ECOLOGICAL RISK ASSESSMENTS OF HEAVY METALS IN SURFACE SEDIMENTS COLLECTED FROM HAQAL COASTAL WATERS (TABUK REGION), SAUDI ARABIA

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Abstract. Haqal is a small city located in north-western part of Arabian Peninsula. The anthropogenic activities in this region are evident. This region is targeted for future development by Saudi government. In this study, the surface sediment of Haqal coastal waters were analysed for heavy metals (Cd, Cu, Fe, Ni, Pb, and Zn). The total concentration (mg/kg dry weight) ranged from 0.012-0.186 for Cd, 0.582-1.13 for Cu, 0.51-2.18 for Ni, 0.68-2.64 for Pb, 1.97-4.52 for Zn while for Fe, it ranges from 0.155 to 0.254%. Based on ecological risk assessment results, the values of PERI were categorised as 'low ecological risk', thus all sampling sites were unpolluted with heavy metals. Despite that, this monitoring study had a positive result for non-heavy metal pollution, future mitigation of the heavy metal pollution in coastal areas of Tabuk should be given priority by the authorities. The present study can be considered as the first effort to monitor the pollution of heavy metals in Haqal. This provides baseline information for future ecotoxicological studies which can involve application of bioindicators to assess the quality of the marine environment in this region.
Keywords: *The Red Sea, marine environment, Arabian Peninsula, pollution*

Introduction

Sediment is an important sink where pollutants are accumulated from the water body, and a secondary pollution source which might impact the surrounding aquatic ecosystem (Birch and Taylor, 2002; Wang et al., 2011; Hahladakis et al., 2016; Kumwimba et al., 2017; Wong et al., 2017). Thus, determining the ecological risks to aquatic ecosystems posed by heavy metal toxicity is crucial to be determined (Kumwimba et al., 2017; Wong et al., 2017). In this study, two approaches were deployed to assess ecological risks for heavy metals in sediments, i.e. 1) sediment total metal concentrations, and 2) metal GFs in sediment. The sub-approaches that are based on total metal concentrations are i) comparisons with sediment quality guidelines (SQGs), ii) geochemical pollution indexes (Igeo), and enrichment factor (EF), iii) contamination factor (Cf), potential risk of individual metal (Er) and potential ecological risk index (PERI), proposed by Hakanson (1980). These SQGs are developed for marine and estuarine ecosystems (Long et al., 1995) via (a) the effect range low

(ERL)/effect range median (ERM)/interim sediment quality value-low (ISQV-low) and (b) the threshold effect level (TEL)/probable effect level (PEL)/interim sediment quality value-high (ISQV-high) values.

Originating from Long and Morgan (1990), the SQG is an informal tool to evaluate sediment chemical data in relation to possible adverse effects on aquatic biota. The SQGs were proposed to be used as benchmark for evaluating sediment chemistry information to identify situations that are potentially harmful to aquatic organisms associated with bad sediments; as well as the benchmark to help set targets for sediment quality for the broader management strategy of long term aquatic ecosystem health sustainability management (CCME, 2002).

Among the basics of SQGs, SQGs based on ERL and ERM are used extensively in assessment of the pollutants' impact on environment. It has been tested against a large dataset gained in US EPA and US NOAA (National Oceanic and Atmospheric Administration, US). These datasets consisted of synchronized measurements of chemical and toxicological variables (O'Connor, 2004). However, O'Connor (2004) has made a clarification that SQG's ERL is not a threshold chemical toxicity in sediment and there is no basis for the assumption of pollutant concentration above ERL increase the probability of toxicity. However, there are still multiple recent heavy metal risk assessment studies that are employing ERL-ERM-based SQG at their assessment (Long and Morgan, 1990; Yap, 2010; Yap et al., 2002; Amin et al., 2009; Garcia et al., 2011; Ali et al., 2015). Therefore, the ERL and ERM approach of risk assessments are still viable approaches of heavy metal risk assessment and will still be used to evaluate the heavy metal risk in this study. In this study, the heavy metal levels in sediments were compared SQGs with multiple approaches (ERL, ERM, TEL and PEL) to assess and deduce the possible environmental impact of the sediment's heavy metals level. Hakanson (1980) has proposed a series of indexes as diagnostic tools for pollution control purposes, i.e. the contamination factor (Cf), risk index of individual metal (Er) and Total Risk Index (RI), and degree of contamination.

