

Fractionation of Cadmium in a Calcareous Sandy Loam Soil Amended with Almond Soft Husk Compost and Biochar

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ABSTRACT

Reducing the mobility of heavy metals in soils is one of the best soil remediation methods to decrease the mobile fraction of the metals. This study was conducted to evaluate the degree of mobility and fractionation of cadmium (Cd) after the addition of almond soft husk compost and biochar in a sandy loam calcareous soil. The efficacy of amendments was evaluated using sequential extraction. This research was factorial and based on a completely randomised design with three levels of cadmium nitrate (0, 40 and 80 mg kg⁻¹ soil) and three levels (0, 2 and 4 wt %) of almond soft husk compost and biochar application rate. It was conducted in three replications. The application of biochar at 4 wt % to soil spiked with 40 mg kg⁻¹ cadmium reduced the exchangeable Cd by 66% and the carbonate-Cd by 23% during the 45 days after incubation compared to the control treatment. Meanwhile, the application of biochar at 4 wt % to soil spiked with 80 mg kg⁻¹ cadmium reduced the exchangeable Cd by 62% and the carbonate-Cd by 17% compared to the control treatment. The application of compost at 4wt% to soil spiked with 40 mg kg⁻¹ cadmium reduced the exchangeable Cd by 39% and the carbonate-Cd by 16% compared to the control treatment, while the application of compost at 4 wt % to soil spiked with 80 mg kg⁻¹ cadmium reduced the exchangeable Cd by 47% and the carbonate-Cd by 13.5% compared to the control treatment. The application of biochar at 4 wt % to soil spiked with 40 mg kg⁻¹ cadmium reduced the exchangeable Cd by 40% and the carbonate-Cd by 7% compared to the compost, while the application of biochar at 4 wt % to soil spiked with 80 mg kg⁻¹ cadmium reduced the exchangeable Cd by 28% and the carbonate-Cd by 2% compared to the compost. The highest mobility factor in soils treated with different concentrations of cadmium was related to control treatment, which was calculated to be 57.4%.

Keywords: Calcareous soil, chemical forms, mobility, sequential extraction, soil pollution.

INTRODUCTION

Soil contamination has emerged as a major environmental problem in many countries in recent years. With increasing population, the request for food has increased enormously, resulting in the adaptation of agricultural practices that not only increase agricultural production but also result in pollution of heavy metals

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in soil (Fernandez-Ondono *et al.* 2017). Anthropogenic input can increase levels of heavy metals in agricultural soils and reduce the quality of agricultural products and crop yield, degrade soil quality and easily transfer the heavy metals from soil to groundwater. The presence of heavy metals in groundwater can result in long lasting toxic effects as they cannot be degraded (Tashakor *et al.* 2014). Cadmium (Cd) is one of the heavy metals that exists in contaminated soils, causing long-term danger to humans and the environments (Liu *et al.* 2013).

In the last years, much research has been done in order to remediate heavy metal polluted soils (Bolan *et al.* 2014). One such innovation is the use of pyrogenic forms of carbon called biochar. Biochar is a carbon-rich black solid produced by pyrolysis of carbon rich residues, manure or dead animals and is used as a soil amendment in agriculture and the environment (Guo *et al.* 2016; Ok *et al.* 2015; Yang *et al.* 2016). The properties of biochar produced from different feedstocks and pyrolysis conditions vary widely. Typically, most biochars have a large surface area, a micro-porous structure, active organic functional groups, high pH and its cost is low. These are very attractive features (Wu *et al.* 2012; Beesley *et al.* 2011). The primary mechanisms of metal immobilisation by biochar in soils include an increase in soil pH, precipitation and co-precipitation, physical sorption, and ion exchange (Park *et al.* 2011). The mobility of heavy metals in soil depends on the form of the heavy metals (Zhong *et al.* 2011; Sahito *et al.* 2015), which can be measured by sequential extraction (Kubová *et al.* 2008).

