TRACE METAL ENVIRONMENTAL CONTAMINATION RECORDS IN CORE SEDIMENTS OF GORGAN BAY IN THE SOUTHEAST OF THE CASPIAN SEA

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Abstract. In this study five sedimentary cores were collected from Gorgan Bay (southeastern part of the Caspian Sea). Concentrations of elements were measured to determine depositional trends and geo-statistical assessment of toxic elements was carried out. Al, Fe, K, Mg, Na and S content increased in all 5 cores, from the bottom to near the top layer, with maximum concentrations at 15–45 cm depths. However, for the toxic elements, maximum concentration of As, Cr, Cu, Pb, Zn, Co and Ni varied in different depth of 5-15 cm and 80-120 cm. Enrichment factors (EFs) and pollution load index (PLI) were calculated to assess the enrichment and pollution of heavy elements in the bay sediments. Except for Ca and S, all studied elements showed positive and significant correlations with Fe and Al, which indicate the same source (human or natural). Principal component analysis (PCA) indicated that sediments were influenced by both geologic and anthropogenic impacts. According to the EFs, most toxic elements showed no significant enrichment and no pollution in the core sediments. Based on PLI average value in all cores, the southeast coast of the Caspian Sea (Gorgan Bay) should be classified as not metal polluted, but in recent decades there has been a dramatic increase of toxic elements that needs more attention and also monitoring.
Keywords: *heavy metals, sedimentary cores, geochemistry, source*

Introduction

The study of core sediments is very useful to detect different natural process such as paleoclimate changes, or anthropogenic activities on sedimentary environments (Harikumar and Nasir, 2010) by examining the geochemical and geo-statistical data (Karbassi et al., 2005; Mohamed et al., 2005; Sun et al., 2012; Vallius, 2014; Veerasingam et al., 2015). The coastal zones with their variable physical and chemical properties are suitable environment for the assessment of pollutant accumulations in aquatic ecosystems, where the bottom sediments are affected by both dissolved heavy elements or on surface absorbed particles transported from land to the basin (Harbison, 1986; Szefer et al., 1995). Heavy metal concentrations from natural and anthropogenic sources in coastal area can be increased through high input after intense rock-minerals weathering in the land or due to urbanization (Harikumar and Nasir, 2010). Anthropogenic pollutions in aquatic environments have direct influence on coastal ecosystems (Alessandro et al., 2006). In fact, core sediments obtained from the coastal areas provide a good chronological record of contamination (Morelli et al., 2012). Understanding trace metal emissions in coastal environments is an important task for researcher sand policy planners, and regulatory actions can be implemented to reduce health risks potential.

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Study area

Gorgan Bay (Southeastern part of the Caspian Sea) is a unique brackish water body, semi-confined triangular-shaped having about 500 km^2 area, maximum depth of 4 m and average depth 1.5 m, high ecological value and hydromorphological properties such as depth variation, freshwater flow and wave exposure. It is located at the south-east of the Caspian Sea in both Golestan and Mazandaran provinces (*Fig. 1*). Gorgan Bay was formed during the late Holocene period by a coastal barrier system (sandy spit) which is named Miankaleh spite (Kakroudi et al., 2012). There are no tides in the Gorgan Bay like in other parts of the Caspian Sea. It is connected to the Caspian Sea through Ashoradeh-Bandar Torkaman mouth, (Approximately; width of 400 m, 3 km long) with strong currents in the area affected by storm surge. Water balance in the Gorgan Bay is influenced by water evaporation, precipitation, intrusion from the Caspian Sea, and to a lesser extent by fresh river water (Sharbaty, 2011, 2012). It receives freshwater from a number of rivers and small streams, Gorgan-rud River from the north of the inlet and Qarehsu river enters from the east.

Several works on distributions of heavy metals and other pollutants have been carried out in this area (Kasymov., 1989; Tait et al., 2004; Parr et al., 2007; Karbassi et al., 2008; Bastami et al., 2012; Nasrolahi et al., 2017), but no studies have focused on the trends of metals on different core sediments in this area. The aims of the current study are: (1) to describe the vertical variation of elements from east to west of Gorgan Bay; (2) to estimate the geochemical sources of major and trace metals in sediments using coefficient correlation and principal component analysis (PCA); and (3) to assess enrichment and possible contamination of trace metals in sediments, using enrichment factors (EFs) and pollution load index (PLI).