Ecological risk assessment (ERA) using above-mentioned approaches have been widely used in ERA-based studies. These studies reported have wide geographical distribution. In Asia region, there are such reports from Malaysia (Yap et al., 2002 and Yap, 2010), Khuzestan coastal waters, Iran (Madiseh et al., 2009), Northern Bohai and Yellow Seas, China (Luo et al., 2010), Dongjiang Harbor, China (Guo et al., 2010), Yangtze Estuary, China (Zhao et al., 2012), Lake Cıldır, Turkey (Kukrer et al., 2014), Ulsan Bay, Korea (Ra et al., 2014), and mangrove sediments of Peninsular Malaysia (Cheng and Yap, 2015).

Tabuk coastal areas including Haqal city supported high floral and faunal diversity. On the other hand, the environment of the coastal areas of Haqal is threatened by several human activities which may increase the pollution burden to the area. Studies on monitoring of heavy metals in these areas are scarce. On the other hand, this area has a promising future as it is targeted for future development with the country development plan. Thus, this study can be considered as pioneer in the assessment of heavy metal contamination of the coastal environment of Haqal. The present study aims to estimate the ecological risk assessments in the surface sediment collected from Haqal coastal waters based on 1) SQGs, 2) two geochemical pollution indexes, and 3) PERI.

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Materials and methods

Study site

Haqal is a small city belongs to Tabuk region and is located in the north-western part of Arabian Peninsula. It is located within geographical coordinates of 29° 17’ 39” North, 34° 57’ 4” East. It is situated in the northwest of Saudi Arabia near the head of the Gulf of Aqaba adjacent to Aqaba across the Jordanian border (lies about 5 km from Jordanian border). This is coastal area is not a port used for the Red Sea shipping. However, coasts of the region are scenic. The climate of this city is arid with annual rainfall of less than 50 mm. The rainy season is in the winter and the annual mean temperature is 24° C. This region is targeted for future development under the NEOM project. Therefore, it is expected that this region will witnessed huge infrastructure development and the environmental assessment is a necessary tool at this stage.

Collection of sediment samples

The surface sediments (3-5 cm from surface sediment) in 15 sites of Haqal coastal areas were collected on early summer of 2018 (*Fig. 1*). The plastic spoon was used to collect the top-sediment to minimize the metal contamination. Sufficient amount of the sediment was collected from each site. The collected sediment samples were transferred into an acid-washed polyethylene bag and brought back to laboratory for temporary storage and analysis. The samples taken back to laboratory were frozen (-20 °C) in a freezer prior to analysis.

Figure 1. *Map showing sampling area in Haqal coastal waters in Tabuk, Saudi Arabia*

Before the analysis, sediment samples were oven-dried at 60 °C for at least 16 h until constant dry weights were achieved. Then the dried sediment particles were sieved

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through a 0.50 mm stainless steel sieve. During the sieving process, the samples were also shaken vigorously to produce homogeneity. Only sediment particles with size below 63 µm in diameter are considered for metal analysis.

The analyses of total Cd, Cu, Fe, Ni, Pb and Zn concentrations in sediment samples were done according to direct aqua-regia method. After sieving, about one gram of each dried sample (Particle size < 63 µm) was weighed and digested in a aqua-regia solution, which is a combination (ratio 4:1) of concentrated HNO_3 (AnalaR grade, BDH 69%) and $HClO_4$ (AnalaR grade, BDH 60%). The digestion was first conducted at low temperature (40 °C) for 1 h and then the temperature was elevated to 140 °C for a minimum 3 h until the samples have been fully digested. The resulting solutions of the digestion were than diluted to a fixed volume of 40 ml using double distilled water. The sample was then filtered filter paper (Whatman no. 1, pore size 11 µm) and the filtrates were stored until metal determination. Flame-Atomic Absorption Spectroscopy (FAAS; Perkin Elmer Model AAnalyst 800) was used to determine the concentration of Cd, Cu, Fe, Ni, Pb and Zn. The resulting data were expressed as mg/kg of samples' dry weight (dw).

