Metal Speciation in Sediment from Muara Angke, Jakarta Bay Using of BCR Sequential Extraction Procedure

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Abstract

The assessment of the biological availability of metals is rarely used only by knowing the total concentration of the metal. Therefore, six sediment samples from Muara Angke, Teluk Jakarta were assessed the chemical speciation of heavy metals. This study aims to determine metal speciation using the BCR sequential extraction procedure and to determine metal speciation to evaluate bioavailability in the sediments of Muara Angke, Jakarta Bay. In sediment from Muara Angke, the ability to move sequence of heavy metals studied was Pb>Zn>Cu>Ni. The mostly accumulated in the non-residual fraction of the total concentrations are Cu, Ni, Pb and Zn which indicated that the mobility and anthropogenic inputs of these metals in Muara Angke were quite high. The Risk Assessment Code (RAC) reveal that Zn and Ni at almost station exist in exchangeable and a fraction of carbonate-bound and therefore high-risk category. Most of the Cu at most of the station is in the oxidizable fraction, except a small portion found at all station is in the exchangeable fraction and fraction of carbonate-bound thus posing a low risk for the waters environment. The patterns of Pb speciation show no to low risk to the waters environment. However, metal observations in the waters are necessary because they are persistent and can accumulate which threatening the water environment.

Keywords: *metal speciation, sediments, Muara Angke, Jakarta Bay*

Introduction

Metals are an appearing and persistent contamination problem faced by the human (Luoma and Rainbow, 2008). As a place to store the main and the metal source in the aquatic environment, sediment participates in the role of transporting and storing heavy metals that have potential hazards (Castillo *et al*., 2013). Notably, studies about total metal concentration in sediment cannot adequate to reflect about mobility, bioavailability, and toxicity. (Campbell *et al*., 1988). Their attributes do not only rely on their total metal content but also on the physicochemical form they happen (Davidson *et al*., 1994). Ure *et al*. (1993) has been described it as speciation. The metals such chromium (Cr), copper (Cu), lead (Pb), zinc (Zn), and mercury (Hg) can become toxic and bioaccumulate in biological tissues and can biomagnify through the food chain (Luoma and Rainbow 2008, García-Alonso *et al*., 2016). The bioavailability and toxicity of metals depend upon their partition and chemical speciation (Luoma and Rainbow 2008, García-Alonso *et al*., 2016).

Sequential extraction procedure is evolved to analyze a different fraction of metals/metals bound in sediment. Sequential extraction procedure used selective chemical reagents to obtain a continuing dissolution of variant mineralogical phases and the next release of metals. The European Union's BCR (The Community Bureau of Reference – BCR, presently as the Standards, Measurements and Testing programme – SM&T) developed a sequential extraction procedure. They are offered to get three fractions named an acid-soluble, a reducible and an oxidizable fraction (Ure *et al*., 1993; Quevauviller *et al*., 1997; Rauret *et al*., 1999; Cuong and Obard 2006; Santos *et al*., 2011; Teuchies *et al*., 2013; Islam *et al*., 2015). A scheme of a BCR sequential extraction procedures requires the several metals extraction in sequence to enlarge of metal reactivity. Metals speciation in sediments applied these procedures to produce three separate fractions (an acid soluble–extractable; a reducible; and an oxidable) (Qiao *et al*., 2013).

Jakarta Bay has been ecologically degenerated because of input biological and chemical pollutants from the sea and mainland areas (Williams *et al*., 2000; Arifin, 2004; Lestari *et al*., 2018). These pollutants were release from highland regions then were carried away through 13 rivers run into Jakarta Bay, including Angke River (Cordova and Riani, 2011). It disrupts water quality in Angke River and creates mass death of fish in

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mid-December 2015. Previous studies of total metals (Hutagalung, 1994; Williams *et al*., 2000; Rochyatun and Rozak, 2007) and metal speciation (Takarina *et al*., 2008 and Lestari *et al.,* 2018) has been carried out. The total metal concentration is infrequently beneficial in defining its biological availability because metals in an environment of sediment happen in both dissolved and particulate condition and a type of chemical forms (Campbell *et al*., 1988). The purposes of this research are to determine metal speciation using the BCR sequential extraction procedure and to assess the bioavailability of metal in the sediments of Muara Angke, Jakarta Bay.

