CHAPTER 3

RESULTS AND DISCUSSION

3.1 Effect of Ratio of Extracting Solution Volume per Sediment Weight

In this work, metal concentrations extracted with increasing volume of extracting solution were used to investigate the extraction efficiency for each extraction step. The effects of the ratio of extracting solution volume per sediment weight (V/m) on metal solubilization were particularly studied for four fractions including exchangeable (F-I), bound to carbonates (F-II), bound to Fe-Mn oxides (F-III), and bound to organic matter (F-IV). For investigating the effect of V/m ratios, sediment samples (S2 and S5) collected for Lot-I was employed. The experimental procedures for the optimization of V/m ratios were absolutely described in Section 2.5.1. The effects of V/m ratio on metal extraction for each extraction step are reported as follows.

For F-I, the volumes of extracting solution (1 M $MgCl_2$, pH 7) were varied in the range of 8 to 64 mL/g. The effects of V/m ratios on metal solubilization are shown in Table 3.1 and 3.2 for Mn and Zn, respectively. Among selected metals, only Mn and Zn concentrations were detected, whereas the extracted Cu, Cd, Pb, and Cr concentrations were not detected for this fraction. It was found that the extracted Mn and Zn concentrations were gradually increased with volumes of extracting solution. The extracted Mn and Zn concentrations from two sediments were plotted as the function of extracting solution volumes as shown in Figure 3.1(a) and 3.1(b),

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respectively. This indicates that the extracted Mn and Zn concentrations were comparable for V/m ratios at 32 to 64 mL/g. The suitable V/m ratio for F-I was therefore chosen at 32 mL/g that is larger than proposed by Tessier *et al.* (8 mL/g). The optimized sequential extraction method required four-time V/m ratios larger than the one of Tessier's method.

For F-II, the volumes of extracting solution (1 M NaOAc/HOAc at pH 5) were varied in the range of 8 to 64 mL/g. The effects of V/m ratios on Mn and Zn solubilizations are shown in Table 3.3 and 3.4, respectively. Both extracted Mn and Zn concentrations obtained from two sediments were high at 32 mL of extracting solution. Other metals were not detected for this fraction. The variations of Mn and Zn concentrations extracted after extraction process for F-II are shown in Figure 3.2 (a), and 3.2(b), respectively. It was found that the variations of Mn and Zn concentrations extracted from both samples were comparable at 32 to 64 mL/g of V/m ratios. The V/m ratio at 32 mL/g was therefore chosen for bound to carbonate fraction that is larger than proposed by Tessier *et al.* (8 mL/g). The optimized sequential extraction method required four-time V/m ratios larger than that of Tessier's method.

For F-III, the volumes of extracting solution (0.04 M NH_2OH•HCl in 25% v/v HOAc) were varied in the range of 10 to 80 mL/g. The results of the extractable Mn, Zn, and Cu concentrations for each extraction V/m ratio are shown in Table 3.5, 3.6, and 3.7, respectively. The Cd, Pb, and Cr concentrations were not detected for this fraction. The results indicate that the extracted Mn, Zn, and Cu concentrations were gradually increased with increasing of extracting solution volumes as can be seen in Figure 3.3(a), 3.3(b), and 3.3(c) for Mn, Zn, and Cu, respectively. It was

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found that the extracted Mn, Zn, and Cu concentrations obtained from both sediments were comparable for 40 to 80 mL/g of V/m ratios. A 40 mL of selected extracting solution was therefore chosen for this fraction. The optimized sequential extraction method required two-time V/m ratios larger than that of Tessier's method.

For F-IV, the volumes of 30% w/w H_2O_2 adjusted to pH 2 with HNO_3 were varied in the range of 10 to 80 mL/g. For both samples, the effects of V/m ratios on the extractable Mn, Zn, and, Cu concentrations are shown in Table 3.8, 3.9, and 3.10, respectively. The variations of Mn, Zn, and Cu concentrations are shown in Figure 3.4(a), 3.4(b), and 3.4(c), respectively. It was found that the extracted Mn, Zn, and Cu concentrations were comparable at 10 to 80 mL/g of V/m ratios. A 10 mL of 30% w/w acidified hydrogen peroxide was therefore chosen for this fraction. The optimized sequential extraction method required the same V/m ratio as used in Tessier's method.

The optimized sequential extraction method required larger extracting solution volumes than Tessier's method for all steps except F-IV. This indicates that V/m ratio for each extraction step proposed by Tessier *et al.* might not completely extract heavy metals from river sediment. Rauret *et al.* [47] reported that V/m ratio for different samples influenced on metal partitioning because metals presented in samples could be bound with many substances. They proposed the suitable V/m ratios for river sediment were larger than Tessier's method, for example, 50 mL/g for F-II, 50 mL/g for F-III, and 50 mL/g for F-IV.

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Table 3.1 Effect of V/m ratios on the extracted Mn concentrations from S2 and S5 (Lot-I) for F-I.

V/m (mL/g)	n	Mn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
8	1	0.054	0.571	56.7	59.4	3.2	0.079	1.082	107.3	102.6	4.9
	2	0.057	0.633	63.0			0.074	0.980	97.5		
	3	0.055	0.592	58.6			0.077	1.041	103.1		
16	1	0.056	0.612	60.9	61.0	1.9	0.082	1.143	113.7	113.8	6.0
	2	0.055	0.592	59.1			0.079	1.082	107.9		
	3	0.057	0.633	63.0			0.085	1.204	119.9		
24	1	0.056	0.612	61.0	65.5	4.0	0.091	1.327	132.0	127.4	4.9
	2	0.059	0.673	67.1			0.089	1.286	128.0		
	3	0.06	0.694	68.4			0.086	1.224	122.3		
32	1	0.066	0.816	81.4	78.6	5.0	0.092	1.347	134.3	138.7	4.0
	2	0.066	0.816	81.6			0.097	1.449	142.0		
	3	0.062	0.735	72.9			0.095	1.408	139.7		
40	1	0.064	0.776	77.0	80.5	4.4	0.096	1.429	136.4	138.6	7.2
	2	0.065	0.796	79.1			0.094	1.388	132.7		
	3	0.068	0.857	85.4			0.101	1.531	146.6		
48	1	0.065	0.796	79.0	81.8	2.4	0.095	1.408	132.8	139.9	6.2
	2	0.067	0.837	83.2			0.098	1.469	143.3		
	3	0.067	0.837	83.0			0.099	1.490	143.7		
56	1	0.068	0.857	84.9	82.7	2.8	0.095	1.408	138.1	137.2	8.5
	2	0.067	0.837	83.6			0.098	1.469	145.3		
	3	0.065	0.796	79.6			0.094	1.388	128.4		
64	1	0.068	0.857	85.0	83.1	5.1	0.095	1.408	128.2	136.5	7.5
	2	0.064	0.776	77.3			0.099	1.490	142.8		
	3	0.069	0.878	86.9			0.096	1.429	138.7		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 100 mL before analysis.

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Table 3.2 Effect of V/m ratios on the extracted Zn concentrations from S2 and S5 (Lot-I) for F-I.

V/m (mL/g)	Zn										
	n	S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
8	1	0.006	0.012	1.2	1.2	0.0	0.006	0.012	1.2	1.1	0.1
	2	0.006	0.012	1.2			0.005	0.008	0.8		
	3	0.006	0.012	1.2			0.006	0.012	1.2		
16	1	0.007	0.016	1.6	1.5	0.1	0.006	0.012	1.2	1.2	0.1
	2	0.007	0.016	1.6			0.006	0.012	1.2		
	3	0.006	0.012	1.2			0.006	0.012	1.2		
24	1	0.007	0.016	1.6	1.5	0.1	0.007	0.016	1.6	1.6	0.0
	2	0.006	0.012	1.2			0.007	0.016	1.6		
	3	0.007	0.016	1.6			0.007	0.016	1.6		
32	1	0.008	0.020	2.0	1.9	0.1	0.008	0.020	2.0	1.9	0.1
	2	0.007	0.016	1.6			0.007	0.016	1.6		
	3	0.008	0.020	2.0			0.008	0.020	2.0		
40	1	0.007	0.016	1.6	1.9	0.1	0.008	0.020	2.0	2.0	0.0
	2	0.008	0.020	2.0			0.008	0.020	2.0		
	3	0.008	0.020	2.0			0.008	0.020	2.0		
48	1	0.008	0.020	2.0	1.9	0.1	0.008	0.020	2.0	2.0	0.0
	2	0.007	0.016	1.6			0.008	0.020	2.0		
	3	0.008	0.020	2.0			0.008	0.020	2.0		
56	1	0.008	0.020	2.0	2.0	0.0	0.009	0.024	2.4	1.9	0.1
	2	0.008	0.020	2.0			0.007	0.016	1.6		
	3	0.008	0.020	2.0			0.007	0.016	1.6		
64	1	0.008	0.020	2.0	2.0	0.0	0.008	0.020	2.0	2.0	0.0
	2	0.008	0.020	2.0			0.008	0.020	2.0		
	3	0.008	0.020	2.0			0.008	0.020	2.0		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 100 mL before analysis.

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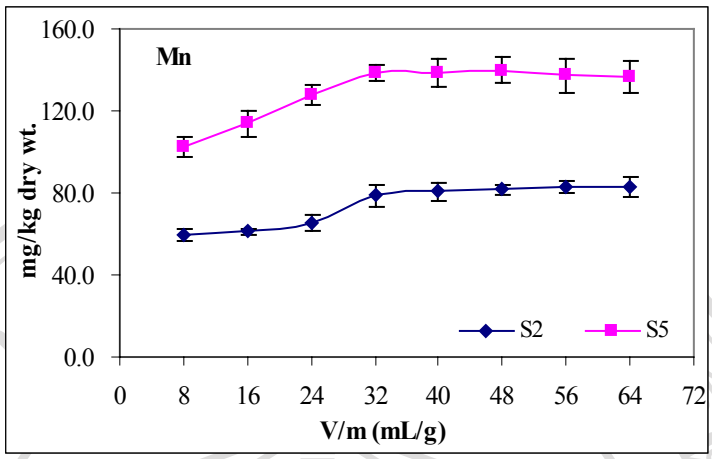
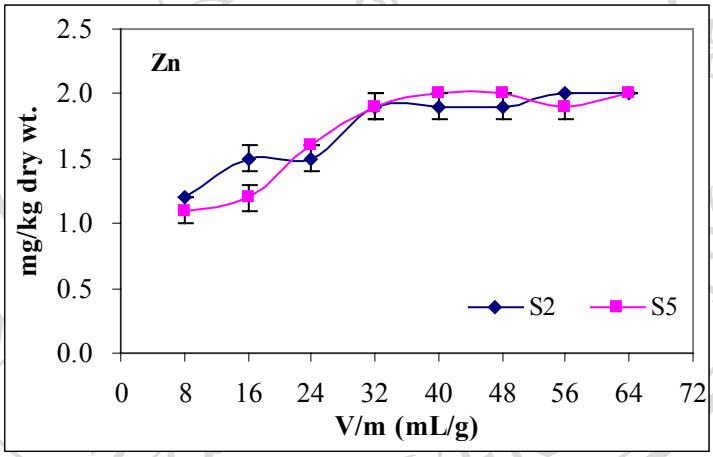


(a)



(b)

Figure 3.1 Effects of V/m ratios on the extracted Mn (a) and Zn (b) concentrations from S2 (Lot-I) and S5 (Lot-I) for F-I.

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Table 3.3 Effect of V/m ratios on the extracted Mn concentrations from S2 and S5 (Lot-I) for F-II.

V/m (mL/g)	n	Mn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
8	1	0.085	1.204	119.4	114.1	7.4	0.087	1.245	123.5	122.9	4.8
	2	0.078	1.061	105.6			0.084	1.184	117.8		
	3	0.084	1.184	117.3			0.089	1.286	127.4		
16	1	0.086	1.224	121.8	121.3	3.3	0.089	1.286	127.9	131.4	3.2
	2	0.087	1.245	124.3			0.091	1.327	132.4		
	3	0.084	1.184	117.9			0.092	1.347	134.1		
24	1	0.091	1.327	132.1	133.6	6.5	0.096	1.429	142.2	141.0	1.1
	2	0.089	1.286	128.0			0.095	1.408	140.2		
	3	0.096	1.429	140.8			0.095	1.408	140.6		
32	1	0.099	1.490	148.6	149.8	9.7	0.100	1.510	150.5	151.1	8.6
	2	0.095	1.408	140.7			0.096	1.429	142.8		
	3	0.105	1.612	160.0			0.105	1.612	160.0		
40	1	0.102	1.551	154.0	154.3	3.9	0.102	1.551	154.4	151.0	4.1
	2	0.104	1.592	158.3			0.101	1.531	152.2		
	3	0.100	1.510	150.6			0.098	1.469	146.4		
48	1	0.103	1.571	156.1	152.1	4.1	0.098	1.469	146.1	150.1	5.3
	2	0.101	1.531	152.3			0.099	1.490	148.2		
	3	0.099	1.490	147.8			0.103	1.571	156.0		
56	1	0.102	1.551	153.7	155.9	2.8	0.106	1.633	161.7	152.4	8.1
	2	0.104	1.592	159.0			0.099	1.490	148.8		
	3	0.102	1.551	155.0			0.098	1.469	146.8		
64	1	0.105	1.612	159.9	155.1	6.4	0.099	1.490	147.7	152.6	9.6
	2	0.099	1.484	147.9			0.098	1.469	146.4		
	3	0.104	1.592	157.7			0.107	1.653	163.7		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 100 mL before analysis.

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Table 3.4 Effect of V/m ratios on the extracted Zn concentrations from S2 and S5 (Lot-I) for F-II.

V/m (mL/g)	n	Zn									
		S-2					S-5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
8	1	0.015	0.049	4.9	4.9	0.4	0.013	0.041	4.1	3.9	0.2
	2	0.014	0.045	4.5			0.012	0.037	3.7		
	3	0.016	0.054	5.3			0.013	0.041	4.1		
16	1	0.016	0.054	5.3	5.3	0.4	0.013	0.041	4.1	4.2	0.2
	2	0.017	0.058	5.7			0.013	0.041	4.1		
	3	0.015	0.049	4.9			0.014	0.045	4.5		
24	1	0.017	0.058	5.7	5.6	0.6	0.014	0.045	4.5	4.5	0.0
	2	0.018	0.062	6.2			0.014	0.045	4.5		
	3	0.015	0.049	4.9			0.014	0.045	4.5		
32	1	0.018	0.062	6.2	6.3	0.2	0.015	0.049	4.9	5.1	0.2
	2	0.018	0.062	6.2			0.015	0.049	4.9		
	3	0.019	0.066	6.5			0.016	0.054	5.3		
40	1	0.018	0.062	6.1	6.2	0.0	0.015	0.049	4.9	4.9	0.0
	2	0.018	0.062	6.1			0.015	0.049	4.9		
	3	0.018	0.062	6.2			0.015	0.049	4.9		
48	1	0.019	0.066	6.5	6.1	0.4	0.015	0.049	4.9	5.0	0.2
	2	0.018	0.062	6.2			0.015	0.049	4.9		
	3	0.017	0.058	5.7			0.016	0.054	5.3		
56	1	0.018	0.062	6.1	6.0	0.6	0.016	0.054	5.3	5.1	0.2
	2	0.016	0.054	5.3			0.015	0.049	4.9		
	3	0.019	0.066	6.5			0.015	0.049	4.9		
64	1	0.018	0.062	6.1	6.0	0.2	0.016	0.054	5.3	5.2	0.2
	2	0.017	0.058	5.7			0.015	0.049	4.9		
	3	0.018	0.062	6.1			0.016	0.054	5.3		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 100 mL before analysis.

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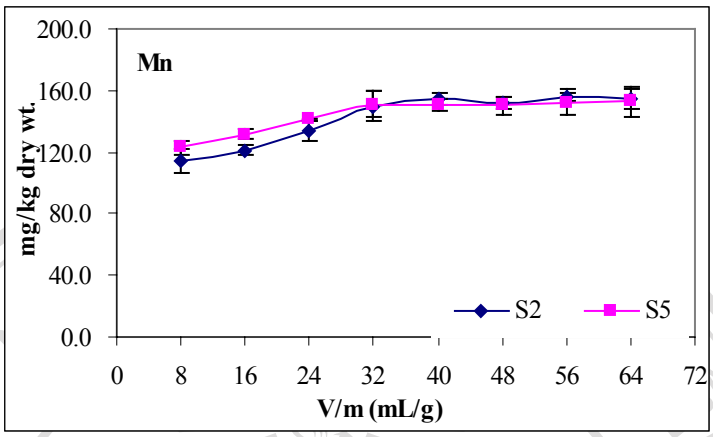
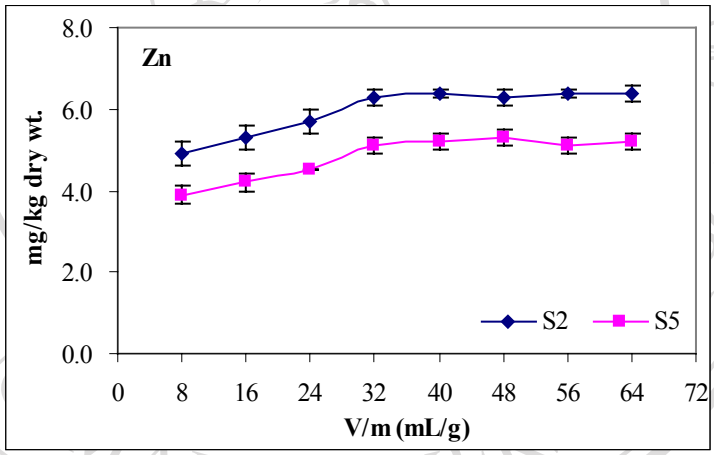


(a)



(b)

Figure 3.2 Effects of V/m ratios on the extracted Mn (a) and Zn (b) concentrations from S2 (Lot-I) and S5 (Lot-I) for F-II.

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Table 3.5 Effect of V/m ratios on the extracted Mn concentrations from S2 and S5 (Lot-I) for F-III.

V/m (mL/g)	n	Mn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
10	1	0.124	2.419	239.8	235.3	7.8	0.124	2.419	239.9	241.5	8.9
	2	0.117	2.273	226.3			0.129	2.523	251.1		
	3	0.124	2.419	239.7			0.121	2.356	233.5		
20	1	0.126	2.460	244.7	251.4	6.4	0.144	2.835	282.0	282.4	10.7
	2	0.129	2.523	252.0			0.149	2.940	293.3		
	3	0.132	2.585	257.5			0.139	2.731	271.9		
30	1	0.130	2.544	253.3	255.9	6.3	0.149	2.940	292.6	288.8	5.7
	2	0.129	2.523	251.3			0.144	2.835	282.3		
	3	0.136	2.669	263.0			0.148	2.919	291.4		
40	1	0.134	2.627	262.0	262.4	8.4	0.154	3.044	303.4	306.6	8.7
	2	0.130	2.544	254.2			0.152	3.002	300.0		
	3	0.139	2.731	271.0			0.161	3.190	316.5		
50	1	0.138	2.710	269.1	262.0	6.8	0.155	3.065	305.0	302.9	9.8
	2	0.134	2.627	261.2			0.149	2.940	292.3		
	3	0.131	2.565	255.7			0.158	3.127	311.5		
60	1	0.139	2.731	271.2	263.0	8.4	0.154	3.044	302.6	306.7	10.7
	2	0.135	2.648	263.4			0.152	3.002	298.6		
	3	0.131	2.565	254.5			0.162	3.210	318.8		
70	1	0.138	2.710	268.5	260.3	8.2	0.161	3.190	315.8	306.6	8.3
	2	0.133	2.606	260.3			0.152	3.002	299.8		
	3	0.129	2.523	252.2			0.154	3.044	304.0		
80	1	0.136	2.669	264.6	265.0	11.6	0.159	3.148	312.0	302.1	8.6
	2	0.130	2.544	253.5			0.151	2.981	297.1		
	3	0.142	2.794	276.8			0.152	3.002	297.3		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 100 mL before analysis.

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Table 3.6 Effect of V/m ratios on the extracted Zn concentrations from S2 and S5 (Lot-I) for F-III.

V/m (mL/g)	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
10	1	0.017	0.054	5.3	5.5	0.2	0.018	0.058	5.8	5.9	0.2
	2	0.017	0.054	5.3			0.018	0.058	5.8		
	3	0.018	0.058	5.8			0.019	0.063	6.2		
20	1	0.022	0.076	7.5	7.3	0.3	0.020	0.067	6.7	6.4	0.2
	2	0.021	0.071	7.1			0.019	0.063	6.2		
	3	0.021	0.071	7.1			0.019	0.063	6.2		
30	1	0.023	0.080	8.0	7.7	0.5	0.021	0.071	7.1	6.8	0.3
	2	0.023	0.080	8.0			0.020	0.067	6.7		
	3	0.021	0.071	7.1			0.020	0.067	6.7		
40	1	0.024	0.085	8.5	8.6	0.2	0.021	0.071	7.1	7.3	0.2
	2	0.024	0.085	8.5			0.021	0.071	7.1		
	3	0.025	0.089	8.9			0.022	0.076	7.5		
50	1	0.025	0.089	8.9	8.9	0.5	0.021	0.071	7.1	7.4	0.3
	2	0.024	0.085	8.4			0.022	0.076	7.5		
	3	0.026	0.094	9.3			0.022	0.076	7.6		
60	1	0.025	0.089	8.8	9.0	0.3	0.021	0.071	7.1	7.4	0.3
	2	0.025	0.089	8.9			0.022	0.076	7.5		
	3	0.026	0.094	9.3			0.022	0.076	7.5		
70	1	0.025	0.089	8.8	8.9	0.4	0.021	0.071	7.1	7.6	0.5
	2	0.024	0.085	8.5			0.023	0.080	8.0		
	3	0.026	0.094	9.3			0.022	0.076	7.6		
80	1	0.025	0.089	8.9	9.0	0.2	0.022	0.076	7.5	7.5	0.0
	2	0.025	0.089	8.9			0.022	0.076	7.6		
	3	0.026	0.094	9.3			0.022	0.076	7.5		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 100 mL before analysis.

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Table 3.7 Effect of V/m ratios on the extracted Cu concentrations from S2 and S5 (Lot-I) for F-III.

V/m (mL/g)	n	Cu									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
10	1	0.004	0.033	3.3	3.2	0.0	0.004	0.033	3.2	3.2	0.0
	2	0.004	0.033	3.2			0.004	0.033	3.3		
	3	0.004	0.033	3.2			0.004	0.033	3.2		
20	1	0.005	0.043	4.2	3.6	0.2	0.005	0.043	4.3	3.6	0.3
	2	0.004	0.033	3.2			0.004	0.033	3.2		
	3	0.004	0.033	3.2			0.004	0.033	3.2		
30	1	0.005	0.043	4.2	3.9	0.3	0.005	0.043	4.3	3.9	0.2
	2	0.004	0.033	3.3			0.005	0.043	4.2		
	3	0.005	0.043	4.3			0.004	0.033	3.2		
40	1	0.005	0.043	4.2	4.9	0.1	0.005	0.043	4.3	4.3	0.0
	2	0.006	0.053	5.3			0.005	0.043	4.3		
	3	0.006	0.053	5.3			0.005	0.043	4.3		
50	1	0.006	0.053	5.3	4.9	0.2	0.005	0.043	4.2	4.3	0.1
	2	0.006	0.053	5.3			0.004	0.033	3.3		
	3	0.005	0.043	4.3			0.006	0.053	5.3		
60	1	0.006	0.053	5.2	4.6	0.0	0.004	0.033	3.3	4.3	0.3
	2	0.005	0.043	4.2			0.005	0.043	4.3		
	3	0.005	0.043	4.3			0.006	0.053	5.3		
70	1	0.006	0.053	5.2	4.9	0.3	0.006	0.053	5.3	4.6	0.2
	2	0.005	0.043	4.3			0.005	0.043	4.2		
	3	0.006	0.053	5.3			0.005	0.043	4.3		
80	1	0.006	0.053	5.2	4.9	0.2	0.005	0.043	4.3	4.6	0.2
	2	0.006	0.053	5.3			0.005	0.043	4.3		
	3	0.005	0.043	4.3			0.006	0.053	5.3		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 100 mL before analysis.

