

SPECIATION AND MOBILITY STUDY OF SELECTED HEAVY METALS IN KOFE DUMPSITE SOIL OF JIMMA TOWN, JIMMA, ETHIOPIA

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Abstract

Speciation and mobility of selected heavy metals including Cd, Pb and Fe were investigated in Kofe dumpsite soils of Jimma Town, Jimma, Ethiopia. To study vertical mobility of the metals, soil samples were collected at three depth profiles comprising surface soil (0-10 cm), subsoil (20-30 cm), and bottom soil (40-50 cm) using soil auger. Similarly, for horizontal mobility assessment, soil samples were collected from 5 m, 10 m and 15 m distances starting from the edge of the dumpsite in east, west, north and south directions. Control soil samples were also collected at the same depth profiles, from 200 m away from the dumpsite. The collected samples were air dried, sieved as well as ground before processing utilizing a Community Bureau of Reference (BCR) sequential extraction procedure. The fraction of the target metals in the extracted samples were then determined by flame atomic absorption spectroscopy (FAAS). The mobility factor (%MF) of the target metals was investigated in both vertical and horizontal variations and the obtained results showed that the target metals have exhibited both vertical and horizontal mobility. The texture, organic matter and pH of the soil samples were also analyzed for correlation study. Variation among obtained results, i.e., %MF and concentration of analytes, were evaluated using one way ANOVA at ($P \leq 0.05$). It was observed that Cd and Pb exhibited vertical mobility and thus could lead to pollution of the underground water. Similarly, the highest amount of Pb in the bioavailable fraction may also indicate its horizontal mobility and its high tendency to pollute surface water, sediment as well as agricultural products.

Key Words: *Speciation, Mobility, Sequential extraction, Heavy metals*

Introduction

Soil is a reservoir of many heavy metals from both natural and anthropogenic sources (Abul, 2010). Metals exist in the soil in various forms and most of them have toxic effects on living organisms when exceeding certain concentration limits (Oluseyi *et al.*, 2014). Dumpsite is one of the major

causes of soil, underground and surface water as well as agricultural crops pollution by releasing toxic chemicals such as heavy metals (Oluseyi *et al.*, 2014). Heavy metals can move in soil profile vertically or horizontally from the point where they are accumulated to other areas via different mechanisms. They may also affect the health of human

beings; from simple poisoning to cancer, heart diseases and genetic abnormalities (Thomas, 2015; Jena *et al.*, 2013).

The toxicity, environmental mobility and potential risk level of heavy metals are strongly dependent on their chemical speciation (Uduma and Jimoh, 2013). Speciation study involves identification and quantification of one or more individual chemical forms of the metals that occurs in a given sample. Such study provides sufficient information about the nature and the fraction of individual chemical species of metals in the soil. It also provides information about the affinity and strength with which metals are bound to the soil components (Fadiran *et al.*, 2014; Zerbe *et al.*, 1999).

The fractions of heavy metals in the soil profile, varies with the origin of the metals, availability, their forms of occurrence and mobility (Thomas, 2015). High fractionation of the total metals in the water soluble, exchangeable, bound to carbonates and reducible fractions is a good indication of anthropogenic pollution (Agbaire and Akporhonor, 2014). Similarly, the mobility of metals in the soil profile is also strongly dependent on their chemical forms (Sungur *et al.*, 2014). It is usually determined based on the relative amount of the metal in water soluble, exchangeable as well as bound to carbonates fraction and their overall potential mobility is usually expressed as mobility factor (% MF) (Wuana *et al.*, 2012).

In general, several attempts were made to assess the mobility of heavy metals in contaminated soils and sediments using sequential extraction procedure so as to separate the mobile fraction which is responsible for environmental pollution (Sungur *et al.*, 2014; Baruah *et al.*, 2011). Even though, disposal of wastes at Kofe dumpsite has used for more than a couple of decades, no study has been conducted regarding the mobility of heavy metals in the dumpsite soils through speciation. Therefore, this study was aimed to assess the speciation and mobility of selected heavy metals including Cd, Pb and Fe using a four steps BCR sequential extraction method (Ure *et al.*, 1993) followed by FAAS.