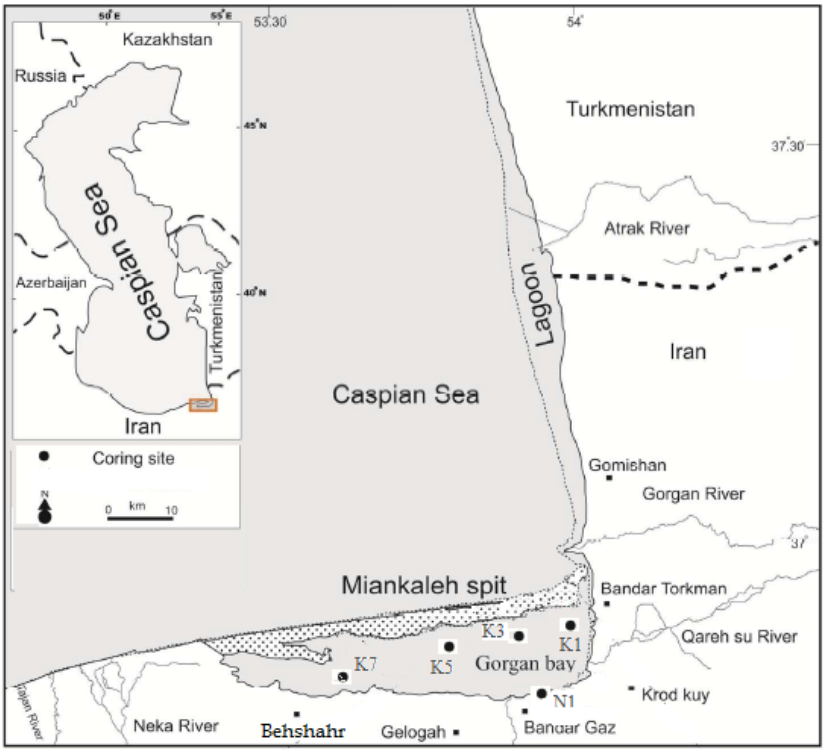

Figure 1. *The locations of the core sampling sites*

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Materials and methods

Five sedimentary cores with different length were collected using a gravity corer on August 2016 (*Table 1*). Samples were packed and carried in ice-boxes to the laboratory and stored at 4 °C until analysis. After opening the cores, about 25 subsamples picked from each core (each 5 cm 1 subsample and totally 120 subsamples). Subsamples were dried in an oven and then powdered by using a mortar then screened with a 0.5 mm sieve to remove large particles. Sediment samples (1 g) were digested using a mixed solution of 1:3 nitric acid : hydrochloric acid for 3 h. The solution was cooled, filtered through Whatman-42 and diluted to 50 ml volume using distilled water (APHA et al., 2005). Concentrations of major elements (Al, Ca, Fe, K, Mg, Na, and S) and trace metals (As, Co, Cr, Cu, Ni, Pb, and Zn) were measured by using inductively coupled plasma-optical emission spectrometry (ICP-OES; Varian 735 ES series), Arsenic (As) analysis was carried out by using atomic absorption spectrometry (model SOLAAR, M5) Data recoveries of the analysed element areas are the followings: Al (100%–101%), Ca (97.6%–97.8%), Fe (100%–101%), K (103%–104%), Mg (95.1%–95.3%), Na (102%–103%), S (98.7%–100%), As (112%–113%), Co (105%–111%), Cr (88.5%–88.7%), Cu (98.3%–98.8%), Ni (97.5%–98.7%), Pb (118%–121%), and Zn (93.2%–93.5%).

Table 1. *Geographic position, length and depth of each core in Gorgan Bay*

Core name	Water depth (m)	Core length (m)	Longitude	Latitude
K1	2.6	1.78	54° 0’ 42.34	36° 51’ 49.9
K3	3.24	1.67	53° 54’ 38.86	36° 50’ 33.5
K5	2.8	1.34	53° 48’ 43.5	36° 50’ 21.35
K7	0.4	1.3	53° 36’ 18.87	36° 48’ 26.31
N1	0.1	0.98	53° 54’ 62.5	36° 47’ 6.19

Grain size analysis was performed using laser particle size analyzer (HORIBA-LA950, France & Japan) in Iranian National Institute for Oceanography laboratory. Before analysis, about 4 g samples were cremated in an oven at 550 °C for 4 h and then at 950 °C for 1 h to remove organic matter and biogenic carbonate, respectively.

Pearson correlation analysis and PCA were conducted to specify geochemical associations and certainly the factors controlling the geochemical origin in coastal sediments (Bastami etal, 2014). A two-tailed Pearson correlation coefficient was used in correlation analysis. PCA was performed on a correlation matrix; N80% of the total variance was computed for principal components. Pearson correlation coefficients and PCA were accomplished using the SPSS 20.0 software. Enrichment factor (EF) as an appropriate index was calculated to determine anthropogenic or geogenic source of the toxic elements by normalizing their concentrations according to the sediment texture properties (Morillo et al., 2004; Selvaraj et al., 2004; Adamo et al., 2005; Vald’es et al., 2005; Shi et al., 2010; Morelli et al.2012; Zhang et al., 2013). To calculate the EF, Al is widely used in the equation as aluminum silicate, it is predominant at the coastal areas. Enrichment Factor is determined as follows (Huang and Lin, 2003; Woitke et al., 2003):

$$\text{Enrichment Factor} = (H / Al)_{\text{sample}} / (H / Al)_{\text{background}} \quad \text{(Eq.1)}$$

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where (H / Al) $_{sample}$ and $H / Al)_{background}$ are heavy metal concentrations in sample and background reference, respectively. In this study, we used background concentrations of heavy metals in K5 sediment core at the depth of 85 cm which are 2.96, 11.50, 53.01, 14.33, 27.43, 11.80 ppm and 41.60 for As, Co, Cr, Cu, Ni, Pb, and Zn, respectively (De Mora et al., 2004).