Materials and Methods

Six samples of sediment were taken from the sediment surface layer from that sites in that period, using stainless steel ponar grab was then subsample using polyethylene spoon and immediately put into an acid pre-cleaned polyethylene sample container (Loring and Rantala, 1992) (Figure 1).

The speciation method of BCR three-step sequential extraction procedure provides information on four fractions: *i.e*., exchangeable and carbonates; reducible; oxidizable; and residual (Table 1.). This method was modified by Cuong and Obard (2006) is followed by a little modification in step 4. The metal content of the residue from step 3 and the total metal content determined using microwave-assisted acid digestion procedure. Dry sediment samples were weighed as much as 1g then digested with 10 ml of HNO3: HCl (1:3) acid mixture in a microwave MarsExpress. Before analyzed by flame atomic absorption (FAAS) using Varian SpectraA 20, the supernatant has to be clear by centrifugation procedure (3000 rpm for 10 min).

The US NOAA's sediment quality guidelines were used to compare with these total metals to estimate the probable environmental impact (Ramirez *et al*., 2005). Using the effects range low (ERL) and effects range median (ERM) for representing minimum level and maximum tolerable level of metals. In general, contrary results happen smaller than 10% of research in which the concentrations were below the respective ERL values, and were observed in greater than 75% of research in which the concentrations exceeded ERM values (Long *et al*., 1995).

The speciation study result showed that metals in the sediments are bound to different fractions with unique strength. An Indication of sediment reactivity to support the risk assessment of the presence of metals in Muara Angke given by these strength value. Evaluating sediment quality used The Risk Assessment Code criteria (RAC) (Jain, 2004; Turki, 2007; Horváth *et al*., 2013; Morelli and Gasparon, 2014). The Risk Assessment Code (RAC) criteria showed that sediment is removing an exchangeable and a carbonate fraction, smaller than 1% of the total metal, was reflected in secure for the environment. Otherwise, the sediment removing more significant than 50% of the total metal in the similar fraction has been thought about considerably hazardous and great probably enter the food chain.

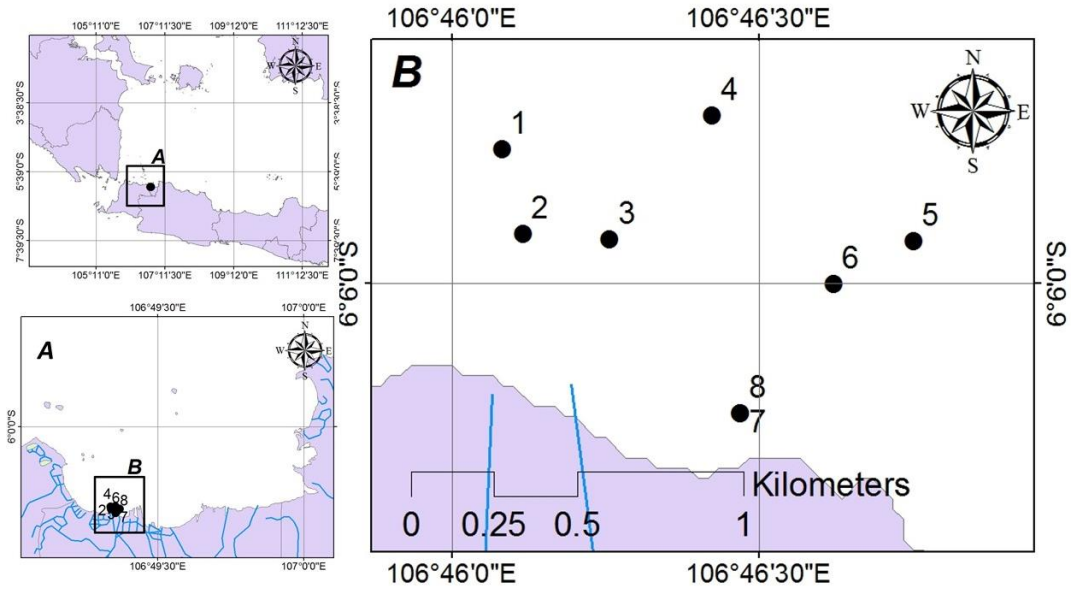

Figure 1. Sampling Location of the study area in Muara Angke, Jakarta Bay

Table 1. The main extraction steps for the BCR Sequential method (Ure *et al.,* 1993 modified by Cuong and Obbard 2006)