Sequential extraction methods can predict the reactivity and availability of different chemical forms of metals in soils and how these metals are transferred from the solid phase to the liquid phase (Altundag *et al.* 2016). Chemical forms of metals in soils include several geochemical forms such as water-soluble, exchangeable, carbonate-bound, Mn-oxide-bound, Fe-oxide-bound, organic-bound, and residue. Water-soluble and exchangeable fractions are considered as bioavailable, but the fractions associated with carbonates, oxides, and organic matter can be potentially bioavailable. However, the residual fraction is not available for plants (Rodríguez *et al.* 2009). Some researchers have suggested that organic matter (OM) could redistribute heavy metals from soluble and exchangeable forms to carbonate, Fe–Mn oxides, and OM-bonded fractions and temporarily reduce their availability to plants (Lee *et al.* 2009). Thus, the aim of the present study was to investigate fractionation of cadmium in a calcareous sandy loam soil amended with almond soft husk compost and biochar.

MATERIALS AND METHODS

Soil Collection and Characterisation

The soil sample was taken from the 0-30 cm layer of a field at the Faculty of Agriculture, Kerman University, Iran. The soil was air-dried and passed through a 2 mm mesh sieve and stored in polyethylene bags prior to use. Selected chemical and physical properties of the soil are shown in Table 1. Particle size was determined by the hydrometer method (Bouyoucos 1962); soil pH was

determined in the saturated extract of the soil using a combination electrode (Thomas 1996); electrical conductivity (EC) was measured in the soil saturated extract using a conductometer (Rhoades *et al.*, 1996); organic matter (OM) content was determined by dichromate oxidation (Nelson and Sommers 1996); CEC was obtained by saturation with 1M ammonium acetate (NH_4OAc) at pH 7 (Sumner *et al.*, 1996) calcium carbonate (CaCO_3) was determined by titration (Loeppert and Suarez 1996); available phosphorus (P) was determined according to the Olsen method (Olsen *et al.* 1954); total nitrogen by Bremner (1996) method; extractable potassium by ammonium acetate molar (Chapman and Pratt 1982) and Fe, Zn, Mn, Cu and Cd concentrations were extracted by DTPA and determined by atomic absorption spectrometry (AAS) (Lindsay and Norvell 1978). This research was factorial based on a completely randomized design with three levels of cadmium nitrate (0, 40 and 80 mg kg^{-1} soil) and three levels (0, 2 and 4 wt%) of either almond soft husk compost or almond soft husk biochar and conducted in triplicate.

TABLE 1
Selected chemical and physical properties of the soil studied

Property (Unit)	Value
Soil texture	Sandy loam
OM (%)	0.69
CCE (%)	8.32
Ec(dS m^{-1})	2.39
pH	7.50
CEC ($\text{cmol}(+) \text{ kg}^{-1}$)	8.93
N (%)	0.03
K (mg kg^{-1})	433
P (mg kg^{-1})	14
Mn (mg kg^{-1})	1.74
Cu (mg kg^{-1})	0.5
Fe (mg kg^{-1})	1.52
Zn (mg kg^{-1})	0.72
Cd (mg kg^{-1})	Insignificant

Biochar Production and Characterisation

The almond soft husk was collected from an orchard in Shiraz (Fars province). Windrow method was used for composting. The almond soft husk was put in a pit and covered with plastic and kept at temperatures of 37 to 60 °C and a moisture level of 50 to 60% for 4 months to prepare the compost. The almond soft husk biochar was air-dried and packed in aluminum sheets to limit oxygenation during the pyrolysis process. Then, the samples were placed in an oven at 500°C for 4 h to produce the biochar (Hall *et al.*, 2008). Almond soft husk was air dried, ground, passed through a 0.5-mm sieve, and analysed for some of its chemical characteristics (Table 2). To perform chemical analysis of the compost and biochar,

TABLE 2
Selected chemical properties of the compost and biochar tested

Property (Unit)	Almond soft husk	Almond soft husk compost	Almond soft husk biochar
pH	3.21	4.83	11.61
EC (dS m ⁻¹)	1.15	1.61	7.93
N (%)	0.34	0.55	0.68
P (%)	0.11	0.13	0.14
K (%)	0.32	0.42	0.63
Fe (mg kg ⁻¹)	68	90	140
Zn (mg kg ⁻¹)	8.37	12.24	28.06
Cu (mg kg ⁻¹)	3.18	4.08	8.03
Mn(mg kg ⁻¹)	5.83	8.67	13.27
Cd (mg kg ⁻¹)	Insignificant	Insignificant	Insignificant

1 g of compost and biochar almond soft husk was placed in an electric furnace for 7 h at 750°C to obtain their ashes. Then it was dissolved in HCl. pH was measured in 1:5 organic matter to water suspension; EC was determined in 1:5 organic matter to water suspension by a conductivity meter; and P was determined by the vanadomolybdate phosphate method in a nitric acid system (Chapman and Pratt 1961), total nitrogen (Bremner1996), and Fe, Zn, Mn, Cu and Cd concentrations were analysed by atomic adsorption spectrometry.

