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An Investigation of the Chemical Composition of Bottom Sediments from Dried Lake Gavur of Turkiye by Using XRF and Multivariate Data Analysis

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Highlights

- The variations of the chemicals were found to be highly dependent on the sampling depth (0-5m).
- The age of the two samples was determined as 4752±33 BP years (2.5 m) and 5470±35 BP (5.0 m).
- The lowest weathering index values were found in the middle part of the core.
- Ca+CaO content was lower and Ti+TiO was higher in the deepest sample indicating rainy periods.
- More multidisciplinary paleogeographical research should be carried out in the region.

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Abstract

Lake sediments offer valuable information about the geographical, climatic and environmental variations. This work was carried out to examine the chemical elements of eleven sediment samples from the old bottom (0-5m; 0.5m increments) of the dried Lake Gavur in Turkiye by applying XRF along with PCA and PLSR data analyses methods. The highest and negative correlations were observed for Sr (-0.89), S+SO3 (-0.74) and Zr+ZrO2 (-0.61) with sampling depths meaning that their concentrations were lower in deeper locations (4.0-5.0 m). In contrast, Ti+TiO2 (0.60), Al+Al2O3 (0.53) and Cu+CuO (0.51) had high and positive correlations and their quantities increased as the depth increased. The age of the two samples (at 2.5 m and 5.0 m) was determined as 4752±33 BP years (2.5 m) and 5470±35 BP (5.0 m). Ca+CaO content was found as lower and Ti+TiO was higher in the deeper sediment (5.0 m) indicating rainy periods. PCA clustered the samples into two groups as deeper samples (4.0-5.0 m) and other samples (0.0-3.5m) while PLSR grouped them into three clusters as deeper (4.0-5.0 m), medium-depth (1.5-3.5 m) and shallower (0.0-1.0m) samples. PLSR model had a good performance to estimate the sampling depth from the chemical elements (R^2cal=0.95; R^2val=0.67) which means that the variations of the chemicals were highly dependent on the sampling depth. A general comment that can be obtained for the lake from the geochemical proxies is that: After a rainy period between 5.0m and 4.0m, there was a gradual drought up to 2.5m and a maximum dry period at 2.5m. It was a wet period between 2.5m and 1.0m and the second period with maximum precipitation was not as effective as seen in the first one.

1. INTRODUCTION

Lakes can be considered as geomorphological units with some crucial properties including good record keeping about climate and ecological variations [1-5]. Climate events (especially precipitation) occurring in a region are one of the most important factors affecting the chemical composition of the sedimentation in lakes and seas [6-8]. The uninterrupted chronological ordering of records from the recent and distant past with certain analyzes makes it possible to read environmental changes from the lakes and seas and their surrounding area [9-11].

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Considering the settlement history of Anatolia, the lakes, which are in an increasing pressure zone for the last 10000 years, are very close to historical and archaeological sites and other water resources. The Lake Gavur in the Kahramanmaras province of Turkiye is one of these areas and there are at least 17 known mounds in and around this province and especially the Domuztepe mound is the most famous. Gavur Lake and its surroundings have been one of the densest settlement areas of the region since the early ages of history. The Domuztepe mound is one of the best examples of this dating back 9000 years [12-16]. Lake Gavur and its surrounding has an important place in terms of paleogeographic studies because it has historical Kahramanmaras elephant (*Elephas maxima asurus*) and relict ash plant (*Fraxinus* sp.). However, this lake was intentionally dried starting from the 1950s to generate lands for agricultural production and today an intensive farming is carried out in this dried lake area.

The geochemical indicators trapped in sediment samples from seas and lakes and their time-dependent changes that can be analyzed with today's advanced technologies constitutes an essential data source about the environment in which the sample is taken [17, 18]. Data sources such as macro and micro fossils, earth materials, archaeological remains, anthropological evidences and historical records can be examined by means of X-ray fluorescence (XRF) in past environmental and climate studies. In XRF analysis, X-ray radiation is emitted and the rays interact with atoms of the chemical elements in the sample during scanning revealing the chemical composition data of the material [19]. Significant research has been conducted on XRF in various disciplines in recent years including geography, marine, lake, river, paleolimnology, etc. [20-23]. Daryin et al. (2005) investigated annual temperature and precipitation time series up to 1200 AD based on the sample cores with 1 mm precision and temporal resolution of about 1 year in Teletskoe lake in Russia [23]. Akcer On et al. (2011) examined regional climate changes based on the analysis of the core sediment samples taken from 0.6 and 4.5 m depths (with 0.2 mm increments) in Kucukcekmece Lagoon in Turkiye with XRF, fossils and dating technology [7]. Roberts (2014) [24] and Mir et al. (2022) [25] discussed the development of core scanners and the use of elements such as iron and titanium as indicators of climate change. Kylander et al. (2013) examined the regional changes during the Younger Dryas period from the Hasseldala Harbor Lake cores in Sweden reporting that geochemical proxies fill an important gap in such studies [26]. Dempster et al. (2013) examined soil formation on glacial tillers and glacial flow directions using XRF in Northern Ireland and determined that the lithological boundaries differed in terms of geochemical content and the soil was formed depending on the bedrock and was of local origin [27]. In another study, Nace et al. (2014) analyzed the changes in northern Brazil for the Late Quaternary Period with XRF data and emphasized that precipitation is largely related to Heinrich events [28]. Duesing et al. (2021) examined modern Homo sapiens with temporal and spatial climatic data by using XRF technology reporting that it is closely related to the climate history of the African continent [29].

Various techniques are employed in multivariate data analysis and chemometrics including principal component analysis (PCA) and various regression methods in the analysis of XRF sediment data. PCA is a dimension reduction and pattern recognition technique in which similar samples are grouped together [30]. It is not a classification method but useful to identify sample clusters [31]. The score plot is employed to study the similarities among the samples while the loading plot is used to identify the inter relationship of the variables. PCA is an important analysis tool used in many different fields (geology, physics, biology, archeology, agriculture, etc.) [32-35]. In case of predicting one dependent variable (Y) from a set of independent variables (X), regression methods are employed. The most three common regression methods are multiple linear regression (MLR), principal component regression (PCR) and partial least squares regressions (PLSR). Among them, the PLSR is the most advanced while MLR is not appropriate when the variables are orthogonal or have high inter correlation. In this case, PCR or PLSR are more suitable while PLSR offers better performance than PCR in most applications as a powerful alternative to PCR [30, 36].

Gavur Lake is very important in terms of paleogeographic studies, particularly, due to the fact that its surroundings have been associated with historical Kahramanmaras elephant (*Elephas maxima asurus*) and relict ash plant (*Fraxinus* sp.) [37, 38]. Recent studies showed that Gavur Lake is the best elephant fossil conservation area in Southwest Asia. Its vicinity has many mounds, such as the Domuztepe Mound, which is a place for late Neolithic settlement (6800-5450 BC). In order to evaluate the paleogeography of the lake and its surroundings more clearly, it is suggested to study the elemental analyses of core samples and radio carbon dating. The deeper cores can provide more detailed data. The region needs this kind of studies to understand the geologic past of the region. However, there has not been enough information about the paleogeography of the dried Lake Gavur and its surroundings, which is located in Kahramanmaras province, Turkiye. Thus, the aim of this study was to obtain information on the paleogeography of dried Lake Gavur and its surroundings by interpreting the XRF chemical data of the sediments taken from the old bottom (0-5m; 0.5m increments) by applying two multivariate data analysis methods of PCA and PLSR.

