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Research Article **Araştırma Makalesi**

Heavy Metal Contamination Status and Risk Assessment in Surface Sediments of Köyceğiz Lagoon Estuary System (KLES) (South-West Anatolia)

Köyceğiz Lagün Östarin Sisteminde (Güney-Batı Anadolu) Yüzey Sedimentlerinde Ağır Metal Kirliliğinin Durumu ve Risk Değerlendirmesi

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Abstract: This study reports the presence of heavy metals (Cr, Ni, Cu, Cd, Pb) in the sediment and water column in the Köyceğiz Lagoon Estuary System (KLES) (South-West Anatolia). The mean heavy metal concentrations (µg L^{-1}) in the water were found as Cr: 3.72-8.36, Ni: 6.51-9.48, Cu: 1.29-11.88, Cd: ND-0.08, Pb: ND-0.34. The heavy metal concentrations in water samples were found to be below the internationally acceptable limits. The mean heavy metal concentrations (mg kg^{-1}) in the sediment were found as Cr: 1.75-4.25, Ni: 5.24-12.69, Cu: 0.25-0.51, Cd: <0.01, Pb: 0.04-0.13. As a result of the analyses conducted concerning contamination factor (CF), geo-accumulation index (I_{geo}), pollution load index (PLI), monomial potential ecological risk index (ER), and total potential ecological risk index (RI) for heavy metals in the sediment, it was deduced that there was not any pollution and moderate or high ecological risks.

Keywords
- Heavy metal
- Environmental pollution
- Ecological risk
- Lagoon

Özet: Bu çalışmada, Köyceğiz Lagün Östarin Sisteminin (Güney-Batı Anadolu) sediment ve su kolonundaki ağır metallerin (Cr, Ni, Cu, Cd, Pb) varlığı ortaya konmuştur. Sudaki ortalama ağır metal konsantrasyonları (µg L^{-1}), Cr: 3,72-8,36, Ni: 6,51-9,48, Cu: 1,29-11,88, Cd: TE-0,08, Pb: TE-0,34 olarak bulunmuştur. Su örneklerinde belirlenen ağır metal konsantrasyonlarının uluslararası kabul edilebilir limitlerin altında olduğu tespit edilmiştir. Sedimentteki ortalama ağır metal konsantrasyonları (mg kg^{-1}) Cr: 1,75-4,25, Ni: 5,24-12,69, Cu: 0,25-0,51, Cd: <0,01, Pb: 0,04-0,13 olarak belirlenmiştir. Sedimentte belirlenen ağır metal konsantrasyonları yönünden yapılan kontaminasyon faktörü (CF), jeobirikim indeksi (I_{geo}), kirlilik yük indeksi (PLI), monomial potansiyel ekolojik risk indeksi (ER) ve toplam potansiyel ekolojik risk indeksi (RI) analizleri sonucunda, herhangi bir kirlilik tespit edilmemiş olup orta veya yüksek ekolojik risklerin olmadığı sonucuna varılmıştır.

Anahtar kelimeler
- Ağır metal
- Çevre kirliliği
- Ekolojik risk
- Lagün

1. INTRODUCTION

Coastal lagoons are important areas for fish species, and due to their abundant nutrient sources, they are compared to nurseries with optimum nutrition and development for juvenile fish (Whitfield, 1999; Innal and Giannetto, 2020). Being under the impact of both marine and freshwater ecosystems, lagoon areas are very rich aquatic habitats. For this reason, fish, sea turtles, and other aquatic animals use these habitats for feeding (Gilabert, 2001). The lagoon management concept emerged when it was understood that the protection of lagoons is extremely important for the development of aquatic animals. The scope of lagoon management includes protecting the ecological characteristics of lagoons, preventing the depletion of valuable biological resources, and destruction of sensitive habitats (Manzo et al., 2016).