Incubation Study

The experiment was performed by adding 1 kg of soil into each pot, to which was added either almond soft husk compost or almond soft husk biochar and mixed completely. The pots were placed in a greenhouse at 25 ± 5 °C for 2 months. After the 2-month incubation, either 40 or 80 mg kg⁻¹cadmium was added to each pot and mixed completely; the pots were then placed in the glasshouse for another 3 months. The treatments of the incubation study are shown in Table 3.

A soil fractionation study was carried out at 45 and 90 days after incubation where different chemical forms of Cd was determined adopted from Singh *et al.*(1988) (Table 4).

TABLE 3
Experimental design for incubation experiment

treatment	pollution with cd	Level of biochar	Level of compost
Cd ₄₀ B ₀	40 mg kg ⁻¹	0 wt%	-----
Cd ₄₀ B ₂	40 mg kg ⁻¹	2 wt%	-----
Cd ₄₀ B ₄	40 mg kg ⁻¹	4 wt%	-----
Cd ₈₀ B ₀	80 mg kg ⁻¹	0 wt%	-----
Cd ₈₀ B ₂	80 mg kg ⁻¹	2 wt%	-----
Cd ₈₀ B ₄	80 mg kg ⁻¹	4 wt%	-----
Cd ₄₀ C ₀	40 mg kg ⁻¹	-----	0 wt%
Cd ₄₀ C ₂	40 mg kg ⁻¹	-----	2 wt%
Cd ₄₀ C ₄	40 mg kg ⁻¹	-----	4 wt%
Cd ₈₀ C ₀	80 mg kg ⁻¹	-----	0 wt%
Cd ₈₀ C ₂	80 mg kg ⁻¹	-----	2 wt%
Cd ₈₀ C ₄	80 mg kg ⁻¹	-----	4 wt%

TABLE 4
Procedure for the sequential extraction of Cd from soil and corresponding phases

Phase/association	Abbreviation	step	Operational definition
Exchangeable	EX	1	4mL 1M Mg(NO ₃) ₂ , shaking 2 h
Carbonated-bound	CAR	2	4mL 1M NaOAc, pH 5, shaking 5 h
Organic-bound	ORG	3	2mL 0.7M NaOCl, pH 8.5, stir occasionally 0.5 h in water bath (Repeat extraction)
Mn oxide-bound	MnOX	4	10mL 1M NH ₂ OH.HCl+HNO ₃ , pH 2, shaking 0.5 h
Amorph Fe oxide-bound	AFeOX	5	10mL 0.25M NH ₂ OH.HCl + 0.25M HCl, shaking 0.5 h at 50°C in water bath
Crystalline oxide-bound	CFeOX	6	10mL 0.2 M(NH ₄) ₂ C ₂ O ₄ + 0.2M H ₂ C ₂ O ₄ + 0.1M C ₆ H ₈ O ₅ , pH 3, stir occasionally 0.5 h in boiling water bath
Residual	RES	7	Conc. HF, conc. HClO ₄ and conc. HCl in sequence

The metal index of mobility(IM) was calculated based on equation (1), adapted from Kabala and Singh (2001):

$$IM(\%) = \frac{EX + CAR}{EX + CAR + ORG + MnOX + AFeOX + CFeOX + RES} \times 100 \quad (1)$$

where IM is the index of mobility; EX, CAR, ORG, MnOX, AFeOX, CFeOX and RES are the chemical fractions of Cd in the soil as explained in Table 3.

Statistical Analysis

All data were processed by Microsoft Excel 2010 and statistical analyses were conducted using the SPSS 11.5 software.