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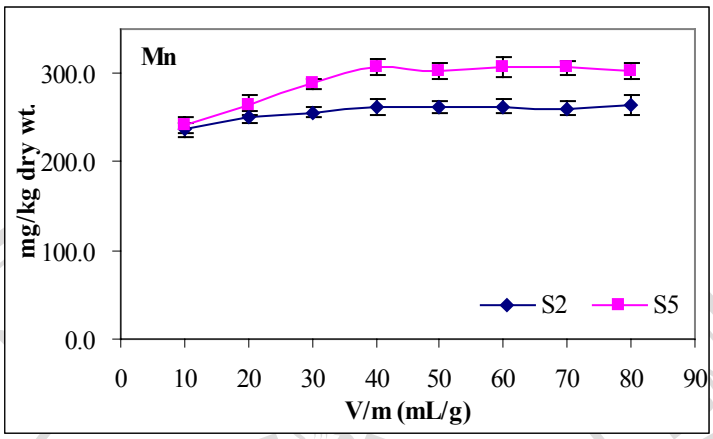

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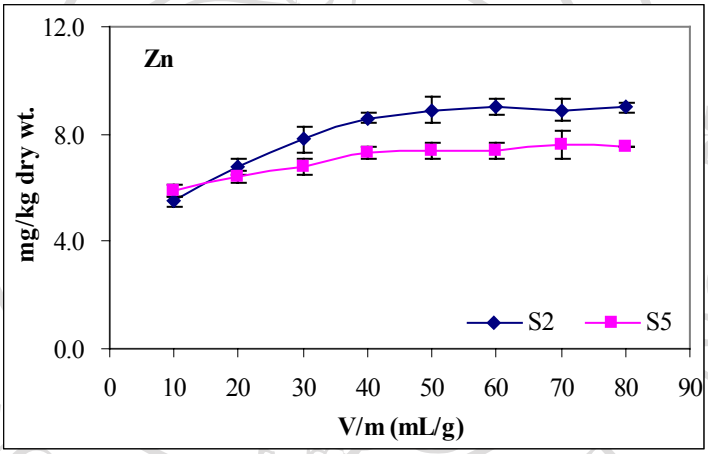

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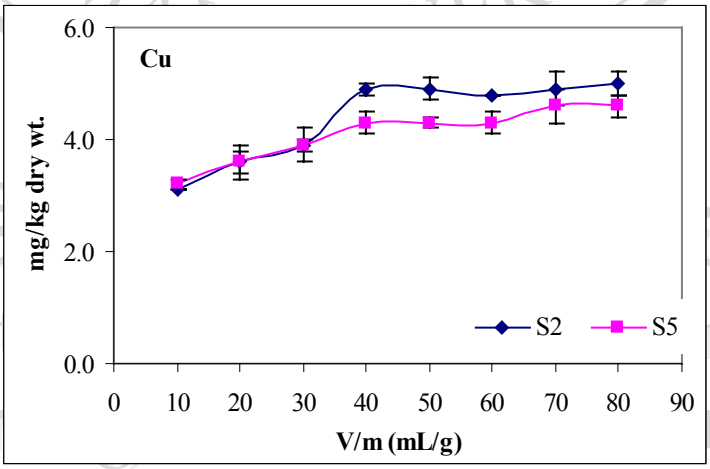

(c)

Figure 3.3 Effect of V/m ratios on the extracted Mn (a), Zn (b), and Cu (c) concentrations from S2 (Lot-I) and S5 (Lot-I) for F-III.

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Table 3.8 Effect of V/m ratios on the extracted Mn concentrations from S2 and S5 (Lot-I) for F-IV.

V/m (mL/g)	n	Mn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
10	1	0.023	0.315	31.2	31.2	2.0	0.031	0.481	47.7	47.8	2.0
	2	0.022	0.294	29.2			0.030	0.460	45.8		
	3	0.024	0.335	33.2			0.032	0.502	49.8		
20	1	0.024	0.335	33.4	31.3	2.0	0.031	0.481	47.9	47.2	1.1
	2	0.022	0.294	29.3			0.030	0.460	45.9		
	3	0.023	0.315	31.3			0.031	0.481	47.9		
30	1	0.022	0.294	29.2	31.9	3.0	0.032	0.502	50.0	49.3	1.1
	2	0.023	0.315	31.3			0.032	0.502	50.0		
	3	0.025	0.356	35.1			0.031	0.481	48.1		
40	1	0.023	0.315	31.4	30.6	3.1	0.032	0.502	50.0	48.6	1.2
	2	0.021	0.273	27.3			0.031	0.481	48.1		
	3	0.024	0.335	33.3			0.031	0.481	47.8		
50	1	0.024	0.335	33.3	31.3	3.5	0.031	0.481	47.9	47.9	2.1
	2	0.024	0.335	33.4			0.030	0.460	45.8		
	3	0.021	0.273	27.2			0.032	0.502	50.0		
60	1	0.022	0.294	29.2	30.6	4.3	0.031	0.481	47.8	49.2	1.2
	2	0.025	0.356	35.4			0.032	0.502	49.9		
	3	0.021	0.273	27.1			0.032	0.502	49.9		
70	1	0.025	0.356	35.3	31.3	4.0	0.032	0.502	49.7	49.3	3.1
	2	0.021	0.273	27.3			0.030	0.460	46.0		
	3	0.023	0.315	31.4			0.033	0.523	52.2		
80	1	0.022	0.294	29.1	30.5	2.3	0.031	0.481	47.7	47.8	1.9
	2	0.022	0.294	29.3			0.030	0.460	45.9		
	3	0.024	0.335	33.2			0.032	0.502	49.7		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes the of extracts were prepared as 100 mL before analysis.

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Table 3.9 Effect of V/m ratios on the extracted Zn concentrations from S2 and S5 (Lot-I) for F-IV.

V/m (mL/g)	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
10	1	0.017	0.054	5.3	5.3	0.4	0.015	0.045	4.4	4.3	0.2
	2	0.016	0.049	4.9			0.014	0.040	4.0		
	3	0.018	0.058	5.8			0.015	0.045	4.4		
20	1	0.018	0.058	5.8	5.5	0.3	0.015	0.045	4.4	4.3	0.3
	2	0.017	0.054	5.3			0.015	0.045	4.5		
	3	0.017	0.054	5.3			0.014	0.040	4.0		
30	1	0.016	0.049	4.9	5.3	0.5	0.015	0.045	4.4	4.4	0.0
	2	0.017	0.054	5.3			0.015	0.045	4.4		
	3	0.018	0.058	5.8			0.015	0.045	4.4		
40	1	0.017	0.054	5.3	5.6	0.3	0.014	0.040	4.0	4.4	0.4
	2	0.018	0.058	5.8			0.015	0.045	4.5		
	3	0.018	0.058	5.8			0.016	0.049	4.9		
50	1	0.018	0.058	5.8	5.5	0.2	0.015	0.045	4.4	4.6	0.3
	2	0.017	0.054	5.3			0.016	0.049	4.9		
	3	0.017	0.054	5.3			0.015	0.045	4.4		
60	1	0.017	0.054	5.3	5.5	0.3	0.016	0.049	4.9	4.7	0.3
	2	0.018	0.058	5.8			0.015	0.045	4.4		
	3	0.017	0.054	5.3			0.016	0.049	4.9		
70	1	0.018	0.058	5.7	5.3	0.4	0.015	0.045	4.4	4.7	0.3
	2	0.017	0.054	5.4			0.016	0.049	4.9		
	3	0.016	0.049	4.9			0.016	0.049	4.9		
80	1	0.017	0.054	5.3	5.3	0.0	0.015	0.045	4.4	4.6	0.2
	2	0.017	0.054	5.3			0.015	0.045	4.4		
	3	0.017	0.054	5.3			0.016	0.049	4.9		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 100 mL before analysis.

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Table 3.10 Effect of V/m ratios on the extracted Cu concentrations from S2 and S5 (Lot-I) for F-IV.

V/m (mL/g)	n	Cu									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
10	1	0.011	0.104	9.8	9.0	0.0	0.010	0.094	8.9	9.4	0.4
	2	0.010	0.094	8.9			0.010	0.094	8.9		
	3	0.009	0.083	8.3			0.011	0.104	10.4		
20	1	0.010	0.094	9.2	9.2	0.2	0.011	0.104	10.1	10.4	0.1
	2	0.011	0.104	9.7			0.012	0.114	10.6		
	3	0.010	0.094	8.7			0.012	0.114	10.5		
30	1	0.011	0.104	10.2	10.0	0.1	0.011	0.104	10.2	9.7	0.3
	2	0.011	0.104	9.7			0.011	0.104	9.8		
	3	0.011	0.104	10.0			0.010	0.094	9.0		
40	1	0.011	0.104	10.3	9.6	0.3	0.010	0.094	9.3	8.7	0.3
	2	0.010	0.094	9.2			0.009	0.083	8.1		
	3	0.010	0.094	9.3			0.010	0.094	8.6		
50	1	0.012	0.114	11.4	10.2	0.5	0.012	0.114	11.4	9.9	0.6
	2	0.011	0.104	9.8			0.010	0.094	8.8		
	3	0.009	0.083	9.3			0.010	0.094	9.4		
60	1	0.009	0.083	9.1	9.8	0.3	0.009	0.083	8.1	8.5	0.7
	2	0.011	0.104	10.0			0.009	0.083	7.3		
	3	0.011	0.104	10.2			0.011	0.104	10.2		
70	1	0.011	0.104	10.1	9.8	0.3	0.012	0.114	11.0	10.1	0.5
	2	0.011	0.104	10.3			0.011	0.104	10.3		
	3	0.010	0.094	9.1			0.010	0.094	9.1		
80	1	0.010	0.094	9.1	9.9	0.4	0.011	0.104	10.0	9.9	0.1
	2	0.011	0.104	10.0			0.010	0.094	10.0		
	3	0.012	0.114	10.7			0.010	0.094	9.8		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 100 mL before analysis.

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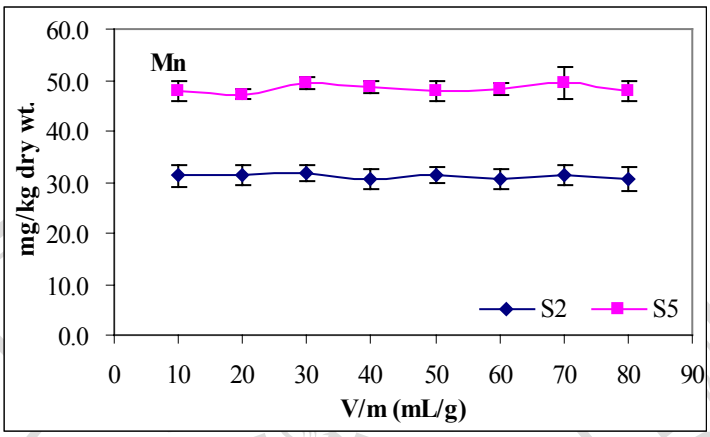
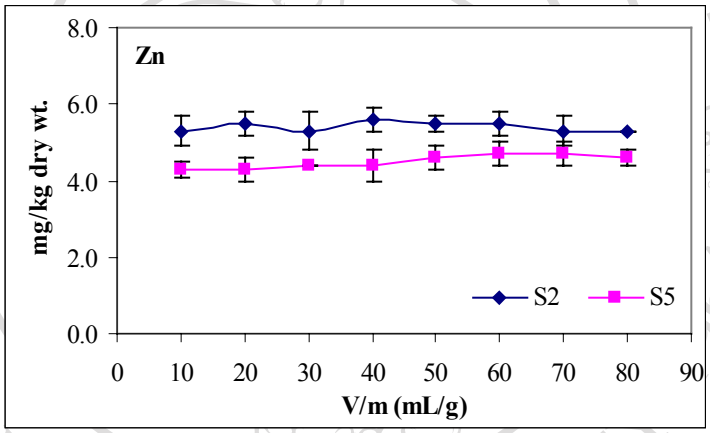


(a)



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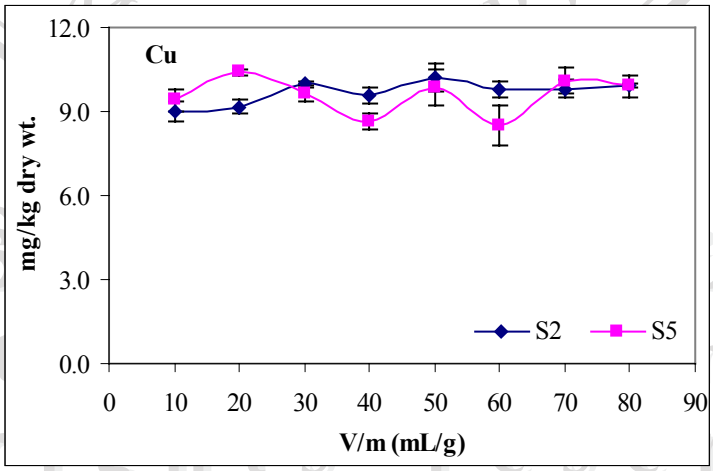

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Figure 3.4 Effects of V/m ratios on the extracted Mn (a), Zn (b), and Cu (c) concentrations from S2 (Lot-I) and S5 (Lot-I) for F-IV.

3.2 Effect of Extraction Times

The aim of this experiment was to examine the extraction time for each step of sequential extraction method. Equilibrium extraction time in each step for different types of sample could be different. The leaching time required for each extraction step depends upon such parameters as particle size, percentage and type of carbonate components, and sample weight [22]. For instance, the coarse bottom sediment with high carbonate contents required longer extraction time for bound to carbonate fraction [20].

In the work, optimal equilibrium time for each extraction step was investigated prior to the determination of metal concentrations using sequential extraction method. The optimal V/m ratios were also used to investigate the optimum extraction times. Although Tessier's sequential extraction method has been widely applied to heavy metal determinations from solid samples, the effects of extraction times were rarely studied in previous works. Two sediment samples (S2 and S5) collected for Lot-I were used to investigate the effect of extraction time for each extraction step of F-I to F-IV. All experimental procedures for the optimization of extraction times for sequential extraction method were described in Section 2.5.2. The extracted solution collected for each extraction time was determined. The effects of extraction times on the extractable metals from S2 (Lot-I) and S5 (Lot-I) for each extraction step are shown as below.

For F-I, both sediment samples were extracted with 32 mL of 1 M $MgCl_2$ at pH 7 for with extraction times in the range of 1 to 6 h. For each extraction time studied, the extractable Mn and Zn concentrations obtained from both samples are shown in Table 3.11 and 3.12, respectively. Only extracted Mn and Zn

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concentrations were detected, whereas Cu, Cd, Cr, and Pb were not detected for this fraction. The effects of extraction times for this extraction step on the extractable Mn and Zn concentrations are shown in Figure 3.5 (a) and 3.5 (b), respectively. The results indicate that the extractable Mn and Zn concentrations obtained from both samples reached the maximum values after 1 h of extraction and they were stable for next 5 h. For investigating in Figure 3.5, the extracted Mn and Zn concentrations from both samples were comparable at 1 to 6 h of extraction times. Therefore, extraction time for this step was chosen at 1 h that is the same extraction time as proposed by Tessier's method.

For F-II, Table 3.13 and 3.14 show the results of extractable Mn and Zn concentrations obtained for each extraction time of 1 to 6 h. The extracted Mn and Zn concentrations obtained from both samples were found increasingly with extraction times. On the other hand, the concentrations of Cu, Pb, Cd, and Cr were not detected. As can be seen in Figure 3.6 (a) for Mn and 3.6 (b) for Zn, the extracted Mn and Zn concentrations were maximum values for both samples at 3 h of extraction and were comparable for 3 to 6 h. The optimum extraction time for this fraction was therefore chosen at 3 h, which is shorter than that proposed by Tessier *et al.* (5 h).

For F-III, the effects of extraction times on the extracted Mn, Zn, and Cu concentrations are shown in Table 3.15, 3.16, and 3.17, respectively. The concentrations of Pb, Cd, and Cr were not detected. The results indicate that the extracted Mn, Zn, and Cu concentrations were increased with extraction times. For both samples, the extracted Mn, Zn, and Cu concentrations were maximum values at 5 h of extraction as shown in Figure 3.7(a), 3.7(b), and 3.7(c) for Mn, Zn, and Cu,

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respectively. Therefore, the suitable extraction time for the metal solubilization was 5 h, which is shorter than that proposed by Tessier *et al.* (6 h).

For F-IV, the extracted Mn, Zn, and Cu concentrations for both samples are shown in Table 3.18, 3.19, and 3.20, respectively. For both samples, the results indicate that the extracted Mn, Zn, and Cu concentrations were increased with extraction times. The variations of the extracted Mn, Zn, and Cu concentrations as the function of extraction times are shown in Figure 3.8(a), 3.8(b), and 3.8(c), respectively. The suitable extraction time for the optimized sequential extraction method was selected at 4 h, which is shorter than that proposed by Tessier *et al.* (5 h).

The results of each fraction indicate that extraction times required for F-I, F-II, F-III, and F-IV were at least 1, 3, 5, and 4 h, respectively. Therefore, these duration times of four extraction steps were used throughout this work. It was observed that total extraction time used for the optimized sequential extraction method were less than that of Tessier's method about 4 h, without hot-acid digestion (residual fraction). The influence of extraction time on efficiency of metal extraction for each extraction step has been widely studied for sequential extraction procedures and also applied to microwave-assisted extraction as reported by several workers [31, 52, 53, 58-61].

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Table 3.11 Effect of extraction times on the extracted Mn concentrations from S2 and S5 (Lot-I) for F-I.

T_E (h)	n	Mn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.131	1.539	73.9	74.3	1.1	0.196	2.455	117.8	121.2	5.6
	2	0.127	1.483	73.5			0.191	2.385	118.2		
	3	0.129	1.511	75.6			0.203	2.554	127.7		
2	1	0.132	1.554	73.2	72.8	1.6	0.205	2.582	121.7	125.4	5.3
	2	0.131	1.539	71.0			0.211	2.666	123.0		
	3	0.134	1.582	74.1			0.221	2.807	131.5		
3	1	0.126	1.469	69.7	73.5	3.5	0.223	2.835	134.5	136.5	6.7
	2	0.134	1.582	76.1			0.215	2.723	131.0		
	3	0.129	1.511	75.3			0.227	2.892	144.0		
4	1	0.127	1.483	71.0	73.6	3.0	0.201	2.525	121.0	133.1	11.2
	2	0.131	1.539	76.9			0.225	2.863	143.0		
	3	0.128	1.497	72.8			0.219	2.779	135.2		
5	1	0.131	1.539	73.3	72.6	3.3	0.226	2.877	137.1	135.7	4.1
	2	0.126	1.469	68.9			0.220	2.793	131.1		
	3	0.132	1.554	75.4			0.225	2.863	138.9		
6	1	0.135	1.596	78.0	75.6	3.6	0.215	2.723	129.7	133.0	4.6
	2	0.128	1.497	74.1			0.220	2.793	131.1		
	3	0.130	1.525	74.6			0.224	2.849	138.2		

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.12 Effect of extraction times on the extracted Zn concentrations from S2 and S5 (Lot-I) for F-I.

T_E (h)	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.017	0.033	1.6	1.5	0.1	0.013	0.023	1.1	1.2	0.1
	2	0.015	0.028	1.4			0.014	0.026	1.3		
	3	0.015	0.028	1.4			0.013	0.023	1.1		
2	1	0.017	0.033	1.6	1.6	0.1	0.014	0.026	1.2	1.2	0.0
	2	0.018	0.036	1.7			0.015	0.028	1.3		
	3	0.016	0.031	1.4			0.014	0.026	1.2		
3	1	0.016	0.031	1.5	1.4	0.1	0.014	0.026	1.2	1.2	0.1
	2	0.016	0.031	1.5			0.013	0.023	1.1		
	3	0.014	0.026	1.3			0.013	0.023	1.1		
4	1	0.015	0.028	1.3	1.2	0.0	0.015	0.028	1.3	1.2	0.1
	2	0.013	0.023	1.1			0.013	0.023	1.1		
	3	0.014	0.026	1.3			0.014	0.026	1.3		
5	1	0.016	0.031	1.5	1.4	0.2	0.015	0.028	1.3	1.3	0.0
	2	0.015	0.028	1.3			0.015	0.028	1.3		
	3	0.016	0.031	1.5			0.015	0.028	1.4		
6	1	0.015	0.028	1.4	1.4	0.0	0.015	0.028	1.3	1.3	0.1
	2	0.015	0.028	1.4			0.016	0.031	1.4		
	3	0.015	0.028	1.4			0.013	0.023	1.1		

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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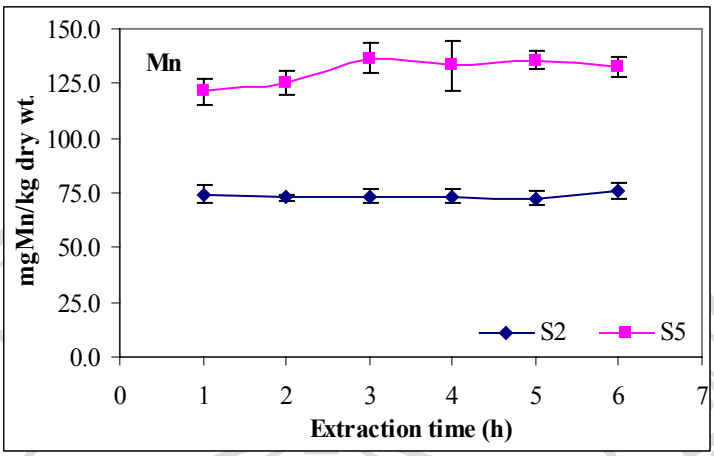
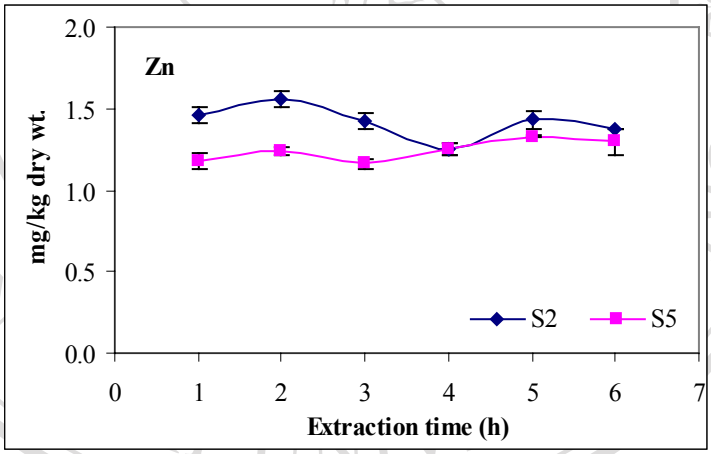


(a)



(b)

Figure 3.5 Effects of extraction times on the extracted Mn (a) and Zn (b) concentrations from S2 (Lot-I) and S5 (Lot-I) for F-I.

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Table 3.13 Effect of extraction times on the extracted Mn concentrations from S2 and S5 (Lot-I) for F-II.

T_E (h)	n	Mn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.196	2.455	113.0	118.7	5.2	0.189	2.356	108.4	112.9	4.6
	2	0.204	2.568	123.1			0.196	2.455	117.7		
	3	0.203	2.554	120.0			0.192	2.399	112.7		
2	1	0.208	2.624	120.1	124.6	7.7	0.205	2.582	127.9	130.3	2.6
	2	0.212	2.680	133.5			0.207	2.610	130.0		
	3	0.204	2.568	120.3			0.212	2.680	133.1		
3	1	0.236	3.018	149.6	148.9	3.0	0.224	2.849	141.2	145.8	4.8
	2	0.230	2.934	145.5			0.230	2.934	145.5		
	3	0.239	3.061	151.5			0.238	3.046	150.8		
4	1	0.241	3.089	151.6	152.8	3.0	0.228	2.906	142.6	146.5	3.5
	2	0.236	3.018	150.7			0.234	2.990	149.3		
	3	0.245	3.145	156.3			0.241	3.089	147.6		
5	1	0.246	3.159	152.8	154.0	1.7	0.242	3.103	150.1	148.8	2.1
	2	0.241	3.089	153.4			0.236	3.018	149.9		
	3	0.249	3.201	155.9			0.235	3.004	146.3		
6	1	0.246	3.159	157.2	155.8	3.2	0.239	3.061	152.3	150.2	5.2
	2	0.242	3.103	152.1			0.245	3.145	154.1		
	3	0.251	3.230	158.0			0.231	2.948	144.3		

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.14 Effect of extraction times on the extracted Zn concentrations from S2 and S5 (Lot-I) for F-II.

T_E (h)	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.024	0.088	4.1	4.0	0.3	0.027	0.098	4.5	4.6	0.3
	2	0.021	0.079	3.8			0.029	0.104	5.0		
	3	0.025	0.091	4.3			0.026	0.094	4.4		
2	1	0.031	0.110	5.0	5.6	0.6	0.034	0.119	5.5	6.1	0.6
	2	0.036	0.126	6.3			0.037	0.129	6.4		
	3	0.034	0.119	5.6			0.040	0.138	6.5		
3	1	0.036	0.126	6.2	6.4	0.1	0.042	0.144	7.2	7.0	0.4
	2	0.037	0.129	6.4			0.038	0.132	6.5		
	3	0.038	0.132	6.5			0.043	0.148	7.3		
4	1	0.035	0.123	6.0	6.3	0.1	0.044	0.151	7.4	7.0	0.5
	2	0.039	0.135	6.7			0.041	0.141	7.0		
	3	0.037	0.129	6.1			0.039	0.135	6.4		
5	1	0.040	0.138	6.7	6.5	0.4	0.044	0.151	7.3	7.1	0.3
	2	0.038	0.132	6.5			0.042	0.144	7.2		
	3	0.037	0.129	6.3			0.040	0.138	6.7		
6	1	0.038	0.132	6.6	6.5	0.2	0.043	0.148	7.3	7.2	0.4
	2	0.037	0.129	6.3			0.040	0.138	6.8		
	3	0.039	0.135	6.6			0.045	0.154	7.5		

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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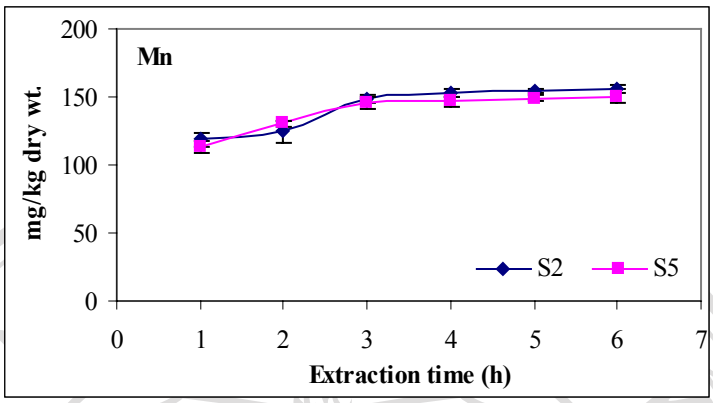
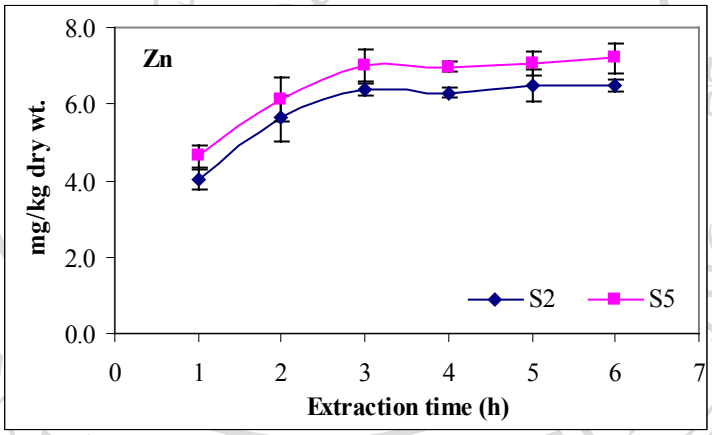


(a)



(b)

Figure 3.6 Effects of extraction times on the extracted Mn (a) and Zn (b) concentrations from S2 (Lot-I) and S5 (Lot-I) for F-II.

