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Three Digestion Methods to Determine Concentrations of Cu, Zn, Cd, Ni, Pb, Cr, Mn, and Fe in Mangrove Sediments from Sai Keng, Chek Keng, and Sha Tau Kok, Hong Kong

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Heavy metals in sediments have received increasing attention in recent years, partly because of the growing scientific and public awareness of environmental issues, and partly because of the improvement in the analytical techniques for accurately measuring their concentrations. Trace metal determinations in soils and sediments commonly involve some type of mineral acid extraction (wet oxidation) followed by atomic absorption analysis. Wet oxidation is normally carried out by digestion of the sample in a mixture of strong acids such as sulphuric, hydrochloric, hydrofluoric, nitric and perchloric acids (APHA 1995). However, sulphuric acid is not recommended for simultaneous extraction of different metals, especially in sediments rich in alkaline earth metals, because of the formation of insoluble sulphate salts (Berrow and Stein 1983). Recently, hydrofluoric and perchloric acids are not recommended to use especially for routine metal analysis in laboratories (De Groot et al. 1982; Berrow and Stein 1983; Abreu et al. 1996). Hydrofluoric acid requires special attention to handle as it is dangerous and highly corrosive, while perchloric acid always poses a danger of explosion especially when the perchlorate vapor is trapped in the wooden or plasticware of the fume exhausted system (Berrow and Stein 1983; Alloway 1995). Nitric acid has been used either on its own or in combination with hydrochloric acids and proved to be effective (Berrow and Stein 1983). Agemian and Chau (1976) reported that sediment samples digested with either *aqua-regia,* hot concentrated nitric acid or hot concentrated nitric-perchloric acid mixture gave comparable results. Therefore, the use of either concentrated nitric acid alone or nitric-hydrochloric acid mixture is a common digestion practice for metal determination in most accredited routine laboratories in Hong Kong including Government and private testing laboratories. Most of the digestion methods for heavy metal determination were developed for bottom sediments in marine environment or soils in terrestrial ecosystems. When these acid extraction methods are applied to coastal sediments, their completeness, accuracy, precision and reproducibility might be different from those of the marine sediments as their matrices were different and need further examinations.

Previous studies have shown that in addition to bottom sediments, coastal sediments such as mangrove sediments act as sinks for heavy metals (Harbison 1986; Lacerda et al. 1993;Tam and Wong 1996). Mangrove ecosystems, being intertidal, are reduced and anaerobic, rich in organic matter and sulfide. All these properties favor the retention of metals from inland freshwater drainage and tidal flushing (Harbison 1986; Lacerda et al. 1991). The concentrations of metals in mangrove sediments could reflect the degree and sources of anthropogenic pollution as mangroves are close to urban development and affected by human activities (Tam and Wong 1995). However, mangrove ecosystems varied significantly spatially. For instance, the 44 mangrove sites found in Hong Kong had different biological and physico-chemical characteristics (Tam et al. 1997). Some sites were more close to human activities and were seriously polluted while others were more remote and had very little human disturbance. In order

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to have a more complete picture of metal contamination in Hong Kong mangroves, sediments from three mangrove sites at different geographical locations, having different properties and receiving various extent of human disturbance were examined. Sai Keng and Chek Keng are situated in the Eastern side of Hong Kong and are popular sites for holiday visitors, while Sha Tau Kok is located at the northern part of Hong Kong and is close to the newly developed industrialized city, the Shenzhen Special Economic Zone, People's Republic of China. The present study aims to compare the effectiveness and suitability of three acid digestion methods, namely *aqua-regia* heating, block and microwave digestion, for the determination of metals in sediments collected from these three mangrove sites in Hong Kong. The first two methods were open digestion while the later was a closed system. Copper, zinc, manganese, nickel, chromium, cadmium, lead and iron were measured in the present work as these metals are commonly found in industrial effluent and other anthropogenic source of contamination. Four Standard Reference Materials of sediments were used for quality control in this study.

