

## **Extraction of heavy metals from compost using a mixture of Na<sub>2</sub>EDTA and Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub>: column studies**

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### **Abstract**

This study reports on a method to extract heavy metals from compost. Compost samples were initially characterized to determine the type and content of heavy metal compounds. Column studies were carried out by solid liquid extraction method using 0.01 M Na<sub>2</sub>EDTA-0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution. Target heavy metal removal efficiencies were achieved for Cu (72%), Zn (77%), Pb (47%), Cd (86%), but not for Ni (12%) after 160 minutes contact time at a solid:liquid ratio of 1:15 g mL and 360 mL solution volume.

**Keywords:** Compost, Heavy metal, Na<sub>2</sub>EDTA, Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub>, Extraction.

## 1. Introduction

Compost can be used as a soil conditioner, as it contains major plant nutrients (N, P, and K), plant micronutrients (Cu, Fe, and Zn), and organic matter, which improve physical properties to achieve better soil aeration and water holding capacity (Zorpas *et al.*, 2002; Zhao *et al.*, 2011; Ingelmo *et al.*, 2012). Besides these positive characteristics, composts can include some inorganic and organic pollutants. Presence of heavy metal content is the main factor restricting the agricul-

tural use of compost (Pinamonti *et al.*, 1997; Ciba *et al.*, 2003; Núñez *et al.*, 2007; Akram Qazi *et al.*, 2009; Zhao *et al.*, 2011). The effect of compost use on heavy metal levels in the environment varies according to soil type, plant species and compost quality (Zorpas *et al.*, 2000; Ciba *et al.*, 2003; Núñez *et al.*, 2007; Zhao *et al.*, 2011). Table 1 lists the maximum permissible limits stipulated by various countries for the heavy metal content of composts.

**Table 1.** Maximum permissible limits stipulated by various countries for the heavy metal content of composts (mg kg<sup>-1</sup> dry compost) (ECN, 2008).

Heavy metal	Germany	Belgium	Canada	France	Sweden
Cu	150	100	100	-	150
Zn	400	1000	500	-	500
Mo	-	-	5	-	5
Co	-	-	34	-	25
Cd	2	5	3	8	3
Ni	50	50	62	200	50
Pb	200	600	150	800	150
Hg	1	-	0.8	8	3
Cr	150	150	210	-	150
As	-	-	13	-	-
Se	-	-	2	-	-

A review of the literature on the reduction of the heavy metal content of composts revealed that “capturing of heavy metals by the use of zeolites which are added to the environment during the composting process” or “removal of heavy metals from bio-waste via physical and chemical processes before the composting process” and similar methods can be used to this end (Zorpas *et al.*, 2000; Zorpas *et al.*, 2002; Veeken and Hamelers, 2003; Casado-Vela *et al.*, 2007). In soil washing techniques, where soil-bound contaminants

are transferred to the liquid phase by desorption and solubilization, acid washing and chelator soil washing are the two most prevalent removal methods (Salomons *et al.*, 1995; Peters, 1999; Kocalkowski *et al.*, 1999; Chaiyaraksa and Sriwiriyanuphap 2004; Kuo *et al.*, 2006; Pocięcha and Lestan, 2009; Zhang *et al.*, 2010; Arwidsson *et al.*, 2010). Using a mixture of Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> and Na<sub>2</sub>EDTA provides more economically optimum solution than using Na<sub>2</sub>EDTA for heavy metal removal (Peters, 1999; Chaiyaraksa and Sriwiriyan-

uphap 2004; Abumaizar and Khan, 1996; Abumaizar and Smith, 1999; Sun *et al.*, 2001).

In this study, the solid: liquid extraction method was investigated for removal of heavy metals from compost. This method is well established in the removal of heavy metals from solid matters such as soil, sediment and fly ash. This study presents the results of column studies on the removal of heavy metals from municipal solid waste (MSW) compost using a mixture of  $\text{Na}_2\text{S}_2\text{O}_5$  (reducing reagent) and  $\text{Na}_2\text{EDTA}$  (chelating reagent).

## 2. Materials and Methods

Compost samples from Istanbul Solid Waste Recycling and Composting Plant in Kismirandra, Istanbul, were used in this study. Characterization of the compost samples used in the experiments is presented in Table 2.

