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Association of trace metals with various sedimentary phases in dam reservoirs

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ABSTRACT: In the present study sediment and water samples collected from Kowsar Dam reservoir in Kohkiluye and Boyerahmad Province, southwest of Iran, are subjected to bulk digestion and chemical partitioning. The concentrations of nickel, lead, zinc, copper, cobalt, cadmium, manganese and iron in water and bed sediment were determined by atomic absorption spectrometry. The concentrations of metals bounded to five sedimentary phases were estimated. On this basis, the proportions of natural and anthropogenic elements were calculated.The anthropogenic portion of elements are as follows: zinc (96 %)> cobalt (88 %)> iron (78 %)> magnesium (78 %)> nickel (78 %)> copper (66 %)> lead (63 %)> cadmium (59 %). The results show sediment contamination by nickel, cadmium and lead, according to the world aquatic sediments and mean earth crust values. Manganese and copper have strong association with organic matter and are of high portion of sulfide bounded ions. Finally, The degree of sediment contamination was evaluated using enrichment factor, geo-accumulation index (Igeo) and pollution index (IPoll). The sediments were identified to be of high cadmium and lead pollution index. The pattern of pollution intensity according to enrichment factor is as follows; manganese (1.25) < copper (1.63) < zinc (1.93) < cobalt (2.35) < nickel (3.83) < lead (12.63) < cadmium (78.32). Cluster analysis was performed in order to assess heavy metal interactions between water and sediment. Accordingly, nickel, cadmium and copper are earth originated. Zinc, copper and manganese are dominated by pH. All the elemental concentrations in water and sediment are correlated except for sedimental copper.

Keywords: *Anthropogenic; Lithogenous; Sediment; Trace metals*

INTRODUCTION

Dams are important structures from the environmental effects point of view. It has been proved that sediments entering a dam reservoir contain materials from neighboring geological formations and organic substances from plants and animal remains decomposed in water (Barretoo *et al.*, 2008). The concentration of organic matter increases gradually in dam reservoir. The accumulation of organic substances can both affect the quality of reservoir water and change the oxidation state into anoxic condition. Reduction condition together with low pH will cause mobilization of metals to upper water levels (Biati *et al.*, 2010). This may lead to serious changes in the water quality of the reservoir. Interactions commonly occur in the sediment-water interface (Van de Guchte, 1992; Chapman, 2000). As a result, investigation of both water and sediment quality is required to examine the quality of reservoir water.

Metal pollution is a serious problem in aquatic systems since their accumulation in the living organisms is toxic and environmentally non-biodegradable (Forstner *et al.*, 1990; Hart *et al.*, 1991; Schuurmann and Market, 1998). Thus, studying water and sediment quality is important to evaluate metal concentrations, since it provides useful information on pollution level in a reservoir. This has already been investigated in various environmnets in several researches (Priju and Narayana, 2007; Opuene *et al.*, 2008; Al-Juboury, 2009; Kho *et al.*, 2009; Taghinia Hejabi *et al.*, 2010). To assess the extent of sediments pollution, determination of the total heavy metal concentration is not sufficient. Identification of the chemical form of the metal in sediments is also required to determine the metal behavior and remobilization in the environment. In addition, the extent of

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contamination can be understood by the determination of the metals distribution between different phases and their origins (natural or anthropogenic) (Forstner, 1985). The five sedimentary phases are: 1) Loosely, 2) Sulfide, 3) Organic-metallic, 4) Resistant, and 5) Within lattice bounded ions.

To evaluate heavy metal loads, usually establishing the contributions of heavy metals in various soluble fractions is also required. These fractions are commonly quantified by a sequential extraction procedure (Horowitz *et al.*, 1999; stamatis *et al.*, 2006).

Kowsar Dam has been constructed on Kheirabad River in Kohkiluye and Boyerahmad Province, southwest of Iran, in 2004. Sampling was carried out in an area between the longitudes of 50° 39' W and 50° 43' E and latitudes of 30° 35' S and 30° 38' N (Fig. 1).

The main goals of the current investigation are: 1) To assess the extent of metals concentrations and their origin and speciation; 2) To identify the association of metals with different sedimentary phases and; 3) To evaluate the aquatic system pollution using Muller's geochemical index (I_{geo}), pollution index (I_{POLL}) and enrichment factor (EF).

The metals selected for the present study include Ni, Pb, Zn, Cu, Co, Cd, Mn and Fe. Furthermore, Loss on ignition (LOI) and pH for sediments were also selected for this investigation.

This research was carried out in the Faculty of Environment laboratory, University of Tehran, between October 2010 and February 2011.

MATERIALS AND METHODS

Twelve sediment samples were collected from different stations in the bottom of the dam reservoir. Also, water samples were collected from the surface, middle depth and bottom in each station. Sampling was carefully performed by professional divers, with the least disturbance in sediment layer. Water samples were stored in 1000 mL polyethylene containers, previously rinsed with the water of the site. The brown to black-colored sediment samples were dried in oven at 50 °C and passed through a 63-μm mesh (ASTM E-11).