Step	Sediment phase	Reagent for extraction	Shaking time and temperature	Volume
F1	acid-soluble fraction – bound to carbonates	40 ml of 0.11 mol.L^{-1} CH_3COOH	16 h at room temperature at 22±5°C and speed 30±10 rpm. The extract was separated from the solid phase by centrifugation at 3000 rpm for 20 min.	40 ml
F2	reducible fraction – bound to Fe and Mn oxides)	40 ml of 0.5 mol.L^{-1} $HONH_2.HCl$ (pH 1.5)	16 h at room temperature 22±5°C and speed 30±10 rpm. The extract was separated from the solid phase by centrifugation at 3000 rpm for 20 min.	40 ml
F3	Oxidizable fraction- bound to organic matter and sulfides	10 ml of 8.8 mol.L^{-1} H_2O_2 (two times), cool and add 50 mL of 1 mol.L^{-1} NH_4OAc (pH 2)	1 h at room temperature 1 h at 85°C 1 h at 85°c 16 h at room temperature 22±5°C and speed 30±10 rpm. The extract was separated from the solid phase by centrifugation at 3000 rpm for 20 min.	50 ml
R	Residual fraction – strongly associated to the crystalline structures of minerals	10 mL of aqua regia (3 HCl : 1 HNO3)	Digesting in a mixture aqua regia	50 ml

Results and Discussions

The determination of heavy metals used testing the accuracy and precision for the extraction procedure. The sums of the metals concentrations get from the sequential extraction of 1.00 g of Certified References Material (CRM). Their total metal contents compared with PACS-2 (Cuong and Obbard, 2006). Table 2 summarized from the value that found to be acceptable (87–98 %) of the get recovery. A marine sediment standard reference material (PACS-2, National Research Council, Canada) was also to use for the accuracy of the microwave assisted acid digestion procedure for total metal determinations. The result showed satisfactory accuracy from this analysis, for the recoveries of all metals in PACS 2 between 91-96 %.

The concentrations of total metal in surface sediments of Muara Angke are shown in Figure 2. Concentrations of Cu, Ni, Pb and Zn varied 29.0-75.9 mg.kg^{-1} dry weight (dw); 16.3-21.6 mg.kg^{-1} dw; 11.7-36.3 mg.kg^{-1} dw; and 83.4-360 mg.kg^{-1} dw, respectively. Compared with the previous study in this area, such as site JK 06 (Williams *et al*., 2000) are lower than this study. The mean concentration of metals in Muara Angke reduce in the sequence Zn>Cu>Pb>Ni. Sediment type in these area study showed the predominance of mud in station 1,2,4 and 7 meanwhile in station 5, and 6 contains sands. The concentration of organic matter (loss in ignition) are varying from 7.5–15.7 %.

The Cu and Zn concentrations were above the ERL values although they were still below the ERM values. However, most of the Pb and Ni concentrations were below both the ERL and ERM values. The total Cu and Zn contents in sediments were above moderately polluted or polluted levels. Table 3 presented the metals concentration in the studied area as well as the ERL and ERM value. Table 4 showed the non-residual and residual portioning of Copper (Cu), Nickel (Ni), Lead (Pb) and Zinc (Zn) in the marine sediments. Associated their total content and speciation give the potential environmental risk of metals in sediments.

