 91-93 Ma from amphibolites in the same area using U-Pb and ^{40}Ar - ^{39}Ar methods, respectively. These are in agreement with the 91-93 Ma age of Çelik et al. (2006) determined from amphibole and mica minerals of the Lycian Nappes, Antalya Nappes and metamorphic basement rocks of the BHN.

Based on the analysis of the amphibolites, P-T conditions of 630-770°C and 6 ± 1.5 kbar, corresponding to a burial depth of 18-20 km, are calculated for the metamorphism of these sub-ophiolitic metamorphic

sole rocks (Elitok and Drüppel, 2008). The amphibolites show two different geochemical characters, alkaline and tholeiitic. According to this, the protoliths for alkaline amphibolites are within-plate type alkaline basalts, while the tholeiitic amphibolites are ocean island basalts (Elitok and Drüppel, 2008; Parlak et al., 2019).

Marmaris Ophiolite (Kmo): The ophiolitic rocks outcropping in the Lycian Nappes and the BHN, which are continuations of each other in southwestern Anatolia, have been defined under different names by various researchers. Various names have been given to the rock association, which consists mainly of dunite, harzburgite and serpentized peridotite were referred to various names such as Beyşehir Ophiolite (Ricou et al., 1975; Çelik and Delaloye, 2006) and Hoyran Ophiolite (Demirkol and Yetiş (1983-1984) within the BHN; ophiolites (Andrew and Robertson, 2002) and peridotites (Elitok and Drüppel, 2008) within the Hoyran Nappes; Peridotite Nappe (Graciansky, 1972),

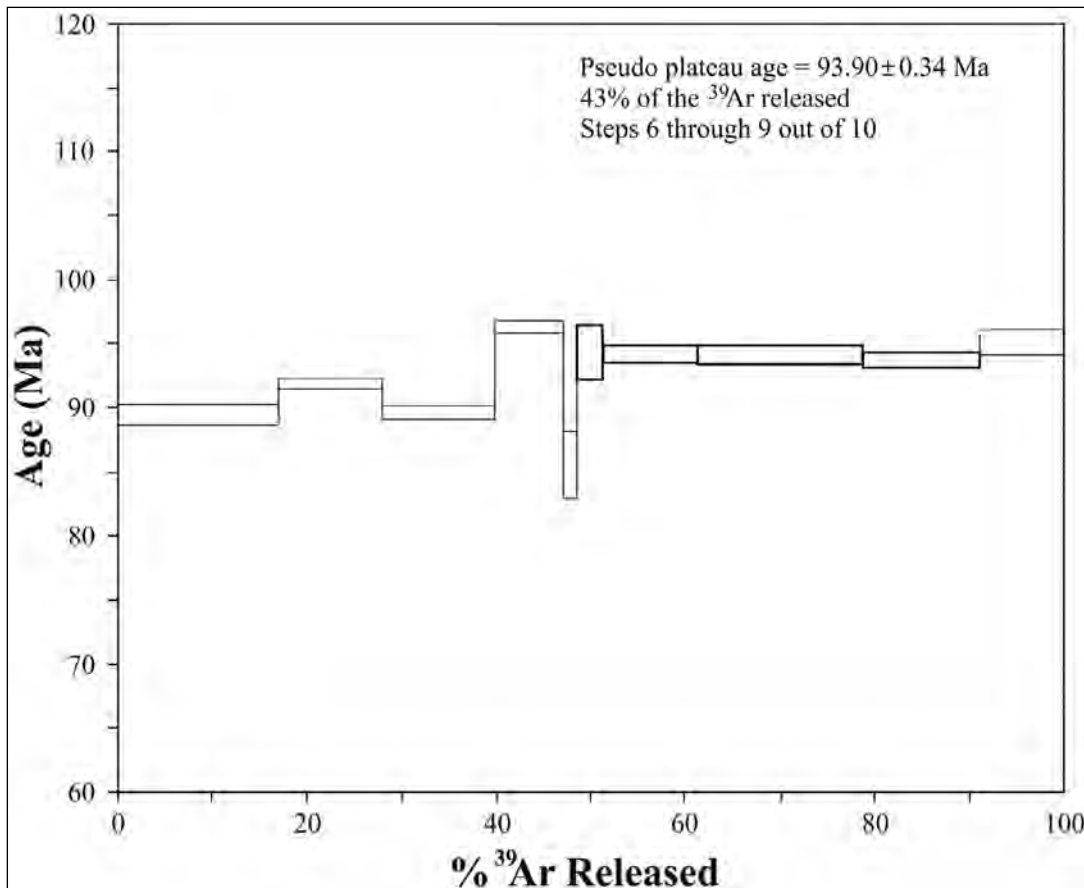


Figure 8- Age spectrum for the amphibolite of the Yenicekale Metamorphics.

Yeşilova-Tefenni Ophiolites (Sarıkaya and Seyrek, 1976), Yeşilova Ophiolites (Sarp, 1976), Marmaris Peridotite (Çapan, 1981) and Marmaris ophiolite (Ergen et al., 2021) within the Lycian Nappes.

The Marmaris Ophiolite, whose lower and upper contacts are tectonic, is up to 1000 m thick. (Şenel, 1997). In the study area, it tectonically overlies the Sultandağı Unit to the south of Şarkikaraağaç and the Anamas-Akseki Autochthon, the Kızılcadağ Mélange and the Yenicekale Metamorphics to the south of Yenicekale (Figure 9c). On the other hand, the units belonging to the Gülbahar and Domuzdağ Nappes rest on the ophiolites with a tectonic contact.

The Marmaris Ophiolite consists of serpentinized dunite and harzburgite and isolated diabase dykes (Figure 9a). The most common rocks observed in this

ophiolitic units are harzburgites, which are medium to coarse grained, with green olivine and grey to black pyroxene minerals, brown on altered surfaces and blackish green to green on fresh surfaces. Dunites, apart from olivine, also include orthopyroxene and chromite minerals, with a mesh texture, light green to greenish grey in color, are less common than harzburgites. Serpentinization is common, especially along fractures. Dunites and harzburgites are intruded by 0.5-2 m thick isolated diabase dykes composed of plagioclase, clinopyroxene and opaque minerals with an ophitic texture. Zircon and titanite minerals from these dykes, which characterize geochemically subduction-related island arc tholeiites, yielded U-Pb ages of 87.5-102 Ma, while hornblende minerals yielded a ^{40}Ar - ^{39}Ar age of 91-93 Ma (Çelik et al., 2006). Stockwork magnesite veins are widely observed in

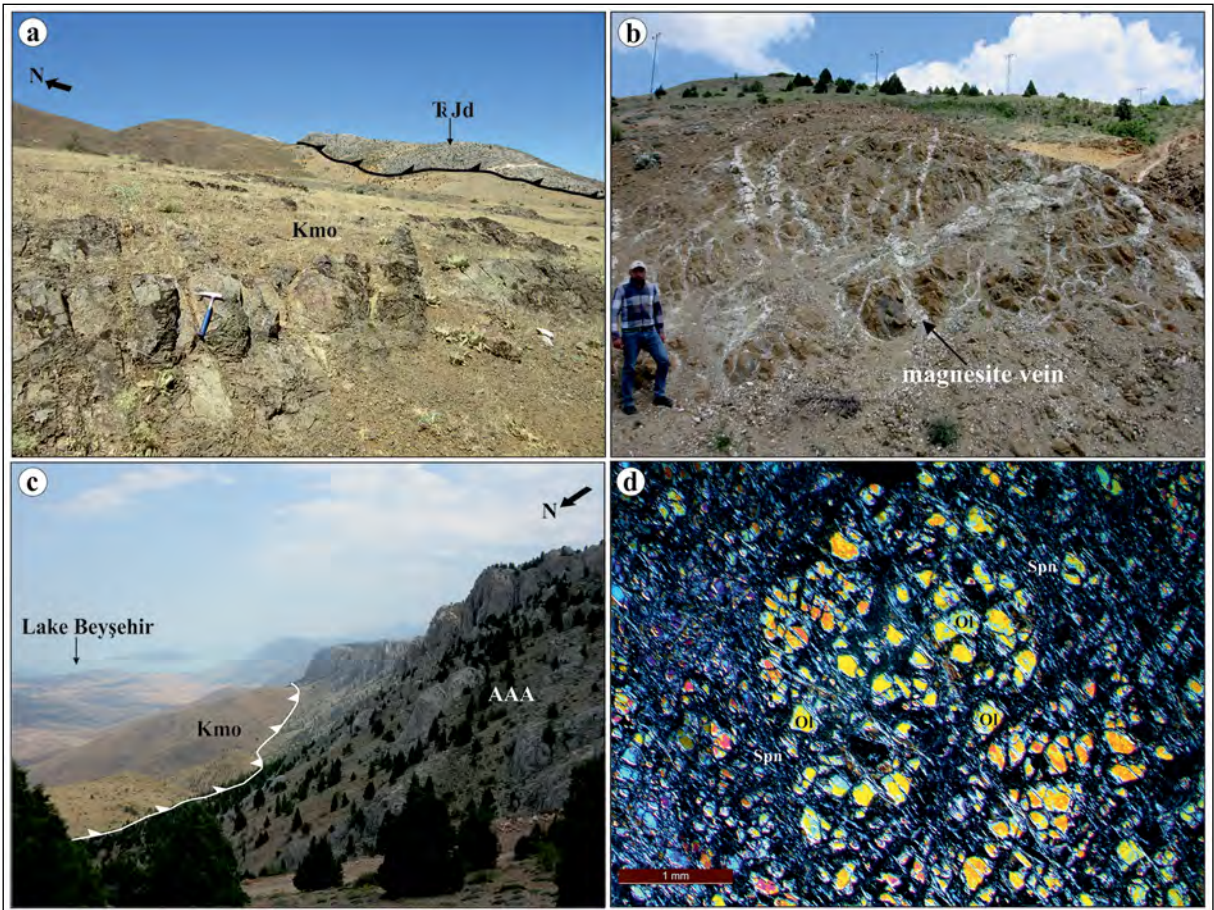


Figure 9- a) Tectonic relationship between the Marmaris Ophiolite (Kmo) and the Dutdere Formation (T_rJ_d) of the Domuzdağ Nappe, NW of Sivri Hill, Yenicekale, b) stockwork magnesite veins in the ophiolites, NW of Eğirler, c) tectonic contact between the Marmaris Ophiolite (Kmo) and the Anamas-Akseki Autochthon (AAA), NW of Lake Beyşehir, d) thin section view of a serpentinized dunite sample under cross polarized light (Ol: Olivine, Spn: Serpentine).

the ophiolites outcropping around Madenli village (Figure 9b).

The unit, which characterizes supra-subduction ophiolites, is accepted as Late Cretaceous in age based on radiometric data and geochemical analysis obtained from its isolated diabase dykes and metamorphic sole (Andrew and Robertson, 2002; Çelik et al., 2006; Elitok and Drüppel, 2008; Parlak et al., 2019; Ergen et al., 2021, Ergen, 2023).

2.4.2. Gülbahar Nappe

The Mesozoic allochthonous sequence of deep marine limestone, chert, radiolarite and mudstone and spilitic basalt is called the Gülbahar Nappe (Graciansky, 1972; Şenel et al., 1994). The Gülbahar Nappe and the Orluca Formation from this nappe have been identified and mapped in this study for the first time in the south of Sultan Dağları.

The Gülbahar Nappe is generally observed as blocks and slices of different sizes in the Kızılcaadağ mélangé. Outcropping west of Ördekçi village and south of Salur (Şarkikaraağaç), the Gülbahar Nappe is represented in the study area by the Orluca Formation. The aforementioned locations where the Orluca Formation is exposed have been misinterpreted by previous researchers (e.g. Elitok and Drüppel, 2008; Parlak et al., 2019) as Upper Cretaceous slope and basin deposits.

Orluca Formation (T_RJo): The formation, which consists of volcanics, radiolarites, cherty micritic limestones, mudstones and sandstones (Şenel, 1989), is exposed west of Ördekçi and south of Salur (Şarkikaraağaç).

The formation is composed of thin to medium-bedded, red, grey, green, intensely folded, deformed micritic limestones with chert bands and nodules, and red, dark grey thin-bedded radiolarites, and green-greyish green, medium- to thick-bedded sandstones and mudstones (Figures 10a-c). They are occasionally accompanied by green, brown, fine- to medium-grained spilitic basalts (Figure 10d), which consist of plagioclase, clinopyroxene, kaersutite, biotite, calcite and opaque minerals with an intersertal texture (Figure

10e). Sericitization and argillization are common in plagioclase minerals, while chloritization is common in pyroxene minerals. Radiolarites consist entirely of fossil radiolarian tests filled with very fine-grained siliceous minerals. The fact that the formation consists of sandstone, mudstone, radiolarite, cherty micritic limestone with basic volcanic intercalations indicates deposition in a slope and basin environment where both volcanism and turbidity currents are occasionally active.

Tectonically overlying the Kızılcaadağ Mélangé (Figures 10 c and 10f), the Orluca Formation is also observed as blocks of various sizes in the mélangé.

A Middle Triassic-Early Jurassic age was assigned to the Orluca Formation by Şenel et al. (1989) on the basis of the fossil assemblages. This age is also accepted in this study.

2.4.3. Domuzdağ Nappe

The unit, which is observed as blocks and slices at various sizes within the Marmaris Ophiolite Nappe and characterized by *Megalodon*-bearing neritic limestones, is recognized as the Domuzdağ Nappe (Poisson, 1977; Ersoy, 1989; Şenel et al., 1994). The “Middle” Triassic-Early Jurassic Dutdere Formation represents the Domuzdağ Nappe in the study area.

Dutdere Formation (T_RJd): The Dutdere Formation (*sensu* Ersoy, 1989) consists mainly of grey, light grey, beyaz and beige-colored, medium- to thick-bedded, *Megalodon* and algae-bearing and occasionally recrystallized limestones, which are very fine-grained biomicrite, microsparite and sparry micrite. The limestones, composed of calcite minerals and poorly preserved fossils in a micritic carbonate cement, are sometimes recrystallized due to the intense tectonics caused by nappe movements. It contains cracks and fractures developed in different directions and is observed as cataclastic and brecciated, especially at the nappe contacts. Şenel et al. (1994) reported that pink to red, thin- to medium-bedded, ammonite-bearing cherty limestones occur outside the study area above the *Megalodon*-bearing lower levels of the formation.

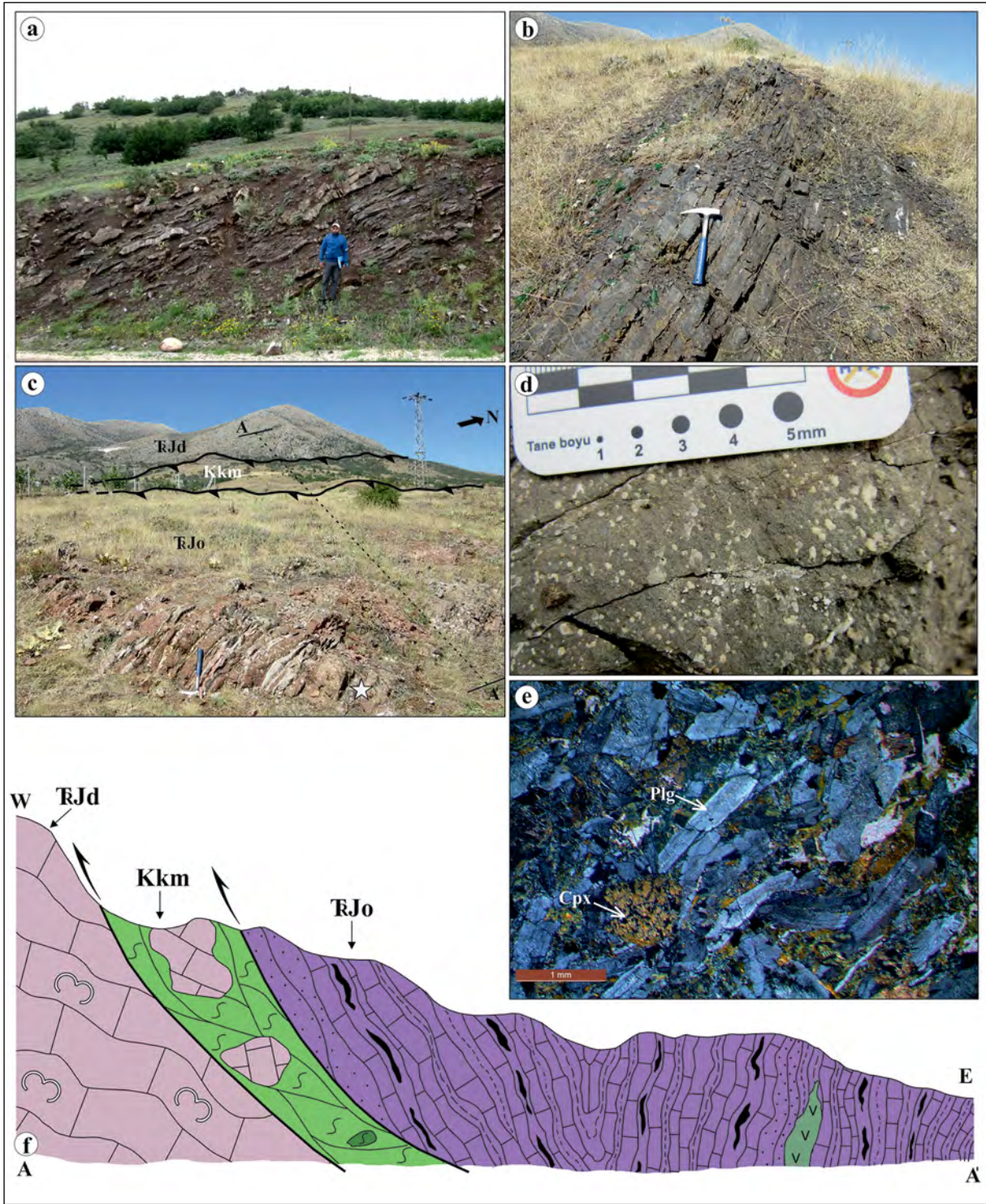


Figure 10- a) Alternation of micritic limestone and mudstone in the Orluca Formation, S of Salur, b) dark grey radiolites, W of Ördekçi, c) tectonic relationships of the Orluca Formation with the Kızılcaadağ mélangé and the Dutdere Formation, NW of Ördekçi, d) a close-up view of the spilitic basalts, e) thin section view of a basalt sample under cross polarized light (Cpx: Clinopyroxene, Plg: Plagioclase), f) a geological cross-section showing relations between the Dutdere Formation (T_RJd), Kızılcaadağ Mélangé (Kkm) and Orluca Formation (T_{Jo}).

In the study area, the formation tectonically overlies the Marmaris Ophiolite, Kızılcaadağ mélangé, and Orluca Formation. The Kızılcaadağ Mélangé is also observed over the Dutdere Formation, depending on the tectonic movements after its primary emplacement. One such contact relationship can be seen at the SE of Yenicekale.

Within the formation, which is easily recognized in the field by bearing *Megalodon* fossils, foraminifera such as *Aulotortus* gr. *sinousus* (Weynschenk), *Aulotortus* sp., *Endoteba* sp., *Reophax* sp. have been determined in this study at the south of Sultan Dağları, indicating a Late Triassic (Norian-Rhaetian) age. Şenel et al. (1989, 1994) assigned a Middle Triassic-Early Jurassic age to the formation, which is also accepted in this study, based on the foraminiferal fauna obtained from the same formation at different locations in the Western Taurides. Based on the lithological characteristics and fossil content, it can be inferred that the formation was deposited in a shallow carbonate shelf environment.

2.5. Cover rocks

2.5.1. Celeptaş Formation (Pgpec)

The Celeptaş Formation (sensu Demirkol, 1977) is mainly composed of thin-bedded limestones, calcarenites, siltstones, mudstones, sandstones and subordinate conglomerates that form different facies and facies associations (Figure 11). The thin-bedded, cream-coloured pelagic limestones and claret-coloured calcarenite and biomicrite with abundant planktonic foraminifera are exposed in the north of the basin (Figures 11a, b). The fine-grained calcarenites are well-sorted and show planar parallel stratification and wave-ripple cross-lamination (Figure 11b). They pass southwards into the interbedded mudstones, siltstones and sandstones, forming mudstone- and sandstone-dominated sequences (Figures 11c, d, e).

Greenish-grey mudstones are laterally extensive and generally massive in the mudstone-dominated sequences (Figure 11c). Very thin-bedded siltstones and very fine to fine sandstones interbedded with mudstones are generally 0.5-5 cm thick, and in lesser amounts up to 15 cm. Sedimentary structures include

planar parallel stratification, normal grading and current ripple cross-lamination.

The sandstone-dominated sequence consists mainly of grey to light brown sandstones and siltstones interbedded with grey mudstones (Figure 11d). The tabular sandstones, which are generally 10-30 cm thick, contain mainly Bouma-type Tbc and lesser amounts of Tabc turbidites. Other sedimentary structures include flute and groove marks, load casts, convolute laminations and some trace fossils. The interbedded sandstones, siltstones and mudstones, form coarsening- and thickening-upward bed packages a few metres thick (Figure 11e). These are erosionally overlain by lenticular channel-fill deposits in the axial part (Figure 11e). The fining upward bedset of the channel-fill deposits consist of coarse sandstones to granule conglomerates, rich in pebble gravels at the base.

The *Nummulites*-bearing pelagic limestones and biomicrites, rich in planktonic foraminifers, indicate a neritic carbonate platform formed on the basin-margin narrow shelf. The calcarenites are interpreted to represent wave-worked, lower shoreface to offshore transition deposits (cf. Clifton, 1981; Dott and Bourgeois, 1982). The mudstones are interpreted to be basin-plain hemipelagic deposits interlayered with thin siltstone and sandstone turbidites in the mudstone-dominated bed packages. The graded sandstone beds with planar stratification are tractional deposits of low-density turbidity currents (Bouma, 1962; Lowe, 1982; Kneller, 1995). The sandstone-dominated coarsening-upwards bed packages are interpreted as turbiditic depositional lobes and the overlying fining-upwards bedsets as channel-fill deposits. The sedimentary facies of these deposits indicate high- and low-density turbidity currents. The composition of the sandstone and the pebbles indicates that the sand grains and clasts are of ophiolitic origin.

The Celeptaş Formation unconformably covers the Sultandağı Unit, the Anamas-Akseki Autochthon and the BHN (Figure 12), while it is tectonically overlain in places by the BHN (Figure 13).

The results of the planktonic foraminiferal analyses performed on the marine mudstone

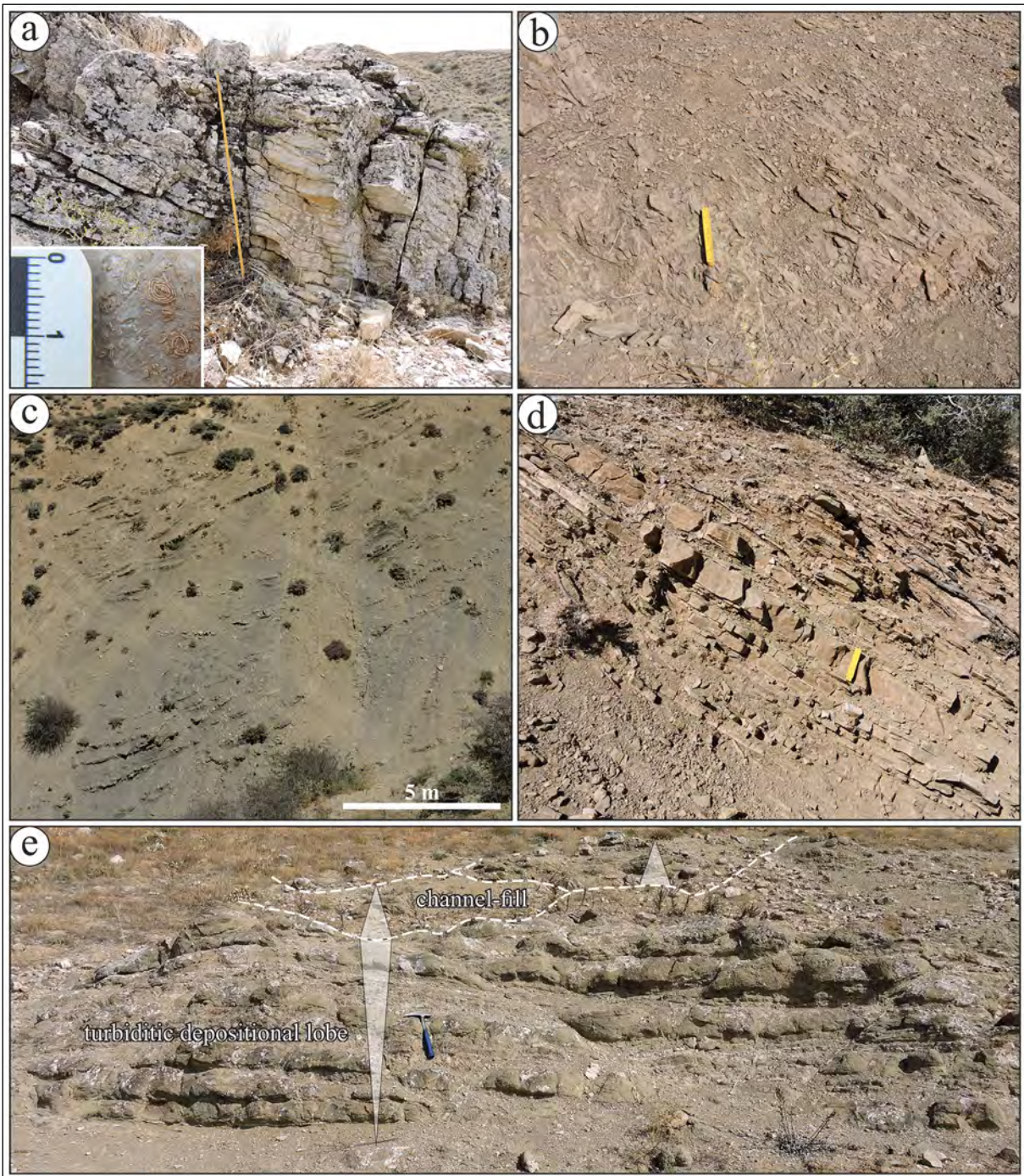


Figure 11- Facies details of Dursunlu Formation, a) Thin-bedded pelagic limestones with *Nummulites* fossils and b) claret-coloured calcarenite and biomicrite, c) Massive mudstones of the basin-plain hemipelagic deposits interbedded with siltstone and sandstone turbidites, d) The sandstone-dominated sequence consists mainly of Bouma-type Tbc and lesser amounts of Tabc turbidites, e) The coarsening- and thickening-upward bed packages of the turbiditic depositional lobes are erosionally overlain by the fining-upwards bedsets of the channel-fill deposits. The ruler is 1 m in figure a and 10 cm in figures b and d, the hammer is 33 cm.

samples from Hodulca Hill northwest of Yalvaç (327382E/4244438N), north of Celeptaş village (327382E/4244438N) and south of Yenicekale (341350E/4214700N) (Figures 2, 3) are given below.

