ORIGINAL ARTICLE

Speciation and mobility of heavy metals in mud in coastal reclamation areas in Shenzhen, China

Jianmin Huang · Runqiu Huang · Jiu J. Jiao · Kouping Chen

Abstract Coastal reclamation has been carried out along the coastal areas near Shenzhen, China in a large scale since 1980s by dumping fill materials over the marine mud at the sea bottom. Usually the area to be reclaimed is drained first and some of the mud is air-dried for a few weeks before it is buried by fill. After reclamation, the terrestrial groundwater, which is relatively acidic and with high dissolved oxygen, gradually displaces the seawater, which is alkaline with high salinity. The changes in the burial conditions of mud and the properties of the pore water in the mud may induce the release of some heavy metals into the mud. Field survey confirms that the pH and salinity of the groundwater in the reclamation site are much lower than the seawater. Chemical analyses of mud and groundwater samples collected from the reclamation sites reclaimed in different years indicate that most of the heavy metals in the mud decrease gradually with time, but the heavy metals in the groundwater are increased. The release of heavy metals into pore water due to reactivation of heavy metals in the mud is of environmental concern. To understand why some of the heavy metals can be released from the mud more easily than others, a sequential extraction method was used to study the operationally determined chemical forms of five heavy metals (Cu, Ni, Pb, Zn, and Cd) in the mud samples. Heavy metals can be presented in five chemical forms: exchangeable, carbonate, Fe–Mn oxide, organic, and residual. Ni and Pb were mainly associated with the Fe–Mn oxide fraction and carbonate fraction; Zn was mainly associated with organic fraction and Fe–Mn oxide fraction, while Cu and Cd were associated with organic fraction and carbonate fraction, respectively. If the residual fraction can be considered as an inert phase of the metal that cannot be mobilized, it is the other four forms of heavy metal that cause the noticeable changes in the concentration of heavy metals in the mud. On the basis of the speciation of heavy metals, the mobility of metals have the following order: Pb (36.63%) > Cu (31.11%) > Zn (20.49%) > Ni (18.37%) > Cd (13.46%). The measured metal mobility fits reasonably well with the degree of concentration reduction of the metals with time of burial observed in the reclamation site.

J. Huang (✉) · R. Huang
Department of Environment and Civil Engineering,
Chengdu University of Technology, Chengdu, China
e-mail: hjianmin9@yahoo.com.cn

J. Huang · J. J. Jiao · K. Chen
Department of Earth Sciences,
The University of Hong Kong,
Hong Kong, China

J. Huang
College of Chemistry, Sichuan Normal University,
Chengdu, China

Introduction

Over the last 20 years, land reclamation has gained much popularity along coastal areas of China due to the rapid development of industry and urbanization. Recently, a reclamation project has been carried out along part of the coast of Shekou, Shenzhen, to provide an area for the construction of the Hong Kong–Shenzhen Western Corridor (Fig. 1). The fill materials

Fig. 1 Schematic map of the study area and location of samples
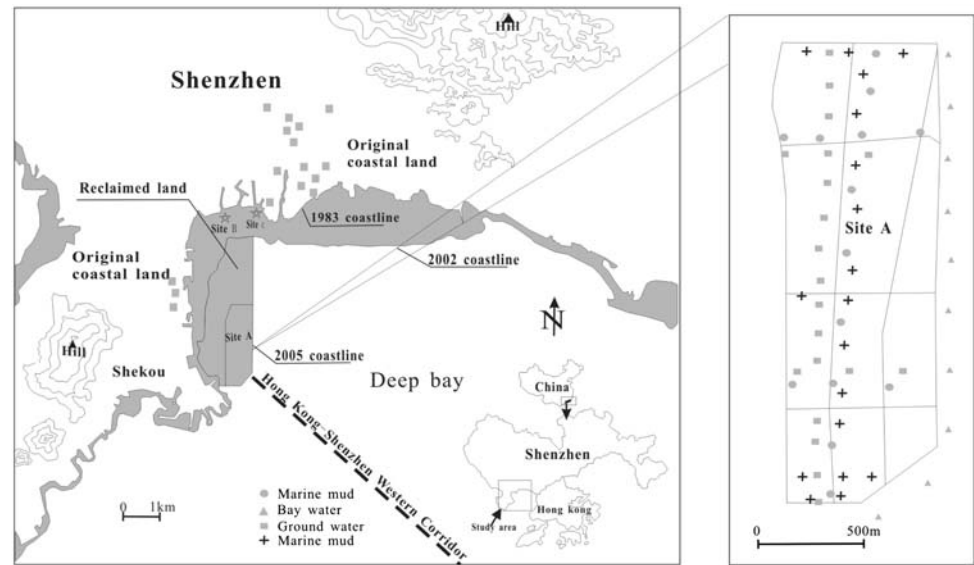
used at this site were largely the reddish, completely decomposed granite, which was excavated and transported from hills nearby. There was a thick layer of gray to dark marine mud at the sea bed.