About 0.5 g of sediment sample was placed in a beaker and reacted with 0.1 mL of 1 N HCl. As the first step of bulk digestion, 7 mL of a mixture of HCl and HNO_3 (1:3) was added. Subsequently, the mixture was heated on a sand bath at 125 °C, and brought into complete evaporation. To dissolve organic bounds, 7 mL of H_2O_2 was added and the flask was placed on heat until it was almost dried. This was followed by the

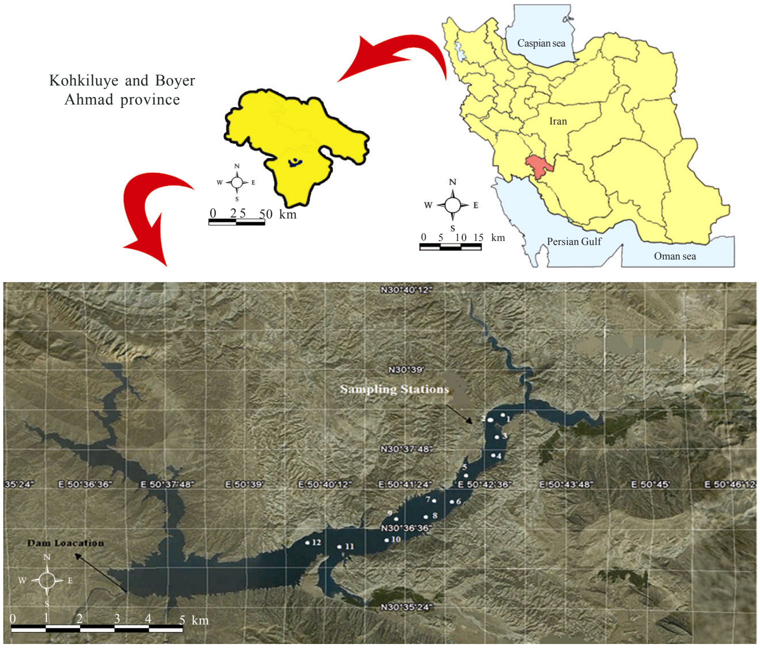

Fig. 1: Location map of Kowsar Dam reservoir in Kohkiluye and Boyer Ahmad province, Iran and the sampling sites

addition of additional 4 mL of H_2O_2, and the sample was allowed to dry. Subsequently, the samples were allowed to cool down to room temperature, then transferred to 50 mL volumetric flasks and brought to volume with distilled water. The samples were further filtered through Whatman .045 µm membrane filters, and stored at 4 °C (Jain *et al.*, 2005).

Chemical partitioning studies were also conducted. The following four steps were implemented: 1) Acetic acid 25 %v/v, 2) Acetic acid 25 % v/v-0.1 M hydroxylamine hydrochloride, 3) 30 % H_2O_2, and 4) Hot 50 % HCl (Gibs, 1973). To assure the accuracy of the results, all containers used for laboratory works were soaked in dilute HNO_3 overnight and rinsed with distilled water prior to use. All acids used in this study were Merck analytical reagent, which supplies the accuracy required for the experiments.

The analysis of metals (i.e., Ni, Pb, Zn, Cu, Co, Cd, Mn and Fe) present in water and sediment samples was carried out using a flame atomic absorption spectrophotometer (model 119 UNICAM).

Blanks and duplicates were run alongside with the samples. The analytical accuracy was approximately ±5% for all elements. Organic matter was determined by measuring the LOI of samples heated for 4 h at 450 °C in a muffle furnace (Carver, 1972).

Cluster analysis was carried out to determine the interrelationship amongst various parameters by the Weighted pair group method (WPG) (Davis, 1973) using MVSP software.

The speciation of the elements was also determined to identify the metal species existing in the water and sediment samples. Eh-pH diagrams were produced by the utilization of metal concentrations and molality and the sampling point's pressure (determined according to the elevation from sea level) in the HSC-Chemistry software. Using the output charts and oxidative-reductive potential (Eh) values measured by a digital voltmeter for the samples, the species present in sediment and water samples were determined.

I_{geo}, I_{POLL} and EF were computed in order to determine the pollution intensity of the Dam sediment using the following formulae:

$$I_{geo} = Log_2\left[\frac{Cn}{Bnx1.5}\right] \tag{1}$$

$$I_{Poll} = Log_2\left[\frac{Cn}{Bn}\right] \tag{2}$$

Where I_{geo} is the geochemical accumulation index, C_n is the metal concentration in sediment and B_n is the metal concentration in shale (Mediolla *et al.*, 2008). I_{POLL} is the pollution index, an optimized form of I_{geo} (Asaah *et al.*, 2005).

