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DETERMINATION OF WATER QUALITY PARAMETERS IN IZMIT BAY (MARMARA SEA) AND CONCENTRATIONS OF HEAVY METALS BY ICP-OES

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Abstract

In this paper, seawater heavy metal concentrations (Al, As, Cu, Ba, Zn, Fe, Cd, Co, Cr, Pb, Mn, Mo and Ni) measured by ICP-OES and water quality parameters (NH_4-N, NO_3^-, N, NO_3, PO_4-P, PO_4^{-3}, suspended solids, temperature, conductivity, pH and dissolved oxygen) were evaluated in Izmit Bay (The Marmara Sea). The study was carried out seasonally between December 2016 and September 2017 at four separate sampling stations. The mean concentrations of heavy metals in mg L^{-1} were: Al: 0.074, As: 0.020, Cu: 0.020, Ba: 0.043, Zn: 0.091, Fe: 0.253, Cd: 0.010, Co: 0.022, Cr: 0.010, Pb: 0.010, Mn: 0.291, Mo: 0.013 and Ni: 0.014. The mean annual value of water quality parameters were: NH_4-N: 0.19, NO_3-N: 1.79, NO_3^-: 8.16, PO_4-P: 0.20, PO_4^{-3}: 0.37, suspended solids:16.61 mg L^{-1}, temperature: 16.47 °C, conductivity: 38.19 µS/cm, pH: 7.52 and dissolved oxygen: 9.52 mg L^{-1}. Generally, when the physical and chemical parameters measured at the sampling stations were evaluated according to above-ground water quality regulation, the water quality was Class II, as defined by national regulations. The correlation between physico-chemical parameters and heavy metals measured at the research stations was investigated. There was a negative correlation between NO_3^- and both Cu and Mn (p<0.05). There was also a positive correlation between suspended solids and As (p<0.05). concentrations of Fe and Mn values were found to be high at the 2st and 4nd stations. This is probably due to the fact that there are many maritime port facilities around the stations and intensive port transportation. Other heavy metal concentrations were found to be in accordance with the criteria..

Key words: heavy metals, ICP-OES, Izmit Bay, physico-chemical parameters

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1. Introduction

The seas have been receiving environmental pollutants for years, which is recognized as a significant problem, particularly in relatively enclosed sea systems such as the Mediterranean and Marmara seas. Pollution sources in the Marmara Sea and associated areas such as Izmit Bay and Gemlik Bay include direct discharges and transport by rivers, agricultural operations, atmospheric precipitation, commercial shipping and leakage discharges. Izmit Bay has an economic value in terms of residential area, commercial sea transportation and industrial activity. However, the aquatic system is also very important ecologically and biologically. Izmit Bay is located between 29° 37.16' and 29° 56.70' E and 40° 39.73' and 40° 49.46' N. The surface area extends to about 261 km^2 area. This bay is located between the cities of Istanbul and Kocaeli and there are many different industrial activities around Izmit Bay. The Bay constitutes the geographical area and the east of the Marmara Sea. Industrial investment, urban expansion, intensive industry and the recent growth of the shipbuilding industry are negative pressures on coastal areas and marine ecosystems. Izmit Bay is at risk of the effects of intense industrialization and 12,000-

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15,000 ship traffic every year (Eyüpoğlu and Eyüpoğlu, 2020). There are 15 organized industrial zones, 2200 industrial establishments, 4 technoparks and research centers and 1 university in Kocaeli. In addition to road and rail transport, maritime transport through the ports located around the bay is also very important (Kahraman et al., 2013).