Results and discussion

Vertical distributions of selected elements in cores K1, K3, K5, K7 and N1 are presented in *Figure 2*. A lot of runoff and eroded sediments from several sources such as human activities and river systems (Bagheri et al., 2012) enters terrigenous materials into the Gorgan Bay. Generally concentration of Al, Fe, K, Mg, Na and S increased from the bottom to the top layers in the cores, and the maximum concentrations of these major elements were observed at depths of 15–45 cm in all cores, while the toxic elements have different trends. In core K1, the highest concentrations of Cr (75.95 ppm), Ni (83.22 ppm) and Zn (83.22 ppm) were determined at 5 cm depth, As (9.39 ppm) at 10 cm, Cu (36.64 ppm) at 15 cm, Co (21.79 ppm) at 30 cm and Pb (22.01 ppm) at 35 cm depth.

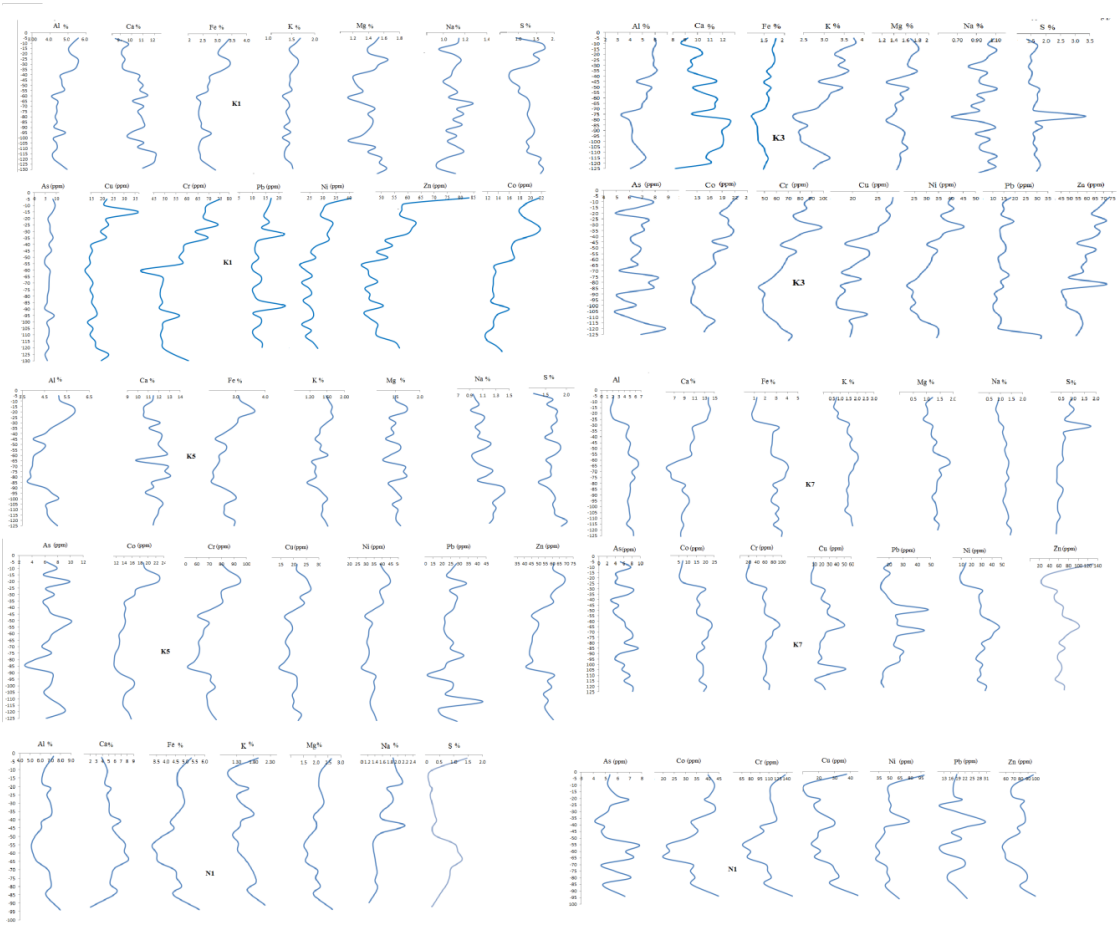

Figure 2. *Vertical distributions of major and trace metals in cores*

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In core K3, the highest concentration of Cu (28.04 ppm), Co (22.04 ppm) and Zn (73.45 ppm) were observed at 5 cm depth, for Cr (98.44) and Ni (45.26 ppm) at 10 cm depth, and for As (8.81 ppm) and Pb (31.38 ppm) at 120 cm depth.