Before land reclamation, temporal dikes around the coastal sea area to be reclaimed were constructed. The seawater in the closed area was pumped out to lower the water level. The mud at the bottom was exposed to the air for a few weeks and partially dried. This air-dried process is a geotechnical measure to speed up the consolidation of the mud. Eventually the fill materials were dumped into the site and the final ground surface was elevated to about 5–7 m above the sea level. The mud was under anoxic conditions because it was under seawater before reclamation, oxidized during the air-dried process at the beginning of reclamation, and was under anoxic condition again after it was buried by fill materials.

After reclamation, the original alkaline seawater in the reclamation site will be gradually displaced by acidic terrestrial groundwater, as shown in Fig. 2 (Jiao et al. 2005). The changes in the anoxic and oxidized conditions of the mud and the properties of the pore water in the mud may result in release of chemicals from the mud into the water.

Chen and Jiao (2006) studied the heavy metals of mud and groundwater samples from sites reclaimed in different years. They found that, mud which was buried earlier, had lower metal concentrations. This means that the concentrations of most of the metals in the mud decrease progressively with time. Table 1 shows the concentrations of heavy metals of mud samples collected from sites reclaimed in 2003, 1998, and 1996. The concentration of most of the metals such as Ni, Cu,

and Zn, were reduced significantly with years of burial. The changes in Cd and Pb were small and the trend was less obvious, but there was still a reduction in concentration by comparing the data of 2003 with that of 1996. To investigate the degree of concentration reduction of the metals with time of burial, a parameter called percentage reduction was introduced, which was defined approximately as the ratio of the difference between the metal concentrations of the mud from the sites reclaimed in 2003 and 1996, respectively, over the metal concentration of the mud from the site reclaimed in 2003. For example, the percentage reduction of Ni was calculated as $(33.12-19.96)/33.12 = 39.7\%$. The percentage reductions of all the heavy metals are shown in the last row of Table 1.

Chen and Jiao (2006) also found that the concentration of metals in groundwater samples collected in the reclaimed area was much higher than that in groundwater samples collected from the original land outside of reclamation area (see Table 2). These observations confirm that heavy metals in the mud were mobilized and released from the soil to groundwater after reclamation.

The reactivation of heavy metals in mud after reclamation is of environmental concern because the release of the heavy metals from the mud to pore water may have adverse effect on the coastal environment (Jiao 2002). The aim of this paper is to investigate why some heavy metals can be released from the mud more easily than others by carrying out laboratory experiments on speciation of heavy metals.

The mobility of heavy metals strongly depends not only on the characters of the pore water, but also on their specific chemical forms or ways of association.
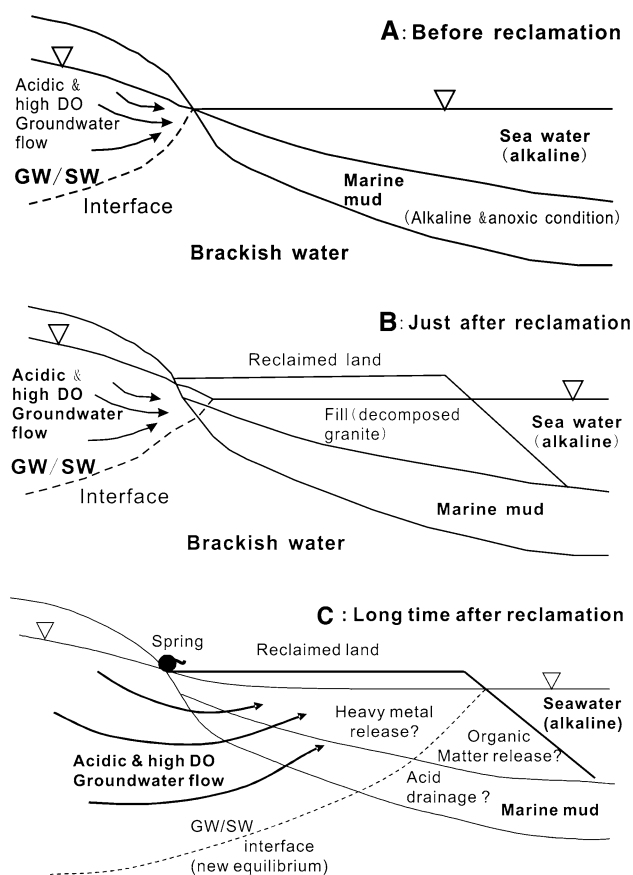
Fig. 2 Conceptual models of land reclamation and possible chemical processes (Jiao et al. 2005)