Materials and Methods

Sample Sites

Soil samples were collected from Kofe dumpsite of Jimma town, which is located in southwestern part of Ethiopia. The dumpsite is located at about 4 km in southwest of the town. Both solid and liquid municipal wastes from hotels, communities and higher learning institutions, like Jimma University and Jimma Teacher Training College have been discharged at the dumpsite for more than two decades. Figure 1, shows the map of Jimma zone, Jimma Town and Kofe dumpsite.

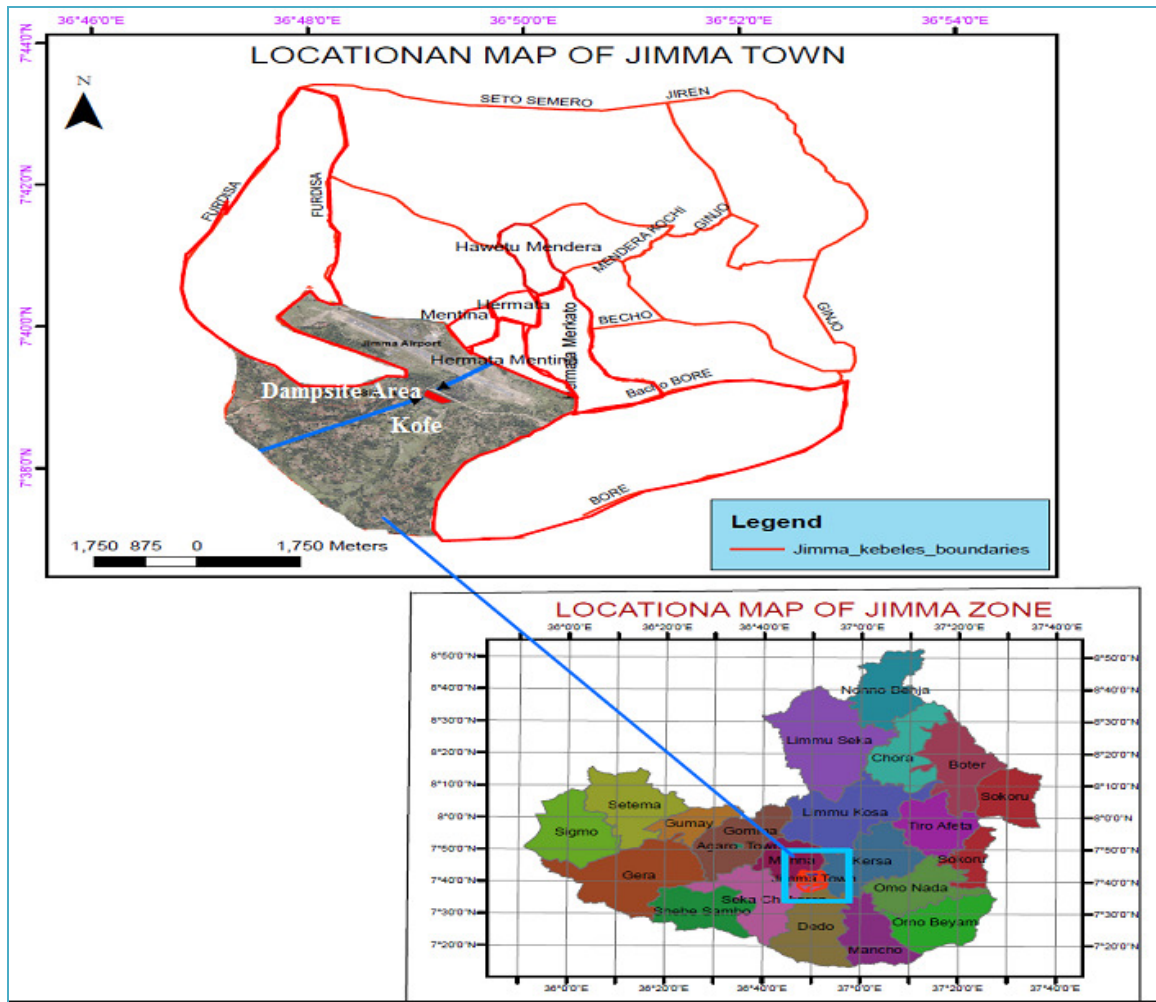


Figure 1: Sketch map of Jimma zone, Jimma town and Kofe dumpsite

Sample Collection

To study the vertical mobility of the metals, soil samples were collected from 12 randomly selected sites, at three depth profiles: surface soil (0-10 cm), subsoil (20-30 cm), bottom soil (40-50 cm) using soil auger. Similarly, for horizontal mobility study, the samples were collected at 5 m variations (i.e., at 5, 10, 15 m), in east, west, north and south directions, starting from the edge of dumpsite. The control soil samples were also collected, at the same depth profiles, from about 200 m distance away from the dumpsite. Subsequently, the collected

