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Pollution distribution of heavy metals in surface soil at an informal electronic-waste recycling site

Takashi Fujimori · Hidetaka Takigami

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Abstract We studied distribution of heavy metals [lead (Pb), copper (Cu) and zinc (Zn)] in surface soil at an electronic-waste (e-waste) recycling workshop near Metro Manila in the Philippines to evaluate the pollution size (spot size, small area or the entire workshop), as well as to assess heavy metal transport into the surrounding soil environment. On-site length-of-stride-scale (~70 cm) measurements were performed at each surface soil point using field-portable X-ray fluorescence (FP-XRF). The surface soil at the e-waste recycling workshop was polluted with Cu, Zn and Pb, which were distributed discretely in surface soil. The site was divided into five areas based on the distance from an entrance gate (*y*-axis) of the e-waste recycling workshop. The three heavy metals showed similar concentration gradients in the *y*-axis direction. Zn, Pb and Cu concentrations were estimated to decrease to half of their maximum concentrations at ~3, 7 and 7 m from the pollution spot, respectively, inside the informal e-waste recycling workshop. Distance from an entrance may play an important role in heavy metal transport at the soil surface. Using on-site FP-XRF, we evaluated the metal ratio to characterise pollution features of the solid surface. Variability analysis of heavy metals revealed vanishing surficial autocorrelation over metre ranges. Also, the possibility of concentration prediction at unmeasured points using geostatistical kriging was evaluated, and heavy metals had a relative "small" pollution scales and remained inside the original workshop compared with toxic organohalogen compounds. Thus, exposure to heavy metals may directly influence the health of e-waste workers at the original site rather than the surrounding habitat and environmental media.

Keywords Surface soil · E-waste recycling · Heavy metal · Pollution distribution · Portable XRF

Introduction

Electronic-waste (e-waste) recycling in developing countries is one of the sources of toxic heavy metals. Lead (Pb), copper (Cu) and zinc (Zn) metals are found in cathode-ray tubes (CRTs), wires/cables, circuit boards and various other electronics. Studies at

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T. Fujimori · H. Takigami
Center for Material Cycles and Waste Management
Research, National Institute for Environmental Studies
(NIES), 16-2 Onogawa, Tsukuba, Ibaraki 305-8506, Japan

T. Fujimori (✉)
Department of Environmental Engineering, Graduate
School of Engineering, Kyoto University, Katsura,
Nisikyo-ku, Kyoto 615-8540, Japan
e-mail: fujimori.takashi.3e@kyoto-u.ac.jp

T. Fujimori
Department of Global Ecology, Graduate School
of Global Environmental Studies, Kyoto University,
Katsura, Nisikyo-ku, Kyoto 615-8540, Japan

uncontrolled e-waste recycling sites have revealed hazardous metal pollution in various environmental media (Wong et al. 2007) and in humans (Huo et al. 2007; Chen et al. 2011). Informal e-waste recycling activities performed in various workshops, such as dismantling, stripping, removing, melting, burning, disposing and selling (Huo et al. 2007; Chen et al. 2011), can increase heavy metal pollution of surface soil in Guiyu, China (Brigden et al. 2005; Leung et al. 2006; Wong et al. 2007; Li et al. 2011a, b), Taizhou, China (Tang et al. 2010), Hong Kong (Lopez et al. 2011), Bangalore, India (Ha et al. 2009), New Delhi, India (Brigden et al. 2005) around Metro Manila, the Philippines (Fujimori et al. 2012), and Accra, Ghana (Asante et al. 2012). General open-air informal workshops consist of an entrance, buying and selling area, working area and piles of e-waste. Surface soil contamination by heavy metals may be influenced by surficial distribution of each e-waste recycling activity area. Therefore, further studies are required on surficial distribution of heavy metals in surface soil at an e-waste workshop to better understand the pollution scale (spot size, small area or surficial entire pollution throughout a workshop) and to assess the transport of heavy metals into the surrounding soil environment. Li et al. suggested that heavy metals remain in the original workplace and are not transported to the surrounding areas (Li et al. 2011b). However, no studies on pollution distribution have been performed inside an informal e-waste recycling site. The size of an informal e-waste recycling workshop is relatively in small size such as 10 m × 10 m scale, and previous studies have generally collected environmental samples representing the entire workshop and have not analysed smaller surficial distributions (~1 m mesh). Studies focused on the transport range of contaminants have always operated on a kilometre (km) scale. Although toxic brominated and chlorinated organic compounds were transported a few kilometres from e-waste recycling sites (more for e-waste dismantling and crushing) (Li et al. 2011b), the transport range of heavy metals is predicted to be shorter. To understand the pollution distribution of heavy metals, a scale-down surficial measurement under metre meshes inside an informal e-waste recycling site is required.

