MALAYSIAN JOURNAL OF APPLIED SCIENCES 2020, VOL 5 (2): 57-68
E-ISSN:0127-9246 (ONLINE)
http://dx.doi.org/10.37231/myjas.2020.5.2.267
https://journal.unisza.edu.my/myjas

ORIGINAL ARTICLE

Fractionation of As, Co, Cu and Zn by Sequential Extraction in Surface Sediment of Kuala Terengganu River Estuary

Muhammad Nazirul Mubin Abd Halim Shah[1], Lee Siang Hing[1,3], Mohd Yusoff Nurulnadia[1,2,3] and Meng Chuan Ong[1,2,3]

[1]Faculty of Science and Marine Environment, Universiti Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia
[2]Institute of Oceanography and Environment, Universiti Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia
[3]Ocean Pollution and Ecotoxicology (OPEC) Research Group, Universiti Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia

*Corresponding author: LSHING@umt.edu.my

Received: 26/10/2020, Accepted: 29/10/2020, Published: 31/10/2020

Abstract

Development and urbanization processes around Terengganu River estuary are expected to release a significant amount of heavy metals into the existing surface sediment. However, information on how and why these metals are attached into specific fraction of sediments is still lacking. Therefore, this study aimed to explain the heavy metal concentration distribution in each available fraction in Terengganu River estuary. In this study, nine surface sediments originated from various human activities area in Terengganu River estuary were collected during four different sampling sessions in 2017. Heavy metal content from the collected sediments were extracted using 3-steps BCR sequential extraction method followed by detection using Inductively Coupled Plasma Mass-Spectrometer (ICP-MS) and we discovered that the total concentration of arsenic (As), cobalt (Co), copper (Cu), and zinc (Zn) ranged from 2.18 to 17.48 mg/kg dry wt., 2.53 to 20.53 mg/kg dry wt., 1.01 to 13.13 mg/kg dry wt., and 6.10 to 65.71 mg/kg dry wt., respectively. Dominance of metals in each fraction can be arranged as follows: As: residual > reducible > exchangeable > oxidizable; Co: residual > exchangeable > reducible > oxidizable; Cu: residual > oxidizable > reducible > exchangeable; Zn: residual > exchangeable > reducible > oxidizable. Availability of metals in the sediment at Terengganu River estuary is limited since that majority of metals resides in non-mobilisable fraction of the sediment. In essence, the sequential extraction provides information regarding the metals' fractionation, availability and mobility, which could be used in assessing the environmental contamination in the area.

Keywords: BCR Sequential extraction, Estuary, Heavy metal, South China Sea

Introduction

Intense development and industrialization around coastal area have the tendency to contribute a significant amount of pollutions into the aquatic environment by ways of anthropogenic input from various human activities. Urbanization and development near the estuary area might not only altered the initial contaminant content, but it is possible that the spatial distribution of the contaminant itself can be changed (Li et al., 2014). As the metals move further away from the point sources, their concentrations became lower.

For the year of 2020, Terengganu's government has targeted a growth rate of about 3.3% for the Kuala Terengganu district alone. Based on the report by FDTP Kuala Terengganu (2008), it has been expected that the district of Kuala Terengganu will accommodate about 536,500 people in 60,528.60 hectares of land; where 12.84% of it will be used for housing area, 0.54% will be used for industrial activities, 0.94% for infrastructure and utilities plus 42.93% for agriculture use. The intense changes in land usage could affect the current contaminant concentrations. It is possible that the pollutants would soon end up in the superficial sediments in the estuary, as the estuary is usually known as a place where important biomass exchange takes place; therefore, results in the accumulation of contaminant in this area (Delgado et al., 2010). Since that all contaminants such as pesticides and heavy metals will sink in the sediments (Ikem et al., 2003). Heavy metal pollutions become one of the major concerns for the estuary area as it is highly persistence in the environment. Since it have the ability to be retained in the aquatic environment, it can be found almost everywhere in the water, sediment and or even in organisms (Doong et al., 2008).

Total metal digestion can only provide an overall view of the metals, and it often serves as the indicator for studying environmental pollution. However, the bioavailability, mobility, toxicity and the reactivity of the metals, is much less discover elsewhere (Saleem et al., 2018). This information gap can be filled by extracting any possible metal fraction that exists in the sediments. Various heavy metal extraction methods had been derived from Tessier et al. (1979) and it is hard to compare various results from various extraction schemes. Therefore, a new and regulated 3-steps BCR method for speciation of metals in soil and sediments samples were introduced by the Standard Measurement and Testing program (formerly European Community of Bureau of Reference, BCR) to solve this problem (Ure et al., 1993). In this extraction protocol, there are three fractions are to be extracted namely, exchangeable, reducible and oxidizable fraction. The last fraction which is the residual fraction can be obtained through the strong acid digestion method after all three steps of the BCR method were done.

Through these procedures, additional information such as the main binding site, the strength of the metal's binding and metal's association with the sediment can be extracted (Yuan et al., 2004). Marcovecchio & Ferrer (2005) believed that the key to understand any possible toxic event that may occur in any environment's sediment was by understanding the available physico-chemical processes. It is because these processes can potentially control the binding of heavy metal and its geochemical partitioning in the sediments. Therefore, this study was done to investigate how As, Co, Cu and Zn are bound to the sediment particles in Terengganu River estuary and to define the fractions of these metals that are highly available to the environment.

Materials and Methods

Study area

Terengganu River estuary is located in the district of Kuala Terengganu in the state of Terengganu, with the river basin area of approximately 4,600 km^2. Terengganu River estuary experiences a wet season during the final and early months of the year; which is the period of the northeast monsoon, with an average rainfall between 200 mm to 600 mm. On the other hand, dry

season in Terengganu River estuary occurs from April to September with an average rainfall between 100 mm to 125 mm (Lee et al., 2016; Sultan & Shazili, 2009). Some of the human activities that can be found in this area include traditional and modern shipbuilding, shipping and marina activities, aquaculture and construction. Figure 1 shows a map of the study area and Table 1 shows the coordinates and locations of the sampling sites.