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Table 3.15 Effect of extraction times on the extracted Mn concentrations from S2 and S5 (Lot-I) for F-III.

T_E (h)	n	Mn									
		S2*					S5*				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.104	1.159	115.5	116.4	4.1	0.129	1.511	145.9	136.5	8.1
	2	0.108	1.215	120.8			0.124	1.441	132.6		
	3	0.102	1.131	112.8			0.115	1.314	131.1		
2	1	0.112	1.272	126.9	132.7	5.5	0.146	1.751	160.3	173.2	11.2
	2	0.117	1.342	133.3			0.157	1.906	178.3		
	3	0.121	1.399	137.9			0.152	1.835	180.9		
3	1	0.158	1.920	187.1	191.6	4.6	0.187	2.328	226.9	232.6	5.5
	2	0.164	2.004	191.5			0.195	2.441	233.2		
	3	0.168	2.061	196.2			0.199	2.497	237.8		
4	1	0.204	2.568	254.8	257.5	2.9	0.224	2.849	282.8	272.8	11.3
	2	0.216	2.737	260.6			0.216	2.737	260.6		
	3	0.208	2.624	257.1			0.221	2.807	275.0		
5	1	0.221	2.807	272.3	273.9	4.5	0.246	3.159	306.5	302.3	11.0
	2	0.217	2.751	270.4			0.231	2.948	289.8		
	3	0.226	2.877	279.1			0.249	3.201	310.5		
6	1	0.219	2.779	270.9	276.4	5.8	0.249	3.201	312.1	305.5	5.7
	2	0.224	2.849	282.5			0.238	3.046	302.1		
	3	0.218	2.765	275.7			0.237	3.032	302.4		
7	1	0.219	2.779	269.6	269.4	7.1	0.248	3.187	309.2	307.8	6.3
	2	0.211	2.666	262.1			0.239	3.061	300.9		
	3	0.224	2.849	276.4			0.251	3.230	313.2		
8	1	0.224	2.849	277.8	272.1	6.0	0.245	3.145	312.7	304.3	7.5
	2	0.212	2.680	265.7			0.238	3.046	302.1		
	3	0.216	2.737	272.8			0.234	2.990	298.1		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL.

* The extracts were further diluted by 2-fold before analysis.

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Table 3.16 Effect of extraction times on the extracted Zn concentrations from S2 and S5 (Lot-I) for F-III.

T_E (h)	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.016	0.063	3.0	3.3	0.2	0.019	0.073	3.5	3.4	0.1
	2	0.019	0.073	3.3			0.019	0.073	3.3		
	3	0.018	0.069	3.5			0.018	0.069	3.5		
2	1	0.025	0.091	4.2	4.2	0.0	0.028	0.101	4.6	4.8	0.4
	2	0.024	0.088	4.1			0.026	0.094	4.6		
	3	0.023	0.085	4.2			0.030	0.107	5.3		
3	1	0.036	0.126	6.1	6.5	0.5	0.030	0.107	5.2	5.4	0.3
	2	0.038	0.132	6.3			0.034	0.119	5.7		
	3	0.043	0.148	7.0			0.032	0.113	5.4		
4	1	0.043	0.148	7.3	7.5	0.3	0.038	0.132	6.5	6.8	0.2
	2	0.045	0.154	7.3			0.042	0.144	6.9		
	3	0.047	0.160	7.8			0.041	0.141	6.9		
5	1	0.051	0.173	8.4	8.3	0.2	0.044	0.151	7.3	7.5	0.5
	2	0.048	0.163	8.0			0.048	0.163	8.0		
	3	0.051	0.173	8.4			0.043	0.148	7.2		
6	1	0.052	0.176	8.6	8.2	0.4	0.043	0.148	7.2	7.5	0.3
	2	0.049	0.166	8.2			0.046	0.157	7.8		
	3	0.046	0.157	7.8			0.045	0.154	7.7		
7	1	0.051	0.173	8.4	8.2	0.3	0.044	0.151	7.3	7.4	0.3
	2	0.047	0.160	7.9			0.046	0.157	7.7		
	3	0.051	0.173	8.4			0.043	0.148	7.2		
8	1	0.049	0.166	8.1	8.3	0.4	0.046	0.157	7.6	7.5	0.1
	2	0.047	0.160	7.9			0.044	0.151	7.5		
	3	0.052	0.176	8.8			0.044	0.151	7.5		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.17 Effect of extraction times on the extracted Cu concentrations from S2 and S5 (Lot-I) for F-III.

T_E (h)	n	Cu									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.008	0.038	1.8	2.0	0.1	0.010	0.044	2.1	2.1	0.2
	2	0.009	0.041	1.9			0.009	0.041	1.9		
	3	0.010	0.044	2.2			0.011	0.048	2.4		
2	1	0.010	0.044	2.0	2.2	0.1	0.015	0.060	2.7	2.6	0.1
	2	0.012	0.051	2.4			0.013	0.054	2.5		
	3	0.011	0.048	2.4			0.012	0.051	2.5		
3	1	0.016	0.063	3.1	3.0	0.1	0.018	0.069	3.4	3.4	0.0
	2	0.015	0.060	2.9			0.018	0.069	3.3		
	3	0.016	0.063	3.0			0.019	0.072	3.4		
4	1	0.012	0.051	2.5	2.9	0.1	0.022	0.082	4.1	3.8	0.1
	2	0.016	0.063	3.0			0.021	0.078	3.7		
	3	0.016	0.063	3.1			0.019	0.072	3.5		
5	1	0.022	0.082	4.0	4.2	0.1	0.022	0.082	4.0	4.0	0.0
	2	0.024	0.088	4.3			0.023	0.085	4.2		
	3	0.025	0.091	4.4			0.022	0.082	4.0		
6	1	0.023	0.085	4.1	4.2	0.1	0.020	0.075	3.6	3.9	0.1
	2	0.024	0.088	4.4			0.023	0.085	4.2		
	3	0.022	0.082	4.1			0.021	0.078	3.9		
7	1	0.025	0.091	4.4	4.4	0.2	0.020	0.075	3.6	3.8	0.1
	2	0.026	0.094	4.6			0.021	0.078	3.8		
	3	0.023	0.085	4.1			0.022	0.082	4.0		
8	1	0.024	0.088	4.4	4.4	0.1	0.023	0.075	4.1	3.9	0.1
	2	0.026	0.094	4.7			0.021	0.078	3.9		
	3	0.023	0.085	4.2			0.019	0.072	3.6		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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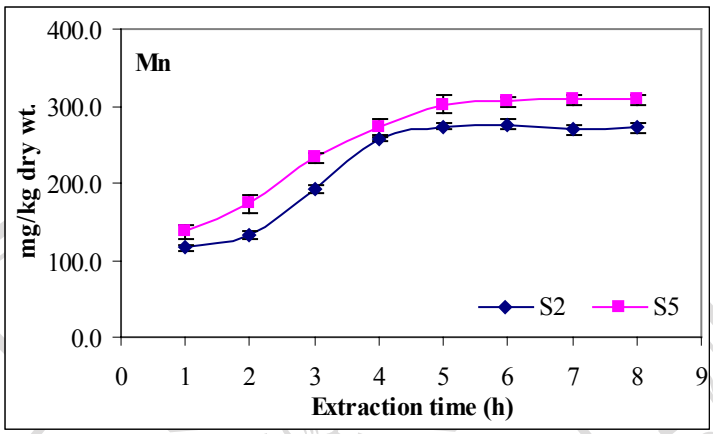

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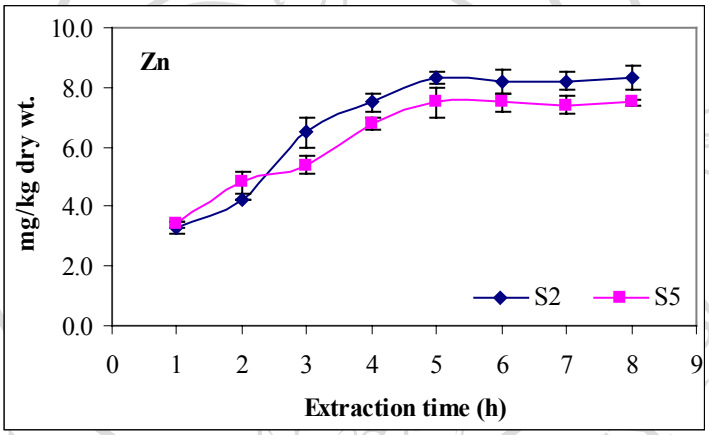

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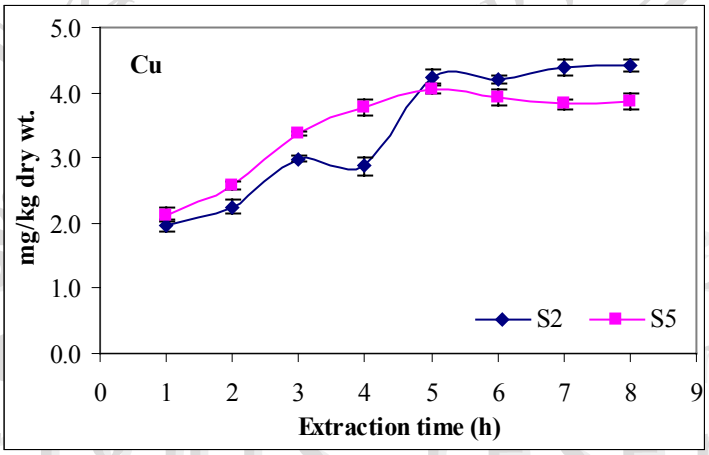

(c)

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Figure 3.7 Effects of extraction times on the extracted Mn (a), Zn (b), and Cu (c) concentrations from S2 (Lot-I) and S5 (Lot-I) for F-III.

Table 3.18 Effect of extraction times on the extracted Mn concentrations from S2 and S5 (Lot-I) for F-IV.

T_E (h)	n	Mn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.051	0.413	20.5	20.3	1.0	0.059	0.525	25.9	26.6	1.2
	2	0.049	0.385	19.2			0.061	0.554	27.2		
	3	0.052	0.427	21.2			0.058	0.511	25.5		
2	1	0.055	0.469	23.4	24.5	1.0	0.062	0.568	28.1	30.5	2.2
	2	0.057	0.497	24.8			0.067	0.638	31.5		
	3	0.058	0.511	25.4			0.068	0.652	32.1		
3	1	0.063	0.582	28.9	29.2	1.0	0.078	0.793	39.0	39.3	1.7
	2	0.062	0.568	28.3			0.076	0.765	37.7		
	3	0.065	0.610	30.3			0.081	0.835	41.1		
4	1	0.069	0.666	33.1	31.5	1.7	0.089	0.948	46.6	47.2	1.1
	2	0.064	0.596	29.7			0.088	0.934	46.5		
	3	0.067	0.638	31.7			0.091	0.976	48.5		
5	1	0.069	0.666	33.1	31.6	2.3	0.089	0.948	47.1	48.4	1.5
	2	0.069	0.666	32.7			0.091	0.976	48.0		
	3	0.063	0.582	29.0			0.093	1.004	50.0		
6	1	0.065	0.610	30.3	31.7	1.4	0.091	0.976	48.0	47.8	2.5
	2	0.067	0.638	31.8			0.094	1.018	50.2		
	3	0.069	0.666	33.1			0.087	0.920	45.2		
7	1	0.070	0.680	33.8	32.2	2.2	0.090	0.962	47.3	47.6	1.0
	2	0.064	0.596	29.7			0.089	0.948	46.8		
	3	0.069	0.666	33.1			0.092	0.990	48.7		
8	1	0.068	0.652	32.4	32.2	0.4	0.094	1.018	50.1	48.1	1.8
	2	0.067	0.638	31.8			0.090	0.962	47.5		
	3	0.068	0.652	32.4			0.089	0.948	46.6		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.19 Effects of extraction times on the extracted Zn concentrations from S2 and S5 (Lot-I) for F-IV.

T_E (h)	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.050	0.169	7.7	7.4	0.2	0.042	0.144	6.8	6.9	0.1
	2	0.041	0.141	7.0			0.041	0.141	6.9		
	3	0.042	0.144	7.5			0.041	0.141	7.0		
2	1	0.052	0.176	8.3	8.3	0.1	0.048	0.163	7.8	7.4	0.2
	2	0.050	0.169	8.2			0.042	0.144	7.0		
	3	0.053	0.182	8.5			0.048	0.163	7.7		
3	1	0.071	0.235	11.1	11.2	0.0	0.048	0.163	8.1	8.1	0.1
	2	0.070	0.232	11.2			0.050	0.169	8.2		
	3	0.072	0.238	11.2			0.050	0.169	7.9		
4	1	0.101	0.329	15.5	15.7	0.1	0.103	0.335	15.8	15.9	0.0
	2	0.100	0.326	15.8			0.101	0.329	15.9		
	3	0.103	0.335	15.7			0.104	0.338	15.9		
5	1	0.096	0.313	14.8	15.7	0.4	0.103	0.335	15.9	16.2	0.1
	2	0.099	0.322	15.8			0.102	0.332	16.3		
	3	0.102	0.332	16.5			0.101	0.329	16.4		
6	1	0.101	0.329	15.5	15.5	0.2	0.104	0.338	15.9	15.8	0.1
	2	0.101	0.329	15.9			0.102	0.332	16.1		
	3	0.089	0.322	15.1			0.101	0.329	15.5		
7	1	0.102	0.332	15.6	15.7	0.3	0.102	0.332	15.7	15.8	0.2
	2	0.104	0.338	16.4			0.103	0.335	16.2		
	3	0.098	0.319	15.0			0.101	0.329	15.5		
8	1	0.097	0.316	14.9	15.4	0.2	0.102	0.332	15.6	15.8	0.1
	2	0.099	0.322	15.6			0.102	0.332	16.1		
	3	0.102	0.332	15.6			0.103	0.335	15.7		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.20 Effect of extraction times on the extracted Cu concentrations from S2 and S5 (Lot-I) for F-IV.

T_E (h)	n	Cu									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
1	1	0.013	0.124	5.9	5.1	0.4	0.014	0.134	6.4	6.4	0.1
	2	0.011	0.104	5.2			0.013	0.124	6.1		
	3	0.009	0.083	4.1			0.013	0.124	6.2		
2	1	0.014	0.134	6.3	7.0	0.3	0.013	0.124	5.8	5.6	0.2
	2	0.016	0.155	7.5			0.011	0.104	5.0		
	3	0.016	0.155	7.3			0.013	0.124	5.8		
3	1	0.019	0.185	8.7	9.4	0.3	0.011	0.104	5.1	5.7	0.3
	2	0.021	0.205	9.9			0.013	0.124	6.0		
	3	0.021	0.205	9.6			0.014	0.134	6.3		
4	1	0.020	0.195	9.2	9.9	0.4	0.019	0.185	8.7	8.5	0.1
	2	0.023	0.226	10.9			0.018	0.175	8.5		
	3	0.021	0.205	9.6			0.018	0.175	8.2		
5	1	0.020	0.195	9.2	9.3	0.2	0.019	0.185	8.8	8.0	0.6
	2	0.019	0.185	9.0			0.014	0.134	6.7		
	3	0.020	0.195	9.7			0.018	0.175	8.7		
6	1	0.021	0.205	9.7	9.4	0.1	0.018	0.175	8.2	6.8	0.6
	2	0.020	0.195	9.4			0.013	0.124	6.0		
	3	0.020	0.195	9.2			0.014	0.134	6.3		
7	1	0.020	0.195	9.1	9.0	0.1	0.014	0.134	6.3	7.0	0.7
	2	0.019	0.185	8.9			0.013	0.124	6.0		
	3	0.020	0.195	9.1			0.019	0.185	8.7		
8	1	0.021	0.205	97	9.3	0.3	0.019	0.185	8.7	8.7	0.1
	2	0.019	0.185	8.9			0.018	0.175	8.5		
	3	0.020	0.195	9.2			0.018	0.175	8.2		

[1] Average absorbance, [2] Concentration (mg/L), [3] Concentration (mg/kg, dry wt.),

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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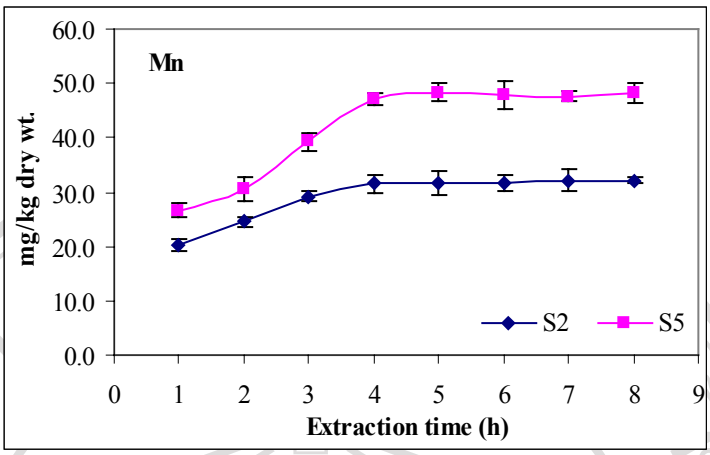
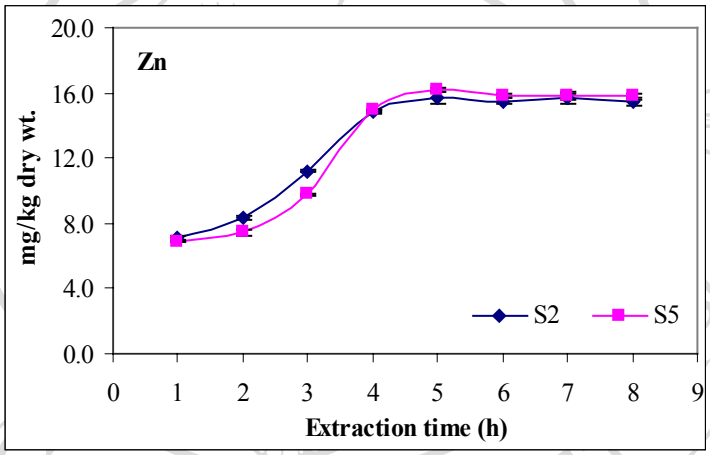
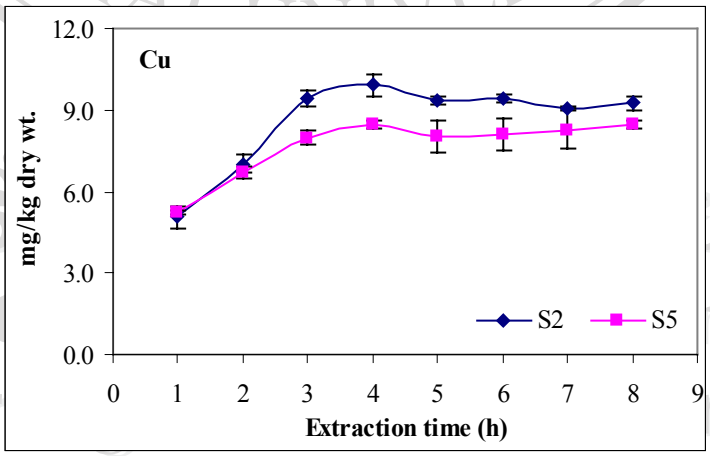


(a)



(b)



(c)

Figure 3.8 Effects of extraction times on the extracted Mn (a), Zn (b), and Cu (c) concentrations from S2 (Lot-I) and S5 (Lot-I) for F-IV.

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3.3 Effect of Repetitive Extractions

In this task, an individual fraction was successively extracted with specific reagents for four times for S2 and S5 (Lot-II). All experimental procedures of repetitive extractions for the sequential extraction method were described in Section 2.5.3. For each extraction step of F-I to F-IV, extraction procedure was performed as sub-fraction for four times. The extracted Mn concentrations using four successive extractions are shown in Table 3.21, 3.22, 3.23, and 3.24 for F-I, F-II, F-III, and F-IV, respectively. For F-I, Table 3.21 shows the extracted Mn concentrations for four successive extractions from both samples. It was found that the extracted Mn concentrations were found for the first, second, and third extractions for each extraction step, whereas those for the fourth extraction were not detected. Extraction efficiency of each successive extraction was evaluated as the percentage that was calculated by comparing the extracted Mn concentration obtained from once extraction with the sum of those obtained from four successive extractions. For each extraction step, the extracted Mn concentration was high for the first extraction, which was more than 80.0, 86.6, 92.6, and 87.7% for F-I, F-II, F-III, and F-IV, respectively. For another extraction step, the extracted Mn concentrations from both samples were highest values for the first extraction the same F-I. As shown in Figure 3.22, 3.23, and 3.24, the percentages of the extracted Mn concentrations in the first extraction were more than 86.6, 92.6, and 87.7% for F-II, F-III, and F-IV, respectively.

Figure 3.9 (a-d) shows the variation of extractable Mn concentration for four successive extractions. For both samples, the extracted Mn concentrations were obviously decreased with increasing the number of extractions. This indicates that the extracted Mn concentrations were high for the first extraction, whereas that of the

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second, third, and fourth successive extractions were very low to non-detectable values. It was observed that the extracted Mn concentrations for the first extraction were high because Mn concentrations might be presented high in sediments. The influence of the extracted Mn concentration found in the second and third successive extractions might be affected from equilibrium constants. Cottenie *et al.* [62] reported that the distributions of different metal forms have been governed by the equilibrium constants of corresponding reactions of precipitation and dissolution, adsorption and de-sorption. Equilibrium displacements, resulting transfer from one form to another, may occur as a consequence of changing physical and chemical conditions.

The results of the extracted Zn concentrations for four successive extractions are shown in Table 3.25, 3.26, 3.27, and 3.28, for F-I, F-II, F-III, and F-IV, respectively. The extractable Zn concentrations were only found in the first extraction, whereas those of the second, third, and fourth successive extractions were not detected. Therefore, only one time of extraction for an individual fraction could absolutely extract Zn from sediments. Figure 3.10 (a-d) shows the variation of the extracted Zn concentration for four successive extractions of each extraction step.

Table 3.29 and 3.30 show the extracted Cu concentrations for F-III and F-IV, respectively. For F-I and F-II, the extracted Cu concentrations were not detected for both samples. It was found that extractable Cu concentrations were found in the first extraction, whereas those of the second, third, and fourth successive extractions were not detected. Figure 3.11 (a-b) shows the variation of the extracted Cu concentration for four successive extractions of F-III and F-IV.

Table 3.31 and 3.32 show the extracted Cd concentration for F-I and F-II, respectively. For both samples, the extracted Cd concentrations were found in the

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first extraction, whereas those of the second, third, and fourth repetitive extractions was not detected. For F-III and F-IV, the extracted Cd concentrations were not detected. For both sediment samples, Figure 3.12 (a-b) shows the variation of the extracted Cd concentration for four successive extractions of F-I and F-II.

The results of the extracted Pb concentrations for four successive extractions are shown in Table 3.33, 3.34, and 3.35, for F-II, F-III, and F-IV, respectively. The extractable Pb concentrations were only found in the first extraction, whereas that of the second, third, and fourth successive extractions was not detected. Therefore, only one time of extraction for an individual fraction could enough to extract Pb concentration from sediment samples. Figure 3.13 (a-c) shows the variation of the extracted Pb concentration for four successive extractions of F-II, F-III, and F-IV.

The results of extractable Mn, Zn, Cu, Cd, and Pb concentrations in each extraction step found that only one time extraction of each extraction step was able to extract these metal concentrations from sediment samples. For investigating the extraction efficiency of each extraction step, the extractable metal concentrations obtained from the first extraction were more than 80.0% for Mn and were nearly 100% for Zn, Cu, and Cd. Only Mn concentration was found in the second and third successive extractions. To perform successive extractions more than a time for each extraction step, although, the extracted Mn concentration was found increasingly, extracting solution volumes and extraction times were increasingly used. The influence of repetitive extractions were studied in order to avoid re-adsorption effect of metal mobilization under equilibrium constants [24]. It was found that re-adsorption effect could be occurred for metals in which existed high concentrations in

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sediments, for example, Mn. It can be seen that high Mn levels could cause the re-adsorption during extraction for all steps. For trace metals, it is not the case.

