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Investigation of Heavy Metal Concentrations in The Gulf of Izmit (Marmara Sea) Altinova Shipyard Region

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Abstract: In this study, heavy metal concentrations (Ca, Mg, Hg, As, Sb, Ag, Al, Co, Cr, Cu, Fe, Mn, Ni, Pb, V, Zn, Ti, Th, U, Mo, Cd) amounts were measured by examining marine sediment samples obtained from the coasts of the Izmit Bay Altınova shipyards region. The natural and anthropogenic pollution levels of the region were revealed by the analysis of sediment samples. The extent to which the marine ecosystem may have been affected by the shipyard activities that have been actively carried out in the region for the last ten years was also investigated. Heavy metal analyses of sediment samples were carried out by the XRF method. In addition, the morphological and surface features of the sediment grains were obtained by SEM analysis, and the element contents of seawater samples obtained from the region were determined with an ICP-OES device. The heavy metal pollution level of the region was revealed by evaluation of the data obtained as a result of the analysis. The results of the XRF analysis showed that the heavy metals, which are toxic for humans, such as cadmium (max 17.984 ppm), lead (max 31.302 ppm), nickel (max 71.725 ppm) and arsenic (max 13.852 ppm) were detected. According to the results of elemental analysis with ICP-OES, Hg, As, Sb, Ag, Al, Co, Cr, Ni, Pb, V, Ti, U, Mo, and Cd were below the measurable limit.

Keywords: Heavy metal, pollution, XRF, SEM, Yalova, Marmara Sea.

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INTRODUCTION

Heavy metals have become one of the most accumulated pollutant groups with the development of industrial activities in marine ecosystems. Because these pollutants cannot be decomposed biologically and chemically, they accumulate in the sediment at the seabed. Environmental monitoring and evaluation of seacoasts is of critical importance in terms of the health of humans and sea creatures. In such monitoring, sediments are used as bioindicators for marine environments. The Sea of Marmara is exposed to chemical and biological pollution from various sources, including a dense population, excessive industrial activities, intense maritime traffic in the straits, marine accidents, and agricultural activities (1-5). This influx has caused pollution, not only in the Marmara Sea, but also in neighboring seas and rivers (1,6). In the marine environment, metals exist in dissolved forms or are adsorbed onto suspended particles (7,8). The movement of heavy metals in the marine environment is dependent on the primary minerals and on the silicate content in the sediment (9). Elements such as Cd, Pb, As, and Hg in marine environments are designated as potentially harmful elements (PHEs) and are reported as priority hazardous substances by Directive (AB) 2020/2184 of the European Commission (10-13). Sediment monitoring has been very important in revealing pollution in marine environments in recent years, and there have been studies on the subject (14-19).

In the study area, there are studies conducted in previous years regarding the pollution status of the region. Okay et al., (2001) showed that there were significant increases in Chlorophyll-a value, which is an indicator of primary producing organisms (phytoplankton) in the Gulf, compared to previous years (20). Morkoç et. al. (1995) studied the effects of wastewater on the water quality of the Bay. Pollution sources that put pressure on the Bay were determined and the situation of the Bay in terms of water quality was determined (21). Sur et al. (2010) determined that the area was polluted according to the metal values measured in the seafloor surface sediment of the study area. It has been determined that the biggest source of this pollution is port and shipyard activities (22). Bayrak et. al. (2018), showed the heavy metal densities of the marine sediments of the Izmit Bay Altinova region and it was determined that it was rich in metals such as Hg, Cu, Ni began (23). In 2007-2008, pollution-monitoring studies were carried out by TÜBİTAK / MAM in the Gulf. As a result of these studies, the water quality of the coastal areas was rated as poor or bad (24).

With these researches on heavy metals conducted in the study area, ways have been evaluated in which the marine ecosystem may have been affected by the shipyard activities, domestic and industrial waste disposal, as well as agricultural activities that have been actively carried out in recent years. Especially from shipyards; pollutants such as ship waste water, ballast water, ship repair oils, chemical paint wastes, wastes arising from leakage are mixed with marine environments. Discharge of these wastes to the marine environment without treatment puts the living life in the region in danger (25-29).

In this study, Marine sediment samples obtained from the shores of the Altınova shipyards region of the Gulf of Izmit were analyzed. Heavy metal analyses of marine sediment and seawater samples were carried out with XRF (wavelength-distributed X-ray fluorescence spectrometry), ICP-OES (inductively coupled plasma-optical emission spectrometry), SEM (scanning electron microscopy).

MATERIALS AND METHOD

Sampling Area

Yalova province is located in the south of Marmara Region, at the entrance of Izmit Bay (Figure 1). It is a developed region in terms of industry, shipbuilding facilities, and population, and there are side-by-side shipbuilding facilities along the coast of the study area. Altınova is built on the Hersek delta where agricultural activities are intense. The region is very active in terms of shipyard activities.