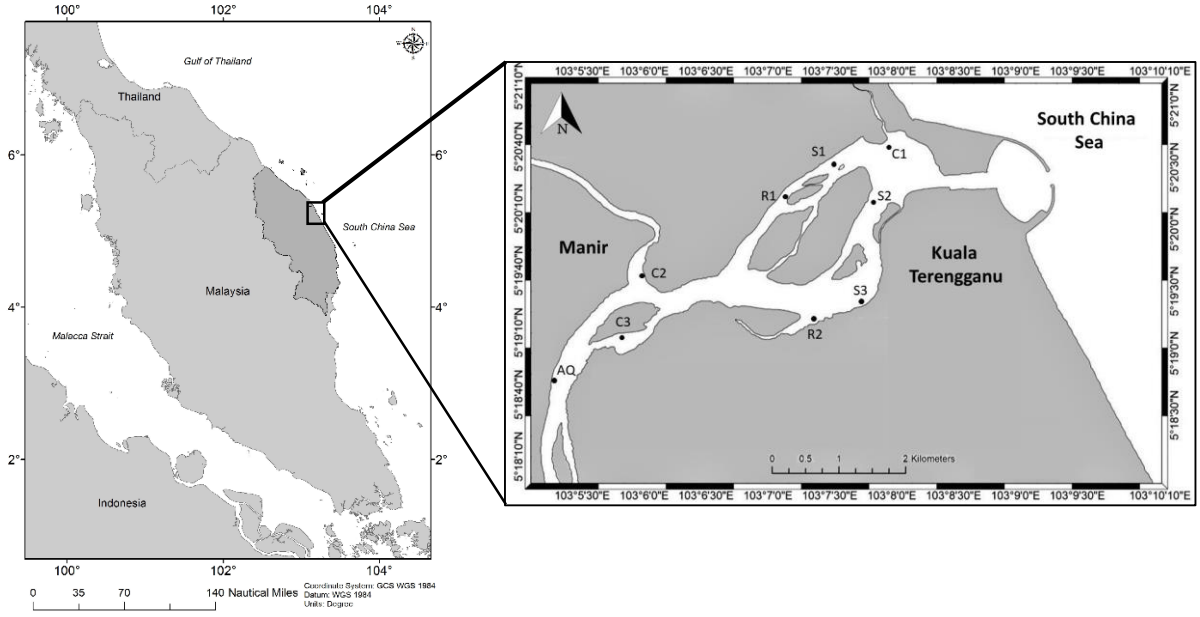

Figure 1: The sampling locations at Terengganu River Estuary

Table 1: Location of sampling sites in Terengganu estuary

Station	Name	Description	Coordinate
AQ	Pulau Rusa	Aquaculture	5°18.864" N, 103°7.866" E
C1	Seberang Takir	Land Reclamation	5°20.634" N, 103°7.866" E
C2	Kmpg. Bkt. Datu	Construction (Housing)	5°19.658" N, 103°5.959" E
C3	Pulau Sekati	Construction (Bridge)	5°19.114" N, 103°5.783"E
R1	Kmpg. Bkt. Tumboh	Residential area (Private Jetty)	5°20.267" N, 103°7.117" E
R2	Kmpg. Losong	Residential area	5°19.211" N, 103°7.263" E
S1	Kmpg. Batin	Shipbuilding (Traditional)	5°20.495" N, 103°7.443" E
S2	Pulau Duyong	Shipping & Marina	5°20.211" N, 103°7.829" E
S3	Pulau Kambing	Fish landing area	5°19.394" N, 103°7.680" E

Methodology

Sediment samples at each location were taken using a Ponar grab. Samples were then collected using plastic scoop and stored in clean vinyl bag. Collected samples were stored inside the ice chest (4°C) and then taken to the lab for analysis immediately. Upon reaching the lab, the collected sediments were transferred into 500 ml plastic containers and were oven dried at 55°C until constant weight was achieved.

Metals in the sediments were extracted using the modified BCR method as described by Nemati et al. (2011). It involved a three-step extraction using the acetic acid extraction, hydroxyl ammonium chloride extraction and hydrogen peroxide/ammonium acetate extraction. After that,

the remaining sediments were digested using mixed acid solution. The detail description of the steps are as follows:

Step 1 – One gram of sediment sample was weighed in 80-100 mL centrifuge tube, then 40 mL of 0.11 mol/L acetic acid (CH_3COOH) was added. The tube was closed and extracted by shaking it for 16 hours at 22 ± 5°C using rotary shaker. Any delay between the addition of the extractant solution and the beginning of the shaking were avoided.

Step 2 - 40 mL of freshly prepared 0.5 mol/L hydroxyl amine hydrochloride ($NH_2OH.HCl$) was added into the residue from Step 1 in the centrifuge tube. The solution was suspended by manually shook, then metals was extracted by shaking the mixture for 16 hours at 22 ± 5°C. Any delay between the addition of the extractant solution and the beginning of the shaking were avoided.

Step 3 - A small aliquot of 10 mL of hydrogen peroxide (H_2O_2) were added carefully to the residue in the centrifuge tube to avoid metal losses due to the chemical reaction. Tubes were covered loosely with its cap and were digested at room temperature for one hour with occasional manual shaking. The digestion were then continued for one hour at 85 ± 5°C with occasional manual shaking for first ½ hour in a water bath, then, the volume of H_2O_2 were further reduced to less than 3 mL through heating of the uncovered tube. An aliquot of 10 mL hydrogen peroxide was added again and the vessel was covered with its cap on. The digestion process was continued for one hour at 85 ± 5°C, with occasional manual shaking for first ½ hour, in a water bath. The tubes cap was then removed, and the solution was reduced to 1 mL by heating. Complete dryness of H_2O_2 was avoided. 50 mL of 1.0 mol/L ammonium acetate (NH_4CH_3COO) was added to the cool moist residue and it was shaken for 16 hours at 22 ± 5°C (overnight), similar to step 1 and step 2.

