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ORIGINAL ARTICLE

Mineralogy, composition and heavy metals' concentration, distribution and source identification of surface sediments from the saline Maharlou Lake (Fars Province, Iran)

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Abstract

This study concerns the mineralogy, spatial distribution and sources of nine heavy metals in surface sediments of the Maharlou saline lake, close to the Shiraz metropolis in southern Iran. The sources for these sediments were studied by comparing the mineralogy and the distribution of heavy metals, using multivariate statistical analysis (correlation analysis and principal component analysis). The geochemical indices, including geo-accumulation index (Igeo), contamination factor (CF) and pollution load index (PLI), were used to assess the degree of heavy metal contamination in surface sediments. Sediment quality guidelines (SQGs) have also been applied to assess its toxicity. The XRD analysis shows that the main minerals of the surface sediments are aragonite, calcite, halite and quartz, with small amounts of montmorillonite, dolomite and sepiolite. The total heavy metal contents in surface sediments decrease in order of Sr > Ni > Cr > Zn > Cu > Co > Pb > As > Cd and the average concentrations of Sr, Ni and As exceeded more than 10, 5 and 3 times, respectively, by comparing with the normalized upper continental crust (UCC) values. The results of pollution indices (Igeo, CF and PLI) revealed that strontium (Sr), nickel (Ni) and arsenic (As) were significantly enriched in those sediments. Based on the sediment quality guidelines (SQGs), Ni would infrequently cause toxicity. Multivariate statistical analysis indicated that the Ni, Co and Cr came mainly from natural geological background sources, while Cd, Cu, Pb, and Zn were derived from urban effluents (especially traffic emissions) and As originated from agriculture activities. Significant relationships of Sr with S, CaO and MgO in sediments suggest that Sr was derived from carbonate- and gypsum-bearing catchment source host rocks.

Keywords Maharlou Lake · Sedimentology · Mineralogy · Pollution · Heavy metals

Introduction

Lakes cover a small portion of the Earth's surface, but play a crucial role in local environmental modifications and their pollution may have serious consequences on sustainability of lake's natural environments (Hairston and Fussmann 2001; Hou et al. 2013). Sedimentology has a major role in basin analysis, petroleum systems and also major role in environmental studies (Karimian Torghabeh et al. 2014, 2015; Karimian Torghabeh and Pimentel 2016). Heavy metals are deemed hazardous pollutants because of their toxicity, durability, non-degradability and accumulation in biological tissues (e.g., El Nemr et al. 2016). Sediment pollution of lake systems by heavy metals around the world is a common environmental problem due to urbanization, industrialization and agricultural activities (e.g., Arnous and Hassan 2015; Lin et al. 2016; Zhang et al. 2016). The source, behavior, mobility, controlling factors, as well as the bioaccumulation and possible health impacts of heavy metals in aquatic sediments, have been discussed in many studies during the last 20 years (Ajima et al. 2015; Bourg and Loch 1995; Liaghati et al. 2004; Malik et al. 2009; Singh et al. 1999; Stead-Dexter and Ward 2004; Yi et al. 2011; Zwolsman et al. 1993).

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Generally, composition of surface sediments is influenced by both geological conditions, (e.g., mineralogy or morphology of the catchment area) and human activities (Dinelli et al. 2005; Sindern et al. 2016). Moreover, mineralogy is the main geological signature on the chemical composition of sediments (Pehlivanoglou 2001). It has been validated that lake environments may be contaminated by heavy metals both from geogenic and anthropogenic sources (El-Amier et al. 2017; Hu et al. 2012; Wenchuan et al. 2001). These elements can be added into the lakes from natural sources, such as rock weathering, erosion, river flows and runoff, and also from anthropogenic activities, including agricultural fertilizers leaching, discharge of untreated industrial and urban wastewaters, energy production, domestic sewage and mining operations (Arnous and Hassan 2015; Kamala-Kannan et al. 2008; Özmen et al. 2004; Yuan et al. 2014).

In aquatic systems, sediments provide a valuable tool for assessing heavy metal pollution because they have great absorption capacity and also are known as the primary accumulator of heavy metals (Wang et al. 2015a). Changes in different physico-chemical characteristics can cause the heavy metals to be released from the sediments to the overlying waters and thereby surface sediments can act as probable secondary sources of heavy metals (Mamat et al. 2016; Wang et al. 2015b, c). Thus, sediments are a good exploratory instrument for initial environmental assessment and also to understand the possible sources and distribution of heavy metals, to distinguish geogenic from anthropogenic contributions to the heavy metal accumulation in surface sediments, necessary for sediment quality assessments and aquatic environmental protection (Lin et al. 2016; Wang et al. 2015a).

Heavy metal contamination in aquatic systems in Iran is becoming a serious problem, due to rapid economic growth in recent years (Abdollahi et al. 2013; Amini Ranjbar 1998; Karbassi et al. 2007). Maharlou Saline Lake is one of the most important water bodies on the Fars province and is facing the threat of wastewater resulting from anthropogenic pollution input. The lake has been exploited for various chemical compounds, especially for the petrochemical industry, and its water is used for salt extraction. Heavy metal pollution is therefore becoming a critical issue in the Maharlou saline Lake management. Comprehending the status of heavy metal pollution in the surface sediments of Maharlou saline Lake is essential for remediating pollutants in the environment. Some studies have attempted to determine and evaluate the heavy metals in salt and sediments of Maharlou saline lake (Forghani et al. 2009; Hatami-Manesh et al. 2014; Moore et al. 2009). However, none of these investigations attempted to determine possible sources of heavy metals in the surface sediments of Maharlou Lake and the relationship they might have been, associated with the sediment mineral composition.

The main goals of this study are: (1) to assess contamination levels of heavy metals such as arsenic (As), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), nickel (Ni), lead (Pb), zinc (Zn) and strontium (Sr) in the surface sediments; (2) to investigate the spatial distributions of the heavy metals; (3) to determine the relationship between metal concentrations in the sediments and their mineral composition, to assess the geogenic influence; and (4) to identify the potential sources of heavy metals by the use of the multivariate statistical analysis.

Geology and study area

The Maharlou lake basin is situated in Iran, about 18 km south east of Shiraz city (municipality of Fars province), between latitudes 29°17′–29°33′ North and longitudes 52°42′–52°59′ East (Fig. 1). The lake is very shallow, with an average depth of 1 m. Nevertheless, in recent years, the maximum depth of the lake is 50 cm due to successive drought stress. In wet seasons, the area of the lake develops up to 280 km^2 (26 km long and 12 km wide). The main flow direction of drainage systems guided by the topographic slope is from northwest to southeast and it receives water from direct precipitation and water inflow from surface runoff, with a few seasonal rivers and several karstic springs compensating for the strong evaporation in a semi-arid climatic context (Dumas et al. 2003). In the study area, there is no permanent river running into the lake, the drainage system being composed of seasonal rivers including Khoshk River and Chenare-Rahdar streams (Fayazi et al. 2007). Khoshk River flowing through the Shiraz city has the largest discharge to Maharlou Lake. In the last 30 years, Shiraz has experienced rapid urbanization and population growth, and large quantities of pollutants have been discharged into the river, meaning that the Khoshk River has become progressively polluted and large volumes of heavy metals have been incorporated into the sediments. Consequently, this river is seemingly the main source of the lake's pollution. The region is subjected to several human activities (e.g., agriculture, pisciculture) and its ecosystem has been significantly impacted by untreated agricultural, industrial, sewage and household effluents. However, based on its special geographical position and function, Maharlou Lake acts as an important preservation area.