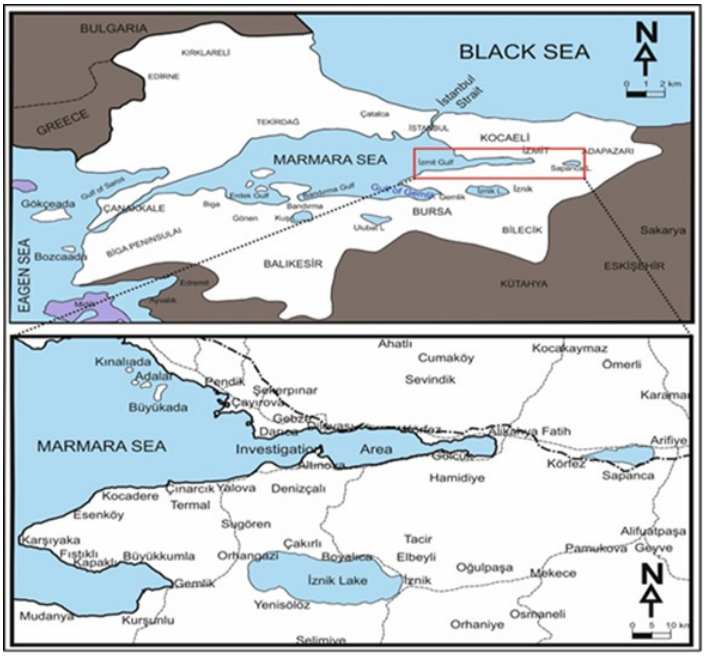

Figure 1: Location map of the study area.

Shipyard Pollution

In the shipbuilding and repair process, especially steel and metals, as well as paint and paint solvents (solvent), blast abrasives, machine oils and cutting oils are used as raw materials. It also contains chemical materials, heavy metals and cyanide used in surface preparation processes such as acid and alkaline cleaners, degreasing solvents and coating solutions. Shipyard wastes generally consist of organic compounds (VOCs), particles (PM), waste solvent materials, oil and resins, metal wastes, dirty water, dirty waste paint and abrasives (30).

Table 1: Shipyard processes and waste stream (30).

Process	Input Material	Air Emissions	Waste water
Surface Preparation	Abrasives (steel shot, grit, copper slag, paint removers and cleaners)	VOC emissions from solvent cleaners and paint removers	Paint chips, cleaning fluids, surface contaminants, oil and scrap oil from cargo and ballast tanks
Metal plating and surface finishing	Coating metals, cyanide solutions, cleaning solvents, rinses	Metal ion vapour, acid vapour, VOC emissions from solvents	Rinse water contaminated with metals, cyanide, acids, alkalis, organics and solvents
Painting	Paint, solvent and water	VOC emissions from paints and solvents and equipment being cleaned	Contaminated water from equipment cleaning and solvents
Fiberglass manufacture	Fiberglass, resin, catalysts, wood and plastic materials	VOC emissions during manufacturing	Waste water generation is very low
Machining and metal works	Cutting oils, solvents	VOC emissions from degreasers and cleaning solvents	Waste water contaminated with used solvents, cutting oils and cooling oils

Sampling and Preparation

Sea sediment samples were collected from sub-seafloor depths varying between 7 m and 38 m. A sediment sample and a seawater sample were taken with a ship at each of six points deemed appropriate for the region (Figure 2). The depths and coordinates of the six core samples obtained by drilling are recorded (Table 1).

Figure 2: Study area where sample locations are specified (31).

XRF Analysis of Sediment Samples

The analysis is based on excitation of atoms with a high-energy radiation such as X-rays. When the excited electrons return to their initial energy levels, they give back the excess energy they have gained in the form of X-rays with wavelengths of 0.1–50 Å. This secondary emission of X-rays is called fluorescence. The wavelengths of the radiations produced by the elements are specific to those elements (32). For XRF analysis, sediment samples were first ground into powder; then 9 grams of each sample was weighed and mixed homogeneously with 6 grams of cellulose. The obtained mixtures were transferred to a mold designed for that analysis and formed into pellets under high pressure. Pellets of approximately 15 grams each were prepared for analysis and were analyzed with the Pro-Trace program (Malvern Panalytical) for 116

minutes. The Pro-Trace program, which is a program that can count at the level of ppm (parts per million), is frequently used in elemental analysis studies today (33).

ICP-OES and Anion Analysis of Seawater Samples

The ICP-OES technique is based on the excitation of the elements in the sample by argon plasma, which is heated to a temperature of 10,000 K by electromagnetic induction, and measuring the optical emission of the excited elements according to their specific wavelengths (EPA Method 200.7) (34,35). ICP-OES analyses were performed on seawater samples taken from the study area. Seawater samples taken from the field were preserved and brought to the laboratory environment in accordance with sample storage standards (34,35). First, anion analyses were carried out on the water samples collected from the region. The device used in that analysis was an ICS-3000 DIONEX brand ion chromatography device. Results were obtained in ppm (3,5).