Step 4 – Residue from step 4 was digested using the ultra-high-purity grade (or equivalent) concentrated nitric acid (HNO_3), hydrofluoric acid (HF), and hydrochloric acid (HCl).

Inductively Coupled Plasma Mass Spectrometer (ICP-MS) was used to determine the concentration of heavy metal. The summary of BCR sequential extraction method and its target phase are shown in Table 2.

Table 2: Summary of sequential extraction method

Extraction steps	Reagent(s)	Target phase
Exchangeable fraction	CH_3COOH	Soil solution, carbonates, exchangeable metals
Reducible fraction	$NH_2OH.HCl$	Oxides of Fe-Mn
Oxidizable fraction	H_2O_2 followed by NH_4CH_3COO	Organic matter and sulphides
Residual fraction	HNO_3+HCl+HF	Remaining metals

Results and Discussion

The total concentration of As is shown in Figure 2a. In general, during the four month of observation, the concentration of total As varied from 2.18 to 17.48 mg/kg dry wt. Among four different studied areas, sampling locations which are closer to the construction and shipping activities yielded quite a high concentration of total As.

In Figure 2a, station C1 (land reclamation activities) which is located nearby the estuary mouth shows the highest average of total As, with the concentration of 13.88 mg/kg dry wt. While, the sampling station closed to small aquaculture cage (station AQ) which is located further away from the estuary mouth exhibited the lowest average of total As with the concentration of 4.59

mg/kg dry wt. In overall, based on Figure 2b, the highest percentage of As in the Terengganu River estuary samples is classified as the residual fraction which is commonly found in the crystal lattice of the sediment. As in the residual fraction made up to 38.6 to 93.0 % of the total As concentration in the sediment samples. The presence of As in the reducible fraction range from 4.84 to 59.98 %, remarks as the second largest fraction. Approximately 0.28 to 12.45 % of total As (Figure 2b) is classified as the exchangeable fraction. In the meantime, a small percentage of As was found in the oxidizable fraction with the percentage range from 0.46 to 4.04 %.

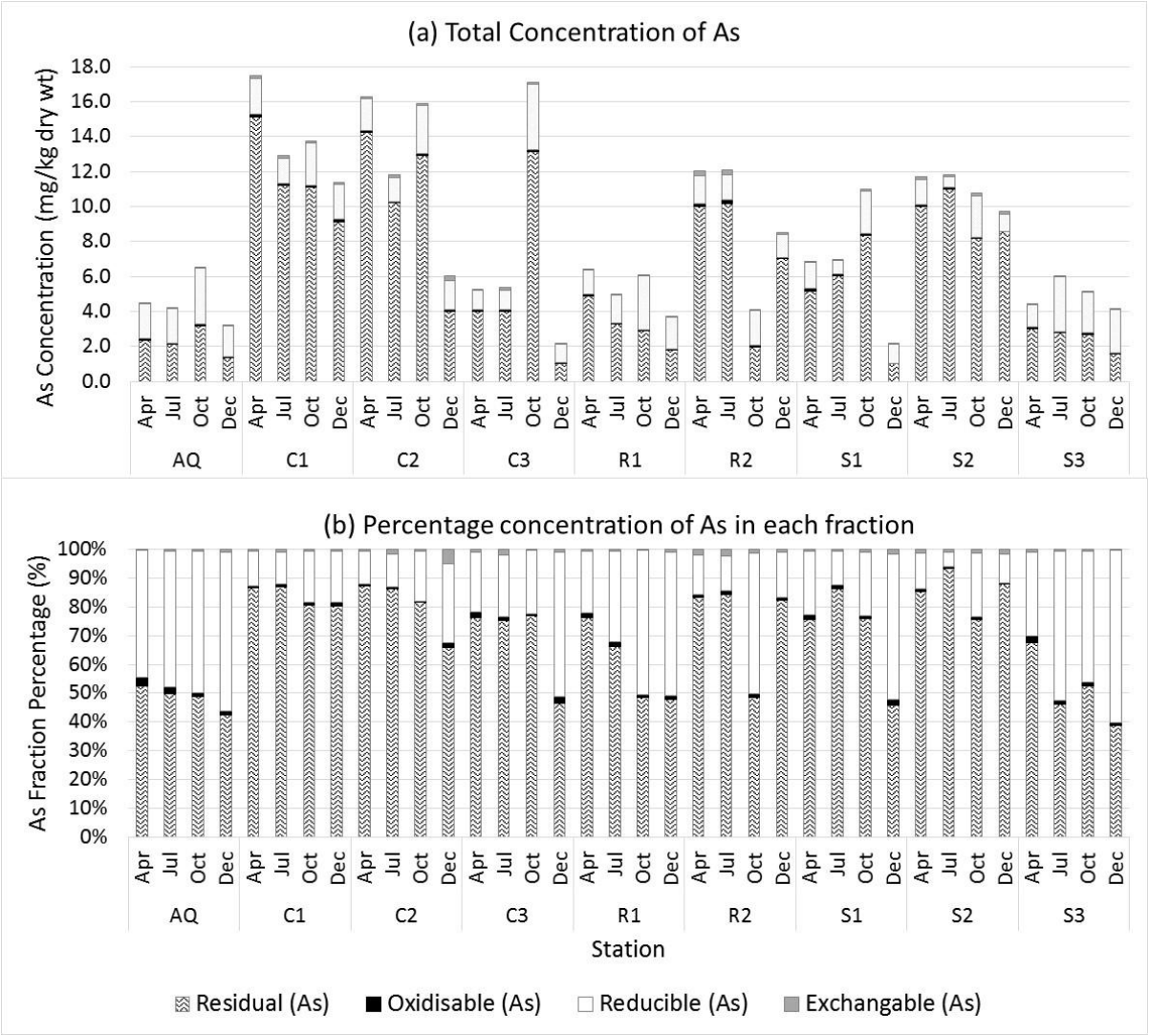

Figure 2: (a) Total concentration of As in mg/kg dry weight and (b) percentage As concentration by fraction in the Kuala Terengganu River estuary.