The measurement of the total concentration of a particular metal provides little indication of potential mobility in the environment. It is necessary to identify and quantify the forms in which a metal is present to gain a better understanding of the potential and actual mobility of metals under changing environmental conditions (Tessier and Campbell 1987). A lot of scholars used the speciation of heavy metals to study the potential mobility of metal (Tessier et al. 1979; Salomons and Förstner 1984; Banat et al. 2005; Hu et al. 2006) because speciation can provide some essential information on mobility. The sequential extraction scheme introduced by Tessier et al. (1979) is one of the most thoroughly studied and widely used procedures to evaluate the possible chemical associations of metals in sediments and soils and was used in this study to understand the mobility of the heavy metals in the mud in the reclamation area.

Mud and pore water in the reclamation site

Characterization of mud

The scanning electron microscope (SEM) images of the mud, collected from the reclamation site, were studied by Shenzhen Research and Design Institute, China Academy of Railway Sciences (2004). It was found that kaolinite was the main mineral in the mud.

Table 1 Metal concentrations in mud from three reclamation sites

		Ni	Cu	Zn	Cd	Pb
Site A (*n* = 22) reclaimed in 2003	Mean	33.12	34.63	119.25	0.40	49.45
	Range	21.63–52.96	21.37–74.21	77.36–192.39	0.28–0.68	34.46–67.77
site B (*n* = 6) reclaimed in 1998	Mean	22.00	25.41	102.31	0.44	56.70
	Range	11.87–29.83	12.43–41.59	73.08–128.36	0.39–0.53	52.05–62.20
site C (*n* = 6) reclaimed in 1996	Mean	19.96	16.06	76.79	0.38	40.20
	Range	15.33–25.57	14.57–17.51	62.63–93.01	0.30–0.45	33.18–48.83
Percentage reduction		39.7%	53.6%	35.6%	5.0%	18.7%

All results are in mg kg^{-1} dry weight

n number of samples

Table 2 Mean values of physicochemical parameters of water samples

	Sampling period	pH	Temperature °C	EC (ms/cm)	Salinity (‰)
Seawater samples (*n* = 10)	July 2004	7.71	30.12	41.31	26.45
	November 2004	7.85	23.22	42.60	27.38
Groundwater in reclamation site (*n* = 22)	July 2004	7.14	26.65	5.68	2.82
	November 2004	7.05	24.69	6.81	3.84
Groundwater outside reclamation site (*n* = 27)	July 2004	6.63	25.87	0.604	–
	November 2004	6.45	24.76	0.614	–

n number of samples

Other minerals, such as chlorate, smectite, and pyrite were also found. Crystals of salt and pyrite exist in the pores of the mud. Some bio-remains were also seen in the SEM images.

The air-dried period of the mud at the beginning of the reclamation has important implication on metal mobility. During this period, air, wind, and rain may accelerate the process of decomposition of the organic compound and sulfide and cause the oxidation of FeS_2 and FeS to H_2SO_4 in the mud, leading to pH reduction in groundwater (Meyer et al. 1994). Consequently, this air-dried process may increase the mobility of heavy metals released from the mud into the groundwater (Lau and Chu 1999).

After being covered by filled materials, the mud was consolidated gradually, due to the weight of the overlying fill and surcharge and again isolated from the atmosphere. The condition turns to anoxic again. The fresh groundwater migrated into the mud and salinity and pH of pore water declined gradually. During this period, pH, salinity, and anoxic condition in the reclamation areas were the key factors influencing the metal mobility.