Figure 3 describes the partitioning of heavy metals varied considerably in every station. The Cu-organic complexes formation contributed to the existence of Copper inorganic fraction (39.6%). Copper (29.3%) was bound to Fe—Mn oxides. Copper in the exchangeable fraction was 5.08% and in residual fraction was 25.3%. Copper bound in the residual fraction was dominated in other regions such as such as Banten Bay and Cirebon Waters (Lestari *et al*., 2018), Benoa Bay (Budiyanto *et al*., 2018). Metals relatively stable and inert, and they are hardly to release into the mobile and bioavailable phases when they bounded with the residual phase. This fraction contains crystalline-bound trace metals and is dissolved with strong acids and specials digestion procedures (Hlavay *et al*., 2005; Turki, 2007).

Table 2. Comparative result of analysis of the BCR sequential extraction procedure and total digestion on the certified reference material PACS-2 (n=3).

	Copper(Cu)	Nickel (Ni)	Lead (Pb)	Zinc(Zn)
Sum of the three extraction step and residual step	278±11	33.0±5.6	157±1	315±1
Total Metal	279±6.6	36±1.8	179±1.5	348±17
Recovery %	98	87	87	89
Certified PACS 2	310±12	39.5±233	183±8	364±23
Recovery % Total metal	91	90	98	96

Table 3. US NOAA's ERL and ERM concentrations for the studied metals (values are in $mg.kg^{-1}$ dry weight)

Station		Element, in $mg.kg^{-1}$ dry			
		Copper(Cu)	Nickel (Ni)	Lead (Pb)	Zinc(Zn)
1		74,5	19,5	36,3	360
2		75,9	21,6	32,3	351
4		52,2	20,1	24,6	225
5		29,0	17,1	11,7	83,4
6		37,7	16,3	17,1	105
7		67,1	18,6	25,0	200
Elemental background concentration		55.0	75.0	12.5	70.0
ERL		34,0	20,9	46,7	150
ERM		270	51,6	218	410
	non polluted	<25	<20	<40	<90
SQG	moderately polluted	25-50	20-50	40-60	90-200
	heavily polluted	>50	>50	>60	>200

Table 4. The average non-residual and residual proportion of elements (%) in the sediments from Muara Angke

Element	Non-Residual [a]	Residual [b]
Cu	74.7	25.3
Ni	56.6	43.4
Pb	94.8	5.2
Zn	82.0	18.0

[a] = non residual fraction, F1(acid soluble fraction) + F2 (reducible fraction) + F3 (organic fraction)
[b] =. residual fraction

Nickel bounded in the residual fraction was the most substantial fraction (43.4%) of total Ni. The other fraction (17.6%) have content Nickel in a reducible fraction. Nickel was partitioned 13.9% in the oxidizable fraction (F3) and 25.2% in the exchangeable and bound to carbonates fraction. Lead (Pb) was partitioned among the reducible (F1=84.3%), oxidizable (F3=9.75%), and residual fractions (F4=5.22%). The Lead percentage in exchangeable and bound to carbonates fraction was lower the method detection limit. The most significant fraction for Zinc (42.3%) was exchangeable and bound to carbonates fraction. The other significant fraction (31.6%) have content Zn was a reducible fraction. Zinc is partitioned 8.0% in oxidizable fraction and 8.0% in residual fraction. Similar with area in Shantou Bay, China (Qiao *et al*., 2013), Zn happen in fractions of weakly- bounded with an average of 41.04% was the most high levels then followed by reducible fraction (21.63%), show that in the bay significant scavengers of Zn was carbonate and Fe/Mn hydrous oxides. Mainly, the percentage of Zn in weakly-bound fractions reduce downstream from 49.76-14.05%, occurred with an increase from 12.62-40.00% in the residual fraction. Metals-related with the exchangeable ions and carbonates were easily remobilized by environmental alteration conditions such as pH, salinity and they are the most labile bonds. Metals in reducible fraction can be liberated in the environment with a pH decrease and alteration from oxic to anoxic conditions in sediment. Similarly, metals bound to organic matter are released under oxidizing conditions. These three fractions are the labile or exchangeable and hazardous phases for the environment. They may alter depending upon the surrounding physical and geochemical conditions (Turki, 2007).

Figure 4 described the metal distribution in percent bar graphs in the four fractions obtained in our analysis. The partitioning showed that the percentages of metals associated with the non-residual fractions (consist of exchangeable also with carbonate+ reducible + oxidizable (Table 4).