Geologically, the Maharlou saline lake is located in the Zagros Fold–Thrust Belt zone (Alavi 1994), developed in a ramp valley tectonic setting (Faghih et al. 2012). The study area is mainly characterized by sedimentary rocks such as conglomerates, limestones, marls and shales. The geologic formations in the study area, from youngest to oldest are: (1) recent alluvium, (2) interbedded buff to red sandstones, silty marls and marlstone of Aghajari

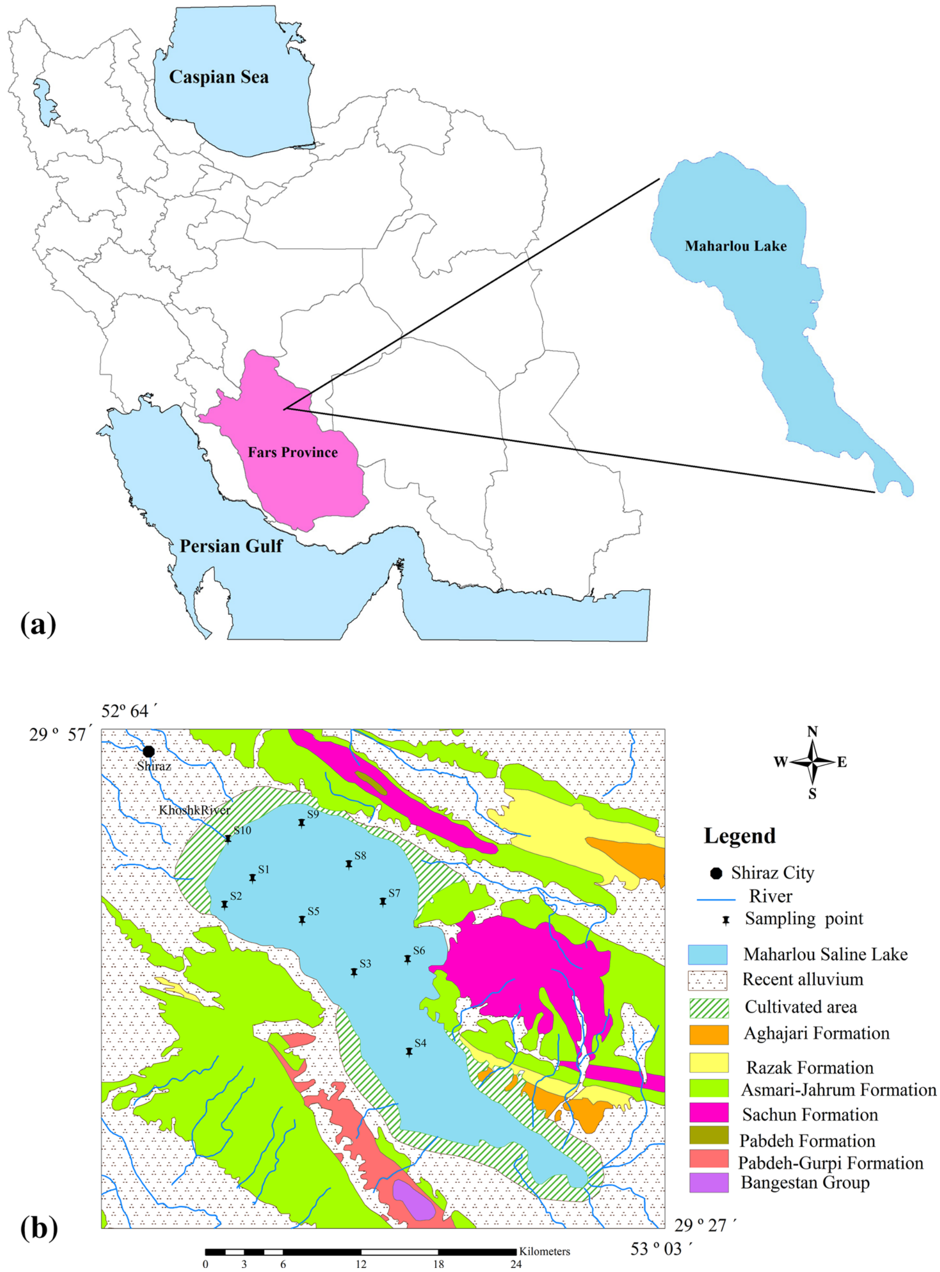


Fig. 1 Location of the study area and geological map of the Maharlou saline lake (Andalibi et al. 2001); S1–S10 are sampling sites

Formation (Late Miocene), (3) marls interbedded with red sandstones, limestone and occasional gypsum beds of Razak Formation (Early Miocene), (4) limestone and marl of Asmari–Jahrum Formation (Miocene to Oligocene), (5) red gypsiferous marls and gypsum beds of Sachun Formation (Paleocene), (6) marl of Pabdeh–Gourpi Formation (Paleogene to Late Cretaceous), (7) limestone and marly limestone of Bangestan Group (Late Cretaceous) and (8) Precambrian evaporates.

Hydrogeochemistry of the lake water is controlled mainly by the abundance of the evaporates, especially by salt domes piercing the surface, which are being eroded and drained, causing excessive salinity (Fayazi et al. 2007).

Sampling and laboratorial analysis

In July 2017, ten surface sediment samples were collected in Maharlou Lake (Fig. 1) at locations within the main body of the lake, selected to provide good area coverage. The sampling sites have been defined to represent different areas of the lake and therefore different proximities and influences of industrial, urban and agricultural activities. The top layer of the sediment was collected from a depth of 0–20 cm using the handheld polyvinyl chloride (PVC) corer whit a diameter of 8 cm and a length of 30 cm. Sediments were placed in sealed polyethylene plastic bags and labeled. Sediment samples were kept cooled prior to laboratory processing. In the laboratory, sediment samples were ground in an agate mortar after drying in air and removing debris. Then, samples passed through a 63-μm sieve to obtain silt and clay fraction for evaluation of total concentration of heavy metals.

Approximately 0.5 g of dried surface sediment samples were weighed and digested under heat with a hydrochloric acid and nitric acid mixture, either the 3:1 true aqua regia (HNO_3:HCl = 1:3). Upon completion of the digestion step, samples were made up to volume with deionized water. Finally, all sediment samples have been analyzed using Inductively Coupled Plasma Mass Spectrometry (Perkin Elmer/NexION 300D) technique at MS Analytical Laboratories Ltd., Canada (accredited under ISO/IEC 17025:2005). To evaluate the quality of chemical analysis, standard reference materials (STD OREAS 24b and STD OREAS 601) and a reagent blank were used. The precision of the analytical procedures, expressed as the relative standard deviation (RSD), ranged from 0.03 to 9.1%.

Mineralogical composition and major element contents of surface sediments were determined using X-ray diffractometry (XRD) and X-ray fluorescence (XRF), respectively.

Generally, there are two types of normalization methods for studying heavy metal contamination in aqueous sediments: (1) methods based on granulometric approaches and (2) methods using geochemical approaches. Most scholars such as Ho et al. (2012) believe that granulometric normalization cannot fully account for metal variability, and therefore the geochemical approach is more commonly used, considering that conservative elements such as Al, Fe, and Sc, represent one or more of the major sedimentary metal carriers (e.g., clay minerals, iron and manganese hydr/oxides, etc.).

