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# A First Record of Metal Fractionation in Coastal Sediment from Ambon Bay, Moluccas, Indonesia

## Catatan Awal Fraksinasi Logam dalam Sedimen Pesisir dari Teluk Ambon, Maluku, Indonesia

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**ABSTRACT:** Five metals in sediment samples at seven sites from the Ambon Bay were analyzed with BCR sequential extraction procedure to determine chemical fractionation of metals and to assess bioavailability of metals with Risk Assessment Code (RAC). The result showed that the percentages of cadmium (100%), lead (82.6-97.08%) and zinc (41.68-76.33%) were mostly accumulated in the non-residual (F1+F2+F3) fraction of the total concentrations. While the copper percentages (44.74-78.91%) and nickel (59.71-74.16%) were mostly accumulated in residual (F4) fraction of the total concentrations. The Risk Assessment Code (RAC) reveals that cadmium, copper, nickel and zinc at locations exist in acid soluble (exchangeable) fraction and therefore, they are in low until very high risk category meanwhile there is no Pb at locations exist in acid soluble (exchangeable) fraction.

**Keywords :** metal fractionation; sediment; RAC; Ambon Bay.

**ABSTRAK :** Lima logam dalam sampel sedimen di tujuh lokasi dari Teluk Ambon dianalisis dengan prosedur ekstraksi berurutan BCR untuk menentukan fraksinasi kimia logam dan untuk menilai bioavailabilitas logam dengan Risk Assessment Code (RAC). Hasilnya menunjukkan bahwa persentase kadmium (100%), timbal (82,6-97,08%) dan seng (41,68-76,33%) sebagian besar terakumulasi dalam fraksi non-residual dari total konsentrasi. Sementara persentase tembaga (44,74-78,91%) dan nikel (59,71-74,16%) sebagian besar terakumulasi dalam fraksi residu dari total konsentrasi. Risk Assessment Code (RAC) mengungkapkan bahwa kadmium, tembaga, nikel, dan seng di lokasi berada pada fraksi yang dapat larut dalam asam (dan dapat ditukar) dan oleh karena itu, mereka berada dalam kategori risiko rendah hingga sangat tinggi sementara Pb di lokasi tidak ada dalam fraksi larut dalam asam (dapat ditukar).

**Kata kunci :** fraksinasi logam, sedimen, RAC, Teluk Ambon

## INTRODUCTION

Pollution of heavy metal is a critical environmental issue because of the nature of the contaminants is persistent and not biodegradable (Sarkar *et al.*, 2014). Sediments are the ultimate sink for heavy metals of both natural and anthropogenic origin (Campbel *et al.*, 1988), also for pollution (Hlavay *et al.*, 2004). Toxicity and mobility of metals are strictly related to their chemical form; for this reason, the knowledge of the total metal content of a matrix does not allow to achieve information on their bioavailability and eco-toxicology. Trace metals in sediments and soils can be associated with several geochemical phases, the most important of which can identify as carbonates, iron and manganese oxides, sulfides, organic matter. Sequential extraction procedures allow the individuation of the metals partitioning along the

fractions of solid environmental matrices, providing therefore, useful information on their bioavailability and mobility (Craba *et al.*, 2004).

Ambon City is the capital of Moluccas Province that surroundings by Ambon Bay. It is separated by a shallow sill ( $\pm 12$ m depth) between the outer and inner part as geographically (Corvianawatie *et al.*, 2016). Ambon Bay is the coastal area that is vulnerable to pollution due to anthropogenic activities such as urban, farming, shipping activities, and ship repair activities from the dockyard around the Ambon Bay that may contribute to metal inputs to the Ambon Bay (Manullang *et al.*, 2017). In Ambon Bay, total metal concentration in sediment has been reported by many studies (Siahaya *et al.*, 2013, Tupan *et al.*, 2013, Manullang *et al.*, 2017).

Metal bioavailability in sedimentary environmental samples may provide a better understanding of the interaction of environment-organisms. The bioactivity and bioavailability of metals is very dependent on the forms of chemical connections, and their speciation. Determination of total metal concentration in environmental substances is not enough to assess the environmental impact of polluted sediments because heavy metals may have different chemical forms and only a small portion can be easily remobilized. Studies on the distribution and speciation of heavy metals in sediments not only provide the information about pollution levels, but also especially actual environmental impacts on the bioavailability of metals and their origin (Benson *et al.*, 2013).