In this study, heavy metals (Pb, Cu and Zn) were measured in surface soil at an informal e-waste recycling site near Metro Manila in the Philippines to know the pollution distribution of heavy metals. On-site length-of-stride-scale (~70 cm) measurements were performed at each surface soil point using field-portable X-ray fluorescence (FP-XRF). Although surface soil was thought to be influenced by surface events such as wiping, cleaning and weathering, e-waste recycling activities (transport, piling up, dismantling, abrasion, etc.) might also influence distribution of heavy metals in surface soil. In addition, on-site FP-XRF measurement was surface-oriented analysis. So, this analytical method was suitable for surface soil. The concentration gradient and ratios of Cu, Pb and Zn are discussed to clarify the pollution status and distribution tendencies. Statistical semivariogram provided surficial variability of heavy metals and could be used for geostatistical prediction.

Materials and methods

Location

In August 2010, an informal e-waste recycling workshop located in the southern region of Metro Manila of the Philippines was visited. The site was a small area of less than 10 m × 14 m (Fig. 1). Various wastes were transported by cars, motorbikes and bicycles with carts through an entrance gate (Fig. 1g) and stored in restricted areas that differed depending on the waste type, such as e-waste (Fig. 1a, b, d), paint cans (Fig. 1, no photo) and plastic bottles (Fig. 1f). E-waste contained CRT-based TVs, circuit boards, cables, audio players and other home electronics. Workers dismantled TV casings, removed electronic components from circuit boards and stripped cable coatings without protecting their nose, mouth or hands. These recycling activities likely contaminate the workshop soil with heavy metals.

FP-XRF measurement and quality control

On-site FP-XRF can be used to measure metal concentrations in small surface soil areas (measurement window, diameter of 2 cm). Previous studies have applied cost-effective (Taylor et al. 2004) FP-XRF for on-site measurements of Zn (Carr et al. 2008; Radu and Diamond 2009; Jang 2010), Cu (Carr et al. 2008; Radu and Diamond 2009; Jang 2010) and Pb (Carr et al. 2008; Hurkamp et al. 2009; Radu and Diamond 2009; Chou et al. 2010; Jang 2010; Schwarz

Fig. 1 An e-waste recycling workshop in the Philippines. *Asterisk* indicates the measurement point. **a**, **b**, **d** Piles of various e-wastes. **c** Various debris of e-waste such as corrugated iron and plastic casings. **e** Measurement by FP-XRF (*soil mode*). **f** Pile of plastic bottles in a cage. **g** Entrance gate of the e-waste recycling workshop

et al. 2012) in surface soil to determine heavy metal concentrations. According to the measurement, these metals were at higher concentrations than other elements. In this study, Zn, Cu and Pb concentrations in the surface soil were measured at 56 points on a length-of-stride-scale (~70 cm) inside and outside the informal e-waste recycling site using a FP-XRF (Innov-X alpha, Innov-X System, Inc.) applied to recent on-site soil (Carr et al. 2008; Jang 2010; Chou et al. 2010) and previous screening (Allen et al. 2008;

Takigami et al. 2009; Kajiwara et al. 2011) studies. Length-of-stride scale expressed representative length between point and point of this study. An uncontaminated location was selected as a control measurement point of surface soil. In *soil mode* (Chou et al. 2010), the FP-XRF detected three heavy metals (i.e. Zn, Cu and Pb) at levels greater than ~10 μg/g soil sample. Approximately 40 s was required for each point measurement. Figure 1e shows a photograph of on-site measurement using FP-XRF.