RESULTS AND DISCUSSION

The relative amounts of sequential extraction of cadmium in the treated samples and the control sample 45 and 90 days after incubation, indicate that the carbonate forms has the largest share of the chemical fractions of cadmium (Figures 1 and 2). Rajaie *et al.* (2008)(not in ref list)?? reported that in calcareous and alkaline soils of Iran, the carbonate form was the dominant soil Cd in soils treated with enriched compost. Khanmirzaei *et al.* (2013)(not in ref list)?? investigated the different form of Cd in the calcareous soils of Iran using a sequential extraction method and found the carbonate form to be the dominant form of cadmium in these soils. Application of amendments at various rate (2 and 4 wt %), reduce the exchangeable and carbonate forms compared to the control treatment. The exchangeable form can be introduced as a determinant factor in increasing environmental hazards caused by the leaching of heavy elements. Therefore, reducing the distribution of the exchangeable form could be an effective factor to reduce environmental pollution caused by the release of heavy metals.

The application of biochar at 4 wt % to soil spiked with 40 mg kg⁻¹ cadmium reduced the exchangeable Cd by 66% and the carbonate-Cd by 23% during the 45 days after incubation compared to the control treatment. While the application of biochar at 4wt% to soil spiked with 80 mg kg⁻¹ cadmium reduced the exchangeable Cd by 62% and the carbonate-Cd by 17% compared to the control treatment. Also, the application of compost at 4wt% to soil spiked with 40 mg kg⁻¹ cadmium

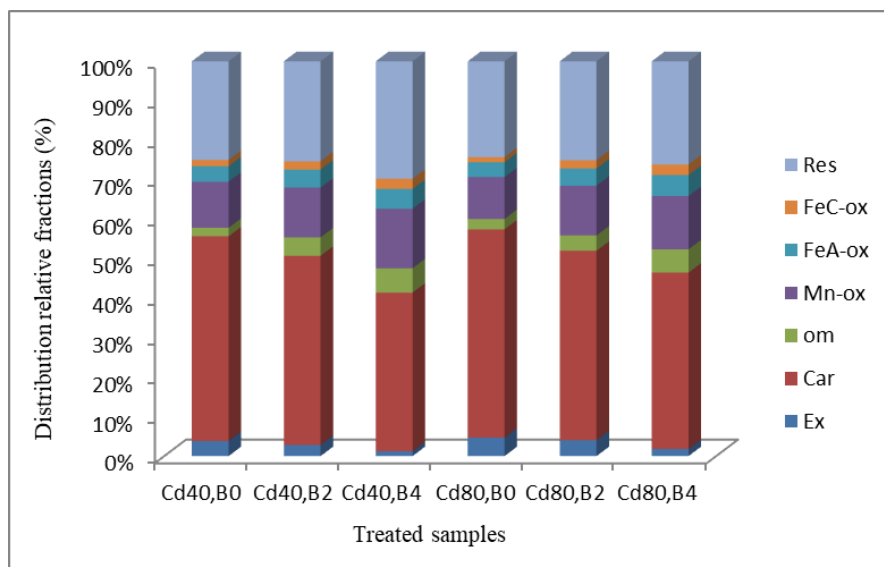


Figure 1: Distribution relative Cadmium fractions, 45 days after incubation in biochar-treated specimens

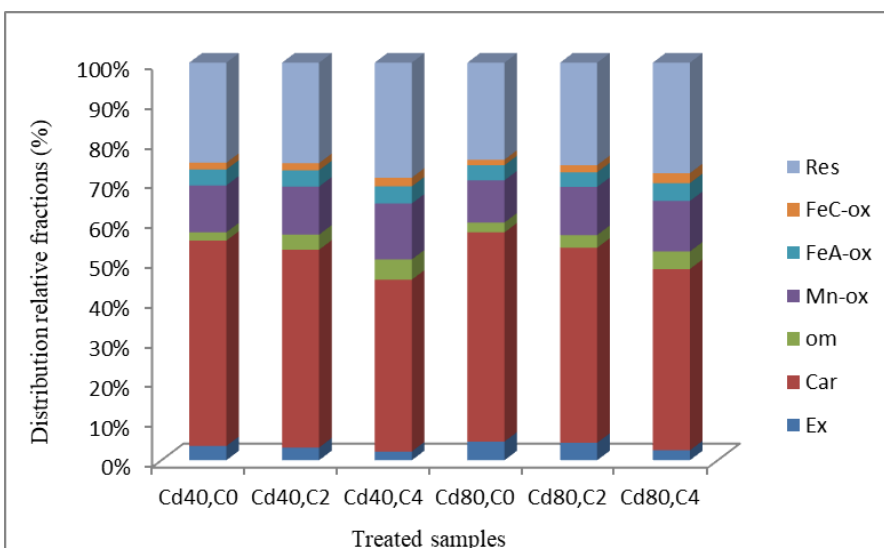


Figure 2: Distribution relative Cadmium fractions, 45 days after incubation in compost-treated specimens