The total concentration of Co ranged between 1.53 to 20.53 mg/kg dry wt (Figure 3a). Similar to total As, the average total Co for four months of observation was higher at Seberang Takir area (station C1), where the land reclamation processes was observed. The average total concentration of Co for all four months is 13.86 mg/kg dry wt. The residential area within Kampung Bukit Tumboh (station R1) exhibited the lowest average of total Co for four months, with concentration of 6.06 mg/kg dry wt. In the meantime, the residential area within Kampung Losong (station R2) exhibited much higher average of total Co (11.94 mg/kg dry wt). Both stations are

located closed to the residential area, however the total concentration of Co is slightly different. This is may be due to the geographical factor, where station R1 is located in an open water flow area which only obstructs by jetty and few houses. Unlike station R1, station R2 is surrounded by sand shelf and mangrove, thus having a restricted water flow.

The concentration of Co by fractions is shown in Figure 3a. The percentage of exchangeable Co is high with 2.04 to 17.74 % out of the total Co extracted. The reducible fraction of Co range within 0.07 to 23.34 % of total Co extracted. In the meantime, the oxidizable fraction of Co range from 1.02 to 7.03 % of the total Co. In overall, the concentration of Co in Kuala Terengganu River estuary derived by the residual fraction. The Co residual fraction comprised approximately 73.0 to 95.5 % of the total Co.

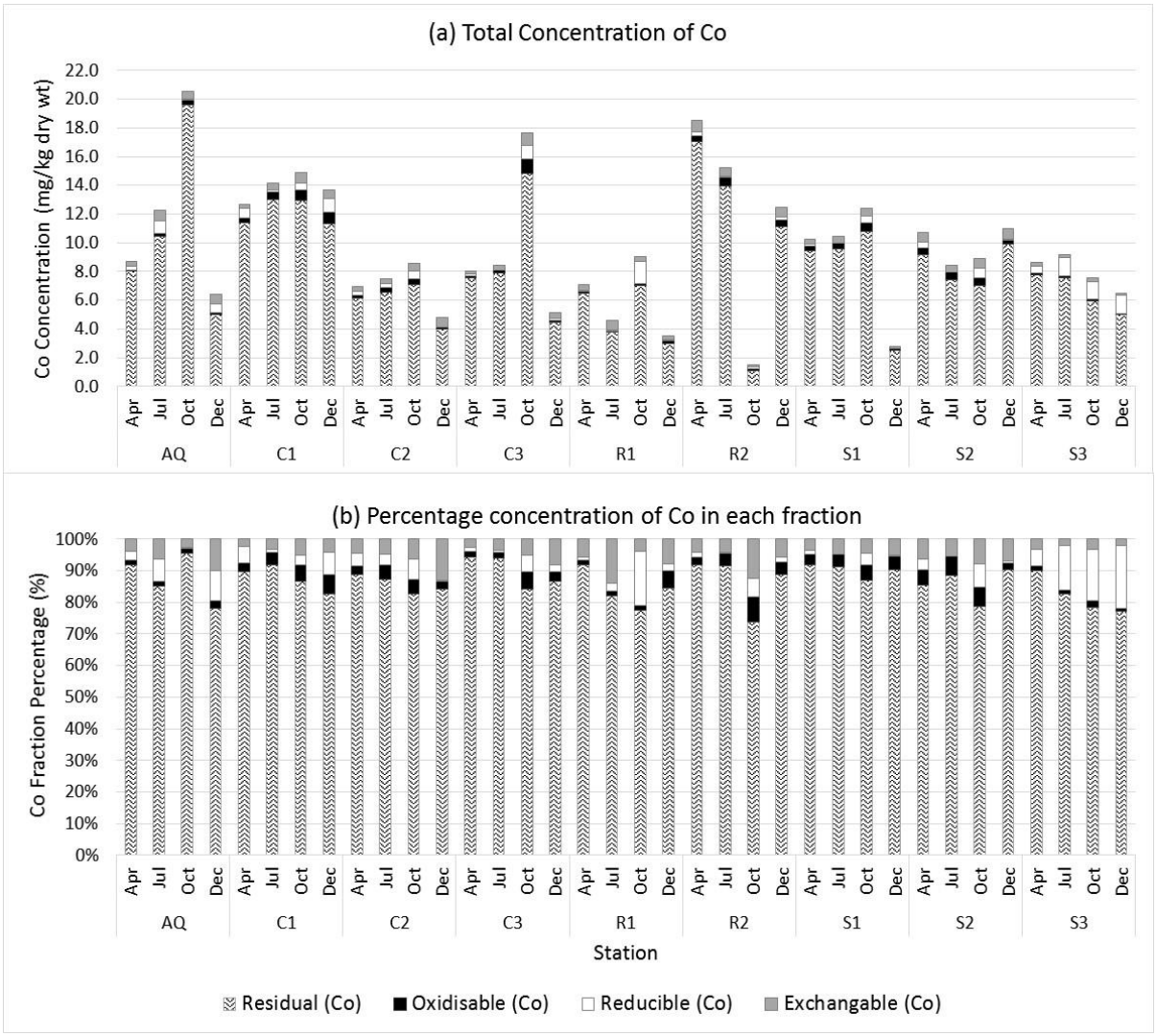

Figure 3: (a) Total concentration of Co in mg/kg dry weight and (b) percentage Co concentration by fraction in the Kuala Terengganu River estuary.

The total concentration of Cu in Kuala Terengganu River estuary varied between 1.22 to 15.98 mg/kg dry wt (Figure 4a). The highest average of total Cu was found at station C1 (10.93 mg/kg dry wt). The minimum average value of total Cu (2.97 mg/kg dry wt) for all four months was observed at station S3, located at Pulau Kambing, where the main jetty for fishermen land their

catches is located. The concentration of Cu in by fractions are shown in Figure 4b. In general, the total concentrations of Cu in the exchangeable fraction ranged from 0.82 to 17.08 %. Meanwhile, the collected sediment samples is consisted of 0.55 to 22.44 % of reducible Cu, which is almost similar to Cu in the oxidizable fraction, with the range of 0.77 to 22.59%. The highest concentration of Cu is found in the residual fraction, varied from 47.2 to 95.7 % of the total Cu.

