ORIGINAL ARTICLE

Contamination assessment of ecotoxic metals in recent sediments from the Ergene River, Turkey

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Abstract The Ergene River is a highly contaminated river that passes through the most heavily industrialized area in Turkey and receives municipal, industrial and agricultural effluents from Edirne, Kırklareli and Tekirdağ cities in the Thrace Basin. In order to investigate the pollution level of selected toxic metals in the river, 20 surface sediment samples were collected and analyzed. Concentrations of 29 elements were determined using ICP-MS in the fine fraction $(<63 \mu m)$ of sediments. The degree of pollution in the sediments of the Ergene River was examined using enrichment factor (EF) and geo-accumulation index (Igeo). Intensification of agricultural and industrial activities within the river basin have caused a considerable increase in metal concentrations, such as Cu $(EF = 12.1)$, Ni $(EF = 7.89)$, Zn $(EF = 5.73)$, As $(EF = 4.63)$, Cr $(EF = 3.62)$ and Ag $(EF = 3.12)$ in the surface sediments of the Ergene River. This result indicated that the investigated samples were moderately contaminated with Ag, Cr and As, moderately to severely contaminated with Zn and Ni, and severely contaminated with Cu. Igeo values also suggest Cu, Ni, Zn, As, Ag and Cr enrichment in the Ergene River sediments.

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Introduction

Among the various contaminants, heavy metals are of particular concern due to their environmental persistence, biogeochemical recycling and ecological risks (Burton [2002](#page-9-0)). Heavy metals can be introduced into rivers and other aquatic environments by natural and anthropogenic processes such as chemical leaching of bedrocks in drainage basins, discharge of urban runoff, domestic and industrial wastewater, mining and smelting operations, and combustion of fossil fuels, processing and manufacturing industries, and atmospheric deposition across the air–sea interface. The highest metal values are generally determined in urbanized and industrialized areas (Schintu and Degetto [1999;](#page-9-0) Rothenberg et al. [2010](#page-9-0); Franciskovic-Bilinski et al. [2011;](#page-9-0) Li and Feng [2012](#page-9-0); Sari et al. [2013](#page-9-0); Franciskovic-Bilinski and Cukrov [2014\)](#page-9-0). Over the past decade, heavy metals have been discharged into the Ergene Rivers as a result of the rapid industrialization of the Corlu, Çerkezköy, Muratlı and Luleburgaz town (Günes et al. [2008](#page-9-0); Hallı et al. [2014](#page-9-0)). In different parts of the world, heavy metals in fluvial systems can be transported along hydrologic gradients for hundreds of kilometers over short periods of times. On the other hand, in many places along the river, heavy metal concentrations in the sediments have been extensively used for the purposes of pollution monitoring (Sarı [2008;](#page-9-0) Zhang et al. [2009;](#page-10-0) Cukrov et al. [2011](#page-9-0), [2014;](#page-9-0) Song et al. [2011](#page-9-0); Shikazono et al. [2012](#page-9-0)). Moreover, knowledge of total heavy metal values in the river sediments can be useful in determining the sources of