To ensure the accuracy and credibility of the result, some pre-caution steps have been done. During metal analysis, a quality control sample was routinely analysed to ensure the accuracy of the analysis. As the result, the metal recovery of the quality control samples was acceptable at 90-110%. All glassware and equipment used during sample processing and metal analysis were soaked in 10% HNO_3 to wash away the possible metal contaminant. Certified Reference Materials (CRM) for Soil (International Atomic Energy Agency, Soil-5, Vienna, Austria) was used to assure the quality of direct aqua-regia method. The CRM was treated exactly the same as the other samples. The result of metal analysis was compared to the certified reference value of the respective metals. The recovery of the CRMs was found to be satisfactory.

Data treatment

Geoaccumulation index (I_{geo})

The values of I_{geo} were calculated according to Muller's (1969) formula (*Eq. 1*):

$$I_{geo} = log_2 \frac{C_n}{1.5 \times B_n} \quad \text{(Eq.1)}$$

where C_n is the sediment heavy metal concentrations and B_n is the "preindustrial reference values" (PRV; unit = mg/kg dw; Hakanson, 1980) which act as geochemical background values for each metal. The PRV for Cd, Cu Pb and Zn was 1.0, 50, 70 and 175, respectively (Hakanson, 1980). Owing to the absence of the PRV of Fe and Ni was taken from "upper continental crust" from Wedepohl (1995) and Rudnick and Gao (2003), in which the values were 3.09% and 47.0 mg/kg dw, respectively (*Table 2*).

To minimize the possible variation in background metal concentrations which might be contributed by lithogenic variations, a factor of 1.5 was introduced into the formula (Al-Haidarey et al., 2010; Hasan et al., 2013). It permits the content fluctuation of the metals in sediment and also some negligible anthropogenic influences (Loska et al., 1997; Hurley et al., 2017). Muller (1969) has classified the resulting Igeo values into six classes according to the extent of the metal geoaccumulation.

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Enrichment factor (EF)

The EF in this study was determined using a formula defined by Buat-Menard and Chesselt (1979), with Fe as a normaliser (*Eq. 2*):

$$EF = \frac{\left(\frac{C_n}{C_{Fe}}\right)_{sample}}{\left(\frac{C_n}{C_{Fe}}\right)_{crust}} \quad \text{(Eq.2)}$$

where (C_n/C_{Fe}) sample is the metal to Fe ratio in the sediments; (C_n/C_{Fe}) crust is the metal to Fe ratio in the earth crust which considered as pre-industrial unpolluted metal value of a sediment.

The normalisation using Fe are necessary to correct for differences in sediment grain size and mineralogy (Schiff and Weisberg, 1999). The use Fe as normaliser was made based on the fact that Fe is a major sorbent phase for trace metals and is a quasiconservative tracer of the natural metal-bearing phases in fluvial and coastal sediments (Schiff and Weisberg, 1999; Hurley et al., 2017). According to Hasan et al. (2013), natural resources (98%) vastly dominated the input of Fe. In this expression, the normaliser (reference element) is assumed to have little variability of occurrence, and is present in trace concentration in the examined environment (Loska et al., 1997). The degrees of EF are categorised by Taylor (1964) and Birth (2003).

Ecological risk assessments

The contamination factor (C_f) was calculated to describe the contamination status of metals in the industrial drainages studied (Hakanson, 1980). C_f was calculated as *Equation 3:*

$$C_f = \frac{C_{sed}}{C_{ref}} \quad \text{(Eq.3)}$$

where C_f is the contamination factor; C_{sed} is the mean metal concentration in the sediment; C_{ref} is the PRV of metals in the sediments. Hakanson (1980) has classified the C_f values into 4 categories.