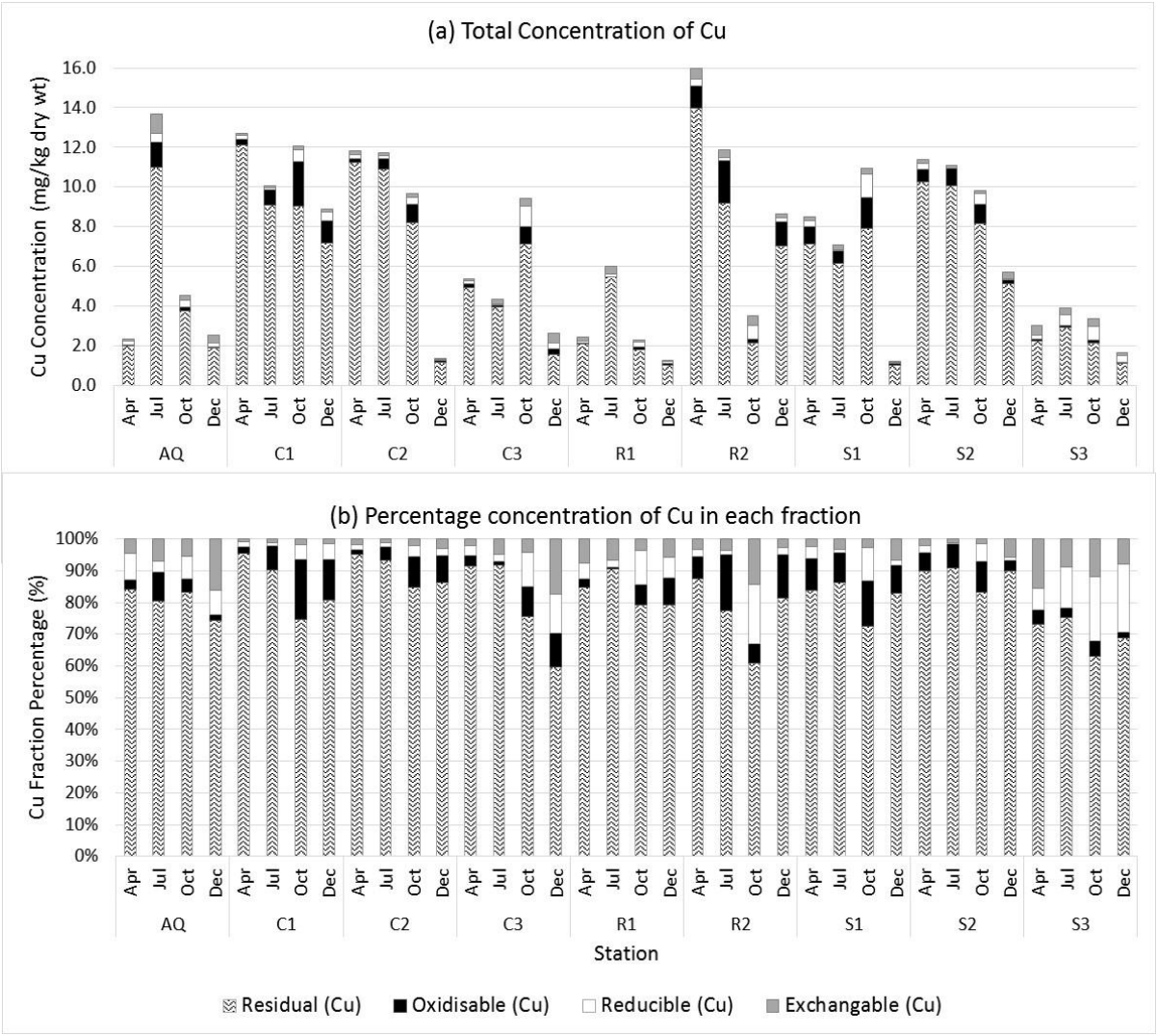

Figure 4: (a) Total concentration of Cu in mg/kg dry weight and (b) percentage Cu concentration by fraction in the Kuala Terengganu River estuary.

The total concentration of Zn for the four months of observation ranged from 6.10 mg/kg to 67.71 mg/kg (Figure 5a). Highest average total concentration of Zn is found at station C1, nearby the land reclamation area in Seberang Takir with the average total concentration of 48.22 mg/kg. Meanwhile, the lowest average for total Zn (12.84 mg/kg) in the sediment of Kuala Terengganu River Estuary is found at station R1, where the least human-impacted area of Kampong Bukit Tumboh residential area.

Based on Figure 5b, approximately 1.63 to 30.75 % of total Zn is classified in the exchangeable fraction. In the meantime, total Zn in the collected sediment samples comprised of 0.55 to 22.44 % in the reducible fraction, whereas 0.81 to 12.53% of the total Zn is in oxidizable

fraction. The residual fraction of Zn is the highest with 53.8 to 92.4% of the total Zn in the surface sediment Kuala Terengganu River estuary.