MATERIALS AND METHODS

Duplicate samples of surface sediments (0-3 cm) were collected from three locations of each mangrove site in Hong Kong. A total of three different sites in the New Territories of Hong Kong, namely Sai Keng (SK), Chek Keng (CK) and Sha Tau Kok (STK), was investigated. Immediately after collection, sediments were air-dried at room temperature (20 ± 2 °C) in the laboratory for two weeks, then ground with a pestle and mortar, and sieved through a 2-mm sieve to remove coarse particles. The sediment samples with diameter less than 2 mm were stored in acid rinsed polyethylene bottles. pH of the samples were measured by stirring 5 g sediment in 10 mL double-deionized water by a pH metre. Total nitrogen concentrations of the sediment samples were determined using Leco CHN-900 Elemental Analyser calibrated with acetanilide. Total Organic Carbon (TOC) was measured according to the method of Walkley and Black (Nelson and Sommers 1982).

Five replicates of each sediment sample were analysed for heavy metals by three digestion methods: microwave digestion (McCarthy and Ellis 1991), *aqua-regia* heating (FAO 1975) and nitric acid block digestion (APHA 1995). The reproducibility of each digestion performed was assessed by calculating the standard deviation of five replicates of each sampling location in each site. To evaluate the accuracy and efficiency of acid digestion method, five replicates of four standard reference materials, namely SRM 1646 (Estuarine sediments from National Bureau of Standard, Washington DC), BCSS-1 (Coastal marine sediments from National Research Council, Canada), PACS-1 (Harbour marine sediments from National Research Council, Canada) and SRM-GBW 07313 (Marine sediments from People's Republic of China), were chosen and analysed by each of the three digestion methods.

For Microwave digestion, air-dried sediments (0.5 g) were digested in concentrated nitric acid (10 mL) in a Microwave Sample Preparation System (MSD-2000, CEM Corporation) equipped with Teflon-coated oven cavity, removable 12-position sample carousels and exhaust fan. Immediately after the acid was added to the Teflon PFA vessels (120 mL capacity, CEM Corp.), the vessels were shaken until all carbon dioxide from the calcium carbonate had escaped. The vessels were then closed tightly (closed vessel digestion) and were capped under pressure using the capping station and disc seals. The vessels were then digested at 95% power with pressure set at 60 psi for 20 minutes. The overall microwave digestion time for one cycle was around 40 minutes. In terms of *aqua-regia* heating, air-dried samples (1 g) were digested in a digestion tube (150 mL) containing 10 mL *aqua-regia* (3 portion of concentrated HCl and 1 portion of concentrated HNO_3). A small glass funnel and a marble were placed at the mouth of the digestion tube to provide a kind of partially closed reflux condition which enhanced the digestion efficiency. The digestion tubes were heated in a boiling water bath for 1 hour. The blocked digestion procedure was similar to *aqua-regia* heating method except 10 mL concentrated nitric acid instead of *aqua-regia* was used, and the digested samples were heated in a block digester with temperature set at 180 °C by a temperature controller. The total digestion time was around 5 hours.

After digestion, the cooled acid digest was filtered into 50 mL volumetric flasks and brought to volume with double deionized water. Concentrations of Cu, Zn, Mn, Ni, Cr, Cd, Pb and Fe in acid digests were determined by the flame atomic absorption spectrophotometer (AA-680, Shimadzu) using a deuterium lamp as the background correction. Working standards were prepared by diluting 1000 mg L^{-1} stock reference solutions to produce concentrations ranging from 0.1 to 4.0 mg L^{-1} and matrix-matched with the same acidity as that in the sample solution (20% v/v HNO_3). The minimum detection limits of the sediment samples for Cu, Zn, Mn, Cd, Pb, Ni, Cr and Fe were 0.43, 0.29, 0.64, 0.48, 4.0, 3.3, 3.5 and 3.0 µg g^{-1}, respectively. Distilled AnalarR® grade nitric acid and Arister® grade hydrochloric acid (BDH) were used. The metal levels in these two acids were determined by digestion the acid blanks (in duplicates) in the same way as the samples for each digestion method. The metal concentrations in all acid blanks were below the detection limits of the conventional flame atomic absorption spectrophotometry (FAAS). All metal concentrations were expressed in µg g^{-1} air-dried weight of the sediments.