2. MATERIAL AND METHOD

2.1. Study Area

The dried Lake Gavur area is located near the Turkoglu district of Kahramanmaras province (Figure 1). It is at the northern end of the tectonic lakes series within the Antakya-Kahramanmaras graben, which was formed under the influence of the Dead Sea and the East Anatolian fault zones [37]. The lake was intentionally dried to create agricultural land in the 1950s but the lake becomes visible again after intense rain. The formation and evolution of the lake developed as a result of tectonic collapses and stream grabbing in the Dead Sea fault zone that developed in the Upper Miocene due to the collisions of the Anatolian and Arabian plates in the Upper Cretaceous [37-38]. As can be seen from the earthquakes (M7.8 and M7.7) that occurred on February 6, 2023 which caused more than 50000 deaths, the region is very active tectonically (Figure 2). The study area (the dried lake and the surrounding area) is in the Mediterranean and Irano-Turanian phytogeographic transition zone according to the plant regions reported by Davis (1965-1985) and it is located in the region where the Anatolian diagonal splits into two branches in the south [39]. Based on the classification of Turkiye's ecoregions by Atalay (2014), the dried lake and its surroundings are included in the Mediterranean subdivision (red pine; *Pinus brutia*) and the Mediterranean mountain division (cedar, larch, fir; *Cedrus*, *Pinus nigra*, *Abies*) [40].

According to the data from the Kahramanmaras meteorological station, which is the closest to the study area, this province receives an annual average of 709 mm precipitation while this value differs from south to north due to topographic, orographic and atmospheric reasons (Figure 1). The region has C2B3S2b3 climate class based on the Thornthwaite climate classification system with semi-humid third degree mesothermal and marine conditions and very high water deficiency in summer [37, 41]. The sampling location is shown in Figure 1 and the lithological units of the study area is presented in Figure 2.

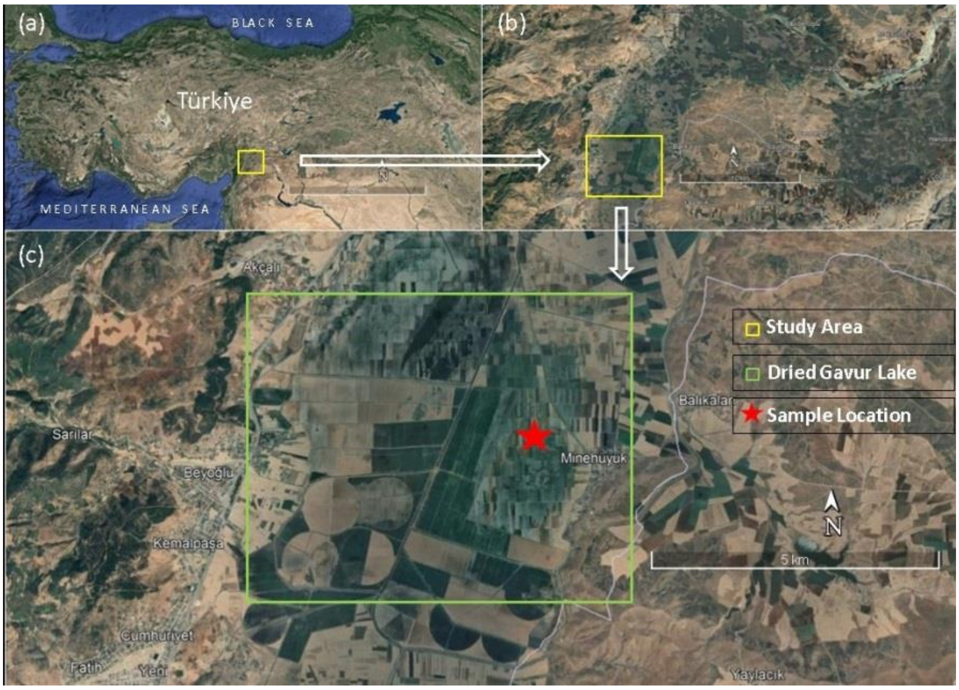

Figure 1. *The location of the Kahramanmaras province on Turkiye map (a), closer view of this province (b), the location of the dried Gavur Lake and the sampling location (c)*

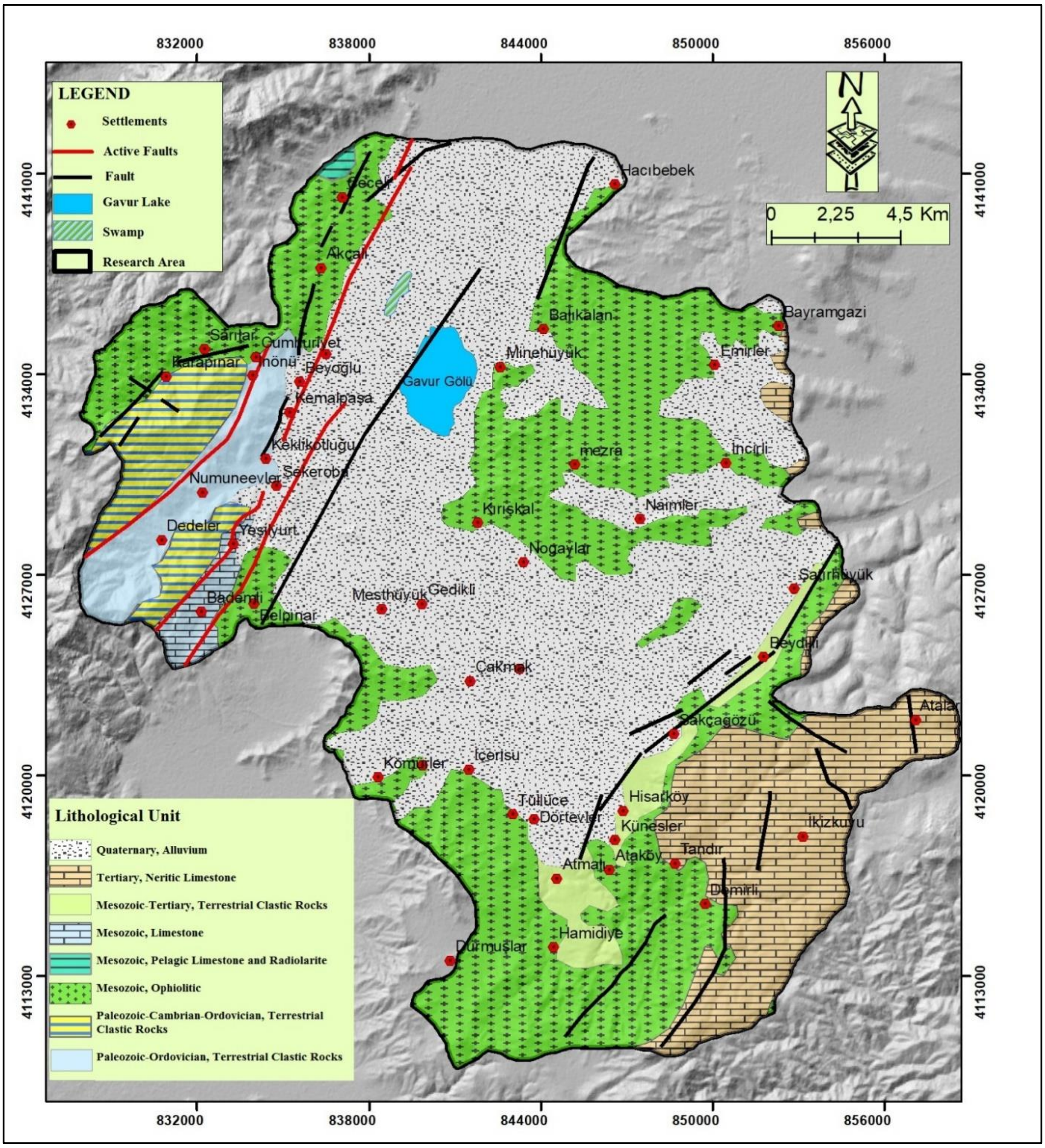

Figure 2. Lithological units map of the study area showing the Lake Gavur and its vicinity [42]