reduced the exchangeable Cd by 39% and the carbonate-Cd by 16% compared to the control treatment, while the application of compost at 4 wt% to soil spiked with 80 mg kg⁻¹ cadmium reduced the exchangeable Cd by 47% and the carbonate-Cd by 13.5% compared to the control treatment. The application of biochar at 4wt%

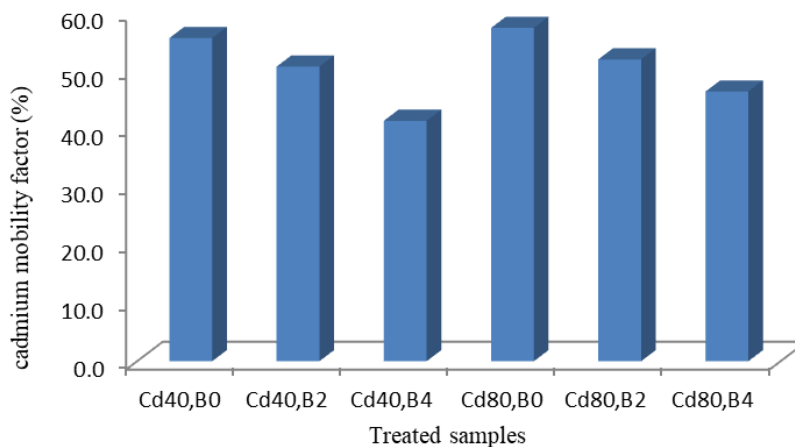


Figure 3: Cadmium mobility factor, 45 days after incubation in biochar-treated specimens

to soil spiked with 40 mg kg⁻¹ cadmium reduced the exchangeable Cd by 40% and the carbonate-Cd by 7% compared to the compost, while the application of biochar at 4 wt% to soil spiked with 80 mg kg⁻¹ cadmium reduced the exchangeable Cd by 28% and the carbonate-Cd by 2% compared to the compost.

Investigating the cadmium mobility factor in soil using sequential extraction of cadmium can complement the positive or negative effects of amendments adopted. The highest mobility factor in soils treated with different concentrations of cadmium in Figure 4 and 5 is related to control treatment, which is calculated to be 57.4%.

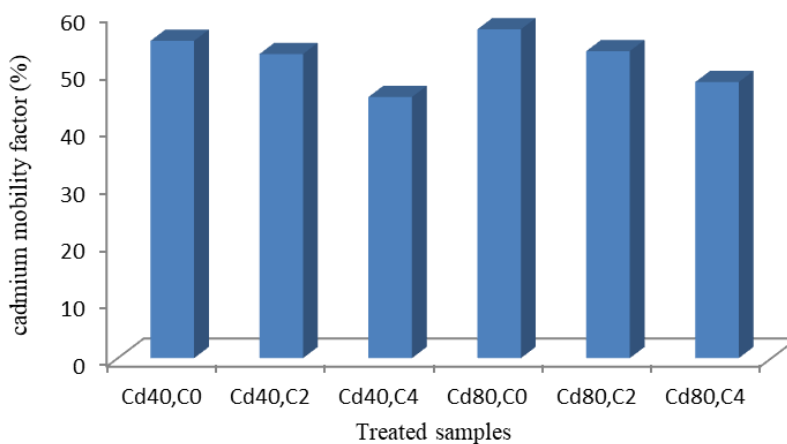


Figure 4: Cadmium mobility factor, 45 days after incubation in compost-treated specimens