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pollution in the aquatic environments. Therefore, sediments have been used to evaluate the status of river pollution around the world. The Ergene River is the last tributary of the Maritza River and carries the pollution load from industrial cities and agricultural areas of the Thrace Basin (Günes et al. [2008](#page-9-0); Seeliger et al. [2014\)](#page-9-0). While the Ergene River is used for drinking water at its source, its water quality deteriorates and the water even loses its irrigation water quality after passing through the polluted urban sites. The high industrial activities are basically concentrated in the upper part of the river basin in Tekirdağ. The Ergene River runs through the rich industrial area of Çorlu, Çerkezköy, Muratlı and Lüleburgaz and receives pollution load from domestic and industrial wastewater (mainly textile, chemical, food, leather, metal, paper, plastic and wood). According to the Ministry of Environment and Forestry of Turkey (MEFT), approximately 2037 industrial plants in the region are concentrated in Corlu, Çerkezköy, Muratlı and Lüleburgaz districts in the Ergene drainage area. As a consequence of the rapid industrialization, urbanization and agriculture activities, environmental problems may have occurred in this basin (Halli et al. [2014\)](#page-9-0). The concentrations of Al, As, Cr, Cu, Fe, Mn, Ni, Pb, V and Zn in freshly deposited sediments of Ergene River were determined in 2014 (Halli et al. [2014\)](#page-9-0), but the spatial distributions of toxic metals in river sediments have not yet been investigated. Within this context, twenty sediment samples from the Ergene and its main tributaries were analyzed for this study to investigate the sources and distribution of heavy metals in the surface sediments of the Ergene River for better understanding of the sources of pollution in the Ergene drainage basin. The objectives of this study were to: (1) determine spatial distribution of ecotoxic metals in modern surface sediments of Ergene River and its tributaries and (2) assess metal pollution level using the EF (enrichment factor) and Igeo (geo-accumulation index) in the Ergene River sediments and sediment quality guidelines (SQGs) (Burton [2002](#page-9-0); Hubner et al. [2009\)](#page-9-0).

Study area

The Ergene River originates in the Strandja Mountains in Tekirdağ City and lies between latitude 40°42' to 41°35'N and longitude $26^{\circ}03'$ to $28^{\circ}03'E$. It flows through Tekirdağ, Kırklareli and Edirne, joins the Maritza River at Adasarhanlı Village and then empties into the northeastern of Aegean Sea near the Gulf of Saroz (Fig. [1](#page-2-0)). The Ergene River has a length of approximately 285 km and a catchment area of approximately $11,325$ km² (Günes et al. [2008\)](#page-9-0). The climate of this region is cold and rainy in winter, while dry and hot in summer. The region has a mean annual temperature and rainfall between

 $3.5-23.4$ °C and $530-726$ mm, respectively (Balcı Akova [2002](#page-9-0)). Land use in the study area is primarily agricultural, covering about 73 % of the basin. In addition, 12 % of wheat, 61 % of sunflower and 54 % of rice productions of Turkey are produced in the basin. Inhabited areas, industrial regions, forest and lake surfaces form the remaining 27 % of the basin. The population and industrialization in Edirne, Kırklareli, Tekirdağ and its surroundings has been increasing since the 1980s. This phenomenon has caused increased amounts of wastewater to be introduced into the Ergene River (Günes et al. [2008](#page-9-0)). Agricultural and industrial activities, such as chemical, food, leather, metal, paper, plastic, textile and wood, are the main sources of river pollution along its drainage basin.

Materials and methods

Sediment sampling and collection

The toxic upper layer $(0-1$ cm) of 20 surface sediment samples was collected from the Ergene River and its tributaries using Ekman grab sampler during a 9-day period from November 21–29, 2012. Samples were numbered E1 to E20, and the geographical positions of the sampling sites were obtained using a Global Position System (GPS model Garmin's 12). The sampling design took into account the discharge points of municipal and industrial wastewater to identify the impacts of anthropogenic activities on sediment quality in the Ergene River. In the field, approximately 1 kg of sediments was collected and stored in polyethylene bags at 4° C to await further analyses. In the laboratory, sediment samples were wet sieved through an acid washed 63 µm nylon mesh to remove coarse-grained particles in order to obtain a homogenous sample for metal analyses. Grain sizes of the fraction smaller than 63 μ m were dried at 40 \degree C in an oven for 48 h and subsequently ground using an agate mortar. All data were calculated on a dry weight basis.