Data analysis

Geochemical indices

In the studied area, three different indices including contamination factor (CF), pollution load index (PLI) and geo-accumulation index (IGeo) were used to assess the degree of heavy metal contamination in surface sediments of the Maharlou saline lake. The background values play a pivotal role in evaluating polluted degree of heavy metals (Rubio et al. 2000; Yang et al. 2009), but the attempt to get unpolluted samples for background determinations has been proved to be difficult at the study area, which has a long history of human activities. In this paper, the elemental abundances in the upper continental crust were used as background referenced (Taylor and McLennan 1985).

The geo-accumulation index (IGeo) method was first introduced by Muller (1969) and has been widely adopted to evaluate the contamination levels of heavy metals in soils or sediments, in terms of their corresponding geochemical baseline. This index was calculated, for each element, as follows:

$$\mathrm{IGeo} = \log_2(\mathrm{Ci}/(1.5\ \times \mathrm{Bi})),$$

where Ci is the concentration of concerned element in sediment sample; Bi is the geochemical background concentration or reference value of the element and the constant 1.5 is the geochemical background matrix correction factor, used to reduce possible variations in the background values due to lithospheric effects. Müller (1981) established seven grades of geo-accumulation index values: IGeo ≤ 0 (Class 0), unpolluted; 0 < IGeo ≤ 1 (Class 1), slightly polluted; 1 < IGeo ≤ 2 (Class 2), moderately polluted; 2 < IGeo ≤ 3 (Class 3), moderately severely polluted; 3 < IGeo ≤ 4 (Class 4), severely polluted; 4 < IGeo ≤ 5 (Class 5), severely extremely polluted; IGeo > 5 (Class 6), extremely polluted.

The contamination factor (CF) and pollution load index (PLI) are commonly used to appraise the status of heavy metal contamination (Xu et al. 2017).

The contamination factor of each element was calculated using the following ratio:

$$\mathrm{CF} = \mathrm{Cx}/\mathrm{Cb},$$

where CF is the contamination factor, while Cx is the concentration of element of interest at a site and Cb is the concentration of the same metal at a background or reference value.

CF is classified into four levels: CF ≤ 1 indicates low contamination factor, 1 < CF ≤ 3 indicates moderate contamination factor, 3 < CF ≤ 6 indicates considerable contamination factor, and CF > 6 indicates very high contamination factor (Hakanson 1980; Thomas 1975).

The pollution load index (PLI) for investigating the pollution status of different samples was calculated using the following equation:

$$\mathrm{PLI} = \ (\mathrm{CF1} \times \mathrm{CF2} \times \mathrm{CF3} \times \cdots \times \mathrm{CF}n)\ 1/n,$$

where n is the number of studied metals and CF is the calculated contamination factor. PLI value of > 1 is polluted whereas < 1 indicates no pollution (Tomlinson et al. 1980).

Toxicity evaluation of heavy metals in surface sediments

Numerical Sediment Quality Guidelines (SQG) have been applied to assess the toxicity of heavy metals in sediments (Macdonald et al. 2000). In the present study, SQGs were developed to determine the adverse biological effects of heavy metal pollution in the surface sediment from the Maharlou Lake. This method provides two levels of concentration: the threshold effect concentration (TEC) and probable effect concentration (PEC). Heavy metal concentrations at each sample sites were compared with the consensus-based SQG values referred to as TEC and PEC (Macdonald et al. 2000; Baran et al. 2016) (Table 2).

Multivariate statistical analysis

Multivariate statistical analysis including Pearson's correlation analysis and Hierarchical cluster analysis were carried out to be used to evaluate the heavy metal concentration relations, and in addition to be used to identify the possible sources of heavy metal contamination in sediments. Statistical analyses were performed using SPSS 19.0, in this paper, to be used to indicate the sources of heavy metals in surface sediments found in Maharlou saline lake.

Results and discussion

Mineralogy and major element concentrations

The mineral composition of five surface sediment samples has been determined using X-ray powder diffractometry (XRD). The data from XRD analysis show that Lake surface sediments are predominantly composed of calcite, argonite, halite, gypsum, quartz, montmorillonite, dolomite and sepiolite (Fig. 2).

Concentrations of major elements (oxides) in surface sediments of the Maharlou saline lake are presented in Table 1. CaO is the most abundant compound, with

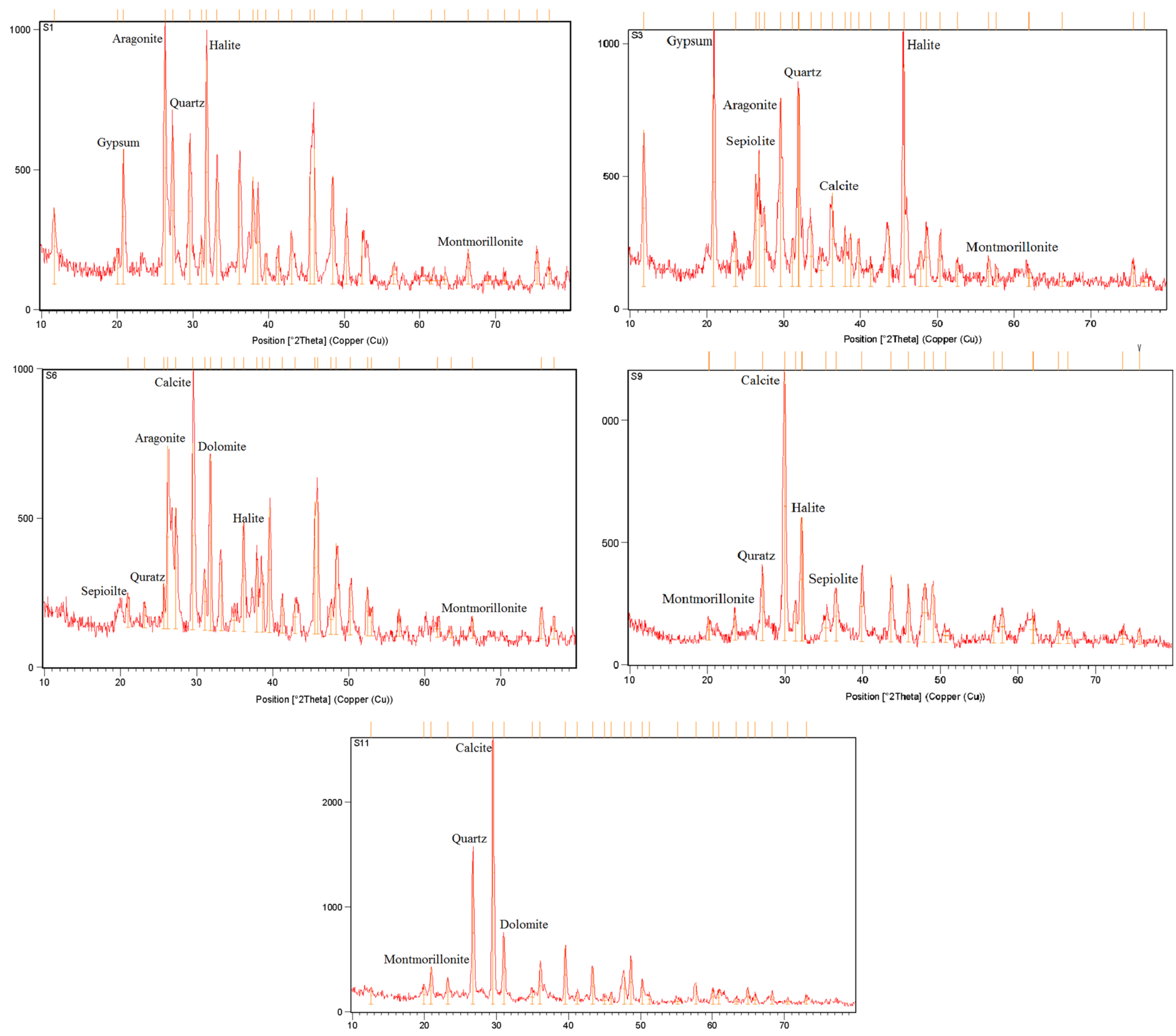


Fig. 2 X-ray diffraction patterns of the samples collected from surface sediment of Maharlou Lake

