USE OF GRAIN-SIZE AND ELEMENTAL NORMALIZATION IN THE INTERPRETATION OF TRACE METAL CONCENTRATIONS IN SOILS OF THE RECLAIMED AREA ADJOINING PORT JACKSON, SYDNEY, AUSTRALIA

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Abstract. Cobalt, Cr, Cu, Fe, Mn, Ni, Pb, Zn and As concentrations in the grain size and elemental normalization using Fe in gravel-free total soil $((X_m/Fe)_{total\,sample}; X_m = Cu, Pb, Zn, etc.)$ were used to determine the influence of variable grain size on the concentration of anthropogenic trace metal contaminants in the reclaimed area adjacent to Port Jackson, Sydney, Australia. Trace metal concentrations in soils in reclaimed area are greatly influenced by the heterogeneous nature of the waste materials buried at the site. The confounding effects of variable grain size in soils can be reduced by analyzing the contaminant concentration of the material after removing >2 mm size in sample. An example from Sydney Harbour is used to demonstrate the comparability between grain size normalized data and elemental normalized data using Fe in soils from reclaimed areas. Results from the current study reveal that the vertical distribution of trace metal concentrations in soils is remarkably similar between the grain size and elemental normalization, using Fe. Normalization methods might be beneficial in overcoming texturally driven variations of contaminant concentrations in soils in the reclaimed or infilled land area.

Keywords: elemental normalization, grain size normalization, reclaimed land, soils, trace metals

1. Introduction

Extensive reshaping of the land surface, deep cultivation and disturbance during preparation of foundations to building and roads, land-filling, importing of materials for parks, gardens and general landscaping have produced young soils with unpredictable complexity (Tiller, 1992). Management and assessment of contaminated urban soils has to recognize its complex heterogeneity. Since colonization in the late 18th century, the shoreline of Port Jackson, Sydney, has undergone major morphological change (Figure 1). Extensive reclamation has markedly reduced mangrove stands, swamps and mudflats that were previously common in the upper estuary (Irvine, 1980). The harbor area has been reduced by 6 km^2 by infilling of bay ends and reclamation of mudflats, mainly using waste (Irvine, 1980). Along the Parramatta River, these activities have resulted in a 25% decrease in total river area and a reduction in the length of the foreshores from 305
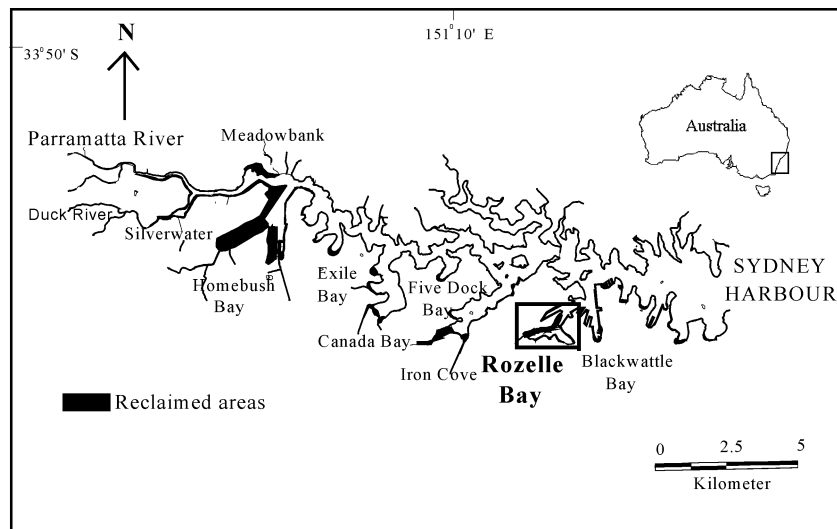
Figure 1. Reclaimed areas in Port Jackson since 18th century, Sydney.

to 245 km (Pitblado, 1978). Swamps and mudflats were infilled with industrial and domestic wastes and contained by seawalls to create large open playing fields for recreational and residential use.

Sediments are important carriers of toxicants and also cumulate toxicants over time and are thus useful in temporal, as well as spatial studies. Sediments are now being used routinely in environmental assessment to locate areas of possible concern and to determine their potential to act as important secondary sources of contaminants in aquatic environments (Birch and Taylor, 2000). Grain size prevents a comparison of sediments with different textures, when total samples are analyzed. Although total sediment analyses are required to assess sediment quality, spatial contaminant distributions expressed as a function of total sediment may not be useful in identification of source, or to determine dispersion pathways. It is therefore necessary to make corrections for differences in grain size using a normalization procedure (de Groot *et al.*, 1982; Salomons and Förstner, 1984; Birch *et al.*, 1998; Birch and Taylor, 2000). Sediment size is governed by ambient energy and sediment supply and can be highly variable over small spatial scales. Trace metals are associated with fine grained sediments (de Groot *et al.*, 1982; Salomons and Förstner, 1984; Luoma, 1987; Barbanti and Bothner, 1993; Birch and Taylor, 1999, 2000). There are two main reasons for this; smaller particles have a greater surface area, which increases their metal scavenging and adsorption capacity, and; greater concentrations of metal adsorbing/binding organic matter and Fe and Mn oxyhydroxides tend to be present in the fine fraction (Buckley and Cranston, 1991; Barbanti and Bothner, 1993; Birch *et al.*, 1996; Birch and Taylor, 2000). Interpretation of sediment contaminant data is, however, complicated by the effects of variable

