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Research Article

Forensic comparison of Soil Samples in Ömerli Dam Region by FTIR and ICP-OES

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ABSTRACT

Soil is a one of the important physical evidence that could be encountered in any objects including car tire, shoes, or clothes. This study presents the comparison of the soils in Ömerli Dam, Istanbul-Türkiye. 35 of soil samples from 7 different regions have been collected around the lake. FTIR and ICP-OES have been utilized for structural analysis and detection of metal contect in soil samples. Moreover, textural analysis, pH, sedimentation, color tests have been systematically investigated. The image of soil samples is also captured under different light of sources by VSC 8000. The outcome shows that discrimination of soil samples could be studied using various techniques including FTIR, textural analysis, metal concentrations and their color. ICP-OES analysis showed that the soil have various elements such as Si, Fe, Mg, Ca, Cu with a different amounts which provide discrimination for soil samples. The natural pH of the samples varied between 6.1 and 8.3 that are from slightly acidic to moderately alkaline character.

1. Introduction

Soil is a complex mixture that containing organic, inorganic and biological components in itself [1]. The characteristic feature of soils is generally affected by the natural events as a result of earth crust movement, rains, climate conditions and human made effect including industrialization, population growth, agriculture and livestock activities and so on [2]. Pedology which came from Greek words pedon- as soil and -logos as study refers to formation, classification, mapping and characterization of soil samples [3]. Due to the variance of samples as a result of natural and artificial dissimilarities, soil is considered as important evidence which can be transferred one environment to another when two surfaces come into physical contact [4].
Soil can be in the form of dust and mud on the clothes of individuals, as well as on different surfaces such as the soles of shoes, burial sites, cars and tires. Therefore, it is encountered on crime scene which provide investigators to valuable evidence [5]. Detection of soil character could be the key information about spatial location and suspect's activities [6]. The first exploit of soil samples as forensic evidence is carried out by Georg Popp in 1904 who used soil on suspect's shoe to support the theory of the crime in the murder of Margarethe Filber [7].

Organic, inorganic and biological changes in the soil provide distinctive information about agricultural activities, population density and industrialization, poor management or inefficient disposal of waste in the region [6].
For instance, some synthetic chemicals such as pesticides could be found in the regions in which agricultural activities are carried out [8]. The percentage of dyes, polyaromatic hydrocarbons (PAHs) and heavy metals could be detected in industrial dense areas [9].In addition, as a result

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of natural processes such as the formation of the world, etc., regional differences may occur in the soil [10]. The oil accidents, asbestos from destroyed old buildings, gold mining operations, volcanic eruptions, agricultural irrigation, sewage systems are some significative natural and artificial parameters in terms of discriminative characteristics of soil samples [11]. Stern et al. examined the soil profiles of United States which have been collected over 20.000 fractions from 7534 sites [12].

It was demonstrated that soil mineral rarity could be important evidence in order to discriminate the soil samples from a region. Testoni et al. have collected various soil samples from different sites to develop and test a Standard Operating Procedure (SOP) for forensic soil sampling [13]. It was found that most of the samples were correctly grouped according to their location by using chemical characterization methods. Dong et al. have discriminated the soil samples according to their color by handheld spectrophotometer [14]. 0.02-0.04 g of fine soil samples was found to be enough for the accurate color determination by instrumental colorimetric equipment.

Discrimination of soils offers many benefits in forensic investigation because it can transfer from many materials, easy to storage, durable, may vary regional differences [15]. Forensic soil specimens could be discriminated according to their mineralogy, textural features, grain size, color, chemical composition, organic matter and so on [16, 17]. Therefore, it is very crucial to determine their divergent features using different characterization techniques. Distinctive features of soil as forensic evidence could be carried out by different methods in terms of color, pH of soil, the chemical and biological compositions, density, particle size [18]. The characterization of the soil evidence can be carried out by X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Thermal gravimetric analysis (TGA), Scanning electron microscope (SEM), Transmission electron microscope (TEM), Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), Inductively Coupled Plasma Mass Spectroscopy (ICP-MS), Atomic Absorption Spectroscopy (AAS) [19].

For instance, decayed human remains can change the chemical structure of the soil by the emergence of various chemicals such as indole, carbon dioxide, hydrogen sulfide, ammonia. The determination of this change in the soil is of critical importance in terms of forensic events [20]. Focant et al. investigated the chemical changes in soil samples containing cadavers using two-dimensional gas chromatography time-of-flight mass spectrometry (GC/GC-TOF-MS). They have found that more than 20 specific compounds such as nitrile, ether, ketone, alcohol, aldehyde have been detected at different depths of the soil in the burial area [21].

