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# Assessing the impact of dewatered faecal matter on mobilization of heavy metals (Cu, Zn, Pb and Cd) in compost and garden soils

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# Keywor ds

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# Introduction

Production and application of compost from Municipal Solid Waste (MSW), including organic wastes is gaining grounds in Ghana.

Composting has now become the most accepted way of municipal solid waste disposal as municipal solid wastes keep increasing and soils continue to lack organic matter. This makes it a better alternative from an economic and environmental point of view as compared to other forms of waste disposal e.g. landfills and incineration (Eriksen et al., 1999; Otten, 2001; Wolkowski, 2003).

In view of these, compost was developed by International Water Management Institute (IWMI) from a well sorted MSW and co-composted with Dewatered Faecal Sludge (DFS) and ammonium sulphate as enrichment for agricultural use. However, the presence of organic and inorganic contaminants in composts, (e.g. pesticides residues and heavy metals) poses grave risk to the environment. These have lead to restricted agricultural use of compost (Merian, 1991; Hargreaves et al. 2007). The total content of heavy metals in compost is of primary importance and may be useful as a global index of contamination (McBride, 1995). It is therefore essential that the amount of these elements in the compost destined for agricultural use does not exceed thresholds which can cause damage to either soil fertility or the food chain (Petruzzelli, 1996). It has been recognized that total soil metal contents alone are not a good measure of bioavailability and do not provide useful tool to assess potential risks and toxicity from soil contamination (Gooddy et al., 1995; Ge et al., 2000; Young et al., 2000). Instead, the different forms in which heavy metals exist in soil, sediment and compost give a better understanding of their behaviour in these media. Different forms of heavy metals have different mobilities and bioavailabilities which are responsible for uptake, contamination of soil and leaching into

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# ABSTRACT

A well sorted municipal solid waste which was composted and co-composted with other materials such as dewatered faecal sludge and ammonium sulphate as enrichment were used in the amendment of a 15 kg soil contained in 147 (43.2 x 35.6 cm) polythene bags for two cropping seasons. Impact of the dewatered faecal sludge on the speciation of heavy metals, Cu, Zn, Pb and Cd in the co-compost (Co), enriched co-compost (Eco) and garden soils (S) were evaluated. The results of the analysis reveal Co and Eco have about 50 % of Cu, 75 – 80 % of Zn and 80 – 85 % Cd highly bound organically. Lead was 60 % bound to organic fraction in Co and Eco, same as the other composts. The amended soils had 30 – 50 % Cu and 30 % Zn organically bound as the dominant fraction. Lead shifted and was 55 – 65 % dominant in the residual fraction whiles Cd was below detection.

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underground water bodies (Lake et al., 1984; Petruzzelli, 1989). These different forms are often referred to as species and the process of identifying them is known as speciation. Speciation is defined as the active process of identification and quantification of the different defined species, forms or phases in which an element occurs in a material (Ure, 1991). The technique often employed is the sequential chemical extraction and this is done by treating the same sample successively with solutions differing in their force of leaching toxic metals. The methods designed by Tessier et al. (1979) and Rudd et al. (1988) are considered to be the most standard ones and used by many authors e.g. Ciba et al. (1998) in analysis of soil, compost and other environmental samples.

The speciation of these metals depends on the physical, chemical and composting process. The amount of the metal form elucidated depends on the pH, redox potential, total metal content, temperature and other inherent factors associated with the complexing ligand forms (Iwegbue et al., 2007). The present study assesses the impact of the addition of dewatered faecal sludge (DFS) on the distribution of heavy metals (Cu, Zn, Pb and Cd) in compost and their effect on the mobility of the heavy metals in amended soils after first and second cropping season.

# Materials and methods

The compost and its derivatives were collected from IWMI/SANDEC co-composting plant at Buobai, Kumasi in the Ashanti Region. They were prepared from market waste, dewatered faecal sludge and ammonium sulphate from the Kumasi market and the metropolis.

## Sample preparation

Sandy loam soil (Vertic Lexisol) collected from the University of Ghana, Department of Crop Science Research Farm in Accra. This was sieved through a 2 mm mesh and about

15 kg was used to fill 147 (43.2 x 35.6 cm) polythene bags. The

compost and its derivatives were applied as treatments. A soil sample (S) was used as control. The rate of application was 150kg N/ha. Each treatment was in triplicate making 48 composite samples. The design used was Randomized Complete Block Design (RCBD) (Gomez and Gomez, 1984). Maize (Zea mays L) was cultivated for two cropping seasons on the amended soils. After harvest the various amended soils were collected and sieved through 2 mm mesh and kept in a well labeled white polythene bags and stored for chemical analysis. The uncultivated soil, compost and its derivative were also analyzed for heavy metal contents.