As a result of these activities and environmental pollutants flowing into aquatic environments, physico - chemical and changes have occurred in the ecosystem. In particular, heavy metals are present which are toxic when they exceed their limit values. Investigation of heavy metal pollution in marine ecosystems is very important for both the environment and human health. Many heavy metals may be pollutants and these include Aluminium (Al), Arsenic (As), Barium (Ba), Cadmium (Cd), Chromium (Cr), Cobalt (Co), Copper (Cu), Iron (Fe), Lead (Pb), Magnesium (Mg), Manganese (Mn), Molybdenum (Mo), Nickel (Ni) and Zinc (Zn). There are many studies of heavy metal pollution in aquatic ecosystems. In a study in the bay of İskenderun, it was concluded that in the prevention of a heavy metal pollution threat, the diversity of organisms living in ecosystem should be taken into consideration and protective measures should be taken as soon as possible (Türkmen and Aras, 2011). A study conducted in Sapanca lake, close to the end of Izmit Bay, investigated the concentrations of the heavy metals Cd, Cr, Cu, Fe, Pb, Mn, Ni, and Zn in the water. These parameters were at acceptable levels according to WHO (World Health Organization) and TS 266 (Turkish Standard, 2005) water standards (Karacoban et al., 2017). However, in a study of the Sakarya River, beryllium and thallium concentrations exceeded the limits given by the US Environmental Protection Agency. Sakarya River sediments were also polluted by Antimony, Tin, Rhodium and Selenium (Dundar and Altundağ, 2018).

Metal pollution (Cd, Cu, Pb, Zn) was determined in harbor sediments on the Island of Lesvos, while no evidence of contamination was detected in the wider coastal zone (Aloupi and Angelides, 2001). Other studies have reported heavy metal levels in the North Aegean Sea with concentrations of Cd; 0.01-0.4, Cu; 0.74–5.93 and Mn; 0.07-44.98 µg/L in the South Aegean Sea with Cd; 0.01-0.17, Cu; 0.48-5.19 and Mn; 0.18-9.31 µg/L and around the island of Favignana, Italy, where concentrations were Cd; 98-183, Cr; 86-144, Cu; 0.51-0.82, Pb; 0.32-0.92 and Zn; 1.76-5 µg/L (Trefry et al., 1996; Voutsinou-Taliadouri et al., 2000).

An earlier study in Izmit Bay showed that discharges of wastes into the surface waters significantly affected biological production and oxygen consumption in the lower layer (Morkoç et al., 2001). Sediment quality standards in Izmit Bay were previously investigated and Cd, Pb and Zn concentrations exceeded the limits in areas where freshwater streams carrying the pollution load entered the bay. A further study of Izmit Bay sediments reported As, Cd, Pb, and Zn concentrations to be raised while Ba, Co, Cr, Cu, Fe, Mg, Mo and Ni concentrations were at background levels. Sediments from the northern coast of Izmit Bay are polluted because both industrial and residential areas are concentrated here and subsequently discharge into the waters of the northern shore. Organic carbon concentrations are also found to be elevated in bay sediments. Studies have shown that road traffic run-off, the paint industry and coal combustion are among the most significant sources (Morkoç et al., 1994; Morkoç et al., 2008; Pekey, 2006; Taşdemir, 2002). After 2004, collection of ship waste, sea surface cleaning, feeder stream water quality improvement and monitoring of seawater quality have been carried out regularly by the Metropolitan Municipality (Kahraman, et al., 2013).

In this study, the heavy metal concentrations and water quality parameters were determined in seawater sampled from Izmit Bay over a period extending from December 2016 to September 2017 from four separate sampling stations. In addition, the relationship between water quality parameters and heavy metal concentrations was determined.

2. Materials and methods

2.1. Apparatus

Inductively coupled plasma optical emission spectrometry (ICP-OES) (Spectro Analytical Instruments, Kleve, Germany) was used for the measurement of elemental concentrations. ICP-OES is an efficient technique, able to simultaneously measure a wide range of metals, including refractory metals and some non-metals. ICP-OES systems have now become the analytical method of choice for a wide range of applications.

2.2. Study area

Izmit Bay is 50 km. long and 1.8-9 km. wide and has a surface area of 261 km^2. The eastern section, the smallest component of the entire system, is about 15 km. in length and relatively shallow with a maximum depth of about 35 m. The central section of Izmit Bay, being the largest component of the system, is about 20 km long and the bottom topography varies considerably in a north-south direction; its northern part is relatively shallow with an average depth of about 60 m. increasing to approximately 180 m towards the southern section.