In 2021, Chauhan et al. have used UV-Vis-NIR spectroscopy to discriminate the soil specimens which is collected from India. The have found the presence of some organic minerals in varying amounts [22]. Pirrie et al. have tested recovering soil trace evidence by using SEM-EDS techniques [23]. The study, published in 2020 by Sangwan et al., conducted many analyzes on soil. In this study, ATR-FTIR, pyGC-MS, SEM-EDX, ICP-MS/OES and XRD analyzes were also performed on the prepared soil samples, apart from physical analyzes such as determining the color, particle size and density of soil solutions [18]. Another study published by Marumo in 2003 revealed similar results and additionally the organic structure and mineral content of the soil were determined. In addition to these results, macroscopic and microscopic imaging of the soil content was also performed [24].

In another study published in 2014, in which soil was considered as trace evidence, ATR-FTIR spectroscopy was used to determine the organic and inorganic compounds of the soil. Soil samples, which were thoroughly crushed and homogenized, were separated according to their color using the Munsell chart, apart from spectroscopic methods [25, 26]. Dawson and Hillier's study in 2010 covered more than 70 soil samples, making it one of the publications that generated the largest sample group. In the study, besides the Energy dispersive spectroscopy (EDS) images, SEM images were also taken, and the inorganic compounds of the soil samples were determined. The structure of soil samples was tried to be fully elucidated by applying X-ray fluorescence (XRF), atomic absorption spectroscopy (AAS), inductively coupled plasma (ICP) spectrometry, neutron activation analysis (NAA) and energy and wavelength dispersive X-ray (EDX and WDX) microanalysis [20]. The relevant section of the book, published by Fitzpatrick et al. in 2009, on soil investigations for forensic purposes, includes detailed physical analyses. SEM, XRF, ICP-MS, DSC, NMR analyzes were performed, especially IR spectroscopy and XRD analyzes, on soil samples separated according to particle size. The pH values of the soil samples strengthened with examples from real events were one of the first factors to be evaluated before all these analyzes [27].

The study which conducted in 1999 by Turner and Wiltshire examined changes in the soil after pig samples were buried in the ground for five months [28]. In this publication, the effect of soil acidity on the buried material after burial in an acidic pH area was evaluated. Although a limited region was chosen as the region, the study showing the change experienced by organic materials due to waiting in the soil has similar features to our current study.

Among these techniques; FTIR come to forefront to discriminate the distinguishing feature of unknown materials because it offers multiple advantage such as rapid, easy to use, portable, do not require further any pretreatment, suitable for different specimens including solid, liquid, paste, powders, films and even gases [29]. Xu et al. Investigated the soil samples from different regions of China by laser-induced breakdown spectroscopy (LIBS) and Fourier transform infrared total attenuated reflectance spectroscopy (FTIR-ATR). They have concluded that obtained soil samples can be easily separated according to their characteristic's differences including elemental, mineral and organic matter compositions via LIBS and FTIR-ATR [17].

This paper aims to characterize and discriminate the physical, chemical characteristics of different soil samples in which they are collected from Ömerli Dam which is one of the most important drinking water sources in the north-east of Istanbul.

The color of soil samples is determined according to the Munsell soil color chart. The structural analysis of soil samples is achieved by Fourier transform infrared spectroscopy (FTIR). The soils also classified according to the pH value of the soil suspensions and ranges between 6.1-8.3 for all regions. VSC 8000 are used to visualization of soil specimens under different light of sources.

Some heavy metals such as are studied by inductively coupled plasma optical emission spectrometry (ICP-EOS). The outlines show that FTIR is effective method to find out distinctive features in terms of forensic investigations. Moreover, the sedimentation character and textural analysis demonstrated that soil samples could be easily separated from each other which make it effective, cheap, and quick options for real crime scene.

Thanks to this study, a region that one of the important part of water source in Istanbul have been systematically explored by using FTIR, soil color test, textural analysis, ph and elemental analysis to discrminate the soil character in Ömerli Dam which provide significant data in crmine scene investigation.