Table 1 Summary statistics of concentration levels of major elements' oxides (%) in surface sediments of Maharlou Lake

	Oxide of silicon (SiO_2)	Oxide of aluminium (Al_2O_3)	Oxide of iron (Fe_2O_3)	Oxide of calcium (CaO)	Oxide of magnesium (MgO)	Oxide of sodium (Na_2O)	Oxide of potassium (K_2O)	Oxide of titanium (TiO_2)	Oxide of phosphorus (P_2O_5)
Mean	21.90	4.26	2.70	24.92	8.85	2.91	1.10	0.23	0.12
Maximum	29.43	6.32	3.97	28.74	11.11	4.83	1.64	0.36	0.17
Minimum	14.09	2.43	1.09	20.46	6.62	0.70	0.27	0.11	0.07
SD	6.31	1.51	1.15	3.36	1.90	1.58	0.54	0.09	0.04
UCC[a]	66	15.4	5.6	3.59	2.48	3.27	2.8	0.64	–

[a]Taylor and McLennan (1985)

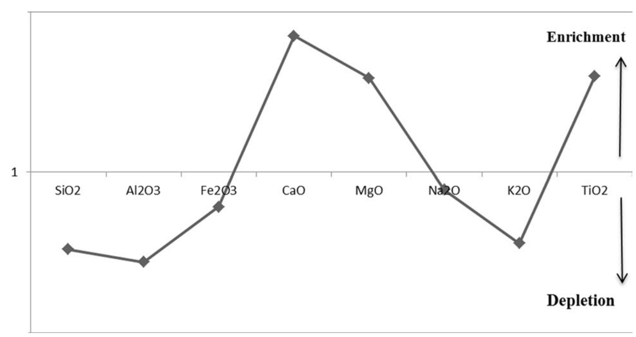


Fig. 3 Major element contents of Maharlou Lake sediments compared to upper continental crust (UCC)

percentages ranging from 20 to 28%, whereas SiO_2 is the second most abundant compound, ranging from 14 to 29% by mass. Total concentrations of MgO range from 6 to 11%, Al_2O_3 from 2 to 6%, Fe_2O_3 from 1 to 3%, Na_2O from 1 to 4%, K_2O from 0.7 to 1.5%, TiO_2 from 0.1 to 0.3% and P_2O_5 from 0.07 to 0.17%.

Major element concentrations suggest, besides the obvious abundance of carbonate and evaporates minerals, the presence of aluminosilicates and heavy minerals, and to a lesser extent a P-containing mineral phase.

To facilitate the interpretation of Maharlou Lake sediments, the values of chemical composition have been compared with UCC—"upper continental crust normalized compositional distributions" (Taylor and McLennan 1985), as presented in a spider diagram (Fig. 3). By comparison with the values of UCC, the contents of SiO_2, Al_2O_3, Fe_2O_3, Na_2O and K_2O are low (depleted) and the contents of CaO, MgO and TiO_2 are enriched. Depletion of SiO_2, Al_2O_3 and Fe_2O_3 may be due to low levels of those compounds in the predominantly carbonated host rocks of the region. On the other hand, enrichment for CaO and MgO indicate the abundance of carbonate minerals in the study area. Depletion of Na_2O represents the formation of an independent salt phase from the sediments in the lake. The higher concentration of TiO_2 throughout the samples in comparison with the UCC could be attributed to heavy minerals present in this region.

Heavy metal concentrations, spatial distribution and background values

Heavy metal concentrations in surface sediments of the Maharlou saline Lake and descriptive statistics are summarized in Table 2. The total heavy metal contents in surface sediments decrease in the order of Sr > Ni > Cr > Zn > Cu > Co > Pb > As > Cd. The highest mean concentration of Sr and As was at S1 sampling site, while Cd, Cu, Pb and Zn showed the maximum level at S10 site. The highest mean value of Cr was found at S4. Similarly, Co and Ni showed the maximum concentration at S7 site.

Figure 4 shows the spatial distribution of As, Cd, Co, Cr, Cu, Mo, Ni, Pb, Zn and Sr in surface sediments from Maharlou saline Lake, using contour maps. These maps are valuable tools for the distinct zones of lower or higher concentration heavy metals illustrated in the studied area.

In the study area, computed coefficients of variation (CV%) for individual elements were as follows: As (12.08%); Cd (42.3%); Co (34.22%); Cr (28.33%); Cu (50.79%); Ni (32.12%); Pb (49%); Zn (39.29%) and Sr (74.60%) (Table 1). However, spatial distribution of these elements in the surface sediments was quite comparable (Fig. 4).

The patches of higher concentrations for As are mainly confined in the North area around Khoshk river (lake inlet), Northeast and South areas (Fig. 4a). The relative low concentration of Arsenic was observed at sites S2 and S5, rather far from agricultural lands. As can be seen from Fig. 4b, the distribution of Cd is quite similar to that of As. The area with higher concentration of Cd was confined to the North portion of the lake, which is close to farmlands. Co, Cr and Ni had generally similar spatial distributions in the lake (Fig. 4c, e, f). Their concentrations decreased from the Northeastern > the Northern > the Southern > the Western parts of Maharlou saline Lake. The spatial distributions of Zn and Pb in surface sediments (Fig. 4g, h) are similar to Cd, with the highest concentrations also appearing in the Northern part of the lake.

Spatial distribution of Cu concentrations in surface sediments is shown in Fig. 4d. Similar to Pb, Cd, and Zn, the

Table 2 Sample site coordinates, summary statistics of concentration levels of heavy metals (mg/kg), sodium and sulfur (%) in surface sediments of Maharlou Lake and guidelines for maximum freshwater sediment concentrations (WCTMRL, mg/kg)