In core K5 maximum concentrations of all heavy metals were observed at a depth of 15-20 cm, expect for As (10.15) at 50 and Pb (43.08) at 110 cm. opposite of this trend in west and south of Gorgan bay in the K7, N1 cores, the highest value of trace elements reported in different depths as As (9.55, 7.80 mg/g) at 85, 55 cm; Co (23.79, 44.79 mg/g); Cr (95.11, 149.4 mg/g) at 65, 94 cm; Ni (47.37, 105.4 mg/g) at 65,5; Cu (52.05, 45.20 mg/g) at 105,94 cm; Pb (48.13, 30.6 mg/g) at 50, 37cm and Zn (131.6, 106.7 mg/g) at 5, 94 cm depth (*Fig. 2*).

Heavy metals show some information about source and migration of sediments. For example, high correlations between heavy metals probably mean these elements reflect similar origin or share analogous transformation and immigration processes in the certain situation. The major factors affecting spatial variation of trace elements in the sediments are organic matter and the grain size (Aloupi and Angelidis, 2001; Huang and Lin, 2003; Liaghati et al., 2004). The fine grains, have high surface/volume ratio and cation absorption potential, and are more effective adsorbents of contaminated organic and inorganic materials (McCave, 1984; Horowitz and Elrick, 1987). Fine-grained sediments, which contain lots of organic matter are more contaminated than coarse-grained sediments (DeMora et al., 2004). However, some studies have reported an inverse relationship between particle size and metal content (Bastami et al., 2014). In our study, in most cores, all elements showed positive and significant correlation (except for Ca and S) with Fe and Al indicating the same origin or input source of these elements (human or natural) into the Gorgan Bay. Also, there was a positive and significant correlation between As, Fe, Zn, Cr, Ni, Cu and K, indicating that these elements have the same origin and the same behavior in the studied area. Sulfur and calcium had a negative correlation with most of the heavy metals which indicates different origin or behavior of these elements in the environment. Also, in core N1, arsenic had a negative correlation with most metals while it was positively correlated with calcium and showed negative correlation with iron and aluminum, which probably indicates the biological origin of this metalloid.

Three principal components (PC) were extracted from the core K1 sediments, which accounted for about 81.74% of the total variance. The first component (PC1) accounted for 58.21%, the second component (PC2) accounted for 14.26% and the third component (PC3) accounted for 8.22% of the total variance (*Fig. 3*). In PC1 high loadings of major elements (Al, Fe, K and Ca) and heavy metals (Co, Cr, Ni, As, Cu, Pb and Zn) were observed (from 0.617 to 0.958). The elements Al, Fe and K mainly represent lithogenic origin after weathering and erosion of rocks and soil parent materials, PC1 described the lithogenic source bound in aluminosilicate minerals. The second component (PC2) also showed Mg and S with moderate to high loadings (0.791-0.901) and Na with high loading counted in the third component (PC3).

In core K3, we extracted 3 PCs, which accounted for about 82.43% of the total variance (*Fig. 4*). Based on the loading distribution of the variable elements, Al, Ca, Fe, K, Mg, Cr, Ni, Cu, Co and Zn constituted a firmly related group (PC1) with 55.81% of the total variance. While another group was composed of Na and S (PC2) with 14.24% of the total variance. The third component (PC3) accounted for 12.37% and had high loadings on As and Pb. These results may indicate the different origins or controlling factors of the heavy metal distributions in the Gorgan Bay sediments. In this area, the

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lithogenic source acted as the main factor accounting for PC1, while PC2 and PC3 may be derived from the urban source.