Among the abundant planktonic foraminiferal assemblages of the Celeptaş Formation, the species such as *Globanomalina pseudomenardii* and *Acarinina soldadoensis* in the Thanetian, *Morozovella*

subbotinae and *Morozovella velascoensis* in the late Thanetian, *Morozovella edgari* and *Morozovella gracilis* in the early Ypresian, *Morozovella lensiformis* and *Morozovella aragonensis* in the Ypresian, *Acarinina bullbrooki*, *Acarinina cuneicamerata* and *Turborotalia frontosa* in the late Ypresian to Lutetian levels have been identified in a stratigraphical order as markers. The age of the Celeptaş Formation has been assigned as Late Paleocene-Lutetian based on this planktonic foraminiferal content.

2.5.2. Yalvaç Basin

The Yalvaç Basin, which is formed of terrestrial and lacustrine deposits, is a triangular molasse basin that extends in a NW-SE direction between the Sultan Dağları and the Anamas Mountains (Yağmurlu, 1991;

Ilgar et al., 2021). Deposits of the basin are alluvial fan, lacustrine clastic and carbonate sediments. The formation of the Yalvaç Basin, which began to open after nappe emplacement as a result of orogenic collapse, is known to have been influenced by the break-up or roll-back of the Southern Neotethys slab during the Early Miocene (Koçyiğit and Deveci, 2007; Koçyiğit et al., 2013; Koç et al., 2016; Ilgar et al., 2021). The sediments of the Yalvaç Basin unconformably overlie all pre-Miocene units in the region.

3. Discussions

The formation and emplacement processes of the BHN are important in revealing the geodynamic evolution of the region. The generally accepted aspect

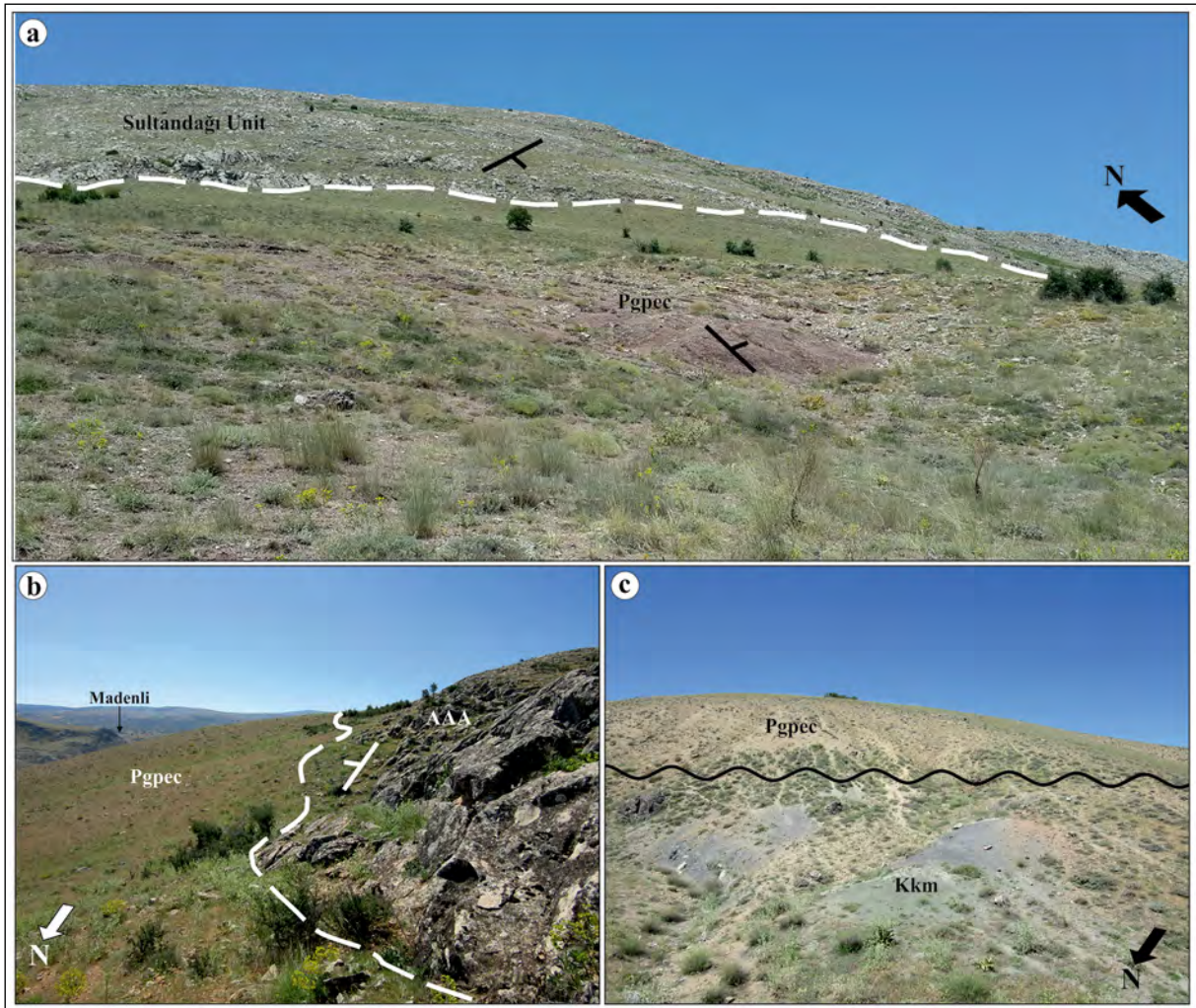


Figure 12- a) The unconformable contact between the Celeptaş Formation and the Middle Jurassic-Upper Cretaceous recrystallized limestones of the Sultandağı Unit, near Hodulca Hill, Yalvaç, b) unconformable contacts between the Celeptaş Formation and Jurassic-Cenomanian limestones of the Anamas-Akseki Autochthon (AAA) and c) Kızılcaadağ Mélange (Kkm) of the BHN, north of Madenli.



Figure 13- a) Thrust contact between the Kızılcadağ Mélange of the BHN and the Celeptaş Formation, SE of Yenice kale, b) close-up of the first figure showing folds.

is that the BHN was formed as a result of horizontal movements due to the closure of the northern branch of the Neotethys Ocean (İzmir-Ankara-Erzincan and/or Inner Tauride Ocean) and settled in the region by thrusting over the Sultan Dağları from north to south during the Late Cretaceous-Eocene (Özgül, 1984; Andrew and Robertson, 2002; Çelik and Delaloye, 2006; Elitok and Drüppel, 2008).

There are several critical issues in dating nappes formed by oceanic subduction processes such as the BHN, the most important of which are the age of the sub-ophiolitic metamorphic rocks, the age of the matrix and blocks of the mélangé and the age of the cover of the nappes. The formation of nappes begins with the development of the sub-ophiolitic metamorphic rocks, which represent the onset of subduction, while it ends with the youngest age obtained from the matrix in the mélangé. The emplacement time, on the other hand, is constrained by the youngest unit the nappes thrust over and the overlying oldest unit.

The sub-ophiolitic metamorphic rocks, which formed during the onset of subduction, are widely accepted as definitive indicators of subduction initiation ages, with metamorphic ages derived from their amphibolites (Çelik and Delaloye, 2006). In this context, the 93.90 ± 0.34 Ma (Cenomanian-Turonian boundary) age data obtained from the Yenice kale Metamorphics is accepted as the onset of the formation of the BHN (Figure 14a).

The ages obtained from the blocks and matrix of the mélangé are another significant parameter related to the formation of the mélangé. Accordingly, the occurrence of Middle Triassic Domuzdağ Nappe, Middle Triassic-Lower Jurassic Gülbahar Nappe and Upper Cretaceous Marmaris Ophiolite Nappe blocks and slices within the Kızılcadağ Mélange and the determination of late Maastrichtian planktonic foraminifera from the matrix of the mélangé indicate that the formation of the BHN lasts possibly from the Turonian to the late Maastrichtian.

The basin type in which the Celeptaş Formation was deposited has been inferred from the sedimentary characteristics of the formation and its tectonostratigraphic relationship with the surrounding units. The rapid transition of the neritic carbonate and offshore transition deposits of the Celeptaş Formation into the deep marine facies indicates a narrow shelf and increased supply of terrigenous sediments to the basin. In addition, intense compressional tectonic deformation during and after deposition suggests that the sediments were deposited in a tectonically controlled basin. Therefore, taken together, we have interpreted the Celeptaş Formation as being deposited in a foreland basin that was fed by and developed in front of the BHN. The flexural subsidence of the basin floor, driven by the crustal load of the BHN, led to the development of sediment accommodation

space. Tectonic loading from the north resulted in the development of a deep-marine environment. Thus, the turbidite deposits of the Celeptas Formation both transgressively overlie the BHN and, in the ongoing

process, the BHN tectonically overlies the Celeptas Formation from north to south (Figure 14d).

The first emplacement age of the BHN must be younger than the tectonically underlying early

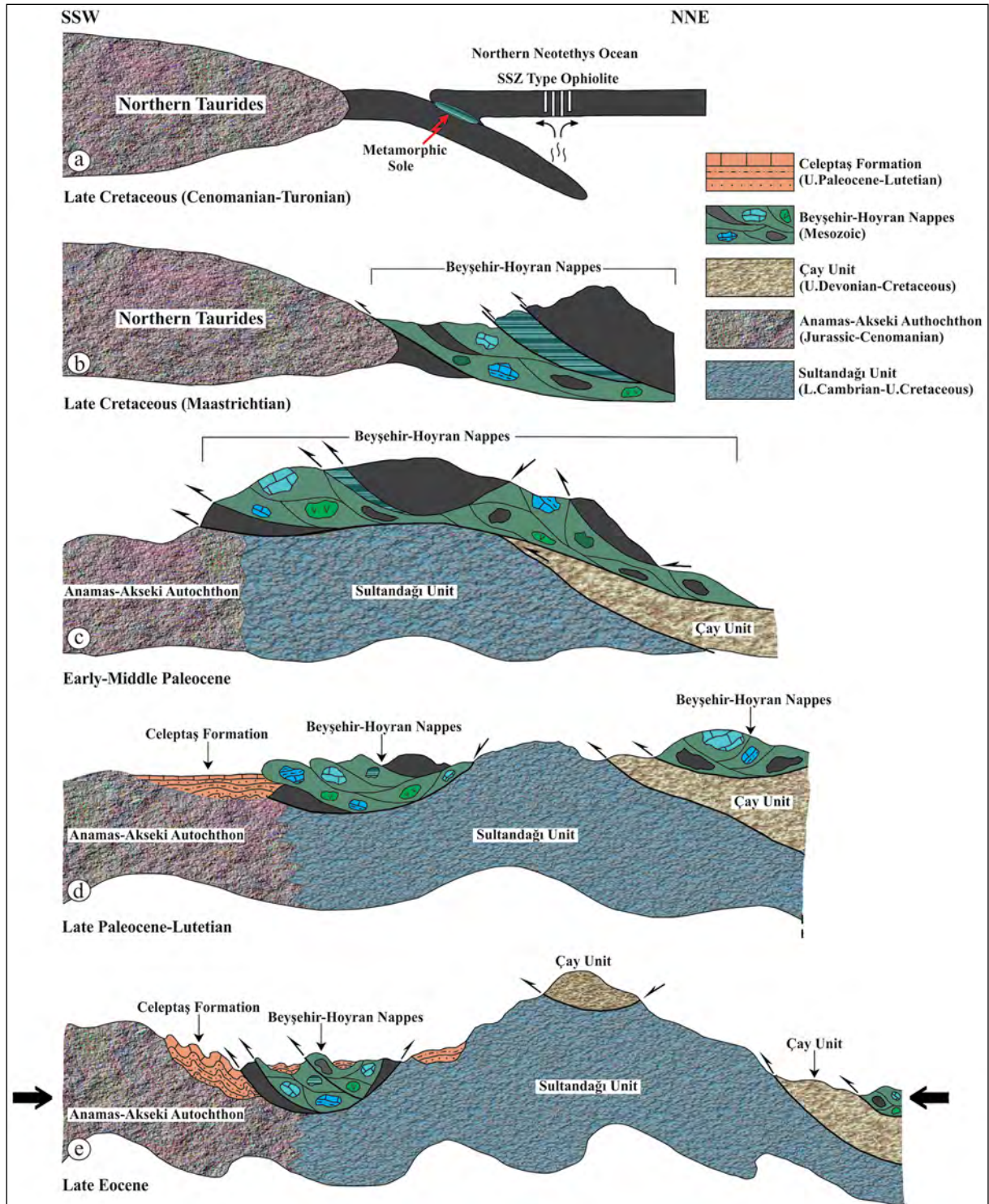


Figure 14- Geological sections illustrating the evolution of the BHN during the Late Cretaceous to late Eocene period.

Cambrian-Late Cretaceous Sultandağı Unit and the Jurassic-Cenomanian Anamas-Akseki Autochthon, while it must be older than the late Paleocene-Lutetian Celeptaş Formation, the common cover of the parautochthonous and allochthonous units in the region, thus pointing to an early-middle Paleocene emplacement age (Figure 14c). The ongoing movements of the BHN and its settlement in the basin caused the syndimentary deformation of the Celeptaş Formation.

Şenel (1991) argued for the presence of a Paleocene-Lutetian common marine basin extending from the Lycian Nappes towards Seydişehir along the Isparta Angle. These data also support the idea that the BHN was not emplaced in the region during late Eocene, contrarily, but already existed there before the late Paleocene.

The stratigraphic studies and geological mapping carried out in the region during this study, as well as the age data obtained from both the BHN and the Celeptaş Formation, helped to reinterpret the Late Cretaceous-Eocene geological evolution of the region. The initiation of the northward subduction of the northern branch of the Neotethys Ocean led to the formation of SSZ-type ophiolites (Marmaris Ophiolite) and consequently to the development of sub-ophiolitic metamorphic sole (Yenicekale Metamorphics) at the Cenomanian-Turonian boundary (Figure 14a). During the Late Cretaceous-early Paleocene, from north to south, the BHN thrust over the Çay Unit and the Çay Unit thrust over the Sultandağı Unit (Figures 14b-c). In the early Paleocene, the BHN crossed the Sultan Dağları and reached the Anamas-Akseki Autochthon (Figure 14c). During this period, the rocks of the Sultandağı and Çay units underwent both deformation and low-grade metamorphism depending on the emplacement of the BHN (Figure 14c). Following this process, the Celeptaş Formation was deposited in the foreland basin, which developed in the south of Sultan Dağları, in the late Paleocene-Lutetian period. Following this process, the Celeptaş Formation was deposited in the foreland basin that developed in the south of Sultan Dağları in the late Paleocene-Lutetian period (Figure 14d).

The Celeptaş Formation was folded due to the tectonic movements, probably occurred in the late Eocene, and subsequently was overthrust in places by units of the BHN. These can be considered as secondary thrusts after the primary emplacement (Figure 14e).

4. Conclusions

The BHN, which extends as a NW-SE belt to the south of Sultan Dağları, consists of allochthonous masses such as the Marmaris Ophiolite Nappe, Gülbahar Nappe and Domuzdağ Nappe. The Marmaris Ophiolite Nappe (Upper Cretaceous) is formed by three subunits as the Marmaris Ophiolite, the Kızılcadağ Mélange and the Yenicekale Metamorphics. The Marmaris Ophiolite is composed of dunite, harzburgite, serpentinite and isolated diabase dykes, while the Kızılcadağ Mélange is composed by blocks of different ages and lithologies contained in an ophiolitic/sedimentary matrix. The Yenicekale Metamorphics are characterized by sub-ophiolitic metamorphic sole rocks such as amphibolites, quartzites and calcschists.

The Gülbahar Nappe is represented by the Middle Triassic-Early Jurassic Orluca Formation, which is composed of deep marine mudstones, cherts, radiolarites, micritic limestone and basic volcanics. The Orluca Formation, which has been identified and mapped for the first time at the south of Sultan Dağları.

In addition, we obtained a ^{40}Ar - ^{39}Ar age of 93.90 ± 0.34 Ma (Cenomanian-Turonian boundary) from the hornblende minerals of amphibolites from the Yenicekale metamorphics. Tectonic processes, which were triggered by and occurred immediately following the formation of the metamorphic sole during the aforementioned time started the formation of the nappes. We therefore accepted that the Turonian time should be the onset of the formation of the BHN.

On the other hand, the late Maastrichtian age, which is obtained from the mudstones in the matrix of the Kızılcadağ mélange, is considered to mark the end of the formation of the BHN.

The evaluation of the stratigraphic, palaeontological and radiometric data together reveals that the formation of the BHN began in the Turonian and

ended in the Late Maastrichtian. The Late Paleocene-Lutetian Celeptaş Formation unconformably overlies the Sultandağı Unit, Anamas-Akseki Autochthon and BHN as common cover in the study area. This clearly indicates that the BHN was emplaced in the region during the early-middle Paleocene. The BHN reached its final position as a result of late Eocene movements.

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Magnetic anomalies and geological signatures in the Black Sea: a comprehensive study of the east and west basins

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Research Article

Keywords:

Curie Point Depth, Heat Flow, Tilt Derivative, Local Wavenumber, Correlation Plot.

ABSTRACT

This study investigates the origin of the Black Sea, focusing on the East and West Black Sea basins. Data such as Curie Point Depth values, heat flow, bathymetry, Moho depth, sediment thickness, and magnetic depth estimation were analyzed to explore the region's geological features. Correlations between these parameters were assessed for each basin, shedding light on their tectonic history and the influence of volcanic rocks. Thinner sedimentary covers enhanced the detectability of rocks with higher magnetic properties, while the interiors showed lower magnetic anomalies due to younger sedimentary rocks. Edge detection techniques, like tilt angle and Local Wavenumber methods, improved magnetic data interpretation. The study computed Curie Point Depth and heat flow using 65 blocks, revealing greater depths in the Western Black Sea (up to 32 km) compared to the Eastern Black Sea (24-28 km). Two profiles (A-A' and B-B') were examined for further insights. Overall, this study provides valuable insights into the geological evolution of the Black Sea basins.

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1. Introduction

To limit the thermal structure and lithospheric rheology, it is significant to assess the spatial discrepancies in temperature across the Earth (Audet and Gosselin, 2019; Sobh et al., 2021). In addition, analysing the variances in magnetic data and the depth of the Moho can provide insight into the tectonic evolution of the region. Calculating the depth of the Curie point, at which crustal rocks reach their Curie temperature [approximately 580° for magnetite (Dunlop et al., 1998)], provides temperature constraints over a region. When rocks are heated above the Curie temperature, they lose their ferromagnetic magnetism and become paramagnetic. Thus, the depth of the magnetised crust can be determined by identifying the

Curie depth, which is the point at which rocks lose their ability to preserve ferromagnetic magnetism (Haggerty, 1978).

Although estimating the Curie depth from magnetic data is significant, uncertainties remain as to whether spectral techniques can produce dependable depth estimates and whether these estimations portray the Curie isotherms or a structural boundary. To clarify this issue, optimum wavenumber ranges for estimating Curie depth was limited by comparing theoretical approximation and linear estimation (Núñez Demarco et al., 2021; Pamuk and Özsöz, 2022). Furthermore, additional constraints such as uncertainties in Curie depth estimation, Moho depth, sediment thickness, and magnetic depth estimation techniques can be

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employed to verify the estimations of Curie depth (Özsöz, 2021).

Numerous researchers have conducted studies on the thermal composition of the Black Sea's crust, specifically within the West and East Black Sea Basins (WBS and EBS). Maden (2013) investigated the EBS and determined that the elevated values of heat flow observed may be attributed to the melting of the lithospheric mantle, triggered by the upwelling of the asthenosphere. Starostenko et al. (2014) noted that the WBS and EBS exhibit a greater depth for the Curie point (CPD) as compared to the Mid-Black Sea Ridge (MBSR) and coastal regions, which are characterised by thinner sediment layers. This finding suggests that areas with thicker sediment deposits correspond to deeper CPD values. In their study, Tütünsatar et al. (2018) presented findings on the fluctuating heat flow values in the Black Sea, ranging from 22 mW/m² to 41 mW/m². They posited that regions with higher heat flow values than the average are indicative of potential deep fault zones and areas where gas migration occurs. Entezar-Saadat et al. (2020) presented a comprehensive lithospheric model of the Black Sea, incorporating thermal structure, sediment thickness, and the depths to the Moho and lithosphere-asthenosphere boundary (LAB). The study utilized a combination of 3D nonlinear inversion of satellite gravity and gravity gradiometry data with 2D iterative forward modelling of potential field data and heat flow measurements. The findings revealed that the average basement depths are 10–11 km in the East Black Sea (EBS) and 11–12 km in the West Black Sea (WBS), with shallower depths of 2–5 km beneath ridges. The mean crustal thickness was determined to be 20 km in the EBS and 21 km in the WBS. Additionally, the surface heat flow was found to be highest on the ridges and lowest over the thick central sedimentary basins. This model offers a detailed and integrated understanding of the Black Sea's lithospheric structure. Bilim et al. (2021) shed light on crustal thickness beneath the Black Sea and surrounding region by gravity data. Their findings indicate that the Shatsky Ridge displays the thinnest crustal thickness at 10 km, while the Sea of Azov boasts the greatest crustal thickness, measuring at 28 km. Akar (2024) conducted a study in the Çameli Basin and its surrounding region in southwestern

Anatolia, Türkiye, utilizing aeromagnetic data and a fractal-based centroid method to estimate the Curie Point Depth, which is essential for understanding regional tectonic and geothermal characteristics. The estimated depths of magnetic sources range between 6.9 and 14.05 km, while the thermal gradient varies from 41.28°C/km to 89.23°C/km, with an average value of 58.59°C/km. The results correspond with NW-SE earthquake distributions and indicate that the Curie isotherm is significantly shallower than the Moho boundary.

The primary objective of this study is to establish a comprehensive theoretical framework elucidating the origin and evolution of the Black Sea, with a specific focus on both the EBS and WBS basins. To achieve this, a wide array of data is employed, encompassing parameters such as CPD (Curie Point Depth) values, heat flow values derived from CPD data, bathymetry, Moho depth, sediment thickness, and magnetic depth estimation techniques (Tilt depth and Local Wavenumber methods). The investigation also examines how the distribution of volcanic rocks in the Black Sea region impacts magnetic anomalies. Furthermore, a meticulous analysis of the correlation between CPD values, bathymetry, Moho depth, sediment thickness, tilt depth, and Local Wavenumber depths is conducted separately for the EBS and WBS basins. By closely examining these correlations and considering the tectonic history and presence of volcanic rocks, the notable discrepancies between the EBS and WBS are thoroughly interpreted. This research aims to shed light on the distinct geological processes and factors that have contributed to the development and divergence of the EBS and WBS basins.

2. Geological Setting

The Black Sea, a large semi-enclosed basin, is surrounded by orogenic belts in the south (Pontides), northeast (Crimea Mountains and Greater Caucasus), and west (Balkanides). Beneath the Neogene to present-day sedimentary succession of the basin fill, representing a unified depocenter, lie two sub-basins, namely the WBS and the EBS, along with a structural high, the Mid Black Sea Ridge (MBSR), comprising Andrusov Ridge and Arkhangelsky Ridge in an

en-echelon arrangement. The WBS and EBS display distinct differences in structural complexity, with the latter featuring a prominent ridge (Shatsky) and a pair of troughs (Tuapse and Sorokin) near its northeastern margin.