#### Compost and soil analysis

The soil samples were collected and air dried for 7 days at temperature of  $29^{\circ}$ C. The samples were sieved and used for physical and chemical analysis of the soils. Compost pH was measured in compost water ratio of 1:5 (TMECC, 2002). Soil pH was measured in soil water ratio of 1:1 (Black, 1965) using digital Metrohm 691 pH-meter. Total organic carbon (TOC) was analyzed by wet oxidation of organic matter according to Walkley and Black (1934). Particle size composition of the soil was analyzed by the Bouyoucos Hygrometer method (Bouyoucos, 1962). The total heavy metal analysis was done by digesting soil samples in 1:1 (HNO3: HCIO<sub>4</sub>) (Hofstede, 1994) and measuring using the AAS (Perkin Elmer A Analyst 400) at the Ecological laboratory, University of Ghana, Legon.

## Speciation and measurement of metal fractions

Using the Rudd et al., 1998 chemical extraction method, the metal fractions were obtained after the treatments in the (Table 2). 0.4g of each replicate sample was successively extracted at  $27^{\circ}$ C after shaking for 16hrs using mechanical shaker.

After each successive extraction, separations were made by centrifugation. Triplicate of each extract were centrifuged at 2500 rpm for 30 minutes and the supernatant were decanted and filtered using 0.45 $\mu$ m membrane filters and acidified with 1% (v/v) nitric acid. Metal analyses were carried out using Atomic Absorption Spectrometer (Perkin Elmer AAnalyst 400). Spiking was done intermittently to ensure precision and accuracy. The residue was washed with 20 ml of deionised water followed by vigorous hand shaking, and then by 30 minutes of centrifugation after each successive extraction.

The metals contents in the various fractions were determined at the following wavelengths (nm): Cu, 324.7; Zn, 213.9; Pb, 283.3 and Cd, 228.8 in air-acetylene flame. The instrument detection limits are 0.002, 0.002, 0.03 and 0.001 mg/l respectively and percentage recovery for spikes were generally between 90 - 110%.

# **Results and discussions**

## Soil physicochemical properties

The compost and its derivatives show an alkaline pH (7.4 – 8.0) while the soil material is slightly acidic (5.1). The amended soils were also slightly acidic with S + C - 6.0, S + Co - 5.8, S + Ec - 5.2 and S + Eco - 5.3. This makes the mobility and availability of metals in the composts very difficult whiles that of the soil materials possible. pH plays major role in determining the availability of metals in solution (Adriano, 1986; Sterckeman 2000; Bradl, 2004). Every unit decrease in pH results in approximately 2- fold increases in the concentrations of metals such as Zn, Ni, Cd etc. (Christensen, 1984). The availability of heavy metals (Cd, Pb, Ni, and Cu etc.) to plants decrease strongly as pH increases from 4 to 6 (Woodbury, 1992). In alkaline soils, Cd, Cu, Pb, Ni and Zn are usually unavailable for plant uptake because they are precipitated due to low solubility

at high pH (Ozores – Hampton et al., 1994). Conversely, acidic environment enhances the mobility and availability of metals. Metals bound to carbonates are very sensitive to pH changes and are therefore leached as the pH is lowered (Perez – Cid et al., 1999).

The percentage total nitrogen in the compost and its derivatives ranges from 1.22 - 3.1 and that of the soil was 0.09. Total percentage phosphorus however was C - 1.80, Co - 1.60, Ec - 0.42, Eco - 1.14 and that of soil was the lowest at 0.02.

Nitrogen and phosphorus can come from many sources, including animal manures, organic fertilizers and commercial fertilizers. Phosphorus (P) is known for being tightly bound to oxidic and particularly andic clays, regardless of pH. Many soil test results have shown extremely high soil P levels in areas where manure or commercial fertilizers have been applied for many years (Robotham et al., 2004). Phosphorus and micronutrients such as copper and zinc also decrease in their plant availability at high pH. In general, many plants will do well in a soil pH range of 5.5 to 7.5.