Table 3.21 The extracted Mn concentrations for four successive extractions from S2 and S5 (Lot-II) for F-I.

R_E	n	Mn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}I_1$	1	0.152	1.213	59.6	61.4	1.9	0.292	2.585	129.4	131.3	2.5
	2	0.155	1.243	61.4			0.301	2.674	132.6		
	3	0.174	1.429	63.5			0.301	2.674	132.8		
$F\text{-}I_2$	1	0.048	0.193	9.5	9.8	0.3	0.056	0.272	13.5	12.7	1.8
	2	0.049	0.205	10.1			0.050	0.213	10.5		
	3	0.051	0.223	9.9			0.057	0.281	14.0		
$F\text{-}I_3$	1	0.041	0.125	6.1	5.8	0.5	0.039	0.105	5.2	4.7	0.5
	2	0.039	0.107	5.2			0.037	0.085	4.2		
	3	0.043	0.139	6.2			0.038	0.095	4.7		
$F\text{-}I_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum ($F\text{-}I_1$+ $F\text{-}I_2$+ $F\text{-}I_3$+ $F\text{-}I_4$) = 77.1							Sum ($F\text{-}I_1$+ $F\text{-}I_2$+ $F\text{-}I_3$+ $F\text{-}I_4$) = 148.7				
$F\text{-}I_1 = \frac{61.4}{77.1} \times 100 = 80.0\%$							$F\text{-}I_1 = \frac{131.3}{148.7} \times 100 = 88.3\%$				
$F\text{-}I_1+F\text{-}I_2 = \frac{(61.4+9.8)}{77.1} \times 100 = 92.4\%$							$F\text{-}I_1+F\text{-}I_2 = \frac{(131.3+12.7)}{148.7} \times 100 = 96.8\%$				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.22 The extracted Mn concentrations for four successive extractions from S2 and S5 (Lot-II) for F-II.

R_E	Mn										
	n	S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-II_1*	1	0.108	0.849	208.9	213.8	4.7	0.162	1.363	270.8	273.0	2.4
	2	0.112	0.888	218.2			0.167	1.406	278.9		
	3	0.120	0.965	214.2			0.161	1.356	269.4		
F-II_2	1	0.080	0.575	28.3	26.7	2.1	0.084	0.618	30.7	31.9	2.2
	2	0.078	0.561	27.6			0.092	0.695	34.4		
	3	0.077	0.547	24.3			0.084	0.618	30.7		
F-II_3	1	0.033	0.132	6.5	6.4	0.1	0.036	0.164	8.1	7.7	0.4
	2	0.033	0.132	6.5			0.035	0.151	7.5		
	3	0.034	0.141	6.3			0.035	0.151	7.5		
F-II_4	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
	Sum (F-II_1+ F-II_2+ F-II_3+ F-II_4) = 246.9						Sum (F-II_1+ F-II_2+ F-II_3+ F-II_4) = 312.6				
	F-II_1 = $\frac{213.8}{246.9}$ x 100 = 86.6%						F-I_1 = $\frac{273.0}{312.6}$ x100 = 87.3%				
	F-II_1+F-II_2 = $\frac{(213.8+26.7)}{246.9}$ x 100 = 97.4%						F-II_1+F-II_2 = $\frac{(273.0+31.9)}{312.6}$ x100 = 97.5%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

* The extracts obtained from S2 was further diluted by 5-fold before analysis.

* The extracts obtained from S5 was further diluted by 4-fold before analysis.

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Table 3.23 The extracted Mn concentrations for four successive extractions from S2 and S5 (Lot-II) for F-III.

R_E	Mn										
	n	S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-III$_1$*	1	0.132	1.077	265.0	259.9	4.9	0.068	0.465	231.0	220.9	4.2
	2	0.130	1.057	259.7			0.065	0.438	217.3		
	3	0.140	1.149	255.1			0.065	0.432	214.6		
F-III$_2$	1	0.049	0.284	13.9	15.0	0.9	0.042	0.213	10.6	12.8	1.9
	2	0.053	0.318	15.6			0.049	0.287	14.2		
	3	0.056	0.348	15.5			0.048	0.273	13.5		
F-III$_3$	1	0.028	0.084	4.1	5.8	1.5	0.031	0.114	5.6	5.6	0.2
	2	0.033	0.134	6.6			0.031	0.116	5.8		
	3	0.035	0.152	6.7			0.030	0.107	5.3		
F-III$_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
	Sum (F-III$_1$+ F-III$_2$+ F-III$_3$+ F-III$_4$) = 280.7						Sum (F-III$_1$+ F-III$_2$+ F-III$_3$+ F-III$_4$) = 239.3				
	F-III$_1$ = $\frac{259.9}{280.7}$ x 100 = 92.6%						F-III$_1$ = $\frac{220.9}{239.3}$ x100 = 92.3%				
	F-III$_1$+F-III$_2$ = $\frac{(259.9+15.0)}{280.7}$ x 100 = 97.9%						F-III$_1$+F-III$_2$ = $\frac{(220.9+12.8)}{239.3}$ x100 = 97.7%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

* The extracts obtained from S2 was further diluted by 5-fold before analysis.

* The extracts obtained from S5 was further diluted by 10-fold before analysis.

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Table 3.24 The extracted Mn concentrations for four successive extractions from S2 and S5 (Lot-II) for F-IV.

R_E	n	Mn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-IV_1	1	0.091	0.741	36.4	37.2	0.7	0.122	1.031	51.2	48.9	2.2
	2	0.095	0.772	37.9			0.113	0.946	46.9		
	3	0.101	0.836	37.1			0.116	0.977	48.5		
F-IV_2	1	0.025	0.112	5.2	5.1	0.4	0.029	0.152	7.6	7.9	0.3
	2	0.023	0.095	4.7			0.030	0.162	8.0		
	3	0.025	0.118	5.2			0.030	0.162	8.0		
F-IV_3	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-IV_4	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
	Sum (F-IV_1+ F-IV_2+ F-IV_3+ F-IV_4) = 42.4						Sum (F-IV_1+ F-IV_2+ F-IV_3+ F-IV_4) = 56.8				
	F-IV_1 = $\frac{37.2}{42.4}$ x 100 = 87.7%						F-IV_1 = $\frac{48.9}{56.8}$ x100 = 86.1%				
	F-IV_1+F-IV_2 = $\frac{(37.2+5.1)}{42.4}$ x 100 = 100%						F-IV_1+F-IV_2 = $\frac{(48.9+7.9)}{56.8}$ x100 = 100%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.25 The extracted Zn concentrations for four successive extractions from S2 and S5 (Lot-II) for F-I.

R_E	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}I_1$	1	0.014	0.028	1.4	1.3	0.1	0.011	0.021	1.1	1.0	0.1
	2	0.015	0.029	1.4			0.010	0.029	0.9		
	3	0.014	0.028	1.3			0.011	0.021	1.1		
$F\text{-}I_2$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}I_3$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}I_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum ($F\text{-}I_1$+ $F\text{-}I_2$+ $F\text{-}I_3$+ $F\text{-}I_4$) = 1.3 $F\text{-}I_1$ = $\frac{1.3}{1.3}$ x 100 = 100%							Sum ($F\text{-}I_1$+ $F\text{-}I_2$+ $F\text{-}I_3$+ $F\text{-}I_4$) = 1.0 $F\text{-}I_1$ = $\frac{1.0}{1.0}$ x100 = 100%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.26 The extracted Zn concentrations for four successive extractions from S2 and S5 (Lot-II) for F-II.

R_E	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}II_1$	1	0.055	0.124	6.1	6.0	0.1	0.050	0.112	5.6	5.5	0.0
	2	0.053	0.120	5.9			0.049	0.110	5.5		
	3	0.060	0.136	6.1			0.050	0.111	5.5		
$F\text{-}II_2$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}II_3$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}II_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		

Sum ($F\text{-}II_1$+ $F\text{-}II_2$+ $F\text{-}II_3$+ $F\text{-}II_4$) = 6.0

$F\text{-}II_1 = \frac{6.0}{6.0} \times 100 = 100\%$

Sum ($F\text{-}II_1$+ $F\text{-}II_2$+ $F\text{-}II_3$+ $F\text{-}II_4$) = 5.5

$F\text{-}II_1 = \frac{5.5}{5.5} \times 100 = 100\%$

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.27 The extracted Zn concentrations for four successive extractions from S2 and S5 (Lot-II) for F-III.

R_E	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}III_1$	1	0.103	0.214	10.5	10.2	0.2	0.081	0.165	8.2	8.4	0.2
	2	0.102	0.212	10.4			0.084	0.171	8.5		
	3	0.106	0.220	9.8			0.084	0.171	8.5		
$F\text{-}III_2$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}III_3$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}III_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum ($F\text{-}III_1$+ $F\text{-}III_2$+ $F\text{-}III_3$+ $F\text{-}III_4$) = 10.2							Sum ($F\text{-}III_1$+ $F\text{-}III_2$+ $F\text{-}III_3$+ $F\text{-}III_4$) = 8.4				
$F\text{-}III_1 = \frac{10.2}{10.2} \times 100 = 100\%$							$F\text{-}III_1 = \frac{8.4}{8.4} \times 100 = 100\%$				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.28 The extracted Zn concentrations for four successive extractions from S2 and S5 (Lot-II) for F-IV.

R_E	n	Zn									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}IV_1$	1	0.038	0.070	3.4	3.4	0.1	0.042	0.077	3.8	3.8	0.0
	2	0.038	0.069	3.4			0.041	0.076	3.7		
	3	0.043	0.080	3.5			0.041	0.076	3.8		
$F\text{-}IV_2$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}IV_3$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}IV_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		

Sum ($F\text{-}IV_1$+ $F\text{-}IV_2$+ $F\text{-}IV_3$+ $F\text{-}IV_4$) = 3.4

$F\text{-}IV_1 = \frac{3.4}{3.4} \times 100 = 100\%$

Sum ($F\text{-}IV_1$+ $F\text{-}IV_2$+ $F\text{-}IV_3$+ $F\text{-}IV_4$) = 3.8

$F\text{-}IV_1 = \frac{3.8}{3.8} \times 100 = 100\%$

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.29 The extracted Cu concentrations for four successive extractions from S2 and S5 (Lot-II) for F-III.

R_E	n	Cu									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}III_1$	1	0.014	0.063	3.1	3.1	0.0	0.012	0.048	2.4	2.9	0.4
	2	0.014	0.063	3.1			0.014	0.062	3.1		
	3	0.015	0.071	3.2			0.014	0.062	3.1		
$F\text{-}III_2$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}III_3$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}III_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum ($F\text{-}III_1$+ $F\text{-}III_2$+ $F\text{-}III_3$+ $F\text{-}III_4$) = 3.1							Sum ($F\text{-}III_1$+ $F\text{-}III_2$+ $F\text{-}III_3$+ $F\text{-}III_4$) = 2.9				
$F\text{-}III_1 = \frac{3.1}{3.1} \times 100 = 100\%$							$F\text{-}III_1 = \frac{2.9}{2.9} \times 100 = 100\%$				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.30 The extracted Cu concentrations for four successive extractions from S2 and S5 (Lot-II) for F-IV.

R_E	n	Cu									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-IV_1	1	0.018	0.095	4.7	4.6	0.1	0.020	0.113	5.6	5.6	0.1
	2	0.017	0.092	4.5			0.019	0.111	5.5		
	3	0.019	0.103	4.6			0.020	0.114	5.7		
F-IV_2	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-IV_3	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-IV_4	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum (F-IV_1+ F-IV_2+ F-IV_3+ F-IV_4) = 4.6							Sum (F-IV_1+ F-IV_2+ F-IV_3+ F-IV_4) = 5.6				
F-IV_1 = $\frac{4.6}{4.6}$ x 100 = 100%							F-IV_1 = $\frac{5.6}{5.6}$ x 100 = 100%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.31 The extracted Cd concentrations for four successive extractions from S2 and S5 (Lot-II) for F-I.

R_E	n	Cd									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}I_1$	1	0.011	0.033	1.6	1.6	0.0	0.010	0.029	1.4	1.6	0.1
	2	0.011	0.033	1.6			0.012	0.037	1.8		
	3	0.012	0.037	1.6			0.011	0.033	1.6		
$F\text{-}I_2$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}I_3$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}I_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum ($F\text{-}I_1$+ $F\text{-}I_2$+ $F\text{-}I_3$+ $F\text{-}I_4$) = 1.6							Sum ($F\text{-}I_1$+ $F\text{-}I_2$+ $F\text{-}I_3$+ $F\text{-}I_4$) = 1.6				
$F\text{-}I_1$ = $\frac{1.6}{1.6}$ x 100 = 100%							$F\text{-}I_1$ = $\frac{1.6}{1.6}$ x 100 = 100%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.32 The extracted Cd concentrations for four successive extractions from S2 and S5 (Lot-II) for F-II.

R_E	n	Cd									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}II_1$	1	0.014	0.045	2.2	2.3	0.1	0.017	0.056	2.8	2.6	0.2
	2	0.015	0.049	2.4			0.015	0.049	2.4		
	3	0.015	0.049	2.2			0.016	0.053	2.6		
$F\text{-}II_2$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}II_3$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}II_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum ($F\text{-}II_1$+ $F\text{-}II_2$+ $F\text{-}II_3$+ $F\text{-}II_4$) = 2.3							Sum ($F\text{-}II_1$+ $F\text{-}II_2$+ $F\text{-}II_3$+ $F\text{-}II_4$) = 2.6				
$F\text{-}II_1$ = $\frac{2.3}{2.3}$ x 100 = 100%							$F\text{-}II_1$ = $\frac{2.6}{2.6}$ x 100 = 100%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.33 The extracted Pb concentrations for four successive extractions from S2 and S5 (Lot-II) for F-II.

R_E	n	Pb									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}II_1$	1	ND	-	-	-	-	0.006	0.045	2.2	2.2	0.0
	2	ND	-	-			0.006	0.045	2.1		
	3	ND	-	-			0.006	0.045	2.2		
$F\text{-}II_2$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}II_3$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}II_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum ($F\text{-}II_1$+ $F\text{-}II_2$+ $F\text{-}II_3$+ $F\text{-}II_4$) = ND							Sum ($F\text{-}II_1$+ $F\text{-}II_2$+ $F\text{-}II_3$+ $F\text{-}II_4$) = 2.4 $F\text{-}II_1$ = $\frac{2.4}{2.4}$ x 100 = 100%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.34 The extracted Pb concentrations for four successive extractions from S2 and S5 (Lot-II) for F-III.

R_E	n	Pb									
		S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
$F\text{-}III_1$	1	0.007	0.180	8.2	8.4	0.1	0.008	0.225	10.2	10.4	0.2
	2	0.007	0.180	8.5			0.008	0.225	10.6		
	3	0.007	0.180	8.4			0.008	0.225	10.5		
$F\text{-}III_2$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}III_3$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
$F\text{-}III_4$	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum ($F\text{-}III_1$+ $F\text{-}III_2$+ $F\text{-}III_3$+ $F\text{-}III_4$) = 8.4							Sum ($F\text{-}III_1$+ $F\text{-}III_2$+ $F\text{-}III_3$+ $F\text{-}III_4$) = 10.4				
$F\text{-}III_1$ = $\frac{8.4}{8.4}$ x 100 = 100%							$F\text{-}III_1$ = $\frac{10.4}{10.4}$ x 100 = 100%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.35 The extracted Pb concentrations for four successive extractions from S2 and S5 (Lot-II) for F-IV.

R_E	Pb										
	n	S2					S5				
		Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs.[1]	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-IV_1	1	0.006	0.081	3.7	3.6	0.2	0.007	0.090	4.1	4.0	0.2
	2	0.006	0.072	3.4			0.007	0.090	4.2		
	3	0.006	0.081	3.8			0.006	0.081	3.8		
F-IV_2	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-IV_3	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-IV_4	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
Sum (F-IV_1+ F-IV_2+ F-IV_3+ F-IV_4) = 3.6 F-IV_1 = $\frac{3.6}{3.6}$ x 100 = 100%							Sum (F-IV_1+ F-IV_2+ F-IV_3+ F-IV_4) = 4.0 F-IV_1 = $\frac{4.0}{4.0}$ x 100 = 100%				

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry wt.).

[4] Average concentration (mg/kg, dry wt.).

Volumes of the extracts were prepared as 50 mL before analysis.

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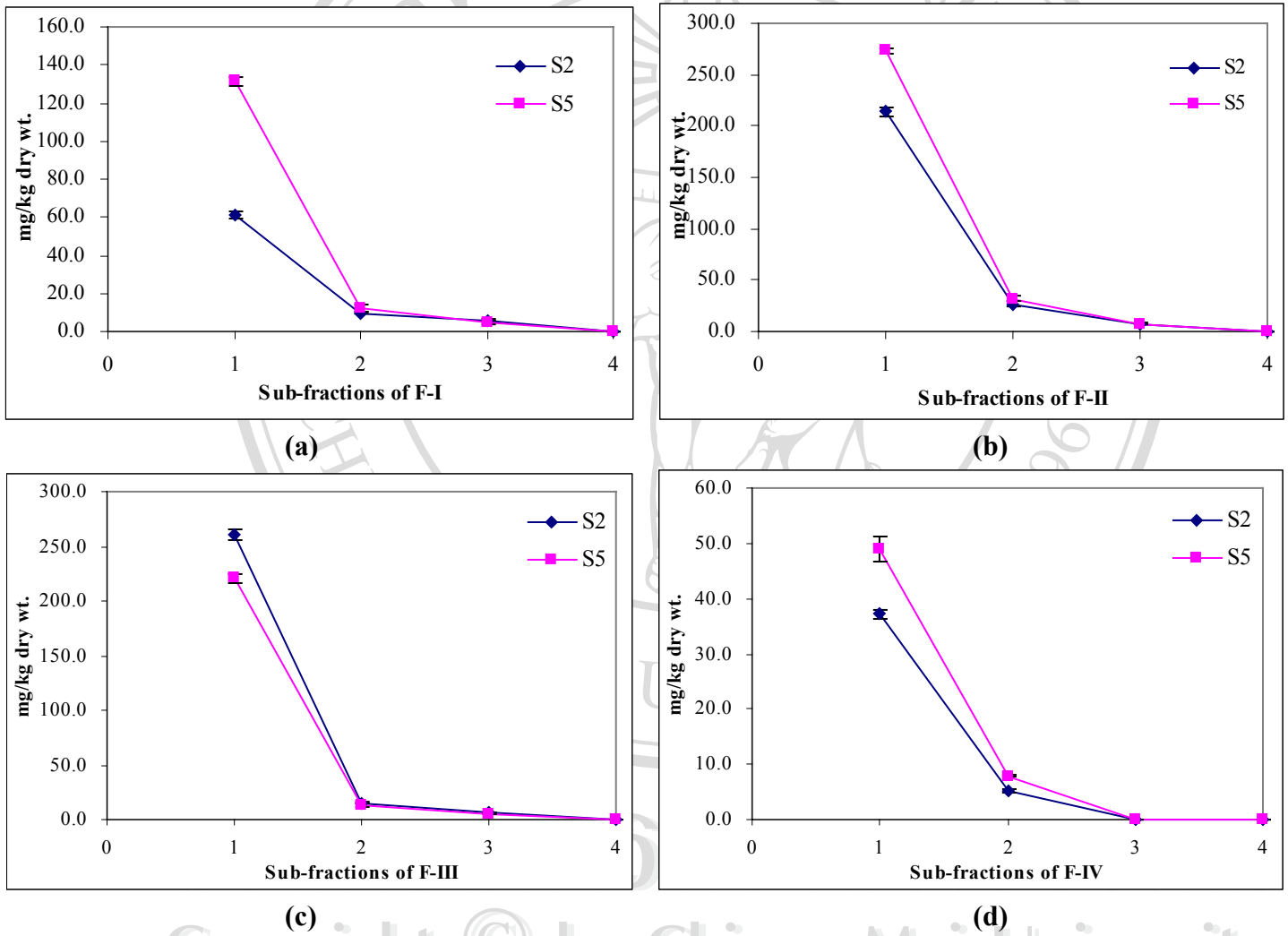

Figure 3.9 Variations of the extracted Mn concentrations by four successive extractions for F-I (a), F-II (b), F-III (c), and F-IV(d).

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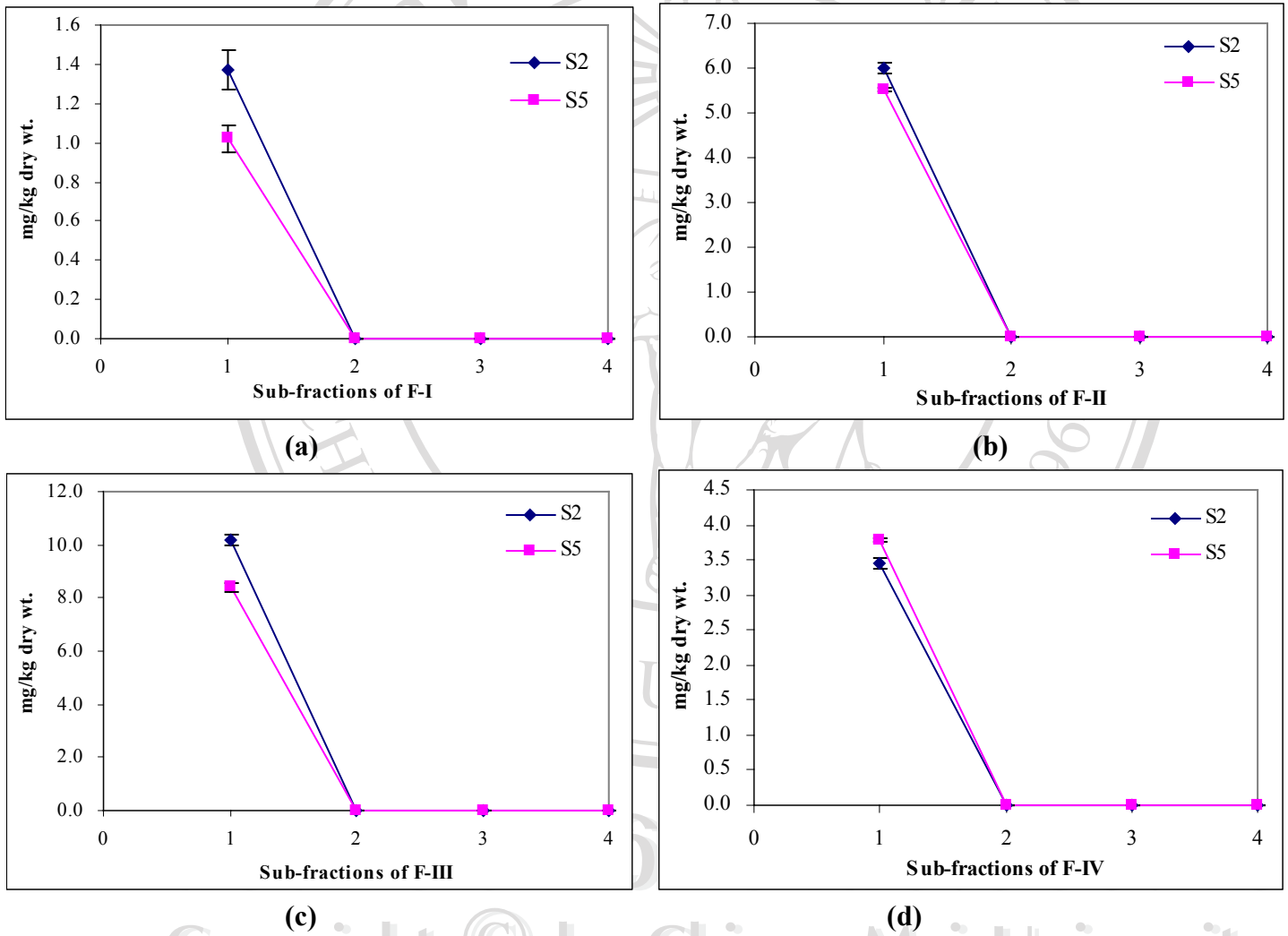

Figure 3.10 Variations of the extracted Zn concentrations by four successive extractions for F-I (a), F-II (b), F-III (c), and F-IV(d).

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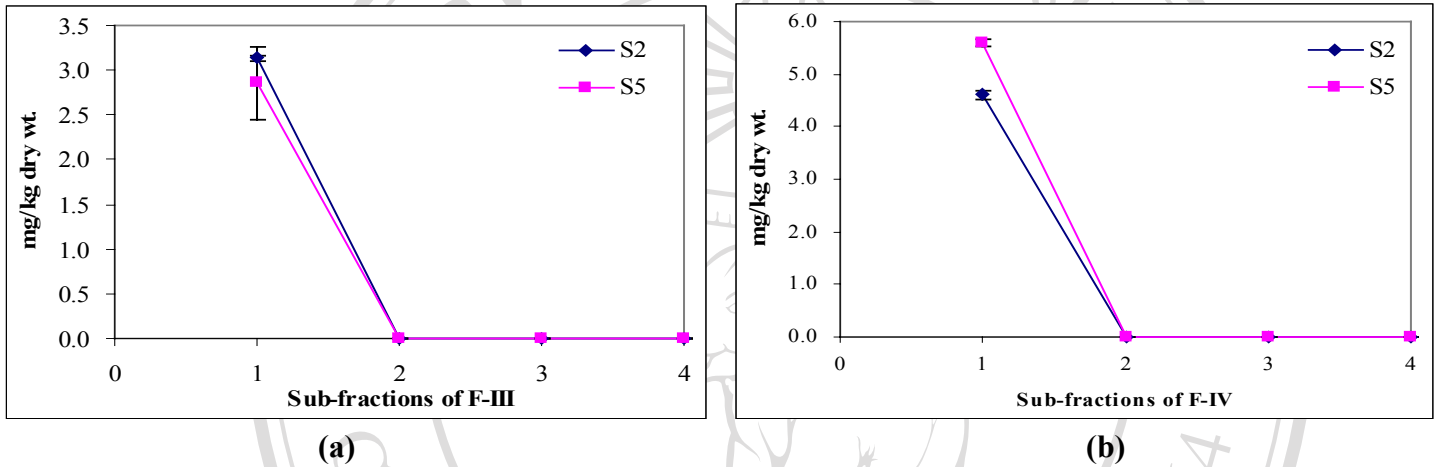

Figure 3.11 Variations of the extracted Cu concentrations by four successive extractions for F-III (a) and F-IV (b).