**Table 2.** Characterization of the compost samples used in the present study.

Parameter	Value
pH	8
Moisture content (%)	17
Bulk density (g 100 mL <sup>-1</sup> )	33.530
C (%)	24.54
H (%)	1.93
N (%)	7.84
S (%)	-
C/N	9.5
TKN (%)	1.34
Total P (mg kg <sup>-1</sup> dry compost)	0.007
Ca (mg kg <sup>-1</sup> dry compost)	0.088
Na (mg kg <sup>-1</sup> dry compost)	0.008
Mg (mg kg <sup>-1</sup> dry compost)	0.007
K (mg kg <sup>-1</sup> dry compost)	0.012
TOC (mg kg <sup>-1</sup> dry compost)	197012

Continued...

Parameter	Value
Cu (mg kg <sup>-1</sup> dry compost)	440
Ni (mg kg <sup>-1</sup> dry compost)	83.22
Zn (mg kg <sup>-1</sup> dry compost)	907.5
Pb (mg kg <sup>-1</sup> dry compost)	151.8
Cd (mg kg <sup>-1</sup> dry compost)	0.695

Distilled water was added to the compost samples at a 2:5 ratio (e.g. 4 g sample/10 mL distilled water) to determine the pH values of the samples. After being mixed by a magnetic mixer for 10 minutes, pH values were read using a pH meter (Jenway 3040 Ion Analyzer) (Methodenbuch zur Analyse von Kompost 1994).

A 1-g compost sample was dried in a drying oven at 105° Celsius for 24 hours. The compost samples were weighed before and after the drying process in order to calculate the moisture as shown in Eq. (1). (Methodenbuch zur Analyse von Kompost 1994):

$$\text{Moisture \%} = (M_1 - M_2)100 / M_1 \quad (1)$$

where  $M_1$  refers to compost weight before drying (gram) and  $M_2$  refers to compost weight after drying (gram).

To determine the amount of heavy metals (Cu, Ni, Zn, Pb, Zn, Cd) and other elements (Ca, Na, Mg, K, P) in the compost samples, a screened, fresh compost sample was dried at 105 °C and ground until reaching a grain size of 0.25 mm. Then, a compost sample weighing 1–2 g was placed in an Erlenmeyer flask and 10 mL 1:1 (volume/volume) water and nitric acid was added to perform the decomposition process. Following completion of the decomposition process, the sample was cooled; 5 mL hydrogen peroxide, 5 mL concentrated hydrochloric acid and 5 mL water were added; and the sample was re-heated. After cooling, the sample was diluted with distilled water and filtered using a 0.45 µm membrane filter. The resulting solution was analyzed to determine the presence and concentration of various elements (Methodenbuch zur Analyse von Kompost, 1994; AWWA, 1995). Samples

were analyzed using at the Advanced Analysis Laboratory (IAL) of Istanbul University using an ICP-MS (Inductively Coupled Plasma-Mass Spectrometer, Thermo-Elemental ICP-MS-X Series).

The percentage (%) of C, H, N and S in the compost samples was determined in the Advanced Analysis Laboratory (IAL) of Istanbul University. Samples were analyzed using Thermo Finnigan Flash EA 1112 Series model equipment.

The Total Organic Carbon (TOC) contents of the compost samples were determined using a TOC analyzer (Shimadzu, TOC-V CSH) at the Chemistry and Environment Institute of Marmara Research Centre (MAM), affiliated to TÜBİTAK (The Scientific and Technological Research Council of Turkey).

Values of orthophosphate and TKN (total Kjeldahl nitrogen) of compost samples were determined in the Analysis Laboratory of the Faculty of Environmental Engineering, Middle East Technical University. Orthophosphate amounts were determined spectrometrically, using the SM 4500-P E:2005 method. TKN values were determined with SM 4500-N<sub>org</sub> B:2005 (Macro-Kjeldahl) method (AWWA, 1995).

To determine the types of heavy metals present in the compost, samples were analyzed using an XRD (X-Ray Powder Diffraction) spectrometer in the Detailed Analysis Laboratory of Istanbul University. Prior to analysis, samples were dried at 50°C for 24 hours and were then ground to a fine powder in a quartz mortar.

A glass column of 4.5 cm inner diameter and 45 cm height was used in the experiments. A fiberglass filter of 0.45 µm was located at the bottom of the column. Glass beads of 5 mm diameter were placed as a 4-cm-thick layer on top of the compost, to ensure homogeneous distribution of the liquid throughout the column. Moreover, glass beads of 5 mm diameter were placed as a 3-cm-thick layer under the compost layer to prevent blocking of the fiberglass filter due to contact with the compost. It was ensured that an amount of liquid was present above the column to ensure uniform distribution of the extraction solution. Solutions were fed into the top of the column (equipped with a tap at the bottom) via a Watson Marlow 505 DI peristaltic pump.