Fig. 1 shows the location of the four sampling stations graphically. Station 1 was located on the Tavşancıl coast of the bay. This station was close to public beachfront houses used in the summer. Station 1 coordinates were 40° 40' 03" N and 029° 34' 12" E. Station 2 was located on the Derince coast, close to a heavily industrialized area and was surrounded by a recreational park area with the coordinates 40° 45' 01" N and 029° 48 '43" E. Station 3 was located in an area of beachfront cafes on the south coast of Izmit Bay with the coordinates 40° 44' 11"N and 029° 56' 30" E.

Station 4 was located on the Kavaklı coast with the coordinates 40° 43' 32" N and 029° 50' 17" E.

2.3. Sample collection and analytical procedure

Water samples were collected from December 2016 to September 2017, in December, May, August and September. A total of 16 collections were conducted as samples were taken every season at each of the four sampling stations. Water samples for chemical analysis were taken from the coastal area into two liter dark glass bottles. Seawater temperature, dissolved oxygen, conductivity and pH were measured in the field with portable instruments (IQ Scientific Instrument Inc., California, USA). Analysis of other water quality parameters, including NH_4-N, NO_3-N, NO_3, PO_4-P, PO_4^{-3} and suspended solids, were determined in the laboratory using standard techniques and methods (APHA, 1999).

For heavy metal analysis, water samples were placed in 500 ml. colored bottles and 5 ml. of nitric acid was added, mixed and then stored in the freezer at -20 ºC until analysis. The trace metals in seawater were chelated with appropriate organic matter at an appropriate pH. Then, using a suitable organic solvent, the chelates were drawn from the water phase into the organic phase. After proper pretreatment (Hot Plate Heating, Microwave Pretreatment), metals were analyzed by ICP-OES. In this study, the EPA METHOD 6010 C - ICP OES analysis method was applied in the environmental laboratory of Barem.

2.4. Statistical analysis

Means were compared using Duncan's multiple range test at a significance level of $p<0.05$. Correlation analysis was performed to determine the relationship of metal concentrations with each other and the physico-chemical parameters that were analyzed. All statistical analyzes were performed using SPSS (IBM Inc., Armonk, NY, USA) and statistical package programs as previously described (Ozdamar, 1999).

3. Results and discussion

One of the most important parameters for living creatures in the seas is dissolved oxygen. Oxygen solubility in water is inversely proportional to the temperature and salinity. The annual ranges of dissolved oxygen concentration was 8.10 - 12.70 mg L^{-1}, water temperature was 10.11- 24.22 °C and salinity was 22‰ - 28‰. This study confirmed that the dissolved oxygen concentration was lower when water temperature and salinity values increased and this was especially evident in summer season sampling. The highest oxygen value was 12.70 mg L^{-1} at Station 1 in May. We hypothesize that phytoplankton activity, which increased during the spring period, also contributed to this situation and our findings are in line with those of a previous study conducted in Izmit Bay (Küçük, 2012). The temperature of seawater in coastal areas depends mainly on the season and degree and strength of incident sunlight but is also affected by winds and currents (Kocataş, 1999). In the present study seawater temperature was the lowest (10.11 °C) at all stations in December and the highest value (24.22 °C) was measured at Station2 in August. Seawater temperatures in our study were within the expected ranges for the season of measurement.

Atmospheric activity, water mixing (seawater, freshwater run-off and rain), temperature and photosynthetic activity are known to all affect the pH of seawater. The normal pH of seawater was reported to be between 7.5 and 8.4 (Ozyurt et al., 2001). In this study pH values varied from pH 7.45 to pH 8.25 during the year and thus was slightly alkaline throughout the year.

The highest pH value was measured at Station 3 in the winter when the water started to cool. This may be due to reduced absorption of atmospheric CO_2 into the seawater because of the difference in air and water temperature. The electrical conductivity, an indicator of the total ion content of seawater, is also very important for aquatic life.

