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# TRANSFORMATIONS OF SOIL Pb AND Cu AMONG THEIR CHEMICAL FORMS IN SOME CONTAMINATED EGYPTIAN SOILS TREATED WITH ORGANIC MATERIALS.*

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**Abstract:** An incubation experiment, using three contaminated Egyptian soils from Helwan, El-Gabal El-Asfar (Cairo governorate) and Arab El-Madabeg (Assiut governorate) was conducted for 16 weeks to evaluate effects of four organic materials including EDTA, poultry litter extract (PLE), vinase (V) and humic acid (HA) solutions on their transformations among various soil forms that contribute in the mobility increase of two heavy metals (Pb and Cu) in these soils. The design of this experiment was completely randomized with 3 replications. The studied chemical forms of metals were the soluble plus exchangeable (S+EXCH), carbonate bound (C-bound), Mn oxide bound (MnO), organically bound (O-bound), poorly crystalline Fe oxide bound (PCFeO), crystalline Fe oxide bound (CFeO) and residual forms.

Soil materials were put in plastic cups, irrigated every week with solutions of these organic materials at 6 mmol/kg for EDTA, 75g/L for PLE, pure vinase and 0.025% for HA as well as distilled water as a control treatment. All investigated organic materials significantly increased levels of the exchangeable form of Pb and Cu in all studied soils compared to the control treatment. The magnitude of these increases depended upon the soil type, metal content and the type of organic material. EDTA was the most effective organic material in increasing exchangeable levels of these metals in all studied soils followed by vinase and then PLE. On the other hand, humic acid was the least effective one in that matter in all studied soils. The low efficiency of PLE and HA in mobilizing the metals and inducing metal transformations may be attributed to the relatively short period used in these experiments as well as their relatively low application levels.

**Key words:** transformations, heavy metals, organic materials, contaminated soils.

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## Introduction

Heavy metals in the environment such as Pb and Cu have been taken considerable attention due to their potential influences on human and animal health. They tend to accumulate in soils and sediments due to industrial activities and mining as well as the use of sludge, pesticides, agriculture chemicals and automobiles (Pandy et al., 2003).

The impact of these problems depends on the chemical forms of the metal, and the capacity of soil and sediments as well as their constituents and properties to immobilize the metals (El-Desoky, 1989).

Chelating agents such as EDTA is effective in mobilizing and removing Pb and Cu from contaminated soils (Chen et al., 2003). Its chelating ability is usually much larger than the naturally occuring ligands (Stevenson, 1991). In a soil contaminated with lead, McBride and Hendershot (1998) studied the effect of levels of soil organic matter on the solubility of Pb speciation within a pH range of 3 to 8. They showed that 30 to 50 % of dissolved Pb were present as soluble organic matter complexes at low pH and up to 80 to 99 % at near neutral pH (6.5-8). Higher pH promoted the formation of soluble organo-Pb complexes, which increases Pb solubility. They concluded that higher contents of organic matter lead to higher concentration of dissolved and labile Pb.

Zhou and Wong (2001) investigated the effects of dissolved organic matter (DOM) on the sorption of Cu in an acidic sandy loam and a calcareous clay loam soil and noticed that increasing the concentration of dissolved organic matter significantly reduced the sorption of Cu by both soils. Dissolved organic matter in the poultry litter and sewage sludge could also contribute effectively in form complexes with heavy metals in soils (McBride, 1989).

The transformation of heavy metals among different forms in contaminated soil profiles is also major environmental concern due to their toxicity and even slow transport through the soil which it may eventually lead to contaminate the groundwater (Li and Shuman, 1997a). Distribution of these metals among their chemical forms is generally dependent on the total content of metals and soil texture (Kabala and Singh, 2001). A sequential extraction of trace metals from soils is potentially valuable in predicting metal transformations between chemical forms in agriculture soils (El-Gendi, 1994). Li and Shuman (1997a) showed that EDTA redistributed Zn, Cd and Pb from the exchangeable, organic matter and Mn oxide forms to the water soluble fraction. Li and Shuman (1997b) also showed that the application of PLE to metal-contaminated soils caused Zn and Cd to be redistributed from the exchangeable to the water soluble fraction and enhance the metal mobility.

The main goal of this study is to evaluate the effect of applying certain organic materials to some contaminated Egyptian soils on the transformations of Pb and Cu among their soil forms.

## Materials and Methods

### I. Characterization of Soils and Organic Materials

Three contaminated soils at Helwan, El-Gabal El-Asfar (Cairo governorate) and Arab El-Madabeg (Assiut governorate) were selected based on their content of heavy metals to evaluate effects of certain organic materials on transformations of Lead (Pb) and Cupper (Cu) forms in some Egyptian contaminated soils. The soils at these locations are receiving a continuous supply of heavy metals as domestic (El-Gabal El-Asfar and Arab El-Madabeg) and/or industrial wastes (Helwan). Soil samples were collected from the surface layer (0-30 cm) of these soils to carry out two different experiments. The collected soil samples were air dried, crushed with a wooden roller, sieved to pass through a 2 mm sieve and kept for analysis and required experiments. Some chemical and physical properties of these soils are present in Table 1.