2.2. Sediment Samples

Samples were taken from the old bottom sediments at the deepest point of the dried Gavur Lake (37°17'53"N, 36°51'8"E, 482 m) by means of a hand auger type Eijkelkamp semi-degraded sediment sampler. The lake boundary was determined when the lake was at its biggest size from the aerial photographs taken in 1948 [38, 43, 44]. A total of 11 sediment samples were taken from 0-5 m depth with 0.5 m intervals.

2.3. Analytical Procedures

Analyzes of the sediment samples (n=11) taken from 11 different depths (0-5 m, 0.5m increments) were carried out using XRF (X-Ray fluorescence) for oxidized and non-oxidized chemical elements at the Center for University and Industry Collaboration of the Kahramanmaras Sutcu Imam University (KSU USKIM). Quality control of the analyzes was performed with duplicate analyses, blank samples and standard reference materials. Two sub-samples (2.5 m and 5.0 m) were also analyzed by the accelerator mass spectrometry (AMS) radiocarbon dating technique in the laboratories of TUBITAK-MAM (Marmara Research Center of the Scientific and Technological Research Council of Türkiye). The results were calibrated to calendar years with a two-standard deviation (2σ) error limit using the program OxCal v4.2 with the IntCal13 atmospheric curve [45].

2.4. Weathering Indices

Six of the most common weathering indices were also calculated separately for each measurement point (Table 1). Weathering indices provide information on the weathering history based on the chemical composition of the weathered materials [46]. Although many indices are put forward in the literature to explain the weathering, the most common six indices were selected and utilized in the present study [47]. All indices are generally based on different ratios between basic cations (K, Ca, Na, Mg) and some other cations such as Si and Al [47]. The chemical alteration index (CIA) index is a number that indicates the ratio of primary and secondary minerals present in the soil based on the process of removing basic cations such as K, Ca and Na from minerals by chemical weathering. It reflects the degree of alteration of feldspars to clays by hydrolytic weathering and indicates the amount of clay relatively [48]. Weathering indices enable inferences about soil formation processes [49, 50]. The CIA value is 100 for the samples mostly decomposed and a large amount of residual clays or minerals such as gibbsite and kaolinite in soils or sediments while its value is 50 for the undissociated top rock crust [51]. The CIW value is around 50 in non-weathered rock and 100 in heavily weathered media and increases with weathering. PIA is an index used as an alternative to CIA in quantifying the degree of decomposition. It also gives detailed information about the alteration of plagioclases. IOL is an index based on the loss of silica during weathering. It manifests itself in a process that occurs due to extreme weather conditions and is dominated by desillation [46]. WIP is used to evaluate the weathering density of silicate rocks depending on the ratio of alkali and alkaline earth elements in weathering [48]. VO index indicates the degree of the residual character of the sediment and is applicable as a residual index [52-54].

Table 1. *Most common weathering indices and their formulas used in the present study*

Weathering Index	Symbol	Formula	References
Index of Laterization	**IOL**	$IOL = 100\text{X}\left[\frac{Al_2O_3 + Fe_2O_3}{SiO_2 + Al_2O_3 + Fe_2O_3}\right]$	*Schellman (1981)* [55]
Weathering Index of Parker	**WIP**	$WIP = 100\text{X}\left[\frac{2Na_2O}{0.35} + \frac{MgO}{0.90} + \frac{2K_2O}{0.25} + \frac{CaO^*}{0.70}\right]$	*Parker (1970)* [56]
Chemical Alteration Index	**CIA**	$CIA = 100\text{X}\left[\frac{Al_2O_3}{Al_2O_3 + CaO^* + Na_2O + K_2O}\right]$	*Nesbitt and Young (1982)* [57]
Plagioclase Index of Alteration	**PIA**	$PIA = 100\text{X}\left[\frac{Al_2O_3 - K_2O}{Al_2O_3 + CaO^* + Na_2O - K_2O}\right]$	*Fedo et al (1995)* [51]
Alumina to Calcium-Sodium Oxide Ratio	**CIW**	$CIW = 100\text{X}\left[\frac{Al_2O_3}{Al_2O_3 + CaO^* + Na_2O}\right]$	*Harnois (1988)* [58]
Vogt's Residual Index	**VO**	VR= $(Al_2O_3 + K_2O)/(MgO + CaO + Na_2O)$	*Vogt (1927)* [59]*;* *Roaldset (1972)* [52]

In the literature, similar studies have been carried out in Burdur, Nigde and Antalya provinces in Turkiye and geochemical compositions have been examined [60-62]. Attention has been drawn to the increasing anthropogenic impact [63]. Similar studies have used multivariate statistical analyses for determining surface water and sediment quality for heavy elements [60], for the analysis of dam lake reservoir soils [61], for examining the geochemical contents of beach sand along the coastline [62], and to infer natural and anthropogenic origin and paleo-environment of past coastal environments [63].

2.5. Statistical Data Analysis

Chemical element contents of the sediment samples were examined by using correlation analysis in Excel spreadsheet (Microsoft Office 2016). The data were also analyzed by using principle component analysis (PCA) and partial least squares regression (PLSR) in UnScrambler multivariate statistics software (v.9.7, Camo, Oslo, Norway). In the PCA method, 17 chemical element concentrations were used as X variables. In the PLSR method, the sediment sampling depth was treated as Y variable (n=1) with the chemical elements as X variables (n=17) to predict the sampling depth from the chemical elements to study the dependency between these two factors. Full cross validation (FCV) was used in both PCA and PLSR. Since the variables had different variances and standard deviations (SD) (Table 2), the data were weighted by using the factor 1/SD with the division of each variable by its SD so that each variable has the same chance to influence the models regardless of units (percent, m) and variance [30]. The root mean square error of calibration (RMSEC), root mean square error of prediction (RMSEP), goodness of fit or coefficient of determination (R^2), scores and loadings of the models were examined in the data analysis.

3. RESULTS AND DISCUSSION

3.1. XRF Chemical Analysis Results

The basic statistical data regarding the results of the XRF analysis including 17 oxidized and non-oxidized chemical element concentrations of the 11 samples taken from the dried Gavur Lake site from 0-5 m depth with 0.5 m intervals are enlisted in Table 2. It was found that the chemical element with the highest concentration was Ca+CaO (38.5%) followed by Si+SiO (27.2%) and Fe+Fe2O3 (18.6%). These three elements constituted about 84.3% of the samples. Regarding the variability of these elements based on the coefficient of variation (CV), Zn+ZnO (68%) followed by Zr+ZrO2 (58%) and Sr (47%) had the highest variability from the surface to the depth of 5.0 m but they had very low concentrations (less than 2%). On the other hand, P+P2O5 (8.2%), Mn+MnO (9.9%) and Fe+Fe2O3 (12.0%) had the lowest variability among the 11 depths in 0-5 m.