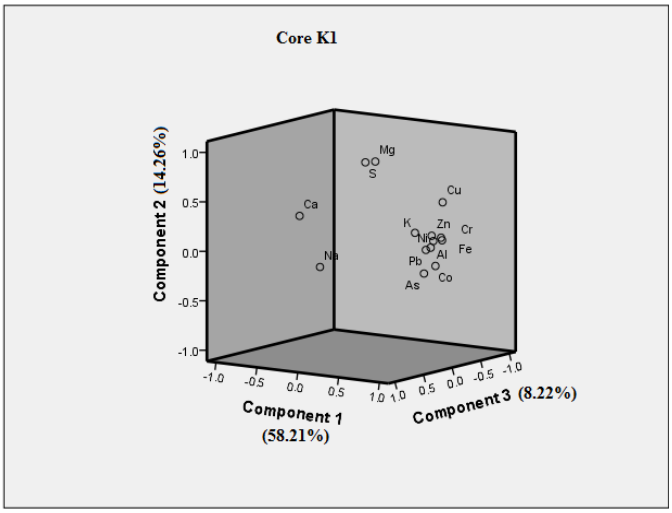

Figure 3. *PCA plot of elements in core K1*

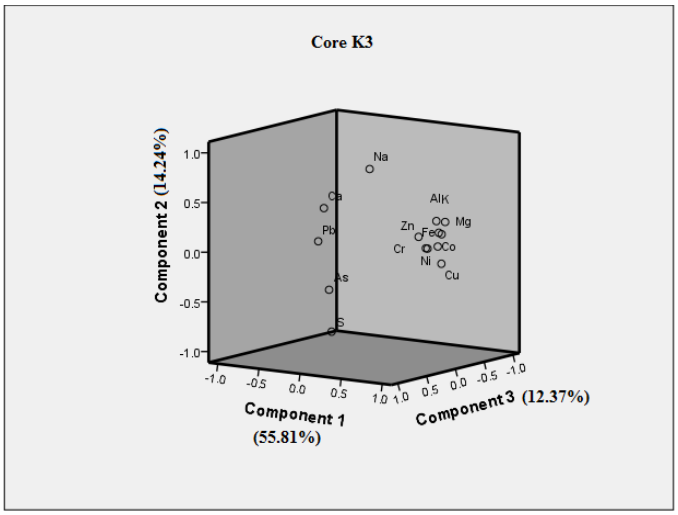

Figure 4. *PCA plot of elements in core K3*

In core K5, we extracted 3 PCs, which accounted for about 80.26% of the total variance (*Fig. 5*). The first component (PC1) accounted for 55.30% of the total variance and included all variable element except for Na, S, Pb and As, while the second component (PC2) accounted for 14.37% with high loading on Na, S and As (0.61-.82) and the PC3 accounted for 10.58% with high loading on Pb.

In core K7 (*Fig. 6*) that was obtained from west of the Gorgan Bay, 3 PCs were extracted and total variance was 86.55% (*Fig. 5*). In this core unlike in the other 4 cores Na and S with other elements (Al, Ca Fe, K, Mg, Cr, Ni, Cu, and Co), Zn with high loading on PC2, and Pb and As show loading on PC3, which accounted 65.11%, 11.79% and 9.65% respectively. In the studied area PCA showed anthropogenic input of Pb and Zn from the industrial activities (anthropogenic source), which are widely used

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as the main components in several industrial processes. In fact, those elements were partially derived from the anthropogenic source.