2. Material and Methods

2.1. Study area

Ömerli Dam is located in the north-east (approximately 30 km) of Istanbul and provides about 43% drinking water requirements of İstanbul which is engaged between 1968 and 1972. The map of region and places where the samples were taken are demonstrated in Figure 1. Google maps have been used to provide a location where soil samples were collected. All soil samples are taken on 5 November 2023. Before sampling, 1*1 m^2 of sampling area is determined. Then, four corner and one center of sampling area is excavated.

The coordinates of the collected soil samples was found to be 41°00'47.6"N 29°17'34.8"E, 40°57'59.8"N 29°20'56.3"E, 41°01'10.1"N 29°21'55.0"E, 41°01'24.4"N 29°22'39.1"E, 41°00'37.8"N 29°24'51.5"E, 41°03'49.8"N

29°21'30.9"E, and 41°04'29.0"N 29°23'20.8"E for S1, S2, S3, S4, S5, S6 and S7, respectively

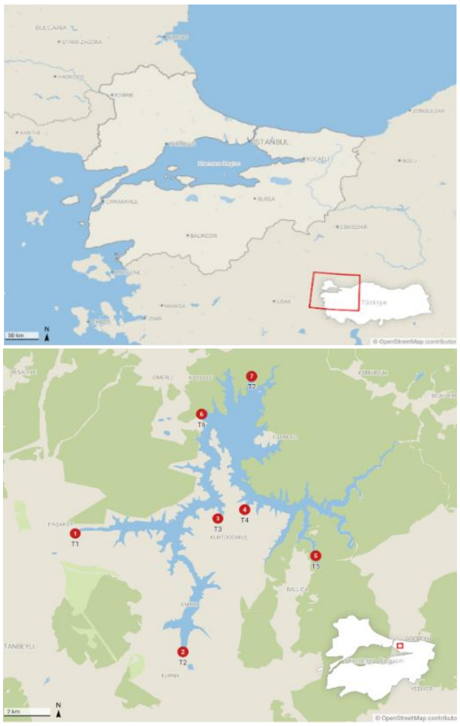

Figure 1. The map of A) Marmara region, Türkiye B) Ömerli Dam in Istanbul

2.2. Sampling

A total of 35 soil samples from 7 region were collected from Ömerli Dam in autumn Figure 2. The samples are collected from 1 meter from the shore at a depth of 20 cm. 5 samples are collected within 1 square meter area (4 corner and 1 center) to get a homogeneous sampling. The collected samples were oven dried for 48 h at 100 ºC then large structural such as rocks, leaves, twigs are gently separated from the soil samples. Then, the soil samples are gently grounded and filtered using <50 micron mesh sieve and then stored in a desiccator.

2.3. Color

The color of soil samples is detected according to the Munsell color system. Soil samples are wetted by spraying of water in order to be moist. White copy paper (A4 paper) was used as background for accurate reading of soil samples. To make a reliable color comparison, some parameter should be taken into consideration including soil comparison should be done under sunlight without using any sunglasses, a white ground should be used, and the soil should be moist. For comparison of color, center of soil samples have been examined.

Figure 1. The images of Omerli Dam region from different perspective

2.4. Textural analysis

Soil textures provides an information to proportion of sand, clay and silt particles. Different textures could be important evidence for the determination of the samples. For this, textural analysis of soil samples as forensic evidence has been investigated. The jar test method has been applied to S1, S6 and S7 samples for determining of silt, sand and rock compositions of samples. Briefly, a known amount of specimen has been added to the jar and water is added up to half of jar. 3 g of dishwashing detergent is added to the jar and the mixture was then vortex for 1 min. The sand, clay and silt compositions have been determined according to the textural triangle [30, 31].

2.5. Gravitational sedimentation of soil

Soil samples consist of sand, clay, silt, organic matter and so on. Depending on the composition, the soil textures can vary [12]. The sedimentation methods give an information on the analysis of gravitational settling of particles in a fluid. To compare the particle size distribution of soil samples, 5% of soil in water (g/mL) is prepared. the suspension first shaken and vortex for 1 min. After that, the suspensions was left tube holder to be rest of suspensions and allowed to sediment against time. The images are captured at different time of intervals (from 1 min to 24 h) in order to observe the color of suspensions.

2.6. Instrumentation

The color of soil samples is classified according to the Munsell soil color chart. The color comparison was held on 19 December 2023 in the middle of the day, in clear weather and natural sunlight. The concentrations of selected elements were determined using Inductively coupled plasma atomic emission spectroscopy (ICP-OES, Thermo Fisher Scientific, Bremen, Germany).