grain size. Contaminants are frequently associated with the fine fraction of aquatic sediments due to an exponential increase in surface area with decreasing grain size and an increase in surface charge (Förstner and Salomans, 1980; Bubb *et al.*, 1990). The effect of variable grain size on the spatial distribution of trace metals has long been recognized in aquatic environments (Förstner and Wittmann, 1981; Brook and Moore, 1988; Krumgalz, 1989; Loring, 1991; Birch and Taylor, 2000). These effects are frequently reduced by either analyzing a uniform sediment size (usually the <62.5 μm or <4 μm fraction), or the total sample data are normalized to a conservative element (Al, Co, Fe, Li, Sc, Cs, Co, Rb) which acts as a proxy for fine material (Grant and Middleton, 1990; Loring and Rantala, 1992; Windom, 1992; Daskalakis and O'Conner, 1995; Matthai and Birch, 2001). An advantage of the elemental normalization of concentrations of trace metals in total sediment is the rapid analysis and greater cost efficiency of conducting regional geochemical mapping. Normalization of trace metals using concentrations of Fe or Al in total sediment is an alternative to grain-size-normalization (e.g. Breslin and Sanudo-Wilhelmy, 1999). Sediment data normalized to Fe and Al successfully distinquished anthropogenic from natural metal concentrations at 350 sites along coastline of the US (Daskalakis and O'Conner, 1995). Concentrations of As, Cr, Cu, Ni, Pb, Sn and Zn covaried with Fe and Al, but correlation of Fe and Al with Ag, Cd, Hg, Sb or Se was weak. Due to high natural concentrations, Fe in estuarine sediment is not expected to be substantially enriched from anthropogenic inputs (Niencheski *et al.*, 1994) and a correlation between Fe concentration and mud content is the basis for normalization by Fe.

Rozelle Bay is situated in the Sydney Basin and the study area (Bicentennial Park) is located on Rozelle Bay foreshore (Figure 2) which comprises an area of 65,000 m^2 reclaimed between 1972 and 1980. Reclamation involved removal of approximately 31,000 m^3 of material from the bed of Rozelle Bay, primarily to provide navigation depth; changes to the shoreline at the head of Rozelle Bay were also provided for land-based facilities. The total area of the study site is 10,100 m^2, the site was used as a timber storage yard until 1989. The study area was divided into two strata, based on the history of infilling, i.e. an area (6500 m^2) infilled prior to 1900 which mainly comprises waste materials, and an area (3600 m^2) infilled between 1972 and 1980 which mainly composes marine sediments (AQUATECH, 1993). Disturbed soil, which surrounds the waterfront and adjacent park area comprises fill and waste materials capped with up to 60 cm of either sandy loam, or compacted clay (Markus and McBratney, 1996). The infill zone in the timber storage yard consists of sands and gravels, some road metals, crushed bricks, charcoal, concrete fragments, and shell fragments (Figure 3). Most of the reclaimed areas in Blackwattle and Rozelle Bays are now designated as contaminated sites by the NSW EPA (Woodlots and Wetlands, 1999).

Two methods were used in the current study to reduce the textural confounding of trace metal distributions:
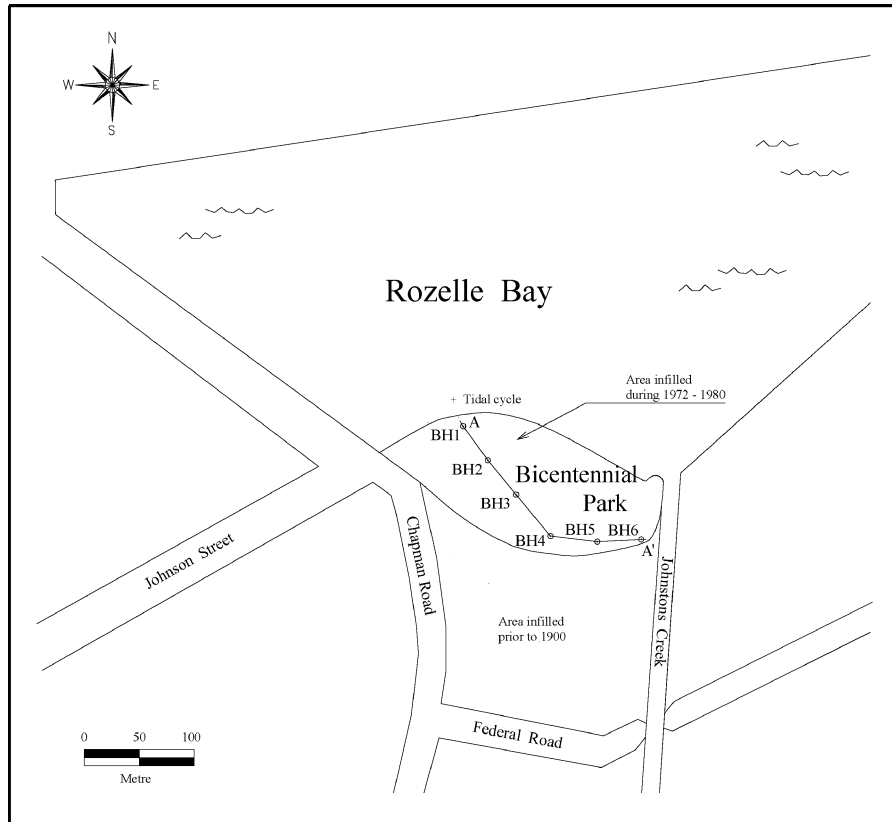
Figure 2. Study area and sampling locations. BH means borehole.