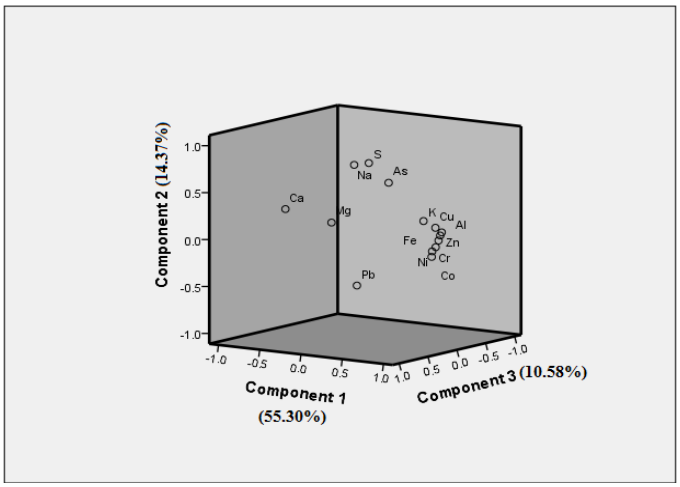

Figure 5. *PCA plot of elements in core K5*

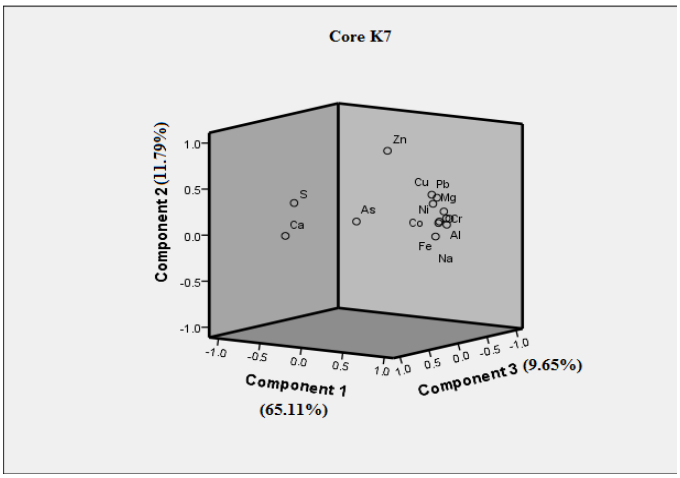

Figure 6. *PCA plot of elements in core K7*

Finally in core N1 (*Fig. 7*), 3 extracted PCs accounted for about 86.89% of the total variance. The PC1, PC2 and PC3 were 54.31%, 20.15% and 12.42% of the total variance, respectively (*Fig. 5*). The first principal component (PC1) included all elements expect for Na and S (that are considered as PC2) and As, Pb (that are considered as PC3) were lithogenic source. The PC2 also showed that Cu, Co, Pb and Zn had loadings on (0.16–0.63), indicating that these elements were partially derived from the anthropogenic source. Overall, all metals in the Gorgan bay sediments were mostly derived from the geologic origin (geogenic), except for western part of the bay, where the toxic elements were mainly derived from terrestrial discharge of industrial activities (anthropogenic).

Enrichment factor is a useful index to assess the heavy metals contamination in the soil and sediments (Feng et al., 2004; Reddy et al., 2004; Han et al., 2006; Chen, 2007; Çevik et al., 2009; Bastami et al., 2012). EF values are interpreted as; EF < 1, no

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enrichment; EF 1 to 3, minor enrichment; EF 3 to 5, moderate enrichment; EF 5 to 10, moderately severe enrichment; EF 10 to 25, severe enrichment; EF 25 to 50, very severe enrichment and EF > 50 extremely severe enrichment (Grant and Middleton, 1990; Loska et al., 1997; Abrahim and Parker, 2008). In this study, EF values in all core samples ranged in no-enrichment (EF < 1) to minor enrichment (EF 1 to 3) suggesting no anthropogenic enrichment of the toxic elements, except for Pb and Zn in core7, which show moderate enrichment of these elements (EF 3-5) and indicate that anthropogenic source of Zn and Pb were significantly polluted the sediments of core 7 (*Fig. 8*).

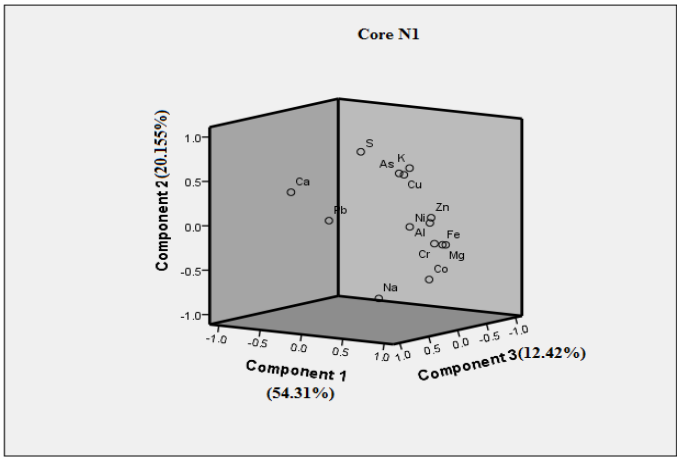

Figure 7. *PCA plot of elements in core N1*

To assess the sediment environmental quality, an integrated pollution load index (PLI) of seven toxic elements was calculated as suggested by Suresh et al. (2011).

$$PLI = (CF1 \times CF2 \times CF3 \times \ldots\ldots CFn)\ 1/n \qquad \text{(Eq.2)}$$

An area with PLI value > 1 is polluted whereas PLI value < 1 indicates no contamination (Chakravarty and Patgiri, 2009; Seshan et al., 2010). Concentrations of the elements in shale (sedimentary rock), which refer to the average concentration of the earth crust (Turekian and Wedepohl, 1961), were used as the reference baselines in this study. The PLI was calculated for all five studied cores and based on seven toxic elements of As, Pb, Co, Cr, Ni, Cu, and Zn (*Table 2*). The maximum and the minimum calculated PLI were 1.07 and 0.26, respectively (*Table 2*).

Table 2. *Comparison of toxic metals in each cores (ppm) with base value and PLI*