Seven contamination ranges have been determined to assess the metal pollution status (Martin and Meybeck, 1979):

Igeo ≤ 0 means unpolluted
0 < Igeo < 1 means unpolluted to moderately polluted
1 ≤ Igeo<2 means moderately polluted
2 ≤ Igeo<3 means moderately to strongly polluted
3 ≤ Igeo<4 means strongly polluted
4 ≤ Igeo<5 means strongly to very strongly polluted
Igeo ≥ 5 means very strongly polluted

Another approach to estimate the amount of pollution in sediment is to compute the Enrichment factor (EF) using the following formula (Karbassi *et al.*, 2008):

$$EF= [C_{metal}/ C_{Fe}]_{\ sediment}/ [C_{metal}/C_{Fe}]_{\ control} \tag{3}$$

Where C_{metal} and C_{Fe} are the concentrations of metal and Fe in sediments of present study and in an unpolluted environment (the earth crust) (Huu *et al.*, 2010). The pollution intensity is to be evaluated by the following categorizations for EF (Karbassi *et al.*, 2008):

EF<2 is deficiency to minimal enrichment
EF 2-5 is moderate enrichment
EF 5-20 is significant enrichment
EF 20-40 is very high enrichment
EF>40 is extremely high enrichment

Larger EF values show more contribution of the anthropogenic origins (Sutherland, 2000).

RESULTS AND DISCUSSION

Elemental concentrations (Ni, Cd, Zn, Pb, Cu, Co, Mn and Fe), along with organic matter content (LOI) and physico-chemical properties in sediment are presented in Table 1. The measured values are compared with the values from Earth crust and world sediments (Turekian and Wedepohl, 1961; Bowen, 1979). The mean concentrations of Ni and Pb in Kowsar Dam sediments were found to be considerably higher than that of these metals in aquatic sediments. The mean concentrations of Cd and Pb are also higher than the metals in mean earth crust (Bowen, 1979).

The results of the five-step sequential chemical partitioning are presented in Table 2. The percentages

Table 1: Concentration of metals and physical properties in Kowsar Dam bed sediments

Station No.	Ni	Cd	Zn	Pb	Cu	Co	Mn	Fe	LOI	Eh	pH
	mg/kg							%		mV	
1	83	6	35	40	25	11	250	1.26	4.04	-189	6.46
2	82	6	40	38	17	13	270	1.22	4.15	-180.2	7.23
3	57	5	30	50	17	10	250	0.86	70.49	-160.3	6.8
4	76	5.5	33	40	24	12	250	0.94	4.22	-169.2	6.59
5	60	6	30	39	19	12	250	1.12	26.15	-185.2	6.92
6	70	5.5	38	39	19	10	250	1.14	4.1	-184.1	7.15
7	82	5.5	37	48	19	11.5	260	1.20	51.91	-177.1	7.20
8	69	6	40	41	22	12.5	260	0.89	21	-180.9	6.49
9	77	6	32	39	23	13	250	0.99	64.7	-163.9	7.10
10	67	5.5	35	43	17	10	269	1.23	4.48	-186	6.56
11	82	5	36	50	17	11	260	1.26	4.16	-162.4	6.50
12	83	6	34	45	17	10	261	1.25	55.34	-179.7	6.94
Min	57	5	30	38	17	10	250	0.86	4.04	-189	6.46
Max	83	6	40	50	25	13	270	1.26	70.49	-160.3	7.23
Mean	74	5	35	42.7	19.7	11.3	256.7	1.11	26.23	-176.5	6.83
Earth crust*	80	0.3	75	14	50	20	850	4.6			
World sediments*	52	-	95	19	33	14	770	4.1			

of elements associated with different sedimentary phases are as follows:

Loose ions:

Cd (54 %)>Co (48 %)>Pb (43 %)>Mn (38 %)>Cu (29 %)>Zn (28 %)>Ni (17.9 %)>Fe (8 %)

Sulfide ions:

Mn (22 %)>Cu (21 %)>Zn (10 %)>Fe (7 %)> Co (6 %)> Pb (2 %)> Ni (1.75 %)> Cd (1 %)

Organic-metallic ions:

Pb (22 %)> Fe (22 %)> Mn (19 %)> Cd (8 %)> Ni (7.5 %)> Co (7 %)> Zn (5 %)> Cu (2 %)

Resistant ions:

Zn (57 %)> Fe (50 %)> Ni (38 %)> Co (34 %)> Cu (24 %)> Mn (8 %)> Cd (6 %)> Pb (6 %)

Within lattice: Cd (31%)> Pb (27%)> Cu (24%)> Fe (13 %)> Mn (13 %)> Ni (8.8 %)> Co (5 %)> Zn (0 %)

The results are also illustrated in Fig. 2.

As it is expected according to several studies (Turekian and Wedepohl, 1961; De, 1987), Mn (22 %) and Cu (21%) as cationic metals have strong associations with organic matter, because of their relatively high portions of sulfide bounded ions.