- grain size normalization,
- gravel-free total soil concentrations normalized to Fe.

The objectives of the current study are to compare grain size and elemental normalization methods, and use of the results of this analyses to better understand the distribution of metals in the soils of a reclaimed area filled with a heterogeneous mixture of waste materials.

2. Methods

Forty-eight soil samples were collected from Bicentennial Park using a truck-mounted drill. Soil samples from six locations were analyzed for a suite of eight trace metals (Co, Cr, Cu, Fe, Mn, Ni, Pb, Zn and As) to establish the vertical distribution of trace metals in soil in the reclaimed area. Polycarbonate storage containers and sampling equipment were cleaned in Pyroneg[TM] and soaked for
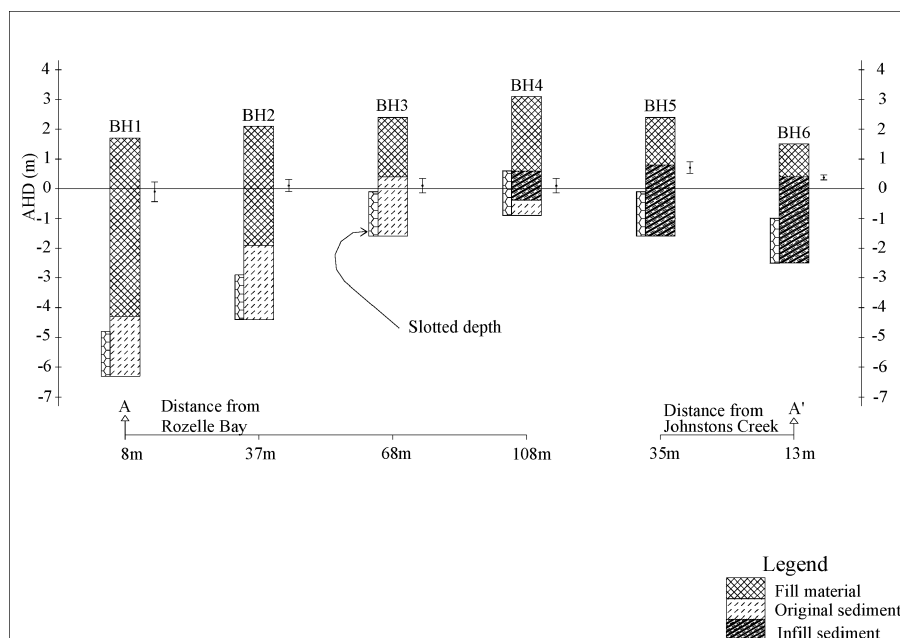
Figure 3. Geological cross section in the study area (A–A'). AHD means Australia Height Datum. Bar means hydraulic head for each borehole.

24 h in 10% (v/v) HNO_3 before repeated rinsing in MilliQ ultrapure water. Samples were oven-dried to a constant weight at 60 °C and the proportions of the fine and coarse fractions were determined gravimetrically. Gravel and sand fractions were separated by dry-sieving through a 2 mm nylon mesh. The fine-fraction samples (<62.5 μm) were ground to a powder with an agate mortar and pestle. Half gram of fine fraction and 1 g of the sand fraction (>62.5 μm) were partially digested with a 2:1 mixture of $HClO_4$:HNO_3 (Birch, 1996). However, digestion in $HClO_4$:HNO_3 will not give a total assay for many sediments. Fine fractions (<62.5 μm) were allowed to settle for 48 h through 80 cm column until the supernatant was clear before decanting the overlying water. Fine fractions (<62.5 μm) were homogenized by grinding in an agate mortar and pestle. The concentrations of Cu, Pb and Zn in the sieve water was below the detection limit of 1 μg L^{-1} and it is therefore unlikely that a loss of trace metals occurred during the sieving procedure.

Soil samples were analyzed for trace metals in a hierarchical fashion. Textural analysis was performed on all soil samples as part of the size-normalization procedure. Trace metal concentrations were determined by analyzing the fine and coarse fractions and recalculating the gravel-free total soil concentrations. All samples were stored and sieved wet to avoid textural and chemical changes during drying (Dalsgaard *et al.*, 1991).