An Attenuated Total Reflection (ATR)-Fourier Transform Infrared Spectroscopy (FTIR) was used to analyze structural characterization of soil specimens. Instrument settings used were 128 scans; 16 cm^{-1} resolution; range 4000-650 cm^{-1}. FTIR results is the average of 5 soil specimens. After analysis, the crystal was cleaned with ethanol. Video spectral comparator (VSC) 8000 have been used as light source which provide soil images in the different light sources including visible light, ultraviolet light and spot (fluorescence).

Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES) was used for quantifying of metals in soils. For preparation, a known amount (approximately 0.2 g) of soil sample which is used center of the soil samples is placed in vessel. Then, 8 mL of HNO_3 and 2 mL of HCI is added. The samples are microwaved for digestion (temperature program: heat to 200 ^{o}C in 15 min and hold to 200 ^{o}C for 15 min and cold to room temperature in 15 min). After digestion, samples in vessels are diluted to 50 mL by using ultrapure water.

3. Result and Discussion

3.1. The color of soil samples

The utilization of Munsell color investigation give a valuable information which enable visual comparison and discrimination of soil samples by investigators [32]. However, some parameters including moisture percentage of soils, lighting, ground color should be considered in order to get a comparable result. For this, a white A4 paper is used for background and the soils are wetted by spraying to get moist samples. According to the Munsell color system, the color of soil samples is found to be 7.5 YR 4/4 (brown) for S1, 5 YR 4/4 (reddish brown) for S2, 7.5 YR 4/6 (strong brown) for S3, 10 YR 4/6 (red) for S4, 2.5 YR 3/4 (dark reddish brown) for S5, 7.5 YR 4/4 (brown) for S6, 5 YR 4/3 (reddish brown) for S7 Table 1.

Table 1. The color characteristics of soil samples

Sample	Color	Color name
S1	7.5YR 4/4	Brown
S2	5YR 4/4	Reddish brown
S3	7.5YR 4/6	Strong brown
S4	10YR 4/6	Red
S5	2.5YR 3/4	Dark reddish brown
S6	7.5YR 4/4	Brown
S7	5YR 4/3	Reddish brown

3.2. Soil images

Video Spectral Comparator 8000 (VSC-8000) was used to visualize the soil samples under different of light sources including visible light, ultraviolet, spot (fluorosence). Soil samples could be discriminated under visible light which confirms the Munsell color test, however, there was no difference under other light sources, even some organic particles could be seen under spot (fluorosence) which shines under fluorosence. The images of soil samples are shown in Figure 3.

3.3. FTIR

A total of 35 samples from 7 different region in the Ömerli Dam were analyzed by FTIR. Sieved and dried samples were used for FTIR analysis. In order to get homogeneity and repeatability, five different spectra are collected and compared which is given in Figure 4. The FTIR spectra of 7 different regions in the Ömerli Dam was demonstrated in Figure 1. According to the spectra, the peaks between 3700-3620 cm^{-1} represent the presence of kaolin mineral which can be especially seen S1-S5 samples. The bands at 1645 shows the C=O streching of amides, and nitrate at 1380 cm^{-1}. The peaks between 3000-2800 cm^{-1} shows the aliphatic methyl and methylene groups [33].

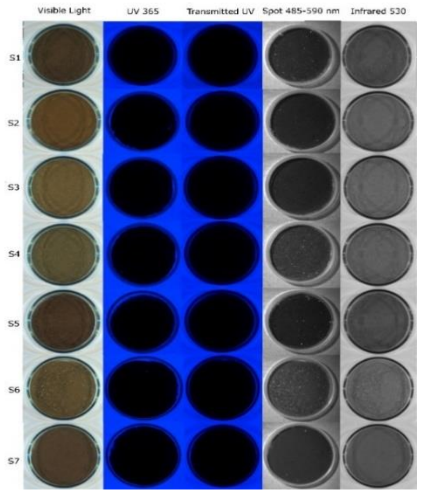

Figure 3. Soil samples under different light of sources

3.4. Textural analysis

A soil textural triangle is used to compare of soils according to their particle size compositions including silt, sand and clay. The relative percentages of components determine the soil texture. For this, some soil specimens are characterized by jar test to provide information about the soil texture. To do this, dried soil samples is placed to jar, and water is added up to half of the jar. Then, dish soap is added. The sediments are shaken and vortex for 1 min and allowed to be settled for 24 h. The bottom (first) phase shows the amount of the sand in the soil. Second phase demonstrated the presence of silt, and the third phase shows the clay composition. The organic matter on the top of water is not taken into account for the determination of soil texture.