Metals	K1			K3			K5			K7			N1		
	Ave±Std	Max	Min	Ave±Std	Max	Min	Ave±Std	Max	Min	Ave±Std	Max	Min	Ave±Std	Max	Min
As	6.65±1.28	9.39	4.70	6.60±1.03	8.81	4.84	6.93±1.60	10.15	2.96	6.11±1.79	9.55	2.89	5.76±1.06	7.80	4.12
Co	15.63±3.16	21.79	11.68	16.78±3.53	22.81	11.68	15.19±3.01	22.81	11.50	16.20±5.34	23.79	5.37	34.63±7.40	44.79	20.24
Cr	56.38±9.68	75.08	38.89	67.41±14.4	98.44	44.88	71.76±10.3	94.24	53.01	57.43±20.92	95.11	15.32	106.35±21.14	149.60	65.89
Cu	17.96±4.99	36.64	12.14	21.72±3.65	28.04	16.80	20.44±3.05	26.63	14.33	22.80±12.23	52.04	6.01	24.09±8.98	45.20	11.31
Ni	27.26±4.44	40.49	21.26	32.07±6.28	45.26	21.99	35.15±4.4	44.58	27.43	28.15±9.92	47.37	8.78	50.32±17.78	105.40	30.01
Pb	13.41±3.31	22.46	9.88	15.43±5.24	31.38	10.16	25.15±5.8	43.08	11.80	22.93±8.60	48.13	14.33	18.34±4.73	30.60	10.95
Zn	52.33±8.72	83.22	42.66	59.36±8.24	73.45	46.09	54.50±7.8	68.37	41.60	66.32±22.01	131.60	23.86	78.14±13.45	106.70	55.45
PLI	0.57			0.63			0.63			0.71			0.83		

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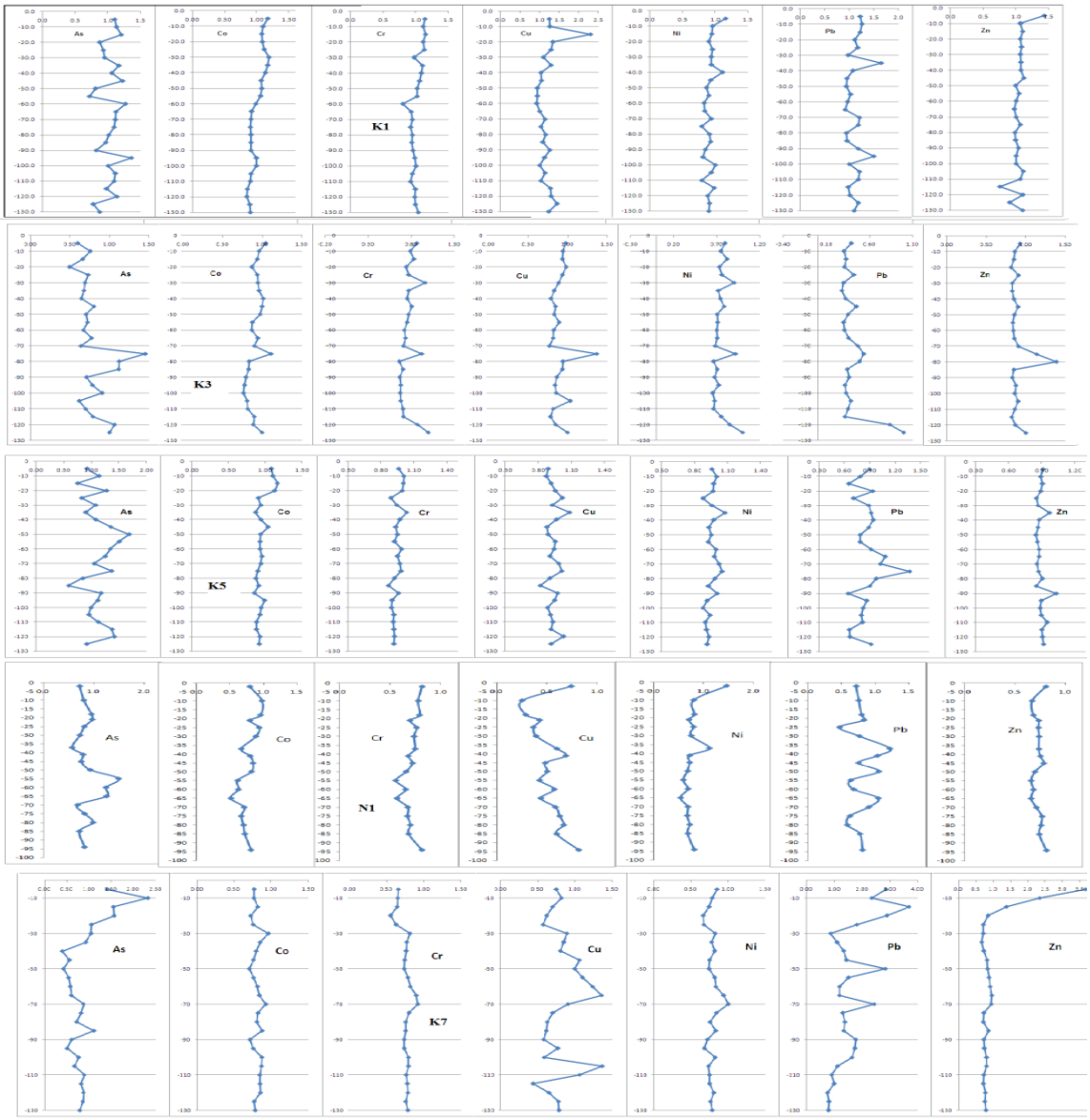