Change of the physicochemical parameters of pore water in the mud

Chen and Jiao (2006) measured in situ pH, temperature, EC and salinity of the seawater and groundwater in the reclamation site soon after reclamation, and background groundwater in the original coastal land (Table 2). Their sampling locations are marked with gray symbols in Fig 1. The mean pH of the seawater in the winter and summer of 2004 was 7.85 and 7.71, respectively; the mean EC was 41.31 and 42.60, respectively. In the reclamation site, the mean pH of groundwater in the winter and summer was 7.05 and 7.14, respectively, and EC in the winter and summer was 5.68 and 6.81, respectively. The lower pH and EC of groundwater in the reclaimed land were due to the fact that seawater was gradually replaced by terrestrial groundwater and that, FeS_2 and FeS in mud were oxidized to H_2SO_4 during the air-dried period (Meyer et al. 1994).

Mud sampling for laboratory experiments

Mud samples were collected from the cores of 18 boreholes drilled for site investigation purposes in site A in June 2004 (Fig. 1). These 18 samples were sealed in plastic bags after extruding the air, and then stored in the refrigerator at –4°C until analysis. The samples were oven-dried at 105°C, disaggregated to pass through 2 mm sieve and ground in an agate mill to a fine powder.

Description of experiments on sequential extraction of heavy metal

The experiments were carried out at National Specialty Laboratory of Geohazard Prevention and Geoenvironment Protection, Chengdu University of Technology. All pipettes, tubes, and containers were washed with detergent in an ultrasonic washer, then soaked for 24 h in the 5% nitric acid (HNO_3) solution and rinsed repeatedly with deionized water. All reagents used were of "analytical grade" or "superpure" quality. International reference materials were used and duplicates of samples and reagent blanks were also used throughout the whole experiment procedure.

The extraction process used in the present study is based on operationally defined fractions: exchangeable, carbonate, Fe–Mn oxides, organic, and residual. Assuming that mobility is related to solubility, then metal solubility decreases in the following order: exchangeable > carbonate > Fe–Mn oxide > organic > residual (Tessier et al. 1979). The residual fraction could be considered as an inert phase corresponding to the part of metal that cannot be mobilized and as the geochemical background values for the elements in the mud (Tessier et al. 1979).

Sequential extraction procedure proposed by Tessier et al. (1979) consists of five steps (Li et al. 1995). The separation of each steps is operated by centrifuging at 3,000 rpm at 20 min, the supernatant solution is separated by pipette. The residue is washed in 10 ml deionized water and again centrifuged. The wash water is discarded. These steps are:

Step 1: To extract exchangeable metal, which is mainly affected by adsorption–desorption processes. Samples were extracted by 16 ml of 1 M magnesium chloride ($MgCl_2$) at pH 7.0 for 40 min at room temperature. Continuous agitation of the samples was maintained during the extraction.

Step 2: To extract the metal, bound to carbonate phase, which is affected by ion exchange and changes of pH. Residue from Step 1 was leached for 8 h with 16 ml of 1 M sodium acetate (NaOAc), which was adjusted to pH 5.0 with acetic acid (HOAc) at room temperature. During the extraction, continuous agitation was maintained.

Step 3: To extract the metal bound to Fe–Mn oxide phase, which is unstable under anoxic condition. This fraction was removed by adding 20 ml of 0.04 M hydroxylammonium hydrochloride ($NH_2 \cdot OH \cdot HCl$) in

25% (v/v) HOAc to the residual of second step. The extraction was performed at 96°C, with occasional agitation and extraction time was 6 h. After extraction, the extract solutions were diluted to 20 ml with de-ionized water and subjected to continuous agitation for 10 min.

Step 4: To extract the metal bound to organic matter and sulphide. Three milliliter of 0.02 M HNO_3 and 5 ml of 30% hydrogen peroxide (H_2O_2; adjusted to pH 2.0 with HNO_3) were added to the residue from Step 3. The sample was heated progressively to 85°C, and maintained at this temperature for 2 h with occasional agitation. A second 3-ml aliquot of 30% H_2O_2 (adjusted to pH 2.0 with HNO_3) was then added, and the mixture was heated again at 85°C for 3 h with intermittent agitation. After cooling, 5 ml of 3.2 M ammonium acetate (NH_4OAc) in 20% (v/v) HNO_3 was added, followed by dilution to a final volume of 20 ml with de-ionized water. The tubes were then continuously agitated for 30 min.