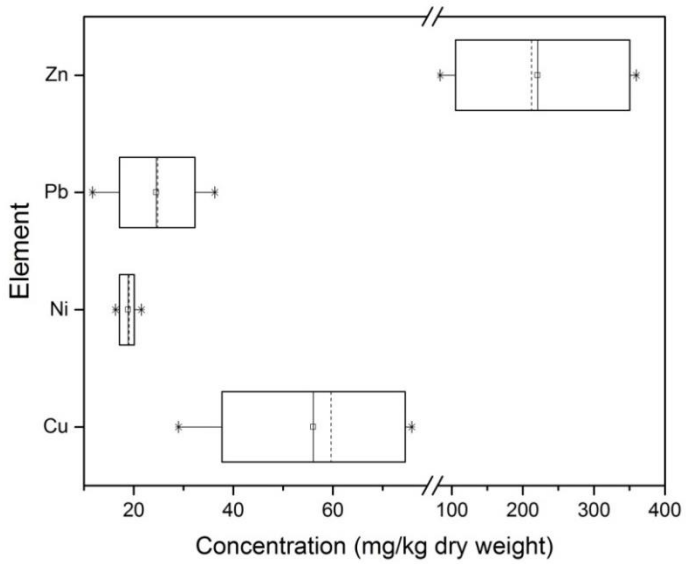

Figure 2. The box plot represented the total metal concentration in Muara angke: the mean (solid line); median (dash-line).