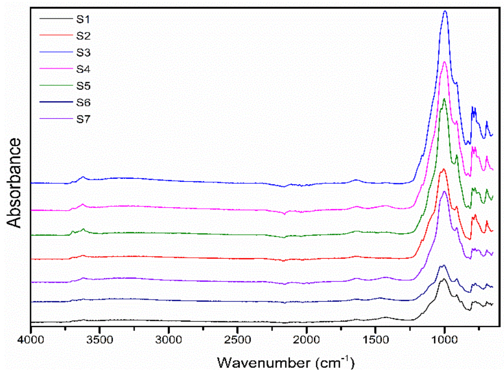

Figure 4. The FTIR spectra of soil samples

The sum of sand, silt and clay give the total amount of soil which assumed to be a 100. After then, Soil texture was determined by intersecting the calculated percentages on the soil content scale. According to the textural analysis S1 samples were found to be sandy clay loam, S6 were found to be loamy sand and S7 were found to be sandy loam which is given in Figure 5. The outcomes show that sedimentation test could be applied for discrimination of soil samples which gives information about the silt, sand and clay composition in the unknown soil samples.

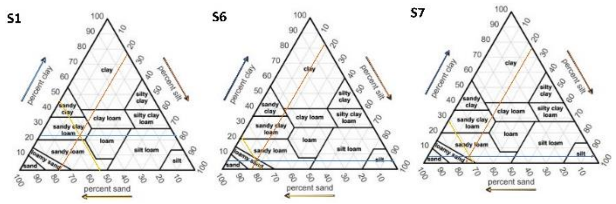

Figure 5. Textural analysis of soil samples

3.5. Sedimentation test

Sedimentation test is studied in order to investigate the stability and sedimentation behavior of soil sample in water [34]. For this, 5% of soil suspensions are prepared in falcon tube. Then, the suspension is shaken and vortex for 2 min. Then, all falcon tubes are allowed to rest, and images are taken at different time of intervals from 1 min to 24 h Figure 6. the color of all suspensions can vary which confirms the Munsell color results. At the end of 1 min, there was no obvious sediment at the bottom for all texture. To do this, dried soil samples is placed to jar, and water is added up to half of the jar. Then, dish soap is added. The sediments are shaken and vortex for 1 min and allowed to be settled for 24 h. The bottom (first) phase shows the amount of the sand in the soil.

Second phase demonstrated the presence of silt, and the third phase shows the clay composition. The organic matter on the top of water is not taken into account for the determination of soil texture. The sum of sand, silt and clay give the total amount of soil which assumed to be a 100. After then, Soil texture was determined by intersecting the calculated percentages on the soil content scale. According to the textural analysis S1 samples were found to be sandy clay loam, S6 were found to be loamy sand and S7 were found to be sandy loam which is given in Figure 5. The outcomes show that sedimentation test could be applied for discrimination of soil samples which gives information about the silt, sand and clay composition in the unknown soil samples.

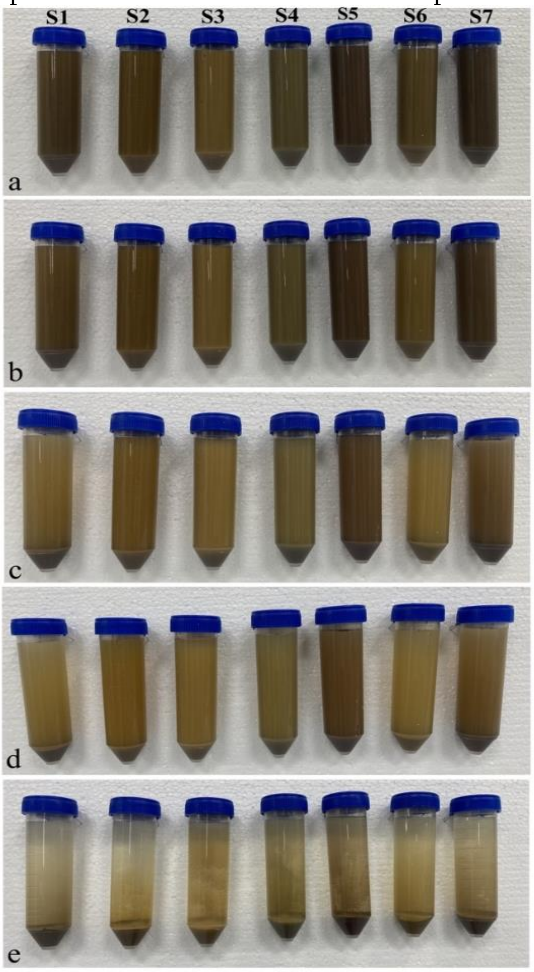

Figure 6. Sedimentation of soil suspensions against to time a) t:0 b) t:1 min c) t: 30 min d) t:1h e) t:24h