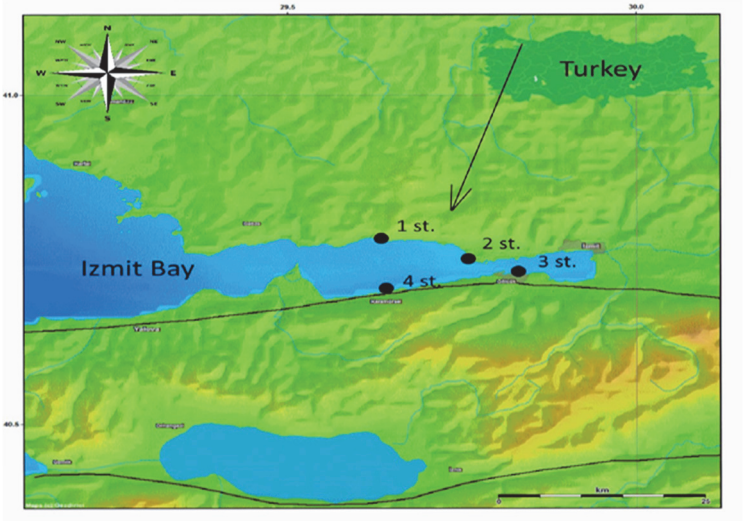

Fig. 1. The study area in Izmit Bay (http://www.koeri.boun.edu.tr.sismo.map)

In this study, electrical conductivity was highest in August at Station 2 (41.80 µS / cm.) and the lowest in December at Station 4 (30.20 µS / cm.). Again, the conductivity values measured in this study are compatible with the results of a previous study conducted in the same area (Küçük, 2012).

In addition to the dissolved oxygen, temperature, pH and conductivity measured, other annual parameters measured at each station were: NH_4-N (mg L^{-1}) 0.09 – 0.50; NO_3-N (mg L^{-1}) 2.10 – 1.30; NO_3^- (mg L^{-1}) 10.20 – 6.40; PO_4-P (mg L^{-1}) 0.11 – 0.45; PO_4^{-3} (mg L^{-1}) 0.15 – 1.30; and suspended solids (mg L^{-1}) 0.80 – 33.50. The N/P ratio varied between 21.18 - 66.53, with the highest ratio being measured at Station 3. This result t Station 3 is thought to be due to the low circulation of water plus nitrate and phosphate input from fishermen's shelters located around the sampling station. The amount of suspended solids in Izmit Bay, as elsewhere, varies according to the two-layer movement of seawater and seasonal characteristics (Algan et al., 1999). The highest mean concentration of suspended solids was measured at Station 2 at 33.50 mg L^{-1}, probably because the research station was close to settlement areas, with both natural and anthropogenic input (Fig. 2). Heavy metal contamination in water may be caused by both natural and human activities. The anthropogenic pollution load consists of both domestic and industrial waste (Altug and Balkis, 2009). Rapid industrial and residential expansion around Izmit Bay has caused pollution an increase in anthropogenic pollution in this region.

In addition, Izmit Bay is also under environmental pressure from heavy shipping and existing large-scale heavy industry. This situation has been shown to have contributed to bad water quality and caused eutrophication in İzmit Bay (Tufekci et al., 2010). Many aquatic organisms need trace amounts of some metals such as Cu, Fe, Co, Ni, Zn, and Mn. However, when present in increased concentrations these metals become toxic to aquatic organisms and whole ecosystems (Balkıs et al., 2016). The present study evaluated heavy metals concentrations in seawater from all time points and sampling stations. Increased concentrations of Ba, Zn, Fe and Mn values were found at Station 2 and station 4. Zn, Ba, Fe and Mn come mainly from industrial and terrestrial sources. These stations are associated with a high density of maritime port facilities, particularly around Station 2, with accompanying intensive port transportation including both maritime and road transport (Fig. 3).