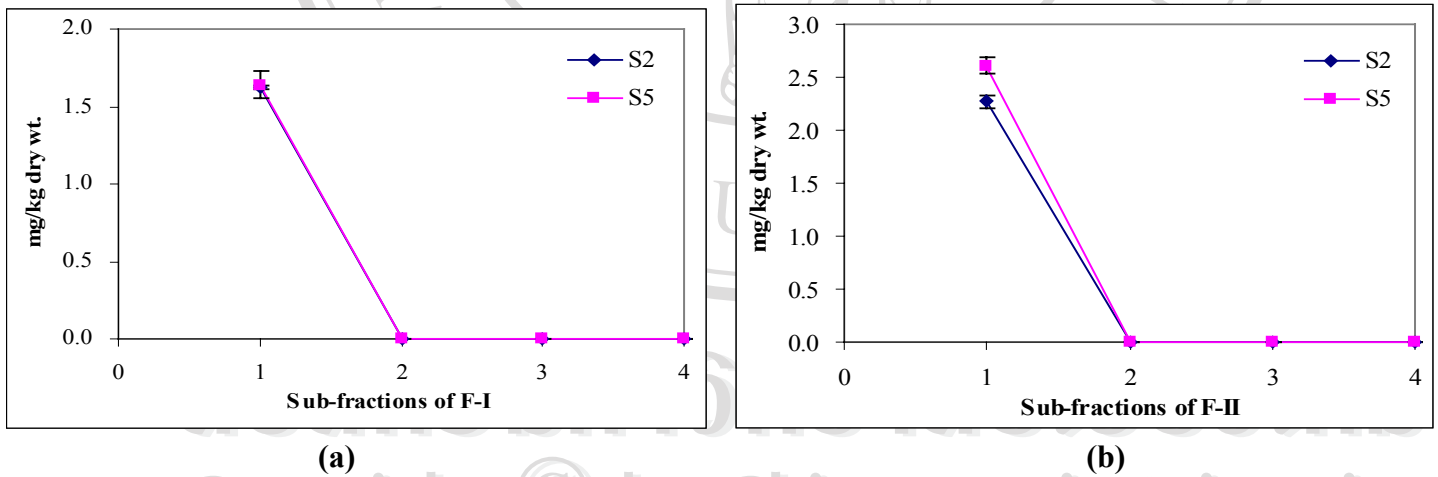

Figure 3.12 Variations of the extracted Cd concentrations by four successive extractions for F-I (a) and F-II (b).

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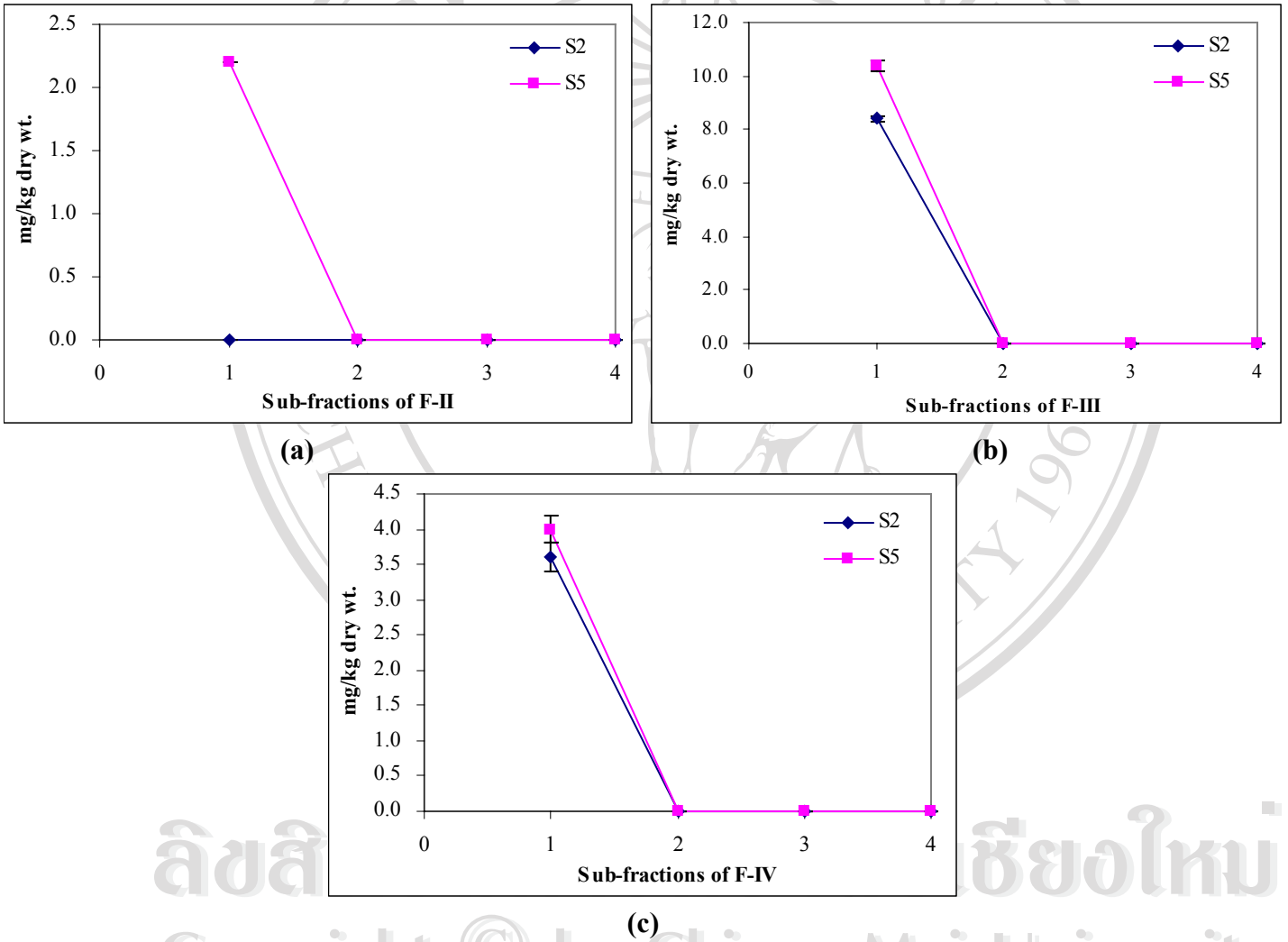

Figure 3.13 Variations of the extracted Pb concentrations by four successive extractions for F-II (a), F-III (b), and F-IV (c).

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3.4 The Optimized Sequential Extraction Method

3.4.1 Comparison between Tessier's and the Optimized Methods

To investigate the extraction conditions for each extraction step of F-I to F-V, the optimized sequential extraction method was described in Section 2.6, whereas proposed Tessier sequential extraction [20] was shown in Table 2.3 (Section 2.7). Comparing to Tessier's method, the optimized sequential extraction method provided shorter total extraction time, but required larger extracting solution volumes. Both Tessier's and the optimized sequential extraction methods were applied to determination of heavy metals from S2 and S5 for Lot-I. For both samples, the results of the extracted metal concentrations obtained from two methods are shown in Table 3.36-3.37, 3.38-3.39, 3.40-3.41, 3.42-3.43, and 3.44-3.45 for Mn, Zn, Cu, Cd, and Pb respectively. Metal distributions in different sediment phases were expressed as percentages by calculating between the extractable metals obtained from each extraction step per the sum of extractable metals obtained from five extraction steps. Using Tessier's and the optimized sequential extraction methods for both samples, the concentrations of Mn, Zn, Cu, Cd, and Pb and their percentage distributions are shown in Table 3.46 and 3.47 for S2 and S5, respectively.

The extracted Mn concentrations obtained from Tessier's and the optimized methods are shown in Table 3.36 and 3.37 for S2 and S5, respectively. As shown in Table 3.46 for S2 and 3.47 for S5, average percentage distributions of the extracted Mn concentrations obtained from the optimized method were 17.1, 25.0, 47.6, 6.5, and 3.6%, whereas those obtained from Tessier's method were 15.5, 22.7, 49.0, 6.2, and 6.5% for F-I, F-II, F-III, F-IV, and F-V, respectively. This indicates that the extracted Mn concentrations obtained from both methods for each extraction step

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were comparable. It was found that Mn concentration was extremely high for bound to Fe-Mn oxide fraction (F-III).

The extracted Zn concentrations obtained from Tessier's and the optimized methods are shown in Table 3.38 and 3.39 for S2 and S5, respectively. As shown in Table 3.46 for S2 and 3.47 for S5, average percentage distributions of the extracted Zn concentrations obtained from the optimized method for S2 were 3.2, 16.0, 21.3, 12.9, and 46.3%, whereas those obtained from Tessier's method were 3.4, 15.5, 21.3, 25.7, and 46.9% for F-I, F-II, F-III, F-IV, and F-V, respectively. It was observed that high proportion of Zn in sediment was associated to the residual fraction (F-V).

The extracted Cu concentrations obtained from Tessier's and the optimized methods are shown in Table 3.40 and 3.41 for S2 and S5, respectively. It was found that the extracted Cu concentrations were not detected for F-I and F-II for both samples. Average percentages distributions of Cu in sediments obtained from the optimized method were 18.6, 50.4, and 32.9%, whereas those obtained from Tessier's method were 19.3, 50.4, and 30.6% for F-III, F-IV, and F-V, respectively as shown in Table 3.46 and 3.47. This indicates that high proportion of Cu concentrations were preferentially associated to organic matter in sediments.

The extracted Cd concentrations obtained from Tessier's and the optimized methods are shown in Table 3.42 and 3.43 for S2 and S5, respectively. For S2, the extracted Cd concentrations were not detected for F-III, F-IV, and F-V while those from S5 were not detected for F-III and F-IV. As shown in Table 3.46 for S2 and 3.47 for S5, average percentage distributions of the extracted Cd concentrations obtained from the optimized method were 44.4, 41.1, and 28.9%, whereas those obtained from Tessier's method were 44.3, 41.4, and 28.4% for F-I, F-II, and F-V,

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respectively. This indicates that the extracted Cd concentrations were high for F-I and F-II, which were considered as mobile forms.

The extracted Pb concentrations obtained from Tessier's and the optimized methods are shown in Table 3.44 and 3.45 for S2 and S5, respectively. For S2, the extracted Pb concentrations were not detected for F-I and F-II, whereas those from S5 were not detected for F-I. As shown in Table 3.46 for S2 and 3.47 for S5, average, percentage distributions of the extracted Pb concentrations obtained from the optimized method were 16.7, 40.6, 16.4, and 34.6%, whereas those obtained from Tessier's method were 16.2, 38.2, 16.7, and 36.9% for F-II, F-III, F-IV, and F-V, respectively. This indicates that the extracted Pb concentrations obtained from two methods were high and comparable for F-III and F-V.

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Table 3.36 Comparison of the extracted Mn concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S2 (Lot-I).

[F]	Mn										
	n	Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	0.138	1.249	62.2	61.3	1.2	0.163	1.525	75.5	76.3	4.4
	2	0.137	1.238	61.6			0.173	1.636	81.0		
	3	0.134	1.205	59.9			0.157	1.459	72.3		
F-II	1	0.236	2.333	116.1	118.5	5.6	0.304	3.085	152.6	147.8	4.2
	2	0.233	2.300	114.5			0.292	2.952	146.2		
	3	0.232	2.510	124.9			0.289	2.919	144.7		
F-III*	1	0.073	0.530	263.7	260.1	3.1	0.073	0.530	262.1	260.6	3.1
	2	0.072	0.519	258.4			0.072	0.519	257.0		
	3	0.072	0.519	258.2			0.073	0.530	262.6		
F-IV	1	0.074	0.541	26.9	27.5	0.6	0.081	0.618	30.6	31.0	1.7
	2	0.075	0.552	27.5			0.085	0.663	32.8		
	3	0.076	0.563	28.0			0.079	0.596	29.5		
F-V	1	0.097	0.795	39.6	39.4	1.9	0.071	0.508	25.1	23.7	1.3
	2	0.100	0.829	41.3			0.068	0.475	23.5		
	3	0.093	0.751	37.4			0.066	0.452	22.4		
				Sum =	506.8	6.8			Sum =	539.4	7.2

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

* The extracts were further diluted by 10-fold before analysis.

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Table 3.37 Comparison of the extracted Mn concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S5 (Lot-I).

[F]	n	Mn									
		Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	0.241	2.388	118.1	117.8	4.8	0.265	2.654	132.6	128.7	3.5
	2	0.230	2.267	112.8			0.254	2.532	125.8		
	3	0.248	2.466	122.5			0.257	2.565	127.7		
F-II	1	0.273	2.742	135.6	137.2	9.0	0.285	2.875	143.6	144.6	3.5
	2	0.292	2.952	146.9			0.283	2.853	141.7		
	3	0.260	2.598	129.1			0.295	2.986	148.6		
F-III*	1	0.080	0.607	300.4	289.5	11.5	0.079	0.596	297.9	300.6	5.8
	2	0.076	0.558	277.5			0.081	0.618	307.2		
	3	0.078	0.585	290.6			0.079	0.596	296.7		
F-IV	1	0.103	0.862	20.6	43.7	1.7	0.112	0.961	48.0	47.5	1.7
	2	0.108	0.917	45.6			0.114	0.983	48.9		
	3	0.103	0.862	42.8			0.108	0.917	45.6		
F-V	1	0.083	0.640	31.7	32.0	2.5	0.060	0.396	19.3	18.9	0.4
	2	0.088	0.696	34.6			0.059	0.375	18.6		
	3	0.079	0.596	29.6			0.059	0.375	18.7		
				Sum =	620.2	15.7			Sum =	640.3	7.80

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

* The extracts were further diluted by 10-fold before analysis.

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Table 3.38 Comparison of the extracted Zn concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S2 (Lot-I).

[F]	n	Zn									
		Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	0.011	0.027	1.4	1.4	0.1	0.012	0.030	1.5	1.3	0.1
	2	0.012	0.030	1.5			0.011	0.027	1.3		
	3	0.011	0.027	1.4			0.010	0.025	1.2		
F-II	1	0.045	0.110	5.5	5.6	0.1	0.046	0.112	5.5	5.8	0.2
	2	0.046	0.112	5.6			0.049	0.119	5.9		
	3	0.046	0.112	5.6			0.049	0.119	5.9		
F-III	1	0.066	0.160	8.0	8.0	0.1	0.068	0.165	82	8.1	0.0
	2	0.065	0.158	7.9			0.067	0.163	8.1		
	3	0.067	0.163	8.1			0.068	0.165	8.2		
F-IV	1	0.043	0.105	5.2	5.3	0.0	0.045	0.110	5.4	5.4	0.0
	2	0.044	0.107	5.3			0.044	0.107	5.3		
	3	0.043	0.105	5.2			0.044	0.110	5.4		
F-V	1	0.107	0.260	12.9	12.9	0.0	0.114	0.277	13.7	13.7	0.0
	2	0.107	0.260	12.9			0.115	0.279	13.8		
	3	0.108	0.262	13.0			0.114	0.277	13.7		
				Sum =	33.1	0.1			Sum =	34.4	0.2

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.39 Comparison of the extracted Zn concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S5 (Lot-I).

[F]	n	Zn									
		Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	0.008	0.020	1.0	1.0	0.1	0.007	0.018	0.9	1.0	0.1
	2	0.008	0.020	1.0			0.009	0.023	1.1		
	3	0.009	0.023	1.1			0.008	0.020	1.0		
F-II	1	0.045	0.110	5.4	5.3	0.0	0.045	0.110	5.5	5.6	0.1
	2	0.044	0.107	5.3			0.047	0.114	5.7		
	3	0.044	0.107	5.3			0.045	0.110	5.5		
F-III	1	0.058	0.141	7.0	6.9	0.0	0.059	0.144	7.2	7.1	0.1
	2	0.057	0.139	6.9			0.059	0.144	7.1		
	3	0.057	0.139	6.9			0.058	0.141	7.0		
F-IV	1	0.030	0.073	3.6	3.6	0.1	0.030	0.073	3.7	3.8	0.1
	2	0.029	0.071	3.5			0.031	0.076	3.8		
	3	0.030	0.073	3.6			0.031	0.076	3.8		
F-V	1	0.170	0.412	20.4	20.4	0.0	0.162	0.393	19.6	19.6	0.0
	2	0.169	0.410	20.4			0.163	0.395	19.6		
	3	0.169	0.410	20.3			0.163	0.395	19.7		
				Sum =	37.3	0.1			Sum =	37.1	0.2

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.40 Comparison of the extracted Cu concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S2 (Lot-I).

[F]	n	Cu									
		Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-II	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-III	1	0.008	0.073	3.6	3.6	0.0	0.007	0.063	3.1	3.1	0.0
	2	0.008	0.073	3.6			0.007	0.063	3.1		
	3	0.008	0.073	3.6			0.007	0.063	3.1		
F-IV	1	0.016	0.144	7.2	7.1	0.1	0.016	0.142	7.0	7.0	0.1
	2	0.015	0.143	7.1			0.015	0.141	7.0		
	3	0.015	0.143	7.1			0.015	0.141	7.0		
F-V	1	0.009	0.083	4.1	4.1	0.0	0.009	0.083	4.1	3.8	0.3
	2	0.009	0.083	4.1			0.009	0.083	4.1		
	3	0.009	0.083	4.1			0.008	0.073	3.1		
				Sum =	14.8	0.0			Sum =	13.9	0.1

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.41 Comparison of the extracted Cu concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S5 (Lot-I).

[F]	n	Cu									
		Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-II	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-III	1	0.005	0.047	2.3	2.3	0.0	0.006	0.048	2.4	2.4	0.0
	2	0.005	0.047	2.3			0.005	0.047	2.4		
	3	0.005	0.047	2.3			0.006	0.048	2.4		
F-IV	1	0.018	0.175	8.7	8.4	0.1	0.016	0.155	7.7	8.0	0.1
	2	0.017	0.165	8.2			0.017	0.165	8.2		
	3	0.017	0.165	8.2			0.017	0.165	8.2		
F-V	1	0.012	0.109	5.4	5.4	0.0	0.012	0.112	5.6	5.6	0.0
	2	0.011	0.108	5.4			0.012	0.112	5.6		
	3	0.011	0.108	5.4			0.012	0.112	5.6		
				Sum =	16.1	0.1			Sum =	16.0	0.1

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.42 Comparison of the extracted Cd concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S2 (Lot-I).

[F]	n	Cd									
		Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	0.019	0.049	2.4	2.4	0.0	0.021	0.055	2.7	2.7	0.0
	2	0.019	0.049	2.4			0.021	0.055	2.7		
	3	0.020	0.052	2.5			0.020	0.052	2.6		
F-II	1	0.017	0.042	2.1	2.1	0.0	0.018	0.045	2.2	2.3	0.1
	2	0.018	0.045	2.2			0.020	0.052	2.6		
	3	0.017	0.042	2.1			0.018	0.045	2.2		
F-III	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-IV	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-V	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
				Sum =	4.5	0.0			Sum =	5.0	0.1

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.43 Comparison of the extracted Cd concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S5 (Lot-I).

[F]	n	Cd									
		Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	0.020	0.052	2.6	2.6	0.0	0.020	0.052	2.6	2.6	0.0
	2	0.020	0.052	2.6			0.020	0.052	2.6		
	3	0.019	0.049	2.5			0.020	0.052	2.6		
F-II	1	0.020	0.052	2.6	2.6	0.0	0.022	0.058	2.9	2.7	0.1
	2	0.020	0.052	2.6			0.020	0.052	2.6		
	3	0.020	0.052	2.6			0.020	0.052	2.6		
F-III	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-IV	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-V	1	0.016	0.038	1.9	2.0	0.1	0.018	0.045	2.2	2.1	0.1
	2	0.017	0.042	2.1			0.017	0.042	2.1		
	3	0.017	0.042	2.0			0.017	0.042	2.1		
				Sum =	7.2	0.0			Sum =	7.4	0.1

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

* The extracts were further diluted by 10-fold before analysis.

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Table 3.44 Comparison of the extracted Pb concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S2 (Lot-I).

[F]	n	Pb									
		Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-II	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-III	1	0.008	0.156	7.7	7.7	0.1	0.008	0.156	7.7	8.4	0.4
	2	0.008	0.156	7.6			0.008	0.156	7.8		
	3	0.008	0.156	7.7			0.009	0.202	9.2		
F-IV	1	0.007	0.064	3.2	3.2	0.0	0.007	0.064	3.2	3.0	0.2
	2	0.007	0.064	3.2			0.007	0.064	3.2		
	3	0.007	0.064	3.1			0.007	0.064	2.9		
F-V	1	0.008	0.156	7.8	7.7	0.1	0.008	0.156	7.1	7.1	0.4
	2	0.008	0.156	7.8			0.008	0.156	7.8		
	3	0.008	0.156	7.6			0.008	0.156	7.1		
				Sum =	18.6	0.1			Sum =	18.5	0.6

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.45 Comparison of the extracted Pb concentration for each extraction step by using Tessier's and the optimized sequential extraction methods for S5 (Lot-I).

[F]	n	Pb									
		Tessier's method					Optimized method				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
F-I	1	ND	-	-	-	-	ND	-	-	-	-
	2	ND	-	-			ND	-	-		
	3	ND	-	-			ND	-	-		
F-II	1	0.007	0.083	3.7	3.6	0.1	0.007	0.083	3.7	3.7	0.2
	2	0.007	0.083	3.3			0.007	0.083	3.9		
	3	0.007	0.083	3.8			0.007	0.083	3.8		
F-III	1	0.008	0.174	7.6	7.8	0.1	0.008	0.174	7.7	7.9	0.2
	2	0.008	0.174	7.8			0.008	0.174	8.1		
	3	0.008	0.174	8.1			0.008	0.174	8.0		
F-IV	1	0.007	0.083	3.5	3.6	0.1	0.007	0.083	3.9	3.7	0.2
	2	0.007	0.083	3.8			0.007	0.083	3.8		
	3	0.007	0.083	3.6			0.007	0.083	3.7		
F-V	1	0.008	0.174	7.6	7.2	0.4	0.008	0.174	7.7	6.8	1.0
	2	0.008	0.128	6.1			0.008	0.128	6.0		
	3	0.008	0.174	7.8			0.008	0.128	5.9		
				Sum =	22.2	0.4			Sum =	22.1	0.9

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

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Table 3.46 Comparison of the extracted concentrations and percentage distributions of Mn, Zn, Cu, and Cd for S2 (Lot-I) by using two methods.

Fraction		Tessier's method		Optimized method	
		mg/kg[a]	%[b]	mg/kg[a]	%[b]
Mn	F-I	61.3 ± 1.2	12.1	76.3 ± 4.4	14.1
	F-II	118.5 ± 5.6	23.4	147.8 ± 4.2	27.4
	F-III	260.1 ± 3.1	51.3	260.6 ± 3.1	48.3
	F-IV	27.5 ± 0.6	5.4	31.0 ± 1.7	5.7
	F-V	39.4 ± 1.9	7.7	23.7 ± 1.3	4.4
Sum (F-I to F-V)		506.8 ± 6.8		539.4 ± 7.2	
Zn	F-I	1.4 ± 0.1	4.3	1.3 ± 0.1	4.0
	F-II	5.5 ± 0.1	16.7	5.8 ± 0.2	16.8
	F-III	8.0 ± 0.1	24.1	8.1 ± 0.0	23.7
	F-IV	5.3 ± 0.0	15.9	5.4 ± 0.0	15.7
	F-V	12.9 ± 0.0	40.0	13.7 ± 0.0	39.8
Sum (F-I to F-V)		33.1 ± 0.1		34.4 ± 0.2	
Cu	F-I	ND	-	ND	-
	F-II	ND	-	ND	-
	F-III	3.6 ± 0.0	24.4	3.1 ± 0.0	22.4
	F-IV	7.1 ± 0.1	48.0	7.0 ± 0.1	50.5
	F-V	4.1 ± 0.0	27.6	3.8 ± 0.3	27.1
Sum (F-I to F-V)		14.9 ± 0.0		13.9 ± 0.1	
Cd	F-I	2.5 ± 0.0	54.0	2.7 ± 0.0	53.3
	F-II	2.1 ± 0.0	46.0	2.3 ± 0.1	46.7
	F-III	ND	-	ND	-
	F-IV	ND	-	ND	-
	F-V	ND	-	ND	-
Sum (F-I to F-V)		4.6 ± 0.0		5.0 ± 0.1	
Pb	F-I	ND	-	ND	-
	F-II	ND	-	ND	-
	F-III	7.7 ± 0.1	41.4	8.4 ± 0.4	45.4
	F-IV	3.2 ± 0.0	17.2	3.0 ± 0.2	16.2
	F-V	7.7 ± 0.1	41.4	7.1 ± 0.1	38.4
Sum (F-I to F-V)		18.6 ± 0.1		18.5 ± 0.5	

[a] Mean ± SD (n=3).

