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Measurement of Heavy Metal Concentrations of Marine Sediments in Yalova Section of Marmara Sea By LIBS Method And Evaluation of Pollution By Principal Component Analysis

Marmara Denizi Yalova Kesiminde Deniz Sedimentlerinin Ağır Metal Konsantrasyonlarının Libs Yöntemi ile Ölçülmesi ve Kirliliğin Temel Bileşen Analizi ile Değerlendirilmesi

Zeki Ünal Yümün [1*], Hızır Aslıyüksek [2], Murat Mücahit Yentür [3], Melike Önce [4]

[1]Namık Kemal University, Çorlu, Tekirdağ, Türkiye, ORCID: 0000-0003-0658-0416, zyumun@nku.edu.tr

[2]İstanbul University, Cerrahpaşa Institute of Forensic Medicine and Forensic Sciences, İstanbul, Türkiye, ORCID: 0000-0001-6845-3717, doktorhizir@gmail.com

[3]İstanbul Sabahattin Zaim University, İstanbul, Türkiye, ORCID: 0000-0003-4382-7502, myentur@hotmail.com

[4]Namık Kemal University, Tekirdağ, Türkiye, ORCID: 0000-0002-3238-3726, e-mail: monce@nku.edu.tr

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Abstract

The wastes formed as a result of increasing settlements, industrial establishments, agricultural activities and shipping activities on the shores of the Marmara Sea cause the

* Corresponding author: zyumun@nku.edu.tr

pollution of the Sea of Marmara. Although the pollution in seawater is diluted in a short time by waves and water currents, heavy metals and other pollutants that precipitate chemically and physically create permanent pollution in the sediments. Heavy metal concentrations in Yalova and its surroundings were analyzed by LIBS (Laser-Induced Breakdown Spectroscopy) method in order to control the pollution in this region. In the analyzes made, the percent availability of heavy metals in the sediments was evaluated. With Principal Component Analysis (PCA), heavy metal contamination levels caused by domestic and industrial wastes in the sediments were analyzed and the results were interpreted. The high levels of Ca, Na, Mg elements in the region depend on the presence of carbonate rocks in the region. The presence of Fe, Al and some trace elements is due to the prevalence of volcanic and magmatic rocks in the region. The fact that the elements with high economic value such as Al, Ag and Ti are especially high in Core 1-6 is due to the granitic rocks outcropping in the eastern part of the study area where the samples were taken in, and it is an indication of the existence of important mineral mineralizations in the region. This situation shows that heavy metal pollution in the marine environment consists of both anthropogenic and natural sources.

Keywords: Sea Sediment, LIBS, PCA, Marmara Sea, Yalova

Öz

Marmara Denizi'nin kıyılarında artan yerleşim yerleri, sanayi kuruluşları, tarımsal faaliyetler ve gemicilik faaliyetleri sonucu oluşan atıklar, Marmara Denizi'nin kirlenmesine neden olmaktadır. Deniz suyunda meydana gelen kirlilikler dalga ve su akımlarıyla kısa zaman içinde seyrelse de kimyasal ve fiziksel olarak tabana çökelen ağır metal ve diğer kirleticiler sedimentlerde kalıcı kirlilikler meydana getirirler. Yalova ve çevresinde oluşan kirliliklerin kontrol altına alınabilmesi açısından bu bölgede ağır metal konsantrasyonları LIBS (Laser-Induced Breakdown Spectroscopy) yöntemiyle analiz edilmiştir. Yapılan analizlerde sedimentlere karışmış ağır metallerin yüzde olarak bulunabilirlikleri değerlendirilmiştir. Temel Bileşen Analizi (PCA) ile de sedimentlerde evsel ve endüstriyel atıkların neden olduğu ağır metal kontaminasyon seviyeleri analiz edilerek sonuçları yorumlanmıştır. Ca, Na, Mg elementlerinin bölgede yüksek oranlarda bulunması, bölgedeki karbonatlı kayaların varlığına bağlıdır. Fe, Al ve diğer eser elementler ise volkanik ve magmatik kayaların bölgede yaygın olmasından ileri gelmektedir. Al, Ag ve Ti gibi ekonomik değeri yüksek elementlerin özellikle Core 1-6'da yüksek olması bölgedeki granitik kayalara bağlı olup, bölgede önemli maden cevherleşmelerinin varlığına bir işarettir. Bu durum denizel ortamda oluşan ağır metal kirliliklerinin hem antropojenik hem de doğal kaynaklardan oluştuğunu göstermektedir.