samples were homogenized according to their profiles at the sampling site and 1 kg composite samples were separately taken into polyethylene bags for each profile. Eventually, the samples were transported to laboratory, where they were oven dried at 105 °C prior to storing below 4 °C.

Sample Preparation Techniques

The oven dried samples were further exposed to air and dried to constant weight. The impurities were removed by sieving with 2 mm pore size sieve. The samples were then divided into half, quartet and so on until the desired

amounts of laboratory samples were taken. The samples were again oven dried at 105 °C and then, ground into fine sizes using mortar and pestle. Finally, the samples were stored in clean plastic bags and kept at room temperature until sample preparation step was carried out.

Physicochemical Properties of the Dumpsite Soil

Physicochemical properties including pH, soil organic matter (% OM) and soil texture were investigated. pH was determined using supernatant suspension of 1:2.5 soil water mixtures. % OM was analyzed using wet oxidation method and after mechanical analysis of soil bulk density by standard procedure (Reeuwijk, 2002). Soil textural classification was investigated using chart method (Antoniadis, 1998).

Sequential Extraction Procedures

For speciation and mobility study of the target metals, a Community Bureau of Reference (BCR) sequential extraction procedure (Ure *et al.*, 1993; Tokalioglu *et al.*, 2000; Katana *et al.*, 2013) was adopted. The procedure involves four fractionation steps: the water and acid

soluble mobile fraction (F-I); reducible fraction (F-II); oxidizable fraction (F-III); and residual fraction (F-IV).

Wet Digestion Procedure

Baker and Amacher method was employed for wet digestion (Zeng *et al.*, 2002).

Analytical Performance Study of Target Metals

For quantitative determinations, calibration curves were constructed by preparing series of solutions containing different concentrations of the target metal standards in 1 M HNO₃. The calibration curves were obtained by plotting the measured absorbance versus concentration and demonstrated good linearity with coefficient of determinations (r²) of 0.999, 0.990 and 0.994 for Cd, Pb and Fe, respectively.

Limit of detections (LODs) and limit of quantifications (LOQs) of the method were determined from the standard deviation of several blank determinations (Christian, 2004). Recovery studies were also performed in two ways: 1) using the method reported by (Uduma and Jimoh; 2013), which is determined as follows.

$$(\%R) \text{ Sequential extraction} = \left(\frac{n \sum \text{Fractions obtained by sequential procedures}}{\text{wet digestion with strong acids}} \right) \times 100$$

2) by spiking known concentration of the target metal standards on to the sample and calculated as follows.

$$(\%R) \text{ total analysis by spiking} = \frac{\text{Aspiked sample} - \text{non spiked sample}}{\text{Amount of metal spiked}} \times 100$$

In addition, the mobility of the target metals was also evaluated in terms of mobility factor %MF (Wuana *et al.*, 2012), which is obtained as follows.

$$\%MF = \frac{\text{con. F-I}}{\sum(\text{con. F-I} + \text{con. F-II} + \text{con. F-III} + \text{con. F-IV})} \times 100.$$

Statistical Analysis

One way ANOVA ($p \leq 0.05$) was employed to evaluate the correlations of the concentrations as well as the mobility factors of the heavy metals fractions.