The Black Sea Basin is categorized as a back-arc basin, resulting from the northward subduction of the Neo-Tethys Ocean under the southern margin of Laurasia (Letouzey et al., 1977; Zonenshain and Le Pichon, 1986; Finetti et al., 1988). The process of rifting in the extensional back-arc led to the separation of a continental fragment (İstanbul Zone) from the Moesian Platform, giving rise to the asymmetric WBS, where the southern margin exhibits a steeper inclination (Okay et al., 1994; Nikishin et al., 2015). On the other hand, the EBS took shape through the separation and rotation (Okay et al., 1994; Robinson et al., 1996) of the Mid MBSR from the Shatsky Ridge, forming a more symmetrical basin (Shillington et al., 2009). The timing of sub-basin formation is generally believed to be late Cretaceous to early Paleogene, with the EBS considered to be the younger of the two, a subject of ongoing debate (Görür, 1988; Görür et al., 1993; Robinson et al., 1995; Okay and Şahintürk, 1997; Hippolyte et al., 2017; 2018).

The two sub-basins are situated on crusts with distinct thermal and mechanical properties (Spadini et al., 1996). After the initial rifting, the WBS evolved into a proper ocean with an inferred oceanic crust, likely during the late Santonian period, indicated by the age of corresponding pelagic sediments and volcanics in the Pontides region (Tüysüz et al., 2012; Tüysüz, 2018). On the other hand, the EBS has a thinned continental or an oceanic crust beneath it (Shillington et al., 2009), and its opening started in the late Paleocene and was completed in the middle Eocene (Robinson et al., 1995). The sedimentary fill thickness in the WBS ranges from 14 to 18 km, whereas in the EBS, it can reach up to 12 km.

Seismic surveys have revealed the presence of multiple buried volcanoes along the southern offshore margin of the Black Sea basin and on the Shatsky and Andrusov ridges (Nikishin et al., 2015). Due to the absence of sufficient well data, the age of these volcanoes is inferred to be likely Santonian-

Campanian based on the correlation with volcanic units in the Pontides and Crimea regions (Nikishin et al., 2003).

Based on seismic reflection studies (Letouzey et al., 1977; Nikishin et al., 2015), the undisturbed post-rift sedimentary layers within the basin indicate that the crust beneath the Black Sea basin possesses sufficient strength to withstand the tectonic forces responsible for the deformation of its margins since at least the Eocene period (Espurt et al., 2014). GPS measurements (Tari et al., 2000) and seismic activity (Neprochnov and Ross, 1978) indicate an active shortening on the EBS. Figure 1 illustrates simplified tectonic map of the study area.

3. Data

The magnetic data used in this study was obtained from the EMAG 2 model. The EMAG 2 model presents a detailed description of the Earth's magnetic field on a global scale. It was calculated using satellite magnetic data as well as land and sea measurements. The EMAG 2 model can identify magnetic anomalies larger than 1 nT with a coverage of 2 arc-minutes and a spatial resolution of approximately 4 km. (Maus et al., 2009).

A robust inversion approach that has been widely validated in multiple regional studies is used to extract the first global model of Curie-point depth (GCDM) from magnetic anomaly inversion based on fractal magnetisation (Li et al., 2017). According to the statistical analysis, the mean depth of the Curie point in the marine zone is lower than in the continental region. The continental Curie depths, on the other hand, have a virtually bimodal distribution, with some old cratons having shallow Curie points. Hydrothermal circulations in young oceanic lithosphere and thermal disturbances in old oceanic lithosphere affect oceanic Curie depths. Furthermore, the oceanic Curie depths are heavily dependent on the spreading rate along active spreading centres (Li et al., 2017). In this research, the estimated Curie-depth points were compared and validated with the global model of Curie-point depth within the same geographical region.

The bathymetry data used in this study, which focused on the Black Sea region, was obtained

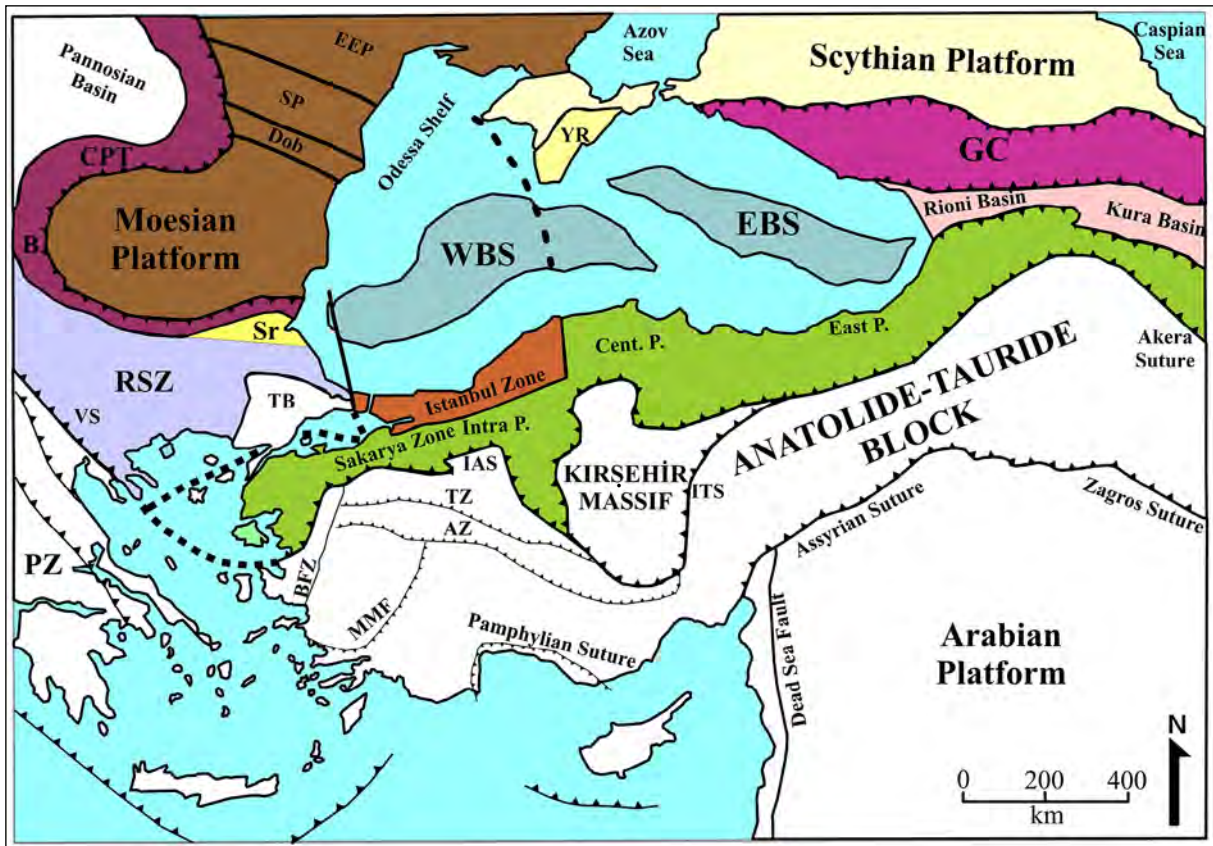


Figure 1- Simplified tectonic map of the Black Sea and its surrounding regions (modified after Şengör, 1984; Okay, 1989; Okay and Tüysüz, 1999; Özsöz and Tokar, 2022). PZ= Pelagonian Zone, VS= Vardar Suture, RSZ= Rhodope-Strandja Zone, B= Balkanides, CPT= Carpathians, Dob= Dobruđa, SP= Scythian Platform, EEP= East European Platform, YR= Yayla Range, WBS=West Black Sea Basin, EBS= East Black Sea Basin, GC= Greater Caucasus, Intra P.= Intra Pontide Suture, Cent. P.= Central Pontides, East P.= Eastern Pontides, IAS= İzmir - Ankara Suture, TZ= Tavşanlı Zone, AZ= Afyon Zone, MMF= Menderes Massif, BFZ= Bornove Flysch Zone, SR= Srednogorie).

from the ETOPO 1 model. ETOPO 1 model includes combination of land topography and ocean bathymetry into a global grid. The model was developed by The National Oceanic and Atmospheric Administration (NOAA). ETOPO1 grid cell spans an area of around 1.8 km² at a resolution of one arc-minute at the equator. The model is constructed with an integration of satellite and marine observations, as well as data from other sources. ETOPO1 is widely used in oceanography, geology, and climate research because it provides a precise depiction of the Earth's topography, including mountain ranges, valleys, and oceanic trenches (Amante and Eakins, 2009).

Moho depth and sediment thickness data were obtained from CRUST1.0 model. CRUST1.0, 1-by-1° global crustal model, which forms the initial framework for the comprehensive database to compile

a worldwide model of the Earth's crust and lithosphere, LITHO1.0 (Pasyanos et al., 2013). The Moho depth and sediment thickness featured in CRUST1.0 is based on 1-degree averages obtained from a recently updated database of crustal thickness data gathered from active-source seismic studies, as well as from receiver function studies (Laske et al., 2013).

4. Methods

4.1. CPD Estimation

The Curie temperature is the fundamental physical phenomenon that controls the exploration of magnetic depth method. This method's ability to detect magnetic bodies is restricted by the depth of the Curie isotherm. It is feasible to utilize magnetic data to deduce heat flow since the magnetic characteristics of rocks are dependent by temperature,

and rocks lose their magnetic properties at the Curie temperature. Nonetheless, this methodology is subject to certain limitations due to its dependence on several assumptions. The technique requires that the magnetic sources' depth be significantly greater than their height, which is vital for spectral separation (Bouguern et al., 2015).

The estimation of CPDs has been accomplished in numerous locations across the globe through the application of magnetic data. Two primary techniques have been employed to calculate CPDs: the spectral peak and the centroid depths. The spectral peak method was initially introduced by Spector and Grant (1970), while the centroid approach was first presented by Bhattacharyya and Leu (1977) and was later improved by Okubo et al. (1985). In this study, the latter approach was used.

Our assumption is that the lithosphere's magnetised layer is limited by two surfaces situated at depths Z_t (the top of the magnetised layer) and Z_b (the bottom of the magnetised layer). It should be noted that if $Z_t = 0$, this would imply that the magnetised layer's top coincides with the measurement plane (Sobh et al., 2021). The thickness of the magnetised layer is represented by ΔZ , which is equal to Z_b minus Z_t (Figure 2).

Rozimant et al. (2009) identified a correlation between heat flow and Curie Point Depth (CPD) values but noted that this relationship does not consistently align with crustal depth. They also emphasized that the Curie point isotherm marks the boundary between the magnetic and non-magnetic regions of the crust.

Spector and Grant (1970) first introduced the method of radially averaging power spectra, which provides information about the depth of the magnetic layer. Subsequently, this method has been improved upon by Bhattacharyya and Leu (1975), Connard et al. (1983), Okubo et al. (1985), Tanaka et al. (1999), Ross et al. (2006). These advancements enable the thermal structure of an area to be determined using magnetic anomaly values. In simple terms, the radially averaged spectrum can be defined as:

$$P(|k|) = Ae^{-2|k|Z_t}(1 - e^{-|k|(Z_b - Z_t)})^2 \quad (1)$$

where A is constant, wavenumber represented by k , and depth measurements for the top and bottom of a magnetic source, denoted as Z_b and Z_t . The next step in the equation 1 involves taking the natural logarithm of both sides.

$$\ln[P(|k|)] = \ln[A] - 2Z_t(k) + 2\ln[1 - e^{-|k|(Z_b - Z_t)}] \quad (2)$$

When the wavenumber components are of high or medium values, the exponential factor in equation

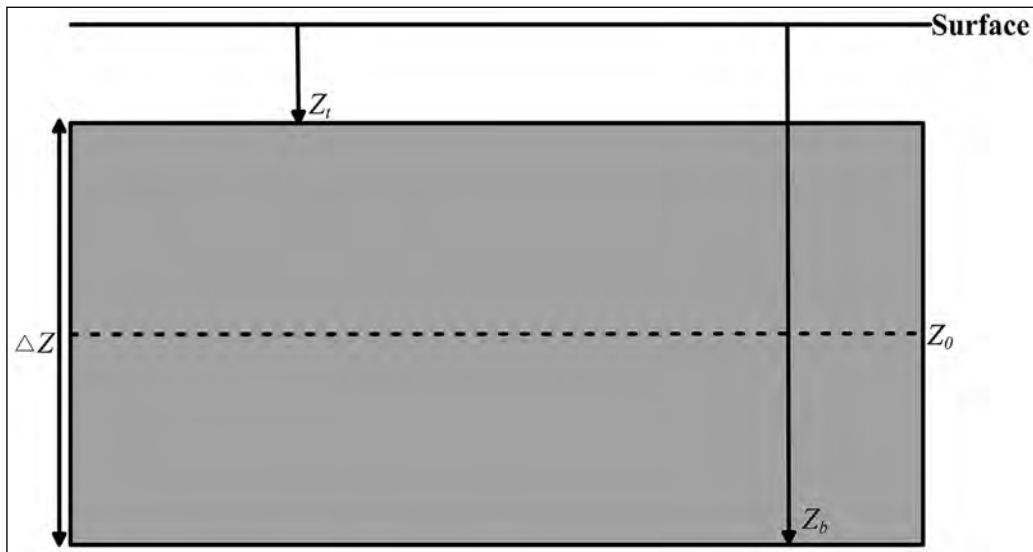


Figure 2- Schematic diagram of magnetic depths where Z_t = top of the magnetised layer, Z_b = the bottom of the magnetised layer, Z_0 = centroid depth that corresponds to $\frac{Z_b + Z_t}{2}$ and ΔZ = thickness of the magnetised layer.

2 becomes insignificant and can be disregarded. As a result, equation 2 can be expressed differently as shown in the following rewritten form:

$$\ln[P(|k|)] = \ln[A] - |k|2Z_t \quad (3)$$

Equation 3 indicates that the slope is equal to $2Z_t$. By dividing equation 3 by 2, a linear estimation model can be derived:

$$\ln[P(|k|)^{1/2}] = C - |k|Z_t \quad (4)$$

The equation 4 involves a constant value, denoted as C . The slope of equation 4 is indicative of the depth measurement to the top of the magnetic layer, represented as Z_t . To obtain an estimation for Z_0 , one can multiply equation 1 by $e^{-|k|(z_0-z_0)}$, and then divide the resulting equation by 2:

$$\ln[P(|k|)^{1/2}] = Ae^{-|k|Z_0}(e^{-|k|(Z_t-Z_0)} - e^{-|k|(Z_b-Z_0)}) \quad (5)$$

The depth to the centroid of the magnetic source is denoted by Z_0 . If we substitute the first terms of the Taylor series expansion for the exponential term in equation 5, an approximate equation for Z_0 can be obtained.

$$\ln[P(|k|)^{1/2}] \approx Ae^{-|k|Z_0}(Z_b - Z_t) \quad (6)$$

The thickness of the magnetic crust (ΔZ) is represented by $Z_b - Z_t$, as defined by Gasparini et al. (1979). By substituting and rearranging the thickness of the magnetic crust with Z_0 , we obtain:

$$\ln\left[\frac{P(|k|)^{1/2}}{|k|}\right] = \ln[C_2] - |k|Z_0 \quad (7)$$

Equation 7 involves a constant value for C_2 , while Z_0 is determined by calculating the slope for the low wavenumber components. The depth of the magnetic layer's centroid is related to the magnetic layer's bottom, as indicated by Okubo et al., (1985) and Tanaka et al., (1999):

$$Z_b = 2Z_0 - Z_t \quad (8)$$

Overall, equation 4 and 7 represent straight-line equations, and ultimately, the estimation of Z_t and Z_0 can be achieved by determining the slope of the linear fit. Once Z_t and Z_0 are estimated, it is possible to articulate the uncertainty (ΔZ_t and ΔZ_0) associated with these estimations in the following manner (Martos et al., 2017; Carrillo-de la Cruz et al., 2020):

$$\Delta Z_t = (RMS_{Z_t}/2\pi) \times [\max(k_{s_{Z_t}}) - \min(k_{s_{Z_t}})] \quad (9)$$

$$\Delta Z_0 = (RMS_{Z_0}/2\pi) \times [\max(k_{s_{Z_0}}) - \min(k_{s_{Z_0}})] \quad (10)$$

The expressions RMS_{Z_t} and RMS_{Z_0} refer to the Root Mean Square (RMS) error of the linear regression model divided by the number of data points used in the analysis. On the other hand, $k_{s_{Z_t}}$ and $k_{s_{Z_0}}$ represent the wavenumber range of the linear segment chosen to estimate Z_t and Z_0 , respectively. Ultimately, the uncertainty in the estimated value of Z_b denoted as ΔZ_b can be expressed as per the definition proposed by Martos et al. (2019):

$$\Delta Z_b = \sqrt{2\Delta Z_0^2 - \Delta Z_t^2} \quad (11)$$

4.1.1. Wavenumber Ranges for Curie Depth Estimations

To ensure the accuracy of the calculated Z_b and Z_t values, it is essential to validate them by comparing with both theoretical approximations and linear estimations. However, many authors tend to overlook this step and manually select the wavenumber ranges. In a recent study, Núñez Demarco et al. (2021) analyzed 72 articles and recommended that the mathematical validity of the computation can be evaluated by comparing the results of equations (2) and (3) for Z_t and equations (5) and (6) for Z_b . The comparison can be done by examining the difference in the slope between the linear estimation model and the theoretical curve.

$$\Delta m = \left[\frac{t(k_{i+1}) - t(k_i)}{k_{i+1} - k_i} - \frac{l(k_{i+1}) - l(k_i)}{k_{i+1} - k_i} \right] \quad (12)$$

The symbol Δm denotes the difference in slope, where $t(k)$ represents the theoretical curve and $l(k)$ denotes the linear estimation. When Δm approaches 0, it implies that the estimated values of Z_0 and Z_t are reliable. Specifically, wavenumber ranges that exhibit a difference of less than 5% between the linear approximation and the theoretical curve can be considered as valid regions for estimating Z_0 and Z_t . Generally, the valid regions for Z_t are larger than those for Z_0 . As the thickness of the magnetic layer (ΔZ) increases, the valid regions tend to be wider for Z_t and narrower for Z_0 . Additionally, when Z_t is deeper for a constant ΔZ , the confidence region becomes broader.

Figure 3 shows that the regions of confidence are estimated for the expected maximum, minimum, and average values of magnetic thickness (ΔZ). In order to achieve the expected maximum value of ΔZ (34 km),

the maximum expected value of Z_b is taken as 36 km, while the minimum expected value of Z_t is taken as 2 km. Similarly, to obtain the expected average value of ΔZ (23 km), the expected average values of Z_b and

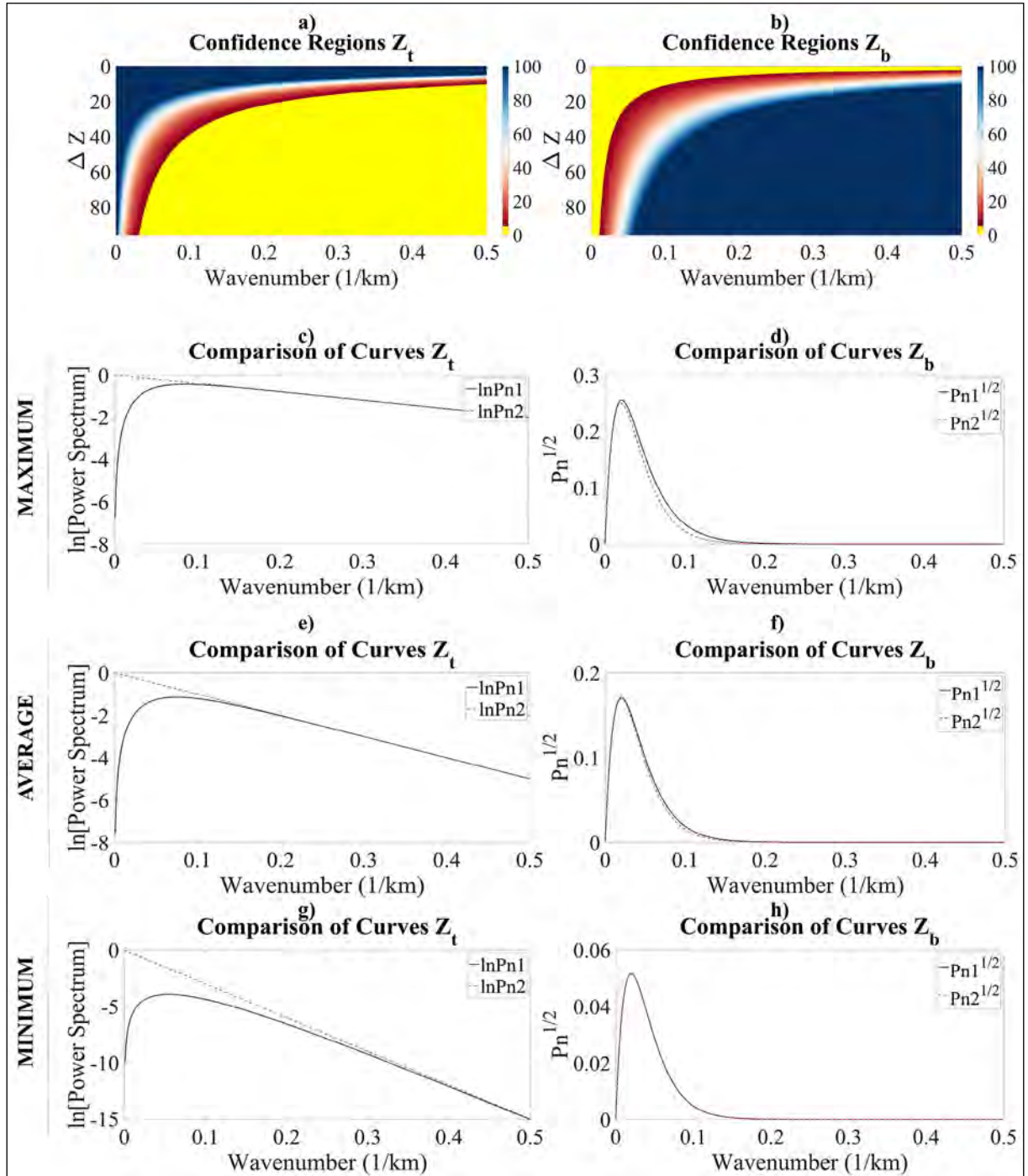


Figure 3- The confidence regions for CPD estimations are presented in the following manner: panels a) and b) display reliable ranges of wavenumbers, represented by the yellow area, where the slope difference (Δm) is less than 5%. Panels c) and d) illustrate the wavenumber ranges associated with the maximum value of ΔZ (34 km), where $Z_b = 36$ km, $Z_t = 2$ km. Panels g) and h) show the wavenumber ranges corresponding to the average value of ΔZ (23 km), where $Z_b = 28$ km, $Z_t = 5$ km. Panels e) and f) depict the wavenumber ranges for the minimum value of ΔZ (7 km), where $Z_b = 22$ km, $Z_t = 15$ km.

Z_t are assumed as 28 km and 5 km, respectively. To achieve the expected minimum value of ΔZ (7 km), the expected minimum values of Z_b and Z_t are taken as 22 km and 15 km, respectively. The assumptions for the expected values of Z_t and Z_b are based on previous studies conducted by Starostenko et al. (2014), Li et al. (2017), Tütünsatar et al. (2018).

4.1.2. Heat Flow Estimations from CPD Results

The estimation of heat flow and thermal gradient is carried out utilizing Fourier's Law (Fourier, 1878). As CPD is connected with magnetite's 580 °C temperature, heat flow can be determined based on the estimated Z_b values. The heat flow can be given as:

$$q(z) = \lambda \frac{dT(z)}{dz} \quad (13)$$

where $q(z)$ is heat flow as a function of depth (z), λ is the thermal conductivity with a value of 2.2 W/mK and $\frac{dT}{dz}$ is the geothermal gradient. To solve equation 13 for conductive heat transfer, which is outlined in (Martos et al., 2017), it is necessary to determine the boundary limits. Thus, the equation can be rearranged and expressed as:

$$q(z) = \lambda \frac{(T_c - T_0)}{Z_b} \quad (14)$$

Equation 10 considers T_c as the Curie temperature of magnetite (approximately 580°C), while T_0 represents the surface temperature, which has a value of 20°C. Notably, the equation 14 does not account for radiogenic heat production, mass advection, temperature dependence of thermal conductivity, and transient cooling, as described in (Ravat et al., 2016).