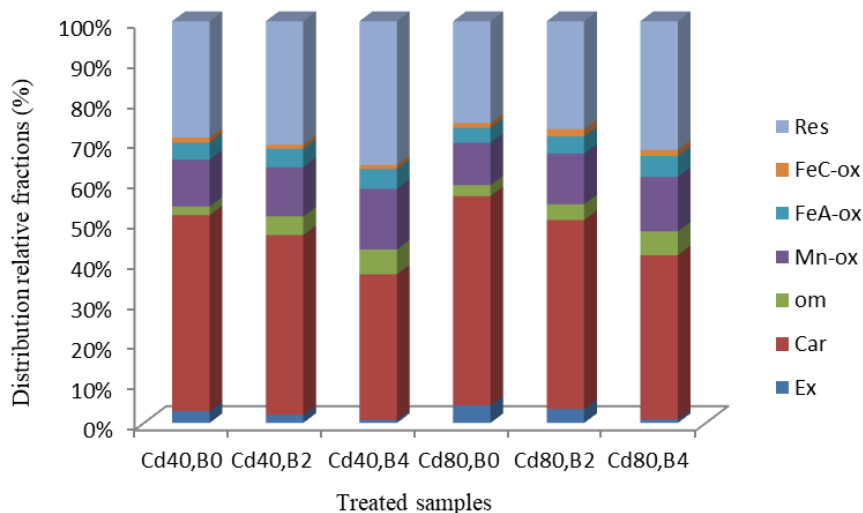


Figure 5: Distribution of relative cadmium fractions, 90 days after incubation in biochar-treated specimens

The application of biochar at 4 wt % to soil spiked with 40 mg kg⁻¹ cadmium reduced the exchangeable Cd by 66% and the carbonate-Cd by 23% during the 45 days after incubation compared to the control treatment. While the application of biochar at 4wt% to soil spiked with 80 mg kg⁻¹ cadmium reduced the exchangeable Cd by 62% and the carbonate-Cd by 17% compared to the control treatment. Also, the application of compost at 4wt% to soil spiked with 40 mg kg⁻¹ cadmium reduced the exchangeable Cd by 39% and the carbonate-Cd by 16% compared to the control treatment, while the application of compost at 4 wt% to soil spiked with 80 mg kg⁻¹ cadmium reduced the exchangeable Cd by 47% and the carbonate-Cd by 13.5% compared to the control treatment. The application of biochar at 4wt% to soil spiked with 40 mg kg⁻¹ cadmium reduced the exchangeable Cd by 40% and the carbonate-Cd by 7% compared to the compost, while the application of biochar at 4 wt% to soil spiked with 80 mg kg⁻¹ cadmium reduced the exchangeable Cd by 28% and the carbonate-Cd by 2% compared to the compost.

Investigating the cadmium mobility factor in soil using sequential extraction of cadmium can complement the positive or negative effects of amendments adopted. The highest mobility factor in soils treated with different concentrations of cadmium in Figure 4 and 5 is related to control treatment, which is calculated to be 57.4%.

Biochar application at 4 wt % to the soils spiked with Cd at 40 and 80 mg kg⁻¹ reduced the metal mobility factor by 41.4 and 46.5%, respectively. The relative amounts of fractions of cadmium by sequential extraction by Sing *et al*, (Year? Not in ref list?) with different treatments 90 days after incubation are shown in Figures 5 and 6. The results show a tangible change in the exchangeable and carbonate forms with an increase in the residual forms compared to the soil 45 days after incubation.