The efficiency between three digestion methods and the variability between sampling sites were evaluated by two way analysis of variance statistical test. Duncan’s multiple range test was conducted if significant differences were found between three digestion methods and/or the sampling sites at 95% confidence level. The relationships between three digestion methods for each metal were determined by simple linear regression analyses.

RESULTS AND DISCUSSION

The concentrations of lead and cadmium in all mangrove sediments were below the detection limits of the atomic absorption spectrophotometry. Among the three digestion methods, microwave digestion technique was slightly more efficient in extracting Zn and Ni from mangrove sediments collected from three sampling sites than the other two methods (Table 1). There was no significant difference between three methods in terms of extracting Cu, Fe and Mn. Similar results had been reported by previous researchers. Kruse (1986) found that microwave digestion showed a comparable result as *aqua-regia* digestion in determining heavy metal concentrations in sewage sludge and industrial wastewater. Nieuwenhuize et al. (199 1) also reported that microwave digestion gave the same or slightly higher results of seven metals (Cd, Cr, Cu, Fe, Mn, Pb and Zn) in sediments, soils and suspended matter than conventional reflux using *aqua-regia*. On the contrary, higher concentration of Cr was extracted by *aqua-regia* heating method (Table 1). Chromium can exist in a number of oxidation states, and the most stable and common forms are Cr(VI) and Cr(III). These two forms have sharply contrasting chemical properties, and Cr(VI) is more toxic and readily extracted from sediment particles whereas Cr(III) is less mobile and adsorbed tightly to siliceous materials (Alloway 1995). The digestion power of *aqua-regia* is arisen from the reactivity of nitrosyl chloride (NOCl) and/or free chlorine formed in freshly prepared *aqua-regia* solution, thus it is a better extracting agent for the refractory metal such as chromium.

Table 1. Concentrations of Cu, Zn, Mn, Ni, Cr (μg g^{-1} air-dried weight) and Fe (% dried weight) in sediments collected from three mangrove sites extracted by three digestion methods.

Mangrove Sites	Digestion Methods	Cu	Zn	Mn	Ni	Cr	Fe
Sai Keng (SK)	Aqua (A)	2.23^{a*} ± 0.3	23.6^{a} ± 2.4	65.8^{a} ± 8.6	9.99^{a} ± 2.2	20.6^{a} ± 1.2	1.11^{a} ± 0.1
	Block (B)	2.49^{a} ± 0.5 (5)	29.9^{a} ± 3.1	82.3^{b} ± 9.9	15.9^{b} ± 1.4 (10)	10.8^{b} ± 1.3	1.35^{ab} ± 0.2
	Micro (M)	ND	33.5^{a} ± 3.8	82.1^{b} ± 10.1	36.8^{c} ± 4.2 (10)	ND	1.02^{a} ± 0.1
Chek Keng (CK)	Aqua (A)	2.65^{a} ± 0.3 (10)	29.8^{a} ± 1.2	232.1^{a} ± 22.4	12.0^{a} ± 1.0	ND	1.82^{a} ± 0.1
	Block (B)	2.69^{a} ± 0.6 (5)	38.7^{b} ± 1.7	224.9^{a} ± 18.0	7.03^{b} ± 1.0	ND	2.04^{a} ± 0.3
	Micro (M)	2.12^{a} ± 0.4 (10)	42.3^{c} ± 3.4	212.9^{a} ± 14.2	21.7^{c} ± 2.7	ND	1.94^{a} ± 0.2
Sha Tau Kok (STK)	Aqua (A)	8.59^{a} ± 0.7	31.8^{a} ± 2.3	93.9^{a} ± 5.3	9.05^{a} ± 1.5 (10)	4.32^{a} ± 0.8 (9)	1.11^{a} ± 0.1
	Block (B)	9.98^{b} ± 0.7	27.9^{b} ± 1.2	94.2^{a} ± 4.4	7.99^{a} ± 1.1 (10)	6.92^{b} ± 1.2	1.11^{a} ± 0.1
	Micro (M)	9.76^{b} ± 0.9	45.7^{c} ± 4.3	82.1^{a} ± 7.1	20.9^{b} ± 2.8 (10)	ND	1.20^{a} ± 0.1
Result of 2-way ANOVA	between digestion method	A=B=M	M>B>A	A=B=M	M>A>B	A>B>M	A=B=M
	between sites	STK>SK=CK	NS	CK>STK>SK	SK>CK>STK	SK>STK>CK	CK>SK=STK