The Electrical Conductivity (EC) of the compost and its derivatives ranges from 4.3 mS/cm to 318 mS/cm and that of the soil material was 2.0 mS/cm. This result reveals that the composts have clay and colloidal materials which will conduct electricity more in solution than the soil materials. Electrical Conductivity of a solution is a measure of the ability of the solution to conduct electricity. The electrical conductivity of soils varies depending on the amount of moisture held by soil particles and this variation follows the order: Sands < silts < clays. Consequently, EC correlates strongly to soil particle size and texture (Grisso et al., 2009).

The percentage organic matter ranges from 0.80 - 17.64 and was in the order: Co > Eco > C > Ec > Soil. Co and Eco have higher organic matter due to the addition of dewatered faecal sludge which is purely an organic material. These two composts may provide more available ligands for organo-metallic complex formation than the other two C and Ec.

Organic matter content is the measure of carbon based materials in compost and soils, and it plays an important role in soil structure, nutrient availability, and water holding capacity (Epstein et al., 1976). Organic matter in soils can decrease the plant availability of cations and does not react with all metals equally, but generally forms complexes decreasing in strength in the following order Cu > Pb > Zn > Cd (Lagerweff et al., 1977). The uptake and availability of plant nutrients, particularly micronutrients such as Cu and Mn, and the effectiveness of herbicides are greatly affected by soil organic matter.

The percentage representation of the texture of soil material used shows 60.15 % sand, 12.35 % silt and 27.5 % clay. This soil is sandy but the percentage clay present has the ability to adsorb more of the metals. The distribution of particles of soil such as clay, silt and sand fractions play an important role in explaining and forecasting the true behaviour of pollutants (Rivière, 2000). Clay fractions in soils are very effective in the adsorption of heavy metals (Kirpichtchikova et al., 2006).

# Total metal content in soil, compost and compost derivatives

The total heavy metal concentration in soil, compost and its derivatives ranges from  $15.58 - 78.71 \text{ mg kg}^{-1}$  for Cu,  $25.71 - 410.21 \text{ mg kg}^{-1}$  for Zn,  $53.54 - 78.62 \text{ mg kg}^{-1}$  for Pb and  $0.00721 - 0.1125 \text{ mg kg}^{-1}$  for Cd. The total Cu, Zn, Pb and Cd concentration found in soil, compost and its derivatives were below the limit (100 mg kg<sup>-1</sup>, 400 mg kg<sup>-1</sup>, 150 mg kg<sup>-1</sup> and 1.5 mg kg<sup>-1</sup> respectively recommended for compost or biosolids to

be used in agriculture (WRAP, 2002) though the latter i.e. 410.21 mg kg<sup>-1</sup> in Zn is slightly above the upper limit of 400 mg kg<sup>-1</sup> recommended. The result however does not give a better understanding of their effect on soil, crops and underground water bodies.

#### Metal Speciation in the various compost samples and soil

Copper extracted from compost and its derivatives are presented in Fig. 1. The predominant copper extracts were in organically bound fractions of the compost and its derivatives. The concentrations 22.43 mg kg<sup>-1</sup> (Co) and 20.32 mg kg<sup>-1</sup> (Eco) were seen to be higher compared to 6.83 mg kg<sup>-1</sup> (C) and 6.10 mg kg<sup>-1</sup> (Ec) fractions. This trend was followed by the order sulphide bound > exchangeable > adsorbed > carbonate bound > residual > water soluble. Cu concentrations in the untreated soils were high as observed in the compost and its derivatives and mainly detected in the organic and residual fractions. Copper was high in the organically bound fraction for compost and its derivatives and the soil due to the high stability complexes Cu forms with organic matter (Gould and Genetelli, 1984). It should be also noted that Eco and Co were higher in organic matter content (Table 3) because of the addition of dewatered faecal sludge in their preparation. The exchangeable fractions which are the next highest fraction are weakly adsorbed metals retain on the solid surface by weak electrostatic interactions and are released by ion-exchange processes. This fraction is most readily released in to the environment and could also be available for plant uptake.