[b] [the extracted metal concentrations from each step / the sum of metal concentrations from all step] x 100.

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Table 3.47 Comparison of the extracted concentrations and percentage distributions of Mn, Zn, Cu, and Cd for S5 (Lot-I) by using two methods.

Fraction		Tessier's method		Optimized method	
		mg/kg[a]	%[b]	mg/kg[a]	%[b]
Mn	F-I	117.8 ± 4.8	19.0	128.7 ± 3.5	20.1
	F-II	137.2 ± 9.0	22.1	144.6 ± 3.5	22.6
	F-III	289.5 ± 11.5	46.7	300.6 ± 5.8	46.9
	F-IV	43.7 ± 1.7	7.0	47.5 ± 1.7	7.4
	F-V	32.0 ± 2.5	5.2	18.9 ± 0.4	2.9
Sum (F-I to F-V)		620.2 ± 15.7		640.3 ± 7.8	
Zn	F-I	1.0 ± 0.1	2.8	1.0 ± 0.1	2.7
	F-II	5.4 ± 0.1	14.4	5.5 ± 0.1	14.9
	F-III	6.9 ± 0.1	18.5	7.1 ± 0.1	19.2
	F-IV	3.6 ± 0.1	9.6	3.7 ± 0.1	10.1
	F-V	20.4 ± 0.0	54.7	19.6 ± 0.0	52.9
Sum (F-I to F-V)		37.3 ± 0.2		37.0 ± 0.2	
Cu	F-I	ND	-	ND	-
	F-II	ND	-	ND	-
	F-III	2.3 ± 0.0	14.3	2.4 ± 0.0	14.9
	F-IV	8.3 ± 0.1	52.2	8.0 ± 0.1	50.3
	F-V	5.4 ± 0.0	33.5	5.6 ± 0.0	38.8
Sum (F-I to F-V)		16.0 ± 0.1		16.0 ± 0.1	
Cd	F-I	2.5 ± 0.0	35.4	2.6 ± 0.0	34.9
	F-II	2.6 ± 0.0	36.2	2.7 ± 0.1	36.2
	F-III	ND	-	ND	-
	F-IV	ND	-	ND	-
	F-V	2.0 ± 0.1	28.4	2.1 ± 0.0	28.9
Sum (F-I to F-V)		7.1 ± 0.1		7.4 ± 0.1	
Pb	F-I	ND	-	ND	-
	F-II	3.6 ± 0.1	16.2	3.7 ± 0.2	16.7
	F-III	7.8 ± 0.1	35.1	7.9 ± 0.2	35.8
	F-IV	3.6 ± 0.1	16.2	3.7 ± 0.2	16.7
	F-V	7.2 ± 0.4	32.5	6.8 ± 1.0	30.8
Sum (F-I to F-V)		22.2 ± 0.4		22.1 ± 0.9	

[a] Mean ± SD (n=3).

[b] [the extracted metal concentrations from each step / the sum of metal concentrations from all step] x 100.

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3.4.2 Accuracy Investigation

3.4.2.1 Accuracy of the optimized sequential extraction method

For this work, the accuracy of the sequential extraction method was evaluated by comparing the sum of metal concentrations found in all fractions [S] with total metal concentrations using hot-acid digestion [T]. The experimental procedure for hot-acid digestion method that was shown in Section 2.8. For S2 and S5 (Lot-I), Table 3.48 shows the result of total concentration of Mn, Zn, Cu, Pb, and Cd obtained from hot-acid digestion. The accuracy of the optimized sequential extraction method expressed as percentage recoveries of heavy metals is shown in Table 3.49. It was found that the recoveries of selected metals for the optimized sequential extraction method were in the range of 89.6-100.9% and 86.8-98.7% for S2 and S5, respectively. Although the recoveries of Pb for both sediments were low (89.6% for S2 and 86.8% for S5), those of Mn, Zn, Cu, and Cd were acceptable in the range of 91.4-100.9%.

3.4.2.2 Accuracy of Hot-Acid Digestion

To check the accuracy of hot-acid digestion for investigation of total metal concentration, spiked samples were prepared as described in Section 2.9. As the results, the recoveries of selected metals obtained from hot-acid digestion are shown in Table 3.50 and 3.51 for S7 and S8 (Lot-I), respectively. It was found that the recoveries of selected metals were in the range of 95.3-102.1% and 93.8-101.5% for S7 (Lot-I) and S8 (Lot-I), respectively. These results show good accuracy for hot-acid digestion method that was used to determine metals participated in residual fraction or total metal contents in original sediments.

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Table 3.48 Total concentrations of Mn, Zn, Cu, Cd, and Pb obtained from hot-acid digestion for S2 and S5 (Lot-I).

metal	n	Total metal concentrations (n=3)									
		S2					S5				
		Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD	Abs[1].	Conc.[2]	Conc.[3]	Ave.[4]	SD
Mn*	1	0.148	1.191	581.5	576.2	2.5	0.162	1.306	643.8	651.1	4.2
	2	0.145	1.166	576.3			0.163	1.314	648.5		
	3	0.145	1.166	570.8			0.166	1.339	661.0		
Zn	1	0.287	0.701	34.2	34.1	0.1	0.321	0.785	38.7	38.5	0.1
	2	0.284	0.693	34.3			0.318	0.778	38.4		
	3	0.282	0.688	33.7			0.318	0.778	38.4		
Cu	1	0.030	0.297	14.5	14.6	0.1	0.035	0.348	17.1	17.0	0.1
	2	0.029	0.287	14.7			0.034	0.338	16.7		
	3	0.030	0.297	14.5			0.035	0.348	17.2		
Cd	1	0.034	0.113	5.5	5.5	0.0	0.043	0.145	7.1	7.1	0.0
	2	0.033	0.109	5.4			0.042	0.144	7.1		
	3	0.034	0.113	5.5			0.042	0.144	7.1		
Pb	1	0.100	0.569	20.8	20.2	0.3	0.009	0.488	24.0	22.7	1.1
	2	0.009	0.488	20.1			0.008	0.407	20.1		
	3	0.009	0.488	19.6			0.009	0.488	24.1		

[1] Average absorbance.

[2] Concentration (mg/L).

[3] Concentration (mg/kg, dry weight).

[4] Average concentration (mg/kg, dry weight).

Volumes of the extracts were prepared as 50 mL before analysis.

* The extracts were further diluted by 10-fold before analysis.

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Table 3.49 Percentage recoveries obtained by comparing the sum of the extracted metal concentrations for the optimized sequential extraction method and hot-acid digestion.

Element	S2 (mg/kg ± SD, n=3)			S5 (mg/kg ± SD, n=3)		
	[S]	[T]	% Recovery[a]	[S]	[T]	% Recovery[a]
Mn	539.4 ± 7.2	576.2 ± 2.5	93.6	640.3±7.8	651.1 ± 4.2	98.3
Zn	34.4 ± 0.2	34.1 ± 0.1	100.9	37.1±0.3	38.5 ± 0.1	96.1
Cu	13.9 ± 0.1	14.6 ± 0.1	95.2	16.0±0.1	17.0 ± 0.1	94.1
Pb	18.1 ± 0.2	20.2 ± 0.3	89.6	19.7±0.2	22.7 ± 1.1	86.8
Cd	5.0 ± 0.1	5.5 ± 0.0	91.4	7.0±0.1	7.1 ± 0.0	98.7
Cr	ND	ND	-	ND	ND	-

[a] [S]/[T]x100.

Table 3.50 Percentage recoveries of Mn, Zn, Cu, Pb, Cd, and Cr obtained from hot-acid digestion for S7 (Lot-I) after spiking method.

Element	S7 (mg/kg ± SD, n=3)			
	C_M (mg/kg)	C_A (mg/kg)	C_s (mg/L)	%Recovery[a]
Mn	1179.0 ± 14.0	1071.7 ± 13.9	100.0	100.7
Zn	132.2 ± 1.9	31.3 ± 1.5	100.0	100.7
Cu	119.7 ± 1.9	21.1 ± 0.4	100.0	98.8
Pb	112.9 ± 6.8	15.3 ± 0.3	100.0	97.9
Cd	102.1 ± 0.8	3.6 ± 0.2	100.0	98.6
Cr	95.3 ± 1.5	ND	100.0	95.3

C_M: metal concentration found for spiked sample (mg/kg).

C_A: metal concentration found for non-spiked sample (mg/kg).

C_S: metal concentration of standard solution added (mg/L).

[a] $[(C_M)/C_S+C_A] \times 100$.

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Table 3.51 Percentage recoveries of Mn, Zn, Cu, Pb, Cd, and Cr obtained from hot-acid digestion for S8 (Lot-I) after spiking method.

Element	S8 (mg/kg ± SD, n=3)			
	C_M (mg/kg)	C_A (mg/kg)	C_s (mg/L)	%Recovery[a]
Mn	1106.8 ± 12.3	1024.0 ± 1.5	100.0	98.5
Zn	128.0 ± 3.1	33.1 ± 2.0	100.0	96.2
Cu	118.6 ± 2.6	20.7 ± 0.6	100.0	98.2
Pb	127.5 ± 14.1	26.8 ± 4.6	100.0	100.5
Cd	103.7 ± 3.1	7.9 ± 0.2	100.0	95.4
Cr	93.8 ± 1.3	ND	100.0	93.8

C_M: metal concentration found for spiked sample (mg/kg).

C_A: metal concentration found for non-spiked sample (mg/kg).

C_S: metal concentration of standard solution added (mg/L).

[a] $[(C_M)/C_S+C_A] \times 100$.

3.4.3 Precision of the Optimized Sequential Extraction Method

Using the optimized sequential extraction method, the precision was also investigated by repeatable extraction for six replicates for all fractions. The repeatabilities of the optimized sequential extraction method were expressed as the relative standard deviation (%RSD). Table 3.52, 3.53, 3.54, 3.55, and 3.56 show the relative standard deviation of the optimized sequential extraction method for Mn, Zn, Cu, Cd, and Pb, respectively. It was found that the relative standard deviation of the optimized sequential extraction method were in the range of 3.1-4.8, 1.4-4.6, 2.3-4.8, 3.4-3.9, and 1.7-4.7% for Mn, Zn, Cu, Cd, and Pb, respectively. This indicates that the optimized sequential extraction method provided good repeatability (<5%, n=6).

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Table 3.52 The results of the extracted Mn concentration obtained for study of the precision of sequential extraction for mixed sample (KN3+KN4).

Mn										
n	F-I		F-II*		F-III*		F-IV		F-V	
	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]
1	1.109	55.1	0.866	215.3	1.364	338.9	0.391	19.4	0.678	33.7
2	1.120	55.6	0.922	228.9	1.408	349.6	0.402	20.0	0.690	34.2
3	1.209	59.8	0.944	233.6	1.452	359.4	0.435	21.5	0.667	33.0
4	1.131	56.6	0.910	227.4	1.319	329.8	0.280	19.0	0.667	33.0
5	1.198	59.3	0.910	225.5	1.441	356.8	0.413	20.5	0.734	36.3
6	1.131	56.2	0.877	218.0	1.364	338.7	0.424	21.1	0.701	34.8
Mean	57.1		224.8		345.5		20.2		34.2	
SD	2.0		6.9		11.7		1.0		1.2	
%RSD	3.5		3.1		3.4		4.8		3.5	

[1] Concentration (mg/L), [2] Concentration (mg/kg, dry wt.).

* The extracts were further diluted by 5-fold before analysis.

Table 3.53 The results of the extracted Zn concentration obtained for study of the precision of sequential extraction for mixed sample (KN3+KN4).

Zn										
n	F-I		F-II		F-III		F-IV		F-V	
	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]
1	0.030	1.5	0.070	3.5	0.146	7.2	0.103	5.1	0.409	20.3
2	0.030	1.5	0.068	3.4	0.150	7.5	0.106	5.2	0.414	20.5
3	0.033	1.6	0.073	3.6	0.148	7.3	0.113	5.6	0.423	20.9
4	0.028	1.4	0.068	3.4	0.153	7.6	0.106	5.3	0.402	20.1
5	0.030	1.5	0.070	3.5	0.150	7.4	0.110	5.5	0.416	20.6
6	0.030	1.5	0.066	3.3	0.157	7.8	0.106	5.2	0.414	20.5
Mean	1.5		3.4		7.5		5.3		20.5	
SD	0.1		0.1		0.2		0.2		0.3	
%RSD	4.6		3.4		2.8		3.1		1.4	

[1] Concentration (mg/L), [2] Concentration (mg/kg, dry wt.).

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Table 3.54 The results of the extracted Cu concentration obtained for study of the precision of sequential extraction for mixed sample (KN3+KN4).

Cu										
n	F-I		F-II		F-III		F-IV		F-V	
	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]
1	ND	-	ND	-	0.048	2.4	0.070	3.5	0.112	5.6
2	ND	-	ND	-	0.048	2.4	0.070	3.5	0.112	5.6
3	ND	-	ND	-	0.048	2.4	0.077	3.8	0.112	6.1
4	ND	-	ND	-	0.045	2.2	0.070	3.4	0.112	5.6
5	ND	-	ND	-	0.048	2.4	0.077	3.8	0.112	5.6
6	ND	-	ND	-	0.048	2.4	0.070	3.4	0.112	5.6
Mean	-		-		2.4		3.6		5.7	
SD	-		-		0.1		0.2		0.2	
%RSD	-		-		2.3		4.8		3.5	

[1] Concentration (mg/L), [2] Concentration (mg/kg, dry wt.).

Table 3.55 The results of the extracted Cd concentration obtained for study of the precision of sequential extraction for mixed sample (KN3+KN4).

Cd										
n	F-I		F-II		F-III		F-IV		F-V	
	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]
1	0.054	2.7	0.047	2.3	ND	-	ND	-	ND	-
2	0.054	2.7	0.047	2.3	ND	-	ND	-	ND	-
3	0.057	2.8	0.050	2.5	ND	-	ND	-	ND	-
4	0.050	2.5	0.044	2.2	ND	-	ND	-	ND	-
5	0.054	2.6	0.047	2.3	ND	-	ND	-	ND	-
6	0.054	2.7	0.047	2.3	ND	-	ND	-	ND	-
Mean	2.7		2.3		-		-		-	
SD	0.1		0.1		-		-		-	
%RSD	3.4		3.9		-		-		-	

[1] Concentration (mg/L), [2] Concentration (mg/kg, dry wt.).

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Table 3.56 The results of the extracted Pb concentration obtained for study of the precision of sequential extraction for mixed sample (KN3+KN4).

Pb										
n	F-I		F-II		F-III		F-IV		F-V	
	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]	Conc.[1]	Conc.[2]
1	ND	-	0.059	2.9	0.142	7.1	0.060	3.0	0.158	7.9
2	ND	-	0.059	2.9	0.145	7.2	0.062	3.1	0.155	7.7
3	ND	-	0.058	2.9	0.149	7.4	0.061	3.0	0.161	8.0
4	ND	-	0.054	2.7	0.142	7.1	0.060	3.0	0.158	7.9
5	ND	-	0.056	2.8	0.146	7.2	0.063	3.1	0.154	7.6
6	ND	-	0.053	2.6	0.148	7.4	0.064	3.2	0.159	7.9
Mean	-		2.8		7.2		3.1		7.8	
SD	-		0.1		0.1		0.1		0.1	
%RSD	-		4.7		1.8		2.5		1.7	

[1] Concentration (mg/L), [2] Concentration (mg/kg, dry wt.).

3.5 The Optimized Sequential Extraction Method for Sample Analysis

The optimized sequential extraction method was applied for the determination of heavy metals in sediments collected from the Kwai Noi River at Kanchanaburi Province. The sediment samples collected on August, 2001 (Lot-I) and March, 2002 (Lot-II) were determined. The extracted metal concentrations and metals distributed in different sediment phases are reported as below.

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3.5.1 Metal Analysis using the Optimized Sequential Extraction Method for Sediment Samples (Lot-I)

After applying the optimized sequential extraction method to 12 samples for Lot-I, it was found that the sums of metal concentrations obtained from

five extraction steps were in the range of 566.6-1139.3, 27.9-46.3, 9.9-22.0, 16.8-25.7, and 4.6-10.0, mg/kg for Mn, Zn, Cu, Pb, and Cd, respectively. The extracted Cr concentrations were not detected for all fractions. The metal distribution in sediments for an individual fraction was expressed as percentage obtained by comparing the extracted metal concentrations for each fraction with the sum of metal concentrations from five fractions. The accuracy of the optimized sequential extraction method was expressed as percentage recoveries calculated by comparing the sum of metal concentrations obtained from five extraction steps [S] with total metal concentration obtained from hot-acid digestion [T].

As shown in Table 3.57, the extracted Mn concentrations obtained from all samples were found in the range of 39.6-168.3, 91.5-401.9, 232.7-488.0, 12.0-63.1, and 12.6-29.2 mg/kg for F-I, F-II, FIII, F-IV, and F-V, respectively. These results indicate that the extracted Mn concentrations were extremely high for bound to Fe-Mn oxide fraction (F-III). As can be seen in Figure 3.14, the percentages of Mn distributed in each fraction were in the range of 8.3-20.3, 19.1-38.4, 36.1-62.7, 1.8-7.5, and 1.8-8.3% for F-I, F-II, F-III, F-IV, and F-V, respectively. These results indicate that most of Mn proportion was associated with oxides of Fe-Mn rather than another phase. This is probably related to abundant of Mn in the earth's crust. Comparing to total Mn concentration, percentage recoveries of the extracted Mn obtained from the optimized sequential extraction method were found in the range of 94.6-100.9%. Navas *et al.* [18] using Tessier's method for soils found that Mn was mostly associated to carbonates and oxides of Fe-Mn (>80.0%). In soil and particulate materials of aquatic environments, Fe and Mn oxides or hydroxides have commonly occurred as coating on minerals. These oxides and hydroxides readily

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sorb or precipitate metal ions. Under reducing conditions, sorbed heavy metals could easily be mobilized into aquatic systems. The mechanism of heavy metal sorption on hydrous oxides could be suggested that metal cations were incorporated in the stren layer of hydroxide structure and hydrogen ions were exchanged [8].

The results of the extracted Zn concentrations for an individual fraction were found in the range of ND-2.3, 2.3-6.0, 3.1-11.9, 4.1-6.7, and 13.1-24.9 mg/kg for F-I, F-II, FIII, F-IV, and F-V, respectively as shown in Table 3.58. The extracted Zn concentrations from all sediments were high for the residue fraction (38.2-68.5%). This indicates that Zn in sediments could be bound into major components of sediments and could not be easily released under natural conditions. On the other hand, the extracted Zn concentrations were low under other extraction conditions such as exchangeable (1.9-7.9%), acid-soluble (5.2-17.5%), reducing (9.1-27.3%), and oxidizing conditions (10.3-20.4%) as shown in Figure 3.15. Comparing to total Zn concentration, percentage recoveries of the extracted Zn obtained from the optimized sequential extraction method were in the range of 96.2-106.5%.

The results in Table 3.59 indicated that the extracted Cu concentrations from different sediment samples were in the range of ND, ND-1.5, 1.5 -7.8, 4.4-9.7, and 2.6-5.5 mg/kg for F-I, F-II, F-III, F-IV, and F-V, respectively. As can be seen in Figure 3.16, a high proportion about 36.4-61.5% of total Cu concentration was associated to organic matter in sediment. This agrees with the results of many studies [31, 41, 45, 51], which concluded that a high proportion of Cu in sediments might form part of organic matter. As reported that copper has been preferentially sorbed by organic materials by complexing mechanism [8]. The order of bonding strength for a number of metal ions onto humic or fulvic acids was Hg^{2+}> Cu^{2+}> Pb^{2+}> Zn^{2+}> Ni^{2+}>

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Co^{2+}> Mn^{2+}. Under other extraction conditions, percentages of Cu distributed in sediment samples were in range of <6.8, 1.5-35.5, and 20.9-35.2% for acid-soluble, reducible, and strong acid conditions, respectively. Comparing to total Cu concentration, percentage recoveries of the extracted Cu concentrations obtained from the optimized sequential extraction method were in the range of 94.6-105.2%.

As shown in Table 3.60, the results of the extracted Pb concentrations were found in the range of ND, ND-3.7, 5.5-10.9, 2.9-5.1, and 5.2-11.3 mg/kg for F-I, F-II, FIII, F-IV, and F-V, respectively. With the exception of exchangeable condition (F-I), the extracted Pb concentrations could be detected in most extraction conditions such as acid-soluble (8.7-16.9%), reducible (28.4-46.3%), oxidizable (14.1-24.3%), and strong acid conditions (26.9-47.5%) as seen from Figure 3.17. Comparing to total Pb concentration, percentage recoveries of the extracted Pb concentrations obtained from the optimized sequential extraction method were in the range of 84.3-108.3%. Hubsher *et al.* [38] used sequential extraction for solid waste samples and found that Pb concentration could be extracted under all conditions studied, but its concentration was high for acid-soluble and strong acid conditions. Under oxidizable condition, they suggested that Pb ions might be bound to mineral substance in sediment such as sulfides which is unstable under this condition.

As shown in Table 3.61, the results of the extracted Cd concentrations were in the range of 1.5-3.5, 2.1-4.6, ND, ND, and ND-2.5 mg/kg for F-I, F-II, FIII, F-IV, and F-V, respectively. As can be seen in Figure 3.18, Cd concentrations could be extracted under exchangeable (25.9-53.4%), acid-soluble (32.5-58.9%), and strongly acidic conditions (19.0-36.1%). This indicates that Cd in sediments might be easily released into environment under natural conditions known as the mobile forms

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(available fraction). Li *et al.* [31] reported that about 5-29% of total Cd content was associated to exchangeable fraction. This agreed to the result proposed by Davidson *et al.* [37] which reported that the extractable Cd concentration was high for exchangeable and carbonate phases. Percentage recoveries of the extracted Cd concentrations obtained from the optimized sequential extraction method in sediments were in the range of 79.3-102.4%. The extracted Cd concentrations were high for both exchangeable and bound to carbonate fractions.

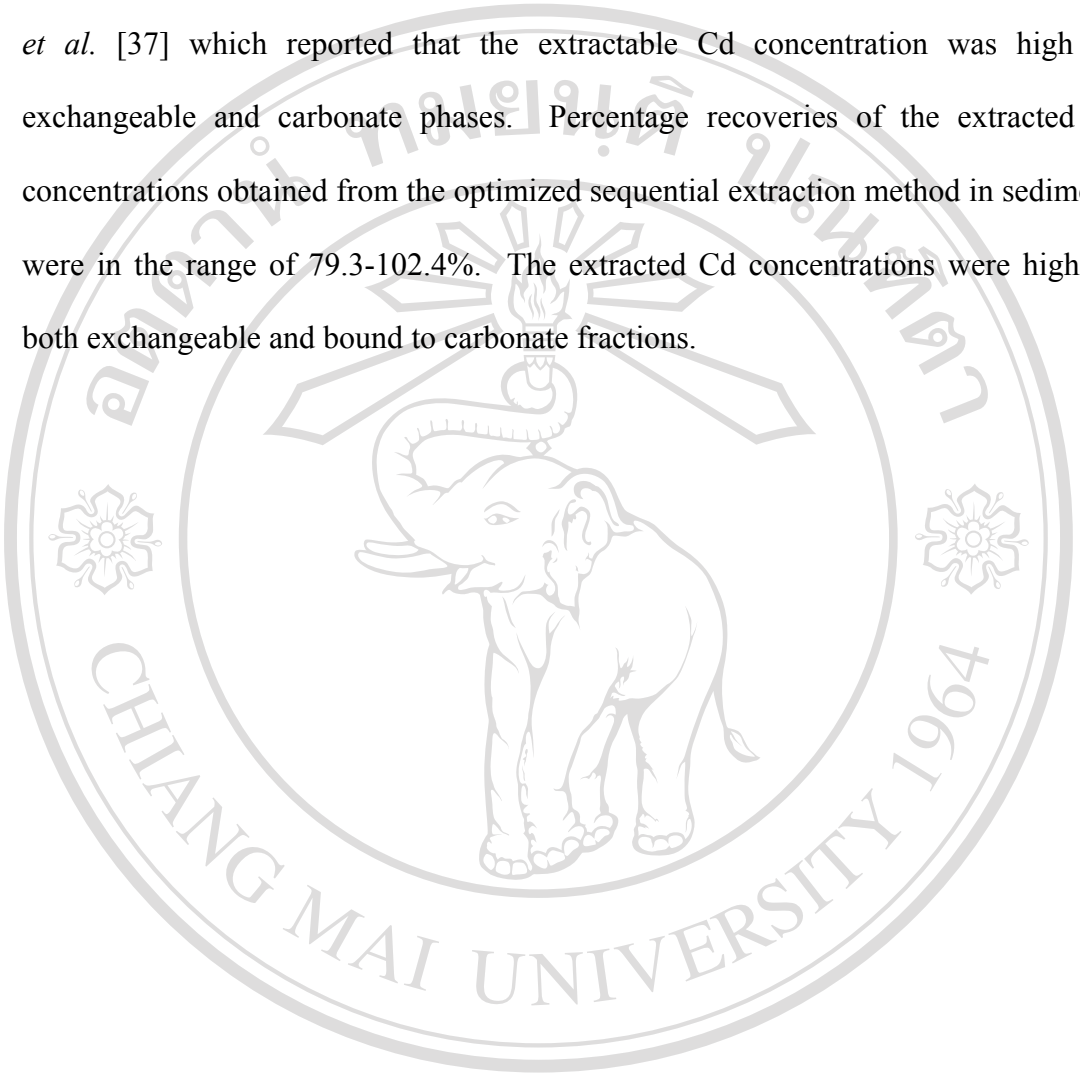

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Table 3.57 Average concentrations of Mn distributed in each fraction of sediments collected from different sample sites (Lot-I), which extracted by the optimized sequential extraction method.