The information on heavy metals speciation studies with different methods has been done in some in polluted areas in Indonesia such as Semarang City (Takarina *et al.*, 2004), Dumai, Sumatera (Amin *et al.*, 2007), Delta Berau East Kalimantan (Arifin *et al.*, 2010), Jenebarang River (Najamuddin *et al.*, 2016). The Community Bureau of Reference (BCR) protocols for sediment and soil give a reasonable basis for most of the solid samples, and the results can be compared among different laboratories (Hlavay *et al.*, 2004). BCR method used in several studies such as Spermonde Island, Makassar (Werolangi *et al.*, 2016) and Banten Bay (Lestari *et al.*, 2018).

The aim of this study to determine the chemical fractionation of metals and to assess the bioavailability of

metals with Risk Assessment Code. The result from this paper will provide a better understanding of environmental risk of metals in the surface sediment of the Ambon Bay.

## METHOD

### Sampling Procedure

Seven sediment samples (0 to 10cm from surface) at 10 to 12m water depth were collected using a stainless-steel grab from 25 to 28 August 2015, located in Ambon Bay. These sampling sites were in the area that is influenced by anthropogenic stress and water circulation. Three sampling areas located in Ambon Inner Bay (AIB), one site in Threshold Galala and the other three in Ambon Outer Bay (AOB), as featured in Figure 1 (Manullang *et al.*, 2017).

Sediment samples that have been collected, were then labelled and stored in a cooler box before being taken to the laboratory. In laboratory, the sediment samples were dried at a temperature of 60°C until it reaches a constant weight, then homogenized using mortar. In the laboratory facility of the Research Centre for Oceanography, Indonesia Institute of Sciences, sediment samples were prepared for fractionation of metal analysis.

### Fractionation of metals analysis

The fractionation method used a BCR three-step sequential extraction procedure from Cuong and Obbard (2006) with a small modification in the amount

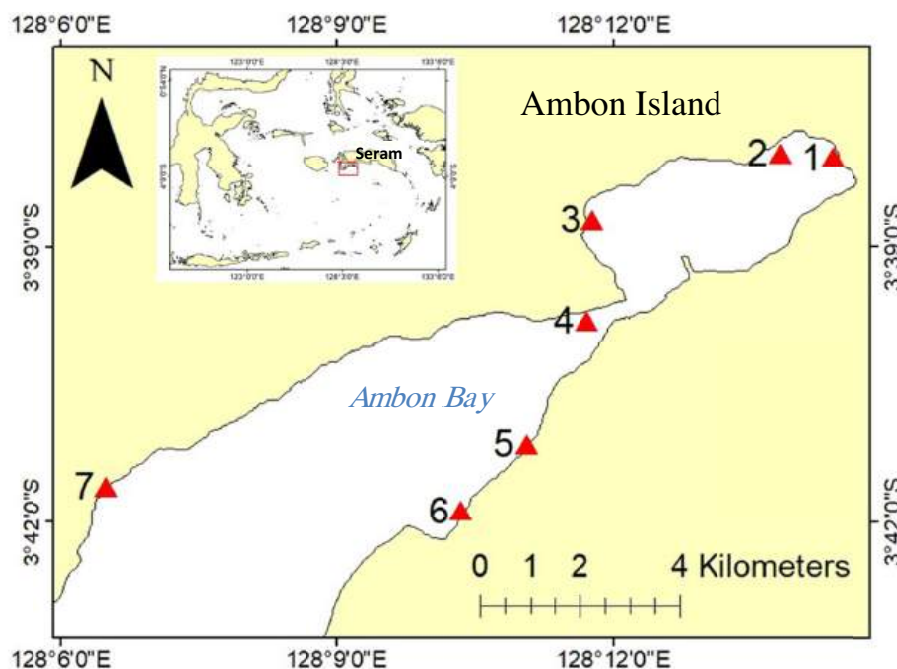


Figure 1. Location of sampling sites in Ambon Bay, Indonesia (Manullang *et al.*, 2017)

of sample and reagent for residual step analysis. This method provides information on four fractions, namely, exchangeable and carbonates (F1); reducible (F2); oxidizable (F3); and residual (F4). The total content is determined by the sum of all fraction (F1+F2+F3+F4). The fractionation method is described in Figure 2.