The test tubes were heated to 160 °C for 12 h, or until acid evaporation left the samples close to dryness (Birch, 1996). After cooling, 30 ml of MilliQ ultrapure water was added and the test tubes were shaken in a rotation stirrer (3000 rpm) to homogenize the extraction solution. After settling, the extraction solutions were transferred to 20 ml polyethylene scintillation vials and stored at 4 °C. Trace metal (Co, Cr, Cu, Fe, Mn, Ni, Pb, Zn) and semi-metal (As) analyses of extractant solutions were carried out using a Varian^{TM} Vista CCD inductively coupled plasma atomic emission spectrometer (ICP-AES). Accuracy was determined by repeated analyses of international reference materials (GXR-6 ($n = 9$) and MAG-1 ($n = 7$), United State Geological Survey) and results were within the range of acceptable values (2–10%). Blanks and international standard reference materials were run on each batch of analyses ($n = 48$).

3. Results and Discussion

3.1. DESCRIPTION OF SUBSURFACE TEXTURE IN SOIL

There are three different materials in the study area, i.e. fill materials, original estuary sediment and dredged infill sediment. The depth of boreholes 1–6 was 8 m, 6.5 m, 4 m, 4 m, 4 m and 4 m beneath the surface, respectively. Fill materials consist of bricks, concrete, road debris, metal, shale fragments, gravel, sand and pebbles. Original estuary sediment consists of sand and clay and does not include any shell fragments, while dredged infill sediment consists of sand and clay and includes a lot of shell fragments (>30%). Fill materials from the surface to 6.5 m, 4 m and 2 m depth in boreholes 1–3 were detected, underlain by original estuary sediment. Borehole 4 shows the order of fill materials, dredged infill sediment and original estuary sediment. However, at boreholes 5 and 6 fill materials are detected from the surface to 1.5 m and 1 m depth, respectively, and then detected dredged infill sediment.

To facilitate the interpretation of vertical distributions of trace metal concentrations in soils and sediments in the reclaimed area, it is necessary to describe the soil grain size (Table I). The variability of the mean mud content is higher at boreholes 5 and 6, with a mean standard deviation of 28 and 13%, respectively, possibly due to the presence of dredged infill sediments. The gravel contents at boreholes 1 (35%) and 2 (39%) are much greater than at the other borehole locations as a result of the deposition of construction materials, whereas the mean mud contents at boreholes 1 and 2 are much lower than at the other borehole locations. These observations are consistent with the nature of waste materials deposited at the site during infilling by construction materials, industrial waste and dredged infill sediment. Birch and Taylor (2000) showed that size normalization of sediment reduces irregularities in small-scale spatial variance in trace metal concentrations and dispersion pathways are more evident in the fine sediment fraction than in total sediment using the grain size normalization method.

TABLE I

Summary statistics of grain size analysis for all boreholes

	Gravel (<2 mm) content (%)	Sand (<2 mm–>62.5 μm) content (%)	Mud (<62.5 μm) content (%)
	BH 1 (8 m)		
Mean	35.3	48.2	16.5
Range	18.6–48.8	31–77.5	3.9–28
S.D.	9.9	14.9	8.2
n	11	11	11
	BH 2 (6.5 m)		
Mean	39.1	43.3	17.6
Range	7.2–59.3	27.6–64.7	9.6–28.1
S.D.	15.8	12.4	6.4
n	11	11	11
	BH 3 (4 m)		
Mean	14.7	67.9	17.4
Range	6–22.1	59.7–78.4	13.3–22.5
S.D.	6.4	6.2	3.7
n	6	6	6
	BH 4 (4 m)		
Mean	15.2	64.0	20.8
Range	2.7–36.9	48.4–74.6	9.3–45.5
S.D.	11.0	11.6	10.9
n	9	9	9
	BH 5 (4 m)		
Mean	11.9	62.8	25.3
Range	2.2–19.6	49–74.5	13.3–41.8
S.D.	7.3	11.1	12.9
n	6	6	6
	BH 6 (4 m)		
Mean	5.9	62.7	31.4
Range	1.6–16.8	27.7–90	5.3–70.7
S.D.	6.3	25.4	27.5
n	5	5	5

S.D., standard deviation; bracket, borehole depth; *n*, number of samples.