Sample site	Mn (mg/kg ± SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	142.7 ± 3.4	237.5 ± 7.3	278.1 ± 7.7	36.1 ± 0.6	12.6 ± 0.6	707.0 ± 11.2	700.4 ± 22.2	100.9
KN2	77.9 ± 2.0	147.5 ± 7.8	266.8 ± 11.0	29.7 ± 1.1	24.3 ± 1.6	546.1 ± 13.8	577.9 ± 33.9	94.6
KN3	78.9 ± 1.0	231.6 ± 9.8	315.3 ± 7.0	12.0 ± 0.8	25.3 ± 1.2	663.0 ± 12.2	685.1 ± 16.2	96.8
KN4	39.6 ± 0.7	91.5 ± 6.4	299.6 ± 6.5	13.4 ± 0.5	36.0 ± 1.3	480.1 ± 9.3	504.6 ± 22.1	95.1
KN5	129.0 ± 3.1	143.2 ± 0.6	297.0 ± 2.8	47.5 ± 0.4	19.2 ± 1.3	635.9 ± 14.4	646.3 ± 24.0	98.4
KN6	88.1 ± 1.0	247.3 ± 6.9	232.7 ± 5.6	41.1 ± 1.3	34.6 ± 1.4	643.8 ± 9.1	735.2 ± 12.2	87.6
KN7	168.3 ± 5.5	401.9 ± 8.7	488.0 ± 14.4	34.7 ± 1.2	46.5 ± 1.5	1139.5 ± 17.8	1162.5 ± 14.1	98.0
KN8	151.2 ± 10.0	342.5 ± 11.1	468.9 ± 7.3	39.1 ± 1.4	51.7 ± 1.8	1053.4 ± 16.9	1052.2 ± 18.2	100.1
KN9	148.0 ± 7.9	342.9 ± 9.8	393.4 ± 8.2	53.7 ± 2.7	70.9 ± 3.2	1008.9 ± 15.2	1068.7 ± 11.3	94.4
KN10	128.7 ± 4.1	347.7 ± 12.0	363.4 ± 6.7	63.1 ± 3.3	81.3 ± 2.3	984.2 ± 14.9	994.0 ± 44.7	99.0
KN11	116.5 ± 12.9	177.6 ± 7.2	273.4 ± 13.6	42.7 ± 2.1	32.7 ± 1.9	642.9 ± 20.3	657.4 ± 15.6	97.8
KN12	70.4 ± 1.7	176.6 ± 13.9	271.3 ± 7.4	31.1 ± 1.6	26.8 ± 1.5	576.2 ± 16.0	568.4 ± 28.0	98.2

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion.

% Recovery = [S]/[T]x100.

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Table 3.58 Average concentrations of Zn distributed in each fraction of sediments collected from different sample sites (Lot-I) , which extracted by the optimized sequential extraction method.

Sample site	Zn (mg/kg ± SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	2.3 ± 0.1	3.7 ± 0.4	5.8 ± 0.1	4.8 ± 0.2	13.1 ± 0.4	29.7 ± 0.7	30.8 ± 0.1	96.4
KN2	1.4 ± 0.1	6.0 ± 0.3	8.1 ± 0.4	5.8 ± 0.2	13.1 ± 0.3	34.3 ± 0.6	32.2 ± 1.3	106.5
KN3	2.1 ± 0.1	2.3 ± 0.1	8.9 ± 0.3	4.5 ± 0.1	15.1 ± 0.3	32.9 ± 0.5	31.7 ± 0.7	103.8
KN4	1.3 ± 0.1	2.3 ± 0.2	10.4 ± 0.3	6.7 ± 0.2	24.2 ± 0.3	44.9 ± 0.6	41.8 ± 0.5	100.2
KN5	0.9 ± 0.1	4.2 ± 0.5	7.9 ± 0.4	4.0 ± 0.1	18.8 ± 0.6	36.0 ± 0.9	37.0 ± 1.0	97.3
KN6	1.8 ± 0.1	3.0 ± 0.2	11.9 ± 0.3	4.8 ± 0.3	24.9 ± 0.5	46.3 ± 0.7	46.7 ± 0.6	99.1
KN7	0.8 ± 0.1	2.8 ± 0.3	7.9 ± 0.3	6.1 ± 0.3	18.7 ± 0.5	36.3 ± 0.7	35.5 ± 0.5	102.1
KN8	0.8 ± 0.1	4.5 ± 0.1	3.4 ± 0.3	4.1 ± 0.2	24.4 ± 0.8	37.2 ± 0.9	37.9 ± 0.6	98.1
KN9	0.7 ± 0.1	2.9 ± 0.1	3.4 ± 0.2	4.1 ± 0.2	24.4 ± 0.2	35.6 ± 0.4	37.0 ± 0.1	96.2
KN10	0.9 ± 0.2	3.6 ± 0.3	4.5 ± 0.2	5.6 ± 0.2	22.4 ± 0.4	37.1 ± 0.6	37.8 ± 0.3	98.1
KN11	0.8 ± 0.0	2.6 ± 0.3	3.1 ± 0.1	6.2 ± 0.3	17.6 ± 0.4	30.4 ± 0.6	30.0 ± 1.1	101.3
KN12	ND	2.5 ± 0.2	4.8 ± 0.1	4.7 ± 0.3	15.8 ± 0.4	27.9 ± 0.5	28.3 ± 1.3	98.6

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion, ND: Not detected.

% Recovery = [S]/[T]x100.

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Table 3.59 Average concentrations of Cu distributed in each fraction of sediments collected from different sample sites (Lot-I), which extracted by the optimized sequential extraction method.

Sample site	Cu (mg/kg ± SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	ND	ND	2.8 ± 0.3	9.7 ± 0.5	3.3 ± 0.5	15.8 ± 0.8	16.7 ± 0.6	94.6
KN2	ND	ND	2.4 ± 0.6	7.0 ± 0.5	3.8 ± 0.3	13.2 ± 0.8	12.5 ± 1.5	103.6
KN3	ND	ND	4.0 ± 0.3	9.5 ± 1.0	4.1 ± 0.5	17.5 ± 0.8	16.9 ± 0.7	104.1
KN4	ND	ND	2.8 ± 0.3	6.6 ± 1.0	3.3 ± 0.3	12.7 ± 1.1	12.5 ± 0.9	101.6
KN5	ND	ND	2.4 ± 0.3	8.8 ± 0.3	4.9 ± 0.3	16.1 ± 1.1	16.8 ± 0.6	95.8
KN6	ND	ND	1.8 ± 0.3	5.3 ± 0.2	3.6 ± 0.0	10.7 ± 0.4	11.1 ± 0.6	96.4
KN7	ND	1.2 ± 0.3	3.7 ± 0.4	8.0 ± 0.3	5.5 ± 0.3	18.4 ± 0.6	18.1 ± 0.5	101.7
KN8	ND	1.5 ± 0.2	7.8 ± 0.2	8.0 ± 0.5	4.7 ± 0.5	21.9 ± 0.6	20.8 ± 1.6	105.2
KN9	ND	ND	3.8 ± 0.4	7.4 ± 0.5	3.7 ± 0.2	14.9 ± 0.7	15.1 ± 0.3	98.7
KN10	ND	ND	2.7 ± 0.0	5.4 ± 0.4	2.6 ± 0.2	10.6 ± 0.4	10.5 ± 1.0	100.9
KN11	ND	ND	2.4 ± 0.5	5.4 ± 0.3	3.2 ± 0.3	11.1 ± 0.6	11.1 ± 0.3	100.0
KN12	ND	ND	1.5 ± 0.0	4.8 ± 0.4	3.4 ± 0.1	9.76 ± 0.5	10.2 ± 0.9	95.7

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion, ND: Not detected.

% Recovery = [S]/[T]x100.

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Table 3.60 Average concentrations of Pb distributed in each fraction of sediments collected from different sample sites (Lot-I), which extracted by the optimized sequential extraction method.

Sample site	Pb (mg/kg ± SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	ND	3.0 ± 1.1	8.9 ± 0.0	4.4 ± 0.0	8.9 ± 0.0	25.2 ± 1.2	29.9 ± 3.4	84.3
KN2	ND	ND	8.8 ± 0.0	2.9 ± 1.0	7.4 ± 1.2	19.1 ± 1.7	19.3 ± 2.5	99.0
KN3	ND	ND	8.9 ± 1.0	3.0 ± 1.0	5.9 ± 1.3	17.2 ± 2.7	17.7 ± 2.3	97.2
KN4	ND	2.3 ± 0.1	10.9 ± 1.4	3.8 ± 1.5	9.5 ± 0.2	26.6 ± 2.1	27.2 ± 2.2	97.8
KN5	ND	3.7 ± 1.4	8.0 ± 2.5	3.7 ± 0.5	6.5 ± 1.2	21.9 ± 1.9	24.1 ± 2.9	90.9
KN6	ND	2.3 ± 0.2	8.1 ± 0.1	3.8 ± 0.1	6.7 ± 0.2	20.8 ± 1.0	23.5 ± 2.5	88.5
KN7	ND	2.3 ± 0.5	8.1 ± 2.5	3.8 ± 0.5	5.2 ± 0.0	19.5 ± 0.2	20.1 ± 0.2	97.0
KN8	ND	ND	8.0 ± 2.9	5.1 ± 0.0	8.0 ± 2.4	21.1 ± 5.5	22.6 ± 4.2	93.4
KN9	ND	ND	7.9 ± 2.3	3.6 ± 0.4	6.5 ± 2.4	18.0 ± 4.1	17.6 ± 3.4	102.3
KN10	ND	ND	5.5 ± 2.5	4.1 ± 0.5	9.9 ± 1.5	19.5 ± 4.3	19.7 ± 4.7	99.0
KN11	ND	ND	6.9 ± 0.0	4.0 ± 0.5	9.8 ± 1.5	20.8 ± 3.5	19.2 ± 1.8	108.3
KN12	ND	ND	8.4 ± 2.4	4.1 ± 0.5	11.3 ± 0.1	23.8 ± 2.5	27.5 ± 0.2	86.5

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion, ND: Not detected.

% Recovery = [S]/[T]x100.

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Table 3.61 Average concentrations of Cd distributed in each fraction of sediments collected from different sample sites (Lot-I), which extracted by the optimized sequential extraction method.

Sample site	Cd (mg/kg ± SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	2.3 ± 0.2	2.2 ± 0.1	ND	ND	2.2 ± 0.1	6.7 ± 0.2	6.5 ± 0.1	102.4
KN2	2.6 ± 0.2	2.3 ± 0.2	ND	ND	ND	5.0 ± 0.2	5.1 ± 0.1	96.7
KN3	2.4 ± 0.1	2.1 ± 0.1	ND	ND	ND	4.6 ± 0.1	5.8 ± 0.1	79.3
KN4	3.5 ± 0.2	4.6 ± 0.3	ND	ND	1.9 ± 0.1	10.0 ± 0.4	10.3 ± 0.2	97.0
KN5	2.5 ± 0.2	2.7 ± 0.3	ND	ND	2.0 ± 0.2	7.2 ± 0.4	7.8 ± 0.2	92.8
KN6	2.4 ± 0.1	2.4 ± 0.3	ND	ND	2.2 ± 0.1	7.1 ± 0.4	7.7 ± 0.0	91.9
KN7	1.5 ± 0.1	2.2 ± 0.3	ND	ND	2.1 ± 0.3	5.8 ± 0.4	6.5 ± 0.2	88.1
KN8	2.3 ± 0.2	3.2 ± 0.3	ND	ND	2.5 ± 0.1	8.0 ± 0.4	8.4 ± 0.2	95.2
KN9	2.0 ± 0.1	2.6 ± 0.1	ND	ND	2.2 ± 0.1	6.8 ± 0.2	7.3 ± 0.4	94.2
KN10	2.2 ± 0.1	3.0 ± 0.2	ND	ND	2.1 ± 0.1	7.3 ± 0.3	7.9 ± 0.1	91.7
KN11	2.2 ± 0.1	3.1 ± 0.3	ND	ND	ND	5.3 ± 0.4	5.3 ± 0.3	98.5
KN12	2.2 ± 0.0	3.1 ± 0.1	ND	ND	2.0 ± 0.1	7.3 ± 0.2	7.7 ± 0.2	94.8

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion, ND: Not detected.

% Recovery = [S]/[T]x100.

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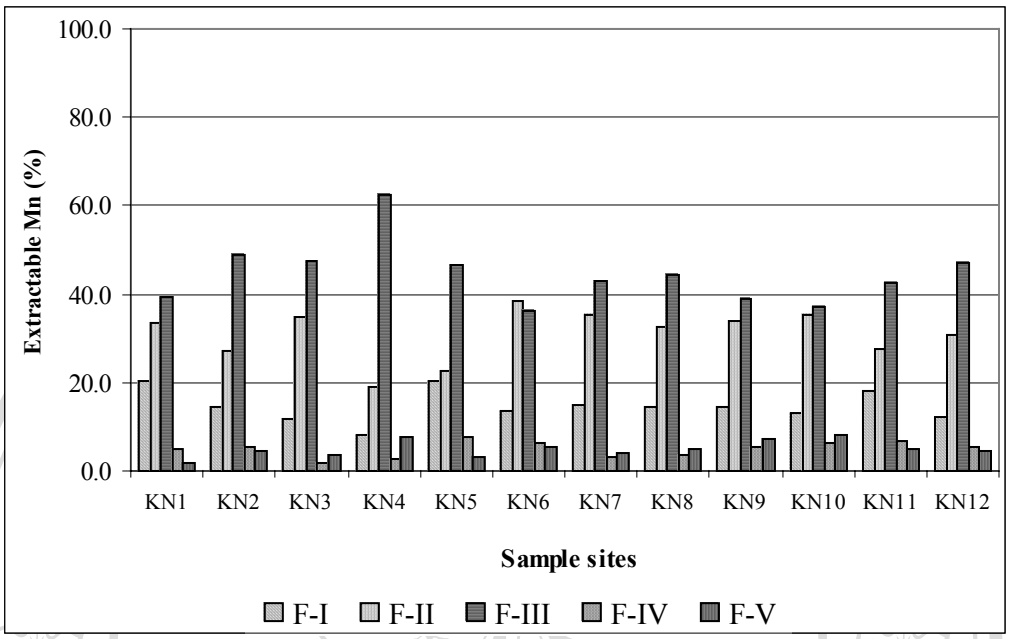

Figure 3.14 Percentages of Mn distribution in each fraction from different sediment samples (Lot-I).

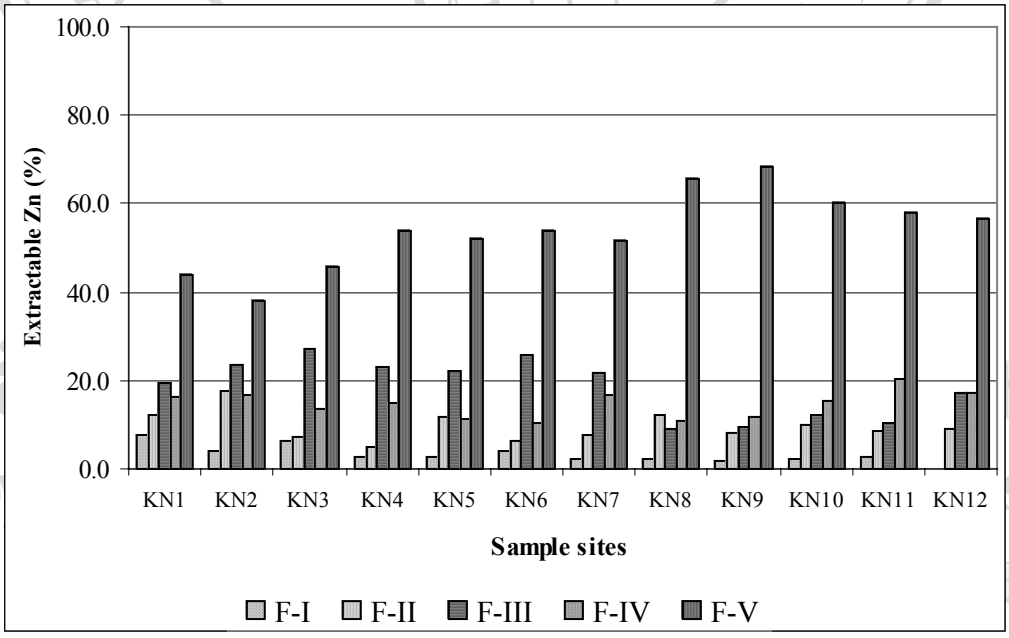

Figure 3.15 Percentages of Zn distributions in each fraction from different sediment samples (Lot-I).

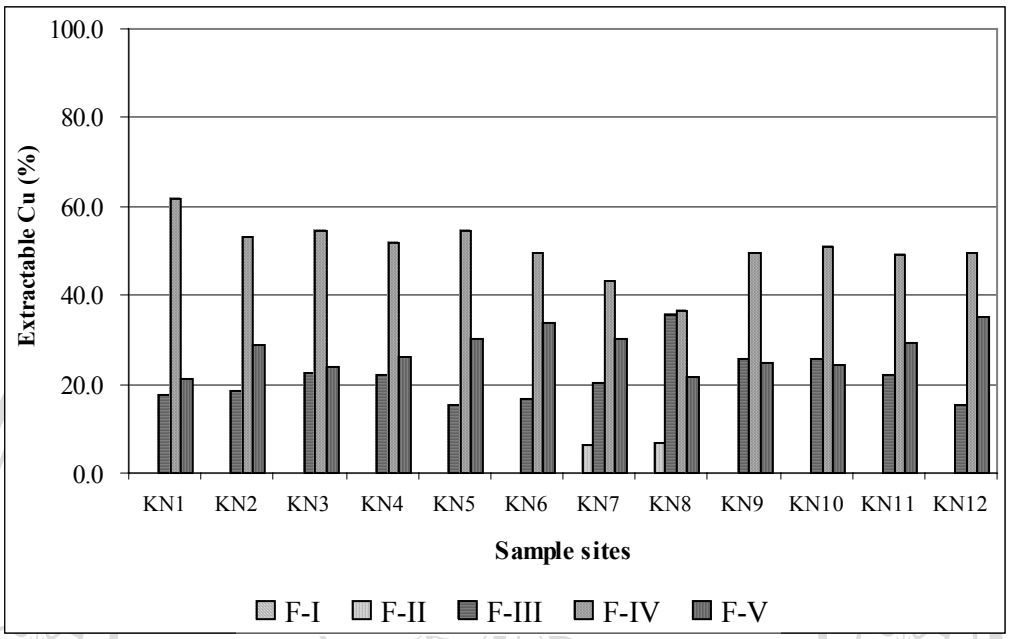

Figure 3.16 Percentages of Cu distribution in each fraction from different sediment samples (Lot-I).

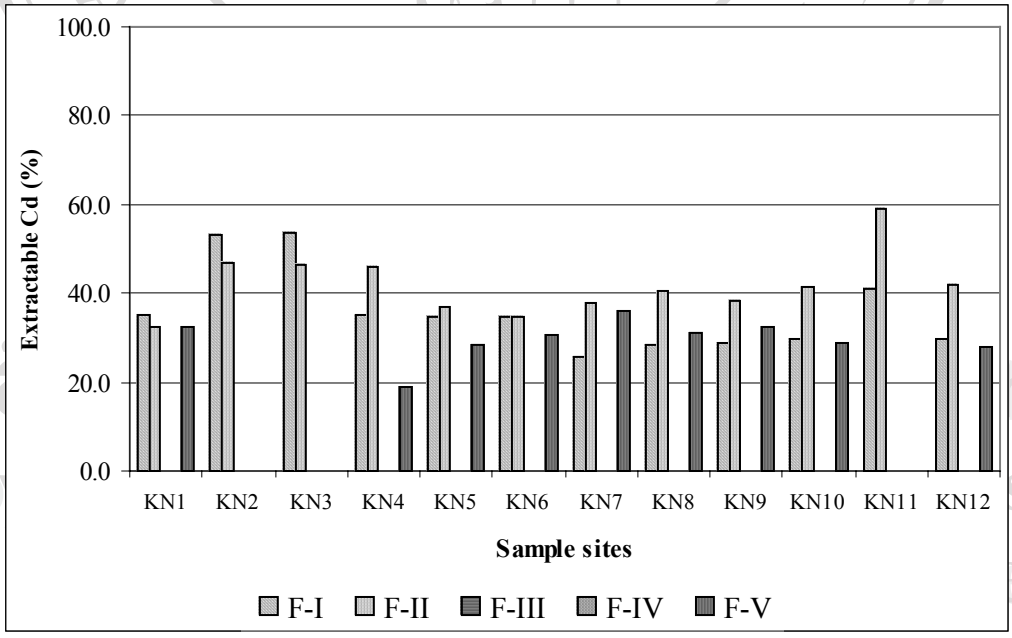

Figure 3.17 Percentages of Pb distribution in each fraction from different sediment samples (Lot-I).

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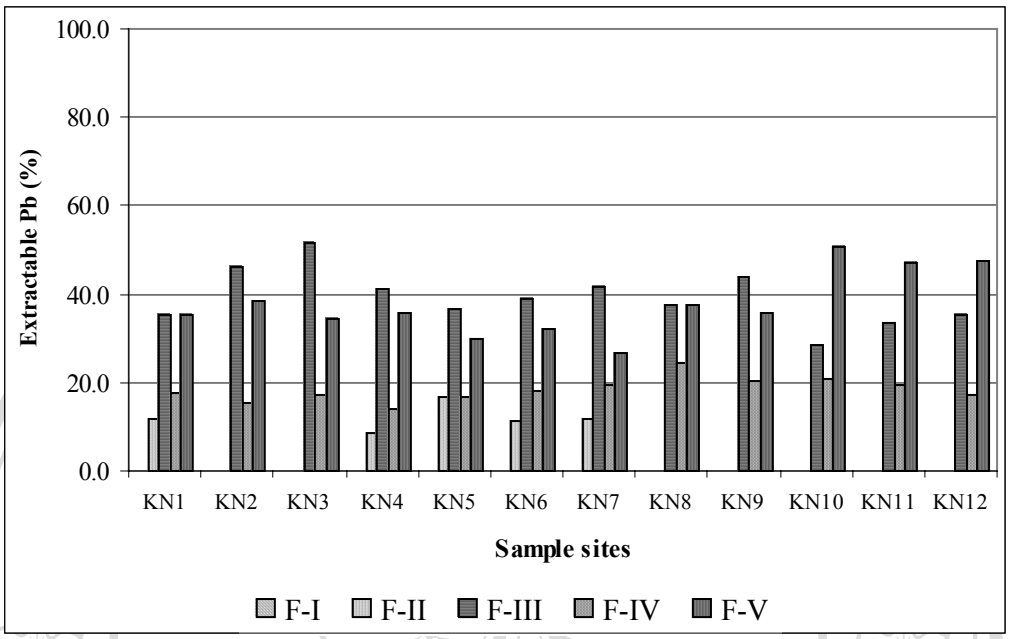

Figure 3.18 Percentages of Cd distribution in each fraction from different sediments samples (Lot-I).

3.5.2 Metal Analysis using the Optimized Sequential Extraction Method for Sediment Samples (Lot-II)

For investigating the extractable metal concentrations from 12 different sediment samples for Lot-II, it was found that the sums of the extracted metal concentrations were found in the range of 539.7-1142.7, 25.3-47.9, 11.4-20.2, 16.5-27.1, and 4.7-8.7, mg/kg for Mn, Zn, Cu, Pb, and Cd, respectively. For all sediment samples, the Cr concentration was not detected

As shown in Table 3.62, the extracted Mn concentrations obtained from all sediment samples were found in the range of 36.0-215.1, 104.1-340.5, 249.9-498.7, 21.7-85.7, and 21.0-86.3 mg/kg for F-I, F-II, FIII, F-IV, and F-V, respectively. Percentages of Mn distributed in each fraction for different samples were in the range of 6.9-21.1, 20.2-36.8, 34.0-60.9, 4.0-9.7, and 3.0-9.1% for F-I, F-II, F-III, F-IV, and

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F-V, respectively (Figure 3.19). Comparing to total Mn concentration, percentage recoveries of the extracted Mn obtained from the optimized sequential extraction method were found in the range of 95.4-99.2%. These results indicate that most of Mn proportion was associated with oxides of Fe-Mn.

The results of the extracted Zn concentrations for an individual fraction were in the range of 1.2-3.8, 2.1-5.7, 4.6-9.9, 3.1-6.3, and 9.0-24.7 mg/kg for F-I, F-II, FIII, F-IV, and F-V, respectively, as shown in Table 3.63. The extracted Zn concentrations from all sediments were high for the residue fraction (38.2-68.5%). On the other hand, the extracted Zn concentrations were low under other extraction conditions such as exchangeable (3.4-9.6%), acid-soluble (4.8-16.6%), reducing (16.9-31.9%), and oxidizing conditions (8.3-18.8%) as shown in Figure 3.20. Comparing to total Zn concentration, percentage recoveries of the extracted Zn obtained from the optimized sequential extraction method were in the range of 92.6-101.6%.