F1+F2+F3+F4 described total Cd, Cu, Ni and Pb in each station. The range concentrations of the five metals varied as follows: Cd (0.07-0.19 mg/kg dw), Cu (11.0-41.1 mg/kg dw), Ni (18.6-197 mg/kg dw), Pb (11.5-17.4 mg/kg dw) and Zn (35.3-147 mg/kg dw). The highest concentration of these metals was varied in these location. Total Cd was found in Passo (Station 1),

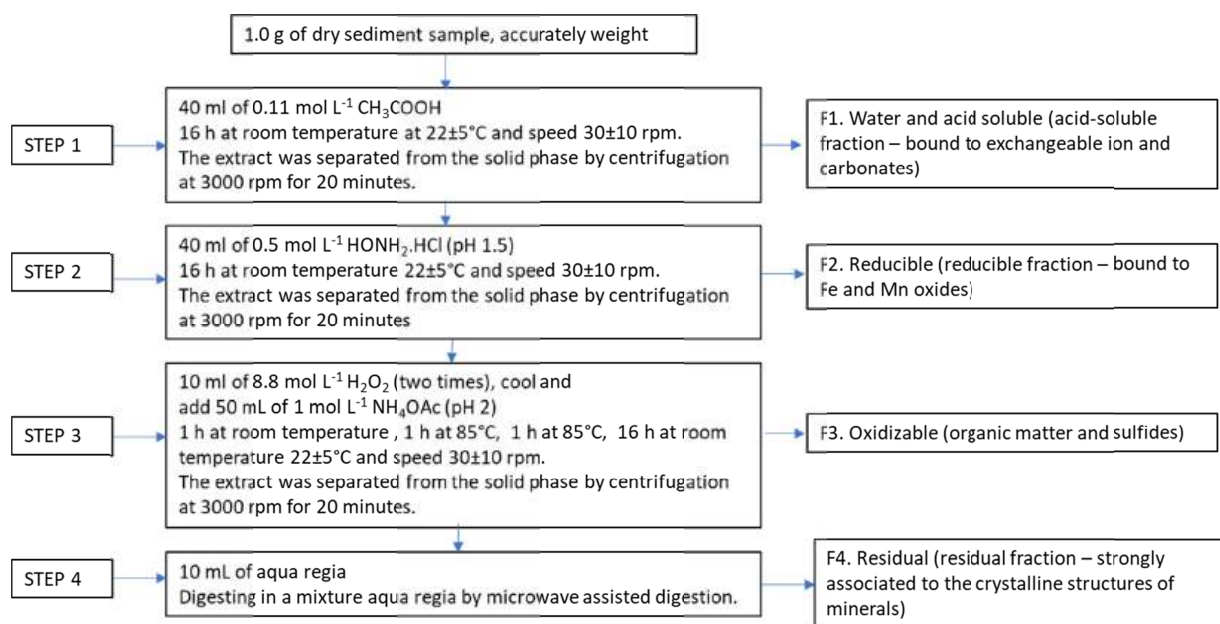


Figure 2. The fractionation method of sequential extraction procedure (Cuong and Obbard 2006 with modification)

### Risk Assessment Code (RAC)

The result of this fractionation study showed that the metals in sediments are bound to different fractions with different strengths. These strength values give a clear indication of sediment reactivity supporting the risk assessment due to the metal existence in Ambon Bay. Many previous studies have used the criteria of Risk Assessment Code (RAC) to evaluate sediment quality (e.g. Jain, 2004; Turki, 2007; Horvath *et al.*, 2013; Morelli and Gasparon, 2014). The criteria of Risk Assessment Code (RAC) indicated that sediment releasing exchangeable and carbonate fractions (F1) less than 1% of the total metal, was considered safe for the environment. On the contrary, the sediment releasing more than 50% of the total metal in the same fraction has been considered highly dangerous and high possibility to enter the food chain (Jain, 2004).

## RESULT AND DISCUSSION

### Total Metals Concentration in Sediment

The concentration of Cd, Cu, Ni, Pb and Zn in total and each geochemical fraction of coastal sediment from Ambon Bay are presented in Table 1. The sum of

meanwhile total Cu was found in Waiheru (Station 2). Total Ni was found in Poka (Station 3), Ambang Galala (Station 4) and Passo (Station 1), total Pb was found in Waihaong (Station 6) and Waiheru (Station 2) while total Zn was found in Ambang Galala (Station 4). The population in Waiheru and Poka are less dense than that of Passo. Meanwhile Batu Merah and Waihaong are area with a large population. The biggest port of Ambon is located around Waihaong, whereis the highest total Pb was detected. Concentration of Cd and Pb in this area were lower than in East China Sea (Andreas & Zhang, 2016). Cu concentration in this location is almost in the same amount as in Cirebon waters (Lestari, 2018), but lower than Muara Angke in Jakarta Bay (Lestari and Budiyanto, 2019), as the Zn concentration status.