Table 2. *Basic statistical data of the 17 oxidized and non-oxidized chemical elements obtained from XRF analysis (11 samples from the surface (0 m) to the depth of 5.0 m with an increment of 0.5 m)*

	Chemical elements	**Basic statistical data of concentration (%)**						
		Min	**Max**	**Median**	**Mean**	**SD***	**Skewness**	**CV%***
1	Ca+CaO	16.67	50.89	38.53	36.65	11.60	-0.27	31.6
2	Si+SiO	20.02	35.19	27.19	27.32	5.10	-0.02	18.7
3	Fe+Fe2O3	16.01	22.54	18.13	18.62	2.24	0.70	12.0
4	Al+Al2O3	2.87	6.28	4.73	4.73	1.14	-0.13	24.1
5	Mg+MgO	2.13	4.54	3.40	3.36	0.64	-0.14	19.1
6	K+K2O	0.75	1.73	1.21	1.23	0.34	0.07	28.0
7	Ti+TiO2	0.57	0.88	0.69	0.72	0.11	0.20	15.0
8	Ni+NiO	0.24	0.55	0.37	0.40	0.09	0.25	22.9
9	S+SO3	0.14	0.31	0.22	0.22	0.05	0.06	22.3
10	P+P2O5	0.27	0.36	0.32	0.31	0.03	-0.02	8.2
11	Cr+Cr2O3	0.15	0.32	0.24	0.23	0.05	0.11	20.9
12	Mn+MnO	0.22	0.30	0.25	0.26	0.03	0.02	9.9
13	Cu+CuO	0.86	1.72	1.23	1.24	0.23	0.45	18.6
14	Rb+Rb2O	0.54	1.02	0.82	0.81	0.16	-0.25	20.0
15	Sr	0.04	0.23	0.13	0.12	0.06	0.31	47.0
16	Zn+ZnO	0.02	1.99	1.89	1.35	0.92	-0.74	68.1
17	Zr+ZrO2	0.12	1.67	0.94	0.85	0.49	-0.10	58.2

*SD: Standard deviation; CV: Coefficient of variation calculated as “(SD/mean)x100”

3.2. Weathering Indices Results

The concentrations of proxy elements reflect the paleoenvironment and temporal changes via relative proportions [9]. In this context, XRF shows specific trends of the relevant elements, such as Ca, Al, Fe, Mg, K, Si, and Ti. The elements of Ca and CaO became major elements mostly available together. The increase in Si in the upper levels is remarkable as a result of two possible causes: Diatoms and contamination as a result of intensive farming in the immediate vicinity of the lake. Elements and proportions are also graphed in the present study (Figure 3). It was preferred to use the interpretation of the means instead of the individual value of each element. Considering the fact that resolution is 50 cm, the general comment that can be taken for the lake towards the surface from the geochemical proxies in accordance with the literature is that: After a rainy period between 5.0m and 4.0m, there was a gradual drought up to 2.5m and a maximum dry period at 2.5m. It was a wet period between 2.5m and 1.0m, and the second period with maximum precipitation was not effective as seen at first one. Geochemical indicators characterize a period of 50 cm in arid conditions and indicate a relatively wet period up to the surface.

For WIP, the highest value was at 2.5-3.0 m in the middle of the core, and the lowest value was at 4.0-5.0 m (Table 3). In the opposite, for the VO index, the highest value was at 4.0-5.0 m and the lowest value was at 3.0 m. While the CIA has the highest (0-1.0 m) at the surface, it was the lowest at 2.0-3.0 m in the middle of the core. The CIW value was the highest at the deepest locations (4.0-5.0 m) and the lowest in the middle of the core (2.5-3.0 m). While the PIA value was the highest on the surface (0-1.0 m), it was the lowest at 2.0-3.0 m in the middle of the core. While the IOL was the highest towards the end of the core (4.0 m), it was the lowest in the middle of the core (2.5-3.0 m). As a result, the lowest values of all indices except WIP were found near the middle depths of the core. In addition, the highest value was reached near the surface in CIA and PIA as the deepest value was obtained in VO, CIW and IOL. The highest value was reached in the middle section only for the WIP (Table 3).

Table 3. *Results of the weathering indices calculations according to the sampling depths*

Depth (cm)	WIP	VO	CIA	CIW	PIA	IOL
0.0	34.61	0.17	19.98	13.04	19.01	31.96
0.5	41.86	0.12	16.21	9.13	15.07	29.24
1.0	23.58	0.22	24.64	17.33	24.32	38.68
1.5	34.73	0.09	12.43	7.48	11.70	27.29
2.0	39.95	0.12	5.99	9.89	5.44	30.76
2.5	44.47	0.09	6.86	7.74	6.43	28.2
3.0	43.74	0.08	8.70	6.77	7.81	27.27
3.5	32.00	0.18	6.59	14.14	5.94	33.98
4.0	17.05	0.37	15.20	28.38	14.45	41.1
4.5	27.46	0.25	8.04	18.61	7.09	34.05
5.0	22.18	0.31	11.34	23.05	9.72	36.82

IOL: Index of laterization, WIP: Weathering index of Parker, CIA: Chemical alteration index, PIA: Plagioclase index of alteration, CIW: Alumina to calcium-sodium oxide ratio, VO: Vogt's residual index.