Results and Discussion**Physicochemical Prosperities of the Dumpsite Soil**

Table 1: The physicochemical properties of the soil samples

Sample	Profile	pH	%OM	Soil texture
Dump site soil sample	Upper	5.80 ± 0.06	2.90 ± 0.05	Silty Clay loamy
	Middle	4.70 ± 0.05	3.80 ± 0.02	Silty loam
	Bottom	4.30 ± 0.10	3.87 ± 0.02	Silty loam
Horizontal soil sample	5 m	4.70 ± 0.02	3.23 ± 0.10	Silty clay loam
	10 m	4.40 ± 0.03	3.90 ± 0.02	clay loam
	15 m	4.10 ± 0.07	4.52 ± 0.01	Clay
Control soil sample	Upper	4.60 ± 0.02	5.68 ± 0.03	Clay
	Middle	4.30 ± 0.03	4.60 ± 0.01	Clay
	Bottom	4.00 ± 0.01	3.42 ± 0.02	Clay

It was observed that pH of soil samples were varied from 5.80–4.30; 4.60–4.00 and 4.70–4.10 for dumpsite, control and horizontal soil samples, respectively. In all cases, pH values were in acidic range (i.e., < 7.00), and their acidity increases vertically as well as horizontally and thus facilitating desorption of the metals from the soil (Ogbemudia and Mbong, 2013). The finding also demonstrated control soil sample has larger %OM than the dumpsite soil samples. Such variation could be attributed to the difference in the level of decomposable plant and waste matter of the soils (Richards *et al.*, 2000; Matos *et al.*, 2000). In contrast to the control soil samples, %OM in dumpsite soil samples exhibited an increment with depth in vertical profiles. This could be related to the availability of non-decomposable materials such as plastics,

rubbers and other materials at the surface of the dumpsite soils (Lawan *et al.*, 2012). The texture of dumpsite soil samples were also varied from the control sample, which were completely clay type, whereas, dumpsite soils have silty clay loam and clay loam textures, which could facilitate the percolation of heavy metals and other colloids to the lower profile (Soriano-Disla *et al.*, 2011).

Limits of Detection Quantification

LODs and LOQs of the method were determined for each fraction (Table 2). It was observed that Cd and Fe exhibited different LODs and LOQs in F-I, but same values in other fractions. In the study, metal concentrations obtained below LOD as well as between LOD and LOQ, at the particular fraction were reported as not detected (ND) and detected not quantifiable (DNQ), respectively.

Table 2: LODs and LOQs of the target metals in µg/g

Fraction	Cd		Pb		Fe	
	LOD	LOQ	LOD	LOQ	LOD	LOQ
F-I	0.02	0.07	0.01	0.03	0.07	0.23
F-II	0.01	0.03	0.01	0.03	0.05	0.17
F-III	0.01	0.03	0.01	0.03	0.05	0.17
F-IV	0.01	0.03	0.01	0.03	0.05	0.17

Speciation and Vertical Mobility of Selected Heavy Metals in the Dumpsite Soils

The concentrations of the target metals in different fractions are presented in Table 3.

Table 3: Concentrations of the metals by fractions in the vertical profiles (Mean ± SD, n = 6)

Profile	Fraction	Dumpsite soil (µg/g)			Control soil (µg/g)		
		Cd	Pb	Fe	Cd	Pb	Fe
Upper	F-I	0.56 ± 0.01	5.00 ± 0.01	5.00 ± 0.04	ND	ND	6.00 ± 0.02
	F-II	2.43 ± 0.02	5.10 ± 0.01	4.20 ± 0.02	ND	ND	24.00 ± 0.03
	F-III	1.32 ± 0.03	4.40 ± 0.02	7.30 ± 0.14	ND	ND	3.20 ± 0.04
	F-IV	0.22 ± 0.03	1.20 ± 0.04	3.50 ± 0.40	ND	ND	ND
	SF	4.34 ± 0.09	15.70 ± 0.08	20.00 ± 1.50	-	-	33.20 ± 0.09
Middle	F-I	0.85 ± 0.02	9.00 ± 0.05	8.10 ± 0.21	ND	DNQ	5.00 ± 0.05
	F-II	1.35 ± 0.03	5.50 ± 0.03	9.50 ± 0.04	DNQ	DNQ	10.00 ± 0.03
	F-III	0.51 ± 0.05	5.50 ± 0.14	5.40 ± 0.06	DNQ	ND	12.00 ± 0.02
	F-IV	0.87 ± 0.10	1.50 ± 0.05	1.50 ± 0.05	ND	ND	1.50 ± 0.03
	SF	2.57 ± 0.20	20.50 ± 0.27	24.50 ± 0.36	DNQ	DNQ	28.50 ± 0.13
Bottom	F-I	0.52 ± 0.30	9.50 ± 0.22	5.20 ± 0.02	ND	ND	1.50 ± 0.12
	F-II	0.68 ± 0.20	3.20 ± 0.04	13.30 ± 0.03	ND	ND	7.30 ± 0.08
	F-III	ND	5.00 ± 0.02	3.50 ± 0.03	ND	ND	8.00 ± 0.40
	F-IV	ND	0.70 ± 0.04	ND	ND	ND	9.00 ± 0.06
	SF	1.20 ± 0.50	18.40 ± 0.12	22.50 ± 0.08	-	-	25.80 ± 0.66