Iztuzu Lagoon has emerged with the set formed by Iztuzu Beach. Located within the borders of Ortaca, Mugla (Southern West Turkey), Iztuzu Beach is a Mediterranean coast with 4.5 km. length. This beach is crescent-shaped and has fine sand. The beach has also lagoons with labyrinth-shaped in its northeastern part through which waters from Köyceğiz Lake reach into the Mediterranean Sea in the west of the beach (Sari et al., 2017). The Times from the UK declared Iztuzu Beach and Lagoon as "The Best Open Space Europe" in 2008 after 6-year research (Anonymous, 2018). In 2013, the beach was selected as the best 7th beach in Europe by TripAdvisor. The beach is one of the important spawning areas of the protected loggerhead turtle *Caretta caretta* and Nile soft-shelled turtle *Trionyx triunguis* species in the Eastern Mediterranean basin. *C. caretta* species is also frequently encountered in the lagoon area. In this special region, sometimes the freshwaters of Köyceğiz Lake, which is located in the north of the lagoon, flows into the seawater, and sometimes seawater flows into the lagoon due to the tide effect in Dalyan mouth location. The lagoon area is overcrowded during the high season (April-October). The transportation from the hotels to the beach is generally provided via boats and this short trip mostly lasts almost 30 minutes. Pollution stress factors for the canal and lagoon are tourism, restaurants, boat traffic, agricultural activities, and transportation from Köyceğiz Lake.

Pollutants in water can be defined as physical, chemical, or biological factors that cause harmful effects on aquatic life and consumers. With the development of urbanization and industrialization, several harmful pollutants are discharged into water (Srivastava et al., 2020). Inorganic and organic pollutants (e.g. nutrients, dioxins/furans, heavy metals) pose serious risks to many aquatic organisms (Scott and Sloman, 2004; Storelli and Marcotrigiano, 2003). Ecological risk assessment is very important to eliminate the negative effects of pollutants in aquatic ecosystems that can affect aquatic life. With the understanding of the importance of ecological risk assessment in recent years, many studies have been conducted in lagoon ecosystems (Maanan et al., 2015; Mendoza-Carranza et al., 2016; Shaheen et al., 2019).

In this study, (1) the heavy metal concentrations detected in the water samples were compared to the internationally acceptable limits. (2) pollution risk assessment in terms of heavy metals was made in sediments by using some indexes and (3) potential ecological risk levels were determined.

2. MATERIAL and METHODS

For heavy metal analyses, surface water and sediment samples were taken with grap (Van Veen type 0.1 m^2 sampling area) twice a year (in winter and summer) from the 5 stations (Figure 1). During the study, the temperature, pH, and salinity values of the lagoon and open sea surface waters were measured with a multiprobe water quality measurement device (YSI Professional Plus).