Quality control of FP-XRF was performed using silicon dioxide (SiO_2, >99 %, Kanto Chemical Co., Inc.) and certified standard materials, such as soils (JSAC 0402 and JSAC 0403, the Japan Society for Analytical Chemistry), a sea sediment (NMIJ CRM 7302-a, National Metrology Institute of Japan), an industrial sludge (NIST 2782, National Institute of Standards and Technology) and indoor dust (NIST 2584). Approximately 2 cm of a powdery sample was uniformly placed into a cup (inner diameter 2.5 cm × height 2.5 cm) with a thin clear film (Mylar X-ray film, Chemplex Industries, Inc.). Polyurethane foam was compacted at the bottom and the sample cup was sealed. The detection window of FP-XRF to Mylar film was held and the trigger was pulled for ~40 s three times. Zn, Cu and Pb were not detected in SiO_2 powder (negative control). Average relative standard deviation (RSD) was 6.5 % (range 2.0–16 %). Of the matrices, average recovery ratios were 82 % (Zn), 80 % (Cu) and 81 % (Pb). During on-site FP-XRF measurements, the moisture content of the soil (Kalnicky and Singhvi 2001; U.S. EPA 2007) required close observation. Moisture content was evaluated using six soil samples, which averaged 20 %. Therefore, the bias by moisture may be minor (Kalnicky and Singhvi 2001; U.S. EPA 2007; Chou et al. 2010). Large biases in on-site FP-XRF measurements were caused by the lack of soil preparation and nonhomogeneous soils (Kalnicky and Singhvi 2001; U.S. EPA 2007). However, FP-XRF can identify small hot spots of heavy metal contamination rapidly on-site (Carr et al. 2008; Hurkamp et al. 2009; Radu and Diamond 2009; Chou et al. 2010; Jang 2010).

Semivariogram

When the available values of variable z at n locations were measured, v_i, $z(v_i)$, $i = 1, 2, \ldots, n$, the semivariogram, $\hat{\gamma}(h)$, is written as

$$\hat{\gamma}(h) = \frac{1}{2N(h)} \sum_{i=1}^{N(h)} \{z(v_i + h) - z(v_i)\}^2 \tag{1}$$

where h represents the geographic distance between two observations, $N(h)$ is the total number of pairs at h, and $\hat{\gamma}(h)$ indicates half of the average squared difference between the paired data values. $\hat{\gamma}(h)$ was calculated using common logarithmic concentrations in Eq. (1) and was simulated by the spherical (Sph) model, as shown in the following equation:

$$\gamma(h) = (s - n)\left(\left(\frac{3h}{2r} - \frac{h^3}{2r^3}\right)1_{(0,r)}(h) + 1_{[r,\infty)}(h)\right) + n1_{(0,\infty)}(h) \tag{2}$$

where h is distance, $\gamma(h)$ is the calculated semivariogram, n is the nugget, s is the sill, r is the range, and $1_A(h)$ function is 1 if $h \in A$ and 0 otherwise. And, we also used linear (Lin) model. Based on the Sph and Lin models, two-dimensional concentrations of elements in soil were computed on a 10 cm × 10-cm grid using ordinary kriging, known as the geostatistical method (Isaaks and Srivastava 1989; Goovaerts 1999). R software for Windows 2.11.1 and the gstat package ver. 0.9–69 (Pebesma 2004; John et al. 2007) were used to calculate the semivariogram and ordinary kriging. They were provided by the comprehensive R archive network (CRAN, http://cran.r-project.org).

Statistical analysis

Statistical analysis was performed using SPSS software (version 19). Continuous data were analysed using the nonparametric Jonckheere–Terpstra trend test to work with unequal sample sizes. Similar groups were divided by hierarchical cluster analysis (Ward method).

Results and discussion

Concentration and surficial distribution

The concentrations of Cu, Zn and Pb in surface soil over the FP-XRF detection limits at every point were shown in Fig. 2. Basic statistics of metal concentrations in surface soil are shown in Table 1. Compared with the average metal concentrations in the upper