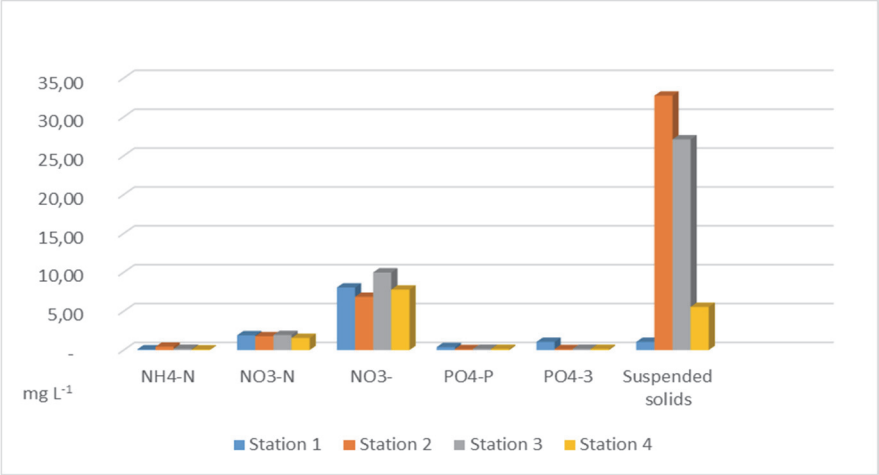

Fig. 2. Mean annual values of physico-chemical parameters in Izmit Bay

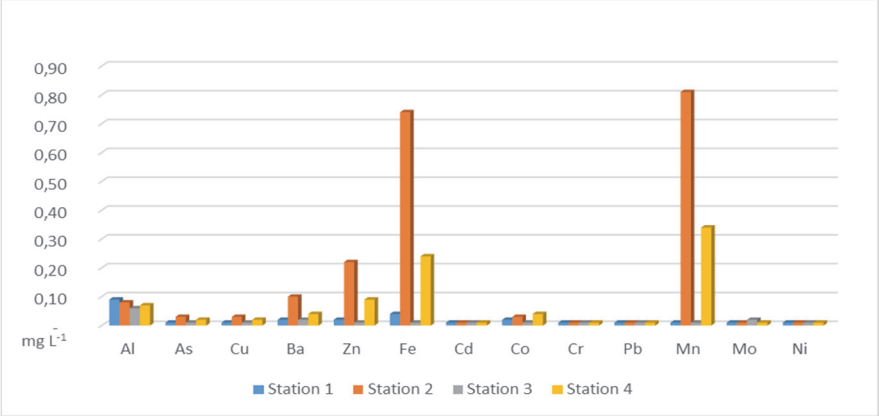

Fig.3. Average annual values of heavy metals in seawater

Heavy metal concentrations differed from station to station. Overall, the mean concentrations of Mn (0.291 mg L^{-1}) was found to be high according to national and international limits including the Institute of Turkish Standards, the European Union, the World Health Organization and the US Environmental Protection Agency (EC Directive, 1998; EPA, 2009; TS 266, 2005; WHO, 2003). In a previous study of the metal concentrations in the surface sediment of the seabed of Izmit bay, it was reported that the sediment was highly contaminated. These authors concluded that this was due to the intense port and shipyard activities in the region (Eyüboğlu and Eyüboğlu, 2020). In aquatic systems, the sediment layer, which over time may become saturated in terms of metal content, will then tend to release the metals back into the water body again (Metin Dereli et al., 2017). We believe that the high concentrations of Mn detected in our study may be due to this mechanism. Other heavy metal concentrations were found to be within national and international limits. In earlier studies concentration ranges of Fe (<4 - 21 mg L^{-1}), Mn (<1 - 123 mg L^{-1}) and Cu (0.4 - 7.4 mg L^{-1}) were reported to be high. However, Cd concentrations were lower than the detection limit of the method (<0.01 mg L^{-1}) along the water column of the Bay (Balkıs et al., 2016). Our results for Cd are in line with the findings of Balkıs et al. (2016). In research conducted on the Dil İskelesi shore of Izmit Bay, concentrations ranges of Zn, Fe, Mn and Pb were 0.007 - 0.033, 0.018 - 0.003, 0.086 - 0.033 and <0.01 µg ml were reported, with metal concentrations being similar although Fe and Mn concentrations showed a summer peak (Ergul et al., 2010). The difference between stations for measured heavy metal concentrations may be due to several factors including differences in volume of industrial wastes, transport to the aquatic system, marine logistics, industrial flue gases, meteorological factors and environmental factors. Comparison of mean measured metal concentrations from Izmit Bay water with national and international standards is given in Table 1.