	Elemental concentrations											Sample site	X	Y
	As	Cd	Co	Cr	Cu	Ni	Pb	Zn	Sr	Na	S	S1	669176.8	3262828.3
Mean	4.67	0.11	10.92	60.81	51.35	104.90	8.0	58.18	4172.5	3.1	1.24	S2	667260.1	3261007.3
Maximum	5.30	0.20	15.20	81.00	107.0	138.90	16.3	101.0	10,000	5.81	4.34	S3	676116.4	3256395.8
Minimum	3.70	0.04	5.0	31.00	22.50	47.0	3.0	28.0	565.0	0.16	0.08	S4	679881.2	3250959.2
SD	0.56	0.48	3.73	17.23	26.08	33.69	3.91	22.82	3112.9	1.7	1.5	S5	672561.9	3259965.8
CV (%)	12.08	42.43	34.22	28.33	50.79	32.12	49.0	39.22	74.60	54.83	120.0	S6	679777.7	3257296.3
UCC	1.5	0.098	17	35	25	20	20	71	350	–	–	S7	678086.6	3261216.5
WCTMRL	–	0.1–1.5	–	20–190	20–90	30–250	10–100	50–250	60–750	–	–	S8	675763.6	3263771.9
TEC	–	0.99	–	43	31.6	22.7	35.8	121	–	–	–	S9	672527.4	3266601.6
PEC	–	4.98	–	111	149	48.6	128	429	–	–	–	S10	667500.7	3265502.5
< TEC %	–	100%	–	10%	30%	0%	100%	100%	–	–	–	–	–	–
≥ TEC > PEC (%)	–	0%	–	90%	70%	10%	0%	0%	–	–	–	–	–	–
≥ PEC	0%	0%	–	0%	0%	90%	0%	0%	–	–	–	–	–	–

UCC: upper continental crust (Taylor and McLennan 1985)

World Common Trace Metal Range in Lake sediment (Förstner and Whitman 1981)

TEC: threshold effect concentration

PEC: probable effect concentration (Macdonald et al. 2000)

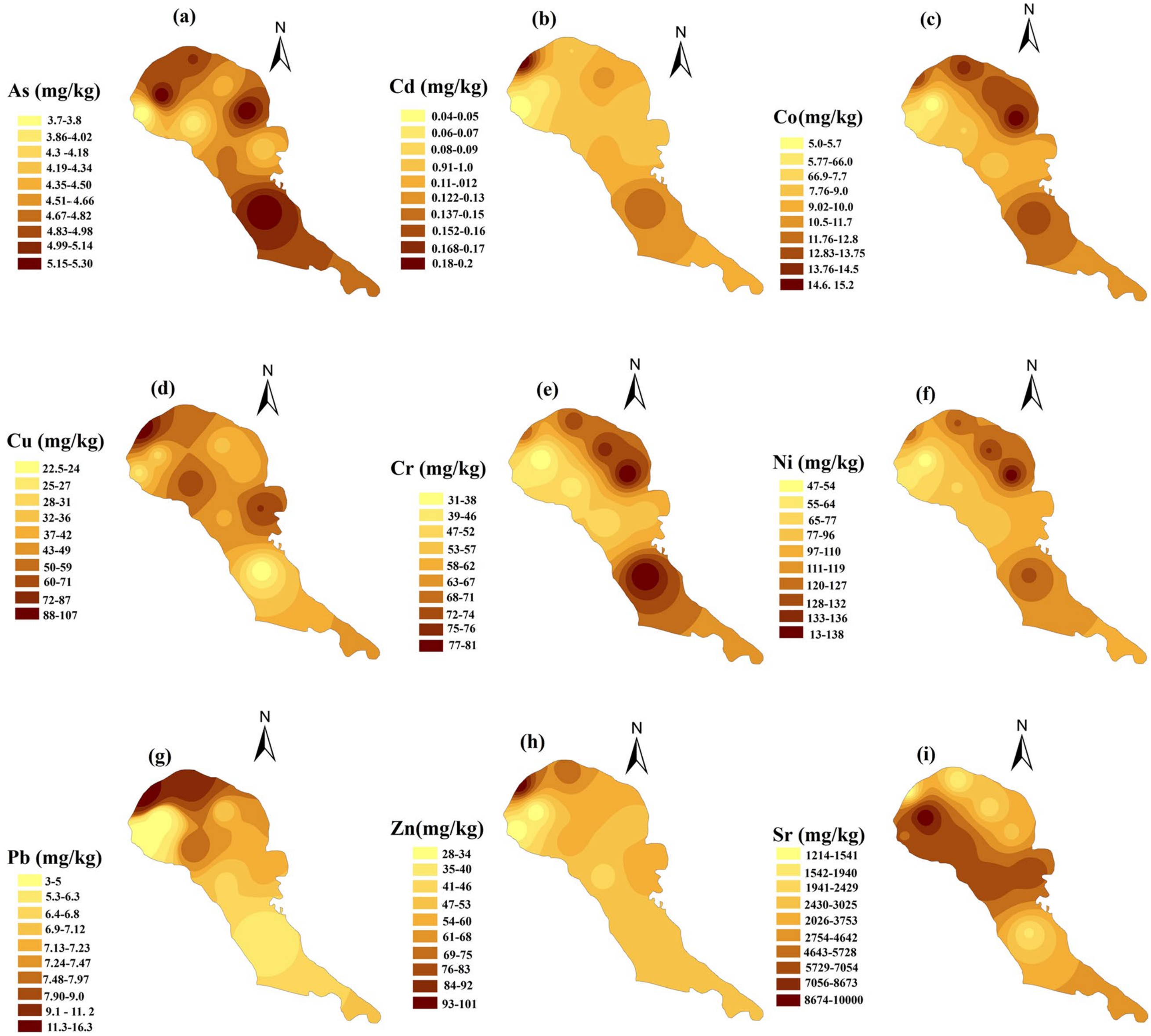


Fig. 4 Spatial distributions of heavy metals in surface sediments of the Maharlou Lake: As (**a**), Cd (**b**), Co (**c**), Cu (**d**), Cr (**e**) and Ni (**f**); Pb (**g**), Zn (**h**) and Sr (**i**)

patch of the highest concentration for Cu also appeared in the inflow area of Khoshk River (Lake inlet). The zones with higher concentrations of Sr are confined to the Northwest and West portions of the lake (Fig. 4i). The similar spatial distribution patterns between elements in the sediments of this lake may indicate that they have the same geological and anthropogenic input sources.

The relative higher level of heavy metals was observed in the inlet lake (Khoshk River-S10), and therefore pollution sources are interpreted to be around this site. There are two possible reasons that might explain the high concentration of Cd, Cu, Co, Pb, Zn and As at this site. The first possibility is that agricultural and industrial wastewater from the surrounding area was discharged into the Maharlou Lake. On the other hand, it may be due to inflows from the Khoshk River (urban runoff in Shiraz city). This is further supported by the fact that concentrations of Cr, Ni, Pb, Zn, and Cd in the sediments of Khoshk River are higher than that in the Maharlou Lake (Salati and Moore 2010). It turns out that sample 4, which is located adjacent to the Khoshk River, has a high concentration of As, Cd, Co, Pb, Zn, Ni and Cr.

To better understand the heavy metal pollution in the study area, concentrations of these pollutants were compared with upper continental crust (UCC) and World Common Trace Metal Range in Lake sediments (WCTMRL) (Table 2). It is evident that the average of all heavy metals measured in these sediments, except Co and Pb, exceeded the corresponding values of UCC. This is especially true for Sr, Ni, As and Cu, which are 11.9, 5.24, 3.2 and 2 times the UCC values, respectively. In addition, it is obvious that the present sediment ranges of Cu and Sr are higher, whereas Cd, Cr, Ni and Pb are lower than the corresponding values of WCTMRL.