ND: Not detected; DNQ: Detected not quantifiable; F-I: Mobile fraction; F-II: Reducible fraction; F-III: Oxidizable fraction; F-IV: Residual fraction; SF: Sum of fractions

It was observed that concentrations of the metals in F-I were varied in vertical profiles ranging from 0.52 – 0.56, 5.00 – 9.50 and 5.00 – 5.20 µg/g for Cd, Pb and Fe, respectively. Specifically, the concentration of Pb in F-I increases down the vertical profile. The ANOVA test (p ≤ 0.05) also demonstrated the existence of significant difference in Cd, Pb and Fe concentrations in vertical soil profiles, indicating the high mobility of Pb and Cd towards the pollution of underground water (Sherene, 2010; Kanmani and

Gandhimathi, 2013). In all fractions of the control soil samples, Pb and Cd were not detected and/or detected below the LOQ, demonstrating the absence of the natural sources of these metals in the control site soils (Tripathi and Misra, 2012). The bioavailable fraction of Fe in the control soil has not exhibited significant difference down the soil profile which might be due to the clay nature of soil and high % OM in the upper profile (Lawan *et al.*, 2012; Violante *et al.*, 2010).

It was also observed that in the different fractions of the dumpsite and control soil samples, the studied heavy metals have exhibited variations. Earlier reports also confirmed the possibility of variations of heavy metals in different fractions of the soil (Zerbe, 1999). Compared to the control soil samples, Pb and Cd were found in higher concentrations in all profiles of the dumpsite soils, indicating the suitable conditions for their existence in the mobile fractions of the dumpsite soils (Jena *et. al.*, 2013; Kanmani and Gandhimathi, 2013). In suitable conditions oxidizable and reducible fractions of Cd and Pb could be easily converted to the bioavailable/mobile fraction (Antoniadis, 1998). The

dumpsite and its surrounding area soil samples have also exhibited higher total concentrations of the studied metals. This could be attributed to the enrichments of the dumpsite soils with the studied metals resulting from uncontrolled solid and liquid municipal as well as commercial wastes dumped on the area (Mohammad and Kenneth, 2012; Bronius and Vaida, 2009).

Vertical Mobility of Target Heavy Metals in the Dumpsite Soil

Vertical mobility study provides important information about the level of toxicity or bioavailability the metals in the environment. Figure 2, shows vertical mobility of target metals in the dumpsite soils.

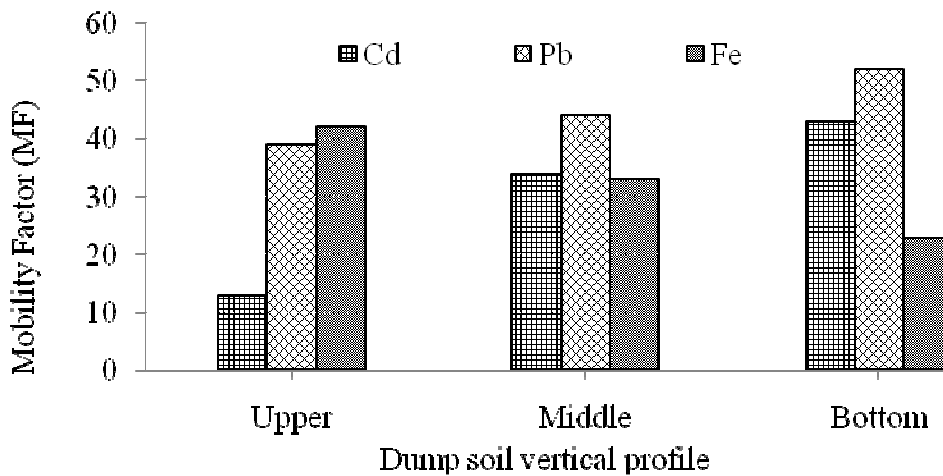


Figure 2: Vertical mobility factors of target metals in the Kofe dumpsite soils.