A 200-mL (67.06 g) sample of compost was poured into the column and 400 mL of 0.01 M Na<sub>2</sub>EDTA - 0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution (ensuring 1:6 g mL<sup>-1</sup> solid-liquid ratio) was passed through the column over a 3-hour contact time (at 1.1 mL min<sup>-1</sup> flowrate); a 5-hour contact time (at 0.66 mL min<sup>-1</sup> flowrate); and a 6-hour contact time (at 0.55 mL min<sup>-1</sup> flowrate) (Table 3). In addition, column studies were also made at 3-hours contact time (0.55 mL min<sup>-1</sup> flowrate) and 1:10 solid-liquid ratio (by using 33.53 g compost and 330 mL 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution); 1:12.5 solid-liquid ratio (by using 33.53 g compost and 420 mL 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution); and 1:15 solid-liquid ratio (by using 28 g compost and 400 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution) (Table 3).

**Table 3.** Conditions of column studies.

Solid:Liquid Ratio (g mL <sup>-1</sup> )	Contact Time (hours)	Flowrate (mL min <sup>-1</sup> )
1:6	3	1.1
1:6	5	0.55
1:10	3	0.55
1:12.5	3	0.55
1:15	3	0.55

The system monitoring produced drawing capacity curves with 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution for 3-hours contact time, 1:15 g mL<sup>-1</sup> solid-liquid ratio, 0.55 mL min<sup>-1</sup> flowrate and 28 g compost and 400 mL 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution. Drawing capacity curves were developed from samples periodically obtained from the column output. The pH of the extraction solution was measured before contact with compost and after leaving the column. Samples collected at the outflow of the column were filtered using a 0.45 µm membrane filter. After filtration, the pH of the samples was adjusted to ≤2.0 by adding 1:1 HNO<sub>3</sub> to the filtrates in order to determine the heavy metals.

### 3. Results and Discussion

The heavy metal contents (Cu, Zn, Pb, etc.) of the compost samples used in the present study were compared with maximum permissible limits given in German compost standards, which are more stringent than those of other countries (Table 1). It is seen that the Cu and Zn levels of the experimental samples would need to be reduced by 66% and 56% respectively. It is also observed that the Pb and Cd contents were within the maximum permissible levels (Pb <200 mg kg<sup>-1</sup>, Cd < 2 mg kg<sup>-1</sup>).

Zorpas *et al.*, (2000) found that the exchangeable fraction was affected by changes in the ionic composition of the water; that the carbonate fraction of the

compost was affected by changes in pH value; that the reducible part was composed of iron and manganese oxides (thermodynamically unstable under anoxic conditions); that the organic part released metals dissolving under oxidation conditions; that the residue part was generally composed of primary and secondary minerals; and that metals were attached to the crystallite structure of these minerals. A study by Peters (1999) reported that metal compounds present in samples should be in the form of exchangeable, carbonate or reducible oxides in order to ensure removal of heavy metals from solid environments via extraction methods.

XRD analysis showed that CdPbO<sub>3</sub>, Cu<sub>2</sub>O (Cuprit), SiO<sub>2</sub> (Quartz), ZnO, NiCO<sub>3</sub> (gaspeite), and NiO heavy metal compounds were present in the compost samples. From the XRD results of the compost samples used in the present study, it was concluded that heavy metals were in the form of reducible oxides and carbonates, and that they were appropriate samples for the extraction method used.

In our previous study, batch experiments showed that the highest efficiency of heavy metal removal from the compost was obtained at 3 hours contact time and 1:6 g mL solid:liquid ratio (Ozbas *et al.*, 2007). However, that work did not include column studies. In the present study, the column studies were conducted under the conditions determined with the batch studies. Under these conditions, the required removal efficiencies could not be achieved in column

studies. Therefore, the solid:liquid ratio was decreased and the column studies were repeated using 1:10, 1:12.5 and 1:15 g mL<sup>-1</sup> solid:liquid ratios (0.55 mL minute<sup>-1</sup> flowrate). The pH values of the solutions, before and after the extraction were measured as 5 and 7 respectively. The results of the column

experiment conducted with 0.01 M Na<sub>2</sub>EDTA-0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution are listed in Table 4. It was found that 3 hours contact time (0.55 mL min<sup>-1</sup> flowrate) and 1:12.5 g mL<sup>-1</sup> solid-liquid ratio provided appropriate conditions for the removal of heavy metals using the column method.