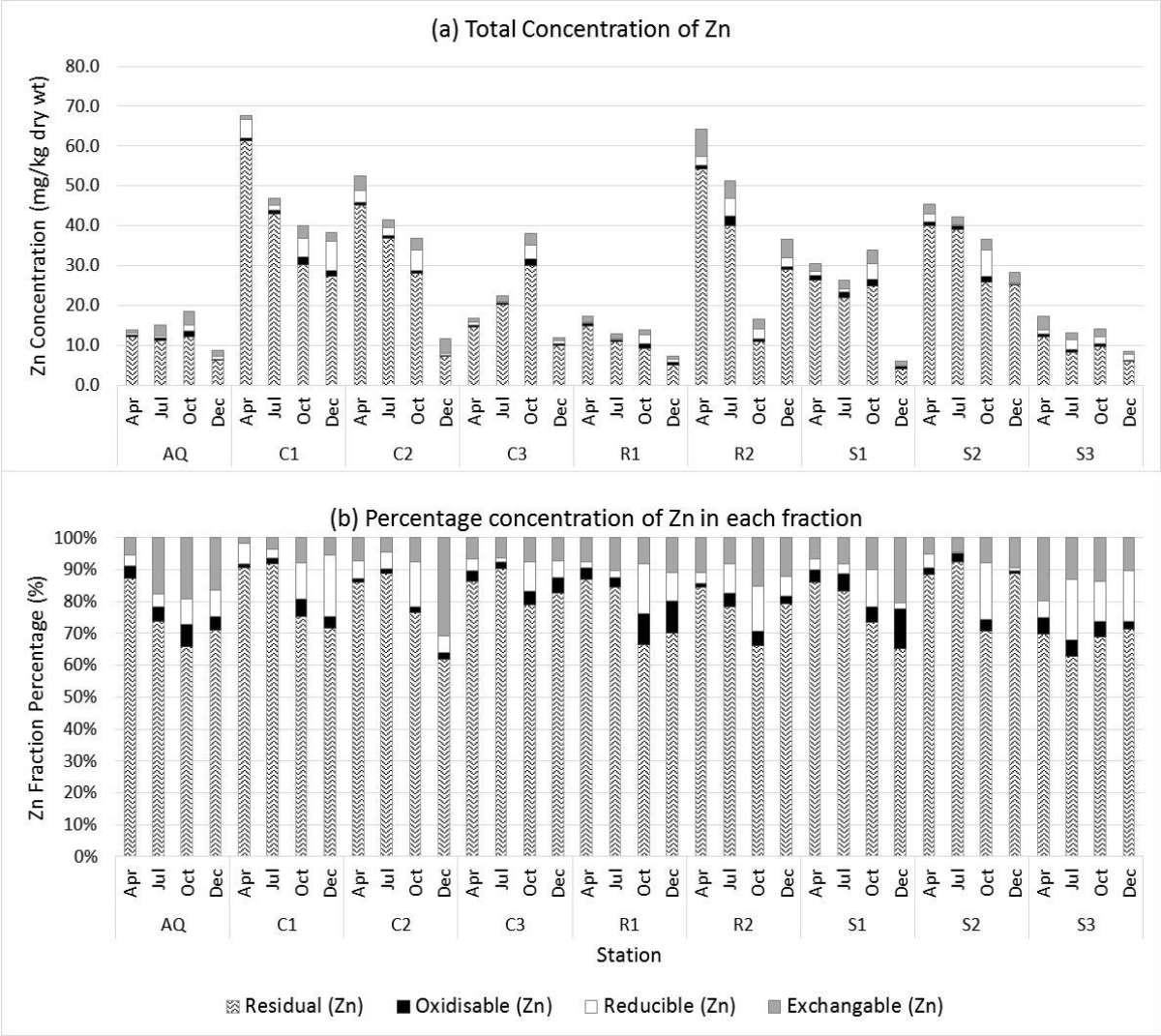

Figure 5: (a) Total concentration of Zn in mg/kg dry weight and (b) percentage Zn concentration by fraction in the Kuala Terengganu River estuary.

One noticeable trend is discovered after the four months of observation. Upon approaching the month of December, a decreasing in metals concentrations was observed. It has been suspected that the changes was affected by the monsoonal period on the surface sediment of the Kuala Terengganu River estuary. During the transitional periods of monsoon (i.e. April and October), the concentrations of total metal were higher at most of the stations. Lesser rainfall was recorded before the monsoons (South-west monsoon and North-east monsoon) start. The rainfall data provided by the Malaysian Meteorological Department (2017) showed that there was less rainfall during this period. Kuala Terengganu district only experienced approximately 32.2 mm of rainfall in 10 days during April, while in October, Kuala Terengganu received only about 309.4 mm of rainfall in 12 days. However, with the presence of north-east monsoon during the month of December, heavy rainfall was brought to Kuala Terengganu district and most probably influenced the total concentrations of metals in the surface sediment of Kuala Terengganu River estuary.

Additionally, during this month, Kuala Terengganu has experienced quite a heavy rainfall, which was 546.2 mm of rainfall in 21 days; which was higher than the rainfall received during October. The rainfall received in July (132 mm in 11 days) was lower than October but higher than April.

Lower concentration of total metals that in the Kuala Terengganu River estuary sediment during the monsoonal season has suggested that the flushing of metals from the estuary to the nearby coastal area might have occurred (Budiyanto and Lestari, 2017). While almost all stations showed a decrease in total concentration of metal due to the heavy rainfall experienced in December, remarkably at station R2, as a very sharp increase in total concentration was observed. In Kuala Terengganu River estuary, the exchangeable fraction of Zn showed high average percentage (Figure 3b) in the sediment samples. This could be explained by the ability of exchangeable Zn to be absorbed into the sediment colloid via the electrostatic attraction with sediment surface (Li et al., 2009). Through the binding of exchangeable Zn with the sediments, it signified that this particular metal fraction is labile and have much lower affinity towards other available metal fraction in the sediment (Pérez-Cid et al., 1996). High presence of exchangeable Zn after residual fraction may suggest that this fraction have the ability to scavenge Zn from the aquatic environment.

High percentage of As reducible fraction (4.84 to 59.98 %) was observed compared to other metals (Figure 2a). Zhang et al. (2017) believed that presence of As in the river sediments might be related to abundance of iron-manganese (Fe-Mn) oxides in the study area itself. Results from this study are similar to those found by Baig et al. (2009). They inferred that the decreases in concentration of As in acid soluble and exchangeable fractions and natural increase in reducible fraction exhibited the possible adsorption of As in the sediment. The presence of As in Kuala Terengganu River estuary is also in agreement with those found by Arain et al. (2008), where the fact of As (V) adsorb onto the sediment and coexists with Fe-Mn oxy hydroxides is bound to happen.