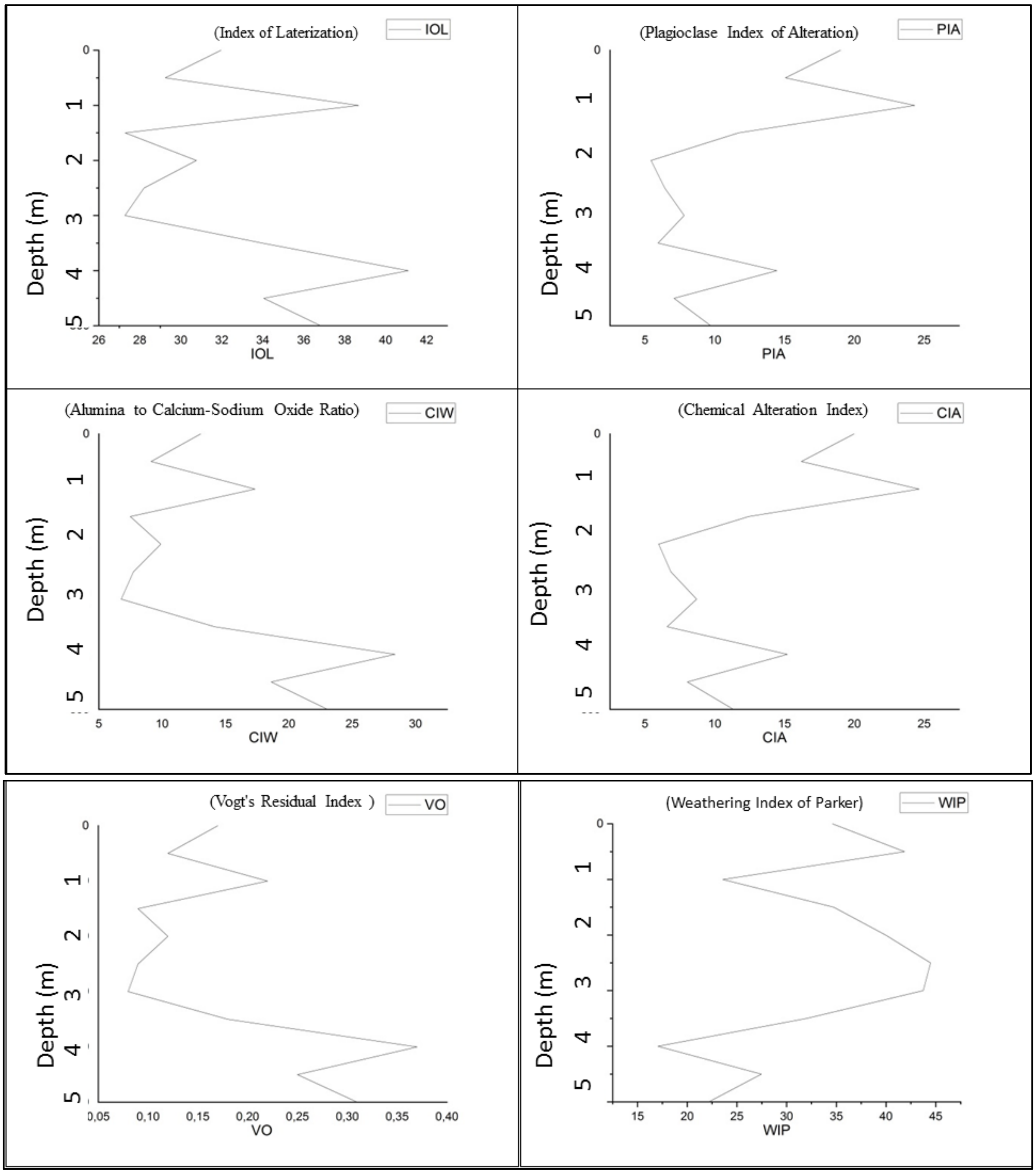

Figure 3. *Graphs of the chemical weathering indices versus sampling depths*

3.3. Lithology and the Accelerator Mass Spectrometer (AMS) Radiocarbon Dating

Based on the lithology of the studied cores, the soil texture of the sediment samples was reported as 0.0-0.6 m: topsoil, 0.6-3.0 m: clay-silt-sand and 3.0-5.0 m: clay [38]. Table 4 shows the calibrated ages of bulk organic carbon in two samples taken from the depths of 2.5 and 5.0 m as determined by the accelerator mass spectrometer (AMS) radiocarbon dating. Based on the data, a time difference of about 718 years was determined between the two samples covering a depth of 2.5 m (Table 4). The chemical compositions (%) of the two sediment samples taken at 2.5 m and 5.0 m were enlisted in Table 5. It was observed that the concentrations of Ca, S, Cr, Sr, Zn and Zr decreased while the quantities of Si, Fe, Al, Mg, K, Ti, Cu and Rb increased from 2.5 m to 5.0 m (Table 5). In particular, the sample at 2.5 m had significantly higher amounts (twice or more) of Ca+CaO (50.9%), Sr (0.13%), Zn+ZnO (1.87%) and Zr+ZrO2 (0.52%) as compared to the sample at 5.0 m (23.5%; 0.06%; 0.02%; 0.14%, respectively).

Table 4. *AMS radiocarbon ages of the two sediment samples from the dried Lake Gavur site*

Laboratory code	Sampling depths	d13C (‰)	Conventional age (BP)	2 sigma calibration (cal BC)
TUBITAK-0391	2.5 m	-26.8±1.0	4752±33	3638-3507
TUBITAK-0392	5.0 m	-27.9±1.0	5470±35	4368-4246

Table 5. *Chemical composition (%) of the two sediment samples at 2.5 m and 5.0 m*

Depth	Ca+CaO	Si+SiO	Fe+Fe2O3	Al+Al2O3	Mg+MgO	K+K2O	Ti+TiO2	Ni+NiO	S+SO3
2.5m	50.9	23.1	17.0	3.81	2.98	0.83	0.61	0.37	0.24
5.0m	23.5	32.3	18.1	6.28	3.74	1.63	0.88	0.34	0.14

Depth	P+P2O5	Cr+Cr2O3	Mn+MnO	Cu+CuO	Rb+Rb2O	Sr	Zn+ZnO	Zr+ZrO2
2.5m	0.29	0.25	0.22	1.00	0.54	0.13	1.87	0.52
5.0m	0.31	0.19	0.25	1.72	1.02	0.06	0.02	0.14

3.4. Correlation Analysis

Correlation coefficients between the sampling depths and chemical element contents were calculated and presented in Figure 4. The highest correlation was observed for Sr (-0.89), S+SO3 (-0.74) and Zr+ZrO2 (-0.61). These three elements had negative correlations meaning that they had decreasing levels from the surface to the depth of 5.0 m. In another words, their concentrations were lower in deeper sampling locations (at the depths of 4.0-5.0 m). On the other hand, Ti+TiO2 (0.60), Al+Al2O3 (0.53) and Cu+CuO (0.51) had relatively high and positive correlations meaning that they had lower quantities in the depths closer to the surface and their quantities increased as the depth increased (4.0-5.0 m). As stated above, Ca+CaO (38.5%) had the highest quantities in the samples and it had a medium level negative correlation (-0.46) (Figure 4) which means that its concentrations had a decreasing trend as the sampling depth increased. A general trend of the changes in the concentrations of the chemicals in the sediments as a function of sampling depth is depicted in Figure 5.

The following comments can be made regarding the correlation results. Strontium (Sr) was negatively correlated and its low concentration in the deeper sediments may be associated with the difficulty in dissolving and probability of salt formation. Also, Sr has been reported as related to pterobod abundance and marker of biogenic origin [8]. Sulphur (S) and Zirconium (Zr) were negatively correlated with the sampling depth and in less concentration in deeper sediments, which is thought to be related to their water solubility and ability to penetrate deeper. Sulphur (S) can be related to the presence of pyrite and bottom water-anoxia while Zirconium (Zr) is related to tephra layers [8]. It has been reported in the literature that volcanic eruptions cause changes in chemical element contents (for example: Zr) in lake sediments [64-65]. The Erciyes Volcano, located about 180 km northwest of the study area, last erupted in 253 BC and the particles may have reached the region through the general atmospheric circulation [64-65]. However, more detailed analyzes are required to obtain a conclusion. Al and Cu, on the other hand, have a positive correlation and are more abundant in deeper sediments and they are soluble in water and can penetrate deeper locations. The relation between the depth and the density of the chemical elements is mostly related to their chemical properties. Their solubility in water and the probability of forming salt appears as two important factors. High Ca content is mostly associated with dry periods while high Ti level is attributed to rainy periods in the literature [9, 50]. In the present study, Ca+CaO content was found to be lower and Ti+TiO was higher in the deeper sediments (Table 5 and Figure 5) revealing that the deeper samples (5.0 m; 5470±35 BP) point to the rainy periods as compared to the shallower sediment (2.5 m; 4752±33 BP).