Fig. 1. Distribution of copper in compost and its derivatives before treatment

Zinc (Zn) was highly bound organically than the other fractions (Fig. 2) and this was more evident in Co and Eco compared C and Ec. The concentration of Zn bound to organic fraction was 38.85 mg kg<sup>-1</sup> for C, 48.43 mg kg<sup>-1</sup> for Ec, 205.0 mg kg<sup>-1</sup> for Co and 168.1 mg kg<sup>-1</sup> for Eco. The other fractions that followed were carbonate bound > sulphide bound > exchangeable > adsorbed > residual. The water soluble fraction was below detection. The distribution in the soil followed the order: organically bound > carbonate bound > sulphide bound > adsorbed > residual > exchangeable but the water soluble fraction was below detection. The higher species of Zn in the organically bound fraction may be attributed to the ability of Zn to be converted into organic combinations during composting (Baker and Bryson, 2002). Ciba et al. (1998) and Hodgson et al. (1965) reported that Zn occurs mainly in the organically bound fraction. Though Qiao and Ho (1996) reported zinc to be evenly distributed in similar study, the composts Eco and Co had higher organically bound Zn than other composts C and Ec due to their higher organic matter content (Table 3) attributed to the dewatered faecal sludge used in the preparations.



Fig. 2. Distribution of zinc in compost and its derivatives before treatment

Lead (Pb) was dominant in the organically bound fraction with C - 31.04 mg kg<sup>-1</sup>, Co - 25.60 mg kg<sup>-1</sup>, Ec - 27.84 mg kg<sup>-1</sup>, Eco  $-28.133 \text{ mg kg}^{-1}$  but the graphical percentage representation showed the organically bound fraction in the same range Fig. 3. The rest of the order was residual > exchangeable > carbonate bound > adsorbed. The percentages of Pb from the exchangeable and adsorbed fractions were less than 15 %. Lead in the soluble and the sulphide bound fractions for each treatment was below detection. In the soil Pb was highly bound to residual fraction instead of the organic fraction. Lead was dominant in organically bound fraction in the compost and its derivatives as well compared with the other fractions. Pb a transition metal forms Pb<sup>2+</sup> which has the ability to combine with ligands and form organic combinations (Baker and Bryson, 2002; Qiao and Ho, 1996). However, within compost and its derivatives C and Eco showed a higher organically bound Pb than Co and Ec although Co and Eco have a higher organic matter than C and Ec (Table 3).



Fig.3. Distribution of lead in compost and its derivatives before treatment

Cadmium (Cd) species were dominant in the organically bound fraction (Fig. 4) with a percentage of between 71.8 to 88.2% in the order Co > C > Eco > Ec. The sulphide bound fraction constitutes 11.8 to 28.1% of the 'pseudo total' with other fractions below detection. Cadmium forms complexes with halogen ligands which have more readily deformable electron sheath and higher polarisability Qiao and Ho (1996). This results in the presence of Cd in halogen substrates in the organic matter.



Fig.4. Distribution of cadmium in compost and its derivatives before treatment

#### Speciation of heavy metals in compost amended soils

The amended soils for first and second cropping showed trends observed in the compost samples. However, in the amended soils, the soluble and carbonate bound Cu fractions were below detection limit.

The species formation of Cu in the amended soils for first and second cropping occurred mainly in the order: organically bound > exchangeable > bound to sulphide > residual >adsorbed (Fig. 5 and 6).

Copper remains highly bound organically in the amended soils after first and second cropping. The change in pH favoured acidity more than basicity and was expected to promote mobility leading to uptake and redistribution.

Copper ion can directly bind to two or more organic functional groups i.e. carboxylic, carbonyl and phenolic since Cu has stronger affinity for organic matter among the transition metals. This makes Cu immobilized in a rigid inner-sphere complex (McBride, 1989). The high stability of Cu complex with organic matter (Cu > Pb > Ni > Zn) made it impossible for its mobility and redistribution under lower pH conditions. Soluble and carbonate bound Cu might have been taken up by maize or the latter leached.



Fig. 5. Distribution of copper in compost and its derivatives amended soils for first cropping





The percentage distribution of zinc in the amended soils were in the order organically bound > sulphide bound > bound to carbonate > adsorbed > residual > exchangeable for first and second cropping as shown in Figure 7 and 8. The soluble fractions were mostly below detection limit.

The stability of  $Zn^{2+}$  ion is however, weak especially under low pH of 5 (Schnitzer and Skinner, 1966, 1967; McBride, 1994). Under acidic and oxidizing condition  $Zn^{2+}$  ions are highly soluble and mobile among the trace metal cations (McBride, 1994) and this could account for the release of the Zn ions from the organically bound fraction.