Anahtar Kelimeler: Deniz sedimenti, LIBS, PCA, Marmara Denizi, Yalova

INTRODUCTION

Today, the most dangerous pollutants that pollute the environment and pose a risk to life are heavy metals. With the development of technology, heavy metals are found in high or trace amounts in almost every material we use in our daily life. One of the most important causes of environmental pollution is the discharge of heavy metal wastes used in an industry into the aquatic environment without being sufficiently separated. Heavy metals are not biodegradable in their environment. When heavy metals and water contaminated with them are discharged into aquatic environments such as streams, lakes, seas and oceans without being treated, they become highly toxic to the living system and its environment in that region. In addition, when these toxic substances are mixed with biological treatment systems, they show toxic properties for microorganisms that have an important role in the biological treatment system (Sağlam & Cihangir, 1995; Önce, Balman, & Kam, 2021.; Dökmeci, 2021). Heavy metals enter into the systems of the proteins in living things that perform their vital functions such as oxygen, nitrogen and sulfhydryl, make bonds and thus precipitate the enzymatic system. In addition, some heavy metals compete with metals with physiological functions and take their place. Considering these properties of heavy metals, their detection in all living environments (sediment, water, air, soil) becomes very important. In this process, the LIBS method is more useful than traditional old methods because of the fast and practical detection of metals. The LIBS method is a clean analysis method that reduces the sample preparation process, does not require reagents, and gives fast results. The fact that the measuring devices give results in a short time facilitates the analysis in the field. Thanks to the LIBS analysis method, environmental pollution is identified without wasting time and the process of taking precautions is accelerated (Yümün & Kam, 2019; Sarah, Jose, & Almiral, 2021; Kumar P., et al., 2011). Today, the LIBS method is used successfully in various fields (environment, health, industrial production, forensics, etc.) (Villas-Boas, et al., 2016; Sankaran, Ehsani, & Morganc, 2015; Guang, Qias, Chen, Ding, & Tian, 2015). In this study, heavy metal analyzes of ten marine sediment samples taken from the Yalova coastal part of the Marmara Sea were carried out by the LIBS method. PCA analysis was applied for the analytical interpretation of the obtained results. There are many studies on the LIBS method in the Marmara Sea and the surrounding areas (Önce, Balnan, & Kam, 2021; Yümün, Kam, & Önce, 2019). In these previous analyzes, heavy metal concentrations that cause marine pollution were evaluated. Conducting this study contributed to the consideration of all studies as a whole.

MATERYAL METOD

Sampling

In the study area, ten core (gravity core) samples were taken within approximately 10-20 m from the sea coast. The location map of the study area and sample locations are given in Figure 1 and their coordinates are given in table 1. The storage, transportation and preservation of sediment samples were carried out according to "TS EN ISO 5667-15 Water Quality-Sampling-Part 15: Protection and Transport Guide of Sludge and Sediment Samples". Sample points were determined in a way and number to characterize the sediment quality in the region and the variation of this quality within the region. In the selection of the sample locations, industrial zones, waste discharge areas and regions where the effluent of wastewater treatment plants are discharged were taken into account. Care was taken not to physically interfere with the sediment to be sampled before sampling. Depending on the sedimentation rate, samples were taken from the top layer of the sediment from a depth of 0 to 50 cm. In regions with high flow velocity, sampling depth was chosen as more than 5 cm (Yümün & Kam, 2019-1; INT-1, 2015; Yümün Z.Ü. and Kam E., 2019-2; Dökmeci, 2021; Kontas, et al., 2020).