Spearman correlation coefficients of metal concentrations in seawater were investigated between pairs of metals. A positive correlation was found between the following pairs (p<0.05): Cu-As; Ba-Al; Ba-As; Ba-Cu; Zn-Al; Zn-As; Zn-Cu; Zn- Ba; Fe-As; Fe-Cu; Fe-Ba; Fe-Zn; Co-Al; Co-As; Co-Cu; Co-Ba; Co-Zn; Co-Fe; Mn-As; Mn-Cu; Mn-Ba; Mn-Zn; Mn-Fe; and Mn-Co. A negative correlation (p<0.05) was found between the following: Mo-Al; Mo-Cu; Mo-Ba; Mo-Zn; Mo-Fe; Mo-Co; Mo-Mn. There was no correlation between Cd, Cr, Pb and Ni concentrations and other metals which have been excluded from the data shown in Table 2. Positive correlations between heavy metals also indicate that water pollution comes from common sources. This sources are as industrial, soil, traffic, urban dusts and similar environmental factors.

When the correlation between physico-chemical parameters and heavy metal concentrations was investigated, a negative correlation of NO_3^- with Cu and Mn (p<0.05) was identified. In addition, there was a positive relationship between suspended solids and As (p<0.05) (Table 3). The transport of heavy metals in aquatic environments, dissolution, sedimentation, absorption and accumulation in aquatic organisms is a very complex process. Metals may be affected by many changes and transformations, which depend on the physical and chemical nature of water. In these reactions, the redox potential and pH of the aquatic environment also have an effect. Metals can also be present in the aquatic environment in the elementary form or as compound colloids, particles, dissolved cations or compounds (complex). Generally, it has been reported that the solubility of metals increases at lower pH and decreases at higher pH (Metin Dereli et al., 2017). However, we found no significant correlation between pH and the metals tested and the mean annual pH was to the alkaline side of neutral. The highest positive correlation was identified between As and suspended solids. This can be explained by the presence of metals in water, in colloids or particles in compound form (Table 3).

Table 1. Comparison of Izmit Bay water with national and international standards (mg L^{-1})

Elements	***This Study***	***Institute of Turkish Standards***	***European Union***	***World Health Organization***	***US Environmental Protection Agency***
Al	0.074	0.2	0.2	0.2	0.2
As	0.020	0.01	0.01	0.01	0.01
Cu	0.020	2	2	2	1.3
Ba	0.043			0.70	2
Zn	0.091				5
Fe	**0.253**	0.2	0.2	0.3	0.3
Cd	0.010	0.005	0.005	0.003	0.005
Co	0.022				
Cr	0.010	0.05	0.05	0.05	0.1
Pb	0.010	0.01	0.01	0.01	0.015
Mn	**0.291**	0.05	0.05	0.4	0.05
Mo	0.013				
Ni	0.014	0.02	0.02	0.02	