Digestion method

For heavy metal content determinations, approximately 0.2 g of dry sediments was totally digested with a $HNO₃$ HF–HClO4 HCl mixed acid solution using a MARS 240/50 microwave digestion system (CEM, USA). The concentrations of all elements, excluding mercury, were measured in dissolved samples by Agilent 7500ce inductively coupled plasma mass spectrometer ICP-MS (Tokyo, Japan). ICP-MS analyses were performed at the Department of Geological Engineering Laboratory of Mersin University, Turkey. The chemical analyses were

Fig. 1 Map of the investigated area with sediment sampling locations

performed as duplicate to estimate sampling error. Analytical precision of replicates $(n = 5)$ was always better than 9 % relative standard deviation (RSD) at 95 % confidence level. The accuracy of analytical procedures for total metal determinations was checked using the reference material NIST SRM 2710 (Montana soil). Replicate analysis of NIST SRM 2710 showed good accuracy, with recovery rates for metals between 93 and 103 %. Our analytical precision of replicates and accuracy of replicate analysis are comparable to other similar studies worldwide. For example, in study of Wang et al. [\(2015](#page-10-0)) based on replicate analysis, the analytical precision expressed as coefficients of variation for each metal was $\&$ %, while the recoveries for each particulate metal ranged from 94 to 102 %.

Total mercury determination

A direct mercury analyzer (DMA 80, Milestone Inc., Italy) was used for mercury determination. Sample measurement was based on thermal decomposition, amalgamation and atomic spectrometric detection. The instrument calibration and analytical procedures were conducted according to US EPA Method 7473. Measurements were taken on \sim 0.1–0.2 g of dried and ground solid material, without any pretreatment.

Statistical analyses

Many researchers used geo-statistical analysis such as EF and Igeo to determine the degree of contamination originating from natural materials or human activities in the sediments (Cukrov et al. [2011,](#page-9-0) [2014;](#page-9-0) Alkan et al. [2015\)](#page-9-0). Either Al or Fe has been used as a conservative element for EF calculations in many studies to reflect the status of environmental contamination. In this study, Al was selected as a reference metal. The EF values of Hg, Cd, Pb, Cu, Zn, Cr, Ni, As, Ag and Sn in the Ergene River sediments were calculated using the following formula:

$$
EF = \left(\frac{\frac{Cn}{AI}\text{ sample}}{\left(\frac{Cn}{AI}\text{ background}\right)}\right)
$$

where Cn and Al are the concentrations of metals and aluminum in the sample of interest and in the background materials, respectively. Background values for studied metals in the region have not yet been established. Although it is informative to normalize the metal concentrations of the sediments with those of shale or soil background values, we have chosen the lowest values of metals in the study area sediments as background values for calculation (Sun and Xu [2012](#page-9-0)). These values were used as the reference baselines and are presented in Table [1](#page-4-0). EF values were interpreted as suggested by Birth [\(2003](#page-9-0)), where: The value of enrichment factor $<$ 1 indicates no enrichment, 1–3 is minor, 3–5 is moderate, 5–10 is moderately severe, 10–25 is severe, 25–50 is very severe, and >50 is extremely severe enrichment.

The Igeo, originally introduced by Muller ([1979\)](#page-9-0), was used as a determination of metal contamination in each sediment sample, by comparing present metal contents with pre-industrial concentrations. The values of Igeo can be calculated using the following mathematical equation:

Igeo =
$$
\log_2 \left(\frac{Cn}{1.5 \times Bn} \right)
$$

where Cn is the measured concentration of the examined metal n in a sample and Bn is the geochemical background value of this metal. As in EF calculations, the lowest values of metals in the study area sediments were used as the background levels for Igeo calculations. Factor 1.5 was used as a background matrix correction due to lithological discrepancies (Muller [1979\)](#page-9-0). The resulting Igeo values were evaluated according to the Muller scale ([1981\)](#page-9-0) which consists of seven grades ranging from class 0 (unpolluted, Igeo \leq 0) to class 6 (extremely polluted, Igeo $>$ 5).

All statistical analyses performed in this paper were obtained using Statistica 6.0 (StatSoft, 2001). The following statistical analyses were performed:

(a) Determination of basic statistical parameters: N (number of cases), mean, geometric mean, median, mode, frequency, minimum, maximum, standard deviation, skewness and kurtosis.