SEM Analysis of Sediment Samples

SEM images were taken with a JEOL brand JSM-6390LV scanning electron microscope. The sample to be SEM imaged first had to be resistant to vacuum; that is, it must not evaporate, and it must be in the solid state (36). (Evaporation of samples under vacuum causes the microscope to become contaminated and may cause errors in the analysis result.) In SEM, a qualified image is obtained by scanning the sample surface in detail with electrons (33,37).

RESULTS AND DISCUSSION

XRF Analysis Results

XRF analysis results of the sediment samples are shown in Table 3.

In the qualitative analyses made with XRF, both element and compound determinations were made for the sediment composition. Because the sediments are composed of organic and inorganic deposits, the elements and compounds obtained in elemental analysis are quite diverse. The results of the analysis revealed that toxic heavy metals such as cadmium, lead, nickel, chromium, and arsenic were detected in high concentrations.

ICP-OES Analysis Results

The ICP-OES analysis results of the seawater samples are given in Table 5.

Table 2. Depths and coordinates of sediment samples obtained from the study area.

Sample No	Sample Depth (m)	Sample Coordinates (WGS-84, 6^0)	
		Y (East)	X (North)
ALT-2	36	707472.83 d E	4508710.82 m N
ALT-3	25	708515.39 d E	4510087.64 m N
ALT-4	38	708213.88 d E	4510457.64 m N
ALT-5	7	710247.21 d E	4511234.23 m N
ALT-6	16	709718.67 d E	4511605.90 m N
ALT-8	27	706038.94 d E	4512214.84 m N

Table 3: Elemental or component concentrations of sediment samples.

	Sample No.						
Analyte	**ALT-2 (ppm)**	**ALT-3 (ppm)**	**ALT-4 (ppm)**	**ALT-5 (ppm)**	**ALT-6 (ppm)**	**ALT-8 (ppm)**	**Average (ppm)**
CaO	40,621.49	58,697.86	45,406.62	64,947.69	79,182.22	47,954.05	56,135
Sc	10.461	10.2	9.131	9.968	5.911	10.102	9.295
TiO_2	8995.155	8769.926	8915.32	8601.837	7829.54	8338.949	8575.11
V	98.913	104.746	97.119	111.393	105.338	104.23	103.6
Cr	78.331	72.632	74.615	99.909	109.665	79.99	85.86
Mn	411.705	431.44	404.828	854.884	658.978	443.219	534.18
Fe_2O_3	55,926.19	56,848.45	54,912.61	62,113.25	56,197.16	53,046.84	56,507.4
Co	30.654	32.384	29.87	28.35	31.592	24.921	29.63
Ni	19.448	15.031	19.078	61.294	71.725	18.162	34.12
Cu	22.823	102.799	21.295	85.177	100.921	80.478	68.915
Zn	76.986	86.662	69.79	90.52	85.467	86.211	82.606
Ga	14.037	13.95	13.595	16.086	14.733	13.677	14.35
Ge	-0.384	-0.726	-0.764	-0.889	-0.567	-0.82	-0.69
As	8.316	9.053	8.518	13.151	13.852	10.698	10.598
Se	0.365	0.819	-0.111	0.506	0.723	-0.174	0.3546
Br	61.64	59.981	49.375	14.268	22.251	63.709	45.204
Rb	79.757	76.005	79.025	92.643	87.778	72.75	81.326
Sr	156.675	262.375	172.795	185.423	216.609	168.63	193.75
Y	22.201	22.751	21.878	22.687	21.243	21.552	22.052
Zr	165.937	142.874	162.801	131.104	128.387	151.585	147.11
Nb	8.488	7.683	8.324	9.414	8.269	7.31	8.248
Mo	1.351	1.803	0.958	2.034	1.266	1.776	1.5313
Ag	-3.65	0.176	-0.923	-2.938	-6.226	-4.271	-2.972
Cd	17.984	18.436	21.026	17.478	17.705	16.412	18.174
Sn	3.232	7.535	5.031	6.784	7.991	8.024	6.432
Sb	4.823	0.802	2.257	5.958	5.761	5.663	4.077
Te	1.602	1.833	3.579	5.085	2.874	6.441	3.569
I	33.411	11.124	19.371	8.253	10.169	16.282	16.435
Cs	-14.354	-14.608	-13.98	-8.719	-7.968	-10.845	-11.75
Ba	199.893	196.735	190.162	253.518	271.294	196.469	218.011
La	34.585	30.899	23.762	26.434	25.248	23.102	31.57
Ce	44.173	47.583	47.242	50.167	39.931	76.717	50.968
Nd	30.514	31.69	26.618	41.402	25.894	33.3	31.569
Sm	0.261	-0.097	2.515	2.114	5.143	5.516	2.5753