pH measurement of soil samples as a discriminant technique also gives a valuable information about the evidence and crime scene. For this, the pH value of soil samples from different region of Ömerli Dam is determined. It was found that the pH value of soil samples was ranged between 6.1 and 8.3. The pH value and description of the soil samples is given in Table 2. According to the results, S3 and S5 was found to be Neutral, while S1, S6, S7 is Moderately alkaline, and S2 is slightly acid. The difference in pH value could be discriminant character for forensic applications Table 2.

Table 2. pH of soil samples in Ömerli Dam region

Sample	pH	Description
S1	8.2	Moderately Alkaline
S2	6.1	Slightly Acid
S3	6.8	Neutral
S4	8.1	Moderately Alkaline
S5	6.6	Neutral
S6	8.3	Moderately Alkaline
S7	8.3	Moderately Alkaline

3.6. ICP-OES

The quantification of metal contents in the soil sample could be potential evidence for comparison of soils. The metal concentration of soil samples was shown in Table 3.

Table 3. Metal concentrations of soil samples by ICP-OES

	ppm				
	Si	**Fe**	**Mg**	**Ca**	**Cu**
S1	<1	139	39.5	265	0.5
S2	<1	229	33.4	22	0.35
S3	<1	268	79.9	30.8	0.60
S4	<1	265	80.2	548.5	0.97
S5	<1	161	22.5	41.4	0.21
S6	<1	94.9	7.5	240.7	0.20
S7	<1	166	62.0	273.2	0.36

The Fe content of soils was found to be 139 ppm for S1, 229 ppm for S2, 268 ppm for S3, 265 ppm for S4, 161 ppm for S5, 94.86 ppm for S6 and 166 ppm for S7. The Fe content was found to be between 94.86 and 268 ppm.

4. Conclusion

It is crucial that identifying the source of the evidence is gaining importance for crime scene investigation. In this study, the comparison of soils in Ömerli Dam which is one of the most important drinking water sources in the north-east of Istanbul have been explored. For this, varios techniques including color test, structural analysis, pH, textural and metal analysis have been studied. The coordinates of studied area is detected by Google maps. The metal content including Si, Fe, Mg, Ca and Cu have been investigated which contains at different ratios.

35 of samples from 7 different regions for analyzed. The natural pH of the soils was varied from 6.1 to 8.3. The color comparison of soil samples is studied by Munsell color chart. The image analysis showed similarities and could not be discriminated from each other. Textural analysis of soil samples demonstrated that it could be effective, easy, and cheap method for studying of soil samples as forensic evidence. Moreover, the sedimentation studies could also be applied and show dissimilarity due to their sand, silt and clay composition which also influence the textural properties.

The outcomes showed that FTIR is inadequate by itself for discrimination of soil samples in Ömerli Dam region. pH of soil samples could give an crucial data for comparison of samples. Moreover, textural anaylsis and heavy metal analtsis have discriminant results in order to compare the soil samples. VSC analysis is also not sufficient for comparing of soil samples. As a result, it is important for forensic sciences to use a combination of different methods to determine the source of the soil.

Article Information Form

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Authors' Contribution

S.K: writing, original draft, methodology, investigation, visualization, conceptualization, supervision, project administration, review, funding and editing I.S.B: writing, original draft, methodology, investigation, visualization, conceptualization, Project administration is added and funding S.A: writing, methodology, conceptualization, review and editing.

The Declaration of Conflict of Interest/ Common Interest

No conflict of interest or common interest has been declared by the authors.

The Declaration of Ethics Committee Approval

This study does not require ethics committee permission or any special permission.

The Declaration of Research and Publication Ethics

The authors of the paper declare that they comply with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that they do not make any falsification on the data collected. In addition, they declare that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic publication environment other than Sakarya University Journal of Science.

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