- (b) A boxplot was used to determine anomalies in the sediment samples. Normal or lognormal boxplots are constructed on bases of the empirical cumulative distribution plots. The box length was of interquartile range, where outlier values were defined between 1.5 and 3 box lengths from the upper or lower edge of the box. Extremes are values more than 3 box lengths from the edge of the box (Tukey [1977](#page-9-0); Reimann et al. [2005\)](#page-9-0).
- (c) Cluster analysis of Q-modality was performed to find groups which contain similar samples. Cluster analysis is a form of multivariate statistics and represents a hierarchical method. There are two modes of cluster analysis: Q-modality, in which clusters of samples are sought, while in the R-modality, clusters of variables (in our case, elements) are desired. More details about cluster analysis can be found in Kaufman and Rousseeuw [\(1990](#page-9-0)).
- (d) Factor analysis was also performed in order to reduce the number of variables and to set up a model of several factors, each of them describing one anthropogenic or natural influence. In factor analysis, the relation between a set of m variables is assumed to reflect correlations of every one of the variables with p mutually non-correlated main factors. The general assumption is that $p < m$. Variance of m variables is derived from the variance of the p factor. More details about factor analysis can be found in Halamić et al. (2001) (2001) and Davis (2002) (2002) .

Results and discussion

Heavy metal distribution

The range and average (in parentheses) concentrations of toxic metals and major elements measured in mg kg^{-1} were 2.1–6.8 (3 ± 1) for Ag, 40,610–91,340 $(72,848 \pm 1)$ 11,730) for Al, $11-52$ (25 ± 10) for As, 1.1-1.7 (1.34 ± 0.2) for Cd, 95-304 (160 ± 49) for Cr, 23-203 (65 ± 46) for Cu, 13,950–39,560 (26,935 \pm 7373) for Fe, 1.8–2.5 (2.2 \pm 0.14) for Hg, 133–865 (356 \pm 167) for Mn, 3.2–6.2 (3 ± 0.7) for Mo, 19–155 (64 ± 35) for Ni, 77–145 (99 \pm 15) for Pb, 258–966 (486 \pm 177) for V and 74–385 (177 \pm 101) for Zn. A comparison of the metal concentrations with average shale values reveals that most of the samples from the Ergene River are polluted with As, Ag, Cd, Cr, Cu, Hg, Mo, Ni, Pb, V and Zn. On the contrary, the samples studied had Al, Fe and Mn values, which indicates that there are no major sources of pollution for these elements in the Ergene River area similar to those for average shale values (Krauskopf [1985](#page-9-0)). The station near

Table 1 Basic statistical

Elements concentrations in μ g/g

the industrial plants $(E2)$ in the Corlu Stream had the highest values of Ag (6.8 mg/kg), As (52 mg/kg), Cr (304 mg/kg) , Mo (6.2 mg/kg) and V (966 mg/kg) . This area is affected by the wastewater and water runoff from local industrial and agricultural activities (Halli et al. [2014\)](#page-9-0). Çorlu, Çerkezkoy and Muratlı town are the industrial area, which are a major source of pollution for the Ergene River. As a result, there is a noteworthy increase in the concentrations of Ag, As, Cr, Cu, Mo, Ni, and Zn in the upper part of the Ergene River (points E2, E3, E5 and E8). Intensification of agricultural and industrial activities within the river basin have caused considerable increase in heavy metals such as Cu $(EF = 12)$, Ni $(EF = 7.9)$, Zn $(EF = 5.7)$ As $(EF = 4.6)$, Cr $(EF = 3.6)$ and Ag $(EF = 3.1)$ in the surface sediments of the upper Ergene River. Arsenic and Cr appeared to be the pollutants with the greatest potential to cause adverse effects on biota, while Cu, Pb and Zn may adversely affect some benthic species occasionally, as suggested by the SQGs.

Basic statistical parameters

Table 1 presents the basic statistical parameters (minimum, maximum, mean, median, range and standard deviation) for the studied elements including Halli et al. ([2014\)](#page-9-0) data

(Al, As, Cr, Cu, Fe, Mn, Ni, Pb, V and Zn) from the Ergene River (Turkey).