Step 5: To analyze the residual phase. Residue from Step 4 was oven-dried at 105°C. About 0.2 g dry residue of each sample was wetted by de-ionized water. Ten milliliter HCl was poured into each polytetrafluoroethylene crucible. These samples were heated to near-dryness on an electrothermal plate. The samples were then digested with 5 ml concentrated HNO_3 (70% w/w), 10 ml perchloric acid ($HClO_4$, 60% w/w), and 10 ml hydrofluoric acid (HF, 40% w/w). This operation was repeated twice. The remaining material was then taken up in 5 ml of 5 M HCl at 70°C for 1 h, and diluted to 50 ml with de-ionized water.

The sequential extractions were carried out in 100 ml polysulphone centrifuge tubes with screw caps, except for the last step, which was carried out in 50 ml polytetrafluoroethylene crucible.

The total metal content was determined in each of the sample sets by flame AAS under the guidance of methods set by the Environmental Protection Agency of the People's Republic of China (GB/T17139-1997, GB/T17138-1997, and GB /T17141-1997). The metal content in each fraction was also analyzed by flame AAS.

Results and discussion

Speciation of the heavy metals in mud

Table 3 and Fig. 3 summarized the average results of the sequential extraction analysis from 18 samples. The average total concentration of Cu in the mud was 32.19 mg kg^{-1}. Cu was largely presented in the residual

Table 3 Sequential extraction results of heavy metals

Cu	Average (mg kg^{-1})	Range (mg kg^{-1})	Percentage of sum
Step 1	ND	ND	ND
Step 2	2.32	1.87–2.889	7.22%
Step 3	ND	ND	ND
Step 4	7.69	2.55–10.77	23.89%
Step 5	22.18	13.52–28.83	68.89%
Sum	32.19	16.94–42.10	100.00%
Pb			
Step 1	2.08	0.39–5.67	3.95%
Step 2	5.26	4.63–9.44	10.00%
Step 3	9.99	6.23–11.77	19.01%
Step 4	1.93	0.65–2.37	3.67%
Step 5	33.32	15.69–68.65	63.37%
Sum	52.57	20.8–90.5	100.00%
Zn			
Step 1	0.79	6.23-nd	0.80%
Step 2	1.26	0.45–3.74	1.27%
Step 3	6.49	4.91–10.91	6.54%
Step 4	11.78	9.19–14.09	11.88%
Step 5	78.85	58.4–98.1	79.51%
Sum	99.17	56–181.4	100.00%
Cd			
Step 1	0.01	ND–0.03	1.69%
Step 2	0.06	0.025–0.274	10.18%
Step 3	0.01	ND-0.033	1.69%
Step 4	ND	ND	ND
Step 5	0.51	0.21–1.130	86.44%
Sum	0.59	0.26–1.4	100.00%
Ni			
Step 1	0.25	ND-1.23	0.73%
Step 2	2.05	0.68–2.79	6.02%
Step 3	2.26	0.142–2.69	6.64%
Step 4	1.70	0.95–2.75	4.99%
Step 5	27.78	18.33–50.36	81.6%
Sum	34.03	20.61–60.78	100.00%

ND not detected

(68.89%) and organic (23.89%) fractions. The Fe–Mn oxide and exchangeable fractions were below detection limit and so they are minor contributors to Cu. As a result, on the average, the percentage of Cu associated with different fractions in the mud cores was in the following order: residual (68.89%) > organic (23.89%) > carbonate (7.22%) > Fe–Mn oxide and exchangeable. Copper can easily complex with organic matters because of the high formation constants of organic-Cu compounds (Stumm and Morgan 1981). In aquatic systems, the distribution of Cu is mainly affected by natural organic matter such as humic materials and amino acids.