Furthermore, the anthropogenic and lithogenus shares of the metals were calculated by summing up the loosely, sulfide and organic bounded ions (Fig. 3). The following pattern shows the mean anthropogenic portions of the studied metals, as a percentage of their mean concentrations:

Zn (96 %)> Co (88 %)> Fe (78%)> Mn (78 %)> Ni (78 %)> Cu (66 %)> Pb (63 %)> Cd (59 %). Based on I_{geo} and the EF (Table 3), the level of pollution in the study area was determined using mean concentrations of metals in the aquatic sediments of the dam. The pollution intensity pattern according to EF is as follows: Mn (1.25) < Cu (1.63) < Zn (1.93) < Co (2.35) < Ni (3.83) < Pb (12.63) < Cd (78.32). According to the aforementioned EF categorizations, Pb and Cd are of "significant" and "very high" enrichment levels while the rest are of minimal to moderate pollution levels. The I_{geo} and I_{POLL} results are partially compatible with the EF results. According to these measures, the sediments are strongly polluted by Cd, while the remaining elements fall within their natural ranges respectively.

Dendogram of cluster analysis (Fig. 4) shows that Ni, Cd, Fe and Cu form cluster "A" show similar behavior. Since nickel is an indicator of oil pollution, it can be concluded that the concentrations of Cd, Cu and Fe are due to contamination by oil. This is supported by the fact that the Gachsaran oil field is located about 30 km from the reservoir. Since iron is known to be a lithogenous index, it can be also concluded that Ni, Cd and Cu are originated from the earth crust. This interpretation is compatible with the results of partitioning (Fig. 3), as the lithogenous portion of Cu and Cd contents is significantly high compared to other elements.

There is also a high correlation between pH and Zn, Co, and Mn in cluster "B". This indicates that these elements are mainly governed by the pH of sediments.

In cluster "C" LOI and lead strongly correlate with the Eh of the sediments. This firstly defines the effect of organic matters on the Eh value, which is related to

Table 2: Chemical partitioning of trace metals in Kowsar Dam bed sediments

Station No.	Ni (mg/kg)					Cd (mg/kg)					Zn (mg/kg)					Pb (mg/kg)				
	a	b	c	d	e	a	b	c	d	e	a	b	c	d	e	a	b	c	d	e
1	17	1.8	10.5	38.3	15.5	2.8	0.3	0.5	0.3	2.3	7.5	5	0.5	24.5	0	17	1	4.5	2.5	15
2	19.3	1	3.5	49.8	8.5	3	0	0.8	0	2.3	0.4	9.6	7	23	0	18.8	1.3	5	0.8	12.3
3	17.5	2.5	8.5	26	2.5	3.5	0	0	0.8	0.8	32.5	0	0	35.8	0	20	0	19.2	4	6.8
4	18.25	2.25	7.5	38	10	3.40	0.2	0.4	0.8	0.8	5.2	0	6	32.5	0	17.5	1.25	5	1	15.25
5	18.7	1	10.5	44.8	0	3.2	0.25	0.35	0.2	1.9	4.8	9.5	5	32	0	18	1.2	6.5	1.5	11.8
6	17	2.5	9.5	44	0	2.95	0.1	0	0.8	1.7	25.6	7.5	0	24	0	20	0	5.3	1	12.75
7	17.2	1	8	45	10.8	2.8	0.1	0.7	0.4	1.5	0.5	0	1.5	23	12	18.50	1.25	17	3.3	8
8	19	2	4.75	29.6	13.7	2.9	0.05	0.5	0	2.5	21	9.6	0.5	23	0	17.25	0	17.4	4	2.35
9	17.4	1.25	3.75	32.7	21.9	3	0	0.75	0	2.3	31.80	8	0	31	0	19	1	4.5	3	11.5
10	19	1	8.75	39	0	3.2	0.05	0	0.7	1.6	7.8	6.9	5	31.3	0	19.8	1	13	1.5	7.7
11	17.34	2.5	5.5	37.8	18.9	3.5	0	0.3	0.5	0.7	9.5	0	0	25.3	1.3	17.40	1.25	9	2.8	19.6
12	17.42	2.25	9.25	31.1	22.9	3	0.2	0.75	0	2.1	15.4	7.50	7	27.8	0	18.75	1	8.5	3.8	13
Min.	17	1	3.5	26	2.5	2.8	0	0	0	0.8	0.4	0	0	23	0	17	0	4.5	0.8	6.8
Max.	19.3	2.5	10.5	49.8	15.5	3.5	0.3	0.8	0.8	2.3	32.5	9.6	7	35.8	0	20	1.3	19.2	4	15
Mean	17.9	1.8	7.5	38	8.8	3.1	0.1	0.4	0.4	1.8	13.5	4.9	2.5	27.8	0	18.6	0.8	9.6	2.4	11.4

anoxic environment, and secondly shows the organic source of Pb in the sediment. This is compatible with the results of chemical partitioning indicating that 22 % of total Pb content is bounded to organic ions.