According to Hakanson (1980), the potential risk for individual metal (E_r) can be calculated using *Equation 4:*

$$E_r = TR \times C_f \quad \text{(Eq.4)}$$

where TR is the toxic-response factor for a metal (*Table 2*) Due to the fact that the absence of TR value for Fe, the E_r for Fe was not calculated. C_f is the contamination factor for the same substance. The E_r for each metals were defined in accordance of Hakanson’s (1980) standard.

The total risk index (RI) or potential ecological risk index (PERI) was calculated as *Equation 5:*

$$RI = \sum_{i=1}^{m} E_r = \sum_{i=1}^{m} (TR \times C_f) \quad \text{(Eq.5)}$$

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where E_r is the risk index of individual metal, TR is the toxic-response factor and C_f is the contamination factor. The PERI can be described according to categories suggested by Hakanson (1980).

The classification of SQGs along with its effects and comparison results are presented in *Table 2*. It is important to determine whether the total concentrations of heavy metals in sediments found pose a threat to aquatic life. In the present study, the sediment risk of each metal investigated is assessed by three sets of SQGs namely (1) Effects Range Low (ERL) and Effects Range Medium (ERM) by Long et al. (1995), (2) lowest effect level (LEL) and severe effect level (SEL) by NYSDEC (1999), and (3) threshold effects level (TEL) and probable effects level (PEL) by MacDonald et al. (1996) and MacDonald (2003). These three sets of numerical SQGs were directly applied (without normalization) to assess possible risk arises from the heavy metal contamination in sediments of the study area.

Results and discussion

The concentrations (mg/kg dry weight; except for Fe in %) of surface sediments collected from Haqal coastal waters are shown in *Table 1*. The surface sediment of Tabuk coastal waters from Saudi Arabia were analysed for Cd, Cu, Fe, Ni, Pb, and Zn. The metal concentrations (mg/kg dry weight) ranges for total concentration are 0.012-0.186 for Cd, 0.582-1.13 for Cu, 0.51-2.18 for Ni, 0.68-2.64 for Pb, 1.97-4.52 for Zn while for Fe, it ranges from 0.155-0.254%. These values of heavy metals are remarkably lower compared to those reported in Jeddah coastal surface by Badr et al. (2009).

Table 1. *Concentrations (mg/kg dry weight; except for Fe in %) of heavy metals of surface sediments collected from Haqal coastal waters (Tabuk, Saudi Arabia)*

Location	Ni	Pb	Cu	Zn	Cd	Fe%
1	0.589	1.996	0.602	4.504	0.081	0.196
2	0.506	1.738	0.873	2.699	0.012	0.216
3	2.179	1.404	0.582	2.172	0.024	0.174
4	1.668	1.330	0.641	1.967	0.032	0.220
5	0.971	1.478	0.885	2.336	0.090	0.168
6	1.042	1.508	0.691	3.955	0.138	0.176
7	1.569	1.502	0.826	1.979	0.064	0.178
8	2.083	2.162	0.802	2.897	0.031	0.205
9	1.966	2.643	1.076	3.398	0.071	0.178
10	1.747	1.727	0.898	2.006	0.125	0.186
11	1.744	1.443	0.955	3.294	0.044	0.206
12	0.704	1.428	0.822	2.407	0.118	0.213
13	0.863	1.206	0.988	2.528	0.186	0.254
14	0.854	0.675	1.094	4.522	0.124	0.172
15	1.306	1.329	1.130	3.364	0.167	0.155

All the values of metals are below ERL, indicating that adverse effects on aquatic biota should rarely occur. There should be no toxicological effects of Cd, Cu, Ni, Pb

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and Zn in the present study area and that may not occasionally be associated with adverse biological effects.

When compared to the LEL–SEL SQGs, among the metals, Cd, Cu, Ni, Pb and Zn concentrations are also below LEL in all sites. According to Thompson et al. (2005), the LEL represents the contaminant concentration below the harmful effects on benthic invertebrates. However, by comparison to the TEL–PEL SQGs, 100% of the concentrations of Cd, Cu, Pb and Zn in all the samples are below TEL. It is interpreted that TEL as the concentrations, below which adverse biological effects rarely occurs. Hence, it is considered to provide a high level of protection for aquatic organisms. This requires a lower level of protection for aquatic organisms.