Krupadam et al. (2007) explained that metals in the aqueous environment could be fixed to reducible fraction by processes like adsorption and co-precipitation; plus, metals could form inactive complex with the mixture. Furthermore, Cu and Zn in Kuala Terengganu River estuary sediment has exhibited highly presented in the reducible fraction. Studies carried out by Ahdy & Youssef (2011), Najamuddin et al. (2016) and Xiao et al. (2015) has found that a huge percentage of Zn was in the reducible phase. However, in this study, according to the fraction percentage of Zn in Figure 5b, approximately 0.55 to 22.44 % of Zn existed in the reducible fraction, which is the third most in percentage abundance after the residual and exchangeable fractions. Similar finding was found with Co and Cu, which suggests that only a small amount of these metals were adsorbed and stored to Fe-Mn oxides. The presence of Cu in the shipping area (station S1, station S2 and station S3) could possibly harm the environment. It may be due to the presence of Cu-based materials that originated from the ships itself such as the ships' paints and corrosion of Cu-based structure (Clement et al., 2010).

Most of the sampling stations showed low percentage of Zn and Cu in the reducible fraction. Krupadam et al., (2007) suggested that metals from the aquatic environment can be scavenged by Fe-Mn oxides by the process of adsorption and co-precipitation, which lead to the formation of inactive complex with the mixture. Generally, the reducible Zn and Cu ranged from 0.55 to 22.44% and 0.38 to 28.57% in the studied area and it is considered as low. High percentage of Cu in the organic fraction is observed in Figure 4b. This could be due to the preference of the metal itself, where Cu is more likely to bind organically with the particulate organic matter in the sediments (Bibby & Webster-Brown, 2006; Li et al., 2009; Yu et al., 2001). Additionally, the particulate organic matters might serve as an important ligand for the metal complexion especially for Cu. Furthermore, Zn that exists in the organic fraction could and might be related to Zn-related organic matter dumping from the nearby domestic and municipal waste and untreated industrial waste (Naji et al., 2010).

The dominancy of As, Co, Cu and Zn in the residual fraction is possibly suggest that these metals were held strongly inside the crystal lattice of the sediments (Xiao et al., 2015), thus justify the unavailability of this fraction to the aquatic environment. This could also be a result of the association of the metals with other mineral such as aluminosilicate minerals (Yuan et al., 2004). High presence of metals in the residual fraction signify that these metals are less likely to be available and absorbed by the living organisms. Thus, there is low possibilities of these metals entering and be accumulated in the food chains. Metal fractions that are mobile may possibility derive from the anthropogenic activities while non-mobile ones usually originate from the natural environment (Najamuddin et al., 2016). High percentage of the residual metals in sediment might be contributed by lithogenic sources and processes such as the weathering of rocks and erosion from nearby terrestrial soil (Yu et al., 2001). Since the residual As, Co, Cu and Zn exhibited the highest dominance among all fractions, it is possible that these metals came from similar sources. Besides that, there are also high chances that these metals came from foreign soil, being unintentionally introduced during the processes of developing the Kuala Terengganu River estuary; such as the land reclamation activities at station C1, bridge construction at station C2 and housing construction near river at station C3. Moreover, sediment that carried metals could have been derived from the river upstream and deposited by river flow and currents (Gómez-Álvarez et al., 2011).

Conclusion

The application of the BCR 3-steps sequential extraction method helps in identifying the behaviour and mobility of As, Co, Cu and Zn in the Kuala Terengganu River estuary sediment. Generally, these metals were mostly bounded to the residual fraction, which makes them highly unavailable to the environment. These metals do not have common binding characteristics. In Terengganu River estuary, As was mostly bounded to the reducible and residual fractions, while majority of Co was found in the residual fraction. In contrast, Cu has the tendency to be attached to the oxidizable and residual fractions of the sediment. Zn was mostly bounded to the exchangeable and residual fractions. Basically, the dominance of metal in available fraction varies depending on the properties of the metals itself. The dominance of each fraction by metals can be arranged as follows:

As: residual > reducible > exchangeable > oxidizable;
Co: residual > exchangeable > reducible > oxidizable;
Cu: residual > oxidizable > reducible > exchangeable;
Zn: residual > exchangeable > reducible > oxidizable.

Through this study, it is proven that the sequential extraction method is important as it can provide extra information on metal's potential availability and other crucial information which is helpful for fully assessing the condition of the environment.

Acknowledgments

Special thanks to the Ministry of Higher Education for funding through the Fundamental Research Grant Scheme (FRGS/1/2016/WAB09/UMT/02/3). We thank our colleagues from the Faculty of Science and Marine Environment, Universiti Malaysia Terengganu who provided insight and expertise that greatly assisted the research.

References

Ahdy, H. H. H., & Youssef, D. H. 2011. Fractionation analysis of some heavy metals in sediments of the north-western part of the Red Sea, Egypt. *Chemistry and Ecology* 27(5): 427–443.

Arain, M. B., Kazi, T. G., Jamali, M. K., Jalbani, N., Afridi, H. I., & Shah, A. 2008. Total dissolved and bioavailable elements in water and sediment samples and their accumulation in Oreochromis mossambicus of polluted Manchar Lake. *Chemosphere* 70(10): 1845-1856.

Baig, J. A., Kazi, T. G., Arain, M. B., Shah, A. Q., Sarfraz, R. A., Afridi, H. I., & Khan, S. 2009. Arsenic fractionation in sediments of different origins using BCR sequential and single extraction methods. *Journal of Hazardous Materials* 167(1–3): 745–751.

Bibby, R. L., & Webster-Brown, J. G. 2006. Trace metal adsorption onto urban stream suspended particulate matter (Auckland region, New Zealand). *Applied Geochemistry* 21(7): 1135-1151.

Budiyanto, F., & Lestari, L. 2017. Temporal and Spatial Distribution of Heavy Metal in Sediment of Urban Coastal Waters: A Case Study in Jakarta Bay, Indonesia. *Bulletin of The Marine Geology* 32(1).

Clement, D., Keeley, N., & Sneddon, R. 2010. Ecological Relevance of Copper (Cu) and Zinc (Zn) in Sediments Beneath Fish Farms in New Zealand. *Marborough District Council*, Report Np. 1805, pg48. Retrieved May 6, 2018 from www.cawthron.org.nz

Delgado, J., Nieto, J. M., & Boski, T. 2010. Analysis of the spatial variation of heavy metals in the Guadiana Estuary sediments (SW Iberian Peninsula) based on GIS-mapping techniques. *Estuarine, Coastal and Shelf Science* 88(1): 71–83.