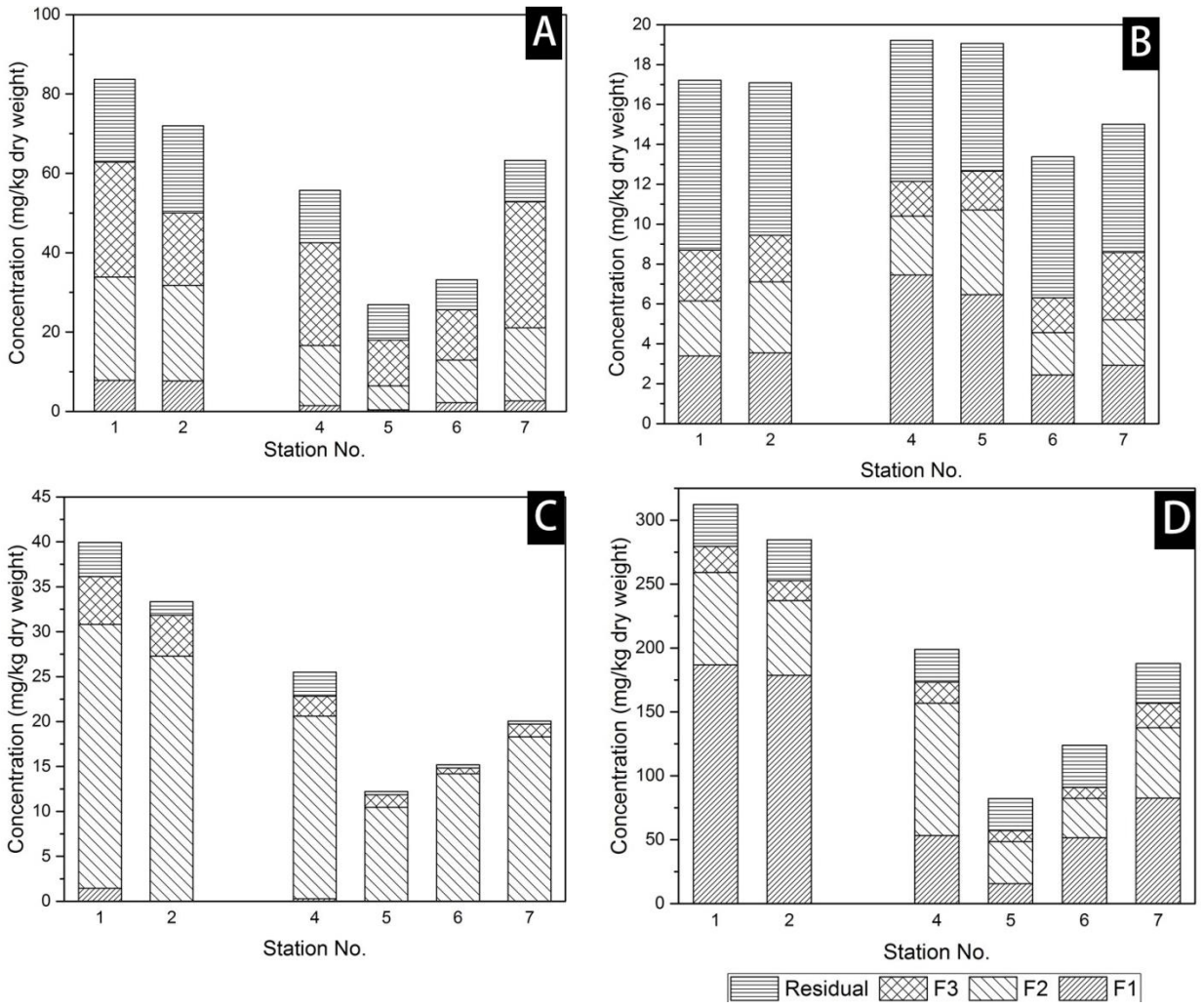

Figure 3. Chemical Partioning of heavy metal (mg.kg^{-1} dry weight) F1:acid soluble fraction, F2:reducible fraction, F3: organic fraction and residual in sediment sample at Muara Angke: Cu (A); Ni (B), Pb (C); Zn(D).