Results of the sequential extraction showed that the residual fraction dominated Zn distribution in mud, accounting for over 79.5% of the total Zn concentration. Among the non-residual fractions, the organic
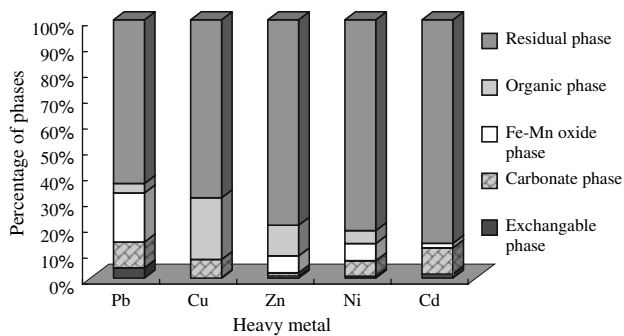
Fig. 3 Sequential extraction results of percentages of heavy metals

fraction and Fe–Mn fraction, which accounted for 11.88 and 6.54% of total Zn, respectively, were more the important fractions than others. The percentage of Zn associated with different fractions in the mud cores was in the following order: residual (79.51%) > organic (11.88%) > Fe–Mn oxide (6.54%) > carbonate (1.27%) > exchangeable (0.8%). The organic fraction and Fe–Mn fraction were reasonably high.

The average total concentration of nickel was about 34.03 mg/kg. The percentage of Ni associated with different fractions in the mud cores was in the following order: residual (81.62%) > Fe–Mn oxide (6.64%) > carbonate (6.03%) > organic (4.99%) > exchangeable (0.72%). Nickel was dominated by residual fraction, and other fractions are relatively small.

The percentage of Pb associated with different fractions in the mud cores was in the following order: Residual (63.37%) > Fe–Mn oxide (19.01%) > carbonate (10.08%) > exchangeable (3.96%) > organic (3.67%). The Fe–Mn fraction was reasonably high.

The results of the extraction experiment show that Cd bound to carbonate (10.2%) was the main fracture besides the residual fraction. The exchangeable fraction was 1.53%. Fe–Mn oxide fraction was 1.73%. The organic fraction was not detected.

Evaluation of mobility of the heavy metals

Mobility of heavy metals based on individual fractions

The sequential extraction of the metals revealed information on metal chemical speciation which is essential to the metal mobility (Tessier et al. 1979). Many previous researchers considered exchangeable fraction to be the most mobile and bioavailable phase present in the soils and sediments, followed by the carbonate phase (Tessier et al. 1979; Ahumada et al. 1999; Howari and Banat 2001). Iron and manganese

oxides exist as nodules, concretions, and cement between particles, or simply as coating on particles. These oxides are excellent scavengers for trace metals and can be mobilized under reducing and acidic conditions (Tessier et al. 1979; Banat and Howari 2005). The organic phase is a relatively stable phase in nature, but it can be mobilized under strong oxidizing conditions due to organic matter degradation, leading to a release of the soluble metal (Tessier et al. 1979). The residual solid should contain mainly primary and secondary minerals, which may hold trace metals within their crystal structure. Metals present in this fraction are a measure of the degree of environmental pollution: the higher the metals present in this fraction, the lower the degree of pollution (Howari and Banat 2001; Ahumada et al. 1999).

As shown in Fig. 4, in this sequential extraction, the exchangeable fraction in the analyzed mud samples is able to retain heavy metals in the following order: Pb (3.95%) > Cd (1.69%) > Zn (0.8%) > Ni (0.73%) > Cu. The carbonate fraction in the analyzed mud samples is able to retain heavy metals in the following order: Cd (10.18%) > Pb (10%) > Cu (7.22%) > Ni (6.02%) > Zn (1.27%). Fe–Mn oxides retain heavy metals in the mud samples of the study area in the following order: Pb (19.01%) > Ni (6.64%) > Zn (6.54%) > Cd (1.69%) > Cu. The organic fraction is able to retain heavy metals in the following order: Cu (23.89%) > Zn (11.88%) > Ni (4.99%) > Pb (3.67%) > Cd.

Mobility of heavy metals based on total mobile fractions

It is expected that there are two factors which may affect the mobility of heavy metals in the reclamation area. One is the chemical speciation of the metals which controls the amount of metals that can be mobilized. The other is the physiochemical features of groundwater in reclamation site.
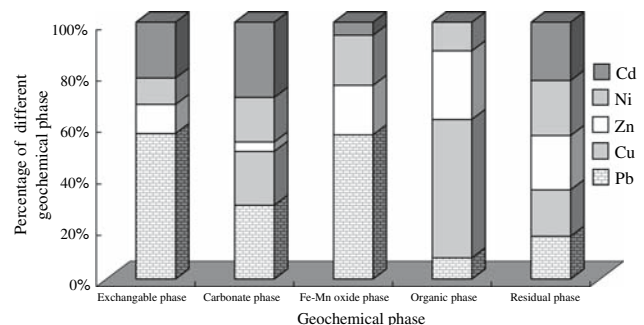