Doong, R. an, Lee, S. hui, Lee, C. chee, Sun, Y. chang, & Wu, S. chee. 2008. Characterization and composition of heavy metals and persistent organic pollutants in water and estuarine sediments from Gao-ping River, Taiwan. *Marine Pollution Bulletin* 57(6–12): 846–857.

FDTP Kuala Teengganu. 2008. *Rancangan Tempatan Daerah Kuala Terengganu 2008-2020*. Kuala Terengganu.

Gómez-Álvarez, A., Valenzuela-García, J. L., Villalba-Atondo, A. I., Meza-Figueroa, D., Almendariz-Tapia, F. J., Whitaker-Bojórquez, T. O., Ochoa-Valenzuela, L. E. 2011. Distribution of heavy metals and their chemical speciation in sediments from the Abelardo L. Rodríguez Dam, Sonora, México. *Chemical Speciation and Bioavailability* 23(4): 201–212

Ikem, A., Egiebor, N. O., & Nyavor, K. 2003. Trace elements in water, fish and sediment from Tuskegee Lake, Southeastern USA. *Water, Air, and Soil Pollution* 149: 51-75.

Krupadam, R. J., Ahuja, R., & Wate, S. R. 2007. Heavy metal binding fractions in the sediments of the Godavari estuary, East Coast of India. *Environmental Modeling and Assessment* 12: 145-155.

Lee, H. L., Tangang, F., Hamid, M. R., Benson, Y., & Razali, M. R. 2016. Modeling the Influence of River Flow and Salt Water Intrusion in the Terengganu Estuary, Malaysia. *IOP Conference Series: Materials Science and Engineering* 136(1).

Li, Jianguo, Pu, L., Zhu, M., Liao, Q., Wang, H., & Cai, F. 2014. Spatial pattern of heavy metal concentration in the soil of rapid urbanization area: A case of Ehu Town, Wuxi City, Eastern China. *Environmental Earth Sciences* 71: 3355-3362.

Li, Jinling, He, M., Han, W., & Gu, Y. 2009. Availability and mobility of heavy metal fractions related to the characteristics of the coastal soils developed from alluvial deposits. *Environmental Monitoring and Assessment* 158: 459-469.

Marcovecchio, J., & Ferrer, L. 2005. Distribution and Geochemical Partitioning of Heavy Metals in Sediments of the Bahía Blanca Estuary, Argentina. *Journal of Coastal Research* 214(4): 826–834.

Najamuddin, Prartono, T., Sanusi, H. S., & Nurjaya, I. W. 2016. Seasonal distribution and geochemical fractionation of heavy metals from surface sediment in a tropical estuary of Jeneberang River, Indonesia. *Marine Pollution Bulletin* 111(1-2): 456-462.

Naji, A., Ismail, A., & Ismail, A. R. 2010. Chemical speciation and contamination assessment of Zn and Cd by sequential extraction in surface sediment of Klang River, Malaysia. *Microchemical Journal* 95(2): 285–292.

Nemati, K., Bakar, N. K. A., Abas, M. R., & Sobhanzadeh, E. (2011). Speciation of heavy metals by modified BCR sequential extraction procedure in different depths of sediments from Sungai Buloh, Selangor, Malaysia. *Journal of Hazardous Materials*, *192*(1), 402–410.

Pérez-Cid, B., Lavilla, I., & Bendicho, C. 1996. Analytical assessment of two sequential extraction schemes for metal partitioning in sewage sludges. *Analyst*. https://doi.org/10.1039/an9962101479.

Saleem, M., Iqbal, J., Akhter, G., & Shah, M. H. 2018. Fractionation, bioavailability, contamination and environmental risk of heavy metals in the sediments from a freshwater reservoir, Pakistan. *Journal of Geochemical Exploration* 184: 199–208.

Sultan, K., & Shazili, N. A. 2009. Distribution and geochemical baselines of major, minor and trace elements in tropical topsoils of the Terengganu River basin, Malaysia. *Journal of Geochemical Exploration* 103(2–3): 57–68.

Ure, A. M., Muntau, H., Quevauviller, P., & Griepink, B. 1993. Speciation of heavy metals in soils and sediments an account of the improvement and harmonization of extraction techniques undertaken under the auspices of the bcr of the commission of the european communities. *International Journal of Environmental Analytical Chemistry* 51(135).

Xiao, R., Bai, J., Lu, Q., Zhao, Q., Gao, Z., Wen, X., & Liu, X. 2015. Fractionation, transfer, and ecological risks of heavy metals in riparian and ditch wetlands across a 100-year chronosequence of reclamation in an estuary of China. *Science of the Total Environment* 517C: 66-75.

Yu, K. C., Tsai, L. J., Chen, S. H., & Ho, S. T. 2001. Correlation analyses on binding behavior of heavy metals with sediment matrices. *Water Research* 35(10): 2417-2428.

Yuan, C.-G., Shi, J.-B., He, B., Liu, J.-F., Liang, L.-N., & Jiang, G.-B. 2004. Speciation of heavy metals in marine sediments from the East China Sea by ICP-MS with sequential extraction. *Environment International* 30(6): 769–783.

Zhang, G., Bai, J., Xiao, R., Zhao, Q., Jia, J., Cui, B., & Liu, X. 2017. Heavy metal fractions and ecological risk assessment in sediments from urban, rural and reclamation-affected rivers of the Pearl River Estuary, China. *Chemosphere* 184: 278–288.

How to cite this paper: Muhammad Nazirul Mubin Abd Halim Shah, Lee Siang Hing, Mohd Yusoff Nurulnadia, Meng Chuan Ong (2020). Fractionation of As, Co, Cu and Zn by Sequential Extraction in Surface Sediment of Kuala Terengganu River Estuary. *Malaysian Journal of Applied Sciences, 5*(2), 57-68.