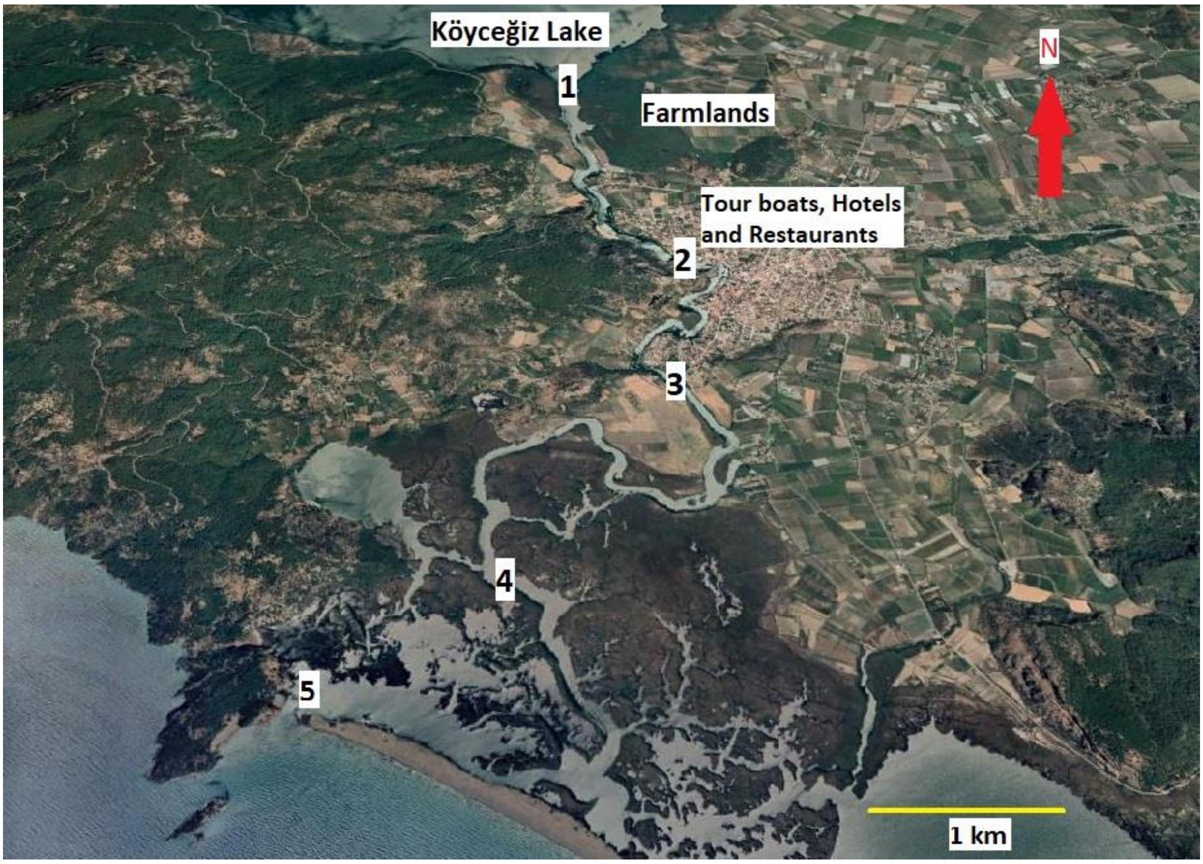

Figure 1. The sampling points for heavy metals.

2.1. Heavy metal analyses in water and sediment samples

The water samples taken from each one of the stations were filtered through Whatman GF/F filter paper in the laboratory, then 15 ml was put into Falcon-type covered tubes. Until the analyses, to prevent metal ions dilution from changing, 5 ml nitric acid (HNO_3 65%, suprapur) per liter was added onto water samples. Water samples were kept in the refrigerator at +4°C until further analysis (ASTM, 1985).

After sediment samples were stirred in a plastic cup, 5 sub-samples with 10 gr. were taken and put into polyethylene containers separately and nitric acid was added onto them (Moody and Lindstrom, 1977). From the samples that had been brought to the laboratory under cool conditions, 0.5 gr for each one of the samples were taken and mineralized in 3 ml $HCl–HNO_3–H_2O$ solution for 1 hour at 95°C. The mineralized sample was then diluted to 10 ml with ultra-pure water and filtered through 0.70 μm GF/F, then it was made ready for the analysis (Sastre et al., 2002). Heavy metal concentrations in the water and sediment samples were determined with Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Agilent 7700x). For elements Cr, Mn, Cu, Zn, Cd, and Pb, the detection limits (LOD) were 0.038, 0.064, 0.016, 0.102, 0.0005, and 0.017 μg kg^{-1}, respectively.

The accuracy and certainty of the results of the heavy metal analysis were checked via standard reference matter (Sigma-Aldrich® CRM016- Fresh Water Sediment 3 for Trace Metals) analysis. The applied method for sediment in determining recovery rate was applied for CRM016-050 with five repetitions. Table 1 presents mean concentrations and recovery rates (%) of certified reference matter used for checking the quality of the applied analytical method in heavy metal analyses. According to the results, a 90% recovery rate was obtained.

Table 1. The analysis results of certificate reference matter (CRM016) for trace metals and recovery rate results (mg kg^{-1}).

Element	CRM016-050	This study	%Recovery
Cr	14.5	13.8 ± 0.8	95.17
Ni	16.7	15.1 ± 1.3	90.42
Cu	15.5	14.2 ± 1.1	91.61
Cd	0.47	0.43 ± 0.07	91.49
Pb	14.1	13.9 ± 0.6	98.58

2.2. The calculation of contamination factor

In the studies which reveal heavy metal contamination in sediment, contamination factor (CF) has been a commonly used method. Although the calculation of CF is extremely simple, it provides strong data about the current situation. The calculation was made according to the formula of Tomlinson et al. (1980) below:

$$CF = \frac{C_{metal}}{C_{background}}$$

In the formula, C_{metal} refers to the obtained metal concentration; $C_{background}$ refers to the estimated mean concentration in sediment for that metal. These reference values are cited from Taylor and McLennan (2001). Contamination factor results were evaluated according to Hakanson (1980) as below:

CF<1 low contamination,
1≤CF<3 moderate contamination,
3≤CF<6 high contamination,
CF>6 very high contamination

2.3. The calculation of the geo-accumulation index

Geoaccumulation index (I_{geo}) is a scale of values used for specifying the level of terrestrial-based heavy metal accumulation in sediment. It was developed by Müller (1969) for revealing anthropogenic modifications in heavy metal concentrations in coastal areas and is calculated via the formula below:

$$I_{geo} = log2\frac{C_{metal}}{1.5 \times C_{background}}$$

In the formula C_{metal} and $C_{background}$ refer to the same concepts as in CF formula. The obtained results were evaluated in relation with 7 levels suggested by Müller (1981). These levels are shown below:

I geo = 0 → level 0→ Unpolluted/practically uncontaminated
= 0-1 → level 1→ Unpolluted to moderated/uncontaminated to moderately contaminated
= 1-2 → level 2→ Moderated polluted/moderately contaminated
= 2-3 → level 3→ Moderated to high polluted/moderately to heavily contaminated
= 3-4 → level 4→ Highly polluted/heavily contaminated
= 4-5 → level 5→ Highly to extremely polluted/heavily to extremely contaminated
= >5 → level 6→ Extremely polluted/ Extremely contaminated

2.4. The calculation of Pollution load index (PLI)

PLI is used for determining pollution load in sediment. It is calculated by the formula below (Tomlinson et al., 1980):

$$PLI = (CF_1 \times CF_2 \times CF_3 \times \ldots \times CF_n) \times 1/n$$

In the formula, CF refers to contamination factor; n refers to metal number. According to the obtained results, for sediment it is suggested as; PLI = 0 “Distinguished”, PLI <1 “No pollution”, PLI >1 “Polluted”.

2.5. Potential ecological risk indexes

The potential ecological risk index developed by Hakanson (1980) was used to evaluate the negative effects of pollutants on the environment and therefore the ecological sensitivity of pollutants. Monomial (for a single element) potential ecological risk index (ER) was calculated according to the formula below:

$$\mathrm{ER} = \mathrm{T}i \times \mathrm{CF}i$$

where CFi is the contamination factor value calculated for metal i, and Ti refers to the toxic response coefficient for metal i. In this study, the toxic response coefficients suggested by Hakanson (1980) were used as Cr = 2, Ni, Cu, Pb = 5, Cd = 30. The obtained ER results were evaluated as ER<40 "low ecological risk", 40≤ER<80 "moderate ecological risk", 80≤ER<160 "considerable ecological risk", 160≤ER<320 "high ecological risk" and ER≥320 "very high ecological risk" (Li et al., 2017).

In addition, total potential ecological risk index (RI) evaluation was made for all studied elements. RI represents the sensitivity of various biological communities and the possible risks posed by heavy metals (Ogundele et al., 2020). RI was calculated according to the formula below:

$$\mathrm{RI} = \sum_{i=1}^{n} \mathrm{ER}$$

where n refers to the number of metals studied. RI values were graded as follows: RI<150 "low ecological risk", 150≤RI<300 "moderate ecological risk", 300≤RI<600 "severe ecological risk" and RI>600 "serious ecological risk" (Hakanson, 1980; Guo et al., 2010; Ogundele et al., 2020).

2.6. Statistical analyses

For the statistical analyses of the data, IBM SPSS version 22.0 was used. To compare stations, one-way ANOVA followed by post hoc LSD was applied.

3. RESULTS and DISCUSSION

The temperature, salinity and pH values measured from the surface of the lagoon and open sea are presented in the Table 2.

Table 2. Minimum and maximum temperature, salinity and pH values of surface water.

	Water Temperature (°C)	Salinity (mg L^{-1})	pH
Lagoon	18.77-19.65	17.92-25.21	9.02-9.33
Open sea	20.75-20.83	34.11-36.29	8.32-8.96

The mean heavy metal concentrations of surface water samples and guideline values based on the WHO (2011), USEPA (1995, 2009), EC (1998) are presented in Table 3.

Heavy metals are accepted as a major anthropogenic contaminant in coastal zones. Due to their toxicity, persistence, and bioaccumulation characteristics, they present a threat to both human beings and aquatic organisms (DeForest et al., 2007; Ruilian et al., 2008). In this study, the highest concentrations in water samples were found as Ni>Cr>Cu>Pb>Cd respectively. In a study conducted in Huelva (Spain) region, in estuaries waters the concentrations were found to be similar to our study as Cu>Pb>Cd (Vicente-Martorell et al., 2009). ANOVA followed by Tukey post hoc LSD tests revealed no statistical differences between stations in terms of heavy metal concentrations in the water samples (p>0.05). It was determined that the heavy metal concentrations detected in the water samples did not exceed the minimum limits set by WHO (2011), USEPA (1995, 2009), and EC (1998) (Table 3).

Table 3. The mean heavy metal concentrations were determined in water samples and guideline values (μg L^{-1} ± s.e.).