Analysis Method

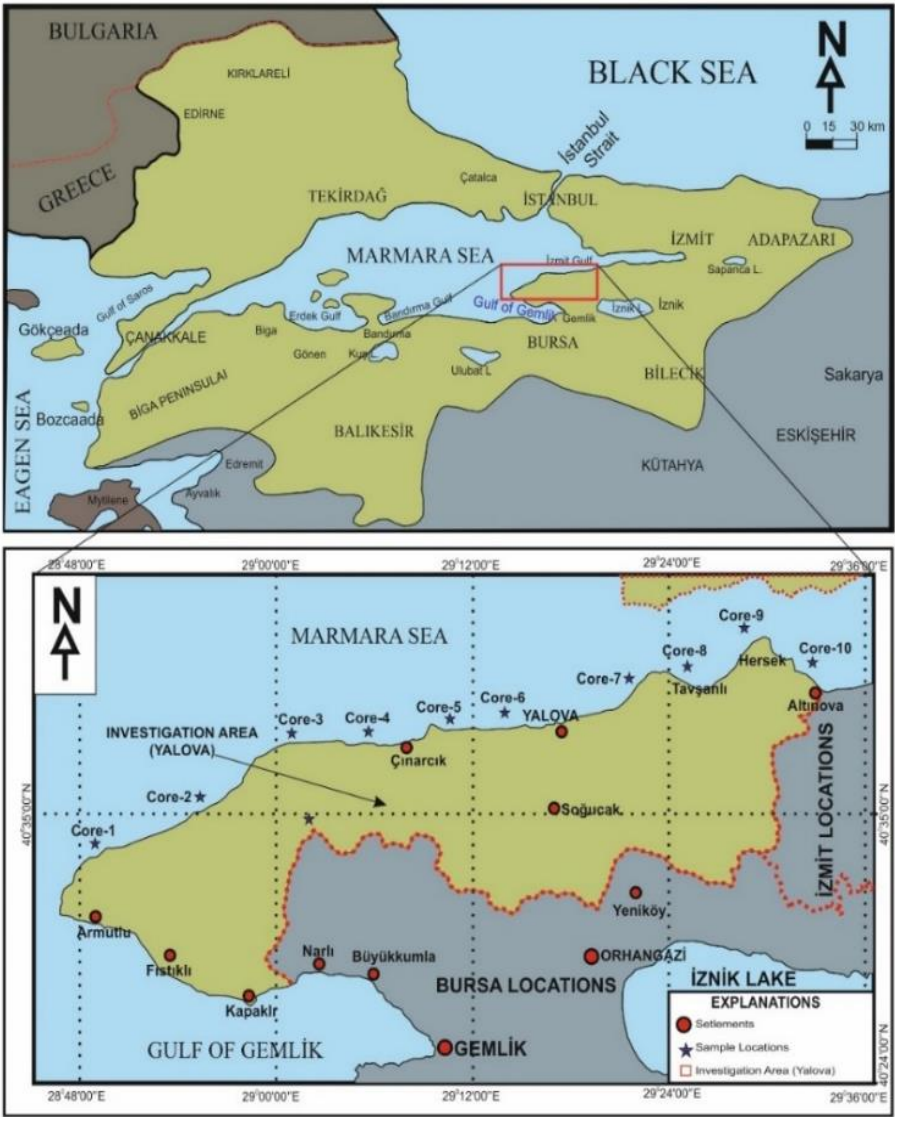
Laser-Induced Plasma Spectroscopy (LIBS) is a method used for multi-element analysis of materials (Barbini et al., 1999; Yang, 2009). In this technique, a focused, high-intensity, powerful laser beam hits the solid surface of the sample under study. As a result, a high-density plasma cloud is formed near the expanding surface (Yang, 2009). The optimum instrumental parameters for soil analysis were obtained when the repetition rate, td and tw, were equal to 10 Hz, 1 μs and 10 μs, respectively. Standard reference material (SRM-2586) was used to prepare the pellets (Daniel, Pereira-Filho, & Konieczynski, 2017; Yümün, Kam, & Önce, 2019-3).

Sample Preparation and Measurements

For elemental analysis, 25 g of each sediment sample was taken from each core. The sediment samples taken were collected in a specially made pellet container with 20-25 gr and compressed in the press machine under a pressure of about 100 bar for 5 seconds. This shape of the samples is called “pellet”. Standard reference material (SRM-2586) uses to prepare pellets for the parameter analysis. The pelleted samples place in sealed bags and sent for LIBS analysis (Yümün & Kam, 2019; Daniel, Pereira-Filho, & Konieczynski, 2017; Imam, Aziz, Chaudhary, Rizvi, & Ali, 2018).

Figure 1

Location Map of the investigation area and samples.