Analyte	Sample No. ALT-2 (ppm)	ALT-3 (ppm)	ALT-4 (ppm)	ALT-5 (ppm)	ALT-6 (ppm)	ALT-8 (ppm)	Average (ppm)
Yb	-18.848	-16.25	-16.46	-18.191	-16.651	-15.65	-17.008
Hf	-1.643	-3.609	-2.504	-4.73	-5.945	-3.971	-3.734
Ta	2.255	-0.058	0.02	-0.053	1.736	-2.017	0.3138
W	58.402	56.682	66.785	24.912	45.015	47.011	49.801
Hg	-118.182	-98.352	-117.369	-87.526	-86.553	-91.486	-99.911
Tl	2.002	0.367	1.911	1.27	2.012	1.06	1.437
Pb	24.174	29.202	21.006	31.302	33.893	30.856	28.405
Bi	-0.024	0.391	-1.202	-0.343	0.057	-0.692	0.302
Th	9.018	8.369	8.417	11.242	10.405	8.088	9.256
U	3.424	3.864	4.418	3.582	3.503	3.492	3.713

Table 4: Anion results of the seawater sample (ppm).

Sample	Fluoride [F^-]	Chloride [Cl^-]	Nitrite [NO_2^-]	Bromide [Br^-]	Nitrate [NO_3^-]	Sulfate [SO_4^{2-}]	Phosphate [PO_4^{3-}]
Seawater	0.9 ±0.1	12,9806 ±0.3	<MDL	36.5 ±0.2	9445.6 ±4.7	1778.2 ±0.3	<MDL

Minimum measurable limits of anions: $MDL_{[F^-]}$ = 0.37 ppb; $MDL_{[NO2^-]}$ = 3.66 ppb; $MDL_{[Cl^-]}$ = 1.62 ppb; $MDL_{[SO4]}$ = 2.83 ppb; $MDL_{[Br^-]}$ = 2.50 ppb; $MDL_{[PO4^{3-}]}$ = 6.97 ppb; $MDL_{[NO3^-]}$ = 7.83 ppb. The ICP-OES technique was used to determine the heavy metal contents of the water samples taken from the region. Analysis results are listed in Table 4.

Table 5: Result of ICP-OES analysis in seawater samples.

Analyte	Analysis result: $\mu = X_{ort} \pm 2$ Stdev	Analysis Method	Std. Dev.	LOQ Measurement Limit (µg/L)	Calibration Standard Solution Range (ppb)
Ca	252.6 ± 12.2 mg/L	ICP-OES	6.10	12.96	0.20/0.50/1.0/2.0 mg/L
Mg	822.7 ± 10.9 mg/L	ICP-OES	5.45	1,168	0.20/0.50/1.0/2.0 mg/L
Hg	< Ö.L.	ICP-OES/Hydride	0.07549	0.8480	5.0/10/20 µg/L
As	< Ö.L.	ICP-OES/Hydride	0.26361	1.700	5.0/10/20 µg/L
Sb	< Ö.L.	ICP-OES/ Hydride	0.06259	0.354	5.0/10/20 µg/L

Analyte	**Analysis result: $\mu = X_{ort} \pm 2$ Stdev**	**Analysis Method**	**Std. Dev.**	**LOQ Measurement Limit (µg/L)**	**Calibration Standard Solution Range (ppb)**
Ag	< Ö.L.	ICP-OES / Stand.	0.4484	2.729	25/50/100
Al	< Ö.L.	ICP-OES / Stand.	1.9685	6.256	25/50/100
Co	< Ö.L.	ICP-OES /Stand.	0.2107	1.427	25/50/100
Cr	< Ö.L.	ICP-OES/ Stand.	0.2047	1.428	25/50/100
Cu	2.914 ± 1.4338 µg/L	ICP-OES/ Stand.	0.7169	2.914	25/50/100
Fe	15.19 ± 0.46 µg/L	ICP-OES/ Stand.	0.2303	1.235	25/50/100
Mn	1.073 ± 0.46 µg/L	ICP-OES/ Stand.	0.0331	0.456	25/50/100
Ni	< Ö.L.	ICP-OES/ Stand.	0.454	2.240	25/50/100
Pb	< Ö.L.	ICP-OES/ Stand.	1.361	8.216	25/50/100
V	< Ö.L.	ICP-OES/ Stand.	0.5349	2.890	25/50/100
Zn	5.472 ± 0.284 µg/L	ICP-OES/ Stand.	0.1420	0.7064	25/50/100
Ti	< Ö.L.	ICP-OES/ Stand.	0.0924	1.233	25/50/100
Th	21.26 ± 4.78 µg/L	ICP-OES/ Stand.	2.3900	3.798	25/50/100
U	< Ö.L.	ICP-OES/ Stand.	8.689	65.86	25/50/100
Mo	< Ö.L.	ICP-OES/ Stand.	1.1415	3.985	25/50/100
Cd	< Ö.L.	ICP-OES/ Stand.	0.0000	0.9810	25/50/100

Table 6: General quality criteria of seawater (38).