The presence of Mn, Co, Zn, and Pb in a separate cluster from Fe can also show that these elements are not earth-originated. Partitioning results are also consistent with this.

The speciation of metals by HSC Chemistry software is indicative of the presence of the following species in sediments:

NiO, Cd^{2+}, Zn^{2+}, $Pb(OH)^+$, Cu, Mn^{2+}, Fe_3O_4, $Co(OH)_2$.

According to HSC Chemistry software, the speciation of metals in water is as follows:

Fe_3O_4, Mn^{2+}, $Co(OH)_2$, $Co(OH)^+$, Cu, $Pb(OH)^+$, Zn^{2+}, NiO,Cd^{2+}.

Therefore, it can be noted that the speciation of metals within the water column and sediments are somehow alike. This is clearly indicative of active water rock inter-action in the area of study. Here are some definitions about the aforementioned species, contributing to determine their characteristics in sediment and water:

Nickel (II) oxide is notable as being the only well characterized oxide of nickel. Several million kilograms are produced in varying quality annually, mainly as an intermediate in the production of nickel alloys (Rosental *et al.*, 1986).

Cadmium prefers oxidation state +2 in most of its compounds. It occurs as a byproduct of zinc production (Lascelles *et al.*, 2005). Cadmium-containing ores are rare and are found to occur in small quantities. However, traces do naturally occur in phosphate, and have been shown to transmit in food through fertilizer application (Kirk and Othmer, 1985).

The only common oxidation state of zinc is +2. The element is normally found in association with other base metals such as copper and lead in ores. The dumps of the past mining operations leach significant amounts of zinc and cadmium. It is also used for galvanization process (Jiao *et al.*, 2004).

The hydrolysis of Pb^{2+}, under progressively increasing alkaline conditions, forms $Pb(OH)^+$, $Pb(OH)_2$(aqueous), $Pb(OH)_3^-$, and other species, including several polynuclear species (Broadley *et al.*, 2007). Metallic lead does occur in nature, but it is rare. Lead is usually found in ore with zinc, silver and (most abundantly) copper and is extracted together with these metals (Wiberg *et al.*, 2001) .

Manganese is found as a free element in nature (often in combination with iron), and in many minerals. As a free element, manganese is a metal with important industrial metal alloy uses, particularly in stainless steels and as an electrode in dry cells. The most stable oxidation state for manganese is +2 (Samans, 1949).

Fe_3O_4 is a ferrimagnetic mineral commonly known as Magnetite, one of several iron oxides and a member of the spinel group. Small grains of magnetite occur in almost all igneous and metamorphic rocks. Magnetite also occurs in many sedimentary rocks, including banded iron formations (Rayner-Canham and Overton, 2003).

Cobalt (II) hydroxide is the chemical compound composed of cobalt and the hydroxide ion (Harrison *et al.*, 2002). It finds the usage as a drying agent for paints, varnishes and inks, in the preparation of other cobalt compounds, as a catalyst, or in the manufacture of battery electrodes (Lide, 1998).

Results of water metal concentrations in the three aforementioned depths are presented in Fig. 5. Although the general trend of the concentrations seems to be ascending from the surface to the bottom level of water in the diagrams, there are still some exceptions such as Ni, Cd and Co. This may indicate that the concentration of most of the metals at the bottom of the reservoir is more than that of metals in the surface. Thus, it is concluded that the concentration of metals accumulated in the water adjacent to sediment layer is more, compared with the accumulation in other water levels.

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Table 3: Comparison of various pollution indices in Kowsar Dam bed sedimants

Metal	Mean concentration (mg/Kg)	I_{geo}	EF	I_{Poll}
Ni	74	-0.46	3.83	0.12
Cd	5.67	3.66	78.32	4.24
Zn	35	-2.03	1.93	-1.44
Pb	42.67	-	12.63	-
Cu	19.67	-1.78	1.63	-1.19
Co	11.33	-1.33	2.35	-0.75
Mn	256.67	-2.31	1.25	-1.73