In comparison to reference values (RVs) (*Table 2*), all the metals levels of all sites are below the levels of pre-industrial reference (PIR) level (Hakanson, 1980), and the upper continental crust (UCC) values proposed by Taylor and McLennan (1995), Wedepohl (1995) and Rudnick and Gao (2003).

Table 2. *Comparisons between total heavy metal concentrations (mean, µg/g dw; except for Fe in %) with those cited from sediment quality guidelines (SQGs) and reference values (RVs). The values of toxic-response factors, employed in this study are also presented*

Site no.	**Cd**	**Cu**	**Fe (%)**	**Ni**	**Pb**	**Zn**	**References**
Haqal coastal sediment	0.012-0.186	0.582-1.13	0.155-0.254	0.51-2.18	0.68-2.64	1.97-4.52	This study
Jeddah (Saudi Arabia)	3.08-3.51	17.47-23.77	20.32-26.71	67.78-85.50	80.30-98.77	52.74-76.36	Badr et al. (2009)
Mangrove area of Peninsular Malaysia	1.11-2.00	5.59-28.7	1.29-4.89	-	25.36-172.6	29.35-130.3	Cheng and Yap (2015)
SQGs	**Cd**	**Cu**	**Fe (%)**	**Ni**	**Pb**	**Zn**	
Effects range low (ERL)	1.20	34.0	-	-	46.7	150	Long et al. (1995)
Effects range median (ERM)	9.60	270	-	-	218	410	Long et al. (1995)
LEL	0.6	16	2	16	31	120	NYSDEC
SEL	9	110	4	50	110	270	NYSDEC
Treshold effect level (TEL)	0.68	18.7	-	-	30.2	124	MacDonald et al. (1996)
Probable effect level (PEL)	4.21	108.2	-	-	112.2	271	MacDonald et al. (1996)
RVs	**Cd**	**Cu**	**Fe (%)**	**Ni**	**Pb**	**Zn**	
Pre-industrial reference level	1.00	50.0	-	68.0*	70.0	175	Hakanson (1980)
Upper continental crust	0.098	25.0	-	44.0	17.0	71.0	Taylor and McLennan (1995)
Upper continental crust	0.102	14.3	3.09	18.6	17.0	52.0	Wedepohl (1995)
Upper continental crust	0.09	28.0	-	47.0	17.0	67.0	Rudnick and Gao (2003)
Toxic-response factor (*Tr*)	30.0	5.00	-	2.00	5.00	1.00	Hakanson (1980)

All concentrations are presented in µg/g dw except for Fe in % and *Tr* values are unitless

The values of enrichment factor (EF), geoaccumulation index (Igeo), contamination factor (CF), ecological risk (ER) and potential ecological risk index (PERI) of heavy metals based on the surface sediments from all sampling sites are presented in *Table 3*.

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All the values of EF for Cd, Cu, Ni, Pb, and Zn are below 2.0, indicating ‘depletion of mineral enrichment’ (Sutherland, 2000; Hsu et al., 2016), except for 5 sites of Cd levels where the EF values are between 2 and 3, indicating ‘moderate enrichment’. Fro Igeo, all values are below 0.0 (in negative values), indicating ‘practically unpolluted’ (Muller, 1969). For CF for individual metal, all values are below 1.00, indicating ‘low contamination factor’ (Hakanson, 1980). For ER, all values are below 40.0, indicating ‘low potential ecological risk’ (Hakanson, 1980). Lastly, for PERI, all values are below 150, indicating ‘low ecological risk’ (Hakanson, 1980).