Various metals	**Maximum allowable result (mg/L)**
Copper	0.01
Cadmium	0.01
Chromium	0.1
Lead	0.1
Nickel	0.1
Zinc	0.1
Mercury	0.004
Arsenic	0.1
Ammonia	0.02

By using the ion chromatography system in conjunction with the ICP-OES system, a much more sensitive and qualified element analysis was performed. According to the results of elemental analysis with ICP-OES, Hg, As, Sb, Ag, Al, Co, Cr, Ni, Pb, V, Ti, U, Mo, and Cd elements were below the measurable limit. The fact that elements such as mercury and cadmium, which are toxic to living beings, are present in very low concentrations indicates that heavy metal pollution does not pose a risk to the ecosystem of the region. It is also seen that the seawater is rich in magnesium and calcium. The elements copper and zinc were determined to be above the maximum limits according to the Water Pollution Regulation.

SEM-EDAX Analysis Results

In this part of the study, count density and elemental weight percentages (wt%) of sediment samples were determined by SEM imaging and EDAX (Energy dispersive X-ray spectroscopy) analysis. During the analysis, the relationships to each other of element groupings selected differently for each sample were examined. The SEM acceleration voltage chosen was 20 kV for all images of the sediments in order to keep the working conditions standard for all samples. SEM images were obtained at 100 times magnification on Sample ALT-2 (Figures 3, 5, 7, 9, 11). Then, the densities of a few elements selected as a result of the EDAX analysis were calculated (Figures 4, 6, 8, 10, 12).

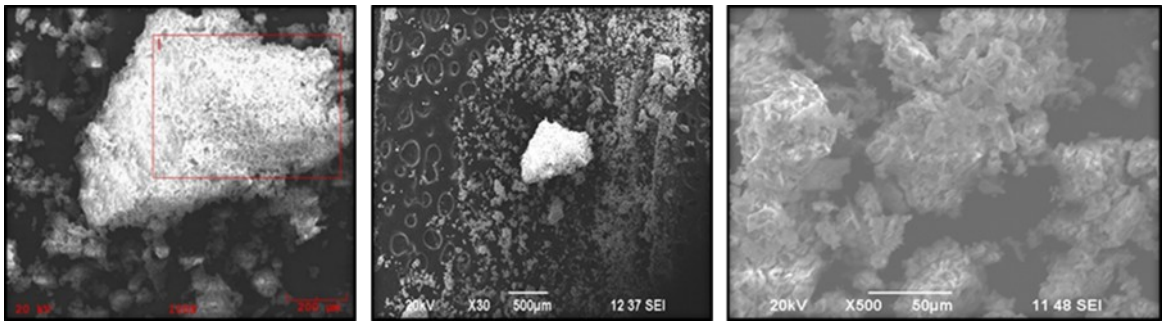

Figure 3: SEM images of Sample ALT-2.

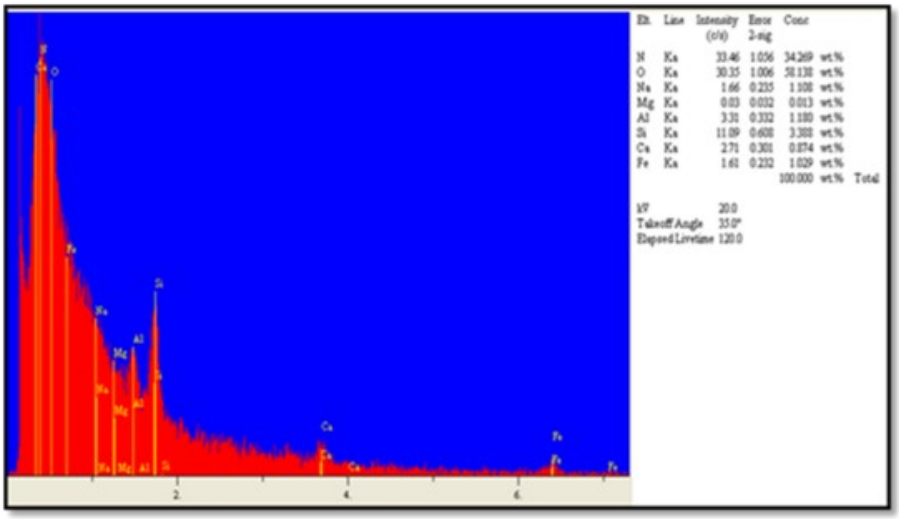

Figure 4: Results of EDAX spectrometric analysis of Sample ALT-2.

SEM images were obtained by 30X magnification of Sample ALT-3 (Figure 5). Then, in the EDAX analysis query, the densities of several selected elements were calculated (Figure 6).

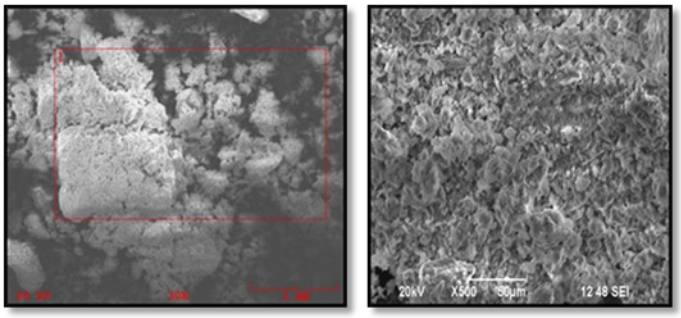

Figure 5: SEM images of Sample ALT-3.