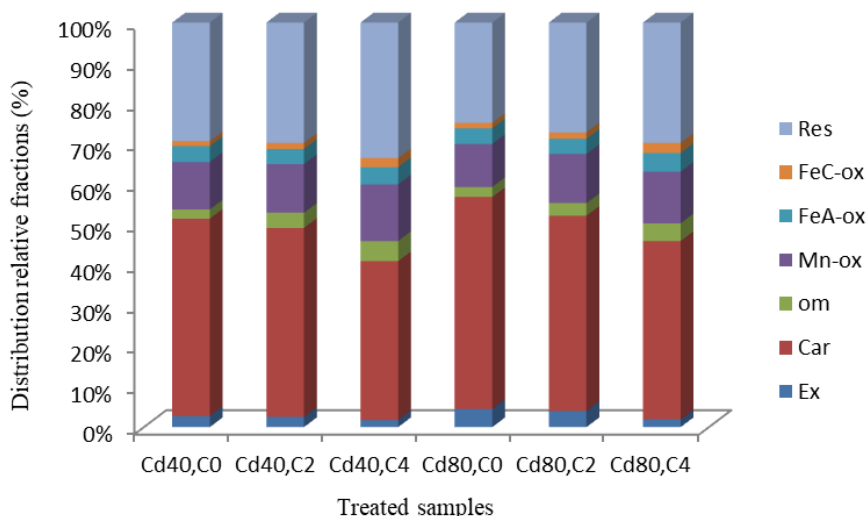


Figure 6: Distribution of relative cadmium fractions, 90 days after incubation in compost- treated specimens

The relative amounts of fractions of cadmium in soils treated 90 day after incubation show that in treated samples and control samples, they were similar to 45 day after incubation with the carbonate form having the largest share of the fractions of cadmium. The results obtained from 90-day incubation soils are similar to the results of 45-day incubation soils, with the difference being a decrease in exchangeable and carbonate forms and an increase in the residual form.

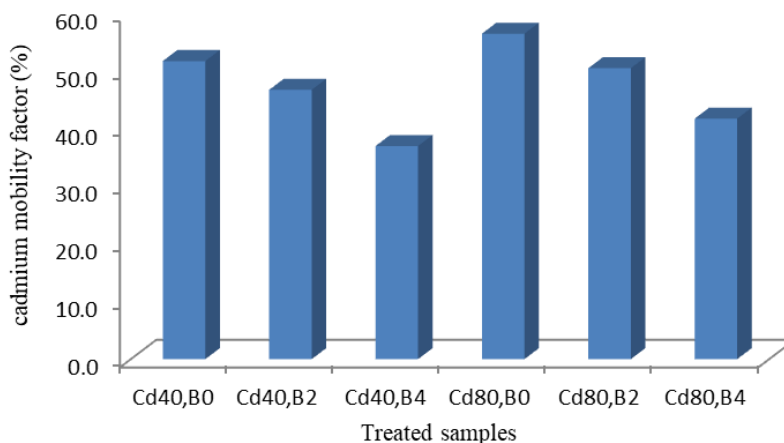


Figure 7: Cadmium mobility factor, 90 days after incubation in biochar-treated specimens

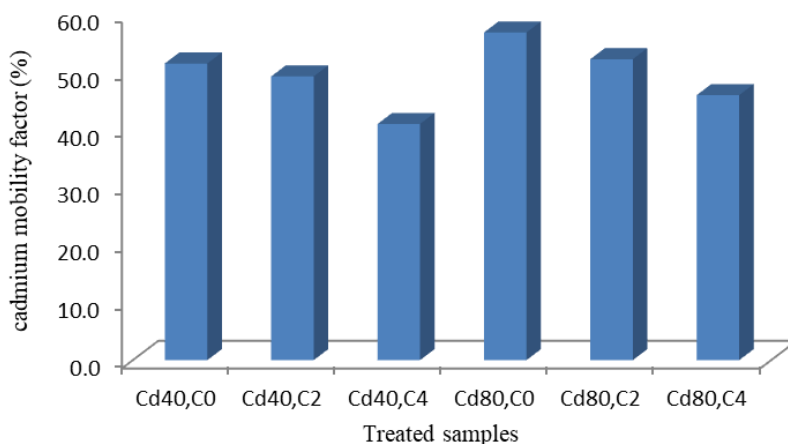


Figure 8: cadmium mobility factor; 90 days after incubation in compost-treated specimens

Generally, the values of chemical forms of Cd in the soil 45 and 90 days after incubation were as follows:

Carbonate-bound<residual<Mn-oxide-bound<exchangeable<Amorphous
Fe- oxide-bound<organic-bound<CrystallineFe- oxide-bound

CONCLUSIONS

The relative amounts of chemical forms of cadmium were influenced by the use of different amendments; cadmium carbonate forms were the most observed chemical forms of cadmium. But the crystalline Fe- oxide-bound had the least amount of chemical forms of cadmium. The use of the almond soft husk compost and biochar increased the formation of organic-bound and cadmium Mn-oxide-bound forms and reduced the forms of exchangeable and carbonate-bound. Soils treated with biochar and compost had the lowest and highest cadmium mobility factor in soil.

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