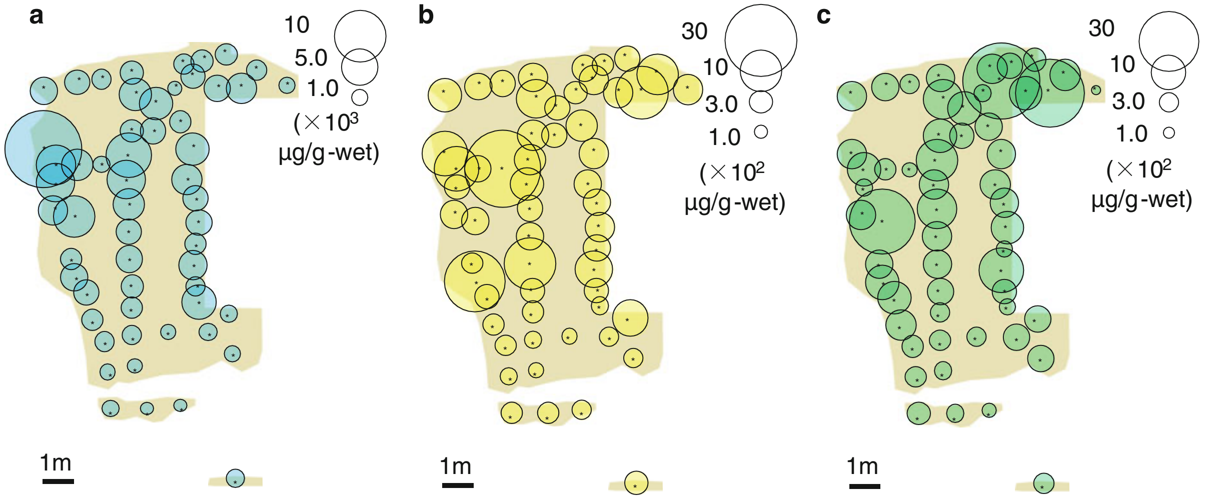

Fig. 2 Surficial distribution patterns of Zn (**a**), Pb (**b**) and Cu (**c**) concentrations in surface soil

continental crust (Cu, 14.3; Pb, 17; Zn, 52 μg/g) (Wedepohl 1995) which were comparable values with local soil in the Philippines (Fujimori et al. 2012), the minimum concentrations of these three metals at inside/outside of the workshop were excessive. In addition, we assessed the influence of suspension of soil dusts by comparison with inside/outside concentrations of heavy metals. As a result, inside of the e-waste recycling workshop was more seriously polluted by the heavy metals than outside. So, major part of heavy metals remained at inside of this workshop. At inside of the workshop, surface soil concentrations of 50 % for Cu, 75 % for Zn and 50 % for Pb exceeded the phytotoxicity levels of Cu (600 μg/g) (NJDEP 1999) and Zn (1,500 μg/g) (NJDEP 1999), and the Pb concentrations put children at risk (400 μg/g to protect 95 % of children at a blood-Pb level of 10 μg/dL) (NJDEP 1999). Therefore, the surface soil at inside of the e-waste recycling workshop in the Philippines was polluted by Cu, Zn and Pb. In addition, concentrations of these heavy metals at inside were similar in soils of other informal e-waste workshops of Bangalore, India (Ha et al. 2009), Taizhou, China (Tang et al. 2010), Hong Kong (Lopez et al. 2011) and around Metro Manila, the Philippines (Fujimori et al. 2012). FP-XRF measurements revealed surficial distribution patterns of Zn, Pb and Cu concentrations, as depicted by the bubble plot in Fig. 2a–c, respectively. Using the surficial distribution patterns, relationships between surficial pollution patterns of heavy metals were statistically estimated. No correlations were observed among the pairs of heavy metals such as Cu versus Zn, Zn versus Pb and Pb versus Cu. These metals were contained in CRTs (Pb), solder (Pb), wires/cables (Cu), circuit boards (Zn and other metals) and various other electronic materials. The three heavy metals were distributed discretely in surface soil, which may have been caused by heterogeneous surficial distribution of e-waste species (Fig. 1) and various recycling activities at this workshop.