Assessment of sediment contamination

Geoaccumulation index (IGeo)

The heavy metal IGeo in Maharlou saline lake's surface sediments is shown in Fig. 5. The IGeo values are the following (presented in decreasing order): Sr with 1.27 to 0.03 (average 0.76), Ni with 0.27 to 0.66 (average 0.51), As with 0.21 to 0.37 (average 0.31), Cu with −0.22 to 0.45 (average 0.08), Cr with −0.22 to 0.18 (average 0.04), Cd with −0.56 to −0.13 (average −0.14), Zn with −0.58 to −0.06 (average −0.29), Pb with −1.0 to −0.26 (average −0.62), and Co with −0.70 to −0.22 (average −0.39). Negative values of IGeo were determined for Cr at sites 1, 2, 3 and 5, for Cu at sites 1,2,4,7 and 8, for Cd at all sites except sites 4 and 10, and for Pb, Co and Zn at all sites, suggesting that these sites were not polluted by these metals. The IGeo values for As and Ni were under 1 in the sediments of all sites, which usually had unpolluted to moderately polluted class values. Among nine studied metals, Sr had the highest IGeo values. The highest Igeo values of metals studied were found in the sediments of site-10. The IGeo class values of As, Cd, Cr, Cu, Ni and Sr was "slightly polluted" for sediments of site-10, while the Igeo class values of Co, Pb and Zn was "unpolluted to moderately polluted". Total Igeo values for the sampled sites followed this decreasing order: S10 > S9 > S6 > S7 > S5 > S8 > S3 > S1 > S2.

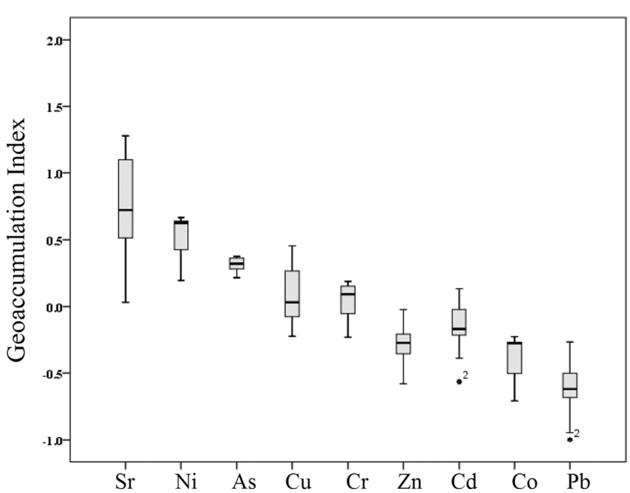


Fig. 5 Box plots of geoaccumulation index (Igeo)

Contamination factor (CF) and pollution load index (PLI)

Figure 6 shows the results of CF. The highest CF values, for Sr (very high contamination), were found at all sites except sites 8, 9 and 10. CF values higher than 6, which indicate "very high contamination", were observed for Ni at sites 4, 7, 8, 9 and 10.

For other elements, the CF values indicated low contamination to considerable contamination (As, Cd, Co, Cr, Cu, Pb and Zn). From the CF values, contamination with the investigated elements can be observed, especially with Sr, Ni and As.

Figure 7 shows the PLI values of the studied elements. The PLI values varied from 1.40 to 1.49 for the investigated sediments, suggesting that pollution does exist.

Sediment quality guidelines

The TEC illustrates chemical concentrations below which toxic effects would be rarely observed, whereas the PEC

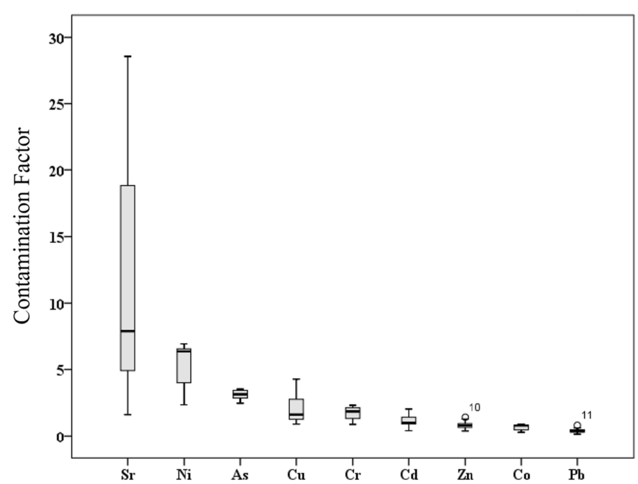


Fig. 6 Box plot of geoaccumulation index (Igeo)

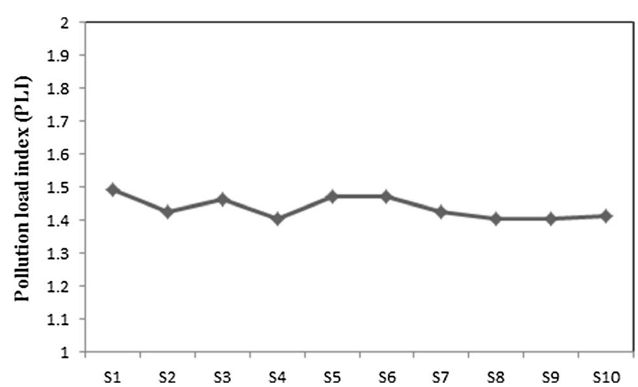


Fig. 7 PLI values of the studied elements in surface sediments of Maharlou Lake

is a concentration above which adverse biological effects are likely to occur (Table 2). According to Macdonald et al. (2000), sediment samples with metal concentrations between the TEC and PEC were predicted to be non-toxic. When compared to the TEC–PEC SQG, 90% (Cr), 70% (Cu) and 10% (Ni) of the samples from Maharlou Lake fell into the range between TEC and PEC (Fig. 8). In the case of all metals in the sediments, 90% (Ni) of samples were above the PEC guideline for this metal. Therefore, adverse biological effects are probable due to the concentrations of this metal in the Maharlou Lake sediments. Concentration of As, Cd, Pb and Zn measured in this study was basically lower than TEC (Fig. 8).

Multivariate statistics

As heavy metals have toxic properties, it is imperative to detect possible sources of pollution to protect the