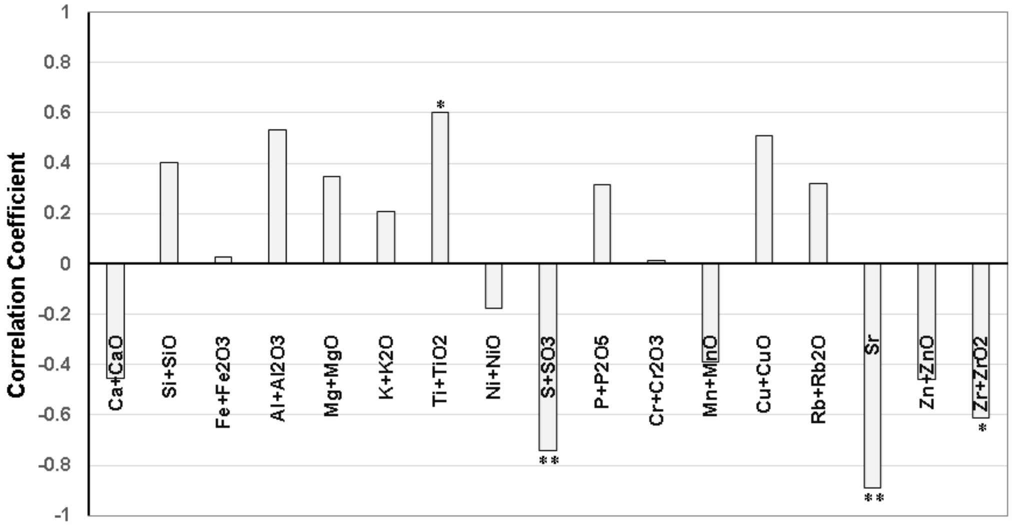

Figure 4. *Correlation coefficients between the sampling depths and chemical element concentrations of the eleven sediment samples (0-5m) (*significant at the 0.05 level, **significant at the 0.01 level)*

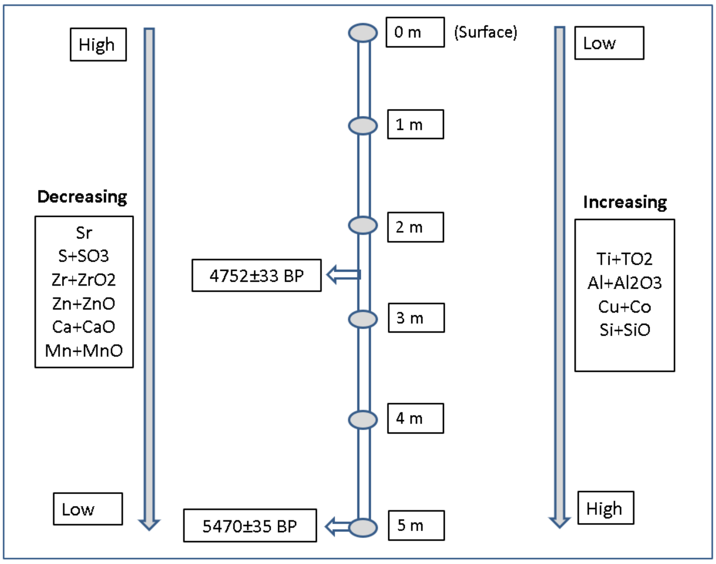

Figure 5. *Changes of the chemical elements based on the sampling depth from the surface to the bottom(0-5m)*

3.5. Principal Component Analysis (PCA) Results

PCA was carried out with 11 sediment samples from 0.0-5.0 m depth (0.5 m increments) and 17 chemical contents as X variables. The aim of the PCA was to see the similarities of the samples and study the relationship between chemical contents. The model had eight principal components (PC) explaining a total of 98% and 75% variability in X variables (chemical contents) in the calibration and full-cross validation stages, respectively. The score and loading plots from the PCA are shown in Figure 6. The first component (PC1) explained about 51% of the variability while the second component (PC2) described about 22% of the variability. The score plot shows a distinct grouping of the samples only for the ones taken from the deepest locations (4.0-4.5-5.0 m) as they positioned on the right hand side of the score graph (Figure 6). These three samples have similar chemical properties and they are different from the remaining eight sediments taken from the shallower depths (0.0-3.5m). Loading plot shows the relationship between the chemical element concentrations (X variables). Three distinct clusters can be seen in the loading plot. Mg, Si, P, Rb, Ti, Cu, Al and K have high and positive loading values on the PC1 axis while their loadings were very small on the PC2 axis. These elements have positive correlations with the deeper sampling depths (4.0-5.0 m) as they are on the right hand size of both the score plot and the loading plot. On the other hand, Zr, S, Zn, Sr and Ca have high and negative loadings on the PC1 axis. Ni, Fe, Cr and Mn have lower loading more close to zero in PC1 axis and thus, they have less importance as compared to the others in the PCA model.

Among the chemical elements, Mn had the lowest loading values close to zero in both PC1 and PC2 axis (Figure 6). The low loading value can be considered as the insignificance of this element for the model and thus can be eliminated to prevent overfitting [30]. A reduced model without Mn with remaining 16 elements revealed lower number of principal components (reduced from 8 to 5) with PC1 explaining 54% and PC2 describing 23% of variability. But the total explained variability in X variables of the reduced model (16 variables) was slightly lower in the calibration (96%) and validation (72%) as compared to the full model (with 17 variables).

In sum, the PCA showed that the samples were clustered into two groups as deeper samples (4.0-4.5-5.0 m) and other samples (0.0-3.5m) (Figure 6). The chemical elements were grouped into three groups as shown on the loading plot. The elements of Ni, Fe, Cr and Mn have lower loadings on PC1 axis meaning less importance as compared to the others.

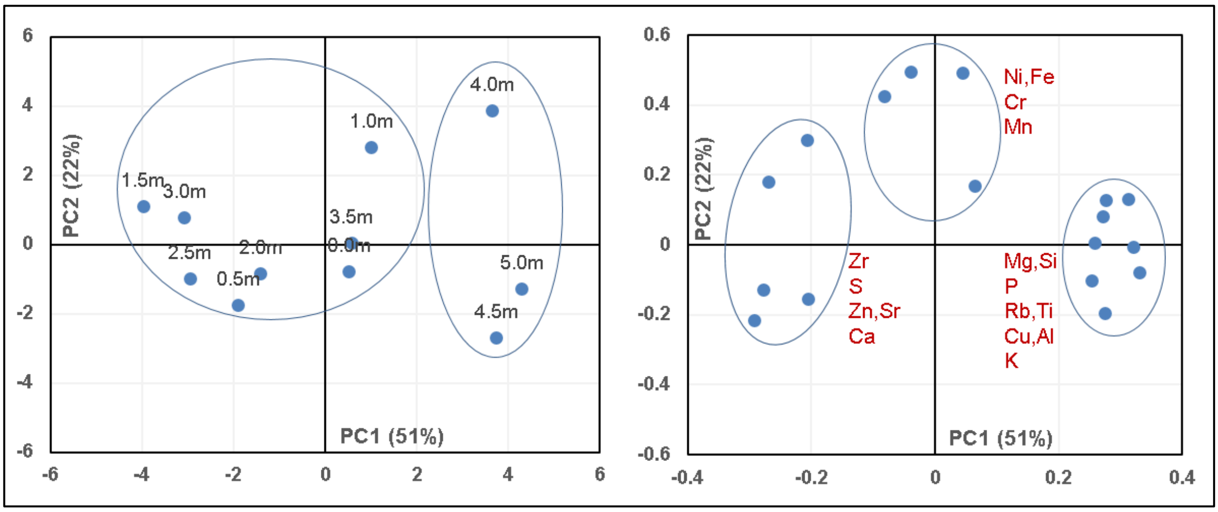

Figure 6. *Score plot (left) and loading plot (right) from the PCA model with eleven sediment samples from 0.0-5.0 m depth (0.5 m increments) and 17 chemical contents (X variables)*