**Table 4.** Results of column experiments conducted with 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub>.

Coloumn conditions	Heavy metal concentrations removed from compost (mg/kg-dry compost)					Heavy metal removal (%)				
	Cu	Ni	Zn	Pb	Cd	Cu	Ni	Zn	Pb	Cd
t=3 h , Q=1.1 mL minute <sup>-1</sup> ), 1:6g mL <sup>-1</sup> solid:liquid ratio	105.6	2.58	430.46	52.37	0.149	24	3.1	47.46	34.5	21.48
t=5 h , Q=0.66 mL minute <sup>-1</sup> ), 1:6 g mL <sup>-1</sup> solid:liquid ratio	126.94	3.41	515.09	55.68	0.161	28.85	4.1	56.79	36.68	23.17
t=6 h , Q=0.55 mL minute <sup>-1</sup> ), 1:6 g mL <sup>-1</sup> solid:liquid ratio	128.83	4.49	587.83	69.65	0.183	29.28	5.4	64.81	45.88	26.32
t=3 h , Q=0.55 mL minute <sup>-1</sup> ), 1:10 g mL <sup>-1</sup> solid:liquid ratio	119.7	5.58	430.95	53.67	0.680	27.2	6.7	47.51	35.35	97.8
t=3 h , Q=0.55 mL minute <sup>-1</sup> ), 1:12.5 g mL <sup>-1</sup> solid:liquid ratio	210.32	6.99	590.47	65.6	0.682	47.8	8.4	65.1	43.44	98.1
t=3 h , Q=0.55 mL minute <sup>-1</sup> ), 1:15 g mL <sup>-1</sup> solid:liquid ratio	306.68	9.15	610.35	74.74	0.690	69.7	11	67.3	49.5	99.3

Studies were conducted at 1:15 g mL<sup>-1</sup> solid-liquid ratio at 0.55 mL minute<sup>-1</sup> flow rate (using 28 g compost, 400 mL 0.01 M Na<sub>2</sub>EDTA-0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution, 3 hours contact time) to produce capacity curves. During the study, a 40-mL sample was collected from each column. Figure 1 gives the capacity curves obtained from the column for 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution.

Examining the capacity curves, it can be understood that 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution

initially removed Cd and Cu, and then Zn and Pb. It was found that the 66% target removal efficiency for Cu could be exceeded (71.91%) using 360 mL solution (at 160 minutes contact time) (Figure 1). A removal efficiency of 60% was achieved for Zn (target 56%) using 200 mL solution (at 90 minutes contact time). The highest removal efficiency for Cd ( $\approx$  100%) was obtained at 240 mL solution (at 108 minutes contact time). The targeted removal efficiency for Ni was not achieved (Figure 1). Examining the change in the pH

values during the column studies, it was concluded that pH value (pH 4 prior to extraction) increased as more solution was passed through the column. When 240 mL solution was passed through the column (at 108 minutes contact time), the pH reached 6 (Figure 1).

The capacity experiments conducted with 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution indicated that when 360 mL solution was passed through the column (160 minutes contact time at 1:15 g mL<sup>-1</sup> solid-liquid ratio), the achieved removal efficiencies

(72% Cu, 77% Zn, 47% Pb and 86% Cd) exceeded the targeted levels for Cu, Zn, Pb and Cd (Figure 1).

In column experiments, 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution at 1:15 g mL<sup>-1</sup> solid-liquid ratios and 0.55 mL min<sup>-1</sup> flowrate (28 g compost, 400 ml molasses hydrolysate, 3 hour contact time) was used. Capacity curves were produced, which indicated that 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution initially captured Cd and Cu and then was effective on Zn and Pb. The maximum removal efficiency values achieved were 91% (Cu), 12% (Ni), 85% (Zn), 51% (Pb) and 100% (Cd).