Table 2. Spearman correlation coefficients in seawater

Sea Water		*Al*	*As*	*Cu*	*Ba*	*Zn*	*Fe*	*Co*	*Mn*	*Mo*
Al	Correlation	1.000								
	Significance									
As	Correlation	0.559	1.000							
	Significance	0.024								
Cu	Correlation	0.361	**0.866**	1.000						
	Significance	0.169	0.001							
Ba	Correlation	**0.779**	**0.823**	**0.778**	1.000					
	Significance	0.001	0.001	0.001						
Zn	Correlation	**0.670**	**0.790**	**0.776**	**0.882**	1.000				
	Significance	0.005	0.001	0.001	0.001					
Fe	Correlation	0.492	**0.712**	**0.850**	**0.829**	**0.747**	1.000			
	Significance	0.053	0.002	0.001	0.001	0.001				
Co	Correlation	**0.729**	**0.641**	**0.627**	**0.819**	**0.698**	**0.680**	1.000		
	Significance	0.001	0.008	0.009	0.001	0.003	0.004			
Mn	Correlation	0.340	**0.850**	**0.992**	**0.768**	**0.778**	**0.839**	**0.642**	1.000	
	Significance	0.197	0.001	0.001	0.001	0.001	0.001	0.007		
Mo	Correlation	**-0.824**	**-0.473**	**-0.505**	**-0.749**	**-0.658**	**-0.661**	**-0.834**	**-0.504**	1.000
	Significance	0.000	0.064	0.046	0.001	0.006	0.005	0.000	0.047	

***** *Bold values indicate a significant statistical relationship (p<0.05)*

Table 3. Spearman correlation coefficients between physico-chemical parameters and heavy metal concentrations

Parameters	*Al*	*As*	*Cu*	*Ba*	*Zn*	*Fe*	*Co*	*Mn*	*Mo*
NH_4-N	0.169	0.371	0.288	0.287	0.221	0.238	-0.033	0.248	0.123
NO_3-N	0.327	-0.125	-0.314	0.098	0.002	-0.128	0.039	-0.361	0.042
NO_3^-	-0.087	-0.468	**-0.660**	-0.427	-0.285	-0.617	-0.418	**-0.665**	0.438
PO_4-P	0.282	-0.386	-0.370	-0.048	0.002	-0.138	-0.100	-0.421	-0.237
PO_4^{-3}	0.284	-0.385	-0.369	-0.039	-0.097	-0.078	-0.100	-0.420	-0.237
Suspended solids	-0.045	**0.516**	0.427	0.214	0.170	0.237	-0.043	0.395	0.287
Temperature	0.155	-0.023	-0.195	-0.007	-0.254	-0.181	0.044	-0.268	0.069
Conductivity	-0.431	-0.253	-0.080	-0.035	-0.378	-0.142	-0.359	-0.105	0.314
pH	-0.035	0.065	-0.179	-0.043	-0.152	-0.318	0.044	-0.268	0.069
Dissolved oxygen	0.061	-0.256	-0.367	-0.108	-0.253	-0.377	0.047	-0.359	0.014

**Bold values indicate that there is strong statistical relationship (p<0.05)*

Generally, when the physical and chemical parameters measured at the sampling stations were evaluated according to above-ground water quality regulation, the water quality was Class II, as defined by national regulations (Turkish Regulations, 2012).

4. Conclusion

Heavy metals may increase the contamination of seawater. This leads to an increase in heavy metal concentrations along the food chain in fish and then humans. In this study, it was demonstrated that there was a significant environmental impact from sources such as terrestrial pollution, increased domestic and industrial activities, which affect the physical and chemical properties of seawater from Izmit Bay. In particular, at Station 2, these effects were more prominent, probably due to the station being located in a position more open to environmental impacts with nearby dense residential development and heavy industry. As Izmit Bay is a semi-closed area, close to a high number of industrial establishments, with intensive maritime activity and high shoreline population we believe this has caused an increase in the level of pollutants in the Bay. In this area, because of the high pollution risk, more environmental measures have been applied in the last 10 years. Advanced biological wastewater treatment plants have been built in different parts of the Bay by Kocaeli Metropolitan Municipality.

In recent years the bay has been monitored by air, land and sea equipment by environmental control teams (Kahraman and Ozkul, 2017). It is important that these activities, carried out with local government and related public institutions, are sustainable.

To this end, it has been concluded that raising awareness and participation of the resident population around the gulf concerning environmental protection will be necessary to achieve and maintain a cleaner İzmit Bay.

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