Elements	Stations					WHO	USEPA	EC
	1	2	3	4	5	(mg L^{-1})	(mg L^{-1})	(mg L^{-1})
Cr	3.78 ± 0.36	3.72 ± 0.11	4.56 ± 0.28	5.73 ± 0.97	8.36 ± 1.04	0.05	0.1	0.05
Ni	9.48 ± 1.27	7.36 ± 1.05	6.61 ± 0.42	6.51 ± 1.01	8.11 ± 0.91	0.02	0.1	0.02
Cu	4.43 ± 0.61	4.53 ± 0.33	1.29 ± 0.14	1.31 ± 0.27	11.88 ± 2.26	2	1.3	2
Cd	0.08 ± 0.02	*N.D.	*N.D.	*N.D.	0.02 ± 0.004	0.003	0.005	0.005
Pb	0.32 ± 0.09	0.34 ± 0.09	0.32 ± 0.10	*N.D.	*N.D.	0.01	0.015	0.005

*N.D.: not detected.

The heavy metal concentrations in water samples reported from previous studies in the Köyceğiz-Dalyan Lagoon basin are given in Table 4.

Table 4. A comparison of the results of the current study with previous studies in the region (min-max μg L^{-1}).

Element	Location	Concentration	Current study	References
Cr	Dalyan channel	27.76 - 209.60	3.72 - 8.36	Avşar et. al. (2017)
	Köyceğiz Lake	25.82 - 38.57		
	Sülüngür Lake	25.34 - 55.63		
	Alagöl Lake	173.20 - 291.40		
	Kocagöl Lake	37.00		
	Köyceğiz Lagoon System	0.71 ± 0.28		Genç and Yilmaz (2017)
Ni	Dalyan channel	1.00 - 32.00	6.51 - 9.48	Avşar et. al. (2017)
	Köyceğiz Lake	2.00 - 9.00		
	Alagöl Lake	< 0.10 - 101.00		
	Sülüngür Lake	7.00 - 12.00		
Cu	Köyceğiz Lagoon System	0.03		Genç and Yilmaz (2017)
	Dalyan channel	< 0.02	1.29 - 11.88	Avşar et. al. (2017)
	Köyceğiz Lake	< 0.02 - 31.00		
	Alagöl Lake	< 0.02		
	Sülüngür Lake	< 0.02		
Cd	Köyceğiz Lagoon System	1.15 ± 0.34	0.02 - 0.08	Genç and Yilmaz (2017)
Pb	Dalyan channel	14.14 - 169.14	0.32 - 0.34	Arslan and Avşar (2020)
	Köyceğiz Lake	14.82 - 91.15		
	Alagöl Lake	149.50 - 173.40		
	Sülüngür Lake	13.69 - 29.00		

Ni and Cu concentrations detected in the water samples in this study were found to be at higher levels when compared to previous studies conducted in the region (Table 4). However, Ni and Cu values in water did not exceed the international limits specified in Table 3. In previous studies conducted in Köyceğiz Lake and Dalyan canal, it was stated that heavy metal pollution in the water may be caused by agricultural activities, thermal springs in the region, and the transport of substances from tributaries waterways (Yılmaz, 2009; Genç and Yılmaz, 2016; Arslan and Avşar, 2020).

3.1. Determined Pollution Indices in Sediment Samples

Table 5 presents the mean values obtained from heavy metal analyses of sediment samples.

Table 5. The mean heavy metal concentrations determined in sediment samples (mg kg^{-1} ± s.e.).

Elements	Stations				
	1	2	3	4	5
Cr	4.25 ± 0.13	3.52 ± 0.41	1.75 ± 0.22	3.79 ± 0.57	2.44 ± 0.32
Ni	12.69 ± 0.59	10.21 ± 1.06	5.24 ± 0.96	12.37 ± 1.14	8.56 ± 0.99
Cu	0.51 ± 0.09	0.46 ± 0.08	0.30 ± 0.06	0.30 ± 0.05	0.25 ± 0.04
Cd			< 0.01		
Pb	0.13 ± 0.01	0.13 ± 0.01	0.04 ± 0.01	0.13 ± 0.03	0.08 ± 0.02

3.2. CF

CF values calculated for each metal in all the stations were found below 1. Accordingly, there was low contamination in terms of sediment in the studied stations (CF<1). In a study conducted in Manaduky estuaries on the western coast of India, likewise our study, low contamination (CF<1) was determined in sediment in terms of the same elements (Kumar and Edward, 2009). In the sediment from Delimi River (Nigeria), significant contamination was found at the level of 3≤CF≤6 in terms of Cd, high contamination was found in terms of Cd and Cr (CF>6) in Emet Stream (Kutahya, Turkey) as well as in terms of Ni in Orhaneli Stream (Bursa, Turkey) (Sabo et al., 2013; Omwene et al., 2018).