mean ± standard deviation of 15 measurements are shown except those specified in the parentheses because the metal concentration in these measurement were below detection limit; ND: not detected; *values with the same letter within the same column of the same site do not differ significantly at $P \leq 0.05$ according to two-way ANOVA followed by Duncan's multiple range test; NS: not significant at $P \leq 0.05$ level; Concentrations of Cd and Pb were not shown as their values were below the detection limits, and metal concentrations in acid blanks were also below the detection limits of FAAS in all cases.

The relationships between digestion methods for each metals were summarized in Table 2. The slope and intercept values were close to 1 and 0 respectively, implying an excellent agreement between two digestion methods. The coefficient correlation value approached to one also indicates significant correlation between two methods. Strong correlations were found between *aqua-regia* and the other two methods in determining Cu, Mn and Fe content (Table 2). A slightly lower correlation coefficient value was recorded between *aqua-regia* and block digestion methods in extracting Cr (r = 0.77). The incomplete dissolution of Cr-rich minerals such as chromite ($FeCr_2O_4$) or chlorite (Cr-rich clay mineral) in block or microwave digestion using only nitric acid as the oxidant (Sinex et al. 1980) might explain the poor correlation between these two digestion methods in determining Cr. In addition, the low detection limit of Cr by the flame atomic absorption spectrophotometry might also contribute to such poor correlation.

Table 2. Relationships between three digestion methods according to linear regression analysis and simple correlation coefficient (r) values.

	Determination range of each digestion method			*Aqua-regia* (A) *vs* Block digestion (B)			*Aqua-regia (A) vs* Microwave digestion (M)			Block (B) *vs* Microwave digestion (M)		
	A	B	M	α_{AB}	β_{AB}	r_{AB}	α_{AM}	β_{AM}	r_{AM}	α_{BM}	β_{BM}	r_{BM}
Cu	0 - 12	0 - 14	0 - 14	1.3	0.7	0.94*	1.3	0.7	0.87*	0.3	0.9	0.95*
Zn	18 - 40	21-48	25-53	9.4	0.6	0.65*	2.0	0.6	0.88*	13.8	0.5	0.54*
Mn	40-422	43-408	38-381	-5.9	1.0	0.98*	-8.4	1.1	0.98*	3.2	1.0	0.99*
Ni	0 - 15	0 - 20	0 - 41	5.1	0.5	0.54*	7.5	0.1	0.36	-0.7	0.4	0.93*
Cr	0 - 24	0 - 14	ND	-6.4	2.2	0.77*	-	-	-	-	-	-
Fe	0.6 - 3.5	0.7 - 3.6	0.7 - 2.8	-0.1	0.9	0.96*	-0.1	1.1	0.84*	0.02	1.1	0.86*

n=45; ND: not detected; *: significant at $P \leq 0.001$ level; α and β are the intercept and slope of the regression line between two digestion methods, respectively; Determination ranges were in $\mu g\ g^{-1}$ dried weight for all metals except Fe which was in % dried weight.