4.2. Edge Detection and Depth Estimation Methods

4.2.1. Tilt Depth Method

The magnetic tilt angle is a mathematical expression that is normalized by comparing the vertical and horizontal derivatives of the reduction to the pole (RTP) field. This approach is called the "tilt depth method" and enables a more intuitive comprehension of the changes in depth of magnetic source bodies (Salem et al., 2007). Tilt angle is initially introduced by Miller and Singh (1994). The most basic version of the tilt depth method assumes that the source formations have perpendicular interfaces, no remnant magnetism,

and that the magnetisation is oriented vertically. Then the method was subsequently refined by Verduzco et al. (2004), and subsequently defined as:

$$\theta = \arctan \left[\frac{\frac{\partial M}{\partial z}}{\frac{\partial M}{\partial H}} \right] \quad \text{where} \quad \frac{\partial M}{\partial H} = \sqrt{\left(\frac{\partial M}{\partial x} \right)^2 + \left(\frac{\partial M}{\partial y} \right)^2} \quad (15)$$

where $\partial M/\partial x$, $\partial M/\partial y$, and $\partial M/\partial z$ represent the first order differentials of the magnetic field with respect to the x, y, and z axes, correspondingly. Additionally, $\frac{\partial M}{\partial H}$ is total horizontal derivative of the magnetic field M. One can observe several interesting attributes of the tilt angle (θ). Specifically, due to the nature of the "tan⁻¹" trigonometric function, the magnitude of all tilts falls within the range of -90° to +90°, independent of the magnitude of the vertical or absolute value of the overall horizontal gradient. In addition to equalizing the amplitude of magnetic anomalies across a grid or profile, this approach preserves the spectral consistency of the signal, thereby enabling additional quantitative analysis such as the determination of local wavenumber (Salem et al., 2007).

Tilt angle over the edges of the contact is 0° ($h=0$), while it reaches 45° when h equals Z_{tilt} and -45° when h is $-Z_{tilt}$. As a result, it is plausible to use contours of the magnetic tilt angle to determine both the location ($\theta=0^\circ$) and top depth (half the physical distance between $\pm 45^\circ$ contours) of contact-like formations (Doğru et al., 2017). This phenomenon quantitatively identified as (Salem et al., 2007):

$$\theta = \arctan \left(\frac{h}{Z_{tilt}} \right) \quad (16)$$

where h is horizontal distance and Z_{tilt} refers to the depth of the top of contact-like structures that have been estimated through the use of the tilt-depth method.

4.2.2. Local Wavenumber Method

The method of determining the local wavenumber requires the calculation of both first and second derivatives in both horizontal and vertical directions. It is the most vulnerable to disturbances from noise and interference. However, if such disturbances are absent, this method can provide precise measurements of the horizontal and vertical positions of individual sources,

as well as the sources' structural characteristics (Phillips, 2000). According to Thurston and Smith (1997) work, they provide a formula for determining the local wavenumber along a 2-dimensional source by measuring a profile. Subsequently, Phillips (2000) derived an equivalent formula for the local wavenumber (LW) of the anomalous magnetic field in a gridded format:

$$LW = \frac{1}{\left(\frac{\partial M}{\partial x}\right)^2 + \left(\frac{\partial M}{\partial y}\right)^2 + \left(\frac{\partial M}{\partial z}\right)^2} \left[\frac{\partial M}{\partial x} \frac{\partial^2 M}{\partial z \partial x} + \frac{\partial M}{\partial y} \frac{\partial^2 M}{\partial z \partial y} + \frac{\partial M}{\partial z} \frac{\partial^2 M}{\partial z \partial z} \right] \quad (17)$$

The LW technique, which employs the second-order derivatives, is susceptible to noise and aliasing in the data, resulting in frequent occurrence of discontinuous contacts and inadequate management of strike direction.

Depth estimations can be conducted by LW with appropriate structural index (N). The numerical value assigned to the structural index differs depending on the geological feature being analysed. A value of zero is assigned to the uppermost portion of a contact or

fault that extends to a significant depth, while a value of one corresponds to the edge of a thin sheet. An extended pipe or cylinder is assigned a value of two, while the centroid of a sphere or dipole is assigned a value of three. The local wavenumber method can accurately determine depths for straightforward sources. However, it fails for thick dikes because of the interfering effects of the two corners, as do all other methods (Phillips, 2000). Depth is estimated using equation 18.

$$\text{At peak} \rightarrow Z_{LW} = \frac{(N + 1)}{LW} \quad (18)$$

where N is structural index which is taken as 0 for this study and Z_{LW} is estimated depth from local peak of LW grid.

5. Results and Discussion

The magnetic data obtained from the EMAG2 model was subjected to the RTP technique, employing a predetermined declination of $6.87^\circ \pm 0.35^\circ$ and an inclination of $61.43^\circ \pm 0.21^\circ$. In Figure 4, the RTP

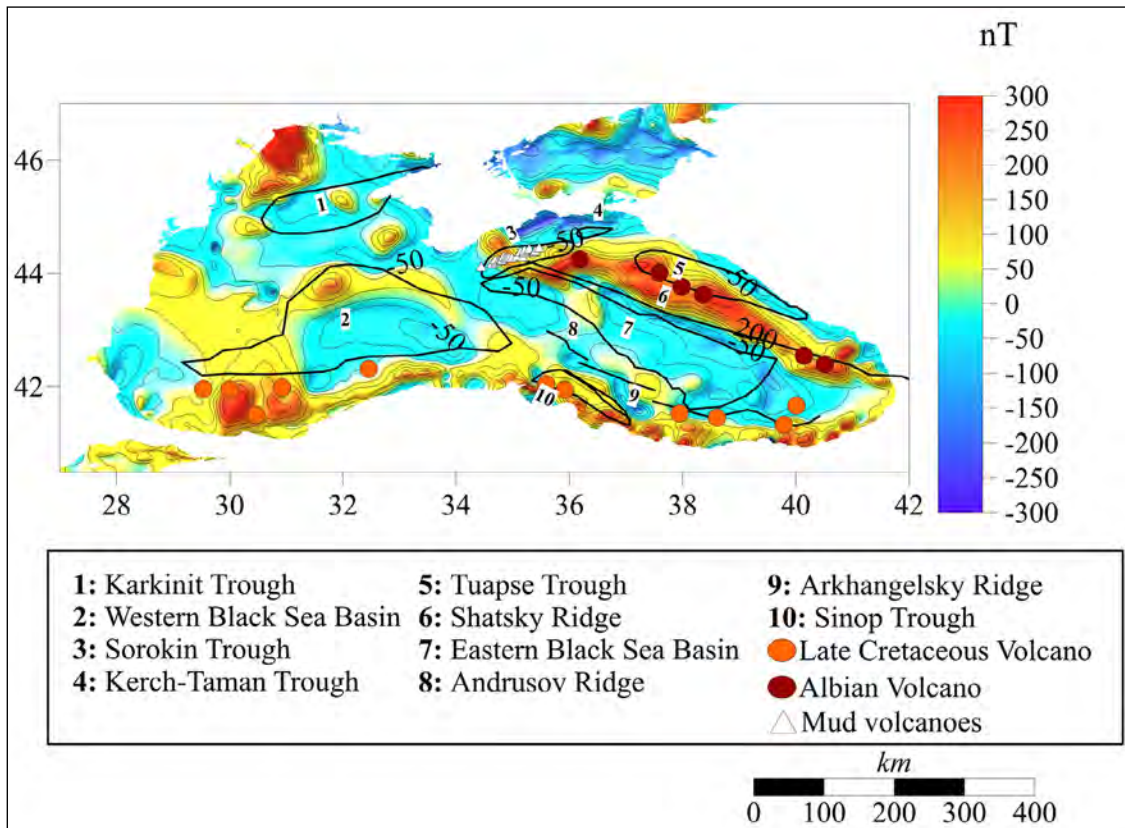


Figure 4- RTP magnetic anomaly with tectonic features in the study area.

magnetic anomaly is depicted, illustrating discernible tectonic features that contribute to the overall magnetic profile.

In each map, the locations of Cretaceous Volcanoes are derived from Nikishin et al. (2015) and Tari and Simmons (2018), while the locations of Albian Volcanoes are sourced from Nikishin et al. (2015). Additionally, the locations of mud volcanoes are digitized based on the data presented in Wagner-Friedrichs et al. (2008).

The range of RTP magnetic anomalies spans from -300 nT to 300 nT, with the most prominent magnetic anomalies localized along the Shatsky Ridge and basin boundaries. Conversely, the EBS and WBS exhibit relatively lower magnetic anomalies. The higher magnetic anomalies observed in the Shatsky Ridge and basin boundaries can be attributed to the prevalence of older magnetic-rich rocks. These regions are predominantly composed of rocks characterized by a high abundance of magnetic minerals, including magnetite, which contribute to the manifestation of larger magnetic anomalies. Additionally, the relatively thinner sedimentary layer in these areas facilitates the detection of the underlying magnetic rocks.

In contrast, the EBS and WBS present lower magnetic anomalies primarily due to their composition of younger sedimentary rocks with reduced magnetic susceptibility compared to the older rocks found in the basin boundaries and the Shatsky Ridge. Furthermore, the presence of a thick sedimentary layer covering the EBS and WBS masks the magnetic basement rocks, leading to a diminished magnetic anomaly.

The Shatsky Swell consists of the Gudanta and Ochamchiri domes, which are aligned with the structural features of the Dzirula Massif in Georgia and are composed of Cretaceous and Jurassic rock sequences. A significant positive magnetic anomaly of approximately 300 nT is depicted in Figure 4, possibly attributable to the presence of volcanic rocks. This observation implies the potential extension of Middle Jurassic volcanics, particularly Bajocian volcanics originating from the Dzirula Massif and the Gagro-Chjawa zone of the southern slope of the Greater Caucasus, into the Shatsky Swell. The Shatsky Swell

is viewed as a connecting region between the areas featuring Jurassic volcanics in the Caucasus and the Crimea Highland. In addition, the analysis of magnetic data reveals a correlation between the magnetic anomalies observed in the Pontides region and the occurrence of Cretaceous-Eocene volcanic rocks, which are extensively documented along the entire coastline. This distinctive feature is characterized by prominently positive magnetic anomalies in the northern part of Türkiye's shoreline.

Edge detection techniques, namely the tilt angle and local wavenumber (LW), were utilized to enhance the interpretation of the magnetic data, as illustrated in Figure 5. The adoption of the tilt angle method was deemed appropriate due to its effectiveness in improving the interpretation of the signal field, which spans a range of ± 1.57 radians. This particular technique proves valuable in accentuating weaker anomalies while equalizing stronger ones, enabling the identification of subtle magnetic anomalies. It is important to note that the sign of the tilt angle is determined by the vertical derivative, as the total horizontal derivative consistently maintains a positive value. Consequently, a direct comparison between the vertical derivative and the total horizontal derivative can be made, further aiding the interpretation process.

The LW technique is used for a variety of reasons. The first reason is that because of it uses second derivatives of the magnetic potential and its capacity to highlight magnetic anomalies with short wavelengths, it is a valuable tool in the analysis of shallower tectonic structures. Secondly, LW map presents higher resolution over the contacts especially narrower structures such as dykes. Finally, the depth of the contact can be estimated by inverse of the local maxima values of the LW (Fairhead, 2016).

The tilt angle map presented in Figure 5a reveals negative readings within the basin and positive values over the ridges and basin boundary. The sign of the tilt angle is influenced by the distribution and orientation of magnetic rocks. Horizontally stratified magnetic rocks within the EBS and WBS contribute to elevated values in the horizontal magnetic field and reduced values in the vertical derivative. Additionally, the presence of younger sedimentary rocks in the basin,

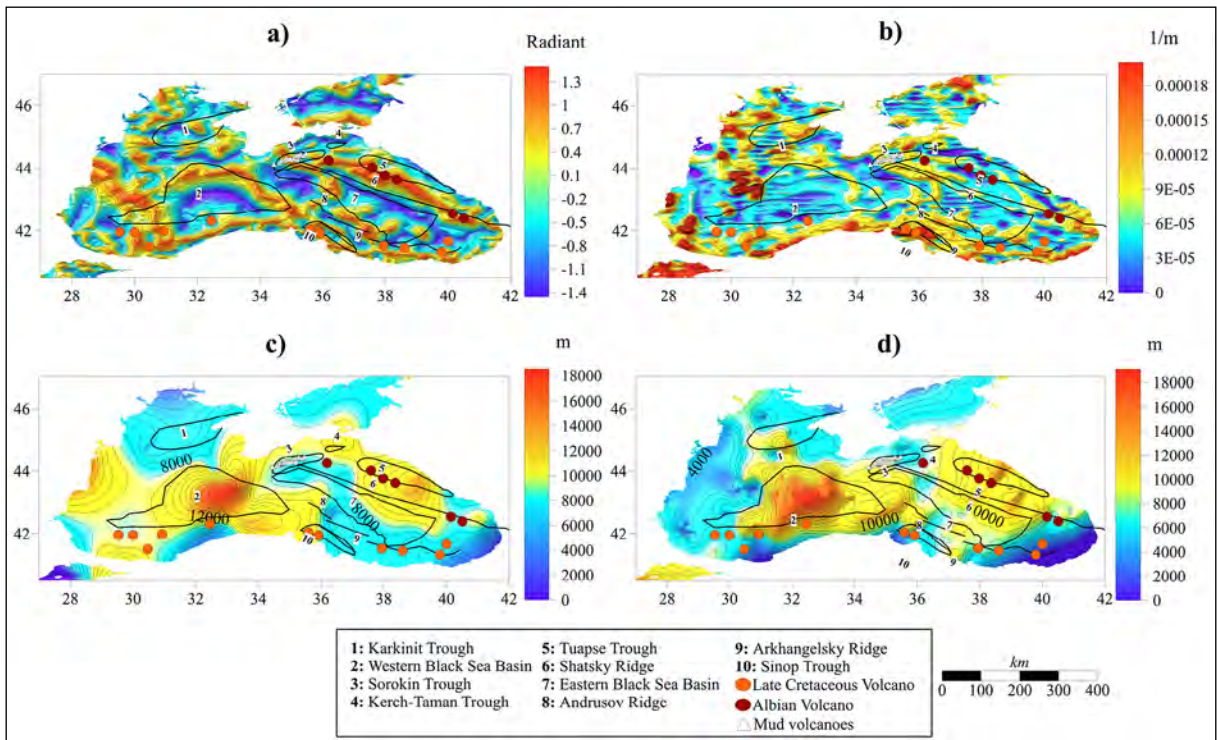


Figure 5- Results of the edge detection methods with tectonic features in the study area: a) Tilt angle map, b) LW map, c) tilt depth, d) LW depth.

exhibiting a negative magnetic susceptibility, results in notable negative anomalies within the tilt angle map. In contrast, ridges often consist of vertically-oriented magnetic rocks, which yield higher values in the vertical derivative and positive tilt angle values. These rocks exhibit a positive magnetic susceptibility. Thus, the determination of the tilt angle map's sign hinges upon the arrangement and quantity of magnetic rocks. At the interface between basement and sedimentary rocks, a sudden shift in magnetic characteristics occurs, leading to an immediate change in the magnetic field and a steep gradient along the edges. Nonetheless, this gradient approaches zero at two neighboring points along the contact boundary. Consequently, tilt angle values either become zero or converge to zero at contact points between distinct rock units.

The LW map, obtained by computing the first and second derivatives of the magnetic field, exhibits local maxima at the contacts between distinct rock types, as demonstrated in Figure 5b. These contacts represent locations where substantial variations in magnetic properties occur, resulting in prominent gradients

that can be detected through the first and second derivatives of the magnetic field. The first derivatives, both in the horizontal and vertical directions, enable the detection of the magnetic field's rate of change, while the second derivatives, also in the horizontal and vertical directions, are particularly responsive to abrupt changes within the LW map. Thus, the presence of local maxima within the LW map serves as a reliable indicator of the existence of notable magnetic gradients.

Figure 5c and 5d provide visual representations of the estimated depth of the uppermost portion of the contact, as inferred from the analysis of the tilt angle and LW maps. Notably, an overestimation of depth by approximately 18 km is observed in the WBS when compared to the EBS. This disparity can be attributed to a combination of factors, including tectonic structures, magmatic content, and sediment thickness.

One factor contributing to the exaggerated depth estimations in the WBS, as opposed to the EBS, is the presence of the Shatsky and Andrusov Ridges. Surrounding the EBS, these ridges generate shorter-

wavelength anomalies, resulting in shallower depth estimations derived from both the tilt depth and LW depth analyses.

Another influential factor is the magma content of the EBS and WBS. The WBS is characterized as magma-poor, while the EBS is known for its magma-rich nature (Kaymakçı et al., 2015). This discrepancy in magma composition directly affects the magnetic properties of the rocks and sediments within these basins, consequently influencing the resulting depth estimations.

Furthermore, sediment thickness plays a crucial role in the wavelength content of the tilt and LW maps. Typically, the WBS exhibits a higher sediment thickness compared to the EBS, leading to the observed overestimation of depth within the WBS.

In addition to employing edge detection methods, such as tilt and LW, the study area involved the calculation of Curie point depth (CPD) and heat flow. The CPD calculations were conducted by dividing

the study area into 65 blocks measuring 200×200 km each, with a 50% overlap between adjacent blocks. The size of these blocks played a crucial role in determining the vertical resolution of Z_b . Following the recommendation of Salem et al. (2014), a block size six times larger than the desired depth was utilized. In this particular study, a block size of 200×200 km resulted in an approximate vertical resolution of 33 km. The CPD values obtained were subsequently employed to infer the heat flow, disregarding the contribution of radiogenic heat production. Consequently, an inverse correlation is observed between the distribution of CPD and heat flow. The outcomes of the CPD calculations, top depth of the magnetic source derived from spectral analysis, and heat flow assessments are visually presented in Figure 6.

Figure 6a provides data indicating a greater Curie point depth (CPD) in the WBS as opposed to the EBS. Furthermore, the CPD values in the EBS exhibit abrupt changes, while the WBS demonstrates a more consistent pattern. These variations can be attributed

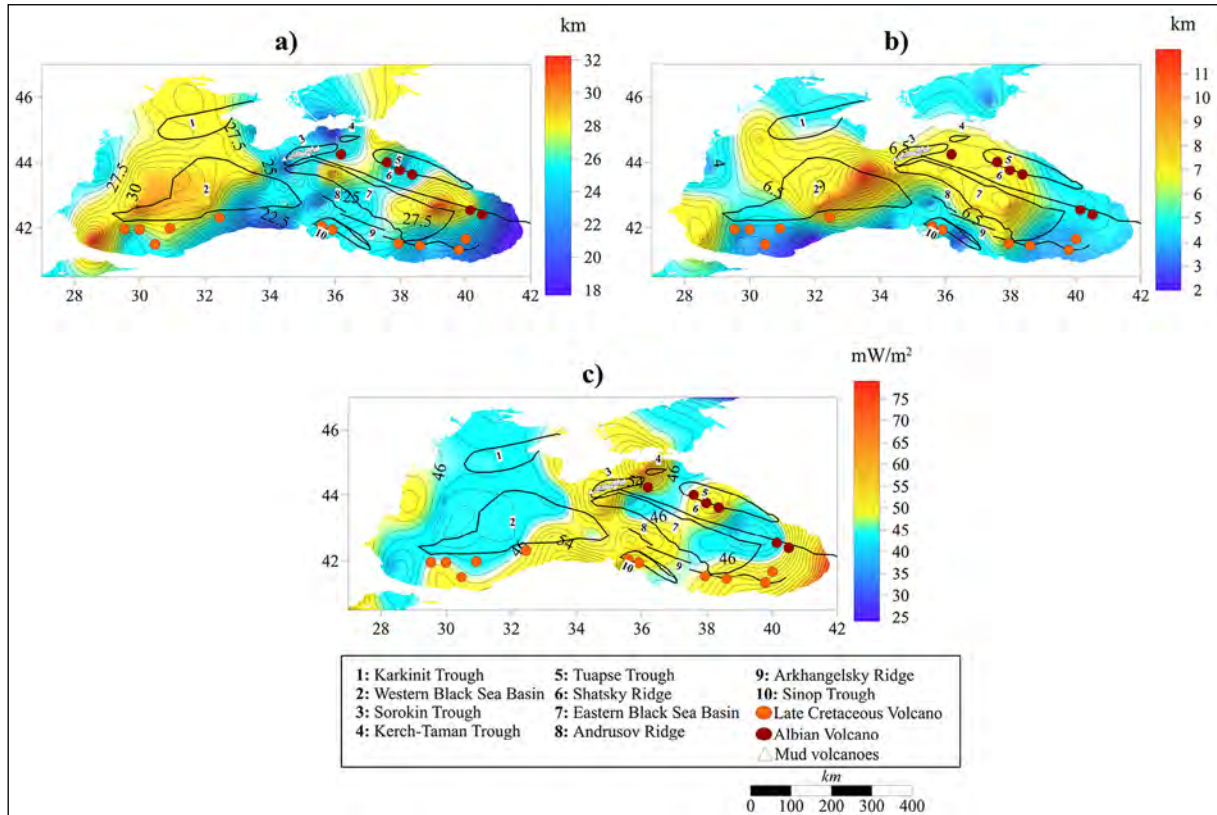


Figure 6- Results of the CPD, Z_t and heat flow with tectonic features in the study area: a) CPD map, b) Z_t map, c) heat flow map.

to various factors, including sediment thickness, the presence of volcanic rocks, and the tectonic history of the EBS and WBS.

To begin with, the thermal insulation provided by sediment thickness hinders heat transfer from the underlying mantle, resulting in deeper CPD values for both the EBS and WBS. Additionally, the WBS exhibits even deeper CPD readings in comparison to the EBS due to its thicker sediment cover.

Moreover, the presence of volcanic rocks enhances the amplitude of higher wavenumber sections, leading to shallower CPD values in the EBS. Furthermore, the abundance of volcanic rocks within the Shatsky Ridge, situated in the northeastern part of the Black Sea, influences the computation of CPD estimates in the EBS. The spectral characteristics of the short-wavelength magnetic anomaly originating from the Shatsky Ridge impact the CPD calculations in the adjacent blocks of the EBS, resulting in underestimated CPD values within the EBS.

Lastly, it is important to note the active tectonic processes occurring in the EBS, while the WBS exhibits a relatively more stable condition. The tectonic instability around the margins of EBS can trigger the upwelling of hot mantle materials. This explanation accounts for the shallower CPD values and higher heat flow observed in the EBS in comparison to the WBS. Letouzey et al. (1977) suggested that the higher heat flow values observed to the south of Crimea and west of the Caucasus might be associated with mud diapirism. Mud volcanoes are known to play a significant role in heat transfer, particularly in geothermal areas.

The Z_t map in Figure 6b provides insights into the top depth of the magnetic basement. Nevertheless, it is important to note that Z_t estimations are more subjective compared to the tilt and LW methods. Therefore, the Z_t estimations were solely utilized in correlation plots to facilitate a comparison of results with sediment thickness, LW maps, and tilt maps.

To facilitate additional investigation, two profiles were designated in the EBS and the WBS as illustrated in Figure 7. Profile A-A' was selected along the WBS, while profile B-B' was chosen along the EBS. These profiles were assessed to evaluate various parameters,

including global CPD (Li et al., 2017), Moho depth (Laske et al., 2013), estimated CPD values (Z_b values), LW depth, tilt depth, sediment thickness (Laske et al., 2013), Z_t values, and bathymetry data (Amante and Eakins, 2009).

The correlation plot depicted in Figure 8 was thoroughly examined to assess the relationships between different parameters along the A-A' and B-B' profiles. Based on the analysis, the following expected relationships between the parameters can be outlined:

- Moho depth and bathymetry are projected to exhibit an inverse correlation. This can be attributed to the requirement for the lithospheric load to be balanced or compensated by adjustments at the Moho interface, resulting in a reciprocal relationship between bathymetry and Moho depth.
- A positive correlation is anticipated between the estimated CPD and global CPD.
- A positive correlation is expected among LW depth, tilt depth, and Z_t values. These parameters provide valuable insights into the uppermost depth of the magnetic basement. Consequently, it is anticipated that at least two of these parameters will exhibit a positive correlation.
- The positive correlation between sediment thickness, LW depth, tilt depth, and Z_t values suggests a potentially rapid sediment accumulation process. The LW depth, tilt depth, and Z_t values are indicators of the top depth of the magnetic basement. If an increase in sediment thickness coincides with an increase in the top depth of the magnetic basement, it can be inferred that sedimentation accumulation rate remains unaffected by deformation, folding, seismic activity or variations in riverbed conditions.

The correlation plots shown in Figure 8 provide a quick and simultaneous comparison of various physical and geometric parameters. These correlation plots are essentially lower triangular matrices, where the comparison of the same geometric and physical parameters results in a value of 1.