3.2. SIZE NORMALIZATION

The mean, range, and standard deviation of the trace metal concentrations in fine and coarse fractions, and in gravel-free total soil of the study area are presented in Table II to compare the confounding effects of variable grain size in soils influenced

TABLE II

Summary statistics of trace metal concentrations in fine fraction, coarse fraction and gravel-free total soil. All values in $\mu g\,g^{-1}$, except Fe in %

Grain size	Cu	Pb	Zn	Mn	Co	Cr	As	Ni	Fe
<62.5 μm				Fine fraction					
Mean ($n = 48$)	74	310	392	528	13	39	42	23	3.1
Range	8.3–336	18–2841	49–2028	73–1729	2.0–33	13–101	8.4–115	3.3–51	1.2–6.4
S.D.	69	592	444	418	8	17	23	12	11
62.5 μm–2 mm				Coarse fraction					
Mean ($n = 48$)	12	52	82	238	5	11	10	9	1.4
Range	0.3–86	9–350	9–531	13–823	0.5–18.5	2–89.2	2.7–25	1.0–46	2.1–4.4
S.D.	15	75	95	227	4	12	6	9	10
<2 mm				Gravel-free total soil					
Mean ($n = 48$)	23	85	150	285	6	18	26	12	1.9
Range	2.8–103	4–790	14–1512	8–1107	0.2–27.1	2.8–95	6.4–64	1.2–44	2.8–5.0
S.D.	19	141	233	245	5	13	14	9	10

S.D., standard deviation; n, number of samples.

by waste materials. Mean concentrations of all the trace metals investigated follow the order; fine fraction ($<62.5\ \mu$m) > gravel-free total soil (<2 mm) > and coarse fraction ($>62.5\ \mu$m).

3.3. ELEMENTAL NORMALIZATION

Normalization of trace metal concentrations in sediments using Fe, or Al is an alternative to grain size normalization (e.g. Breslin and Sanudo-Wilhelmy, 1999). The effectiveness of normalization to reduce textural control of trace metal concentrations is evident for Fe, an element that is not substantially enriched by anthropogenic contamination due to high natural concentrations (Niencheski *et al.*, 1994). In general, a correlation between the Fe concentration and fine fraction is the basis for normalization by Fe. Figures 4a–4c present the correlation between Cu, Zn and Fe concentrations in gravel-free total soil (<2 mm fraction) and mud content (<62.5 μm). In the current study, the correlations of Cu, Zn and Fe concentrations in gravel-free total soil and mud content indicate 0.06, 0.11 and 0.41, respectively (Figures 4a–4c). As a result, concentrations of Fe in gravel-free total soil relatively correlate to mud content ($r^2 = 0.41$, $p < 0.001$, $n = 44$). Concentrations of trace metals in gravel-free total soil were normalized to Fe and plotted along a linear relationship between trace metal concentrations in fine fraction and trace metal concentrations in gravel-free total soil/Fe concentration in gravel-free total soil (Figure 5).

The relationship between trace metal (Cu, Pb, Zn, Mn, Ni, Co, Cr, As) concentrations in fine fraction and trace metal (Cu, Pb, Zn, Mn, Ni, Co, Cr, As) concentrations in gravel-free total soil/Fe concentration in gravel-free total soil is relatively correlated ($r > 0.5$, $p < 0.001$, $n = 48$). These results reveal that the relationship between trace metal concentrations in fine fraction and trace metal concentrations in gravel-free total soil/Fe concentration in gravel-free total soil shows good efficiency in interpretation the distribution of trace metal concentration in reclaimed soil.

3.4. COMPARISON BETWEEN GRAIN SIZE NORMALIZATION AND ELEMENTAL NORMALIZATION USING Fe

Subsurface distributions of Cu, Cr, Pb, Zn, Mn, Ni, Co and As concentrations in fine fraction, in gravel-free total soil, in gravel-free total soil/Fe in gravel-free total soil show the same trend at all borehole locations. For example, subsurface distributions of Cu, Pb, Zn and Ni in fine fraction, in gravel-free total soil, in gravel-free total soil/Fe in gravel-free total soil and mud content at BH 4 and BH 5 are presented Figures 6a–6d and 7a–7d, respectively.