The concentrations of heavy metals in sediments varied significantly between sampling sites (Table 1). Sediments collected from Sha Tau Kok had significantly higher concentrations of Cu than the other two sites. The geographical location of Sha Tau Kok is near Shenzhen, a newly developed city and receives more discharge from industrial sources. When compared with the other two site, Sha Tau Kok sediments had a relatively higher percentage (43%) of the particles in the fine grain fraction (<63 µm) and higher organic matter content (1.1%) which favored the accumulation of metals in sediments. On the other hand, Chek Keng, being more remote and less disturbed by human activities, had lower content of Cu, Ni and Cr but higher content of Mn and Fe. These differences in metal concentrations might also be related to variations in other chemical properties between sites. Chek Keng had a significantly higher pH (5.34) than Sha Tau Kok (5.19) and Sai Keng (4.71), and lower total organic carbon and nitrogen concentrations (0.63% TOC and 0.034% N) than the other two sites (1.06% TOC and 0.079% N in Sha Tau Kok; 1.11% TOC and 0.07% N in Sai Keng). Despite the spatial variations between sites, the metal concentrations recorded in the present study were generally comparable to or even lower than the values reported by previous workers on mangrove sediments (Harbison 1986; Lacerda et al. 1993; Tam and Wong 1995). The mangrove sediments from these three sites were belonged to the uncontaminated sediments (Class A) according to the classification of sediments based on metal concentrations by the Hong Kong Environmental Protection Department (EPD 1992), which concluded the Class A sediments should have Cu, Zn, Cr, Ni, Cd and Pb content lower than 10, 70, 25, 15, 0.1 and 25 µg g^{-1} dried weight, respectively.

The accuracy of the three digestion methods was evaluated by comparing the heavy metal concentrations obtained from the analysis with the certified values of four SRMs having different sediment matrix and different concentrations. Almost all the eight investigated elements exhibited satisfactory yields with respective to the certified values upon the three digestion methods, with recoveries higher than 70% in most case, except for Cr and Pb (Table 3). These recoveries percentages were lower than those reported by previous workers on SRM1646 (Mahan et al. 1987) and PACS-1 (Nakashima et al. 1988) as the certified values of these two SRMs are based on the results of determinations by a comprehensive digestion using a combination of mineral acid with HF (Mahan et al. 1987; Nakashima et al. 1988; Alloway 1995), therefore, it is reasonable to obtain results with lower percentage yield using the present digestion methods.

Differences between the four SRM were not obvious in most cases although a slightly higher recovery was observed in GBW07313 which had a lower TOC concentration (0.32%) than that of the PACS-1(4.08%) and BCSS-1 (2.06%). In GBW07313 SRM, their percentage recoveries were greater than 90% in most metals especially for *aqua-regia* and microwave digestion methods except Pb and Cr. The low percentage of Pb recovery was attributed to the fact that some Pb is held in aluminosilicate such as the potassium feldspars, and is moderately resistant to acid dissolution (Berrow and Stein 1983). The recovery of Cr was found to be relatively low, probably related to the fact that chromium, among all metals, is mostly retained by siliceous materials. Lacerda and his co-worker (1991) examined the accumulation of chromium in a mangrove ecosystem and found that chromium was likely buried in mangrove sediment due to the formation of immobilized refractory Cr-organic compound. Abreu et al. (1996) using *aqua-regia* also recorded a low recovery percentage (<50%) for Cr in the pond sediment certified reference material (NIES-CRM) as the digestion procedure proposed by the NIES-CRM was based on complete sample dissolution in HP (De Groot et al. 1982). It has been suggested that wet digestions excluding HP do not attack the aluminosilicate matrix nor destruct the metalliferous minerals in the sediment (De Groot et al. 1982). Therefore extraction using nitric acid might only achieve partial destruction as the siliceous bound metals will not be released, thus resulting a low recovery percentage. The recovery of Zn was lower in PACS-1 sediment than the other three SRM as the former SRM had significantly higher certified Zn content. These results suggest that the native concentrations and matrix of the sediments did affect the digestion efficiency to some extent.

The precision of three digestion methods was comparable and was satisfactory. The standard deviation values of the five replicates for each digestion method were small for all metals except Pb and Cd (Table 3). The large standard deviation (with reference to the mean) values of Cd and Pb might be related to the low sensitivity of the atomic absorption spectrophotometric measurement to these two metals, and the instrumental error became more obvious under this

Table 3. Comparison of the effectiveness of three digestion methods based on recovery percentages of four SRM