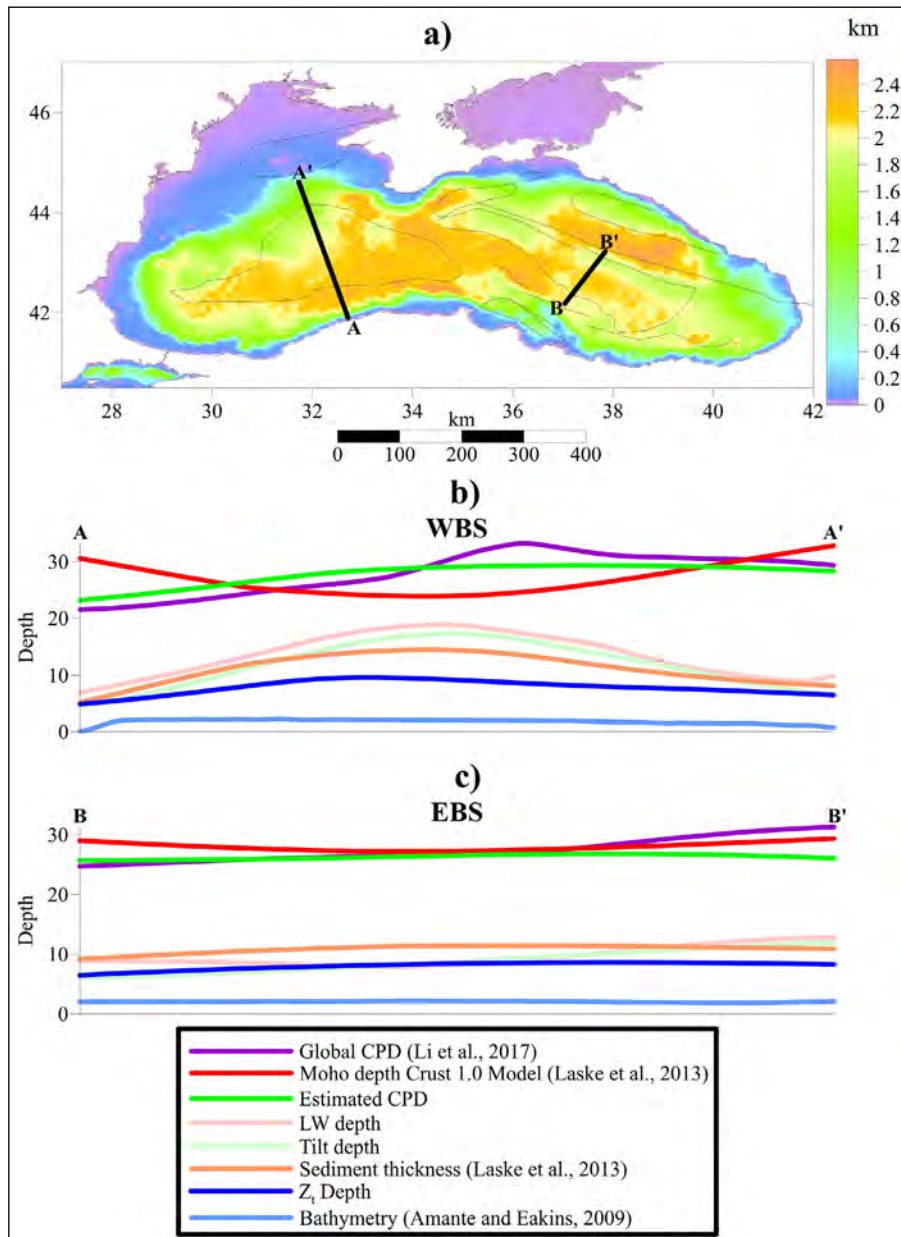


Figure 7- The selected profiles along the WBS and EBS: a) profile locations overlaid on the bathymetry, b) profile A-A' along the WBS, and c) profile B-B' along the EBS. Each profile includes the Global CPD, Moho depth, Estimated CPD, LW depth, Tilt Depth, sediment thickness, Z_r , and bathymetry.

Figure 8 presents the correlation analysis conducted for each variable along the A-A' profile representing the Western Black Sea (WBS) and the B-B' profile representing the EBS. The correlation coefficients range from -1 to 1, with 1 indicating a perfect positive correlation, 0 denoting no association, and -1 representing a complete inverse correlation. Specifically, the focus was on examining the correlation between the estimated Curie point depth (E CPD) and

the global CPD (G CPD) to compare our estimations with the findings reported by Li et al. (2017). Along the A-A' profile (WBS), a strong correlation of 0.93 is observed between G CPD and E CPD, while along the B-B' profile (EBS), a moderate correlation of 0.58 is found between these variables. It is anticipated to observe a high correlation between E CPD and G CPD. However, the relatively lower correlation along the B-B' profile may be attributed to various factors,

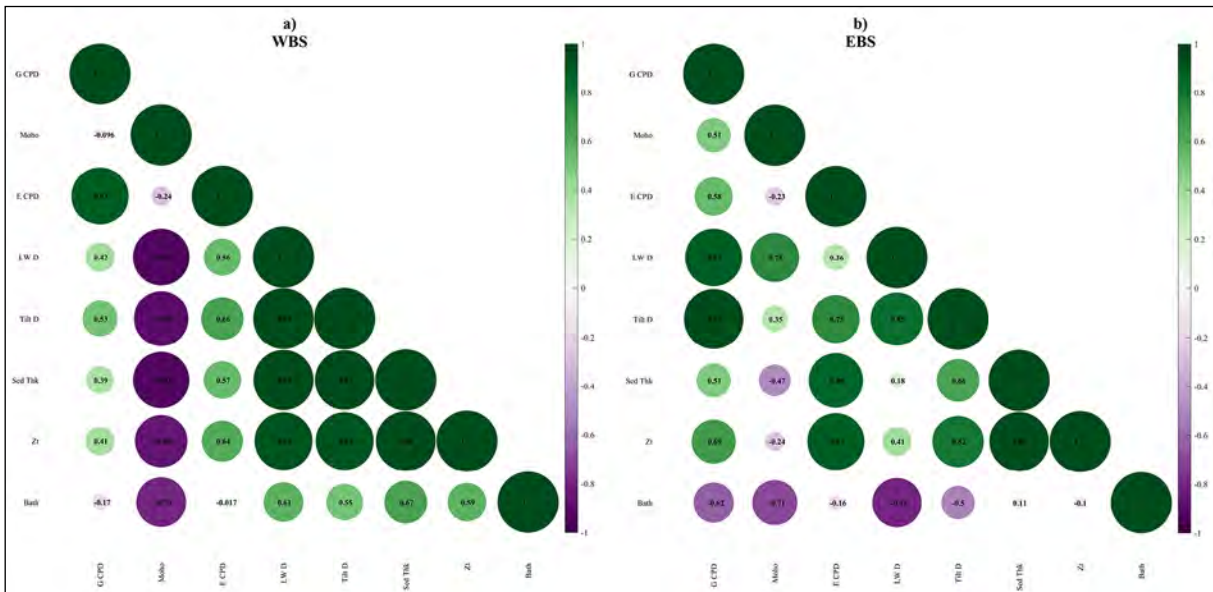


Figure 8- Correlation plots of WBS (A-A' profile) and EBS (B-B' profile). The radius of the circles is controlled by the correlation value. G CPD: global Curie point depth, Moho: Moho depth, E CPD: estimated Curie Point Depth, LW D: Local Wavenumber depth, Tilt D: Tilt depth, Sed Thk: sediment thickness, Z_t: top depth of the magnetic basement and Bath: bathymetry.

including the grid cell size, chosen block size, range of selected wavenumber components during CPD estimation, or the positioning of overlapping adjacent blocks.

As mentioned previously, Z_t, tilt depth (Tilt D), and LW depth (LW D) are indicators of the uppermost depth of the magnetic basement. Therefore, a strong correlation is expected among these variables, approaching a value of 1. Along the A-A' profile, the correlation between these variables exceeds 0.94, indicating a strong positive correlation. However, along the B-B' profile, while Tilt D - Z_t and Tilt D - LW D exhibit a high correlation, the correlation between LW D and Z_t is relatively low at 0.41. This lower correlation may arise from distinct characteristics of LW D and Z_t in the south-western part along the B-B' profile (Figure 7c).

The correlation between sediment thickness (Sed Thk), Tilt D, LW D, and Z_t plays a crucial role in understanding sediment accumulation rates. Along the A-A' profile, the correlation values between Sed Thk and LW D, Sed Thk and Tilt D, and Sed Thk and Z_t are above 0.97. These findings suggest a strong association between the top depth of the magnetic basement inferred by Tilt D, LW D, Z_t, and Sed Thk.

It is possible that the sediment deposition process remains uninterrupted by deformation, folding, seismic activity, or variations in riverbed conditions.

Furthermore, the higher sedimentation rate can be attributed to Balkanide Orogeny during Eocene. The Western Black Sea, being older, had more time for sediment deposition and provided a more suitable environment for sediment accumulation. Therefore, the expected outcome of a higher sediment accumulation rate in the WBS is justified by the correlation values of Sed Thk, Tilt D, LW D, and Z_t. Additionally, Nikishin et al. (2015) emphasized a higher sedimentation rate in the WBS from the Cretaceous to Eocene period, further supporting these findings.

The outcomes of Spadini et al. (1996) findings have yielded significant insights into the origins of the WBS and EBS regions, highlighting fundamental differences in their lithospheric properties. Notably, the WBS's formation can be attributed to the rifting of a thick lithosphere (approximately 200 km), characterised by lower temperatures and higher mechanical strength, thereby contributing to a substantial depth of necking (around 25 km). This thick lithosphere corresponds to the stable continental Moesian Platform. Conversely, the EBS's genesis is associated with the rifting of a

thinner lithosphere (approximately 80 km), resulting in elevated temperatures and weaker mechanical properties, leading to a shallower depth of necking (approximately 15 km). This scenario aligns well with the development of the EBS on the site of a prior (Mesozoic) back-arc basin (Spadini et al., 1996). To summarize, the geological history of the Black Sea region exhibits notable differences between its western and eastern basins. In the WBS, the rifting process was primarily responsible for the development of the stable Moesian Platform. On the other hand, in the Eastern Black Sea, a younger extensional basin was formed, overlaying a pre-existing area that had previously acted as a back-arc basin since the Early Jurassic period. This intriguing contrast in geological evolution sheds light on the diverse tectonic processes shaping the Black Sea basin's geology. These distinctive lithospheric properties and structural features offer plausible explanations for the observed variations in correlation plots between the Eastern and Western Black Sea regions.

6. Conclusion

This study presents a comprehensive theoretical framework aimed at unravelling the origin of the Black Sea, with a specific emphasis on both the East and West Black Sea basins. By leveraging a diverse range of data, including CPD values, heat flow values derived from CPD data, bathymetry, Moho depth, sediment thickness, and magnetic depth estimation techniques (Tilt angle and LW methods), a detailed examination of the region's geological characteristics has been conducted. Particularly, the impact of volcanic rock distribution on magnetic anomalies within the Black Sea area has been investigated. Moreover, an extensive analysis of the correlations between CPD values, bathymetry, Moho depth, sediment thickness, tilt depth, and LW depths was performed independently for the EBS and WBS basins. By scrutinizing these correlations in light of the tectonic history and presence of volcanic rocks, the significant disparities observed between the EBS and WBS have been thoroughly interpreted. This research has provided valuable insights into the distinct geological processes and contributing factors that have shaped the development and divergence of the EBS and WBS.

On the whole major findings in this study can be summarised as:

- Edge detection techniques, namely the tilt angle and LW methods, have played a crucial role in enhancing the interpretation of magnetic data. The analysis of tilt angle and LW maps reveals a significant difference in the estimated uppermost contact depth between the WBS and the EBS, with the WBS showing an approximately 18 km higher depth than the EBS.
- The WBS exhibits a higher Curie Point Depth (CPD), ranging up to 32 km, compared to the East Black Sea (EBS), where CPD values range between 24 to 28 km. Additionally, the CPD values in the EBS demonstrate abrupt changes, while the WBS displays a more consistent pattern, indicating the presence of thermal and magnetic property variations between these two regions.
- The correlation between Sed Thk, Tilt D, LW D, and Z_c is crucial for assessing sediment accumulation rates. Along the A-A' profile, the correlation values between these variables are all above 0.97, indicating a strong association between the top depth of the magnetic basement and sediment thickness. This suggests undisturbed sediment deposition, unaffected by deformation, folding, seismic activity, or variations in riverbed conditions.
- In aggregate, the findings derived from this empirical inquiry delineate that the two basins underwent divergent tectonic processes.

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Sediment thickness in the Thrace Basin estimated from the three-dimensional inversion of magnetotelluric data

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Research Article

In Memoriam

by Ahmet Tuğrul Başokur

The first author, Cemal Kaya, passed away in February, 2022. The Turkish geophysical community will always remember Cemal Kaya's friendship, honesty and sincerity. He graduated from İstanbul Technical University (ITU) Faculty of Mines, Department of Geophysical Engineering in 1981. He started his career as a geophysical engineer at the General Directorate of Mineral Research and Exploration. He completed his MS and PhD under my supervision at Ankara University. We completed many research projects in the 1990s, including the controlled-source audio-magnetotelluric (CSAMT) method for massive sulfide exploration and the magnetotelluric method in geothermal resource exploration, the first examples in Türkiye. We also initiated the systematic use of the MT method in crustal research as a joint TÜBİTAK project of Ankara University and the General Directorate of Mineral Research and Exploration in 1995. This article is the first profile of national crustal research project. Since only two-dimensional modelling could be applied in the 1990s, a conductive artefact zone appeared under the Hisarlıdağ uplift in the resistivity model since the Thrace Basin surrounded by the Aegean, Marmara and Black Seas on three sides. Now, we have constructed a more realistic model of the Thrace Basin using the three-dimensional modelling that permits including the seabed topography.

Keywords:

Three-Dimensional
Magnetotelluric Inversion,
Thrace Basin, Sediments
Thicknesses.

ABSTRACT

The time-variation of the magnetotelluric (MT) fields was recorded at 37 measurement stations in the Thrace Basin (North-western Türkiye) by a remote-reference MT system that records the MT data in two frequency bands allowing a data acquisition between 320–7.5 Hz and 6–0.00055 Hz, respectively. The measurement stations were located from the Aegean Sea to the Black Sea seashores along a 207 km profile in the southeast-northwest direction. Three-dimensional inversion of MT data outlined the main geological elements of the region, the interface between sedimentary cover and basement rocks. The total thickness of the sedimentary layers in the basin is interpreted using the inflection and deflection points of the three-dimensional resistivity model in the vertical direction.

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1. Introduction

MT studies have been used to investigate oil, gas and geothermal reservoirs, deep sedimentary basins, the upper mantle and the crustal thickness for more than six decades (Swift, 1967; Vozoff, 1972; Jupp and Vozoff, 1977; Beblo et al., 1983; Wannamaker, 1983; Hersir and Björnsson, 1991; Lezaeta et al., 2000; Gokarn et al., 2008; Kaya, 2010; Hacıoğlu et al., 2020; Başokur et al., 2022; Hacıoğlu et al., 2023). We used the MT method to investigate the thickness of the sedimentary layer in the Thrace Basin of northwestern Türkiye because of its cost-effective implementation along a profile. The basin is one of the most important hydrocarbon production areas in Türkiye (mainly natural gas). The existing drill holes ended in the sedimentary cover around the MT line. An estimation for the total sediment thickness was published by Siyako and Huvaz (2007) using seismic and well data as a contour map. Three-dimensional inversion of MT data provides additional information about the

shape and depth of the basement along the 207 km line between the Aegean and Black Seas seashores.

2. Geology of the Thrace Basin

The Thrace Basin is considered to be an intramontane Tertiary basin surrounded by the Strandja in the north, the Rhodope in the west, the İstanbul Zone in the east, and the Sakarya Continent and Intra-Pontid Suture in the south, as shown in Figure 1. The basement of the surrounding massifs consists of metamorphics, İstanbul Paleozoic-Mesozoic sedimentary sequences, ophiolitic melanges and occasional igneous bodies of the pre-Upper Cretaceous age (Doust and Arıkan, 1974; Turgut et al., 1983; Siyako and Huvaz, 2007). Okay et al. (2023) partitioned the sedimentary layers into three sequences separated by major unconformities: Lower-middle Eocene, Middle/upper Eocene-Oligocene, and Miocene-Pliocene. The basin is elliptical, and its long axis extends in the southwest-northeast direction. A series of basement step faults limits the

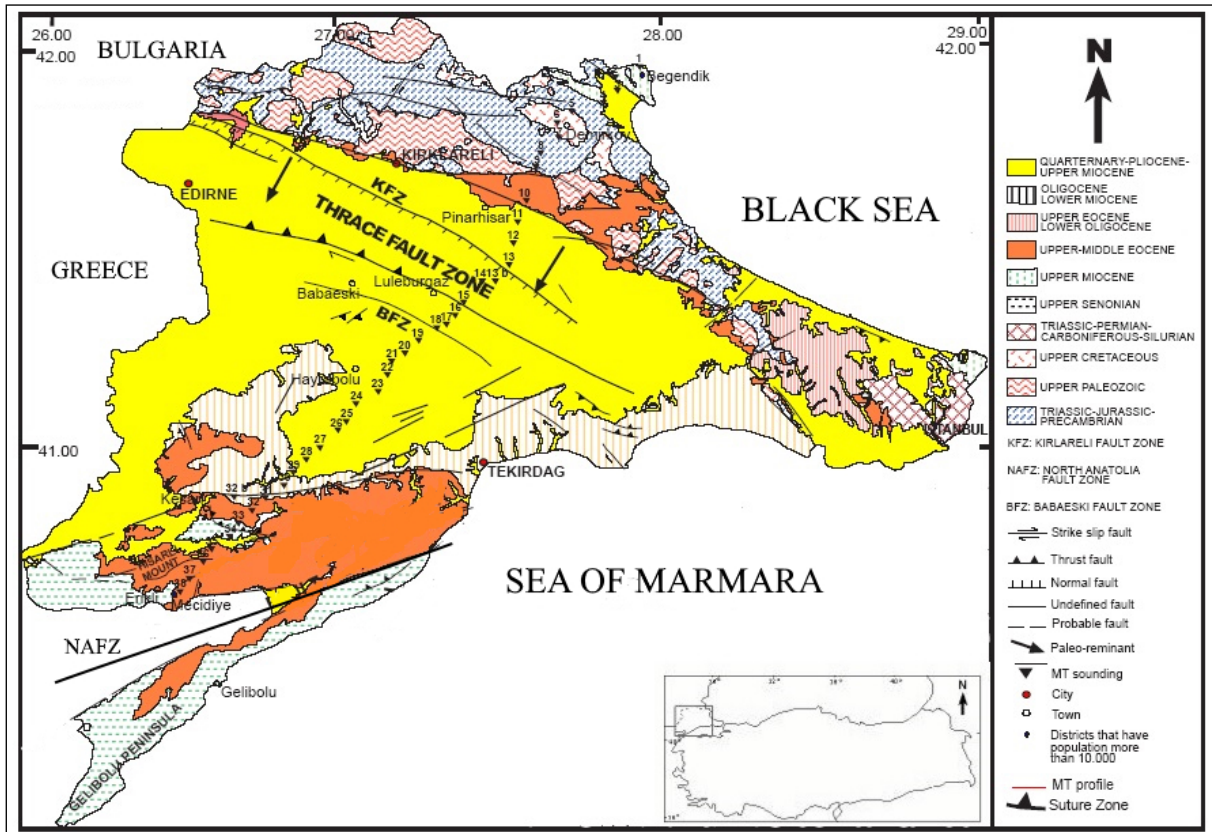


Figure 1- Geological map of Thrace Basin and MT stations (station numbers over triangles), modified from the Geological Map of Türkiye, General Directorate of Mineral Research and Exploration.

basin in NE, and a fault scarp controls the flanks of the basin in SE. Very thick flysch-type clastics were due to a very fast subsidence. Some clastic materials were deposited in the Lower Oligocene because of continuous transgression that caused the submerging of the southwestern parts of the basin. In contrast, the carbonates were deposited on the northern shelf simultaneously. An active volcanism caused dacitic and andesitic ashes. In the Middle Oligocene, the subsidence slowed, and the basin's depositional environment became lacustrine and fluvial. The deposition continued without interruption until the Upper Oligocene. Volcanic activities lost intensity in the Upper Oligocene (Turgut et al., 1983; Siyako, 2006).

Doust and Arıkan (1974) postulated that a major erosional phase caused the removal of much of the Lower and Upper Oligocene sediments into the centre and southern part of the basin. The Lower and Upper Oligocene sedimentary sequence demonstrates a transgressive-regressive depositional cycle that includes shallow marine sediments. A major tectonic event in the Middle Eocene gave rise to faulting and folding in the SW-NE direction. The late Pliocene tectonic activities shaped the present configurations of the basin (Turgut et al., 1983). On the other hand, according to Perinçek (1991) and Perinçek et al. (2015a), the final most important tectonic event in Thrace Basin occurred before the deposition of Ergene formation. Thrace fault system became active before the Ergene formation and continued during the sedimentation of it. Thrace fault systems cause uplift and erosion along the fault zones (Perinçek et al., 2015b).

Two tectonic trends are dominant in the study area. 1) The Ganos Mount trend is in the NE-SW direction parallel to the Ganos Mount and constitutes the southward limit of the basin. 2) The Strandja trend has an NW-SE strike parallel to the Strandja basement ridge, limiting the north basin (Doust and Arıkan 1974). Coşkun (1997) postulates that the older structures trending in the NE-SW direction were influenced by basement paleotopography, and the younger structures trending in the NW-SE direction were under the influence of the North Anatolian Fault (NAF). Perinçek (1991) interpret

the Thrace fault system as the oldest strand of the North Anatolian Fault, releasing-bend extensional structures. The Thrace strike-slip fault system consists of the west-northwest-striking Kirklareli, Lüleburgaz, and Babaeski fault zones that are inactive presently (Perinçek, 1991).

The collision between the Sakarya and Rhodope continents leads to lateral movements and normal basement faults, which become detachment faults during the subsequent opening of the Thrace Basin (Turgut et al., 1991; Tüysüz et al., 1998). The Bouguer gravity map clearly shows these trends and, the thick basin fills are represented by a low value of gravity anomalies (Figure 2). Sakıncı et al. (1999) suggested that the basin principally developed by the activities of the Thrace and Ganos strike-slip fault systems following the continental collision during the Late Oligocene-Early Miocene period. Thrace Basin was opened during the Middle Eocene on Strandja Massif as a post-collisional fault-controlled extensional basin and turned into an intramontane basin in the Middle Miocene (Tüysüz et al., 1998). However, a fore-arc origin for the Thrace Basin has been proposed, which is linked to the subduction of the Intra-Pontide Ocean between the Middle Eocene and Oligocene. The Rodope-Pontide magmatic arc was developed on the overriding Strandja zone and started to rift and extend, forming the Thrace Basin (Görür and Okay, 1996).

3. Data Acquisition and Processing

The MT profile lies in the southwest-northeastern direction from the Aegean Sea to the Black Sea and consists of 40 measurement stations (Figure 1). Station intervals ranged from 4 to 5 km, depending on the accessibility of the territory. The total length of the profile was 207 km. The first and last measurement stations were close to the Black Sea and Aegean coast.

The magnetotelluric (MT) method simultaneously measures orthogonal components of the natural horizontal electric and magnetic fields. The electromagnetic variations, lasting longer than 1 second, consist of micropulsations originating from the earth's magnetosphere's interaction with charged particles ejected from the sun and other sources. Lightning strokes almost entirely generate

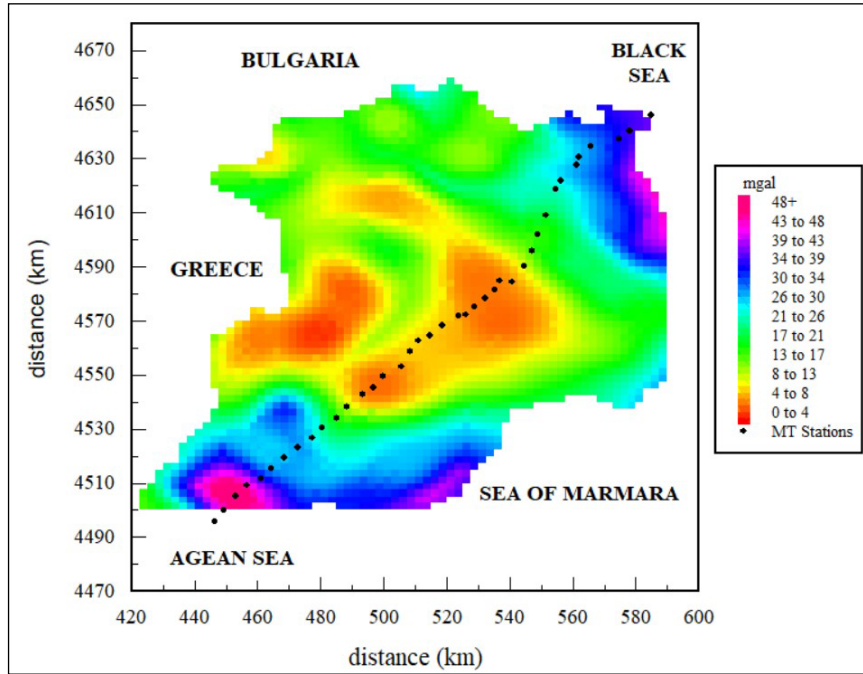


Figure 2- Location of MT stations used in this study over Bouguer gravity anomaly map (MTA, 1999).

electromagnetic variations below one second. Phoenix-V5 equipment was used to acquire MT data consisting of three orthogonal magnetic (H) and two orthogonal electric (E) field components. MT electrical dipoles of 100 m, equal to the length of one side of the TEM transmitter loop, were applied to measure E-fields in NS and EW directions. Nonpolarizable lead-lead chloride ($Pb-PbCl_2$) electrodes were used for E-field measurement because of their long-term stability of polarization (Petiau and Dupis, 1980). Induction coils and an air loop on the ground were used to measure the horizontal and vertical components of the magnetic fields, respectively. Additional two orthogonal magnetic fields were measured at a remote reference station.