### Fractionation of Metal in sediment

The potential environmental risk of trace elements in sediments is associated with their total content either their fractionation. The chemical partitioning of the considered metals (Cd, Cu, Ni, Pb and Zn) from each extraction step has been described in Figure 3 and showed that the metals in each location were fractioned differently.

Table 1. Concentration of Cd, Cu, Ni, Pb and Zn in each geochemical fraction of Ambon Bay coastal sediment (in mg/kg dw)

No	Location	F1 Acid Soluble	F2 Reducible	F3 Oxidizable	F4 Residual	$\Sigma$ Cd Fraction
1	Passo	0.14	0.02	0.03	nd	0.19
2	Waiheru	0.02	0.02	0.12	nd	0.17
3	Poka	0.02	0.02	0.12	nd	0.17
4	Ambang Galala	0.10	0.02	0.03	nd	0.15
5	Batu Merah	0.01	0.06	0.07	nd	0.15
6	Waihaong	0.02	0.02	0.03	nd	0.07
7	Tawiri	0.01	0.02	0.03	nd	0.07

No	Location	F1 Acid Soluble	F2 Reducible	F3 Oxidizable	F4 Residual	$\Sigma$ Cu Fraction
1	Passo	0.47	1.85	1.25	13.3	16.9
2	Waiheru	2.75	7.51	4.11	26.7	41.1
3	Poka	1.45	3.95	1.27	11.6	18.2
4	Ambang Galala	2.74	6.72	5.57	12.2	27.2
5	Batu Merah	0.36	0.79	2.58	7.24	11.0
6	Waihaong	0.26	1.88	2.89	16.3	21.3
7	Tawiri	0.37	1.23	5.16	9.59	16.3

No	Location	F1 Acid Soluble	F2 Reducible	F3 Oxidizable	F4 Residual	$\Sigma$ Ni Fraction
1	Passo	9.21	14.4	14.1	69.1	107
2	Waiheru	1.79	3.08	3.85	25.1	33.8
3	Poka	15.4	26.2	31.7	124	197.0
4	Ambang Galala	14.5	13.0	17.1	77.5	122.1
5	Batu Merah	3.67	1.76	1.61	11.5	18.6
6	Waihaong	3.24	2.43	2.44	21.8	29.9
7	Tawiri	6.43	4.02	3.63	20.9	34.9

No	Location	F1 Acid Soluble	F2 Reducible	F3 Oxidizable	F4 Residual	$\Sigma$ Pb Fraction
1	Passo	nd	9.25	1.33	2.23	12.8
2	Waiheru	nd	13.5	1.73	1.89	17.1
3	Poka	nd	9.44	2.11	0.35	11.9
4	Ambang Galala	nd	13.4	0.97	2.26	16.7
5	Batu Merah	nd	10.6	0.59	0.34	11.5
6	Waihaong	nd	14.2	0.60	2.66	17.4
7	Tawiri	nd	12.4	3.21	1.11	16.7

No	Location	F1 Acid Soluble	F2 Reducible	F3 Oxidizable	F4 Residual	$\Sigma$ Zn Fraction
1	Passo	3.01	17.3	4.37	27.5	52.2
2	Waiheru	11.1	15.7	14.8	58.1	99.7
3	Poka	36.8	18.0	5.87	36.8	97.5
4	Ambang Galala	67.9	30.1	14.4	34.9	147
5	Batu Merah	9.78	9.06	2.13	14.3	35.3
6	Waihaong	13.9	11.3	4.93	40.2	70.3
7	Tawiri	5.64	26.9	9.71	26.2	68.5