As indicated in Figure 2, mobility of Pb and Cd increase down the vertical soil profile. Therefore, the bottom dumpsite soil contains higher mobile fractions of toxic heavy metals, whose fate might be resulted in pollution of the surrounding underground water (Sherene, 2010; Kanmani and Gandhimathi, 2013).

However, Fe mobility decreases down the vertical profiles, indicating the presence of the majority of its mobile fraction at the upper surface.

In general, mobility of the metals down the dumpsite soil profiles follows the trend: Pb > Cd > Fe. The ANOVA test ($p \leq 0.05$) also confirmed the presence of

significant variation of the metals down the soil profile. Similarly, the mobile fractions at each profile follow various trends, i.e., in upper Fe > Pb > Cd; middle Pb > Fe ≈ Cd; and bottom Pb > Cd > Fe. In all cases, the highest mobile fraction was observed for Pb, indicating a serious environmental concern of dumpsite soils.

Speciation and Horizontal Mobility of Target Heavy Metals

Speciation and horizontal mobility study can provide information about how far the mobile fractions of toxic metals and/or plant nutrients could migrate in soil surface from their point source to the surrounding areas. The concentrations of the studied metals in the horizontal soil profiles are presented in Table 4.

Table 4: Concentration of target metals in horizontal soil samples

Profile	Fraction	Concentration (µg/g)		
		Cd	Pb	Fe
5 m	F-I	0.25 ± 0.02	7.80 ± 7.80	9.20 ± 0.04
	F-II	0.62 ± 0.10	5.20 ± 0.04	10.40 ± 0.07
	F-III	0.79 ± 0.03	5.20 ± 0.04	2.10 ± 0.30
	F-IV	ND	0.78 ± 0.05	ND
	SF	1.66 ± 0.15	18.98 ± 0.15	21.70 ± 0.41
10 m	F-I	0.65 ± 0.10	12.70 ± 0.05	9.80 ± 0.05
	F-II	0.64 ± 0.10	4.20 ± 0.30	10.20 ± 0.30
	F-III	0.45 ± 0.10	5.00 ± 0.20	5.00 ± 0.23
	F-IV	0.26 ± 0.12	ND	ND
	SF	2.00 ± 0.42	21.90 ± 0.55	25.00 ± 0.58
15 m	F-I	0.82 ± 0.02	28.80 ± 0.04	10.00 ± 0.03
	F-II	0.24 ± 0.10	4.00 ± 0.10	15.30 ± 0.50
	F-III	0.44 ± 0.15	1.20 ± 0.02	ND
	F-IV	ND	2.50 ± 0.05	ND
	SF	1.50 ± 0.27	36.50 ± 0.21	25.30 ± 0.53

The findings indicated that the target metals were shown different concentrations of mobile fractions ranging from 0.25 – 0.82 µg/g, 7.8 – 28.8 µg/g and 9.2 – 10.00 µg/g for Cd, Pb and Fe, respectively, as the distance increases from the dumpsite. The increase in the concentration of Cd and Pb in mobile fraction might be attributed to the acidity and higher % OM of the soil, which facilitate migration of the metals in the

mobile fraction, as has been also indicated in the literature (Ashworth and Alloway, 2008). The total concentrations of Fe also increase with distance up to 10 m away from the dumpsite and then, become constant for longer distances. However, its bioavailable fraction has not shown significant variation, may be due to the preference of Fe to bound in reducible fraction (Tokalioglu *et al.*, 2000).

Horizontal Mobility of the Target Heavy Metals

As has been mentioned earlier, mobility is described in terms of % MF

(Ashworth and Alloway, 2008). Figure 3, illustrates horizontal mobility of the studied metals.