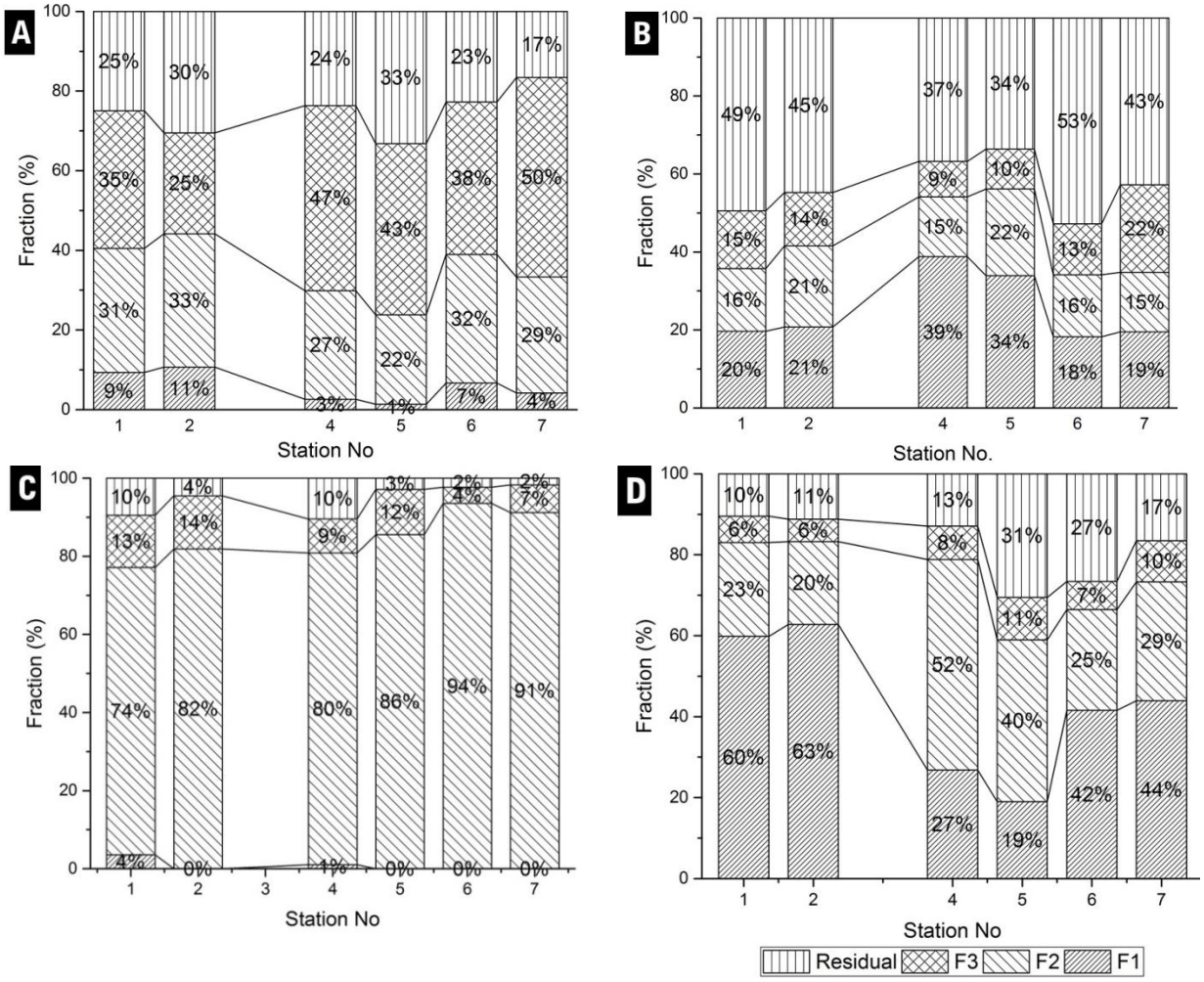

Figure 4. Removal percentage of (A) Cu, (B) Ni, (C) Pb, and (D) Zn in each step of the sequential extraction procedure (F1:acid soluble fraction, F2:reducible fraction, F3: organic fraction and residual) applied for marine sediment at Muara Angke

Notably were higher (94.8% for Pb; 82% for Zn; 74.7 for Cu and 56.6% for Zn) than those of the residual fraction (5.2% Pb; 18.8% for Zn; 25.3% Cu and 43.3% for Ni). These show that these metals were firstly obtained from an anthropogenic feed in even of the background of geochemical. The potentially of metals more available for exchange and release into the marine environment was indicated from our result. The non-residual fractions increased in the order of Pb<Zn<Cu<Ni in this studied of metal.

Using of Risk Assessment Code (RAC) criteria to assess sediment quality as given below Table 5. The code applied in our study revealed that about 41.6-62.8% of Zn at nearly all station was exchangeable and carbonate bound and enter the food chain. About 34-38.8% of Ni at station Angke 4 is exchangeable and carbonate bound and beneath category of high-risk. Most of the copper at most of the stations are in the oxidizable fraction, except a small portion (1.37-10.7) found at all stations is in the exchangeable and carbonate bound fraction, so that gives a low risk for the environment of aquatic. Patterns of Pb speciation (0-3.6% in exchangeable and carbonate bound) show no to low risk to the aquatic environment. However, metal observations in the waters are necessary because they are persistent and can accumulate which threatening the water environment.

Table 5. Risk Assessment Code (after Jain, 2004)

Risk Assessment Code (RAC)	Criteria (%)
No Risk	< 1
Low Risk	1–10
Medium Risk	11–30
High Risk	31–50
Very High Risk	> 50

Conclusions

Total Copper and Zinc contents in sediments were above moderately polluted or polluted levels based on SQG. Marine sediments in Muara Angke in

consequence of anthropogenic impact were indicated in this study. Evaluation of the potential mobility and the possible transfer of heavy metals from sediments to the surrounding environment from Muara Angke, Jakarta Bay was applied using the BCR sequential extraction procedure. Copper, Nickel, Lead, and Zink were generally accumulated in a non-residual fraction which indicated that the mobility and anthropogenic inputs of these metals in Muara Angke were quite high. The mobility order of heavy metals studied was Pb>Zn>Cu>Ni in sediment from Muara Angke. The Risk Assessment Code (RAC) reveal that Zink and Nickel at nearly all stations be in exchangeable and carbonate bound fraction and therefore high-risk category. Most of the copper at most of the stations are in the oxidizable fraction, except a small portion found at all stations is in the exchangeable and carbonate bound fraction, so that gives a low risk for the aquatic environment. Patterns of Pb speciation show no to low risk to the aquatic environment.

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