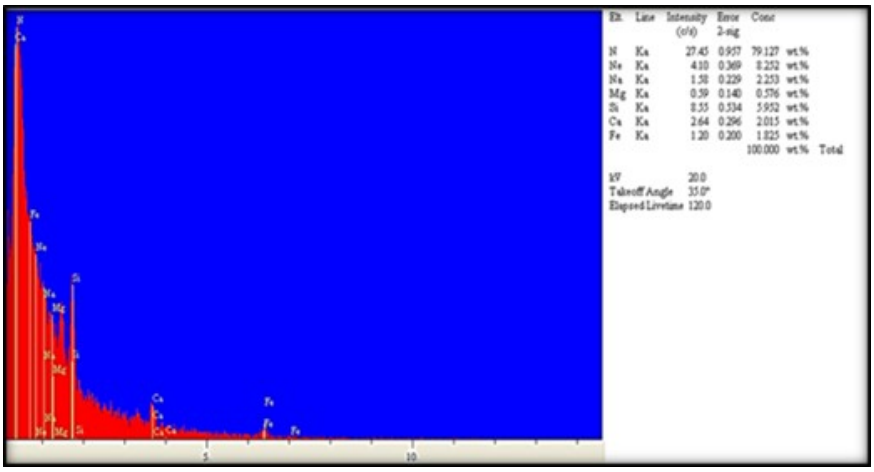

Figure 6: Results of EDAX spectrometric analysis of Sample ALT-3.

SEM images were obtained by magnifying Sample ALT-4 by 30 times (Figure 7). Then, in the EDAX analysis query, the densities of several selected elements were calculated (Figure 8).

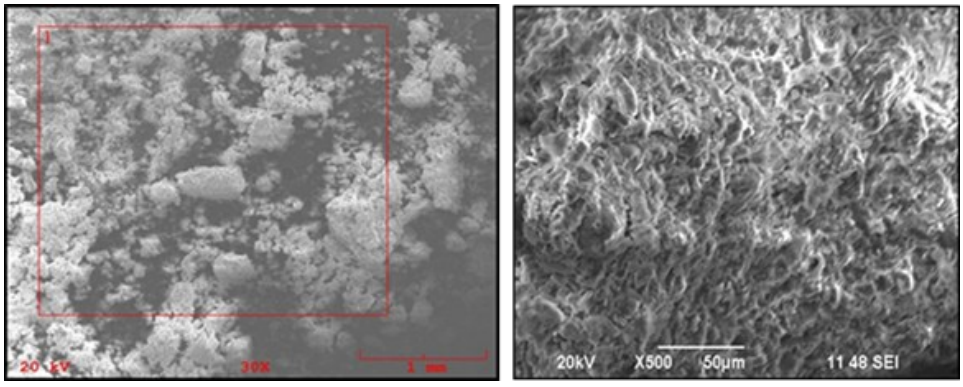

Figure 7: SEM images of Sample ALT-4.

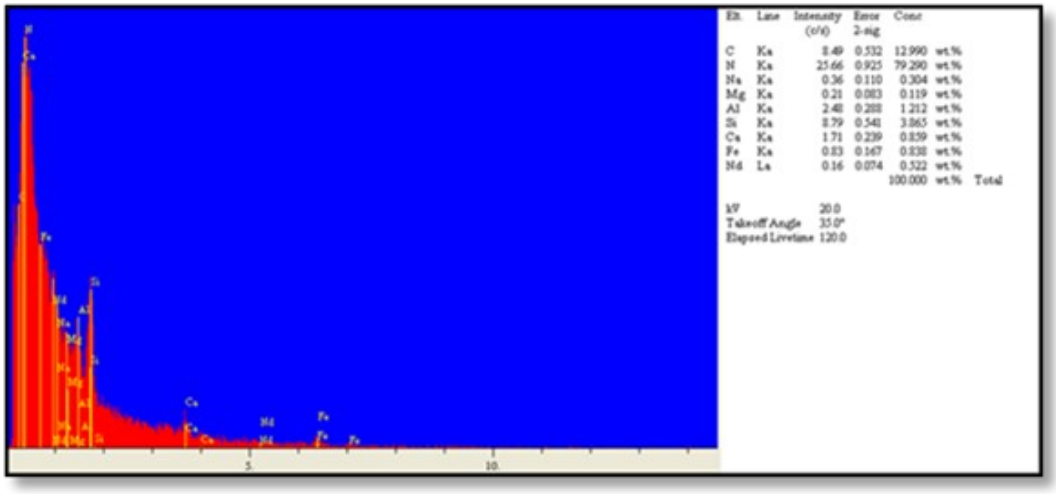

Figure 8: Results of EDAX spectrometric analysis of Sample ALT-4.