Determination of anomalies using boxplot method

The anomaly data from the boxplot statistical method are presented in Fig. [2](#page-6-0) and Table [2](#page-7-0). In total, 32 parameters were evaluated, including the chemical elements and three additional parameters (silt, clay and TOC). Of those, 16 parameters had a very regular distribution, demonstrating no anomaly, while 16 other parameters had at least one anomaly. Statistically speaking, those anomalies were mostly of low intensity, i.e., all were outliers, while only four elements (Si, Sr, Mo and Ag) showed extremes, staggered on only two locations (E1 and E2). Of the 20 locations, only E9 had no anomalies. The location of greatest interest was E2, which had three extremes and four outliers, some of which were potentially toxic heavy metals. E2 could be under significant anthropogenic influence, while minor anthropogenic influence could also be present at locations E5, E6, E8, E18 and E19. The minor anomalies present at other locations were most probably of natural origin. The Si anomaly at the E1 location was probably of natural origin, derived from Strandja Massif including metamorphic and granitoid rocks which can be observed in the northeastern most part of the study area. These rocks are highly enriched by Si (53 %), Sr (936 ppm), Mo (4.3 ppm) and Ag (1.2 ppm) (Eraslan [2010\)](#page-9-0). Additional river enrichment by Si, Sr, Mo and Ag may also be a result of surface drainage of both solid and liquid wastes, having limited treatment for organic matter, originating from the large industrial plants surrounding the Ergene River. Storage tanks for various solvents used in petroleum, paint, plastic and pesticide industries are located mostly along the northeast of the river (Günes et al. [2008](#page-9-0); Halli et al. [2014](#page-9-0)).

Q-modality cluster analysis

Table [3](#page-7-0) presents the results of Q-modality cluster analysis with members of each of three extracted clusters and mean values of each studied parameter and element for each cluster. Cluster 1, containing only three samples, could be considered a ''carbonate cluster,'' because it contained the highest concentrations of calcium. The locations in this cluster were mostly unpolluted, indicating it was not under high anthropogenic pressure, as concentrations of almost all heavy metals were lowest in this cluster. Carbonate gravels predominated this cluster as evidenced by the large grain size and low clay percentages. Cluster 3 was under significant anthropogenic influence since the concentrations of the majority of heavy metals were the highest at these locations. Most of the element concentrations for Cluster 2 were ''transitional,'' i.e., between the concentrations of those found in Clusters 1 and 3. Cluster 2 also had the highest concentration of Si, obviously under the influence of silicate rocks such as flysch, metagranite and gneiss schist in the Thrace Basin (Gorur and Okay [1996](#page-9-0); Turgut and Eseller [2000;](#page-10-0) Siyako and Huvaz [2007](#page-9-0)).

Factor analysis

Table [4](#page-8-0)a, b presents factor loadings and factor scores, respectively, for each sampling station. Three factors (varimax normalized) were extracted, and the results fall well within the requirements (Morrison [1967\)](#page-9-0) that main components should explain at least 75 % of the total variance (in our dataset, 75.83 % of the total variance were explained with three factors).

Factor 1 correlated positively with clay, Al, Fe, Co, Zr and Hg and negatively with gravel. It could be a natural factor bound to some coal rocks and lignite occurrences and igneous rocks of Istranca Mountains north of the Thrace Basin close to Saray, Vize, Kırklareli and Demirhanlı (Eraslan [2010\)](#page-9-0). There are also some anthropogenic factors bound to some industry such as chlorine–alkali plants, paper, pulp and antifouling plants, which have been discharging effluents into river. Factor 2 correlated with V, Cr and As. The main anthropogenic sources of arsenic are agricultural and industrial activities; chromium is used in stainless steel and other alloys, pigment manufacturing in the textile industries which are mostly concentrated in study area. On the other hand, vanadium is used in alloys for making rust-resistant steel, manufacturing tools, engines and gears in the drainage basin of the Ergene River. Factor 3 was correlated with TOC, Cu, Zn and Cd and is an obvious anthropogenic factor, but associated with pollutants other than those associated with Factor 1.

The source of these elements is agricultural, municipal and industrial (electroplating, steel works and textile) wastewater discharges in Corlu, Cerkezköy and Muratlı.