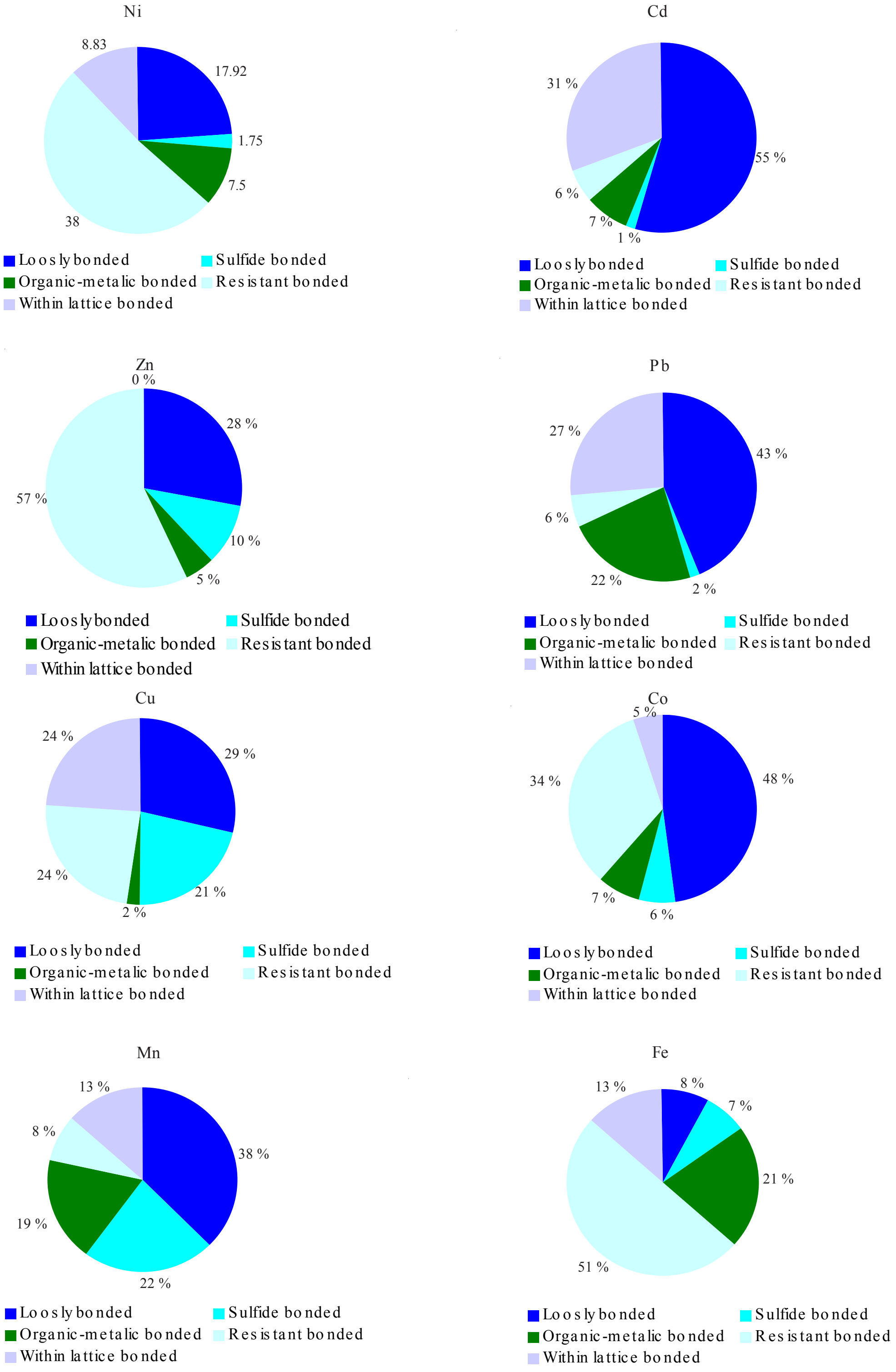

Fig. 2: Association of metals with various sedimentary phases in Kowsar Dam

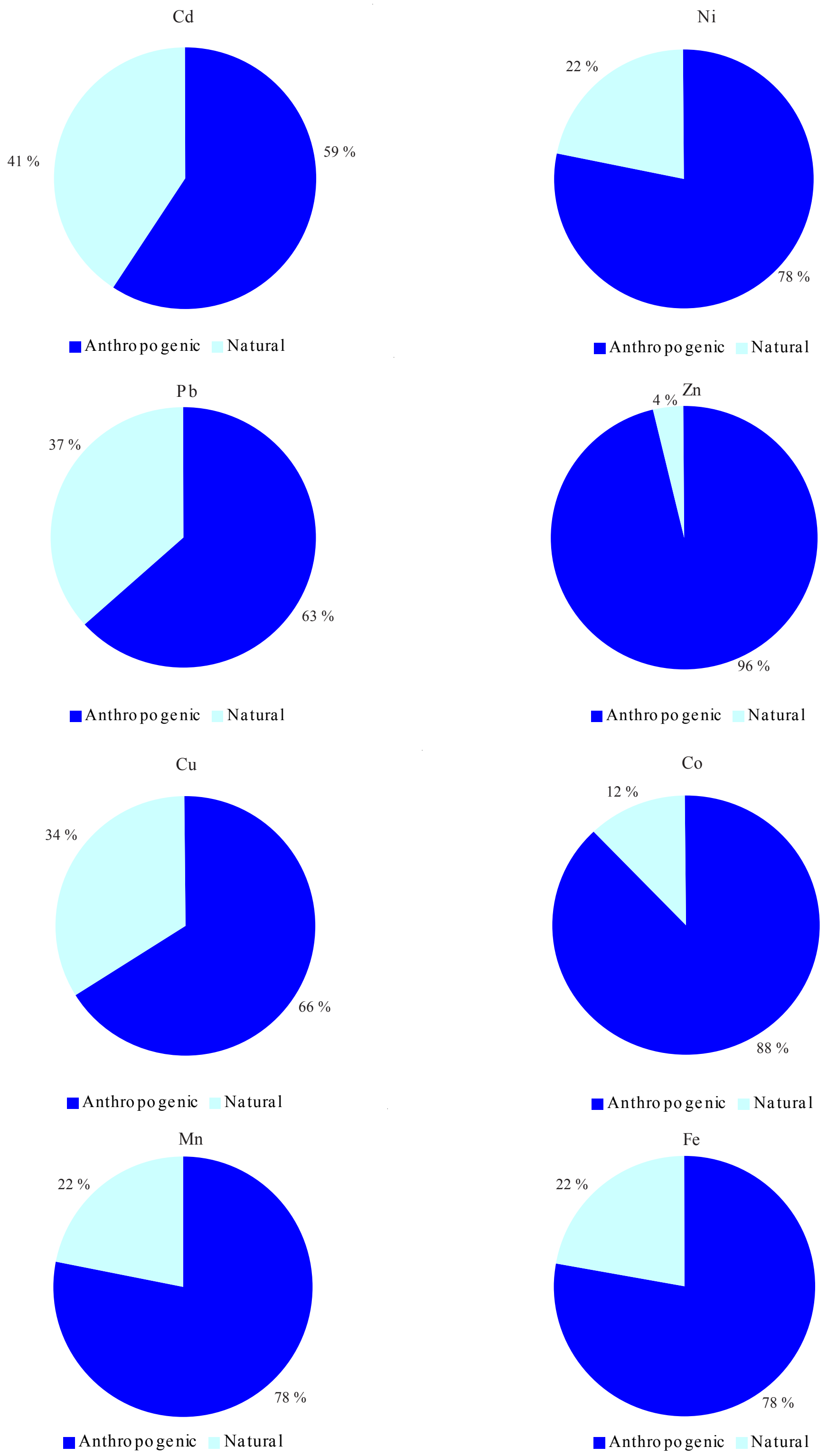

Fig. 3: Pie diagrams showing natural and anthropogenic portions of metals in the bottom sediments of Kowsar Dam

contributing to determine their characteristics in sediment and water:

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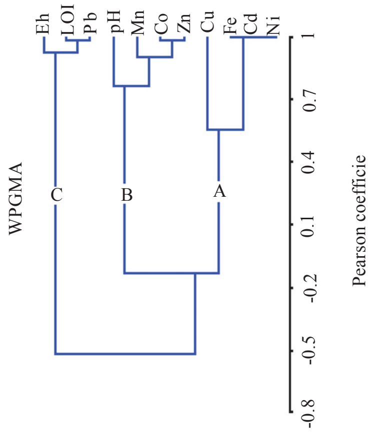

Fig. 4: Dendogram of cluster analysis amongst trace metals in Kowsar Dam bed sediments

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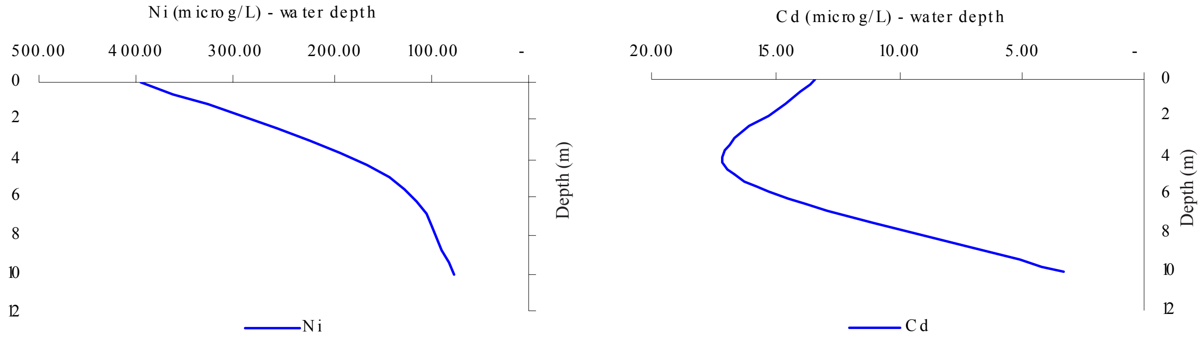

Fig. 5: Diagrams of water metal concentrations in various water depths of Kowsar Dam