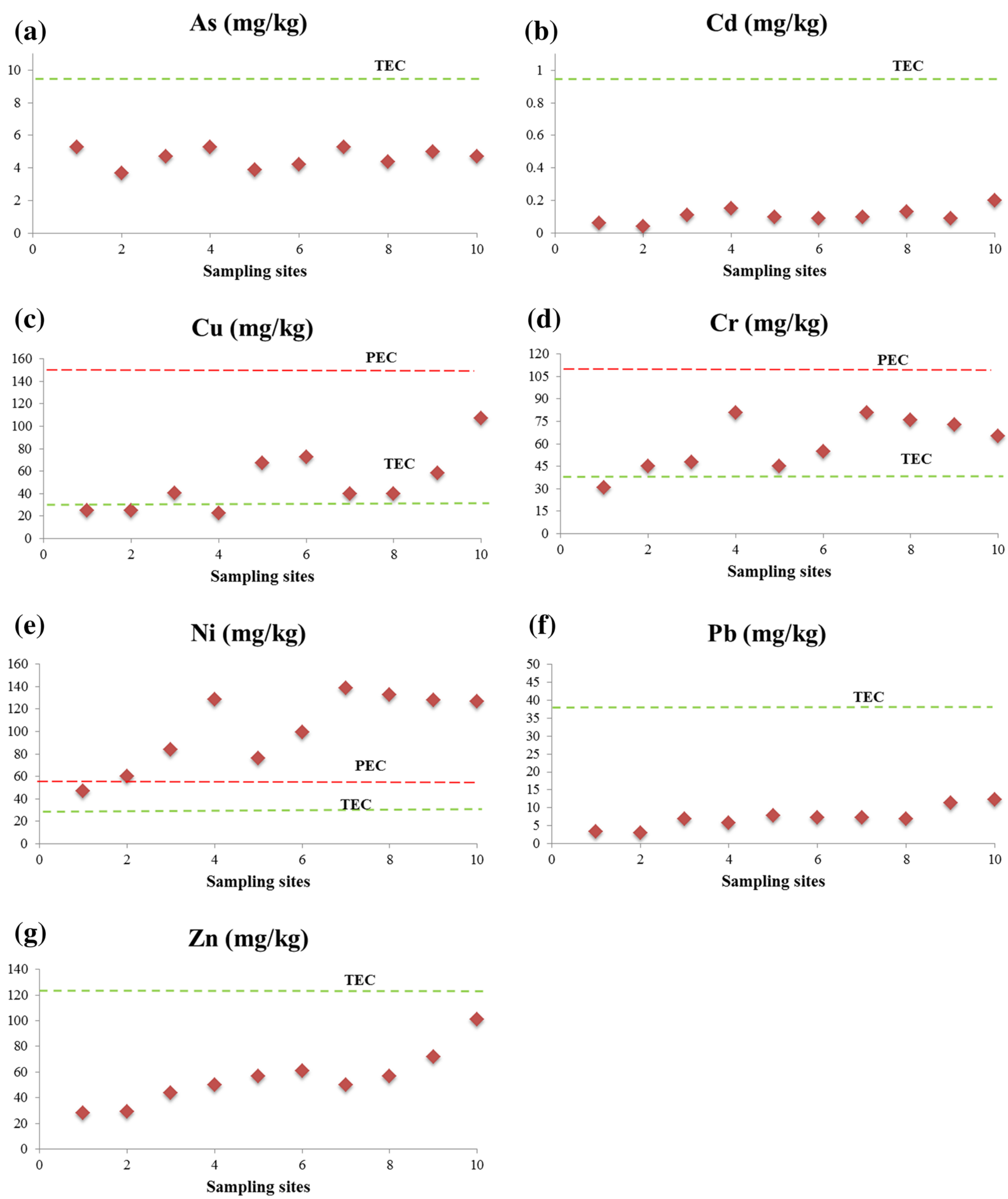


Fig. 8 Comparison of heavy metal concentrations with SQGs: As (**a**), Cd (**b**), Cu (**c**) and Cr (**d**); Ni (**e**), Pb (**f**) and Zn (**g**)

environment and improve its environmental and living conditions. The possible anthropogenic and/or geogenic sources of heavy metals in the surface sediments of Maharlou Lake were explored by combining the spatial distribution patterns of the heavy metals with multivariate statistical results (correlation analysis and hierarchical cluster analysis). In this case, correlation analysis was used to evaluate the correlations among nine elements in the surface sediments. Furthermore, to determine the most common pollution sources, the sediment heavy metal content and its mineral composition (based on major oxides) were analyzed by hierarchical cluster analysis (CA).

Correlation studies

Pearson's correlation coefficients of heavy metals in surface sediments from the Maharlou Lake were determined and the results are given in Table 3. Concentration of As was not significantly correlated with any of the studied heavy metals, suggesting that As had a unique anthropogenic source. As is closely related to the intensive use of pesticides, herbicides and chemical fertilizers (Lin et al. 2016), and the Maharlou Lake catchment is dominated by agriculture (Djamali et al. 2009). Based on spatial distribution pattern of As, this most polluted area is the near-shore area of the Northern, Southern and Eastern parts of the lake, which is intensively occupied by agricultural land. In addition, the use of pesticides, herbicides and chemical fertilizers might be responsible for the As pollution in Maharlou Lake. However, very significant correlations were found between Zn and Cd ($r = 0.828$) Co and Cr ($r = 0.959$), Cr and Ni ($r = 0.958$), Cu and Zn ($r = 0.852$), Pb–Cd ($r = 0.737$) and Zn and Pb ($r = 0.926$) at the $p < 0.05$ level. Three elemental pairs, Cd–Co ($r = 0.680$), Ni–Cd ($r = 0.704$), and Pb–Cu ($r = 0.724$), had a significantly positive correlation at $p < 0.01$. Further, Cd, Cu, Zn and Pb were positively correlated among each other and might have common sources.

In urban areas, sediments of rivers are important sinks for heavy metal pollution. In this region, typical sources of heavy metal contamination, such as Cd, Cu, Zn and Pb, are traffic related including motor vehicles, vehicle exhaust particles, particles from roads, tyres and brakes; on the other hand, pavement surface, coal combustion, municipal wastes and industrial emission can contribute significantly to the heavy metals's concentration (Kabata-Pendias and Mukherjee 2007). In consequence, the anthropogenic load of Cd, Cu, Zn and Pb in Khoshk river sediments leads to an elevated level of these elements in the surface sediments of the Maharlou saline lake. This is confirmed by results presented by Qishlaqi et al. (2008) and Salati and Moore (2010), who also reported on heavy metal contamination of the Khoshk river sediments.

Similar spatial distribution patterns and good positive correlations between Ni, Cr and Co suggest that the content of these heavy metals share a geogenic source.

The negative correlations between Sr and other metals in the surface sediments (Table 3) suggest that the Sr had a different behavior or source.

Hierarchical cluster analysis (CA)

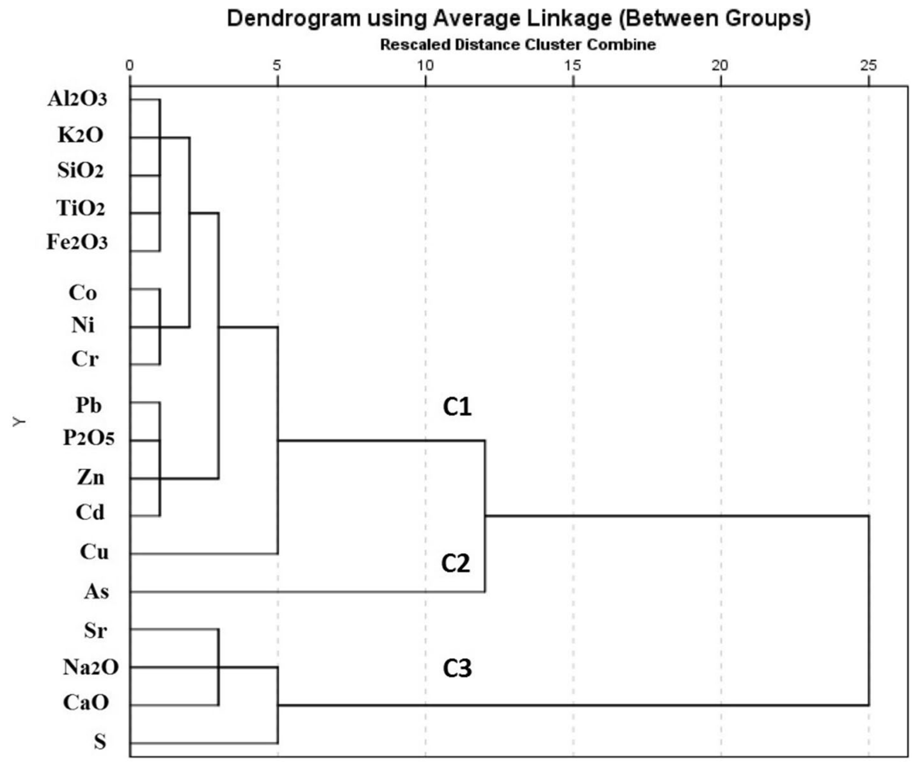
To further identify the possible sources of heavy metals in sediments, hierarchical cluster analysis (CA) was performed on As, Cd, Co, Cr, Cu, Ni, Pb, Zn, Sr, S, SiO_2, TiO_2, Fe_2O_3, Al_2O3, K_2O, P_2O_5, CaO, MgO and Na_2O concentrations. Cluster analysis of the concentration of heavy metals and major oxides data, generated dendrograms (Fig. 9) that revealed three distinct clusters (C1–C3). SiO_2, TiO_2, Fe2O3, Al_2O_3, K2O, P_2O_5 and Co, Cr, Ni, Pb, Zn, Cd, Cu are clustered as C1, while As occur in the C2 group. This reaffirms the findings of the correlation analysis that Cd, Co, Cr, Cu, Ni, Pb, Zn and As are derived from different sources. Sr, Na_2O, CaO and S from cluster 3, suggesting that sedimentary host rocks are influencing Sr concentration on surface sediment in the study area. These details will be fully discussed in the next section ("Source identification").