EF and Igeo

Results of EF and Igeo are presented in Table [5](#page-8-0) for 10 toxic metals: Hg, Cd, Pb, Cu, Zn, Cr, Ni, As and Ag. For each toxic metal, basic statistical parameters (minimum, maximum, mean, median and range) are presented to provide a brief overview of the studied region.

Based on EF values, the majority of elements showed minor or moderate enrichment at all locations. Only three elements (Cu, Zn and Ni) show higher EF values. The maxim EF value for Cu (12.1) was at location E6; according to the EF scale, it belongs to severe enriched sediments, indicating significant pollution at this location. At E8 and E14 locations, EF values for Cu indicate moderately severe enriched sediments. At most locations, Zn

Fig. 2 Boxplot of selected elements which showed anomalous values

Table 2 Anomalies (extremes and outliers) determined by the boxplot method

Table 3 Cluster means for three obtained clusters by Q-modality cluster analysis

Location	Extreme	Outlier
E1	Si	Se
E2	Sr, Mo, Ag	V, Cr, As, Sb
E3		
E4		
E5		Pb
E6		Ca, Cu
E7		
E8		Zn
E9		Ca
E10		-
E11		Si
E12		
E13		
E14		
E15		
E16		Sr
E17		Rb
E18		Mn, Rb
E19		Zn, Rb, Tl
E20		

EF values indicated minor or moderate enrichment, but at two stations (E8 and E14), the values were characteristic of moderately severe enriched sediments. At seven locations (E7, E10, E11, E14, E15, E19 and E20), Ni had EF values characteristic of moderately severe enriched sediments.

Igeo values were low for all studied elements. For Hg, Cd, Pb and Sn, Igeo values indicated no enrichment (values are \le 1). All other elements' Igeo values ranged from 1 to 3, characteristic of minor enrichment.

EF was shown to be a more sensitive tool for the estimation of toxic metal enrichment in sediments.

Sediment quality criteria

There have been numerous sediment quality guidelines developed during the past 20 years to assist regulators in dealing with contaminated sediments. Unfortunately, most of these have been developed in North America and have errors of 25 % or greater (Burton [2002](#page-9-0)). Due to the dearth of Turkish regulations, for use in this study, metal concentrations in the sediments were compared to various sediment quality guidelines (Burton [2002;](#page-9-0) Hubner et al. [2009](#page-9-0)).

Although SQG alone should not be applied to causeand-effect evaluations (Chapman et al. [1999\)](#page-9-0), we use them here for basic toxic evaluation.

All Hg (Table [1\)](#page-4-0) values obtained in the sediments were above higher limits using any SQG (Burton [2002\)](#page-9-0)

indicating a significant possibility of negative impacts upon the biota. This is in contrast to sediment cadmium concentrations where SQG indicates a very low possibility of negative effects. For lead and copper, negative effects are possible, but are not very probable. Zinc, chromium and nickel, each has a greater possibility of having a negative impact when compared with copper and lead. Similar to mercury, all measured concentrations of arsenic and silver were above ERM guidelines, indicating a great possibility of negative effects.

Bold values indicates significant correlations

Conclusions

The River Ergene is subjected to varying degrees of pollution caused by numerous untreated and partially treated waste inputs of Çorlu, Çerkezköy, Muratlı and Lüleburgaz Districts and industrial effluents. The river is highly influenced due to ecotoxic metals, which enter the river

Table 5 Enrichment factor and Igeo for selected toxic metals

system by direct discharges of municipal, agricultural and industrial effluents and surface runoff.

The metal enrichment factor (EF) and geo-accumulation index (Igeo) of Cu, Ni, As, Cr and Ag indicated concentrations above background levels in the eastern part of the study area in proximity to Corlu, Cerkezköy and Muratlı districts, while concentrations of other ecotoxic metals were generally within background concentration ranges.

Factor and cluster analyses results indicated that Cu, Ni, As and Cr were associated with anthropogenic activities, whereas enriched Si, Sr, Mo and Ag in the study area were most probably of natural origin, derived from Strandja Massif.

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