The data were acquired in two steps by measuring high and low frequency bands in 1995. The high band consisted of 12 frequencies between 320 and 7.5 Hz. There were 28 frequencies in the low band in the range of $6-5.493 \times 10^{-4}$ Hz. MT records were acquired for more than 24 hours in the lower band. The impedance values in 40 frequencies were combined in the frequency domain after the Fourier transformation of time-domain data using cascade decimation (Wight and Bostick, 1980).

The orthogonal components of the horizontal electric and magnetic fields measured in the time-domain are linearly dependent. Consequently, their Fourier transform leads to a 2×2 tensor, and its elements are called impedances (Z). Impedance values are estimated from a complex electric field (E) ratio over the magnetic field (H) in the frequency domain. The tipper (T) connects the vertical and two horizontal magnetic fields. The impedance tensor and tipper vector are given in the frequency domain after the application of Fourier transformation as follows (Equations 1, 2) :

$$\begin{pmatrix} E_x \\ E_y \end{pmatrix} = \begin{pmatrix} Z_{xx} & Z_{xy} \\ Z_{yx} & Z_{yy} \end{pmatrix} \begin{pmatrix} H_x \\ H_y \end{pmatrix} \quad (1)$$

$$\begin{pmatrix} H_z \end{pmatrix} = \begin{pmatrix} T_x & T_y \end{pmatrix} \begin{pmatrix} H_x \\ H_y \end{pmatrix} \quad (2)$$

where x and y denote the orthogonal components in the north and east directions, and z indicates the vertical component. The impedances are always a decreasing function of the period, and the shape of MT curves is independent of subsurface resistivity distribution. Then, the general behaviour of impedance data may not give any idea about the resistivity variation

in the subsurface. The following normalization (Equation 3) improves the physical significance of the data (Başokur, 1994; 1999):

$$F = \frac{Z}{\sqrt{i\omega\mu}} = \frac{I}{\sqrt{i\omega\mu}} \frac{E_x}{H_y} \quad (3)$$

where $i = \sqrt{-1}$. F is named as the frequency normalized impedance or shortly FNI. The unit of the FNI function is the square root of ohm-m ($\sqrt{\text{ohm-m}}$). μ and ω denotes the magnetic permeability of free space and angular frequency, respectively. This normalization connects the measured data and the subsurface resistivity distribution and directly reflects resistivity variations versus depth as the function period (Başokur, 1999; Başokur, 2023). The validity of the measured MT data can be tested using the causality concept. Başokur (2023) proved that the real and imaginary parts of the FNI function can be calculated from each other via the Hilbert transform. This procedure help check if the data satisfies natural EM field conditions. As an example, Figure 3 shows the measured and Hilbert-transformed FNI data for the station 33 and proves the data validity for this station.

4. Three-dimensional Inversion

The purpose of geoelectrical data inversion is to estimate the subsurface resistivity distribution. A resistivity model is derived indirectly by searching for a fit between theoretical data computed for a conceptual model and the measured data. The most commonly used conceptual models are 1D, 2D or 3D subsurfaces in historical order. The reliability of the derived model is dependent on the consistency of the conceptual model with the actual surface conditions. The type of conceptual model should be decided before geophysical exploration so that the survey design can be planned according to the selected conceptual model. All constants defining a particular conceptual model are named ‘model parameters’. A 3-D model consisting of a huge number of cube-shaped cells is preferred for a better description of subsurface physical conditions. Usually, the inversion algorithm is initialized by a homogeneous earth model by assigning the same resistivity value to all model cells. The model parameters only comprise the resistivity values of each cell. Keeping the geometry of cells constant during iterations drastically reduces the number of model parameters to be estimated.

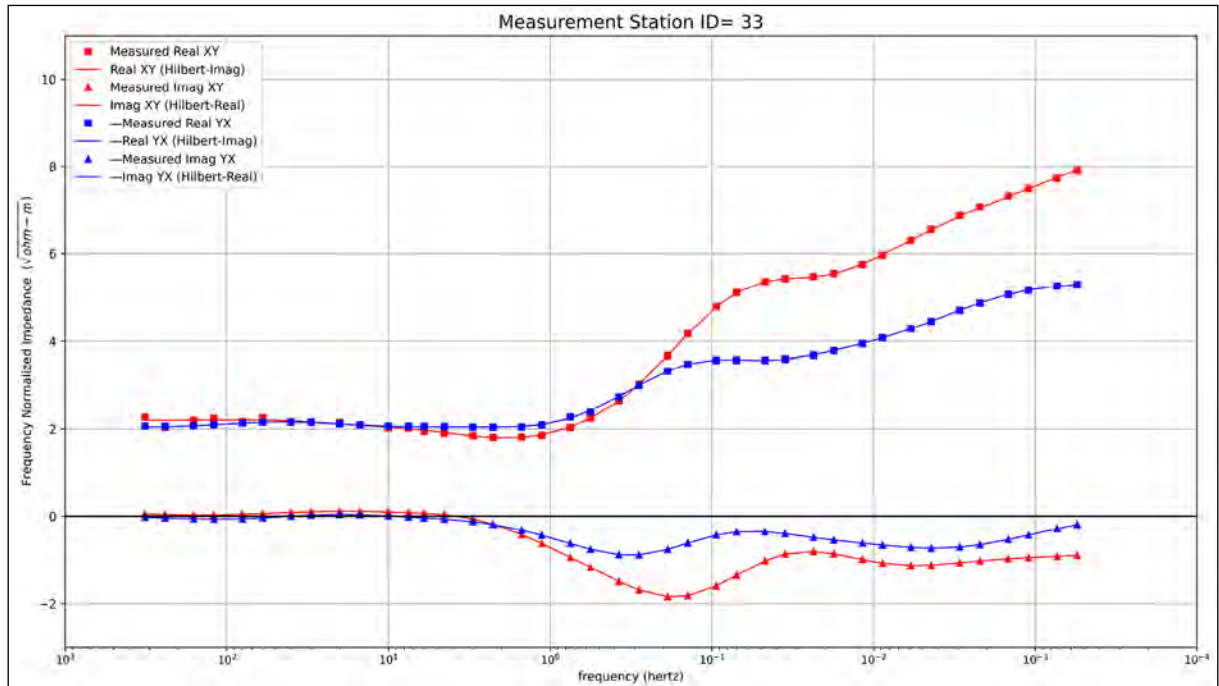


Figure 3- Frequency normalized impedance, real (squares) and imaginary (triangles) components, and their corresponding Hilbert transforms (continuous curves).

Then, the cell resistivity values are updated iteratively by computing a parameter update vector at each iteration from the differences between measured and theoretical data sets (Başokur and Akca, 2011). Models corresponding to misfit values $E(p)$ lower than a threshold are considered a possible solution to represent subsurface resistivity distribution. The reduction of misfits along successive iterations is not sufficient to obtain a smooth and geologically acceptable model. Also, preventing random resistivity changes among neighbouring cells is necessary. For this purpose, a stabilizer function $S(p)$ is added in the objective function $\phi(p)$ to control the model resistivity variations as follows (Equation 4):

$$\phi(p) = E(p) + \alpha S(p) \quad (4)$$

where α is the regularization coefficient. The misfit function is defined as the sum of the square of the differences between measured and calculated data values at each frequency (Equation 5) :

$$E(p) = \sum_{i=1}^n \frac{(d_i - f_i)^2}{\sigma_i^2} \quad (5)$$

d_i and f_i are the measured and theoretical data, respectively. σ_i is the standard deviation corresponding to i th frequency value. n is the number of measured data values. The $nmrs$ value quantifies the degree of misfit, and an $nmrs$ value which is lower than three is expected in the final step of inversion (Equation 6):

$$nmrs = \frac{1}{n} \left[\sum_{i=1}^n \frac{(d_i - f_i)^2}{\sigma_i^2} \right]^{1/2} \quad (6)$$

The 3D non-linear conjugate gradient (NLCG) software was used and named ModEM (Egbert and Kelbert, 2012; Kelbert et al., 2014). ModEM is a modular system of parallel computer codes for the inversion of electromagnetic (EM) geophysical data and uses the following stabilizer function (Equation 7):

$$S(p) = \sum_{j=1}^m \frac{(m_j - m_j^0)^2}{\sigma_j^2} \quad (7)$$

where m_j is the resistivity value of j th cell and m_j^0 corresponds to the initial resistivity value of a priori model. σ_j represents the model covariance value corresponding to the j th cell. m is the number of model parameters. A 3-D MT inversion requires a lot

of computing time despite using a multi-core cluster and parallelized calculations. The computation was performed using an HP ProLiant DL580 Gen10 computer with 80 cores.

Bayrak et al. (2006) applied a dimensional analysis to the current MT data. They concluded that the high-frequency part, corresponding to the younger sedimentary fills, shows a 1D behaviour, while the data at the low frequencies show a 3D behaviour. Consequently, we applied 3D inversion and the bathymetry of the Black Sea, Marmara and Aegean Seas were inserted into the model and a sea resistivity value of 0.33 ohm-m was assigned to represent their contribution to the data. The topography is modelled from SRTM90 data. An average elevation is used for the z coordinate of the top of the corresponding cell. Figure 4 shows the 3D modelling grid in the x-y plane comprising 85 and 80 cells, respectively. The model is also extended to 95 cells in the z -direction. The total number of parameters to be solved is 646.000. The ModEM software has been executed in a cluster so that one processor serves the calculation of the theoretical data for one frequency. The inverted data consists of 40 sample values in the range of frequencies between 320 and 5.493×10^{-4} Hz.

The Thrace Basin consists of a sediment layer with low resistivity, so the industrial noise was attenuated relatively short distances. Because of its higher resistivity values, the noise contribution over Strandja Massif was higher than that of the basin in intermediate and long periods. The two off-diagonal components of the impedance tensor have been used in the inversion. However, the diagonal components corresponding to the low frequencies were scattered while almost zero at the high-frequency band because of the thick 1D sedimentary cover. For these reasons, the diagonal components were not used in the inversion. The tipper values have been disregarded because of high noise contamination. The algorithm was initialized with homogeneous resistivity values of 100 ohm-m. Meanwhile, various error levels and smoothing factors for horizontal and vertical directions were used for each initial-guess model. Finally, the model produced the smallest misfit ($nmrs$) value of 1.8 after 55 NLCG iterations. The measured

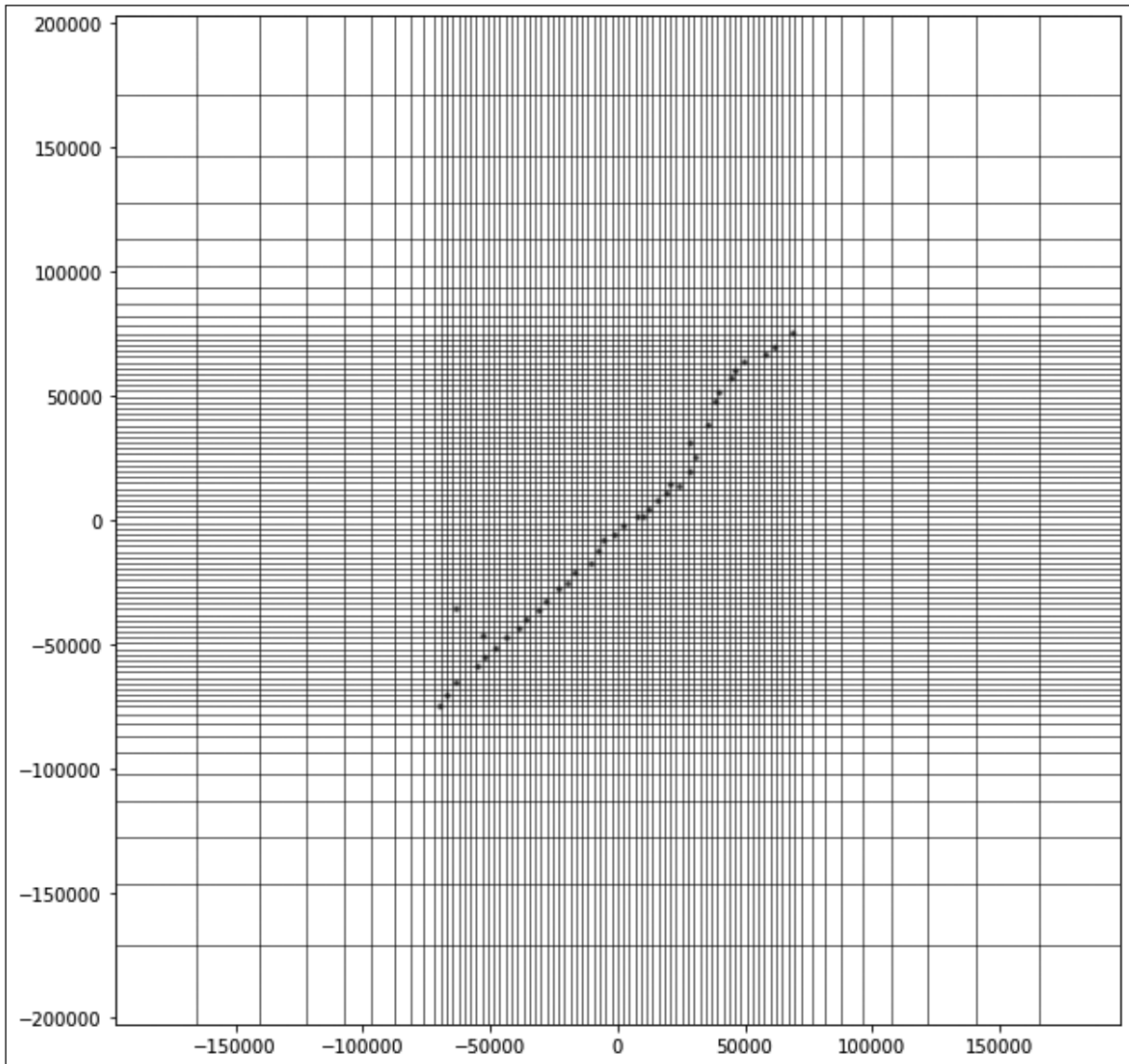


Figure 4- Plan view (x-y plane) of the modelling grid used in the 3-D inversion. The yellow dots show measurement stations. The survey area is subdivided into 646,000 cells. There are 85, 80 and 95 cells in the north, east and depth.

and calculated curves corresponding to station 33 are shown in Figure 5.

Figure 6 shows the FNI pseudo-sections of the measured and calculated data in a distance-frequency (x - f) plane for the YX component. It can be concluded that the final 3D model produced a satisfactory fit between the measured data and model response, and there are no artefacts in the model due to noise or surrounding seas. The pseudo-section derived from the measured data indicates the shape of the basement since the MT profile is almost perpendicular to the geological strike of the basin.

5. Interpretation

Figure 7a illustrates the final model obtained from the 3D inversion. A cell was colourized by a specific colour proportional to its intrinsic resistivity value. The resistivity limits were selected in the range $[0,3]$ $\log_{10}$ ohm-m corresponding to the resistivity values of 1 and 1000 ohm-m in a linear scale, respectively. The selected resistivity scale better reflects the shape of the basin due to the high resistivity contrast between relatively low resistivity young sediments and high resistivity basement. However, interpreting the depth of interfaces using a colour scale will

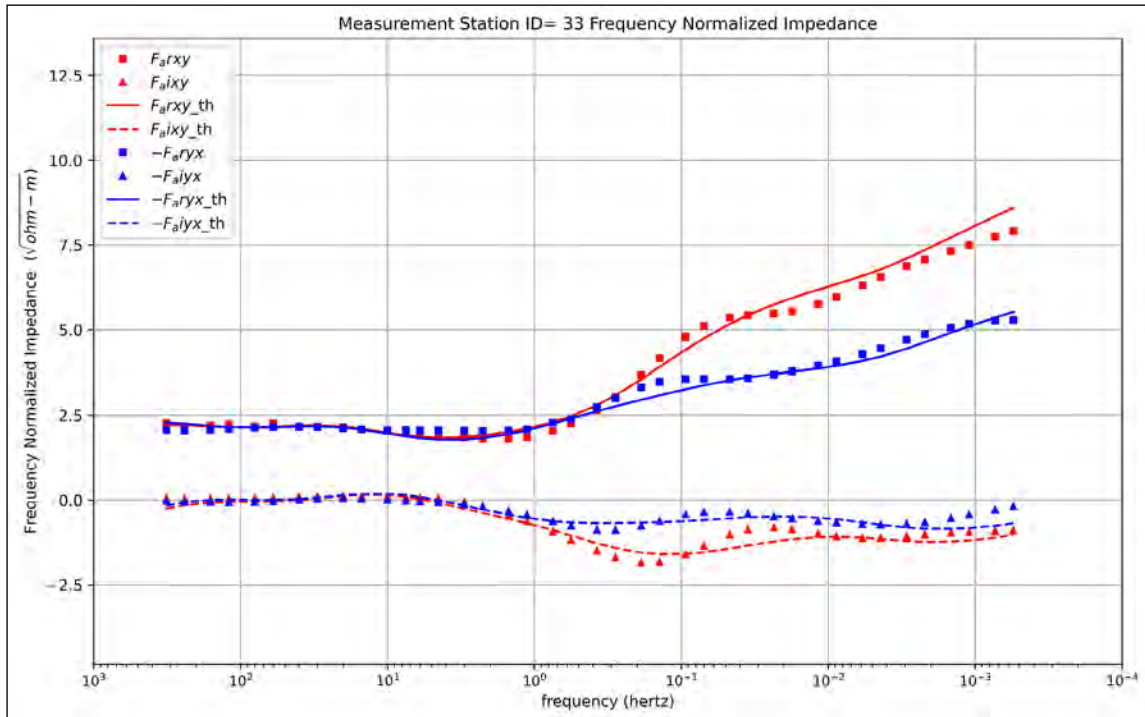


Figure 5- Data fit for the 3D inversion at station 33. The red and blue colours indicate the xy and yx components. Squares and triangles show the measured real and imaginary components, respectively. The continuous curves are the model responses.

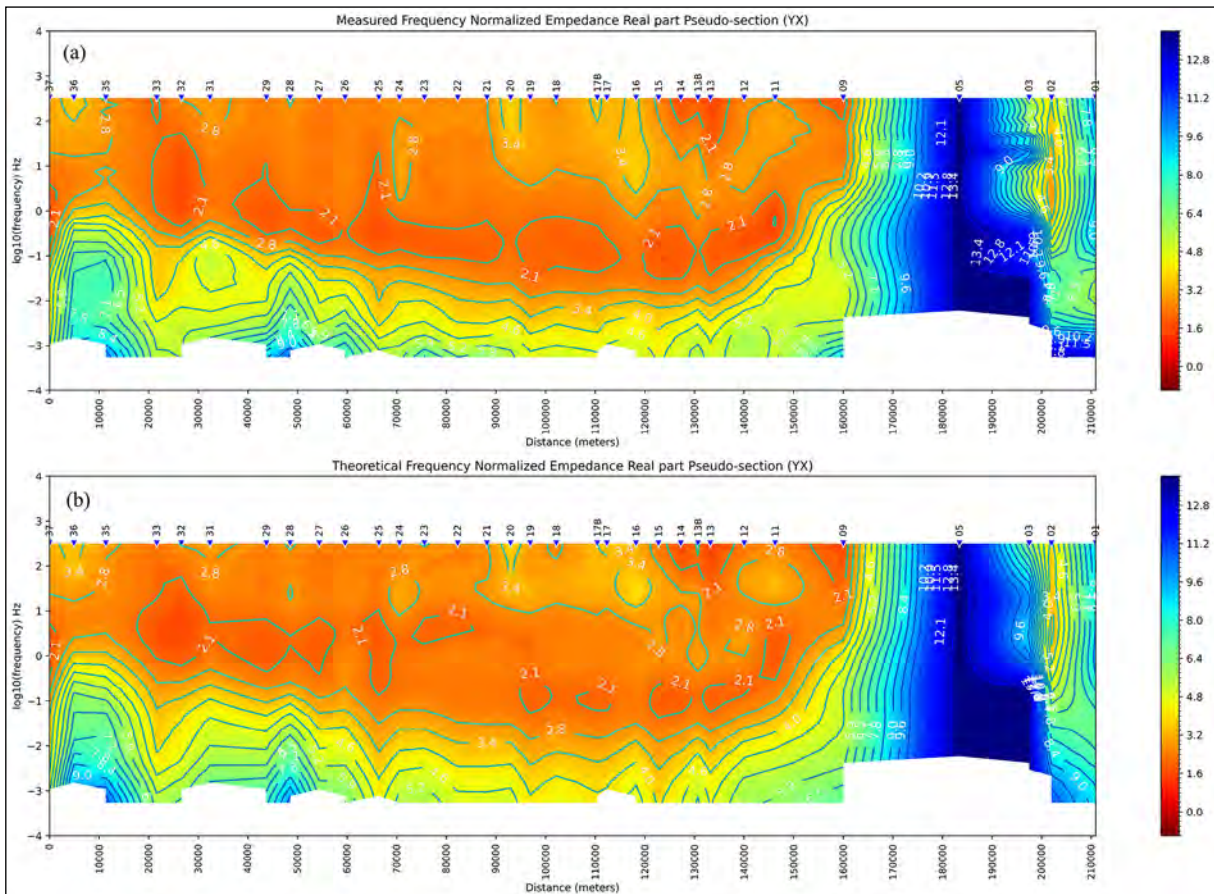


Figure 6- Comparison of the measured a) and calculated b) real parts of the FNI function for the YX component pseudo-sections.

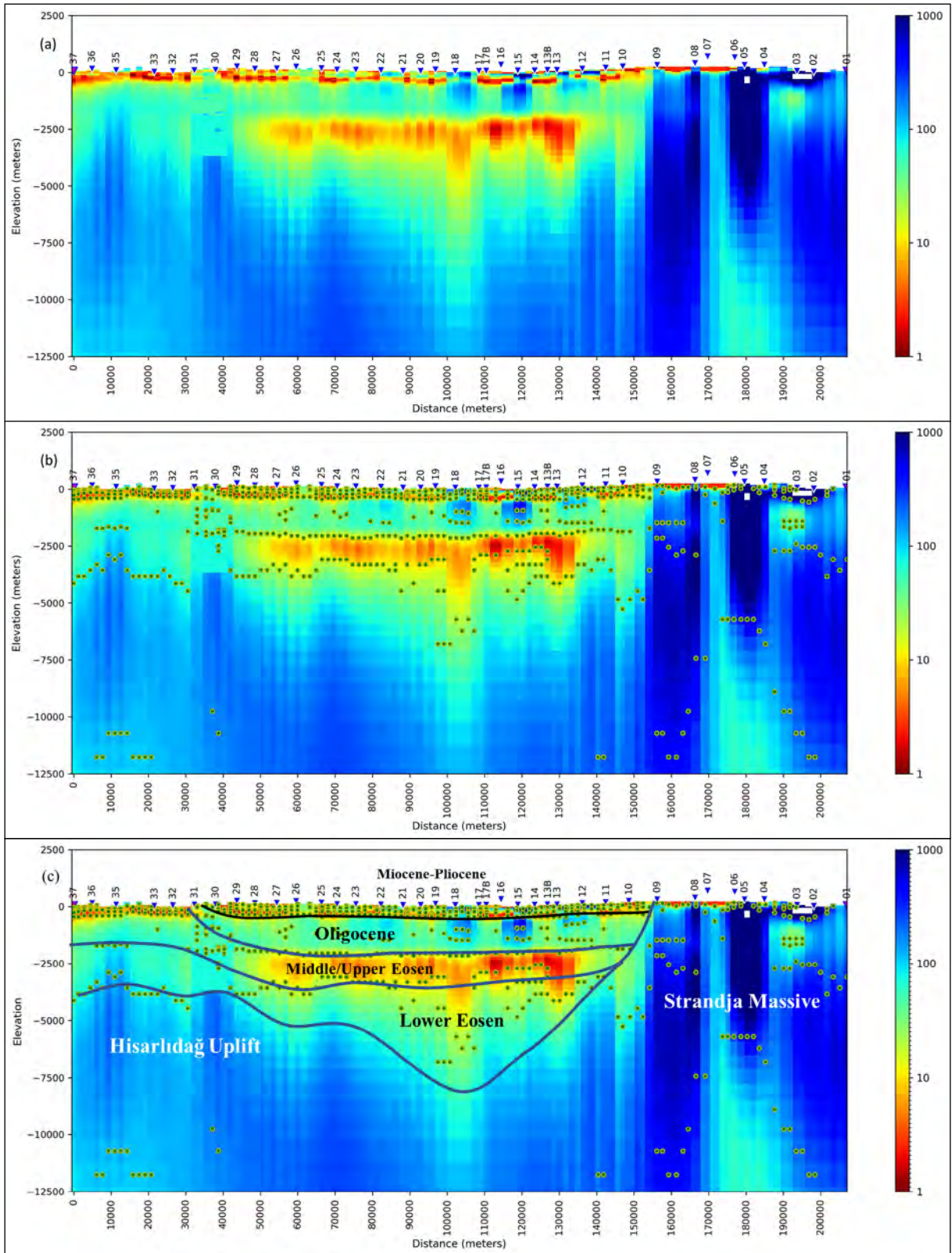


Figure 7- a) Resistivity section derived from the 3D inversion, b) the inflection and deflection points of the model in the vertical direction, c) possible interpreted interfaces.

remain subjective. Changing the resistivity scale of the colour map will produce images in which the probable location of interfaces will be perceived differently. This is the main difficulty in identifying the layer interfaces by visual inspection. Akca and Başokur (2010) developed the concept of the structure-based model in which the model parameters simulate undulating interfaces in 2D subsurface. However, the conventional derivative-based inversion methods could not be applied to the structure-based model parameterization. Searching for a model that produces minimum misfit can be performed by model verification algorithms that generate and verify many distinct and complicated models. For cases where many models should be verified, the genetic algorithm is one of the most suitable for verifying large model sets (Başokur et al., 2007). However, the computation of 3D models is expensive and time-consuming, so the next computer generation can tackle constructing structure-based 3D models.