Tablo 1

Core coordinates, depths and lithologies of samples

Sample No	Coordinat		Depth (m)	Lithology
	Y (North)	X (East)		
Core-1	4490806.68	650935.56	12.00	Sandy Silt- Clay
Core-2	4495209.07	660644.89	13.00	Sandy Silt- Clay
Core-3	4500426.93	666593.4	12.50	Sandy Silt- Clay
Core-4	4502037.00	670824.00	13.50	Sandy Silt- Clay
Core-5	4502616.00	676534.00	16.20	Sandy Silt- Clay
Core-6	4503311.00	682500.00	18.60	Sandy Silt- Clay
Core-7	4504259.00	689241.00	20.00	Sandy Silt- Clay
Core-8	4507026.00	698699.00	17.40	Sandy Silt- Clay
Core-9	4510209.00	708649.00	19.00	Sandy Silt- Clay
Core-10	4508757.00	715575.00	14.50	Sandy Silt- Clay

ANALYSIS RESULTS

LIBS Analysis and Evaluation of Analysis Results With PCA Analysis

LIBS analysis results (%) of ten Core samples taken from the study area are given in table 2. Principal Component Analysis (PCA) was used to evaluate these results empirically and analytically. Principal Component Analysis (PCA); It is one of the multivariate data analysis techniques used to reduce the data size (Yalçın, Kılıç, Nyamsari, Yalçın, & Kılıç, 2016). In this technique, the total variation in the data set, it is explained through new 'principal components' that allow the original variables to be restated in several linear combinations and have no correlations between them. The best results in PCA are obtained when the variables that make up the data set have the highest negative or positive correlation with each other (Caneque, ve diğerleri, 2004; Panthija, Rai, Rai, & Thankur, 2010; Yoon, Choi, Moon, & Choi, 2021; Özkan, Büyükışık, Kontaş, & Türkdoğan, 2017). Table 3 contains the results of the PCA analysis. Looking at the cumulative sum, 9 samples provided 100% of the variance for 21 elements. The first sample accounted for 30.638% of the variance, the second sample 27.828%, the third 14.344%, the fourth sample 10.603%, the fifth sample 8.322, the sixth sample 4.816%, the seventh sample 2.205, the eighth sample 1.1% and the ninth 0.188% explains.

Table 2

LIBS analysis results

Sample Locations	Yalova 1 (%)	Yalova 2 (%)	Yalova 3 (%)	Yalova 4 (%)	Yalova 5 (%)	Yalova 6 (%)	Yalova 7 (%)	Yalova 8 (%)	Yalova 9 (%)	Yalova 10 (%)
Ba	92	91	89	80	92	91	88	86	84	88
Li	90	91	88	89	89	91	88	91	83	90
Mg	89	88	86	86	87	90	88	90	90	89
O	88	87	85	89	90	87	88	88	89	88
Si	88	86	86	82	88	87	88	82	84	86
Ga	85	89	93	75	87	78	79	49	90	74
Rb	74	75	-	76	76	70	71	52	74	69
Al	74	73	73	72	73	85	84	74	74	75
Na	73	94	72	72	90	78	93	70	72	76
Sr	65	60	87	77	88	82	50	76	61	77
K	65	59	-	63	65	64	65	63	67	65
Ag	64	54	-	61	-	66	72	-	-	-
H	55	50	46	-	56	-	-	57	-	-
Fe	95	95	95	95	95	95	95	95	95	95
Ti	71	71	77	69	68	73	76	66	80	72
Ca	71	73	74	78	79	66	74	72	75	77
Be	-	-	72	-	46	64	50	-	54	63
N	-	-	63	68	68	71	69	59	75	78
Mn	-	-	49	-	-	-	-	43	-	-
F	-	-	-	57	-	-	-	-	-	70
Cl	-	-	-	-	-	-	-	-	69	-

The chart of the Eigenvalues is given in Figure 3. Using Eigenvalues, there are factors to be interpreted in PCA analysis. These factors which obtained from the analyzes greater than 1 are considered significant, and factors less than 1 are not considered. The total variance associated with each factor is shown in the Scree Plot Chart. The break occurred after the 5th point in the graph given in Figure 3 and a certain decrease has been observed.

Table 3

Explanation of total variance on elements in the Yalova coastal sediments

PC	*Eigenvalue*	*%Variance*	*Cumulative %*
1	2302.2	30.638	30.638
2	2091.03	27.828	58.466
3	1077.8	14.344	72,810
4	796.712	10.603	83,413
5	625.301	8.3217	91,735
6	361.856	4.8157	96.550
7	165.657	2.2046	98.755
8	79.3972	1.0566	99.812
9	14.1579	0.18842	100.00

Hence, the factor number was determined as four. No significant downward trend is seen after five and other factors. In other words, the contributions of the four and the following factors to the variance are close to each other. The results of the principal component analysis (PCA) of the elements as shown in Table 3 indicates that the six components retained have a good representation of all the elements.