As shown in Table 3.64, the results of the extracted Cu concentrations from different sediment samples were found in the range of ND, ND-1.6, 2.2-5.3, 4.6-9.8, and 2.4-5.4 mg/kg for F-I, F-II, F-III, F-IV, and F-V, respectively. As can be seen in Figure 3.21, a high proportion about 36.4-61.5% of total Cu concentration was preferentially associated to organic matter in sediment. The detected Cu concentrations were low for other extraction conditions such as acid-soluble (<9.3%), reducible (14.3-28.3%), and strong acid (18.6-36.5%). Comparing to total Cu concentration, percentage recoveries of the extracted Cu concentrations obtained from the optimized sequential extraction method were in the range of 81.4-106.5%.

As summarized in Table 3.65, the extracted Pb concentrations were found in the range of ND, ND-2.3, 6.1-10.7, 2.3-5.0, and 3.7-10.6 mg/kg for F-I, F-II, FIII, F-

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IV, and F-V, respectively. The extractable Pb concentrations could be detected for most extraction conditions such as acid-soluble (5.3-9.0%), reducible (35.2-50.4%), oxidizable (14.0-23.4%), and strong acid conditions (22.4-53.5%), except for exchangeable fraction (Figure 3.22). Comparing to total Pb concentration, percentage recoveries of the extracted Pb concentrations obtained from the optimized sequential extraction method were in the range of 91.1-103.4%.

As shown in Table 3.66, the results of the extracted Cd concentrations were found in the range of 1.4 -2.6, 2.1-3.6, ND, ND, and ND-2.7 mg/kg for F-I, F-II, FIII, F-IV, and F-V, respectively. It was found that Cd concentrations could be extracted under exchangeable (21.4-46.7%), acid-soluble (31.0-53.1%), and strongly acidic conditions (26.7-35.1%) as seen from Figure 3.23. This indicates that Cd in sediments might be easily released into environment under natural conditions. Percentage recoveries of the extracted Cd concentrations obtained from the optimized sequential extraction method in sediments were in the range of 86.0-103.2%.

For samples from Lot-II, the variation of selected metals distributed in different sediment phases was comparable to the results obtained from Lot-I.

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Table 3.62 Average concentrations of Mn distributed in each fraction of sediments collected from different sample sites (Lot-II), which extracted by the optimized sequential extraction method.

Sample site	Mn (mg/kg ± SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	124.6 ± 9.1	231.9 ± 11.8	255.9 ± 17.8	45.3 ± 2.7	25.4 ± 4.5	683.1 ± 23.8	700.8 ± 23.6	97.5
KN2	92.6 ± 7.7	214.6 ± 11.9	258.6 ± 11.6	24.7 ± 2.8	21.0 ± 2.8	611.6 ± 18.7	627.5 ± 25.5	97.5
KN3	102.8 ± 9.5	238.2 ± 9.5	320.0 ± 7.0	34.5 ± 1.0	21.7 ± 0.7	717.3 ± 13.4	723.2 ± 11.3	99.2
KN4	36.0 ± 2.0	104.1 ± 6.8	314.1 ± 9.7	21.7 ± 1.2	39.8 ± 1.0	515.8 ± 12.1	539.7 ± 21.6	95.6
KN5	157.9 ± 6.0	300.1 ± 1.7	277.6 ± 1.7	55.3 ± 2.3	24.8 ± 1.7	815.7 ± 7.1	844.3 ± 21.1	96.6
KN6	118.5 ± 12.1	167.8 ± 4.1	277.7 ± 13.4	64.7 ± 2.7	37.1 ± 3.4	665.8 ± 19.0	676.3 ± 12.9	98.4
KN7	163.1 ± 9.6	332.6 ± 13.5	498.7 ± 10.5	81.5 ± 6.8	66.8 ± 1.7	1142.7 ± 20.8	1154.2 ± 20.2	99.0
KN8	186.6 ± 5.9	321.9 ± 8.8	455.9 ± 9.1	85.7 ± 2.6	63.1 ± 2.2	1113.2 ± 14.4	1133.0 ± 19.1	98.2
KN9	215.1 ± 6.4	332.1 ± 8.9	365.5 ± 11.8	57.0 ± 4.4	50.0 ± 1.8	1019.7 ± 16.8	1032.0 ± 27.3	90.1
KN10	123.7 ± 8.9	340.5 ± 7.1	325.8 ± 15.8	69.3 ± 0.7	86.3 ± 2.0	945.6 ± 19.6	990.7 ± 23.3	95.4
KN11	126.8 ± 5.9	255.6 ± 9.8	293.7 ± 11.9	56.3 ± 1.5	44.6 ± 2.9	776.9 ± 16.8	794.3 ± 22.2	97.8
KN12	87.9 ± 10.3	231.9 ± 8.4	249.9 ± 9.3	32.2 ± 0.3	32.1 ± 1.0	634.0 ± 16.3	646.4 ± 15.0	98.1

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion.

% Recovery = [S]/[T]x100.

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Table 3.63 Average concentrations of Zn distributed in each fraction of sediments collected from different sample sites (Lot-II), which extracted by the optimized sequential extraction method.

Sample site	Zn (mg/kg ± SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	2.5 ± 0.1	3.5 ± 0.1	4.6 ± 0.3	4.8 ± 0.2	10.2 ± 0.1	25.6 ± 0.4	25.3 ± 0.3	101.2
KN2	1.3 ± 0.0	5.5 ± 0.1	9.5 ± 0.2	3.6 ± 0.1	11.2 ± 0.2	33.1 ± 0.3	33.2 ± 0.2	93.7
KN3	2.3 ± 0.0	2.4 ± 0.1	8.4 ± 0.3	4.4 ± 0.2	9.0 ± 0.2	26.5 ± 0.4	27.9 ± 1.0	95.0
KN4	2.6 ± 0.1	3.0 ± 0.1	9.9 ± 0.3	5.6 ± 0.3	16.6 ± 0.6	37.7 ± 0.7	38.8 ± 0.3	97.2
KN5	1.2 ± 0.0	5.4 ± 0.1	9.0 ± 0.2	4.2 ± 0.1	15.0 ± 0.2	34.8 ± 0.3	36.5 ± 2.5	95.3
KN6	3.8 ± 0.4	5.7 ± 0.3	7.4 ± 0.3	4.0 ± 0.2	21.9 ± 0.7	42.8 ± 0.9	45.9 ± 1.2	93.2
KN7	1.8 ± 0.1	3.8 ± 0.1	9.6 ± 0.2	3.4 ± 0.2	17.2 ± 0.1	35.9 ± 0.3	38.1 ± 0.6	94.2
KN8	2.3 ± 0.0	5.4 ± 0.1	6.7 ± 0.1	3.3 ± 0.1	21.8 ± 0.4	39.4 ± 0.4	40.6 ± 1.3	97.0
KN9	2.3 ± 0.1	2.1 ± 0.1	8.6 ± 0.4	6.1 ± 0.3	24.7 ± 0.5	43.8 ± 0.7	47.3 ± 0.3	92.6
KN10	1.3 ± 0.0	3.8 ± 0.1	8.4 ± 0.3	6.3 ± 0.5	18.0 ± 0.4	37.8 ± 0.7	38.8 ± 0.6	97.4
KN11	1.3 ± 0.0	3.3 ± 0.1	6.0 ± 0.1	3.4 ± 0.2	15.1 ± 0.1	29.1 ± 0.3	28.9 ± 0.6	100.7
KN12	1.2 ± 0.1	3.4 ± 0.2	7.0 ± 0.2	3.1 ± 0.0	10.6 ± 0.3	25.3 ± 0.4	24.9 ± 0.2	101.6

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion.

% Recovery = [S]/[T]x100.

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Table 3.64 Average concentrations of Cu distributed in each fraction of sediments collected from different sample sites (Lot-II), which extracted by the optimized sequential extraction method.

Sample site	Cu (mg/kg ± SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	ND	1.4 ± 0.5	2.9 ± 0.5	9.3 ± 0.6	3.1 ± 0.6	16.7 ± 1.1	17.2 ± 0.6	97.1
KN2	ND	ND	2.6 ± 0.3	4.6 ± 0.1	4.2 ± 0.1	11.4 ± 0.4	14.0 ± 0.7	81.4
KN3	ND	ND	2.6 ± 0.2	7.7 ± 0.1	3.7 ± 0.1	14.0 ± 0.3	17.0 ± 0.6	82.4
KN4	ND	ND	3.1 ± 0.1	6.7 ± 0.4	3.4 ± 0.3	13.1 ± 0.5	13.8 ± 0.3	94.9
KN5	ND	ND	3.1 ± 0.1	6.7 ± 0.1	4.7 ± 0.3	14.5 ± 0.3	15.4 ± 0.6	94.2
KN6	ND	ND	2.5 ± 0.6	5.7 ± 0.3	3.5 ± 0.3	11.6 ± 0.7	12.0 ± 0.4	96.7
KN7	ND	1.6 ± 0.3	4.0 ± 0.2	7.2 ± 0.3	4.7 ± 0.0	17.6 ± 0.4	18.7 ± 0.3	93.6
KN8	ND	1.3 ± 0.0	3.5 ± 0.3	9.4 ± 0.5	5.4 ± 0.2	19.6 ± 0.6	21.2 ± 0.9	92.4
KN9	ND	1.0 ± 0.1	5.3 ± 0.7	9.8 ± 0.5	4.2 ± 0.5	20.2 ± 1.0	22.9 ± 0.6	88.2
KN10	ND	ND	3.3 ± 0.2	5.8 ± 0.6	2.4 ± 0.2	11.5 ± 0.7	10.8 ± 0.8	106.5
KN11	ND	ND	3.3 ± 0.5	8.6 ± 0.5	4.0 ± 0.3	15.9 ± 0.8	16.5 ± 0.1	96.4
KN12	ND	ND	2.2 ± 0.2	8.4 ± 0.3	4.6 ± 0.6	15.2 ± 0.7	16.3 ± 0.5	93.2

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion, ND: Not detected.

% Recovery = [S]/[T]x100.

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Table 3.65 Average concentrations of Pb distributed in each fraction of sediments collected from different sample sites (Lot-II), which extracted by the optimized sequential extraction method.

Sample site	Pb (mg/kg ± SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	ND	2.0 ± 0.4	8.9 ± 0.4	3.4 ± 0.2	8.5 ± 0.2	22.9 ± 1.9	24.2 ± 2.0	94.6
KN2	ND	1.1 ± 0.0	8.1 ± 0.5	2.3 ± 0.4	6.4 ± 0.7	17.9 ± 1.8	18.9 ± 0.5	94.7
KN3	ND	1.0 ± 0.0	8.7 ± 1.6	2.4 ± 1.1	5.1 ± 1.4	17.2 ± 2.4	18.7 ± 0.1	92.0
KN4	ND	2.3 ± 0.1	10.7 ± 0.2	4.4 ± 0.4	9.7 ± 0.1	27.1 ± 0.5	28.2 ± 2.3	96.1
KN5	ND	2.3 ± 0.1	9.2 ± 1.2	3.9 ± 0.0	8.8 ± 1.1	24.3 ± 2.5	23.5 ± 1.6	103.4
KN6	ND	2.3 ± 0.0	9.8 ± 0.2	4.6 ± 0.0	7.4 ± 1.2	24.2 ± 2.2	23.5 ± 1.9	103.0
KN7	ND	ND	9.4 ± 0.2	3.4 ± 0.0	3.7 ± 0.2	16.5 ± 0.3	16.5 ± 1.6	94.3
KN8	ND	1.6 ± 0.2	7.6 ± 0.8	5.0 ± 0.2	7.3 ± 1.7	21.5 ± 1.9	23.6 ± 0.1	91.1
KN9	ND	1.3 ± 0.2	8.3 ± 0.0	4.5 ± 0.0	6.5 ± 0.4	20.6 ± 0.5	21.4 ± 1.9	96.3
KN10	ND	ND	6.1 ± 1.0	3.1 ± 0.2	10.6 ± 0.2	19.8 ± 1.0	20.3 ± 2.5	97.5
KN11	ND	ND	7.6 ± 2.0	3.3 ± 0.2	9.5 ± 0.2	20.4 ± 0.1	20.7 ± 1.9	98.5
KN12	ND	1.1 ± 0.2	8.6 ± 1.8	4.6 ± 0.2	6.9 ± 1.2	21.3 ± 1.9	22.7 ± 2.4	93.8

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion, ND: Not detected.

% Recovery = [S]/[T]x100.

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Table 3.66 Average concentrations of Cd distributed in each fraction of sediments collected from different sample sites (Lot-II), which extracted by the optimized sequential extraction method.

Sample site	Cd (mg/kg±SD, n=3)							%Recovery
	F-I	F-II	F-III	F-IV	F-V	[S]	[T]	
KN1	1.8 ± 0.2	3.1 ± 0.1	ND	ND	1.9 ± 0.0	6.9 ± 0.3	8.0 ± 0.1	86.0
KN2	1.4 ± 0.1	2.9 ± 0.3	ND	ND	2.1 ± 0.1	6.4 ± 0.3	6.2 ± 0.2	103.2
KN3	2.3 ± 0.1	2.1 ± 0.1	ND	ND	2.4 ± 0.1	6.8 ± 0.2	7.3 ± 0.2	92.6
KN4	2.6 ± 0.0	3.6 ± 0.1	ND	ND	2.5 ± 0.1	8.7 ± 0.2	9.3 ± 0.1	94.0
KN5	1.8 ± 0.1	2.6 ± 0.1	ND	ND	2.3 ± 0.1	6.7 ± 0.2	7.3 ± 0.2	91.9
KN6	1.9 ± 0.1	2.6 ± 0.2	ND	ND	2.7 ± 0.2	7.1 ± 0.3	8.8 ± 0.0	81.1
KN7	2.2 ± 0.0	2.9 ± 0.3	ND	ND	2.0 ± 0.0	7.1 ± 0.3	7.7 ± 0.1	92.5
KN8	2.6 ± 0.1	3.3 ± 0.1	ND	ND	2.6 ± 0.2	8.5 ± 0.2	9.7 ± 0.2	87.5
KN9	2.2 ± 0.1	2.4 ± 0.1	ND	ND	2.2 ± 0.2	6.8 ± 0.2	7.3 ± 0.2	92.4
KN10	2.0 ± 0.1	2.6 ± 0.2	ND	ND	1.9 ± 0.0	6.6 ± 0.2	6.8 ± 0.4	96.3
KN11	2.2 ± 0.1	2.5 ± 0.1	ND	ND	ND	4.7 ± 0.2	5.0 ± 0.2	93.7
KN12	2.3 ± 0.2	3.1 ± 0.1	ND	ND	2.0 ± 0.1	7.3 ± 0.2	7.5 ± 0.1	97.7

[S]: The sum of metal concentrations from all extraction steps, [T]: Total metal concentrations found using hot-acid digestion, ND: Not detected.

% Recovery = [S]/[T]x100.

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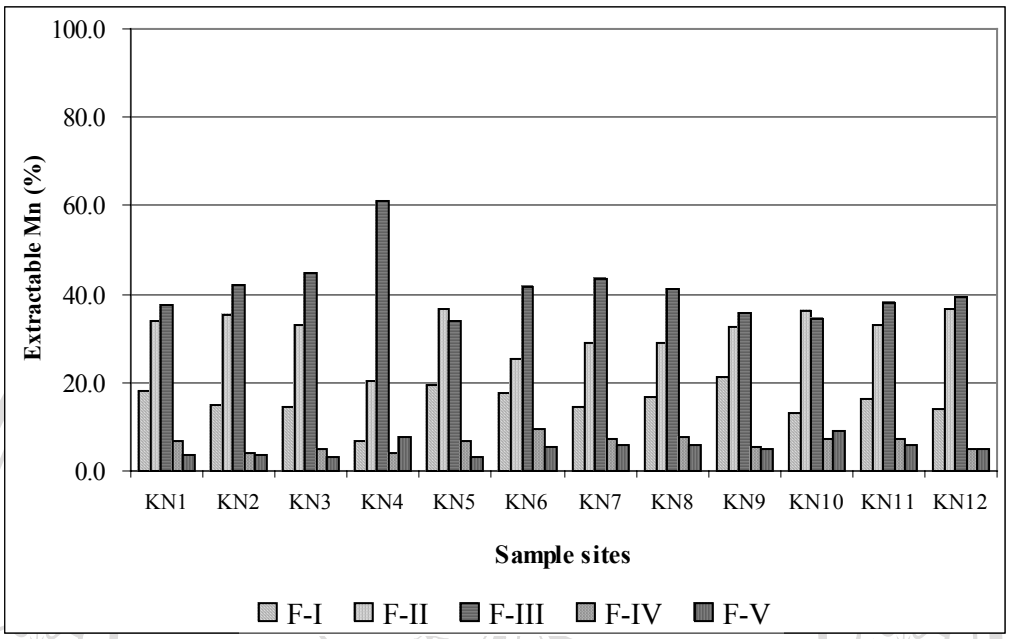

Figure 3.19 Percentages of Mn distribution in each fraction from different sediment samples (Lot-II).

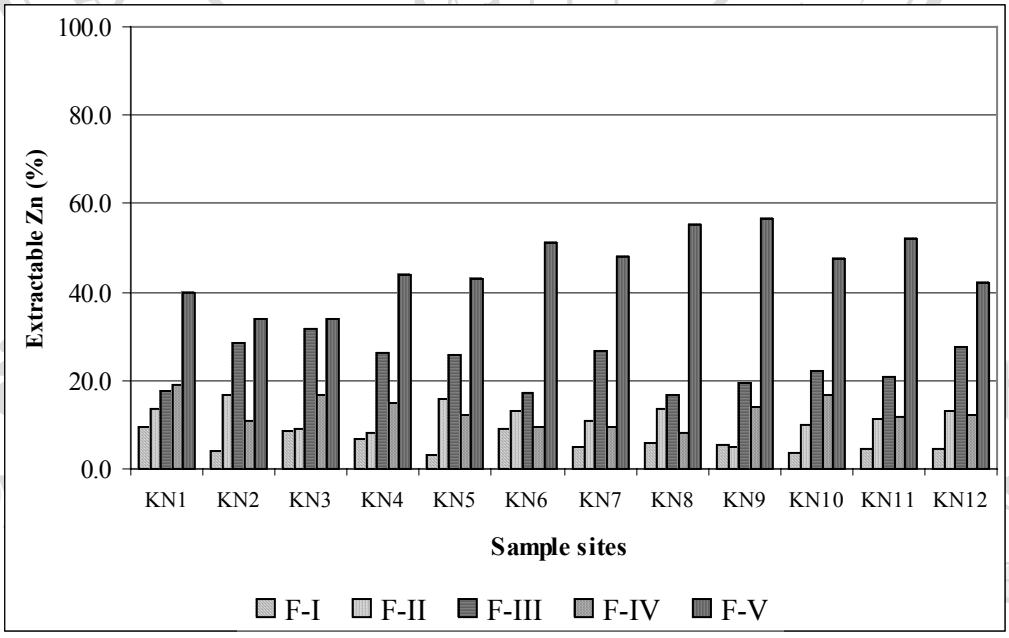

Figure 3.20 Percentages of Zn distribution in each fraction from different sediment samples (Lot-II).

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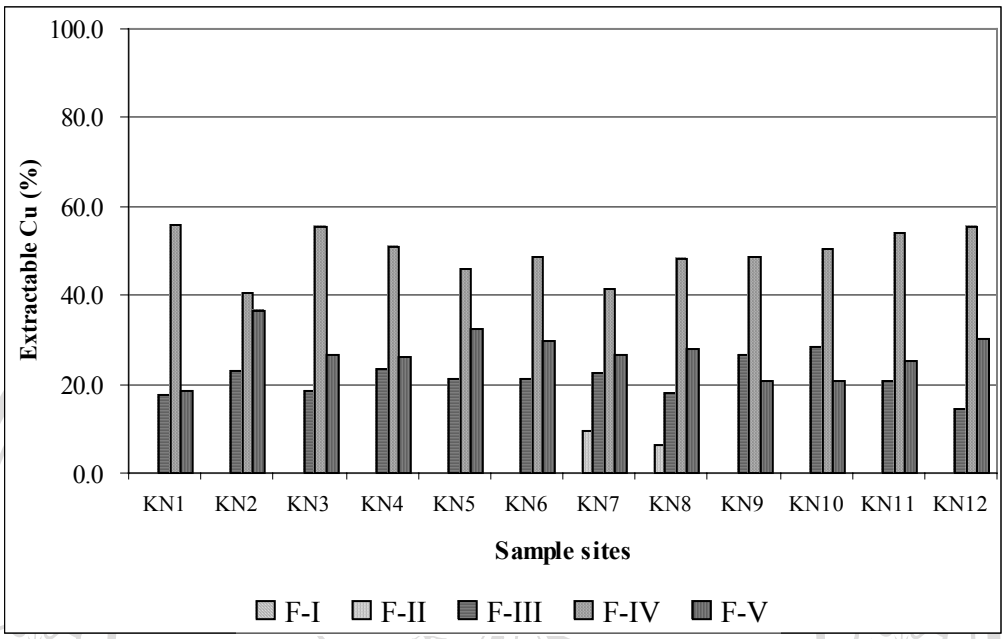

Figure 3.21 Percentages of Cu distribution in each fraction from different sediment samples (Lot-II).

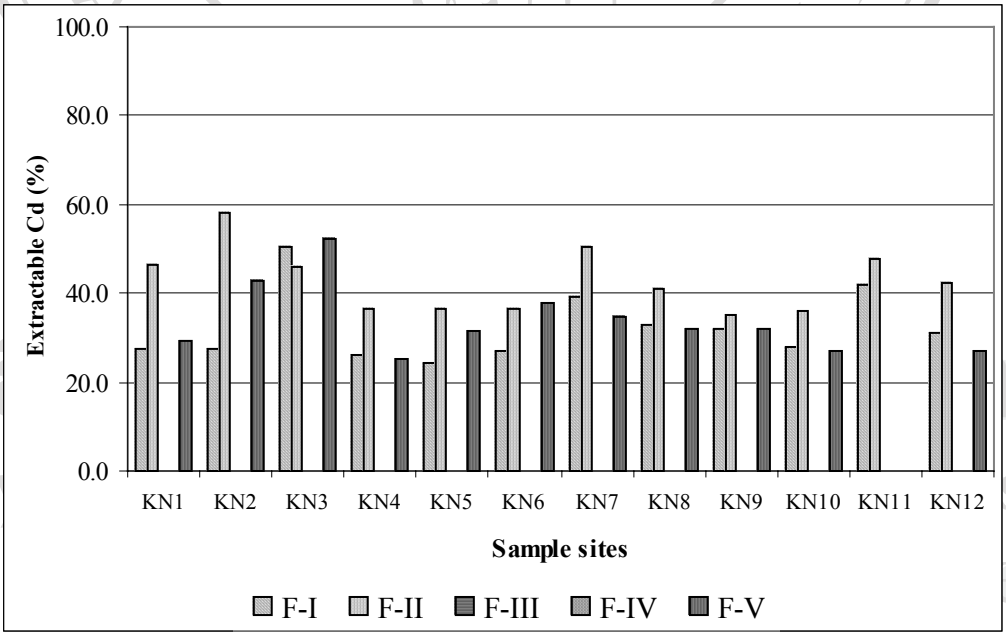

Figure 3.22 Percentages of Pb distribution in each fraction from different sediment samples (Lot-II).

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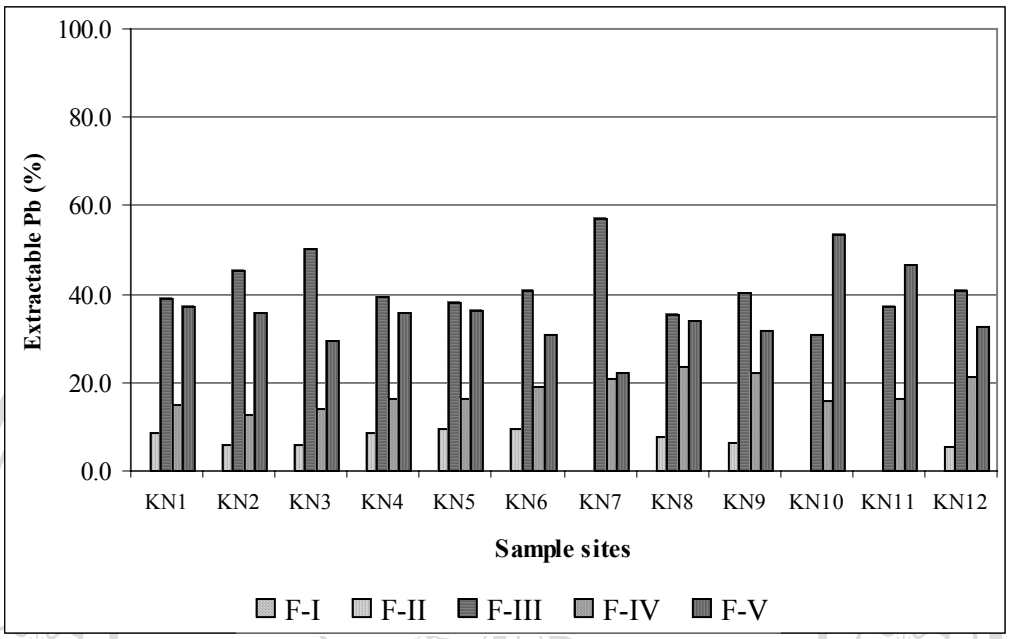

Figure 3.23 Percentages of Cd distribution in each fraction from different sediment samples (Lot-II).

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