Subsurface distributions of Cu, Pb, Zn, and Ni concentration in fine fraction, in gravel-free total soil, in gravel-free total soil/Fe in gravel-free total soil and mud content at BH 4 and BH 5 show the same trend in fill materials, infill sediments, and estuary sediments between the surface and 4 m depth (Figures 6a–6d and 7a–7d).
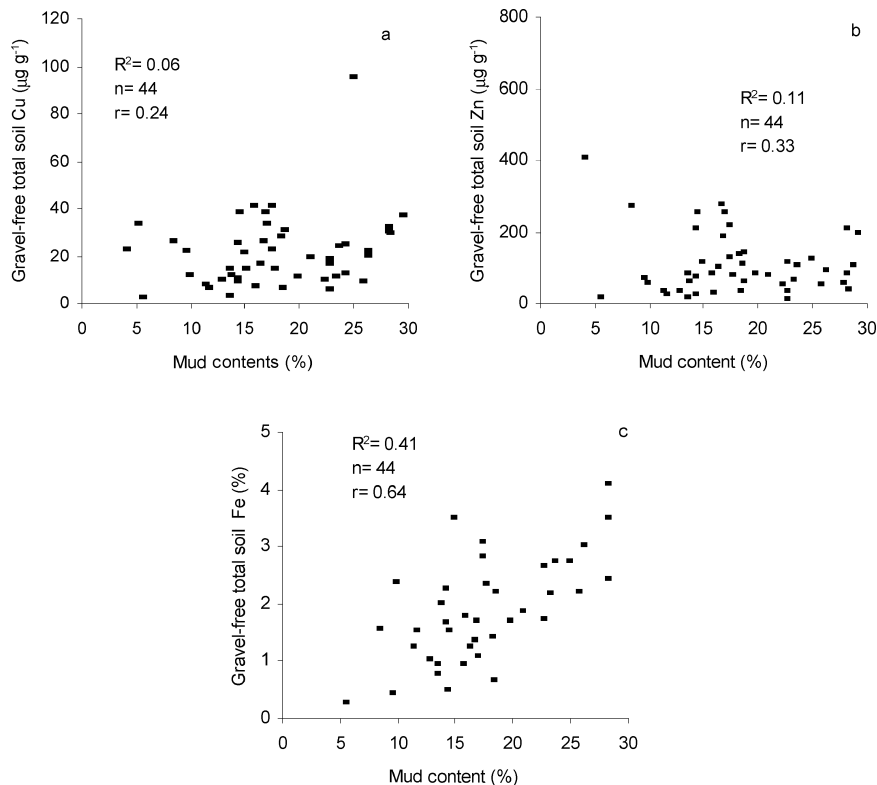
Figure 4. a–c: Correlation between Cu, Zn and Fe concentrations in gravel-free total soil and mud content.

In general the subsurface trend of Cu, Pb, Zn and Ni concentration at BH 4 is not consistent with mud content owing to artificially reclaimed various contaminants, while the subsurface trend of Cu, Pb, Zn and Ni concentration BH 5 is relatively consistent with mud content owing to artificially reclaimed marine sediment.