SEM images were obtained by 30X magnification of Sample ALT-5 (Figure 9). Then, in the EDAX analysis query, the intensities of several selected elements were calculated (Figure 10).

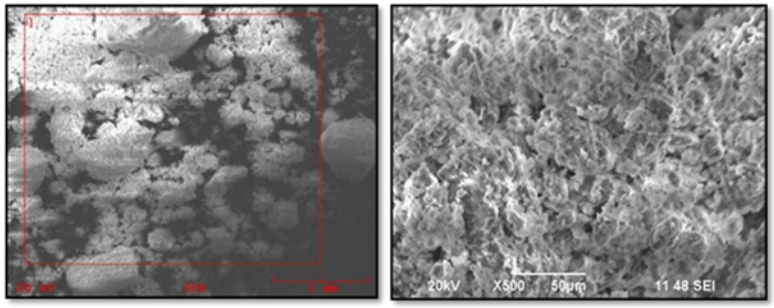

Figure 9: SEM images of Sample ALT-5.

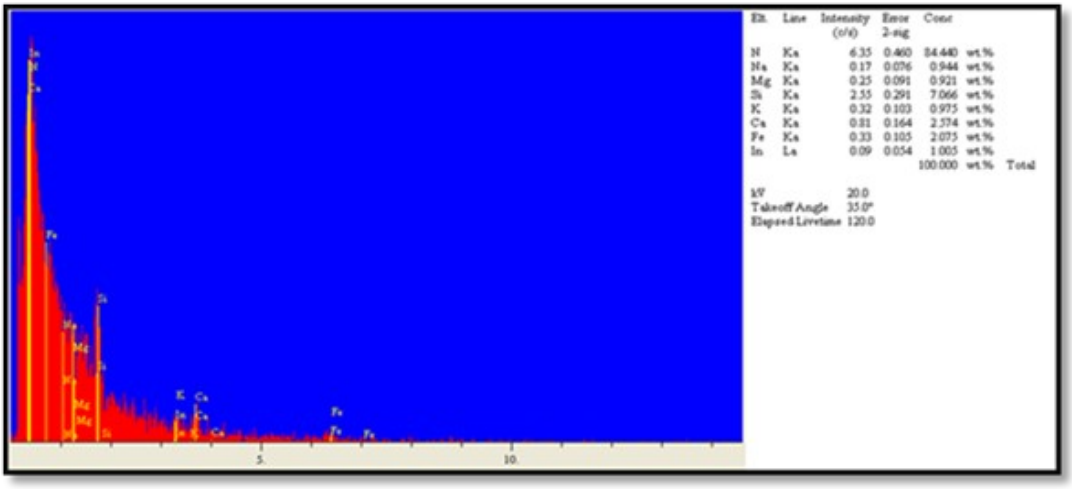

Figure 10: Results of EDAX spectrometric analysis of Sample ALT-5.

SEM images were also obtained by magnifying Sample ALT-6 by 30 times (Figure 11). Then, in the EDAX analysis query, the densities of several selected elements were calculated (Figure 12).

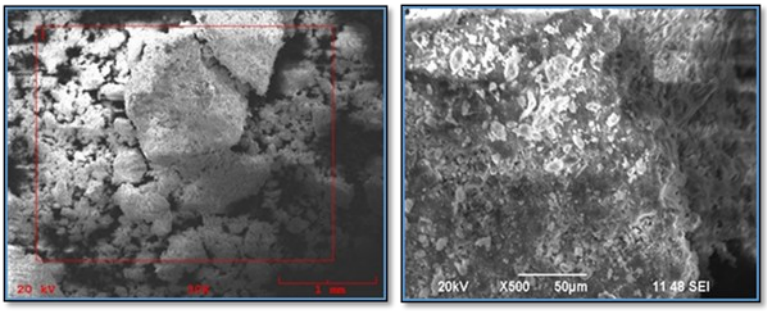

Figure 11: SEM images of Sample ALT-6.

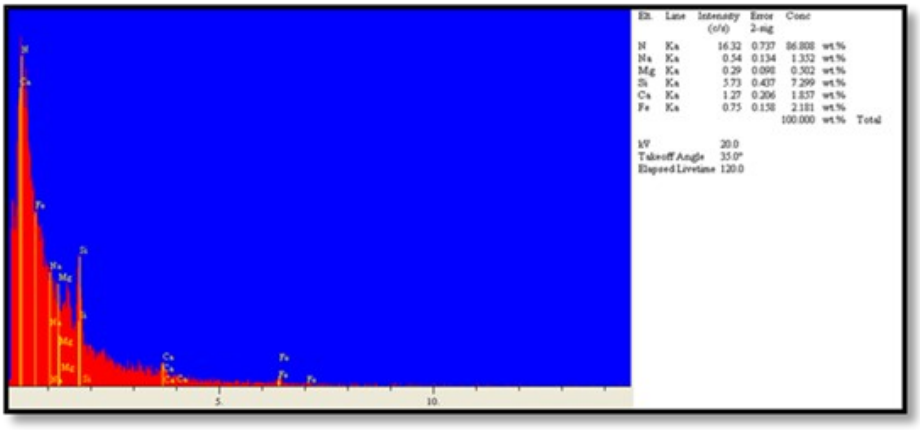

Figure 12: Results of EDAX spectrometric analysis of Sample ALT-6.