Table 3. *Summary results (mean) enrichment factor (EF), geoaccumulation index (Igeo), contamination factor (CF), ecological risk (ER) and potential ecological risk index (PERI) of heavy metals based on the surface sediments from the present study*

	Ni				Pb				Cu				Zn				Cd				Total ER/ PERI
Sites	**EF**	**Igeo**	**CF**	**ER**	**EF**	**Igeo**	**CF**	**ER**	**EF**	**Igeo**	**CF**	**ER**	**EF**	**Igeo**	**CF**	**ER**	**EF**	**Igeo**	**CF**	**ER**	
1	0.14	-7.44	0.01	0.02	0.45	-5.72	0.03	0.14	0.19	-6.96	0.01	0.06	0.41	-5.86	0.03	0.03	1.28	-4.21	0.08	2.44	2.68
2	0.11	-7.66	0.01	0.01	0.36	-5.92	0.02	0.12	0.25	-6.42	0.02	0.09	0.22	-6.60	0.02	0.02	0.17	-6.98	0.01	0.36	0.60
3	0.57	-5.55	0.03	0.06	0.36	-6.22	0.02	0.10	0.21	-7.01	0.01	0.06	0.22	-6.92	0.01	0.01	0.42	-5.99	0.02	0.71	0.94
4	0.34	-5.93	0.02	0.05	0.27	-6.30	0.02	0.09	0.18	-6.87	0.01	0.06	0.16	-7.06	0.01	0.01	0.45	-5.56	0.03	0.96	1.17
5	0.26	-6.71	0.01	0.03	0.39	-6.15	0.02	0.11	0.32	-6.41	0.02	0.09	0.24	-6.81	0.01	0.01	1.66	-4.05	0.09	2.71	2.95
6	0.27	-6.61	0.02	0.03	0.38	-6.12	0.02	0.11	0.24	-6.76	0.01	0.07	0.40	-6.05	0.02	0.02	2.42	-3.44	0.14	4.14	4.37
7	0.40	-6.02	0.02	0.05	0.37	-6.13	0.02	0.11	0.29	-6.50	0.02	0.08	0.20	-7.05	0.01	0.01	1.10	-4.56	0.06	1.91	2.15
8	0.46	-5.61	0.03	0.06	0.47	-5.60	0.03	0.15	0.24	-6.55	0.02	0.08	0.25	-6.50	0.02	0.02	0.47	-5.58	0.03	0.94	1.26
9	0.50	-5.70	0.03	0.06	0.65	-5.31	0.04	0.19	0.37	-6.12	0.02	0.11	0.34	-6.27	0.02	0.02	1.23	-4.40	0.07	2.14	2.51
10	0.43	-5.87	0.03	0.05	0.41	-5.93	0.02	0.12	0.30	-6.38	0.02	0.09	0.19	-7.03	0.01	0.01	2.09	-3.58	0.13	3.76	4.04
11	0.39	-5.87	0.03	0.05	0.31	-6.19	0.02	0.10	0.29	-6.29	0.02	0.10	0.28	-6.32	0.02	0.02	0.66	-5.10	0.04	1.31	1.58
12	0.15	-7.18	0.01	0.02	0.30	-6.20	0.02	0.10	0.24	-6.51	0.02	0.08	0.20	-6.77	0.01	0.01	1.71	-3.67	0.12	3.54	3.76
13	0.15	-6.89	0.01	0.03	0.21	-6.44	0.02	0.09	0.24	-6.25	0.02	0.10	0.18	-6.70	0.01	0.01	2.26	-3.02	0.19	5.57	5.79
14	0.23	-6.90	0.01	0.03	0.17	-7.28	0.01	0.05	0.39	-6.10	0.02	0.11	0.46	-5.86	0.03	0.03	2.22	-3.60	0.12	3.71	3.92
15	0.38	-6.29	0.02	0.04	0.38	-6.30	0.02	0.09	0.45	-6.05	0.02	0.11	0.38	-6.29	0.02	0.02	3.34	-3.17	0.17	5.02	5.28

Conclusion

Based on the monitoring survey of the metal concentrations in the surface sediments of Haqal coastal waters of Saudi Arabia, the values of PERI were categorised as ‘low ecological risk’, thus all sampling sites were unpolluted by heavy metals. Nevertheless this monitoring study showed a positive outcome of non-heavy metal pollution, future mitigation of the heavy metal pollution at the study area should be given priority by the enforcement. Further studies concerning the heavy metal assessment in this region are needed to better understand the accumulation of heavy metals and their transfer in the food web of the living organisms.

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