ModEM software produces smooth models; consequently, the resistivity versus depth is a smooth curve. For this reason, we apply the scheme developed by Başokur (2022) to delineate possible interfaces. The variation of resistivity along a column in the vertical direction with the same central x and y coordinates is extracted as a function of z values from the smooth model. For example, Figure 8 shows the depth-resistivity curve derived from the 3D model cube for particular x-y coordinates corresponding to station 23 (black lines). The maxima, minima, (magenta dots in Figure 8) inflection and deflection points (red dots) can be derived using the Numpy library of Python (<https://numpy.org/>) from the first and second numerical derivatives, respectively. The inflection and deflection points of the depth-resistivity (z - ρ) curve are the most probable boundaries of the interfaces since they indicate sharp resistivity variations. The applied procedure is similar to edge detection algorithms. A subsurface model is constructed by connecting the points (green lines in Figure 8). The method can be repeated for all z - ρ curves along a profile, and possible layer interfaces can be plotted as dots over a resistivity section (Figure 7b). This numerical strategy helps the interpreter identify the layer interfaces from a cell-based smooth 3D model.

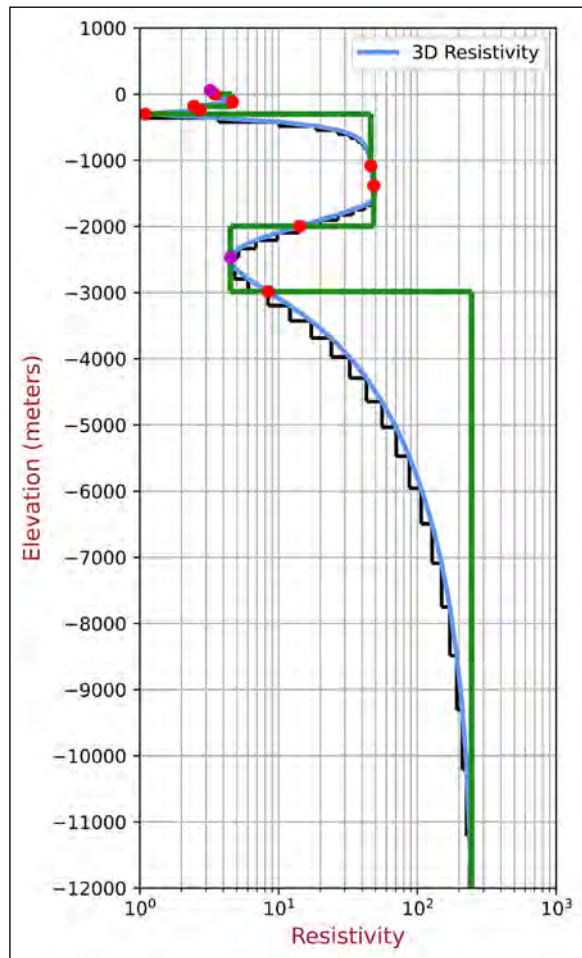


Figure 8- Resistivity variation with depth. Black lines are the resistivity values of each cell. Green lines indicate the probable depth of interfaces.

Figure 7c illustrates our interpreted model derived from the dotted inflection and deflection points. There is no significant sedimentary cover on the top of the Strandja massif. The slope variations in the basement floor were well delineated because of the high resistivity contrast between sedimentary units and crystalline basement. The overall interpretation could imply that major structural elements of the region could be outlined with the help of the MT method without much difficulty. Moreover, the main fault zones may be identified with the help of the information obtained from the surface geology. The depth to the basement along the MT profile reaches 8000-8500 meters. However, the total sediment thickness can reach 10-11 km at the east of the MT profile. A depth estimate using the power spectrum of the gravity data produces a similar result.

6. Discussion and Conclusion

There have been three different suggestions for the location of the Intra-Pontid suture zone representing the boundaries of former Sakarya and Rhodope continents: (1) It has been suggested that a suture of the Intra-Pontide Ocean existed between Strandja Massif and Sakarya continent as noted by Şengör and Yılmaz (1981). The ocean closed during the Cretaceous to Palaeocene period. However, there has been a controversy over the existence and location of this suture zone. It has been proposed that the suture was located at the south of the Thrace Basin, extending towards the west-east direction along the north coast of the Aegean Sea towards the Marmara Sea, as reported by Şengör and Yılmaz (1981). (2) The north of the Strandja massif has been suggested to be the location of the suture zone (Yılmaz et al., 1997). (3) Bayrak et al. (2004) reported that the suture was an N-S-oriented zone between İstanbul and Çatalca in the north. They have also proposed an alternative location close to the surface contact between Thrace Basin and the Strandja massive in the north based on the interpretation of their nine magnetotelluric stations. Their short profile extended approximately in the N-S direction from Strandja Massif towards the Sea of Marmara. It crossed our MT line around station 13 and contained no information about the south of station 13. A possible location may correspond to the low resistivity anomaly beneath stations 12 and 13 in Figure 7c. Additional MT profiles parallel to the existing line are required to resolve the exact location. Modelling over an MT profile data with distant stations would not allow a detailed geological interpretation. Still, we demonstrate that the MT method could map sedimentary cover and undulations in the basement. Also, the present study shows that the main differences in the depth of the conductive units of Thrace and other similar sedimentary basins can be inexpensively delineated over large areas using the MT method.

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Discussion: Determination of Atterberg Limits Using the Vane Shear Test Method [Bull. Min. Res. Exp. (2024) 174: 1–10]

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Discussion

Keywords:

Fine-Grained Soil, Liquid Limit, Plastic Limit, Vane Shear Strength, Regression Analysis.

ABSTRACT

This article presents a discussion of the original research reported in the paper by Kayabalı et al. (2024) (the Authors) that was recently published in the Bulletin of the Mineral Research and Exploration Vol. 174, pp. 1–10. Using liquid limit (LL), plastic limit (PL) and vane shear strength against water content [i.e., $s_{u(VST)}-w$] results obtained for 100 fine-grained soil samples, the Authors performed multiple regression analyses to produce four strength-based correlations for predicting the LL and PL values based on $s_{u(VST)}-w$ measurements. The Authors' dataset primarily consisted of residual soils formed through the weathering of igneous rocks, along with a few lacustrine soil samples, all sourced from the vicinity of Ankara, Türkiye. This discussion article examines the veracity of the Authors' claims regarding the predictive performance of their proposed correlations when applied to other fine-grained soils. Using a sizable independent database of dissimilar fine-grained soils compiled from the research literature, it is conclusively shown that, contrary to the Authors' assertions, their proposed correlations generally produce poor LL and PL predictions when employed beyond the calibration soil types. This outcome is not unexpected, since the Authors' data-driven correlations were deduced based on a specific dataset with limited diversity in terms of soil physico-chemical and mineralogical attributes. The article closes with a discussion of the plausible explanations for the poor applicability of the Authors' correlations when applied to dissimilar fine-grained soils.

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ABBREVIATIONS

AS	Australian Standard
ASTM	American Society for Testing and Materials
BS	British Standard
FC	Fall cone (LL testing method)
HR	Hand rolling (PL testing method)
IS	Indian Standard
LL	Liquid limit

PL	Plastic limit
RP	Rolling plate (PL testing method)
VST	Vane shear test

NOTATIONS

a	y -axis intercept of best-fitting exponential curve to $s_{u(VST)}$ against w data plot
b	Gradient of best-fitting exponential curve to $s_{u(VST)}$ against w data plot

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I_p	Plasticity index [= $LL_{(FC-BS)} - PL_{(RP)}$ or $LL_{(FC-BS)} - PL_{(HR)}$]
$LL_{(FC-BS)}$	British Standard fall-cone liquid limit
N	Number of investigated fine-grained soils
$PL_{(HR)}$	Standard hand-rolling plastic limit
$PL_{(RP)}$	ASTM rolling-plate plastic limit
$s_{u(VST)}$	Saturated remolded vane-shear strength
x, y	Exponent parameters
w	Water content
σ	Standard deviation

1. Introduction

This article presents a discussion of the original research reported in the paper by Kayabalı et al. (2024) (hereafter referred to as the Authors) that was recently published in the Bulletin of the Mineral Research and Exploration Vol. 174, pp. 1–10. Following standard testing procedures, the Authors determined the water content values corresponding to the liquid limit (LL) and plastic limit (PL) state transitions, as well as the variation in vane-shear strength $s_{u(VST)}$ with water content w , for 100 fine-grained soil samples (henceforth denoted as Dataset 1). This dataset primarily consisted of residual soils formed through the weathering of igneous rocks, along with a few lacustrine soil samples, all sourced from the vicinity of Ankara, Türkiye. Specifically, the Authors performed their LL [i.e., $LL_{(FC-BS)}$], PL [i.e., $PL_{(RP)}$] and vane shear test (VST) measurements following the British Standard (BS) fall cone (FC) method (BS 1377–2 1990), the ASTM rolling-plate (RP) device approach (ASTM D4318, 2005), and by using a miniature VST apparatus for determining the $s_{u(VST)}$ of the saturated soil specimens in accordance with ASTM D4648 (2000), respectively. Applying multiple regression analyses to their compiled database of $LL_{(FC-BS)}:PL_{(RP)}:s_{u(VST)}-w$ measurements, the Authors deduced four strength-based empirical correlations (presented as *Equations 4–7* in their paper, and reproduced herein as *Equations 1–4*) for predicting the $LL_{(FC-BS)}$ and $PL_{(RP)}$ parameters, based on either a single VST measurement (i.e., *Equations 1 and 2*), or by employing the fitting parameters (a and b) that define the $s_{u(VST)}-w$ regression relationship as $s_{u(VST)} = ae^{-bw}$ (i.e., *Equations 3 and 4*).

$$LL_{(FC-BS)} = 0.902 \times w^{0.997} \times s_{u(VST)}^{0.138} \text{ (Authors' Equation 4)} \quad (1)$$

$$PL_{(RP)} = 0.609 \times w^{0.959} \times s_{u(VST)}^{0.139} \text{ (Authors' Equation 5)} \quad (2)$$

$$LL_{(FC-BS)} = 3.62 \times a^{0.106} \times b^{-0.92} \text{ (Authors' Equation 6)} \quad (3)$$

$$PL_{(RP)} = 1.72 \times a^{0.129} \times b^{-0.91} \text{ (Authors' Equation 7)} \quad (4)$$

For the 100 soils of Dataset 1, the Authors demonstrated that their *Equations 4–7* produced good predictions of the measured $LL_{(FC-BS)}$ and $PL_{(RP)}$ values. This discussion article examines the veracity of the Authors' claims regarding the predictive performance of their proposed equations/correlations when applied to other fine-grained soils, i.e., different from the soils comprising Dataset 1. For this purpose, the Discussers employ a separate $LL_{(FC-BS)}:PL_{(RP)}:s_{u(VST)}$ dataset (hereafter referred to as Dataset 2) comprising 180 dissimilar fine-grained soils to re-examine the predictive performance/applicability of the Authors' *Equations 6 and 7*. Employing routine statistical techniques, it is demonstrated that, contrary to the Authors' assertions, their proposed correlations generally produce poor $LL_{(FC-BS)}$ and $PL_{(RP)}$ predictions when applied for fine-grained soils dissimilar to those comprising their calibration dataset (i.e., Dataset 1). This finding is not unexpected, since the Authors' data-driven correlations were deduced based on a specific dataset with limited diversity in terms of soil physico-chemical and mineralogical attributes. In other words, when considering a wide range of fine-grained soil types, the fitting parameters a and b are not unique. The article closes with a discussion of the plausible explanations for the poor/limited applicability of the Authors' *Equations 4–7* (to dissimilar fine-grained soils).

2. Critical Statistical Appraisal of the Authors' Proposed Correlations for $LL_{(FC-BS)}$ and $PL_{(RP)}$ Estimations

Table 1 summarizes Dataset 2, which comprises of $LL_{(FC-BS)}$ and $PL_{(RP)}$, and the a and b coefficients (obtained from exponential curve fitting of experimental $s_{u(VST)}-w$ results) for 180 fine-grained soils reported in two earlier investigations performed by the research group of the lead author for the paper under discussion [i.e., Kayabalı et al. (2015a, 2015b)]. Compared to the ranges of $LL_{(FC-BS)} = 29.3\text{--}117.0\%$

Table 1- Summary of Dataset 2 employed by the Discussers in assessing the predictive performance/applicability of the Authors' Equations 6 and 7.

Reference	Source in Reference	N	$LL_{(FC-BS)}$ (%)	$PL_{(RP)}$ (%)	I_p (%)	$\log a$	$b (\times 10^{-2})$	Remarks
Kayabali et al. (2015a)	Table 3 (pp. 128–129)	60	29.0–117.0	15.4–29.1	12.1–90.0	2.50–7.95	5.76–62.2	Prepared from natural soil mixed with fine sand and commercial bentonite at various dry mass ratios.
Kayabali et al. (2015b)	Table 1 (pp. 719)	120	46.0–91.0	22.0–44.0	20.0–57.0	2.41–3.12	3.50–8.50	Remolded soil samples from different regions of Türkiye.
Overall	—	180	29.3–117.0	15.4–44.0	12.1–90.0	2.41–7.95	3.50–62.2	—

Note: N = number of investigated fine-grained soils; $LL_{(FC-BS)}$ and $PL_{(RP)}$ determined in accordance with BS 1377–2 (1990) and ASTM D4318 (2000), respectively; I_p = plasticity index [= $LL_{(FC-BS)} - PL_{(RP)}$]; and $s_{u(VST)}-w$ data (used for a and b determination) obtained in accordance with ASTM D4648 (2000).

and $I_p = 12.1$ –90.0% [where I_p denotes the plasticity index, defined as the difference of $LL_{(FC-BS)}$ and $PL_{(RP)}$] for these 180 soils, the Authors' 100 soils of Dataset 1 had comparable ranges of 23.3–106.0% and 7.5–49.8%, respectively.

Figure 1 illustrates the 180 soils (of Dataset 2) plotted in the standard plasticity chart, with 132 and 48 of them classifying as *clay* and *silt*, respectively (i.e., only 27% are of *silt* type). Whereas in the case of the Authors' 100 soils (of Dataset 1), 30 and 70 of them classified as *clay* and *silt*, respectively (i.e., 70% were *silt* type). In other words, the fine-grained soils comprising Datasets 1 and 2 are quite different.

Employing the Authors' Equations 6 and 7, the Discussers calculated the values of $LL_{(FC-BS)}$ and $PL_{(RP)}$ predicted for each of the 180 soils (of Dataset 2) by inputting their reported experimental values of

a and b . Note that Kayabali et al. (2015a) reported values of a and b based on the equation $\log_{10} s_{u(VST)} = \log_{10} a - bw$ while the required form for the current analysis is $s_{u(VST)} = ae^{-bw}$ (or $\log_{10} s_{u(VST)} = \log_{10} a - bw \log_{10} e$), necessitating a conversion of the reported b parameter [in Kayabali et al. (2015a)] to $b/\log_{10} e$. Figures 2a and 2b illustrate the predicted against measured values of the $LL_{(FC-BS)}$ and $PL_{(RP)}$ parameters, respectively. The Authors reported that for their 100 soils (of Dataset 1), the overall absolute percent errors for the $LL_{(FC-BS)}$ and $PL_{(RP)}$ predictions were 6.3% and 3.9%, respectively, for the Dataset 2 soils. However, when the Authors' Equations 6 and 7 are applied to the 180 soils of Dataset 2, it is clear from Figures 2a and 2b that they are poor $LL_{(FC-BS)}$ and $PL_{(RP)}$ predictors, generally overestimating, often seriously, the measured $LL_{(FC-BS)}$ and $PL_{(RP)}$ values, with the aforementioned absolute percent errors significantly increasing to 44.8% and 60.6%, respectively. To put this in context, ASTM D4318 (2005) deems acceptable PL measurement variations of up to $\pm 3.5\%$ and $\pm 7.0\%$ (both in terms of water content) for fine-grained soils of low ($LL < 50\%$) and high ($LL \geq 50\%$) plasticity, respectively. However, for the 180 soils of Dataset 2, 74% of the $PL_{(RP)}$ values predicted by the Authors' Equation 7 differed by more than 7% water content from their measured counterparts, which is deemed unacceptable according to the ASTM D4318 (2005) standard.

Additionally, Figure 3 presents the predicted $I_p:LL_{(FC-BS)}$ data pairs [with I_p defined as the difference of the predicted $LL_{(FC-BS)}$ and $PL_{(RP)}$ values] for the 180 soils of Dataset 2 plotted in the standard plasticity chart.

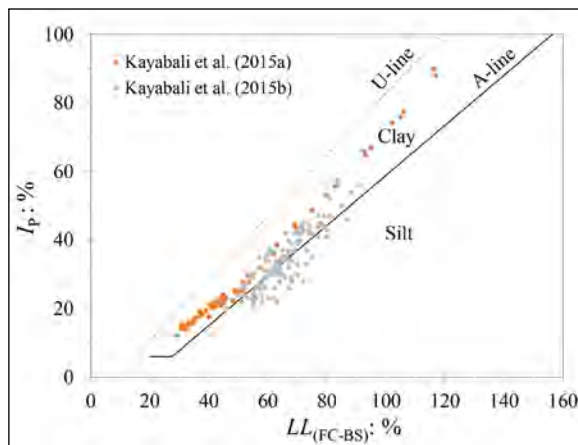


Figure 1- Dataset 2 of 180 fine-grained soils plotted in the standard plasticity chart. Note: $LL_{(FC-BS)}$ and I_p [= $LL_{(FC-BS)} - PL_{(RP)}$] are measured values.

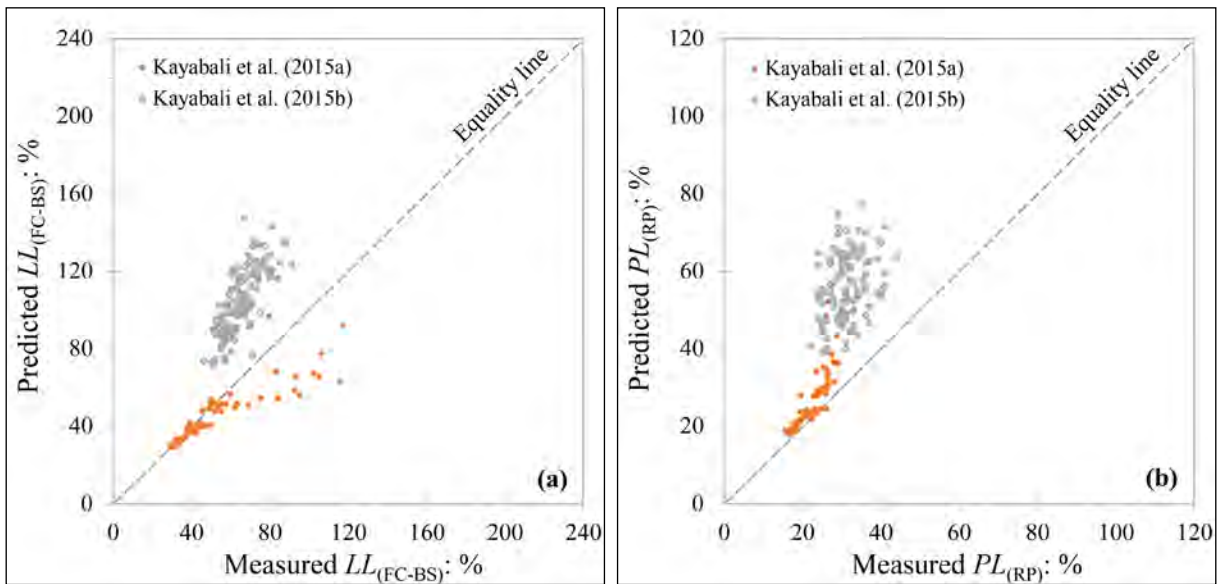


Figure 2- Predicted (by the Authors' Equations 6 and 7) against measured values of (a) $LL_{(FC-BS)}$ and (b) $PL_{(RP)}$ for the 180 fine-grained soils of Dataset 2.

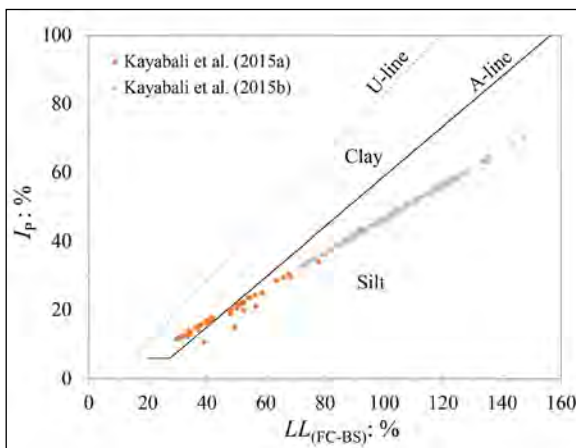


Figure 3- Predicted values of I_p against $LL_{(FC-BS)}$ (deduced using the Authors' Equations 6 and 7) for the 180 fine-grained soils of Dataset 2.

A comparison of Figures 1 and 3 clearly indicates that the predicted soil classes (based on the computed results from the Authors' Equations 6 and 7) are generally different to the measured soil classes, as determined using the standard plasticity-chart classification framework. For instance, whereas Figure 1 indicated that only 27% of the 180 soils actually classify as *silt* type (i.e., 73% being *clay*), their $I_p:LL_{(FC-BS)}$ data pairs predicted by the Authors' Equations 6 and 7 incorrectly suggest that 83% of the 180 soils should be classified as *silt* type (i.e.,

plotting below the A-Line demarcation boundary). Furthermore, as observed in Figure 3, the predicted $I_p:LL_{(FC-BS)}$ data exhibit a general linear relationship, on which the Discussers note the following. The measured data (in Figure 1) themselves exhibit a general linear $I_p:LL_{(FC-BS)}$ tendency, which naturally carries over in applying the Authors' Equation 6 and 7 to obtain the predicted $I_p:LL_{(FC-BS)}$ data. More importantly, however, the power exponents for a and b in the Authors' Equations 6 and 7 for $LL_{(FC-BS)}$ and $PL_{(RP)}$, respectively, are very close (i.e., 0.106 versus 0.129 for a , and -0.91 versus -0.92 for b). Consequently, the predicted $LL_{(FC-BS)}$ -to- I_p ratio can be estimated as follows. Given that $LL_{(FC-BS)} \approx 3.62 a^x b^y$, $PL_{(RP)} \approx 1.72 a^x b^y$ and $I_p = LL_{(FC-BS)} - PL_{(RP)} \approx 3.62 a^x b^y - 1.72 a^x b^y$, it follows that $LL_{(FC-BS)}/I_p \approx 3.62/(3.62 - 1.72) = 1.91$. This implies that the predicted $I_p:LL_{(FC-BS)}$ data approximately follow a linear trend with a slope close to $1/1.91$ (in practice, however, an intercept parameter would also exist, which would slightly adjust/modify this slope to a different value). In other words, because of the structure of the Authors' Equations 6 and 7, the predicted $LL_{(FC-BS)}$ -to- I_p ratio is expected to remain fairly constant (see Figure 3).

Considering all the above, the predictive performance of the Authors' Equations 6 and 7 when applied to other fine-grained soils (i.e., different from

the 100 soils of Dataset 1) is generally considered unreliable and therefore unacceptable for routine geotechnical engineering practice. Moreover, for all the same reasons discussed in this section, the predictive performance of the Authors' *Equations 4* and *5*, which are simplified forms of their *Equations 6* and *7*, is anticipated to be equally (if not more) unreliable.

3. Discussion on Poor Predictive Performance/ Applicability of the Authors' Proposed Correlations for $LL_{(FC-BS)}$ and $PL_{(RP)}$ Estimations

This section discusses various reasons for the poor predictive performance of the Authors' *Equations 4–7* when applied to the dissimilar fine-grained soils of Dataset 2 (compared to the soils in the Authors' Dataset 1). The first and most obvious reason is that these equations were deduced/calibrated using experimental results for residual soil samples formed through the weathering of igneous rocks, along with a few lacustrine soil samples, all sourced from the vicinity of Ankara, Türkiye. Whereas Dataset 2 concerned natural soil mixed with fine sand and commercial bentonite at various dry mass ratios, as well as remolded soils sampled from different regions of Türkiye. Accordingly, with the Authors' *Equations 4–7* deduced (optimally calibrated for) the particular soil types comprising Dataset 1, it is not unexpected that these equations produce sub-optimal predictions for the dissimilar soils (in terms of their particle-size and mineralogical compositions and hence plasticity characteristics) comprising Dataset 2. Notably, the Authors investigated mostly *silt* type soils (70%), whereas the fine-grained soils comprising Dataset 2 were mostly clay type (73%).