Digestion Methods	Cu	Zn	Mn	Ni	Cr	Cd	Pb	Fe
SRM 1646, Estuarine sediment, USA								
Certified value (μg g^{-1})	18 ± 3	138 ± 6	375 ± 20	32 ± 3	76 ± 3	0.36 ± 0.07	28.2 ± 1.8	33500 ± 1000
Aqua-regia recovery (%)	87^{a} (0.8)	77^{a} (0.5)	63^{a} (1.2)	60^{a} (3.7)	40^{a} (0.2)	ND	68^{a} (2.0)	78^{a} (0.9)
Block recovery (%)	84^{a} (0.9)	75^{a} (0.8)	61^{a} (2.9)	63^{a} (2.0)	37^{a} (0.7)	ND	51^{b} (1.5)	79^{a} (2.5)
Microwave recovery (%)	89^{a} (0.9)	77^{a} (3.8)	65^{a} (2.7)	73^{b} (3.8)	36^{a} (1.7)	ND	44^{c} (5.5)	87^{b} (5.1)
BCSS-1, Coastal sediment, Canada								
Certified value (μg g^{-1})	18.5 ± 2.7	119 ± 12	299 ± 15	55.3 ± 3.6	123 ± 14	0.25 ± 0.04	22.7 ± 3.4	32900 ± 980
Aqua-regia recovery (%)	82^{a} (3.2)	82^{a} (2.1)	61^{a} (0.9)	85^{a} (0.8)	29^{a} (0.1)	ND	82^{a} (2.7)	83^{a} (0.6)
Block recovery (%)	79^{a} (1.3)	76^{a} (1.0)	54^{b} (1.4)	78^{a} (3.7)	25^{b} (0.5)	ND	56^{b} (9.8)	81^{a} (0.6)
Microwave recovery (%)	80^{a} (3.9)	76^{a} (2.4)	63^{a} (0.7)	86^{a} (4.2)	21^{c} (0.1)	ND	42^{c} (8.8)	88^{b} (1.1)
PACS-1, Harbour sediment, Canada								
Certified value (μg g^{-1})	452 ± 16	824 ± 22	470 ± 12	44.1 ± 2.0	113 ± 8	2.38 ± 0.20	404 ± 20	48720 ± 840
Aqua-regia recovery (%)	96^{a} (2.1)	49^{a} (2.4)	60^{a} (0.8)	79^{a} (3.3)	44^{a} (3.2)	98^{a} (6.4)	95^{a} (5.3)	78^{a} (2.0)
Block recovery (%)	87^{b} (0.8)	33^{b} (1.0)	57^{a} (1.9)	65^{b} (0.9)	42^{a} (0.1)	ND	67^{b} (1.4)	70^{ab} (1.0)
Microwave recovery (%)	86^{b} (0.9)	65^{c} (1.6)	56^{a} (1.0)	68^{b} (4.0)	39^{a} (0.7)	102^{a} (6.4)	77^{c} (2.8)	80^{a} (0.2)
GBW 07313, Marine sediment, PRC								
Certified value (μg g^{-1})	424 ± 8	160 ± 3	3330 ± 78	150 ± 4	58.4 ± 1.3	NF	29.3 ± 1.1	43820 ± 490
Aqua-regia recovery (%)	92^{a} (1.0)	82^{a} (0.6)	91^{a} (0.2)	122^{a} (0.6)	37^{a} (0.7)	ND	62^{a} (4.1)	88^{a} (1.0)
Block recovery (%)	86^{b} (0.6)	80^{a} (0.1)	89^{a} (2.8)	80^{b} (1.7)	31^{b} (0.6)	ND	50^{b} (5.9)	84^{a} (1.0)
Microwave recovery (%)	94^{a} (1.2)	80^{a} (0.3)	93^{a} (2.4)	90^{c} (0.7)	29^{b} (0.3)	ND	48^{b} (6.8	99^{b} (1.5)

NF: not found in this SRM; ND: not detected; Mean recovery % and the standard deviation (in parentheses) of five replicates are shown; Values with the same letter within the same column of the same SRM do not differ significantly at $P < 0.05$; Metal concentrations in acid blanks were below the detection limits of FAAS in all cases.

situation (Real et al. 1994). In addition, if the sediments were heterogenous or if the amounts of heavy metals present were low, the variability between replicates would be higher. This explains why the variations found in mangrove sediments were generally larger than that in SRM. In summary, the accuracy and reproducibility of the digestion methods examined in the present study were acceptable.

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