Pollution scale

The study site was divided into five areas based on distance from the entrance gate (ref. Fig. 1g) of the e-waste recycling workshop: <0, 0–3, 3–6, 6–9 and 9–12 m (*y*-axis in Fig. 3a). Although analysis of the *x*-axis direction had no clear pattern in heavy metal concentrations, the three heavy metals showed similar concentration gradients in the *y*-axis direction, which suggested that distance from an entrance significantly affected heavy metal concentrations in surface soil. The maximum concentration of Zn in surface soil was 6–9 m from the entrance ($p < 0.05$) (Fig. 3b). Median Zn concentrations at 6–9, 3–6 and 0–3 m areas were 3,800, 2,400 and 1,100 μg/g-wet, respectively. Based on this information, we estimated half the distance of the highest concentration zone ($d_{1/2}$). For Zn, the highest concentration zone was 6–9 m, and a representative distance was selected for this zone as a median value (7.5 m). The $d_{1/2}$ of Zn was estimated as

Table 1 Statistics of metal concentrations in surface soil at an e-waste recycling workshop in the Philippines

	Zn (µg/g-wet)		Pb (µg/g-wet)		Cu (µg/g-wet)	
	Inside ($n = 52$)	Outside ($n = 4$)	Inside ($n = 52$)	Outside ($n = 4$)	Inside ($n = 52$)	Outside ($n = 4$)
Median	2,300	830	420	260	680	320
Geometric mean	2,300	850	450	250	630	290
Arithmetic mean	3,000	890	580	250	850	310
Max	22,000	1,300	3,400	300	5,000	450
75 % quartile	3,600	1,100	560	280	900	390
25 % quartile	1,600	630	330	230	400	240
Min	720	620	130	200	67	160

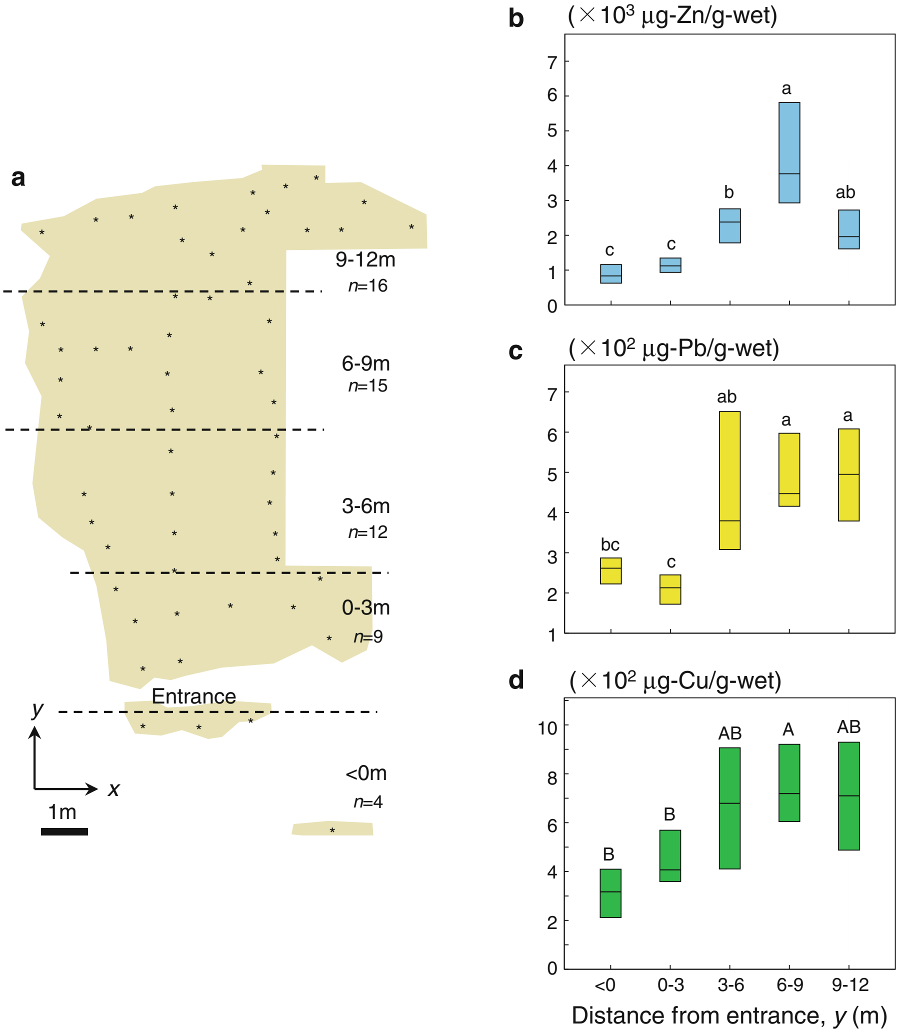

Fig. 3 Concentration gradients of heavy metals from the entrance. **a** Dividing five areas (*y*-axis; <0, 0–3, 3–6, 6–9 and 9–12 m). **b**, **c**, **d**, Zn, Pb and Cu concentrations (lower 25 % quartile, median and upper 75 % quartile), respectively, of each area. Differences in *lowercase letters* (*a*, *b* and *c*) and *capital letters* (*A*, *B* and *C*) indicate $p < 0.05$ (significant difference) and $p < 0.20$, respectively, by means of nonparametric multiple comparison (Jonckheere–Terpstra trend test)