Figure 8. *Enrichment factor trend of heavy metals in cores*

Results showed that the PLI value of the elements in average sediment was lower than that in surface sediment in cores K1, K3, K5, K7 and N1, which can be attributed to direct discharges from urban area and fisheries boats carrying toxic elements, contaminants in study areas, especially in core N1 area. Based on average of PLI value in all cores, southeast coast of the Caspian Sea (Gorgan Bay) should be classified as no toxic pollution (*Table 3*), but in recent decades there has been a dramatic increase that needs more attention and consideration. Finally, comparison of toxic elements with the quality guidelines (NOAA, ISQG) showed the range between the two guideline (level of some elements as As, Ni, Cu, Pb was higher than ERL and lower than ERM). However, because of ecosystem condition, tourism and aquatic resources, this area, must be prevented, managed and monitored properly before they constitute a serious threat to the health of the environment and the organisms.

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Table 3. *Comparison of this study with other studies and quality guidelines*

Region	Pb (ppm)	Cu (ppm)	Zn (ppm)	As (ppm)	Ni(ppm)	Co(ppm)	Cr(ppm)	Reference
Gorgan Bay	9.88-43.8	6.01-45.2	23.8-106.18	2.89-10.15	8.78-105.4	5.37-44.8	15.3-149.6	This study
Southeast coast of the Caspian Sea	13.00±3.14 (9-16)	9.09±3.16 (5-14)	28.00±5.71 (22.33-36.33)	4.11±1.27 (2.36-5.16)	17.42±3.78 (16.33-24)	-	-	Bastami et al. (2014)
Caspian Sea (Iran)	18.0±4.17 (11.3-24.6)	34.7±11.9 (13.2-50.9)	85.3±17.9 (55.9-146)	12.5±3.04 (6.97-20.1)	51.6±11.8 (29.4-67-8)	15.9±4 (6.9-24.2)	85.2±15.3 (59.6-128)	De Mora et al. (2004)
Jurujuba sound (Brazil)	61±35 (5–123)	51±40 (5–213)	158±97 (15–337)	-	48±19 (15-79)	-	89±19 (15-79)	Baptista Neto et al. (2001)
Black Sea	19.44±12.48 (2.1-43.5)	30.67±18.76 (4.61-75.72)	69.00±57.03 (1-174)	-	49.8±34.81 (1-117)	20.76±18.55 (1.57-71.59)	57.2±41.85 (1-120)	Secrierus and Secrierus (2002)
Mediterranean Sea	23.81±12.80 (8-54)	85.87±65.63 (10-208)	115.75±62.75 (38-227)	9.43±4.80 (5-24)	16.31±5.85 (8-29)	-	17.43±5.83 (10-26)	Moreno et al. (2009)
ERL	46.7	34	150	8.2	21	-	81	Long et al. (1995)
ERM	218	270	410	70	52	-	370	Long et al. (1995)
ISQG	30.2	18.7	124	7.24	-	-	52.3	ISQG
PEL	112	108	271	41.6	-	-	160	ISQG

ERL = Effect range low (NOAA); ERM = Effect range medium (NOAA); ISQG = Interim sediment quality guideline (Environment Canada); PEL = Probable effects level (Environment Canada)

Conclusion

In this study concentrations and geochemical trends of some major elements (Al, Fe, K, Mg, Na and S) and heavy metals (As, Cu, Ni, Co, Pb, Cr, Zn), in 5 cores from Gorgan Bay were determined. in all K1, K3, K5, K7, N1 cores major element concentrations increased from the bottom to the top layer, and the maximum concentrations of these major elements in cores were observed at depths of 15–45 cm. All elements showed a positive and significant correlation (except Ca and S) with Fe and Al and represent the same input source (human or natural), also there was a positive and significant correlation between arsenic, Fe, Zn, Cr, Co, Ni, Cu and K, indicating that these metals have the same origin and the same behaviour in this area. Sulfur and calcium had a negative correlation with most of the heavy metals which indicates different behaviour and origin of these elements in the environment. PCA represent all metals were mostly derived from the geological origin in sediments of the Gorgan Bay, except for western part which was mainly derived from terrestrial discharge of industrial activities. EF values in all cores ranged no enrichment (EF < 1) to minor enrichment (EF 1 to 3) suggesting no anthropogenic enrichment, expect Pb and Zn in core 7 showed moderate enrichment (EF 3-5) which indicated that anthropogenic Zn and Pb enriched and polluted significantly sediment core 7. Based on the average of PLI values for these 5 cores and 7 trace metals (As, Co, Cr, Cu, Ni, Pb, and Zn), the southeast coast of the Caspian Sea (Gorgan Bay) should be classified as no metal pollution but in recent decades there has been a dramatic increase specially in the coastal area of K7, that needs more attention and consideration.

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