Additionally, as a fundamental overriding point, Atterberg's PL is defined as the water content corresponding to the plastic/ductile–brittle state transition, determined experimentally using the standard hand-rolling [i.e., $PL_{(HR)}$] or rolling-plate [i.e., $PL_{(RP)}$] methods, and deduced by a significant change in soil deformation behavior occurring for water contents either side of this state transition. It should be noted that comprehensive statistical investigations

by Soltani and O'Kelly (2021) for a database of 60 diverse fine-grained soils demonstrated that these two standard PL testing methods produce essentially similar results, with the likelihoods of $PL_{(RP)}$ underestimating and overestimating $PL_{(HR)}$ found to be 50% and 40%, respectively. Given that standard PL testing evaluates the soil's ductility properties, strength-based approaches (such as the Authors' *Equations 4–7*) are fundamentally inappropriate for making such assessments and in determining Atterberg's PL (Haigh et al., 2013; Sivakumar et al., 2016; O'Kelly et al., 2018; O'Kelly, 2021, 2024). For instance, when applied to Dataset 2, the Authors' *Equations 4* and *5* predict the values of $LL_{(FC-BS)}$ and $PL_{(RP)}$ for the 180 soils based on mean $s_{u(VST)}$ values of ~ 2.31 kPa (standard deviation of $\sigma = 0.01$ kPa) and 94.72 kPa ($\sigma = 5.96$ kPa), respectively (where the standard deviation arises from insufficient precision of the equations' fitting coefficient values). However, when considering diverse fine-grained soils, the strength mobilized at Atterberg's PL state transition has been shown to vary over a much wider range (Nagaraj et al., 2012; Haigh et al., 2013; O'Kelly, 2013). Regarding the $LL_{(FC-BS)}$ parameter, strength-based approaches are appropriate for its determination, with the BS 1377–2 (1990) FC test linking the $LL_{(FC-BS)}$ state transition to an $s_{u(VST)}$ value of ~ 1.7 kPa (O'Kelly et al., 2018; O'Kelly, 2024).

Finally, the Discussers would point out that *Equation 2* in the Author's paper (used for calculating the vane blade constant K) is not consistent with a cruciform vane used for $s_{u(VST)}$ determination. The correct form of the equation, as reported in the ASTM D4648 (2000) and BS 1377–7 (1990) standards, is given as follows:

$$K = \pi D^2 \left(\frac{H}{2} + \frac{D}{6} \right) \quad (5)$$

where H and D are the vane height and diameter (width), respectively.

For the 12.7×12.7 mm blade dimensions employed in the Authors' study (assuming a cruciform vane), the Authors' *Equation 2* would yield a K value of $3.38 \times 10^{-4} \text{ m}^3$, whereas *Equation 5* would give a value of $4.29 \times 10^{-6} \text{ m}^3$. It is not possible for the Discussers

to know whether there are typographical errors in the Authors' *Equation 2*, or whether this incorrect equation (as written) was used by them in performing their $s_{u(VST)}$ calculations.

4. Summary and Conclusions

The Authors developed their *Equations 4–7* from regression analyses of experimental results obtained for residual soil samples formed through the weathering of igneous rocks, along with a few lacustrine soil samples, all sourced from the vicinity of Ankara, Türkiye. Being calibrated for these particular soils, *Equations 4–7* gave good predictability of the investigated soils' measured $LL_{(FC-BS)}$ and $PL_{(RP)}$ parameters. However, using a sizable independent database compiled from the research literature, the Discussers have carefully demonstrated that the Authors' *Equations 4–7* generally have poor $LL_{(FC-BS)}$ and $PL_{(RP)}$ predictive performances when applied to dissimilar fine-grained soils, generally overestimating, often seriously, their measured values. Accordingly, the use of the Authors' *Equations 4–7* should be strictly limited to the calibration soils (i.e., they should not be employed for dissimilar soil types, as they generally produce unreliable predictions).

Finally, because repeatability and reproducibility remain significant challenges in PL determination using standard thread-rolling techniques [i.e., $PL_{(HR)}$ or $PL_{(RP)}$], the idea of exploring alternative, ductility-based PL measurement methods is certainly warranted. However, this need not be the case for LL determination, as standard FC testing methods offer reasonably high repeatability and reproducibility, with the added benefit of being able to establish the LL state transition by standard (e.g., IS 2720–5 1985; BS 1377–2 1990; AS 1289.3.9.2 2006) or non-standard (e.g., Kayabalı et al., 2023; O'Kelly and Soltani, 2024) single-point measurement methods. In other words, there appears to be little justification in attempting to establish the LL parameter by means of $s_{u(VST)}$ measurements.

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Reply to: “Discussion: Determination of Atterberg Limits Using the Vane Shear Test Method [Bull. Min. Res. Exp. (2024) 174: 1–10]”

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Reply

The main focus of the criticisms raised by O’Kelly and Soltani (2025) (hereafter referred to as discussers) to the authors’ article in Bulletin of Mineral Research and Exploration (174, 1-10) is to examine the veracity of the strength-based equations presented by Kayabali et al. (2024). The discussers applied the predictive equations proposed by the authors to dissimilar soils in the two articles (Kayabali et al., 2015a; Kayabali et al., 2015b) published earlier by the lead author with his research group. The discussers showed that the predictive equations 6 and 7 given by Kayabali et al. (2024) yield extremely high values of liquid limit (LL) and plastic limit (PL) when applied to dissimilar soils. Further, discussers argued that the approach proposed by Kayabali et al. (2024) led to erroneous results in determining soil classes.

First of all, authors need to emphasize that 500 sets of tests were conducted for the 100 soils examined in the article under discussion. It should be appreciated that such a voluminous data set cannot be presented in full detail in an article that is expected to be of a reasonable length. Therefore, the experimental results were presented as only a and b coefficients of vane

shear tests (VST) for space-saving considerations. The same approach was adopted by Kayabali et al. (2015a) and Kayabali et al. (2015b), where only a and b coefficients of VST were preferred for presenting test data in place of lengthy details. Among the predictive equations in article under discussion, authors’ priority of use is surely the first two ones (i.e. equations 4 and 5), since they estimate the two important Atterberg limits (namely LL and PL) with only one set of data comprising only water content and undrained shear strength from VST. Obtaining the a and b coefficients for equations 6 and 7 requires at least a few- to several sets of data for any fine-grained soil. Since the authors could not present the experimental data in full detail in the article under discussion, the discussers naturally had to use prediction equations 6 and 7 presented in the paper.

The detailed data sets produced by the lead author’s research group in the past and published in Kayabali et al. (2015a) and Kayabali et al. (2015b) were retrieved from the archive and the predictive performance of especially the equations 4 and 5 presented in Kayabali et al. (2024) were re-evaluated using those

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dissimilar soils. The data in Kayabali et al. (2015a) originally belonged to 60 soils. Approximately 300 sets of test data were produced by performing 3-7 sets of experiments for each soil comprising LL, PL, water content, and undrained shear strengths. The authors applied Equation 4 in Kayabali et al. (2024) to the data sets in Kayabali et al. (2015a). In this way, the LLs obtained for each soil by employing the predictive equation, varying in number between 3 and 7, were compared with the LLs of that soil determined through the falling cone method (Figure 1). This figure comprises only 60 pairs of LLs instead of some 300+ pairs of LLs for the sake of simplicity. That is to say, the mean value of the LLs for the 3-7 pairs of water content and undrained shear strength predicted by the strength-based equation of 4 was compared with the LL value determined by the fall-cone test. According to this figure, the prediction performance of our strength-based equation yields quite reasonable comparisons up to about LL = 60%. Liquid limits calculated by predictive equations are generally 15% higher than the experimentally found LLs. Similar steps were employed using equation 5 to PL as shown in Figure 2. A quick glimpse shows that while equation 5 in Kayabali et al. (2024) gave reasonable values when the plastic limits were low, it overpredicted PL for higher plastic limits. Plastic limits calculated by the prediction equation are generally 18% higher than those determined experimentally.

After retrieving some 600+ sets of experimental data employed by Kayabali et al. (2016b) from the archive for 120 dissimilar soils, the LLs again were predicted by Equation 4 by Kayabali et al. (2024) as shown in Figure 3. It is observed from Figure 3 that the proposed predictive equation 4 performs better for this data set. According to Figure 3, the prediction equation in our 2024 article overpredicted the liquid limit by 9% in 103 of 120 soils and underpredicted it by 4% in 17 of them. When looking at the performance of the prediction equations in predicting the plastic limit for the last data set (Figure 4), it is seen that the plastic limit is generally overpredicted by about 33%.

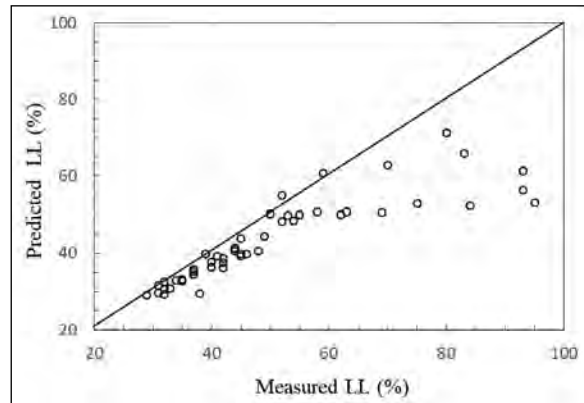


Figure 1- Comparison between the mean values of predicted LLs and experimental LLs for each soil by employing Kayabali et al. (2015a) data.

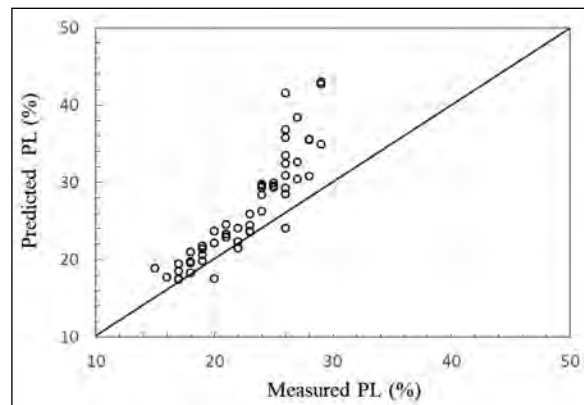


Figure 2- Comparison between the mean values of predicted PLs and experimental PLs for each soil by employing Kayabali et al. (2015a) data.

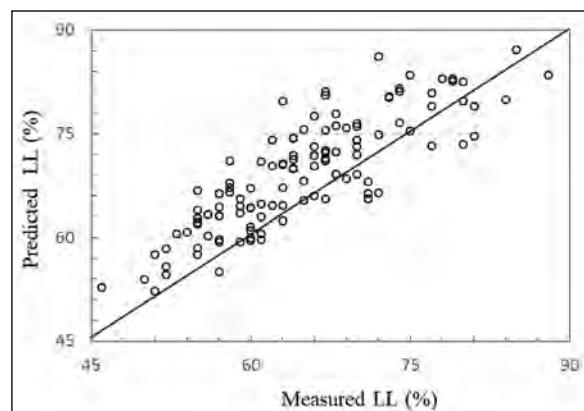


Figure 3- Comparison between the mean values of predicted LLs and experimental LLs for each soil by employing Kayabali et al. (2015b) data.

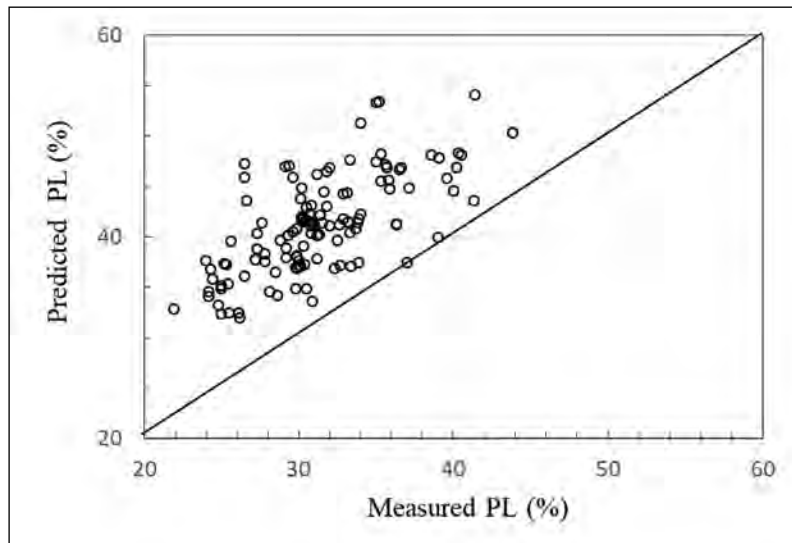


Figure 4- Comparison between the mean values of predicted PLs and experimental PLs for each soil by employing Kayabali et al. (2015b) data.

The authors acknowledge that there is a typo in Equation 2 as pointed out by the discussers. It should have been given as follows:

$$K = \frac{\pi D^2 H}{2 \times 10^6} \left[1 + \frac{D}{3H} \right] \quad (1)$$

This equation presented in the paper under discussion is given only as background information. It was not used directly in the computations. The software of the VST apparatus directly gave the undrained shear strength values.

The evaluations of the proposed predictive equations rendered within the context of this reply show that the predictive equations 4 and 5 yielded reasonably acceptable results for dissimilar soils of our research group's previous studies. It can be seen that the discrepancy in the scatter of predicted LLs and PLs in this reply gets significantly narrower compared to the scatter of the results obtained by the discussers. An important point the authors need to mention here is that the undrained shear strengths obtained by our research group in both 2015 investigations were carried out using the VST mechanism shown in Figure 5. In this device, calibrated springs are used to determine the undrained shear strength. The VST mechanism used in current article under discussion is servo-controlled and provides much more precise results. Therefore the authors are of the opinion that



Figure 5- The old-fashioned vane shear test apparatus used in the earlier investigations of the lead author and his research group.

the scatter obtained in the previous set of data that tested with our predictive equations mainly may be due to the mechanism involved in apparatus set being used. Other minor effects may be the use of unusual soils such as bentonite mixtures in experimental

studies and the fact that the operators performing the experiments are different for each of the study.

It was emphasized in a great number of earlier studies that the hand-rolling or the plate-rolling method used in determining the plastic limit is somehow irrational for being not quantitative. Because it is akin to the liquid limit, several research was performed in the past on determining the PL together with the LL by using the fall-cone method. The authors believe that research will continue for a longer time to establish the plastic limit on a more rational basis. Despite everything, the authors are of the opinion that the method used in the paper under discussion has a significant premise in determining both the liquid limit and plastic limit of fine-grained soils. So, the authors recommend prospective researchers employ a VST apparatus giving more precise undrained shear strength values and avoid the use of a spring-type VST machine for further investigations of Atterberg limits.

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- O'Kelly, B. C., Soltani, A. 2025. Discussion: Determination of Atterberg Limits Using the Vane Shear Test Method [*Bulletin of the Mineral Research and Exploration* (2024) 174: 1–10].

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- To contribute to the establishment of scientific communication issues in earth sciences both in Türkiye and internationally.
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- Corradini, C., Serpagli E. 1999. A Silurian conodont biozonation from late Llandovery to end Pridoli in Sardinia (Italy), In Serpagli (Ed.), *Studies on conodonts: Proceedings of the 7th European Conodont Symposium*. *Bollettino della Società Paleontologica Italiana* 37 (2-3) (1998), 255-273.
- Corradini, C., Corriga, M. G. 2010. Silurian and lowermost Devonian conodonts from the Passo Volaja area (Carnic Alps, Italy). *Bollettino della Società Paleontologica Italiana* 49 (3), 237-253.
- Corradini, C., Corriga, M. G. A. 2012. Pridoli – Lochkovian conodont zonation in Sardinia and the Carnic Alps: implications for a global zonation scheme. *Bulletin of Geosciences* 87 (4), 635-650.
- Corradini, C., Leone, F., Loi, A., Serpagli, E. 2001. Conodont Stratigraphy of A Highly Tectonised Silurian-Devonian Section in The San Basilio Area (Se Sardinia, Italy). *Bollettino Della Societa Paleontologica Italiana* 40 (3), 315-323, 1 Pl.
- Corradini, C., Pondrelli, M., Serventi, P., Simonetto, L. 2003. The Silurian cephalopod limestone in the Monte Cocco area (Carnic Alps, Italy): conodont biostratigraphy. *Revista Española de Micropaleontologia* 35 (3), 285-294.

Corradini, C., Corrigan, M. G., Männik, P., Schönlaub, H. P. 2015. Revised conodont stratigraphy of the Cellon section (Silurian, Carnic Alps). *Lethaia* 48 (1), 56-71.

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- All illustrations should be submitted in separate files regardless of the text.
- Abbreviations should not be used in illustration explanations in the text and should be numbered in the order of mention within the text.
- Photographs and plates must be submitted as a computer file in which all details can be seen for the examination of the article, with EPS, TIFF or JPEG extension and at least 300 dpi resolution.

6.1. Figures

- Drawings and photographs other than the plate to be included in the article are evaluated together as “Figure” and numbered in the order of mention in the text.
- The figures should be prepared in computer considering the dimensions of a single column width as 7.4 cm or double column width as 15.8 cm. The figure area with its caption should not exceed 15.8x21 cm.
- While preparing the figures, unnecessary details should not be included and care should be taken not to use more space than necessary for the transfer of information.
- In figure descriptions, a space should be left after the word “Figure” is written, and the number is given in the usual sequence number, followed by a hyphen (-) and a space again, and a description of the relevant figure should be written. If the figure legend exceeds the bottom lines, the following lines should to be written after the “Figure 1-” statement alignment. Figure descriptions should be created as follows, without exceeding the edges of the figure and justified on both sides.

For example:

Figure 1- The district of Sandıklı (Afyon); a) geological map of the southwest, b) the general vertical section of the study area (Seymen, 1981), c) Türkiye’s most important neotectonic structures (modified from Koçyiğit, 1994).

- Drawings should be drawn in computer properly, clean and with care.
- The use of thin lines that may disappear when minimized in figures should be avoided.
- Symbols or letters used in all drawings should not be less than 2 mm (7 pt.) in Times New Roman.
- All standardized symbols used in the drawings should preferably be explained in the drawing, if they are too long then they should be explained in the figure below.
- Bar scale should be used in all drawings and the north direction should be indicated on all maps.
- The name of the author, description of the figure, figure number should not be included in the drawing.

- Photographs should reflect the aims of the subject and should be in adequate numbers.
- Figures should be framed.

6.2. Plates

- Plates should be used in cases where multiple photographs are required to be printed together on a special paper.
- Plate dimensions must be equal to the size of the bulletin's usable area of the page.
- Figure numbers should be written under each of the figures on the plate and bar scale should be used.
- Original plates must be attached to the final copy to be submitted in the case of acceptance of the manuscript.
- Figures and plates should be numbered among themselves and independently. Figures should be numbered with Latin numerals and plates with Roman numerals (eg Figure 1, Plate I).
- There should be no explanation text on the figures inside the plate.

6.3. Tables

- All tables should be arranged in word format and should be prepared in Times New Roman.
- Tables should not exceed the size of 15x8 cm together with the table caption.
- Table explanations should be created without exceeding the edges of the figure and justified as in the example below.

For example:

Table 1- Hydrogeochemical analysis results of geothermal waters in the study area.

7. Nomenclature and Abbreviation

- Abbreviations must be in the accepted international or national form. Unusual nomenclature and abbreviations that are not standardized in the article should be avoided. In cases where it is deemed necessary to use such nomenclature and abbreviations, the way and method followed should be explained.
- There should not be a dot between the words initials used in standard abbreviations (such as MTA, DSİ).
- Abbreviations of geography aspects should be made in English (N, S, E, W, NE and etc.).

The word group to be abbreviated should be written clearly where it is mentioned first time and the abbreviation should be given in parentheses, then only the abbreviated form should be written throughout the article.

- Systems with international validity (m, inch, etc.) should be used as the unit of measure. Decimals should be separated with commas in Turkish articles and with a period in English articles.
- The names of figures, plates and tables in the article should not be abbreviated. For example, "As seen in the generalized stratigraphic section of the region (Figure 1)"

7.1. Chronostratigraphic and Geochronologic Nomenclature

- "International Chronostratigraphic Chart" (<https://stratigraphy.org/chart>), which is updated annually by the International Stratigraphic Committee, should be taken into consideration in chronostratigraphic and geochronological nomenclature.
- Position within a chronostratigraphic unit can be expressed in adjectives indicating the position, for example: lower, middle, upper and etc. When using these adjectives, it should be decided whether the lower, middle and upper distinction is formal / informal in the International Chronostratigraphic Chart.

For example:

lower Miocene, Upper Holocene and etc.

- When stating the time where a geochronological unit is, temporal adjectives such as; early, middle, late and etc. are used. When using these adjectives, the International Chronostratigraphic Chart should be taken into consideration to decide whether the adjectives begin with capital or lowercase letters.

For example:

early Miocene, Late Holocene etc.

7.2. Paleontological Nomenclature and Spelling of Fossil Names

- Original names of fossils should be used.

For example:

Nummulites with limestone

- Fossil genus and species names are written in italics, cf., aff. and gr. etc. expressions are written as normal (perpendicular). When writing fossil names for the first time, the surnames of the people who identify them and the year in which they were first defined should be written. In later uses, the surnames and the year in which they are defined may not be written. The surnames and dates of identifiers coming at the end of the fossil names are not references, they should not be included in the mentioned documents.

For example:

Alveolina aragonensis Hottinger, 1960 not a reference.

Alveolina cf. *aragonensis* Hottinger, 1960 not a reference.

Alveolina aff. *aragonensis* Hottinger, 1960 not a reference.

Alveolina gr. *aragonensis* Hottinger, 1960 not a reference.

- After the first use of the same genus in the text is written clearly, it can be abbreviated as in the example so that it will not be confused with another genus in later use.

For example:

Alveolina aragonensis, *A. polathensis*, *A. ellipsoidalis* etc.

- If the date is in parentheses after the person describing it after the name of the fossil in the text, this is a reference and should be included in the mentioned documents.

For example:

Alveolina aragonensis Hottinger (1960) is a reference.

- The following rules should be taken into account when writing the systematic paleontologic section.
 - a. First of all, genus, species and subspecies to be identified should be written in hierarchical order like the order, upper family, family type species and so on. Later, the species to be described should be written together with the surnames and date of the people who defined the subspecies name. If there is a photograph of the described fossil, the plate or figure with the photograph should be added under the fossil name. The names of the authors listed here are not references, so they are not included in the reference.

For example:

Order: Foraminiferida Eichwald, 1830

Superfamily: Alveolinacea Ehrenberg, 1839

Family: Alveolinidae Ehrenberg, 1839

Type Genus: *Borelis* de Montfort, 1808

Type Species: *Borelis melenoides* de Montfort, 1808
= *Nautilus melo* Fichtel and Moll, 1798

Borelis vonderschmitti (Schweighauser, 1951)

(Plate II, Figure 3-5 or Figure 3A-H).

- b. Similar or synonyms (synonym) list should be left-aligned in chronological order. The page and figure number of the synonymous fossil in the relevant study should be included in the synonymous list. Authors in the synonymous list are references and must be included in the references.

For example:

1951 *Neoalveolina vonderschmitti* **Schweighauser**, page 468, Figures 1-4.

1974 *Borelis vonderschmitti* (Schweighauser), **Hottinger**, page 67, plate 98, Figures 1-7.

- c. After the synonymous list is given, the definition, explanations (similarities and differences), dimensions, material, stratigraphic distribution (according to the characteristics of the fossil) should be written.
- d. If the fossil is defined for the first time (new species) in the systematic paleontology section, the origin of the name, holotype, type locality, material, description, explanation (similarity and differences), age and geographical distribution, dimensions (according to the features that define the fossil) should be written. Photographs of the fossil identified for the first time by the authors must necessarily be placed in plates or figures.
- e. Bar scale indicating the size of fossils must be used definitely in plates / figures.

8. References

- In the references to be made in the Main Text, only the surnames of the authors and the publication year of the mentioned article should be specified. Referencing should be arranged according to one of the following examples:
- Referring to a publication with a single author (in chronological order):

-Altınlı (1972, 1976) defined the Bilecik sandstone in detail.

It is known that the fold axes of the Devonian and Carboniferous units around İstanbul are N-S trending (Ketin, 1953, 1956; Altınlı, 1999).

- Referring to a publication with two authors:
- The upper parts of the unit include Ilerdian fossils (Sirel and Gündüz, 1976; Keskin and Turhan, 1987, 1989).
- Referring to a publication with more than two authors:
- According to Caner et al. (1975), the Alıcı formation reflects the conditions of fluvial environment.
- Unit disappears by wedging towards E (Tokay et al., 1984).
- Referring to a reference in another publication:

It is known that Lebling mentioned about the existence of Liassic around Çakraz (Lebling, 1932; Charles, 1933).

- When referring to the works of the authors with the same surname in the same year, referring the authors' first names by writing their initials:
- Many studies have been done in the field of structural geology in the study area (Gutnic et al., 1979; Yılmaz A., 1983; Yılmaz, İ., 1983; Poisson et al., 1984 etc.).

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