Fig. 7. Distribution of zinc in compost and its derivatives amended soils for first cropping



Fig. 8. Distribution of zinc in compost and its derivatives amended soils for second cropping

In the amended soils for first and second cropping (Fig.9 and 10) there was a major shift in lead distribution compared to the distribution in compost and its derivatives. The Pb species were detected mostly in the residual fractions in contrast to what was observed in the compost and its derivatives. The exchangeable fraction and the organically bound fraction had almost the same percentage distribution although the latter was higher in all the treatments. The soluble and the sulphide bound fractions were below detection limit. The complexation of Pb with organic matter, chemisorptions on oxides and clays, and precipitation as carbonates, hydroxide or phosphate are all favoured at higher pH (McBride, 1994). Lead has a higher affinity to the binding sites of clay (Hofstede, 1994; Alloway and Ayres, 1997) and this was evident in the amended soils where majority of Pb shifted to the residual fraction.



Fig. 9. Distribution of lead in compost and its derivatives amended soils for first cropping



Fig. 10. Distribution of lead in compost and its derivatives amended soils for second cropping

#### Conclusion

The study reveals that Copper and zinc were mostly bounded in the organic fraction of the compost and its derivatives in the order Co > Eco > C > Ec. Lead was also dominant in the organic bound fractions of composts and its derivatives in the order C > Eco > Ec > Co. Cadmium was observed in only two fractions, organic bound and sulphide bound with the former fraction dominating. The order of Cd in the composts and its derivatives were Co > C > Eco > Ec. The addition of dewatered faecal sludge to the compost in the preparation of Co and Eco resulted in higher organic matter in which promoted a higher organic bound Cu and Zn though the same order could not be inferred in the case of Pb and Cd.

For the amended soils; Copper was highly bound to organic fraction same as in the compost and its derivatives. Zinc was evenly distributed in the amended soils because is a soluble metal and its stability complexes with organic matter is weak. Higher Pb concentrations were found in the residual fraction than was observed for the organically bound fraction attributed to the high affinity of Pb for clay materials. The behaviour of heavy metals in the soils depends on individual metal and other characteristics. The complexation of metals with organic matter does not depend solely on the content of organic matter but other factors such as the characteristics of the metals and the concentration as well.

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 Table 1: Composition of the various compost materials

Materials	Designation	Composition
Compost	С	Municipal solid waste
Co-compost	Со	Municipal solid waste + Dewatered faecal sludge
Enriched compost	Ec	Municipal solid waste + ammonium sulphate
Enriched co-compost + ammonium sulphate	Eco	Municipal solid waste + Dewatered faecal sludge

Table 2. Description of the procedure used in speciation					
Metal form	Treatments				
	Reagent	pН	Volume of reagent	Time	
Soluble form	de-ionized water		20 ml	16 hrs	
Exchangeable form	0.1 KNO3		20 ml	16 hrs	
Adsorbed form	0.5 M KF	6.5	32 ml	16 hrs	
Organically bound form	0.1 M Na4P2O7		32 ml	16 hrs	
Bound to Carbonate	0.1 MEDTA	6.5	32 ml	16 hrs	
Bound to Sulphide	6.0 HNO3		20 ml	16 hrs	
Residual fraction	1:1 (HNO3:HCO4)		10 ml		

Table 2: Description of the procedure used in speciation

Parameters	Units					
T unumotors	Ching	С	Co	Ec	Eco	Soil
pН		8.0	7.8	7.4	7.6	5.1
EC (x10)	mS/cm	0.61	0.43	31.8	25.6	0.20
Total P	%	1.80	1.60	0.42	1.14	0.02
Total N	%	1.22	1.35	2.92	3.1	0.09
Organic ma	tter %	14.66	17.64	11.0	16.83	0.80
Texture:						
Sand	%	-	-	-	-	60.15
Silt	%	-	-	-	-	12.35
Clay	%	-	-	-	-	27.5
Heavy metals:						
Copper	mg kg <sup>-1</sup>	30.62	78.71	24.50	69.54	15.58
Zinc	mg kg <sup>-1</sup>	130.62	410.21	119.21	311.25	25.71
Lead	mg kg <sup>-1</sup>	71.62	69.25	69.583	68.00	53.542
Cadmium	mg kg⁻¹	0.1125	0.0875	0.0625	0.00721	****
**** below	detection					
- not app	plicable					

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Table 3: Phy	sco-chemical	characteristics of	compost	samples	and soi	l used	in experi	ment
Parameters	Units							

EC electrical conductivity