3.6. Partial Least Squares Regression (PLSR) Results

PLSR was applied with 11 sediment samples from 0.0-5.0 m depth (0.5 m increments) and 17 chemical contents as X variables and sampling depth as Y variable. The aim of the PLSR was to see if it was possible to predict the sampling depth from the chemical concentrations of the sediment samples. A high predictability means a high relationship between the sampling depth and chemical composition of the samples. The PLSR model revealed three principal components (PC) explaining a total of 95% and 67% variability in Y variable (sampling depth) and a total of 78% and 50% variability in X variables (chemical contents) in the calibration and full-cross validation, respectively. The score plot and loading plot of the PLSR model are shown in Figure 7. The first component (PC1) explained about 49% (X) and 54% (Y) of the variability while the second component (PC2) described about 11% (X) and 39% (Y) of the variability (Figure 7). The score plot shows three distinct grouping of the sediments as the deeper samples (4.0-4.5-5.0 m), medium-depth samples (1.5-3.5 m) and the shallower samples (0.0-1.0 m). The deeper samples are located on the right hand side of the plot while the medium-depth samples are on the left-top of the graph and the shallower samples are located on the left-bottom side. The samples in each group have similar chemical properties. Besides, the loading plot shows the relations between the chemical element contents (X variables) and sampling depth (Y variable). Three distinct clusters can also be seen in the loading plot. Cu, Ti, Al, Rb, Mg, Si, P and K have high and positive loadings on the right hand side in PC1 axis. These elements have positive correlations with the sampling depth as they are on the same side (right hand side) of the loading plot (Figure 7). On the other hand, Ca, Zn, S, Zr and Sr have high and negative loadings on PC1. Cr, Ni, Fe, and Mn have lower loadings and thus, they have less importance as compared to the others in the PLSR model. Also, sampling depth and the elements of Ca, Zn, S, Zr and Sr are on the opposite sides of the loading plot meaning they have negative correlation. It was also seen that the PLSR model had a good performance to estimate the sampling depth from the chemical composition (R^2cal= 0.95; R^2val=0.67; RMSEC=0.37m; RMSEP=1.0m). This means that the variation of the chemicals in the sediments have a high dependency on the sampling depth. Accurate estimation of the depth and chemical element relation is important in revealing the past climatic and environmental characteristics of the basin [51]. However, taking high number of samples from more frequent depths and their dating analysis was not possible for an entire lake floor and is was very costly.

As similar to the PCA results, PLSR analysis revealed that Cr, Ni, Fe and Mn have the lowest loadings close to zero in PC1 axis (Figure 7). The low loading value can be regarded as the insignificance of these variables for the model and hence they can be eliminated to prevent overfitting [30]. A reduced PLSR model without these four chemicals with remaining 13 elements had four PCs explaining a total variability of 99% and 77% of the Y variable and 87% and 50% of the X variable for the calibration and validation, respectively which is higher than the full model with all 17 X variables. The prediction performance was also higher with the reduced model (R^2cal= 0.99; R^2val=0.77; RMSEC=0.19m; RMSEP=0.84m) as compared to the full model.

In sum, the PLSR showed that the samples were clustered into three groups as deeper (4.0-4.5-5.0 m), medium-depth (1.5-3.5 m) and shallower samples (0.0-1.0m) (Figure 7). The chemical elements were also clustered into three groups as shown on the loading plot. The elements of Ni, Fe, Cr and Mn had lower loadings on PC1 axis meaning less importance as compared to the others. PLSR model had a good performance to predict the sampling depth from the chemical composition data (R^2cal= 0.95; R^2val=0.67; RMSEC=0.37m; RMSEP=1.0m) which means that the variation of the chemicals in the sediments have a high dependency on the sampling depth.

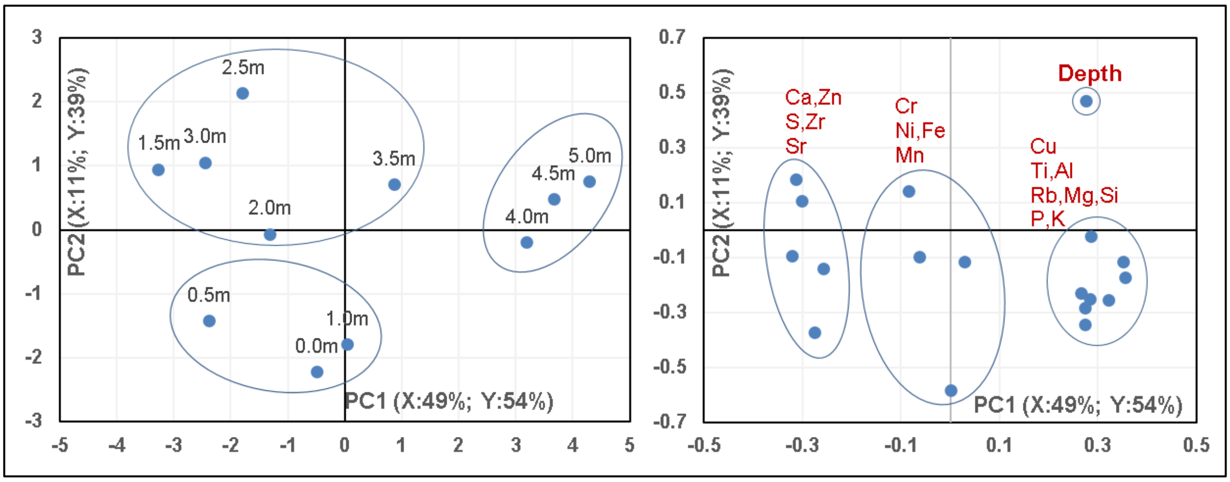

Figure 7. *Score plot (left) and loading plot (right) from the PLSR model with eleven sediment samples from 0.0-5.0 m depth (0.5 increments), 17 chemical contents (X variables) and sampling depth (Y variable)*

4. DISCUSSIONS

Valuable information can be acquired from lake and sea sediments about the geographical, climatic and environmental changes related to the surrounding area [3, 9]. Analysis of chemical elements, pollen and archeological findings are usually used for this purpose covering the area around a lake [52]. Climatic events, primarily precipitations, are crucial influencers of the chemical composition of the sediments [7-9].