Figure 2

Principal Component Analysis (PCA) Results

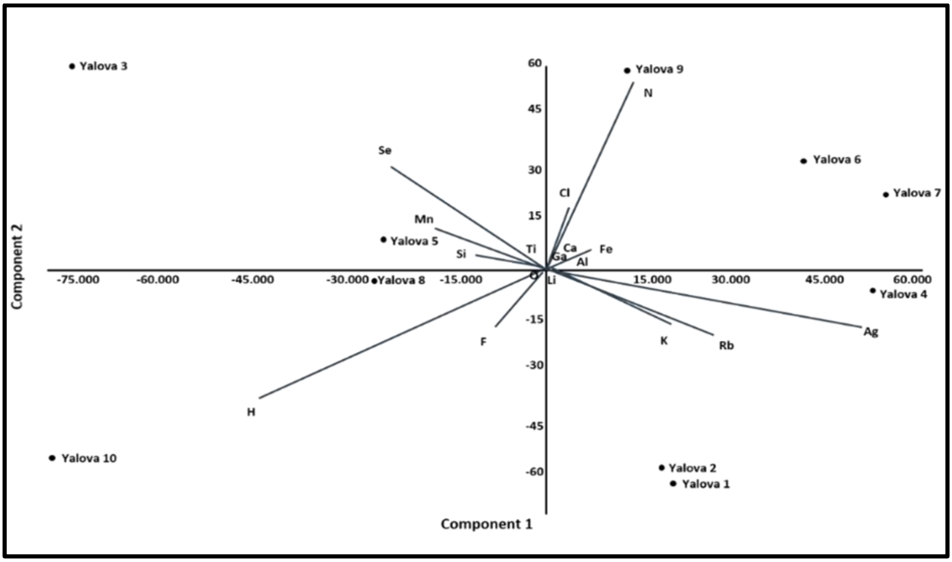
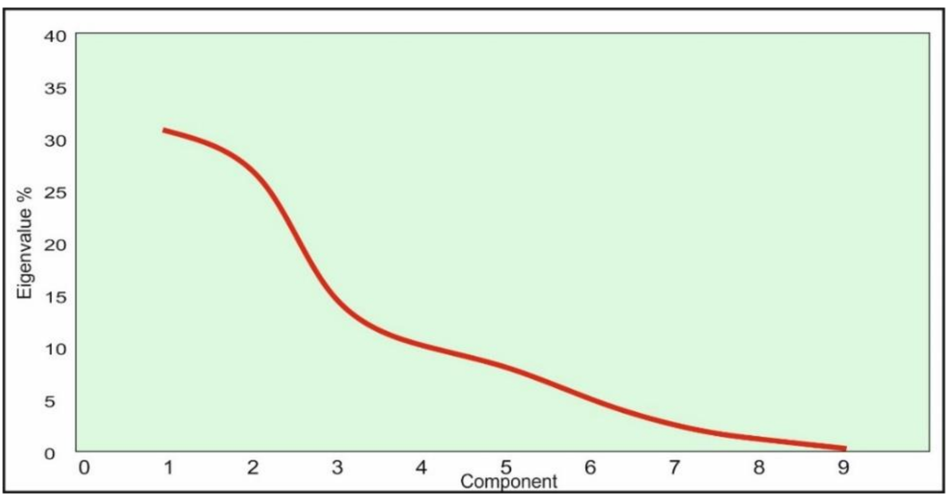


Figure 3

Scree Plot Chart Showing the Factor Analysis of the Elements in the Composition of Marine Sediments



Study Area Geology and Evaluation of the Effect of Geological Structure on Element Presence