Cd concentrations in sediments are a major concern because of their high toxicity. BCR procedures have been used to obtain information about the distribution of Cd in sediments. The acid soluble fraction (F1) contributed a higher portion for Cd 33% in this study as well as several studies in other areas (Andreas & Zhang, 2016, Nemati *et al.*, 2011, Qiao *et al.*, 2013, Lin *et al.*, 2014, Yang *et al.*, 2014, Cao *et al.*, 2015, Lin *et al.*, 2016, Gu, 2018). Cadmium in this fraction is easy to move, unstable, bioavailability, toxicity, and is therefore easily extracted from sediment by biota (Andreas & Zhang, 2016). Contribution of Cu only 5%, Ni 12%, Zn 22%, and there is no Pb in this fraction (F1). The F2 fraction is portion of a metal that is enclosed in Fe/Mn oxide or hydroxide precipitation, that is hard to remove in consequence of hard ionic bonds. However, this can be deoxidized and lead to second pollution, if potential redox and oxygen in seawater declined (Lin *et al.*, 2014). The reducible fraction (F2) contributed the highest portion to Pb (80%), meanwhile contribution to Cd only about 23%, Cu 14%, Ni 11%, and Zn 24%. In the F3 fraction, metal ions as the center of action are bound with reactive groups of organic materials or produce materials that are not dissolved in water with sulfur ions, as a result, it is complicated to be liberated under normal moderate reduction or weak oxidation environment (Lin *et al.*, 2014). The organic fraction (F3) contributed highest portion for Cd 45%, while for Cu only about (16%), Ni (12 %), Pb (10%), and Zn (9%). In the F4 fraction, metal ions are bound to the aluminosilicate lattice, due to their strong stability, lowest bioactivity and toxicity so they are considered to have no bioavailability (Lin *et al.*, 2014). The residual fraction (F4) is the most important fraction for Cu (65%), Ni (66%) and Zn (44%) whilst for Pb only 10% and no Cd contribution. Thus, Cd fractionation mainly consists of F1 and F3 fractions, Pb bound to F2, Cu, Ni, and Zn are dominated by F4 in surface sediments in Ambon Bay. The result of this study is almost similar to the condition in Xiamen Bay (Lin *et al.*, 2014). In Muara Angke, Jakarta Bay, the main content of Zn is F1, Pb is strongly tied to F2, Cu is bound to F3 while Ni dominates F4 (Lestari and Budiyo, 2019). While in the waters of Cirebon and Banten Bay, Cu, Ni and Zn are dominated by F4 (Lestari *et al.* 2018).

Figure 3 showed that the distribution of the heavy metals in percent bar graphs in the four fractions (F1 is acid soluble fraction, F2 is reducible fraction, F3 is oxidizable fraction, and F4 is residual fraction) obtained in our analysis. The partitioning showed of metals associated with the non-residual fraction (consists of F1 + F2 + F3) and the residual fraction. The partitioning showed that the percentages of 3 metals (Cd, Pb and Zn) are more associated to the non-residual

fractions (100% for Cd; 90% for Pb and 55.9% for Zn) than to the residual fraction. This indicates that these metals are derived from anthropogenic inputs instead of geochemical background (especially for Cd and Pb). In contrast, two metals (Cu and Ni) are greater in residual fraction (F4) with percentage 64.7% and 65.7% respectively. Therefore, the results indicated that the metals are potentially more available for exchange and/or release into the marine environment. The studied metals in the non-residual fractions increased in the order of Cd>Pb>Zn>Cu> Ni.

The proportion of cadmium 100%, lead 82.6-97.08% and zinc 41.68-76.33% were mostly accumulated in the non-residual fraction of the total concentrations, while some parts of copper 44.74-78.91% and nickel 59.71-74.16% were mostly accumulated in residual fraction of the total concentrations (Figure 3).

### Risk Assessment Code

The Risk Assessment Code (RAC) reveals that about 10-72.73% of cadmium, 1.2-10.1% of copper, 5.30-19.75% of nickel, 8.24-46.12% of zinc and <1 of lead at the location exist in the acid soluble fraction or exchangeable fraction. Therefore, Cd can be categorized as medium risk – very high risk, Zn is in low risk – high risk category, nickel and copper are considered low – medium risk and no risk for lead (Table 2). Therefore, monitoring of the presence of metal in the waters is need to be done because it is persistent, it can be accumulated, and it endanger the aquatic environment.

Table 2. 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

Sediments from the coast of Ambon Bay have been analyzed using sequential extraction procedures BCR. Speciation studies of Cd, Cu, Pb, Ni, and Zn in Ambon Bay sediments showed that Cd fractionation mainly consists of soluble acid fraction (F1) and oxidized fraction (F3), Pb is bound to the reducible fraction (F2), whereas Cu, Ni and Zn are dominated by residual fraction (F4), however contribution of total non-residual fractions (F1+F2+F3) to element Zn is actually still higher compared to the contribution of F4.