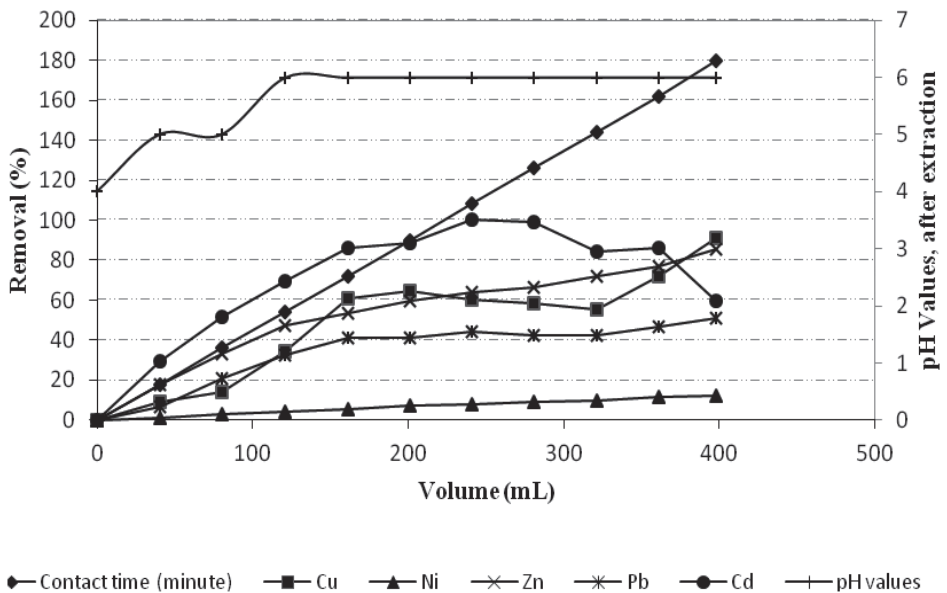


Figure 1. Capacity curves obtained by using 0.01 M Na<sub>2</sub>EDTA -0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution.

In the experiments conducted with 0.01 M Na<sub>2</sub>EDTA-0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution, it was found that the pH value of the solution was approximately 7 after the extraction. Previous studies reported that pH is an important factor in extraction (Apak, 1997; Abumaizar and Smith, 1999; Peters, 1999; Chaiyaraksa and Sriwiriyanuphap, 2004); and that in cases of high pH in solutions, metal ions will turn into hydroxide form, and that Na<sub>2</sub>EDTA will not be able to extract hydroxide forms (Peters, 1999; Chaiyaraksa and Sriwiriyanuphap, 2004). Therefore, the high pH of the media (approximately 6) in the present study is suggested as the reason for the low removal of Ni using 0.01 M Na<sub>2</sub>EDTA-0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution.

In cases where Na<sub>2</sub>EDTA solution is used alone or Na<sub>2</sub>EDTA and Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> are used in combination, Na<sub>2</sub>EDTA can show different reactions to the metals in the medium. While Na<sub>2</sub>EDTA is able to easily extract some metals according to the condition in the medium, it performs very poorly in extracting some others (Kocalkowski *et al.*, 1999; Abumaizar and Khan 1996; Sun *et al.*, 2001).

In studies that analyzed the adsorption of metal mixtures in wastewater, it was reported that adsorption capacities of metal ions in the same elemental group were quite similar; however, whereas those of metal ions in different classes were different (Tüzün *et al.*, 2005). Cu belongs to IB, Ni VIIIIB, Zn and Cd IIB, Pb IVA group. In the present study, in continuous experiments conducted with solutions containing Na<sub>2</sub>EDTA and Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub>, removal yields of 72%, 11%, 77%, 47% and 86% were obtained for Cu, Ni, Zn, Pb and Cd, respectively. It is suggested that these differing extraction ratios are because the target metals are in different groups. The results suggest that when a solution containing 0.01 M Na<sub>2</sub>EDTA-0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> was used, among the metals in the compost, Cu, Zn, Pb and Cd were extracted first, and that

Ni was extracted later. However, in this case, it is thought that, as the pH of the solution increases and becomes close to 6, the expected Ni removal could not be achieved, because pH must be lower than 5.5 for Ni mobility.

### 3.1 Cost Estimation

It was found that 1:12.5 g mL<sup>-1</sup> (compost/0.01 Na<sub>2</sub>EDTA-0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution) solid:liquid ratio provided appropriate conditions for the removal of heavy metals using the column method. In Turkey, the representative costs of Na<sub>2</sub>EDTA and Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> are US \$46 and \$0.45 per kg, respectively. In this case, the cost of treating compost using Na<sub>2</sub>EDTA and Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> is estimated at approximately US \$2700 per ton.

## 4. Conclusion

In the experiments conducted with 0.01 M Na<sub>2</sub>EDTA-0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution, it was observed that targeted removal efficiencies were achieved for Cu, Zn, Pb and Cd but not for Ni. Cost estimates show that the use of 0.01 M Na<sub>2</sub>EDTA-0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub> solution is not economical for the extraction of metals from compost. Future studies are planned to provide further post-treatment characterization in order to determine the quality of the compost material.

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