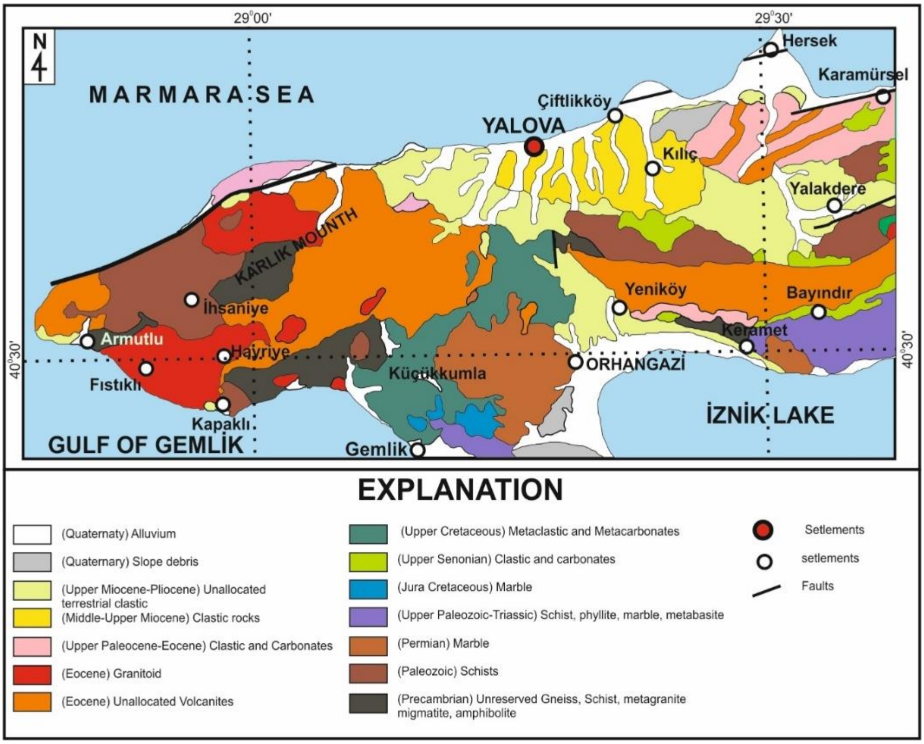
The research area has a slightly sloping topography, and the adjacent land areas are at different heights between 10-15 meters compared to sea level. Various lithological units representing different times from the Paleozoic to the Present are exposed in the study area (Figure 4). At the basement of the field are the Pamukova metamorphics, which are thought to be Precambrian-Lower Paleozoic aged, and the Lower Triassic?-Cretaceous aged Iznik Metamorphics, which show less metamorphism (Göncüoğlu, et al., 1986; Göncüoğlu, et al., 1992).

At the top, the Upper Cretaceous Bakacak formation, Upper Paleocene-Middle Eocene aged İncebel formation, and Eocene aged Sarısu formation overlying the basement are sedimentary and volcano-sedimentary units. During the Eocene period, the Fistikli Granitoid was emplaced in the region. In the upper part, Miocene aged Kılıç formation, Upper Miocene-Lower Pliocene aged Yalakdere formation, Pleistocene aged marine terrace deposits and current alluviums are located. The coastline gained its current position in the Holocene and the marine successions in the flow direction of Safran Stream continue to develop today (Erendil, et al., 1991; Günay, 2018).

The high levels of Ca, Na, Mg elements in the region depend on the presence of carbonate rocks such as Limestone ($CaCO_3$) and Magnesium ($MgCO_3$). Fe, Al and other trace elements depend on the prevalence of igneous and igneous rocks in the region. The reason why elements such as Al, Ag and Ti are especially high in the first 6 samples (Core 1-6) is thought to be due to the proximity of granitic rocks to these locations. The presence of these elements with high economic value is an important finding and detailed studies are recommended for detailed extraction of mineral enrichments.

Figure 4

Geological map of the study area and its surroundings (Bayrak, Yümün, & Kam, 2018; Turkecan & Yurtsever, 2002)



CONCLUSION

In this study, LIBS analysis was used to analyze heavy metal pollution of marine sediments of the Yalova coast. PCA analysis was performed to evaluate the results of the LIBS analysis. Due to the redundancy of the spectral data of the ten samples obtained, it becomes difficult for us to analyze them. It is also difficult to analyze spectral lines from LIBS spectra. Therefore, PCA analysis was used to help us reduce and compress data by selecting only the principal components (PCs). As a result of the analysis, it was seen that the LIBS technique is capable of performing the separation and differentiation of various samples, no matter how small the differences in element composition. In PCA analysis based on LIBS spectra, common points were found in the elemental composition of the sediment samples. It can comment on the pollution of the Yalova seacoast, whose element composition is obtained. By determining the lithological characteristics of the sediments around the study area, a more realistic evaluation of the LIBS analysis results was provided. Here, it is understood that there may be significant mineral enrichment, especially in the locations where Core 1-6 samples are found.

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Author Contributions

All authors contributed jointly at every stage of the study.

Conflict of Interest

No conflict of interest was reported by the authors.

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Ethical Statement

Ethics committee approval is not required for the study.