Table 3 Pearson correlation matrix of elements' variable in surface sediment of Maharlou Lake

Variable	As	Cd	Co	Cr	Cu	Ni	Pb	Zn	Sr
As	1								
Cd	0.29	1							
Co	0.46	0.68*	1						
Cr	0.41	0.55	0.95**	1					
Cu	− 0.18	0.56	0.24	0.29	1				
Ni	0.41	0.70*	0.98**	0.95**	0.26	1			
Pb	0.17	0.73**	0.61*	0.46	0.72*	0.63*	1		
Zn	0.09	0.82**	0.65*	0.47	0.85 **	0.67*	0.92**	1	
Sr	− 0.25	− 0.71	− 0.92	− 0.89	− 0.29	− 0.90	− 0.64	− 0.71	1

*$p < 0.05$; **$p < 0.01$

Fig. 9 Dendrogram showing the three main clusters of variables (chemical elements)



Source identification

Three main sources of the studied heavy metals in Maharlou surface sediment can be identified, according to their distribution, correlation coefficient analysis and hierarchical cluster analysis (CA, Fig. 9). Source I—Co, Cr and Ni mainly originate from geogenic source; Source II—Pb, Cd, Cu, Zn and As represent anthropogenic sources; Source III—Sr is controlled by sedimentary host rocks.

Source I includes SiO_2, TiO_2, Fe_2O_3, Al_2O_3, K_2O, and heavy metals Co, Ni and Cr. The association of Cr, Ni and Co in this cluster is consistent with the result obtained from spatial distribution patterns and correlation studies. Harami et al. (2003) and Salati and Moore (2010) reported that the Razak Formation has a high concentration of metals such as Ni and Cr in the Maharlou catchement area, thus suggesting that these elements may be of geogenic origin.

Source II includes P_2O_5, and heavy metals Pb, Cd, Cu, Zn. The observed well correlated relationship of Cd–Zn, Cd–Pb, Cu–Pb, Cu–Zn, and Zn–Pb may indicate possible identical sources of heavy metal contamination. Thus, this group can be interpreted as pollution related. The association of Cu, Pb, Cd and Zn in this cluster could be from nonpoint sources of urban effluent, such as urban runoff and traffic emission from Shiraz city (Moore et al. 2011). The presence of Cd and P_2O_5 in this group is explained by agriculture activities around the lake. Cd is typically considered as an indicator element of agricultural activities because it is one of the usual components in the phosphate-rich fertilizers (Arnous and Hassan 2015; Wang et al. 2015a), which are also a well-known source of As (Hartley et al. 2013).

Source III includes CaO, MgO and Na_2O, S and Sr, which are controlled by sedimentary host rocks of the study area. Sachun Formation plays an important role in the catchment area sedimentary rocks (Arzaghi et al. 2012). The mean concentration of Sr and Na in the Sachun Formation range from 90 to 890 and 250 to 8720 mg/kg, respectively (Shabafrooz et al. 2010). Therefore, association of Sr with S, CaO and MgO in the sediments suggests that Sr was derived from Sr-bearing evaporates (gypsum) and their carbonate-rich (aragonite, calcite and dolomite) host rocks of Sachun Formation. Both CaO and Na_2O exhibit a good association with Sr. According to Fig. 4 (spatial distribution pattern of Sr) and Table 2 (content of Na), concentrations of Sr and Na increase in the intermediate and central parts of the lake, and therefore increase in salinity towards the lake center. These results are in accordance with Önalgil et al. (2015), who considered that throughout the Seyfe Lake basin the association of Strontium with in situ gypsum crystals lead to a relative increase in salinity towards the lake center. However, based on this cluster and Table 1, carbonate minerals (dolomite, aragonite and calcite), gypsum and halite are the major factors to increase the levels of Sr concentration in surface sediments of Maharlou Lake.

Conclusions

This study analyzed the mineralogy, spatial distribution, contamination assessment and sources of nine heavy metals including As, Cd, Co, Cr, Cu, Ni, Pb, Zn and Sr in surface sediments from Maharlou Lake. The XRD analysis indicated that the main minerals of the surface sediments were aragonite, calcite, halite and quartz, with small amounts of montmorillonite, dolomite and sepiolite. Total heavy metal concentrations in the sediment samples from the Maharlou Lake followed the order: Sr > Ni > Cr > Zn > Cu > Co > Pb > As > Cd. The field study showed that Sr, As, Cd, Cu, Cr, Ni, and Zn in sediments of the Maharlou Lake were clearly indicating contamination, especially Sr, As and Ni which exceeded the UCC values more than 11, 5 and 3 times, respectively, while no significant Co and Pb pollution has been observed in the area.

The IGeo results suggested moderate pollution for As, Ni and Sr, while the CF study revealed low to considerable contamination by As, Cd, Co, Cr, Cu, Pb and very high contamination by Sr. Toxicity assessment of heavy metals in surface sediments, based on the SQGs, suggested that toxicity affected by Cr and Cu would occasionally occur. Ni would infrequently cause toxicity. Based on correlation matrix and hierarchical cluster analysis, the heavy metal sources could be divided into three: Source 1 includes Cr, Ni and Co which originated from Razak formation in the catchment area; Source 2 includes As, Cd, Cu, Pb and Zn related with anthropogenic sources, Cd, Cu, Pb and Zn mainly originated from the Khoshk River and other coastal rivers, and As might be affected by the agriculture activities; and Source 3 with Sr showing significant relationship with S, CaO and MgO in sediments, suggesting provenance from Sr-bearing evaporates and their carbonate host rocks, likely from the Sachun Formation. According to the spatial distribution maps, the northern part of the Maharlou Lake is identified as the primary region in need for environmental protection and management, because of the high contamination levels by heavy metals. In addition, controlling agricultural and industrial point/non-point sources pollution is required to decrease toxicity affected by As and Cd pollution in the future. Therefore, main strategies should focus on monitoring and reducing the discharge of urban, industrial and agricultural wastewater into the Maharlou lake basin, especially by the Khoshk River watershed.

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