~3 m inside the workshop based on a linear regression model ($r^2 = 0.99$). The outside (<0 m area) concentration of Zn in the 0–3 m area was similar to the indoor concentration. Pb concentrations peaked in the 6–9 and 9–12 m areas (i.e. 6–12 m; $p < 0.05$; Fig. 3c). Generally, Pb concentrations decreased as a function of distance from the 6–12 m area. Median Pb concentrations of 6–12, 3–6 and 0–3 m areas were 450–500, 380 and 210 µg/g-wet, respectively. Based on this information, we estimated $d_{1/2}$ of Pb as ~7 m inside the entrance (linear regression, $r^2 = 0.94$). The outside (<0 m area) concentration of Pb did not differ from the 0–3 and 3–6 m areas. Figure 3d suggests that Cu concentrations in surface soil showed the same trend in the *y*-axis direction as other heavy metals, although Cu concentrations at each area showed no statistical difference ($p < 0.20$). Analysing median Cu concentrations at 6–9, 3–6 and 0–3 m areas (720, 680 and 410 µg/g-wet, respectively), $d_{1/2}$ of Cu was estimated to be ~7 m inside (linear regression, $r^2 = 0.84$). Generally, heavy metals used in various e-wastes had lower mobility than organohalogen compounds. Polybrominated diphenyl ethers (PBDEs) and polychlorinated biphenyls (PCBs) were reported to transport from e-waste recycling sites for only a "short range" (i.e. distance of a few kilometres) (Li et al. 2011b). Present our study suggests that the transport range of heavy metals was only a few metres, that is, 10^3 times the "short range" of PBDEs and PCBs. Thus, these results suggest that heavy metals remain at the original e-waste recycling site. Heavy metals trapped in an e-waste recycling site may directly influence the health of e-waste workers at the original location rather than the surrounding habitat and environmental media.

Metal ratio

We calculated the ratio of Cu, Pb and Zn concentrations in surface soil to characterise pollution features at the e-waste recycling site (Fig. 4a). Pollution feature means character of balanced/unbalanced metal ratio. If unbalanced ratio compared with average representative soil, a source of specific heavy metal possibly exists near the point of unbalanced ratio. So, pollution sources of heavy metals were also discussed in this section. The metal ratio patterns were divided broadly into four groups (general, Cu-rich, Pb-rich and Zn-rich) by hierarchical clustering, as shown in Fig. 4b. In total, 70 % of the measurement points were classified as general, in which the concentrations of the three metals were similar to the average upper continental crust (Wedepohl 1995). Although the majority of areas are categorised as a general metal ratio pattern, the three heavy metals showed a variety of concentrations at each measurement point. The remaining 30 % of the measurement points belonged to unbalanced groups of metal ratios. Cu- and Pb-rich patterns were distributed in limited spots in the workshop (Fig. 4a). In this workshop, Cu-rich patterns were observed near where an e-waste worker was stripping coated Cu cable. Therefore, Cu-rich surface soil may be produced by the fine fragments derived from stripping activity. In contrast, Fig. 4a showed that Zn-rich patterns were found in the 6–9 m area (ref. Fig. 3a). Various e-wastes were covered with a corrugated iron board beside the 6–9 m area, as shown in Fig. 1c. The Zn-rich pattern may be due to Zn fine fragments from the corrugated iron board. Surficial distribution patterns of metal ratios (Cu, Pb and Zn) may be reflected by e-waste recycling activities and the species of e-waste products. However, few studies have evaluated the metal compositions in various e-waste products.