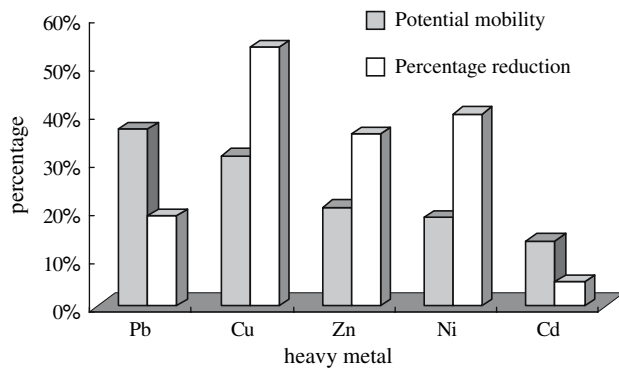**Fig. 4** Heavy metal percentages in different geochemical phases

Fig. 5 Comparison of observed percentage reduction and potential mobility of metals

The changes in pH and salinity of groundwater was observed in the reclamation site. The air-dried period of mud during the reclamation process may also have some effect on the mobility of heavy metals. This drying process had been reported in literature, may increase the mobility of contaminant released from the sediment into the water (Salomons et al. 1987; Liang and Wong 2003). Under an oxidation environment, the organic fraction of metals bound to the mud can be easily released. It is expected that the oxidation in the air-dried period may play a key role in the release of Cu into the pore water, because Cu is dominated by organic fraction. This may also apply to Zn, which has a large organic fraction. Similar behavior of Zn was also reported earlier (Liang and Wong 2003). After the mud was buried by the filled materials, the environment changed to anoxic condition again. Fractions of metals which can be easily influenced by anoxic condition should be active.

The sum of different phases of metals can be used to express the potential mobility of metals. The potential mobility of a metal can be assessed by adding up the results of the exchangeable phase, carbonate phase, Fe–Mn oxide phases, and organic phases of that metal. The potential mobility of heavy metals calculated in this way is illustrated using gray columns in Fig. 5, which shows that the mobility of the five metals have the following order: Pb (36.63%) > Cu (31.11%) > Zn (20.41%) > Ni (18.38%) > Cd (13.58%).

Discussion about the potential mobility
and the observed percentage reduction
of heavy metals

To compare the potential mobility of heavy metals estimated from laboratory experiments with the observed behavior of the metals in the mud samples collected from the different reclamation sites

reclaimed in different years, both potential mobility and percentage reduction are shown in Fig. 5. Except for Pb, the observed behavior matches reasonably well with the theoretical mobility: large percentage reduction of a metal has a large potential mobility. This indicates that the observed behavior of most of the heavy metals can be explained by the potential mobility from the speciation analysis.

Pb has relatively high mobility from the speciation analysis, but the percentage reduction is small. The reason for this is unclear. This suggests that the actual behavior of Pb may be controlled not only by its speciation, changes in pH, salinity and Eh, but also by other unknown factors.

Conclusion

Field sampling of mud and ground water samples in a coastal reclamation site in Shenzhen, China, were carried out to understand the physical and chemical changes of the mud and groundwater. The chemical analyses of the samples indicated that overall the concentrations of the heavy metals in the mud decreased with the years of burial of mud because of the changes in the physicochemical environment of the mud, but the heavy metals in groundwater increased with time. It was believed that the heavy metals were released into the pore water during and after land reclamation. The release of heavy metals of mud to pore water is of concern because eventually this may affect the coastal environment. The sequential extraction scheme was used to understand the mobility of the heavy metals Cu, Ni, Pb, Zn, and Cd in the mud in the reclamation area. Five chemical forms such as exchangeable, carbonate, Fe–Mn oxide, organic, and residual, were investigated. The total amount of a heavy metal in the first four forms was used as an indicator of the mobility of that metal. Based on the speciation of heavy metals, the mobility of the metals have the following order: Pb (36.63%) > Cu (31.11%) > Zn (20.41%) > Ni (18.38%) > Cd (13.58%). The measured mobility matched reasonably well with the observed degree of concentration reduction of the metals with time except for Pb. The theoretical mobility of Pb was large but the observed reduction in Pb concentration was low. This suggests that the actual behavior of Pb may be controlled not only by its speciation, changes in pH, salinity, and Eh, but also by other unknown factors.

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