Significant correlations exist between Cu, Pb, Zn, Mn, Ni, Co, Cr and As concentrations in fine fraction and Cu, Pb, Zn, Mn, Ni, Co, Cr and As in gravel-free total soil/Fe in gravel-free total soil ($r = \geq 0.5$; $p < 0.001$, $n = 48$) (Table III). In particular, the correlation between Pb in fine fraction and Pb in gravel-free total soil/Fe in gravel-free total soil yielded a significant correlation ($r = 0.92$, $p < 0.001$, $n = 8$). An advantage of the elemental normalization method is that is a rapid analysis and has more cost effective for conducting regional geochemical mapping (Matthai and Birch, 2001), while the elemental normalization method has disadvantage of requiring suitable element for elemental normalization. An advantage of the grain size normalization is that is identification of source, dispersion pathways and sinks of trace metal contaminants (Birch and Taylor, 2000), while the grain size normalization requires analyses of different grain size fractions of the soil (Matthai and Birch, 2001).
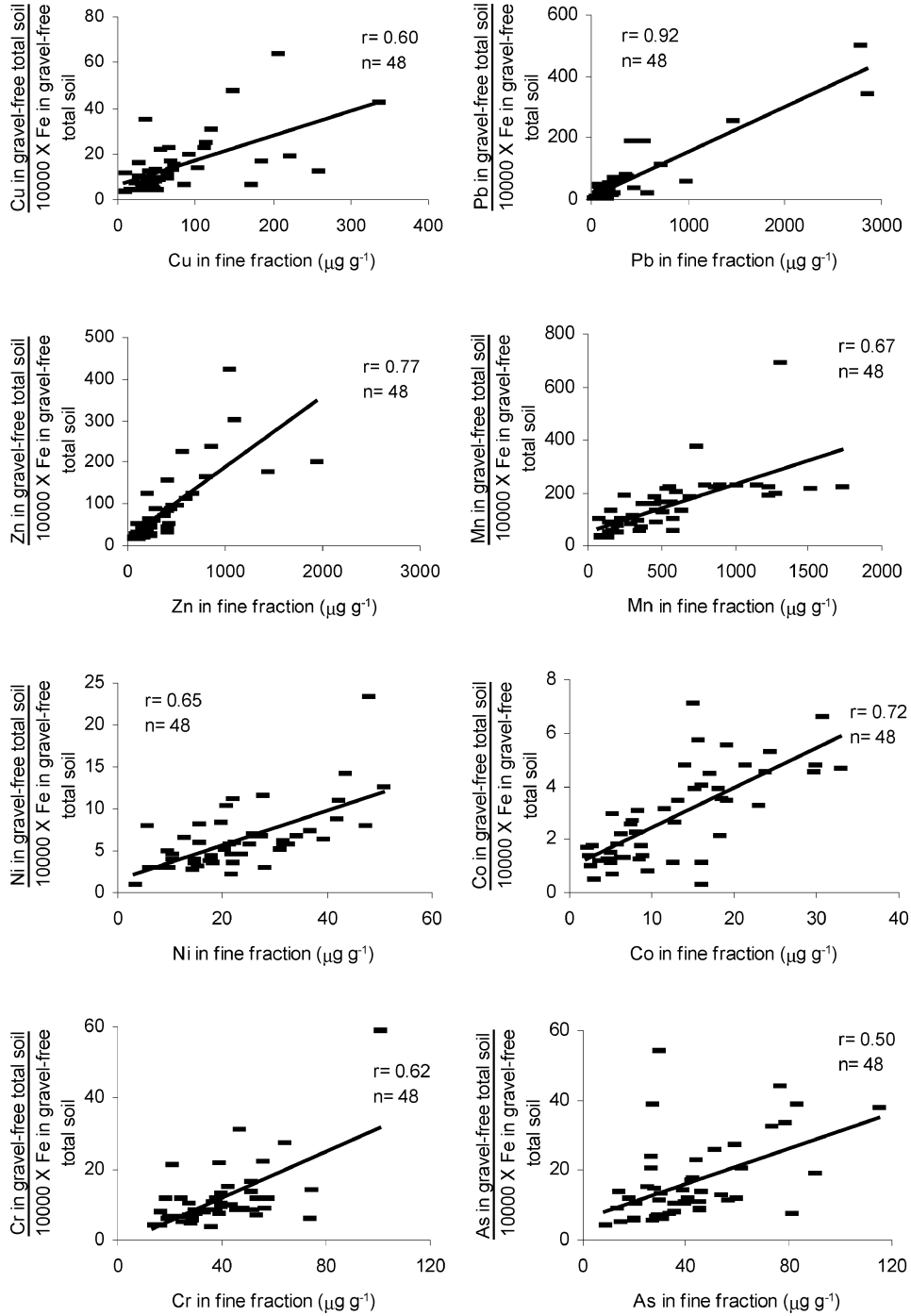
Figure 5. Correlation between metal concentrations in fine fraction and metal concentrations in gravel-free total soil/Fe in gravel-free total soil.
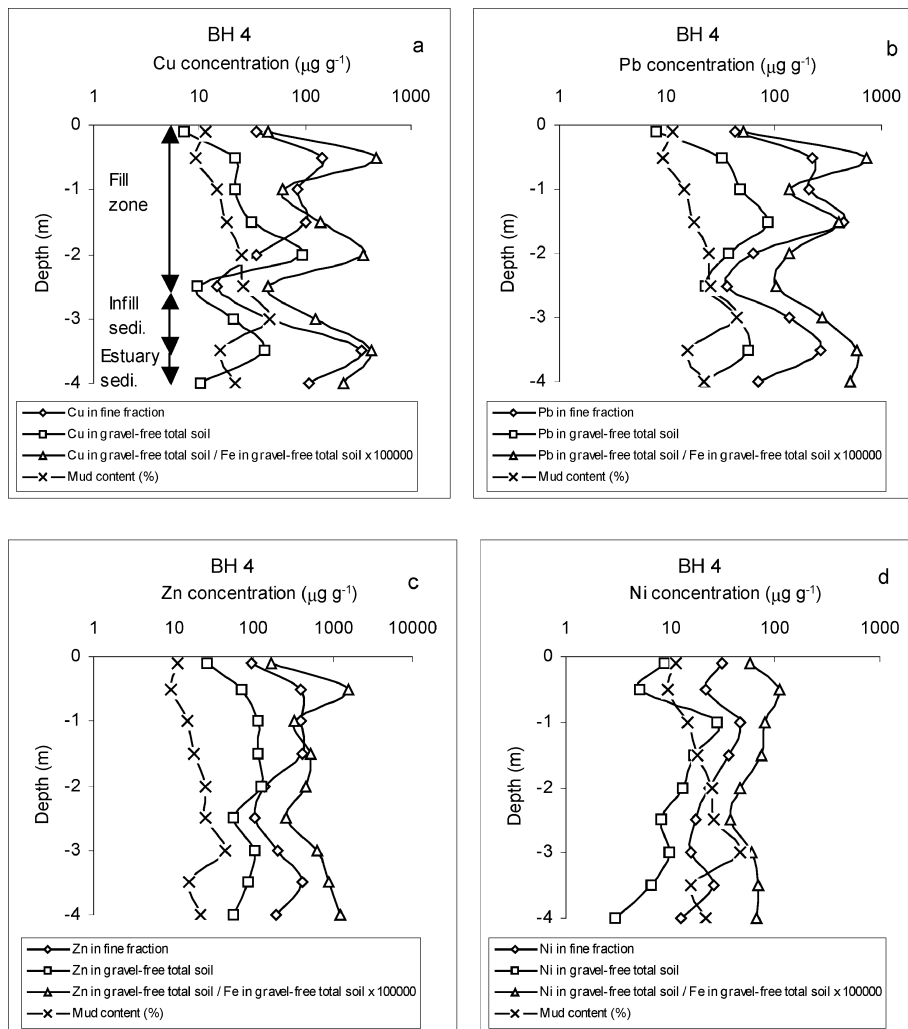
Figure 6. a–d: Subsurface distribution of Cu, Pb, Zn and Ni concentrations in fine fraction, in gravel-free total soil and in gravel-free total soil/Fe in gravel-free total soil at BH 4.