3.3. I-geo

According to I-geo results, there was not any heavy metal contamination observed in sediment samples taken from the studied stations (I-geo = 0). In the sediment from Köyceğiz Lagoon System, a highly polluted in terms of Cr was observed in the summer season (Genç and Yılmaz, 2016). In a study carried on Mangkabong lagoon (Malaysia), there was not found any contamination in terms of copper in sediment samples (level 0) like this study, however in terms of lead, level 1 I-geo was determined (Praveena et al., 2008). The I-geo results determined for Seyhan Dam Lake (Adana, Turkey), report that sediment was moderated polluted (level 2) and moderated to high polluted (level 3) in terms of cadmium and unpolluted in terms of chromium and copper (Çevik et al., 2009). When I-geo results calculated for surface sediment of Gulf of Suez were examined, extreme contamination in some of the stations especially in terms of nickel was observed (Khaled et al., 2006). In another study conducted in the Central Black Sea (Turkey), it was claimed that there was not any contamination in the sediment in terms of Cr, Cu, and Pb but moderately high contamination was detected in terms of Cd (Şimşek and Bakan, 2017).

3.4. PLI

As a result of calculations made in terms of evaluated heavy metals, PLI value was found as 0.04 which indicated that the sediment was "unpolluted" as it was below 1. Genç and Yılmaz (2016), determined metal pollution caused from anthropogenic activities in Köyceğiz Lagoon System as a result of PLI calculations and claimed that this pollution increased during summer months. Omwene et al. (2018) found PLI values between 0.65-2.58 which they obtained as a result of different calculations from different stations in Mustafakemalpaşa, Orhaneli, and Emet Streams in North-Western Turkey. These high PLI values were reported to be potentially caused by industrial and mining activities in the region as well as by geological conditions. In another study which had similar results with the current study, PLI values were detected as <1 in an effluent channel and Dikrong River in India (Chakravarty and Patgiri, 2009; Kumar and Thakur, 2017).

3.5. ER and RI

According to the potential risk index calculations for a single heavy metal, ER was found as <40 in sediment samples taken from all the stations for each heavy metal under consideration. Therefore, low ecological risk was determined in terms of the studied heavy metals and it was concluded that there was no hazardous ecological risk for the aquatic life of Iztuzu Canal and Lagoon. Similarly, some researchers reported ER values as <40 for all the elements under consideration except Cd, in the coast of Tamil Nadu (India) (Devanesan et al. 2017), coastal lagoons in south-east Brazil (Fernandes, 1997), and a lagoon in southern Mexico (Mendoza-Carranza et al., 2016). Genç and Yılmaz (2018), found ER values as 80≤ER≤160 for Cd during the winter season in Köyceğiz Lagoon System.

In this study, the RI values obtained for all stations are <150, and there is a low ecological risk for the lagoon and the canal. In contrast to this study, moderate ecological risk in port sediment in Tianjin (China) and a lagoon in southern Mexico (Guo et al., 2010; Mendoza-Carranza et al., 2016), a very high ecological risk at some stations in Nador Lagoon (Morocco) (Maanan et al., 2015) were observed.

4. CONCLUSION

Ranking of heavy metals in water and sediment for the study area was determined as Ni>Cr>Cu>Pb>Cd according to the sum of the mean element concentrations of all the stations for the channel and lagoon. As a result of the analysis, it can be concluded that the heavy metal concentrations determined in the water samples do not exceed the internationally acceptable limits. According to CF, I-geo, PLI, ER, RI index calculations made for heavy metal concentrations detected in sediment, there is not any significant pollution and moderate or high ecological risk observed in this study.

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CONFLICT of INTEREST

The authors declare that they have no conflict of interest.

AUTHOR CONTRIBUTIONS

Murat YABANLI, Aykut YOZUKMAZ, İdris ŞENER and Tülin ÇOKER performed the samples collection, laboratory process, interpretation of the data and writing the paper. Hatice HASANHOCAOĞLU YAPICI and Esra ÇETİN KASA contributed in samples collection and data processing.

ETHICAL STATEMENTS

Not applicable.

DATA AVAILABILITY STATEMENT

The data used during the current study are available from the corresponding author on a reasonable request.

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