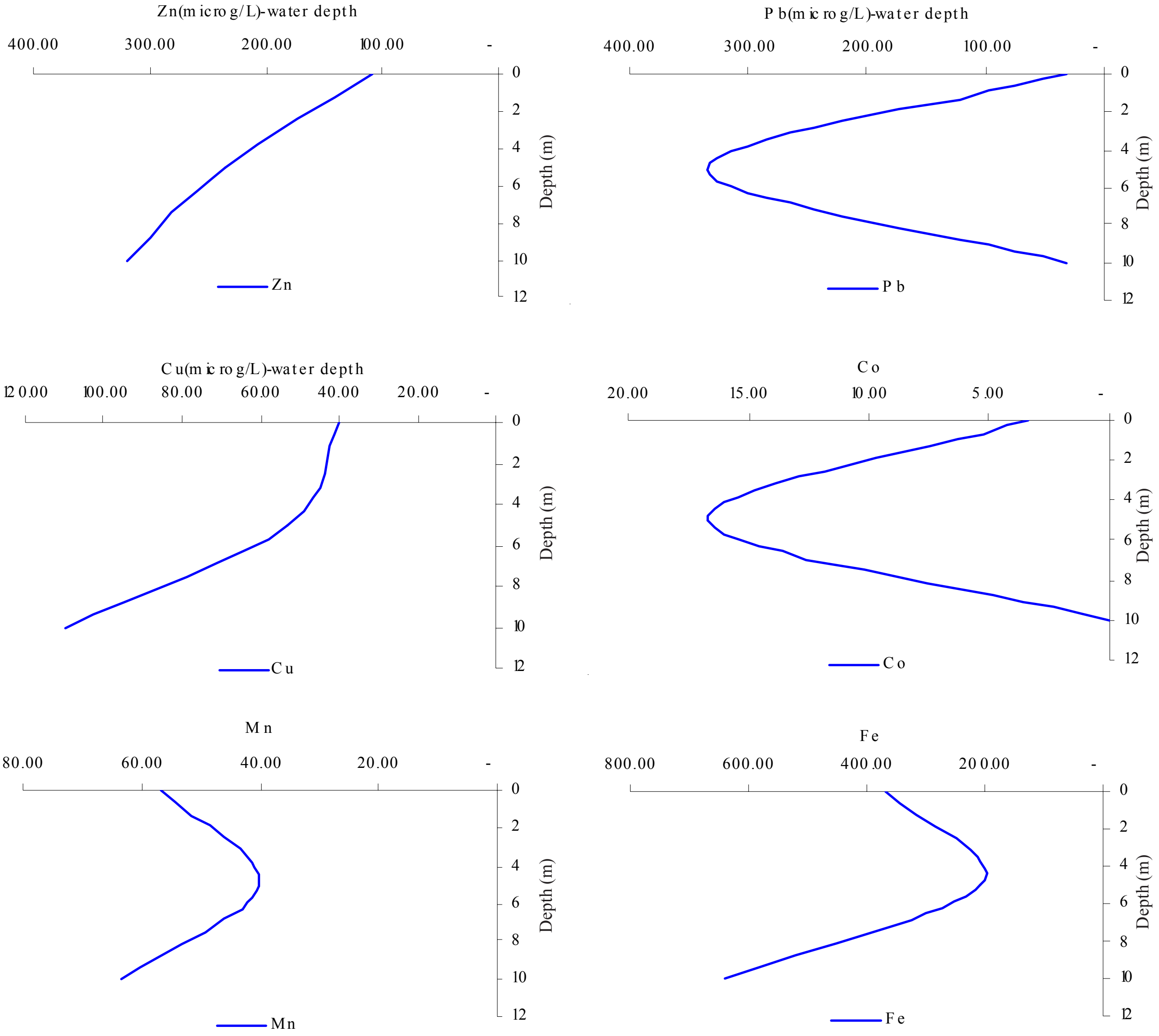

Fig. 5 (continues): Diagrams of water metal concentrations in various water depths of Kowsar Dam

level of water in the diagrams, there are still some exceptions such as Ni, Cd and Co. This may indicate that the concentration of most of the metals at the bottom of the reservoir is more than that of metals in the surface. Thus, it is concluded that the concentration of metals accumulated in the water adjacent to sediment layer is more, compared with the accumulation in other water levels.

CONCLUSION

Based on the results of this study, the portions of all metals bounded to anthropogenic phase were higher than that of them bounded to natural phase in sediments. This exhibits that a great portion of metal contents originates from anthropogenic origins such as municipal wastewater discharges to the reservoir rather than lithogenous origins. Sediments are contaminated with Ni, Cd and Pb when compared to the world sediments and mean earth crust. Strong association of Mn and Cu with organic matter shows that these elements tend for organo metallic bounds. The higher concentrations of Cd and Pb are clearly shown by various pollution indices. According to the cluster analysis, Ni, Cd and Cu are lithogenous, i. e originate from sources such as neighboring terrestrial formations. This clearly shows that in the absence of chemical partitioning studies, the statistical analysis may lead to wrong interpretations.

Zn, Co and Mn are governed by pH. All elemental concentrations in water and sediment are correlated with each other (except for Cu content of sediments). Cd, Fe and Cu are due to oil contamination with the provenance of Gachsaran oil field near the reservoir. Since the speciation of metals within the water column and sediments are somehow alike, it might be inferred that equilibrium exists as a result of water rock interaction in the area of study.

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