4. Conclusions

This study was performed to compare grain size and elemental normalization methods, and use of the results of this analyses to better understand the distribution of metals in the soils of a reclaimed area filled with a heterogeneous mixture of waste materials, resulting that the interpretation of trace metal concentrations in the total soil is influenced by variable grain size and texture.

Normalization methods might be beneficial in overcoming texturally driven variations of contaminant concentrations in soils in the reclaimed or infilled land area.
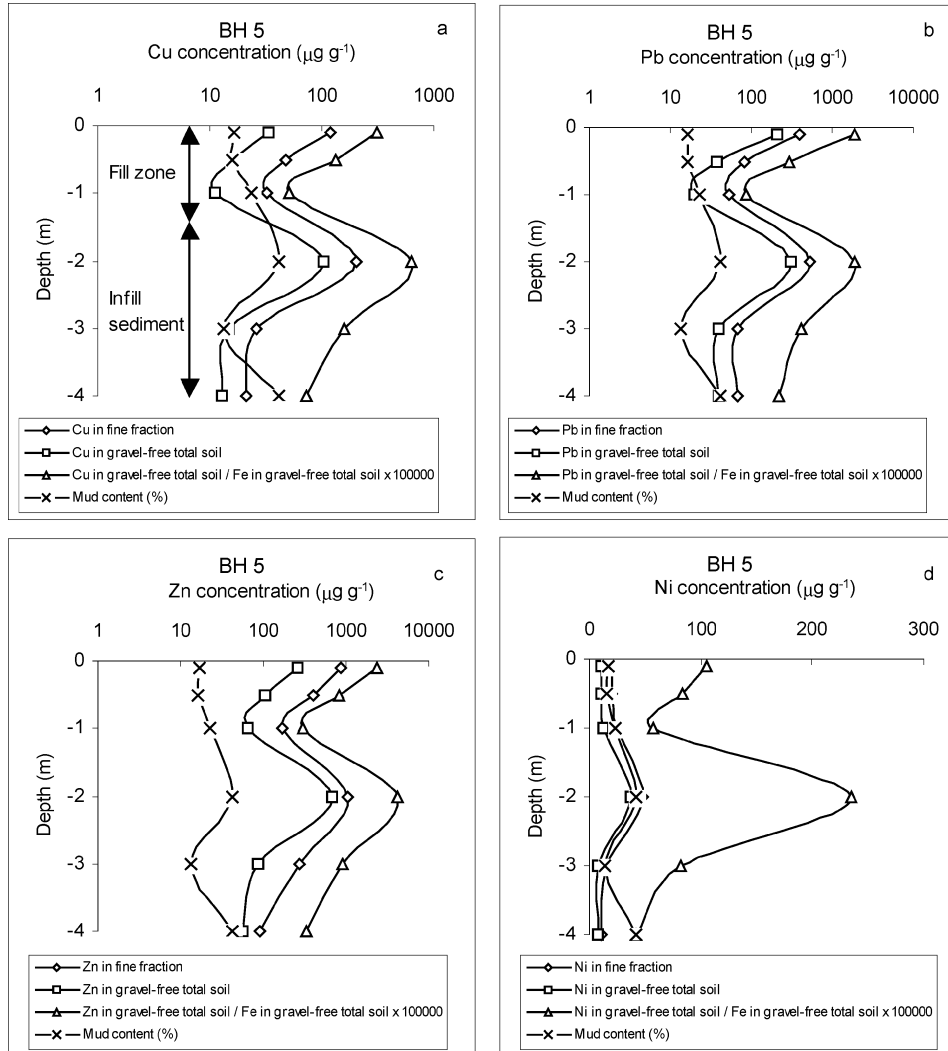
Figure 7. a–d: Subsurface distribution of Cu, Pb, Zn and Ni concentrations in fine fraction, in gravel-free total soil and in gravel-free total soil/Fe in gravel-free total soil at BH 5.

The majority of contaminants are associated with the fine fraction of the sediment and analyzing this fraction considerably reduces grain effects and increases data comparability. However, possible contaminant loss during the separation process has retarded the use of this technique. Results of the current study indicate minimal loss to the coarse sediment component and no detectable loss to sieve water during the fractionation procedure. Normalization of the trace metal data using Fe is more time cost-effective than grain size normalization, which requires analyses of different grain size fractions of the soil. An example from Sydney Harbour is used to

TABLE III

Correlation of each metal in gravel-free total soil/Fe in gravel-free total soil and each metal in fine fraction

	Cu	Pb	Zn	Mn	Co	Cr	As	Ni
Cu	0.6[a]							
Pb		0.92[a]						
Zn			0.77[a]					
Mn				0.67[a]				
Co					0.72[a]			
Cr						0.62[a]		
As							0.5[a]	
Ni								0.65[a]

[a]Indicates $p < 0.001$, $n = 48$.

demonstrate the comparability of size-normalized data and elemental normalized data (Fe) in interpretation of source, dispersion and accumulation in soils in the reclaimed area.

Acknowledgments

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