Finally, SEM images were obtained by 30X magnification of Sample ALT-8 (Figure 13). Then, as a result of the EDAX analysis, the densities of several selected elements were calculated (Figure 14).

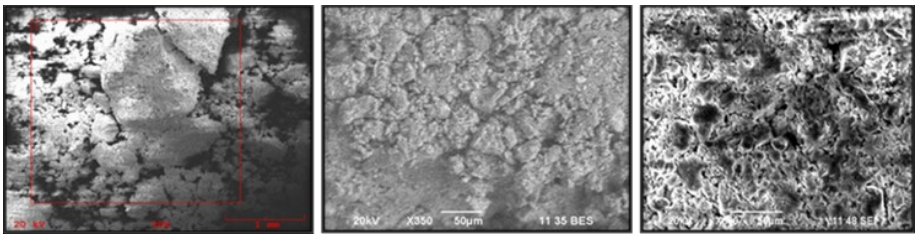

Figure 13: SEM images of Sample ALT-8.

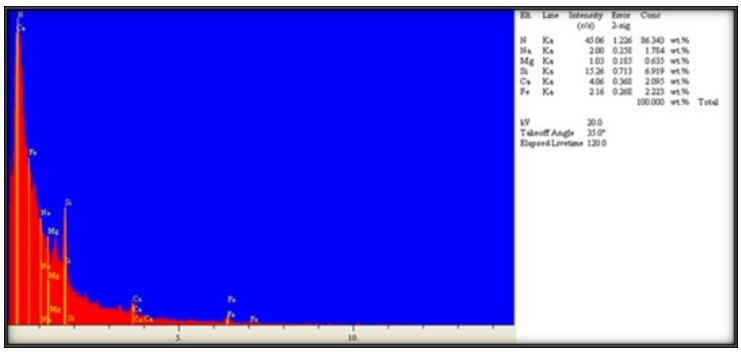

Figure 14: Results of EDAX spectrometric analysis of Sample ALT-8.

The selected elements in the analysis of Samples ALT-6 and ALT-8 were kept constant in the study and were compared for better interpretation of the SEM analysis. It was observed that the elemental abundance in Sample ALT-8 was higher. According to the analysis results, the element with the highest count was determined to be nitrogen (N). That situation is explained by the facts that some of the sediments are composed of organic materials and that the proteins in organic materials have nitrogen in their structures. Nitrogen also may have originated from the fertilizers used in the large agricultural lands in the hinterland of the study area. Another element that stands out in the results is silicon (Si). Silicon is one of the most abundant elements in nature, as 27.7% of the earth's crust by weight consists of silicon. Silicon, which is of vital importance for plants, is used in fertilizers to produce quality products on agricultural lands. It is thought that some of the silicon detected in the sediments may have originated from fertilizers (39). The percentage of the element neodymium (Nd) in the elemental composition of Sample ALT-4 sediment was investigated. Neodymium is one of the rare earth elements. According to the SEM results, the count was 0.16 c/s, while the XRF results were calculated as 0.22 c/s, so it is seen that the concentration results are almost in agreement with each other (40). When the SEM images were examined in terms of their topographic features, it was concluded that the sediments were not comprised of standard element compositions, which resulted in the images having complex structures.

CONCLUSION

In the study, heavy metal ratios were measured in sediment and seawater samples taken from the coasts of Altınova shipyards region of Yalova province. Heavy metal analyses were carried out in the sediment by XRF. In addition, the morphological and topographic properties of the sediments were obtained by SEM. Heavy metal contents in seawater were determined by ICP-OES. The study evaluated the extent to which the marine ecosystem may have been affected by the shipyard activities that have been actively carried out in the region for the past ten years. In future environmental studies, researches about this region will serve as a reference. Contamination on the coastline of the Altınova region originates from the regional sewage treatment plant, industrial zones, ferry piers, residences, a small number of restaurants, and, most importantly, the shipyards operating along the coast. In the qualitative analyses of the sediment content done with XRF, both elemental and compound determinations were made. As the sediments are both organic and inorganic deposits, the elements and compounds found in elemental analysis are quite diverse. According to the results of the analysis, heavy metals that are toxic for humans (Arsenic poses a threat to the human body after 1 mg. Mercury and Cadmium are toxic even at very low concentrations such as 0.001 - 0.1 ppm. The lethal dose for mercury is in the range of 10 - 60 mg/kg. When lead is above 80mg/dl in the blood, it has toxicity (41).), such as cadmium (max 17.984 ppm), lead (max 31.302 ppm), nickel (max 71.725 ppm) and arsenic (max 13.852 ppm) were detected. By adding the ion chromatography system to the ICP-OES system, a much more sensitive and qualified element analysis was performed.

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