Variability and possibility of kriging

Surficial variability of heavy metal concentrations was assessed using a semivariogram, and the semivariances

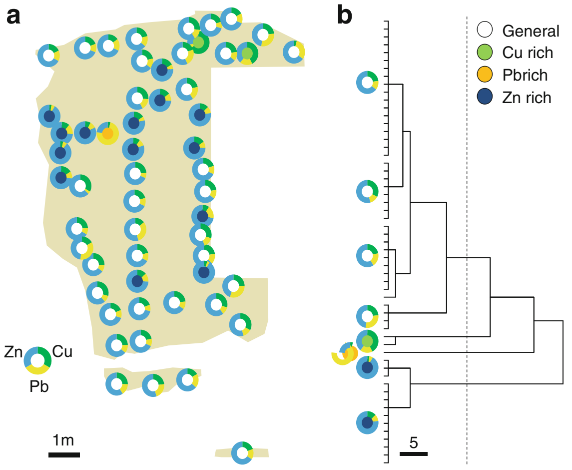

Fig. 4 Distribution pattern of the concentration ratio of heavy metals. **a** Concentration ratio of heavy metals (Cu, Pb and Zn). **b** Hierarchical clustering (Ward method) of heavy metal components in surface soil

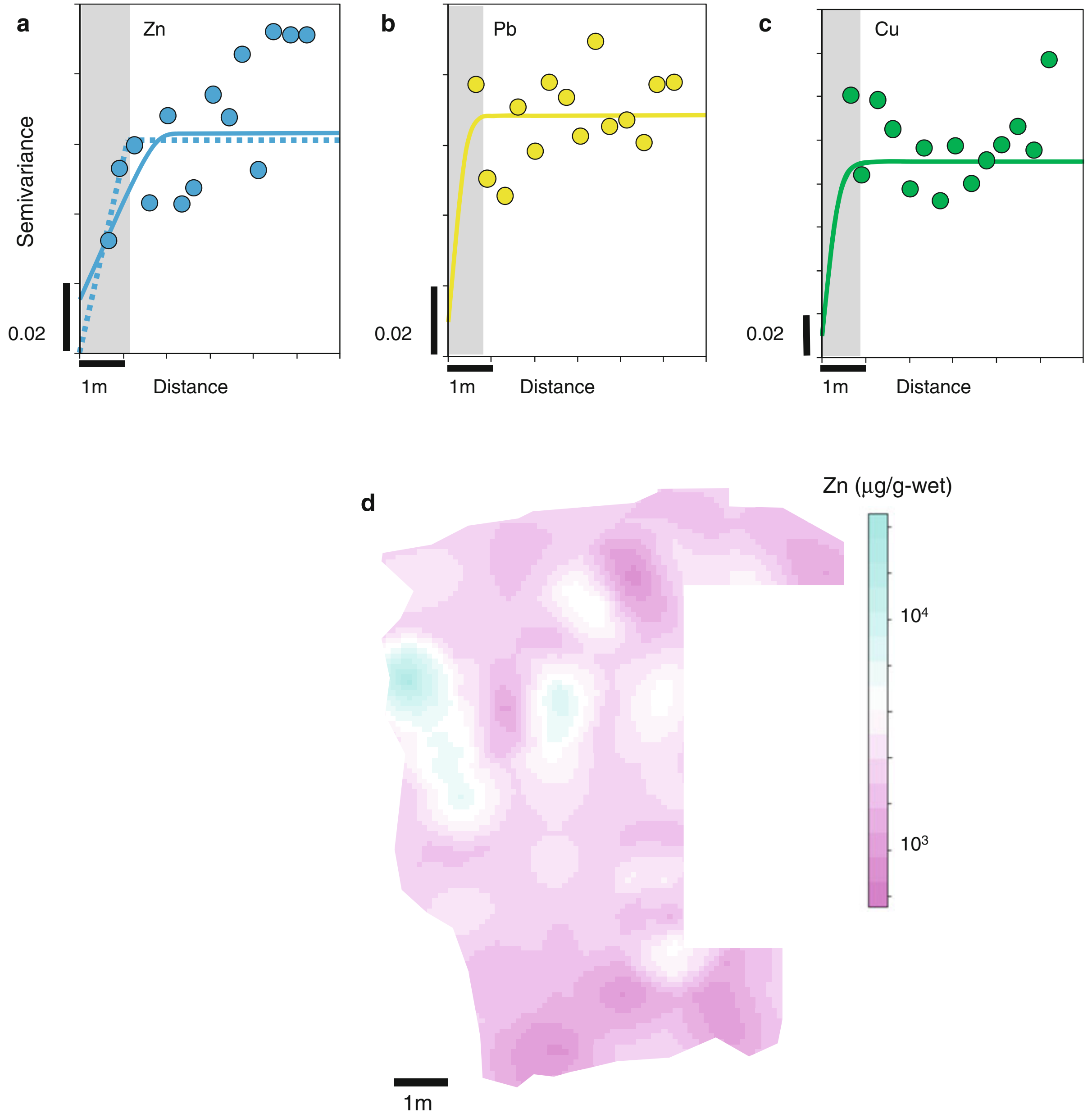

Fig. 5 Surficial variability of metal concentrations. Semivariograms of Zn (**a**), Pb (**b**) and Cu (**c**) in logarithmic concentrations. *Solid* and *dashed lines* are spherical and linear models, respectively. **d** Predictable distribution of Zn concentrations in inside soil calculated using kriging

of Zn, Pb and Cu were calculated by Eq. (1), as shown in Fig. 5a–c, respectively. The common logarithmic concentration was used, $z = \log_{10}$ (*concentration of metal*), due to the histograms of heavy metals showing improved lognormal distribution patterns, as well as the Sph model in Eq. (2), which fit better than the nonlogarithmic case. According to the results of structure analysis using the Sph model, the ranges of Zn, Pb and Cu were calculated as 2.0, 0.8 and 0.8 m, respectively (shaded area in Fig. 5a–c). Although surficial autocorrelation generally occurs at a specific range, no measurement points existed at <0.8 m for Pb and Cu. Therefore, the variance of the concentration between two points measured for Pb and Cu in surface soil diverged to the plateau by length-of-stride-scale stepping. Thus, the concentrations of Pb and Cu were not correctly estimated at an unmeasured point at a distance shorter than the length-of-stride scale by kriging. In contrast, Fig. 5a shows the concentration range of Zn (2.0 m) between two measurement points. However, Sph model sowed high nugget effect for Zn data as shown in Fig. 5a (solid line). So, we also considered Lin model. Lin model did not show nugget effect (i.e. zero) as shown in Fig. 5a (dashed line). Then, range of Zn indicated 1.1 m. Since some measurement points existed at <1.1 m for Zn, we attempted to estimate the concentrations of Zn at an