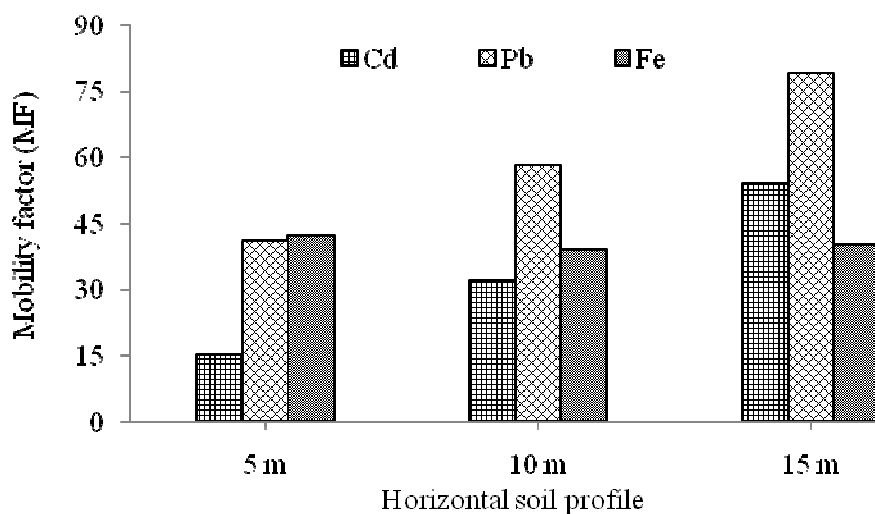


Figure 3: Horizontal mobility factor of the target metals.

The finding demonstrated linear increment in the mobility of Pb and Cd with distance from the dumpsite. This could be happened due to the increased in the acidity and the % OM of the soils as one goes away from the dumpsite (Ogbemudia and Mbong, 2013; Richards *et al.*, 2000). The AOVA test (at $p \leq 0.05$) also revealed the existence of significance differences in Cd and Pb mobile fractions along horizontal profiles. The trend of their horizontal

mobility shows the possibility of contamination of the soil, sediment, surface water and agricultural products in nearby areas as dumping is continued for longer time (Mohammad and Kenneth, 2012; Wuana *et al.*, 2012; Bronius and Vaida, 2009).

Recovery Study of the Target Heavy Metals

The results of recovery studies for both sequential extraction and total analysis are presented in Table 5.

Table 5: Speciation recovery and wet digestion recovery of the target metals

Sample type	Dumpsite Soil (vertical) samples									Horizontal soil sample									Control soil sample											
Profile	Upper			Middle			Bottom			5 m			10 m			15 m			Upper			Middle			Bottom					
Analyte	Cd	Pb	Fe	Cd	Pb	Fe	Cd	Pb	Fe	Cd	Pb	Fe	Cd	Pb	Fe	Cd	Pb	Fe	Cd	Pb	Fe	Cd	Pb	Fe	Cd	Pb	Fe	Cd	Pb	Fe
%R	93	90	69	71	106	97	77	108	87	87	97	99	84	100	104	80	99	92	-	-	98	115	81	111	-	-	86			
%RS	80	84	92	80	106	92	89	92	106	94	90	94	80	90	102	88	92	90	99	100	106	104	87	92	100	100	96			

%R: Recovery for speciation; % RS: Recovery for spiking

The recovery values of the target metals, by both methods: sequential extraction procedure and spiked acid digested samples were ranging from 69–115% and 80–106%, respectively, indicating acceptable recoveries. Comparison of the two recovery studies indicates that the total amounts of analytes obtained by summation of the individual fractions in the speciation analysis and total analysis by wet digestion procedure are almost the same.

Conclusion

In this study, speciation and mobility of heavy metals including Cd, Pb and Fe in Kofe dumpsite were investigated. The findings demonstrated that the studied metals exist in the dumpsite soils in different form and also exhibited vertical and horizontal mobility. As compared to the control soil sample, the dumpsite contain higher amount of Cd, and Pb, attributing to anthropogenic sources resulting from the availability of solid and liquid wastes at the dumpsite. The variation in the physicochemical properties of soils such as pH, %OM and texture might be the cause for the variations of concentrations of bioavailable fractions of the metals in the dumpsite soil. Cd and Pb exhibited both horizontal and vertical mobility and thus, they pollute the nearby surface water, sediments and agriculture crops. Similarly, larger concentration of Fe was observed in reducible fraction in the dumpsite and oxidizable fraction in the control soils, indicating its low bioavailability relative to Cd and Pb. The findings of the study demonstrated that the studied dumpsite soil is not suitable or safe for agricultural purposes.

Acknowledgments

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