It was emphasized that the use XRF offers time and cost savings in paleoenvironmental studies and can also be used to assess pollutants [51, 53]. Sun et al. (2008) analyzed the processes affecting the geochemical composition of sediments from the South China Sea, compared the data with cave sediment records and reported that the findings were closely related to regional and global climate change [54]. Evans et al. (2019) utilized XRF and PCA for the samples of a core from Kanono Lake in New Zealand in a dataset covering 2230 years and stated that precipitation was associated with oscillations such as El Nino southern oscillation (ENSO) [66]. Topuz (2019) stated that the density of arboreal species decreased as the time approached the present day and herbaceous species increased due to human influence by reporting the presence of pollens of beech (*Fagus*), chestnut (*Castanea*), hornbeam (*Carpinus*), elm (*Ulmus*), and hazelnut (*Corylus*) trees around the Lake Gavur area [42].

Chemical analysis of the sediments from different depths can lead to information about the history of the geological and environmental changes of the nearby basin area [9, 11]. Hence, the current research examined the chemical elements of the eleven sediment samples from the old bottom (0-5m; 0.5m increments) of the dried Lake Gavur by applying XRF along with PCA and PLSR multivariate data analysis techniques.

It is mentioned that Strontium (Sr) was related to pterobod abundance and marker of biogenic origin [8]. In the current study, Sr had lower concentrations in the deeper sediments that may be related to the difficulty in dissolving and probability of salt formation. The same authors [8] also stated that Sulphur (S) can be related to the presence of pyrite and bottom water-anoxia while Zirconium (Zr) is related to tephra layers. In the present study, both elements were negatively correlated with the sampling depth with lower quantities in deeper sediments.

It was reported that high Ca content is mainly associated with dry periods while high Ti is related to rainy periods [9]. In the present study, the ages of two samples from 2.5 and 5.0 m depths were determined as 4752±33 BP years (2.5 m) and 5470±35 BP (5.0 m). Ca+CaO content was found to be lower and Ti+TiO was higher in the deeper sediments revealing that the deeper sample (5.0 m) indicate rainy periods as compared to the shallower sediment (2.5 m). It was also seen that the sample at 2.5 m had significantly higher amounts (twice or more) of Ca+CaO, Sr, Zn+ZnO and Zr+ZrO2 as compared to the sample at 5.0 m. However, temporal resolution was lower in the current study with only two samples at 2.5 and 5.0 m to reveal detailed short-term variations in climate since it can take long times for a chemical element to reach and gravitate in the lake bottom. Still, the data can give some basic information about climatic variations. This finding is consistent with the findings of the pollen study conducted in the same sediment samples [52].

PCA is a common method used in many diverse research areas including geochemical analysis. PCA showed in the present study that the samples were clustered into two groups as deeper samples (4.0-5.0 m) and other samples (0.0-3.5m) while the chemical elements were grouped into three clusters. The PCA model with first two components explained about 73% of the total variability. Similarly, Schreiber et al. (2014) used PCA on 13 variables of XRF core scanning (0-80cm) from a lake in the center of Copenhagen and reported a distinct trend over sampling depth with a model with two or three PCs explaining about 80% of the total variability. The clustering of chemical elements is an important indicator in terms of geochemistry [18]. The elements of Ni, Fe, Cr and Mn had lower loading values meaning less importance as compared to the other elements.

PLSR was also applied in the current study and it showed that the samples were clustered into three groups as deeper samples (4.0-4.5-5.0 m), medium-depth samples (1.5-3.5 m) and shallower samples (0.0-1.0m). Similar to the PCA results, the chemical elements were also grouped into three groups while Ni, Fe, Cr and Mn had lower loadings indicating less importance as compared to the other elements. Furthermore, PLSR model had a good performance to estimate the sampling depth from the chemical composition data (R^2cal= 0.95; R^2val=0.67; RMSEC=0.37m; RMSEP=1.0m) which means that the variation of the chemicals had a high dependency on the sampling depth.

Some researchers used chemical analysis of sediments from lakes or seas to study environmental pollution especially with heavy metals as a result of natural and/or anthropogenic factors [56]. It was stated that heavy metal availability in sediment samples (Cu and Ni) is an indication of human activities and the use of fertilizer [57]. In the present study, among the heavy metals, Cu+CuO had the highest but medium-level and positive correlation (0.51) with sampling depth (Figure 4) which means that its quantity was higher at deeper sediments while Zn+ZnO (0.46) and Mn+MnO (0.39) had slightly lower but negative correlation indicating their contents were higher in shallower sampling locations. On the other hand, Ni+NiO (0.18), Fe+Fe2O3 (0.03) and Cr+Cr2O3 (0.00) had much lower or no correlations.

Chemical weathering indices were also used in the present study. WIP is divided into certain classes in the literature (Koralay et al., 2014). Lower WIP values indicate stronger chemical weathering, in contrast to the way CIA values are generated [48]. CIA values increase in parallel with the increase in clay minerals in the rock [48]. High LOI values indicate relatively more segregation [48].

5. CONCLUSIONS

This research examined the variations of chemical elements of eleven sediment samples from the old bottom (0-5m; 0.5m increments) of the dried Gavur Lake, Turkiye by applying XRF technique along with PCA and PLSR multivariate data analyses.

The highest correlation coefficients were observed for the elements of Sr (-0.89), S+SO3 (-0.74) and Zr+ZrO2 (-0.61) against the sampling depth. These three elements had negative correlations meaning that they had decreasing levels from the surface to the depth of 5.0 m and their concentrations were lower in deeper sampling locations. PCA indicated that the samples were clustered into two groups as deeper samples (4.0-4.5-5.0 m) and other remaining samples (0.0-3.5m). On the other hand, PLSR analysis showed that the samples were clustered into three groups as deeper samples (4.0-4.5-5.0 m), medium-depth samples (1.5-3.5 m) and shallower samples (0.0-1.0m). Similar to the PCA results, the chemical elements were also grouped into three groups while Ni, Fe, Cr and Mn had lower loadings indicating less importance as compared to the other elements. PLSR model had a good performance to estimate the sampling depth from the chemical composition data (R^2cal= 0.95; R^2val=0.67; RMSEC=0.37m; RMSEP=1.0m) which means that the variation of the chemicals had a high dependency on the sampling depth.

The age of two samples from 2.5 and 5.0 m depths was determined as 4752±33 BP years (2.5 m) and 5470±35 BP (5.0 m). Ca+CaO content was found to be lower and Ti+TiO was higher in the deeper sediments revealing that the deeper sample (5.0 m) indicate rainy periods as compared to the shallower sediment (2.5 m).

The weathering indices were also examined in 11 depths with the help of the chronology obtained from the age-depth curve. The area, which is supported by archaeological findings in the literature, has been the scene of a dense settlement since the past (9000 years ago) and the disappearance of the *Elephas Maxima Asurus* is related to these changes in the lake and its surroundings. The differentiation that took place in 3800 AD, excluding WIP, corresponds to the extinction of the *Elephas Maxima Asurus*. This might be the chemical weathering properties of the elephants as a result of pollution in the lake.

It is thought that the results of this research can provide a data source for future studies on dried Lake Gavur and similar lakes in the region in the context of paleogeography studies. Regional paleo changes, especially at times corresponding to the boundary levels of sampling depth groups, can be examined in more detail through multidisciplinary context with historical, archaeological, geochemical, fossil pollen and other proxies. This will contribute greatly to the better definition of the paleogeography of the region. In order to evaluate the paleogeography of the lake and its surroundings with more detail, it is suggested to take more core samples and make more radio carbon dating. It is thought that the deeper cores can provide clearer results for this region in terms of paleogeography.

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CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

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