unmeasured point using kriging, as shown in Fig. 5d. The kriged Zn map (Fig. 5d) was consistent with the above-mentioned Zn concentration gradient (Fig. 3d). High Zn pollution (>3,500 μg/g-wet) was detected in the 6–9 m area (ref. Fig. 3a). Near the entrance gate (0–3 m area) and in the 9–12 m area, Zn concentrations decreased. Concentrations of heavy metals had only a few-metre range variability in surface soil. This variability represented the difficulty in obtaining representative metal concentrations during sampling. In addition, this study demonstrated that a kriging map was generated based on on-site length-of-stride-scale measurements of Zn. However, assumption of spatial autocorrelation of Zn was thought to be rough by this kriging because of low resolution of on-site measurement. We will need to study optimisation of measurement to estimate autocorrelation in the future.

Conclusions

Length-of-stride-scale measurements of surface soil at e-waste recycling sites revealed a “small” pollution scale of heavy metals compared with toxic organohalogen compounds. The $d_{1/2}$ of Zn, Pb and Cu were estimated as $\sim$3, 7 and 7 m, respectively, inside the informal e-waste recycling workshop. Thus, distance from an entrance may affect heavy metal concentrations in surface soil. This is more predominantly noted in the case of e-waste dismantling and crushing where heavy metals remained at the original workshop and were not scattered in the surrounding environment. The on-site FP-XRF technique was also applied to study the metal ratios and to increase our understanding of the characteristic features of solid surfaces. Variability analysis of heavy metals showed decreasing surficial autocorrelation over metre ranges. The current on-site survey allowed Zn concentrations to be predicted; however, predicting Pb and Cu concentrations was not possible using kriging. The appropriate mesh size to construct a kriging map depends on the metal species and surficial distribution of metals. Furthermore, this on-site procedure is applicable to assess the pollution scale of other heavy metals in surface soil.

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