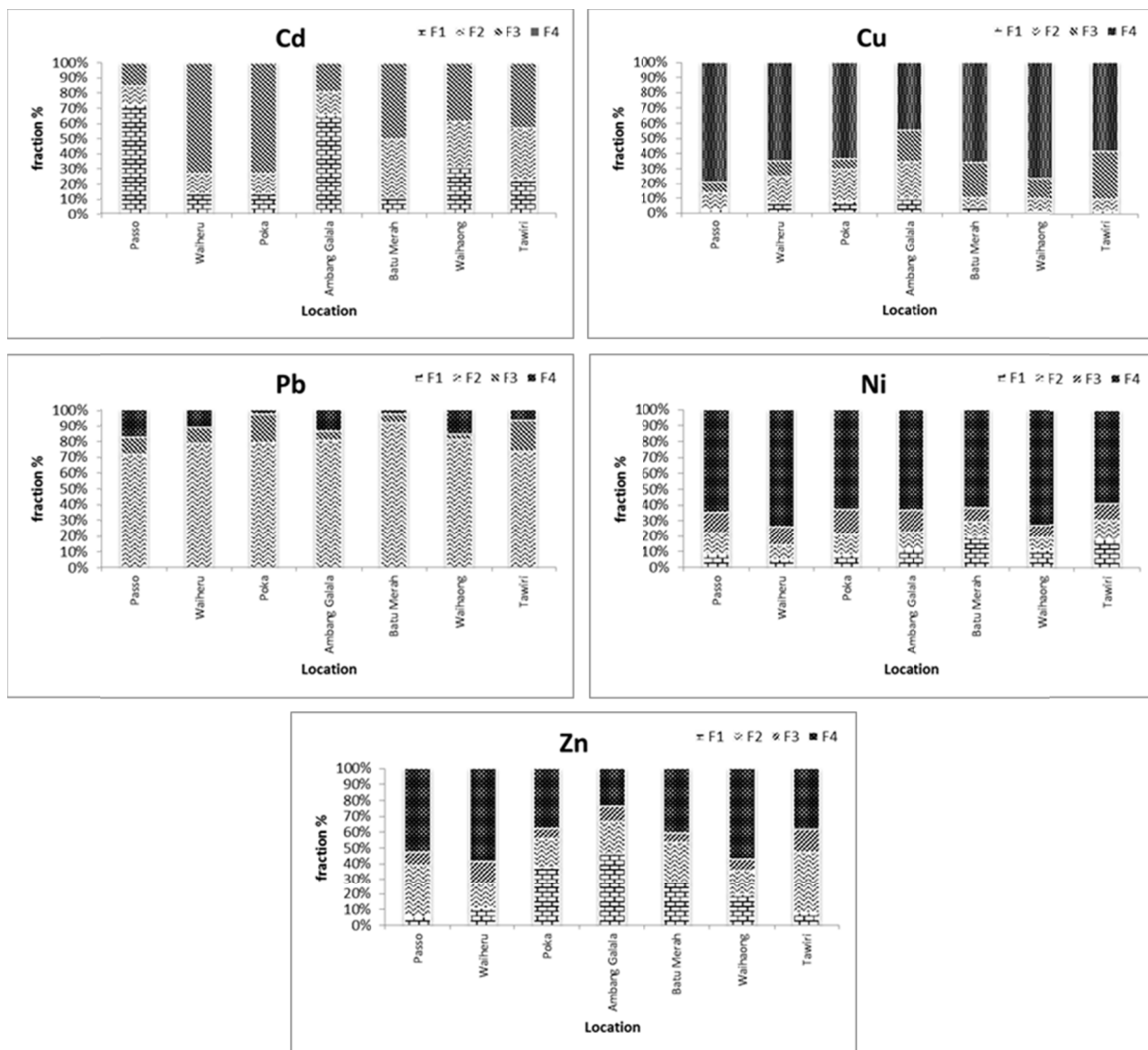


Figure 3. The distribution of metal fractionation in coastal sediment from Ambon Bay

Therefore it can be concluded that cadmium, lead and zinc are mostly accumulated in non-residual fractions meanwhile Cu and Ni were mostly accumulated in residual fraction of total concentration. The studied metals in the non-residual fractions increased in the order of  $Cd > Pb > Zn > Cu > Ni$  indicating they are originated from anthropogenic inputs rather than geochemical backgrounds. The Risk Assessment Code (RAC) reveals that cadmium, copper, nickel and zinc in these area are in fractions that are soluble in acids (exchangeable). In general they are in the low to very high risk category for the environment. There is no Pb in the acid soluble fraction (exchangeable) in the area.

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