CHAPTER 4

CONCLUSION

The effects of extracting solution volume per sediment weight ratios and extraction times were studied for four extraction steps in order to obtain the best extraction efficiency for the proposed method. The experimental results showed that the suitable V/m ratios for the optimized sequential extraction method were 32, 32, 40, and 10 mL/g, whereas the optimum extraction times were 1, 3, 5, and 4 h for exchangeable, bound to carbonate, bound to Fe-Mn oxide, and bound to organic matter fractions, respectively. The optimum V/m ratios and extraction times were further applied for the investigation of heavy metal partitioning in sediment samples.

The extraction efficiency of each extraction step of the optimized sequential extraction procedure was investigated by using repetitive extractions for four times. As the number of extractions were employed, it was found that the extracted Mn, Zn, Cu, Pb, and Cd concentrations were extremely high for the first extraction. The extracted Cr concentration was not detected for all fractions. In case of Mn, its concentrations were found only for the first, second, and third extractions for each fraction. The extraction efficiency of Mn concentration obtained from the first extraction was more than 80.0, 86.6, 92.6, and 87.7% for F-I, F-II, F-III, and F-IV, respectively. On the other hand, the extracted Zn, Cu, Pb, and Cd concentrations were only detected for the first extraction as nearly 100%. Only one time extraction was enough to extract the concentrations of Zn, Cu, Pb, and Cd for each extraction

step of F-I to F-IV. Although percentage extraction of Mn was quite low for once extraction, its extraction efficiency was acceptable for extraction method.

For investigating the accuracy of the optimized sequential extraction method, the sum of extractable metal concentrations from five extraction steps was compared with total metal concentration obtained from hot-acid digestion. It was found that percentage recoveries were in the range of 93.6-98.3, 96.1-100.9, 94.1-95.2, 86.8-89.6, and 91.4-98.7% for Mn, Zn, Cu, Pb, and Cd, respectively. The percentage recoveries for the optimized sequential extraction method showed good agreement for all metals.

To evaluate the accuracy of hot-acid digestion method for determining total metal concentration, original sample spiked with standard solution at known concentration was employed. It was found that percentage recoveries were nearly 100 %. To investigate the precision of the optimized sequential extraction method, experimental procedures were performed repeatedly for six replicates for each extraction step of F-I to F-V. It was found that the relative standard deviation of this procedure was lower than 5 %. This indicated that the optimized sequential extraction method provided good precision.

The extractable metal concentrations obtained from the optimized sequential extraction and Tessier's methods were compared. It was found that information of Mn, Zn, Cu, Cd, and Pb distribution in sediments obtained from two procedures were comparable. Using sequential extraction methods, the extracted Mn concentrations were found in all fractions, but they were extremely high for bound to Fe-Mn oxide fraction (F-III). On the other hand, extracted Zn and Pb concentrations were considerably high in the residual fraction (F-V). The extracted Cu concentrations

were high for bound to organic matter fraction (F-IV). To compare the results obtained from two extraction methods, the metal concentrations extracted from sediments were comparable for each extraction step of F-I to F-IV.

For evaluating the metal distribution in different sediment phases, the extracted metal contents obtained from exchangeable (F-I) and bound to carbonate fractions (F-II) have been considered as mobile forms or available fractions. On the other hand, the extracted metal contents obtained from bound to Fe-Mn oxide (F-III) and bound to organic matter fractions (F-IV) have been considered as slowly mobile form, whereas those found in residual fraction (F-V) has been considered as immobile form.

After applying the optimized sequential extraction method to sediment samples collected from the Kwai Noi River at Kanchanaburi Province, it was found that the most of extracted Zn (68.5%), Cd (37.3%), and Pb (53.5%) concentrations were strongly retained in the residual phase. These metals presented in crystal lattices of minerals with strong binding; and consequently, it would not be released easily in the environment. In case of Mn, its high proportion (about 62.4% of total contents) was associated with oxides of iron and manganese. Theoretically, the metals bound to iron and manganese oxides were released if the sediment were subjected to more reducible conditions. However, the extracted metal levels in this stage would be influenced by the efficiency and selectivity of reagents used in previous stages. Therefore, metals found for this fraction might be high if the carbonates were not completely dissolved or low if parts of iron and manganese hydroxides were already extracted. Moderate proportion of the extracted Mn (about 38.4%) was bound to carbonates in sediment. The Mn ions would be released from carbonates into the

environment if conditions became more acidic. A high proportion of Cu (about 61.5% of total contents) was found in the bound to organic matter fraction. The Cu ions could preferentially be bound to organic ligands by complxation. Under oxidizing condition, organic matter could be degraded and its Cu bound could be released. This agreed to the results of many studies which concluded that a high proportion of Cu in sediments might form part of organic matter.

The exchangeable phase represents mobile and bioavailable heavy metal fractions. In this phase, heavy metals have been retained on sediment surfaces by relatively electrostatic interactions and could be released easily into the environment under electrolyte or weak acidic conditions. The presence of heavy metals in this phase where they could be taken by plants from soils and sediments is most hazardous to the ecosystem. It was found that low concentrations of Mn, Zn, and Cd were found in this phase. Although the extractable metal concentrations found in exchangeable phase were considered as mobile form, their low concentrations detected did not danger to the ecosystem.