Four organic materials were used to investigate their effects on the transformations of Pb and Cu forms of these studied contaminated soils. They included ethylene diamine tetra acetic acid (EDTA) as a synthetic organic material as well as poultry litter extract (PLE), vinase (V) and humic acid (HA) solutions as natural organic materials.

The poultry litter (PL) was collected from the poultry farm of Assiut University, Assiut. Vinase, a by-product of sugar industry, was obtained from Abu-korkas Sugar Factory, El-Minya governorate. Moreover, the humic acid solution was brought from the Agriculture Company for Recycling Agriculture Residues, El-Minya governorate. Table 2 shows some properties of these investigated organic materials and their total content of Pb and Cu.

**Table(1):** Some chemical and physical properties of the soils at the different studied locations.

| Property | Soil location | | |
|---|---|---|---|
| | Helwan | El-Gabal El-Asfar | El-Madabeg |
| Particle size distribution | | | |
| Clay (%) | 24.74 | 12.00 | 4.91 |
| Silt (%) | 17.32 | 12.64 | 7.35 |
| Sand (%) | 57.94 | 75.36 | 87.74 |
| Texture | Silty clay loam | Loamy sand | Sand |
| $CaCO_3$ (%) | 5.37 | 2.50 | 6.8 |
| pH (1:2.5) | 8.11 | 6.71 | 7.59 |
| Organic matter (%) | 2.18 | 5.70 | 2.80 |
| EC (1:1 dS/m) | 5.18 | 1.86 | 1.7 |
| Soluble cations and anions (mmol/L) | | | |
| $Na^+$ | 17.31 | 7.97 | 4.18 |
| $K^+$ | 12.46 | 3.79 | 4.37 |
| $Ca^{+2}$ | 12.75 | 3.75 | 3.97 |
| $Mg^{+2}$ | 0.48 | 0.39 | 0.22 |
| $HCO^-_3$ | 4.12 | 2.89 | 3.9 |
| $Cl^-$ | 16.62 | 6.83 | 6.31 |
| $SO_4^{-2}$ | 17.74 | 5.15 | 3.36 |
| DTPA-extractable metals (mg/kg) | | | |
| Pb | 2.70 | 45.51 | 6.16 |
| Cu | 3.21 | 3.75 | 6.07 |
| US.EPA-extractable metals (mg/kg) | | | |
| Pb | 45.60 | 247.20 | 86.10 |
| Cu | 36.10 | 191.20 | 38.30 |
| Total metals (mg/kg) | | | |
| Pb | 56.80 | 261.00 | 95.00 |
| Cu | 55.00 | 195.00 | 45.00 |

**Table(2):** Some properties of the investigated organic materials and their total content of Zn and Mn.

| Organic Material | Pb | Cu | EC | pH | OM |
|---|---|---|---|---|---|
| | mg/kg | | (dS/m) | | (%) |
| PLE (25g/L) | 0.40 | 0.10 | 1.65 | 7.46 | 1.90 |
| PLE (50g/L) | 0.40 | 0.13 | 3.02 | 7.50 | 2.10 |
| PLE (75g/L) | 0.51 | 0.15 | 4.20 | 7.66 | 2.25 |
| Vinase | 0.39 | 1.15 | 14.70 | 4.45 | 5.11 |
| Humic acid | 0.12 | 0.10 | 25.90 | 12.90 | 3.10 |

**II. Experiment**

- **Preparation of organic material treatments.**

EDTA was applied every week at a level of 6 mmol/kg soil. The respective powder amount of EDTA was dissolved in the required amount of distilled water that brought each soil to its saturation capacity resulting in a respective level of 48 mmol/kg soil at the end of the experiment.

The collected Poultry litter (PL) was air-dried, sieved through a 2 mm sieve and stored in a plastic bag. The poultry litter extract (PLE) was prepared by using 75 g of the PL suspended in one litter of distilled water, steered for 2 h., filtered by filter paper, and stored in the refrigerator at 6 C for use. The amount of PLE to make each soil to reach its saturation capacity was added weekly to each soil columns. Pure vinase was used in this experiment. The weekly added amount of each level was to make each soil to reach its saturation capacity.

The humic acid solution that contained 2% HA was kept in a plastic container at 6 Co in the refrigerator for use. This solution was used to gave a HA level of 0.2% in the soil at the end of the experiment. The right amount of HA was divided into 8 doses for 8 weeks to give weekly respective level of 0.025%. Each dose of HA was mixed with the required amount of distilled water to make each soil to reach its saturation capacity and then added weekly to each soil column.

**An incubation experiment**

This experiment aimed to show the soil form(s) of Pb and Cu that involved in this mobility. In another meaning, it could show the transformations among various soil Pb and Cu forms that contribute in the mobility increase of these metals. The design of this experiment was completely randomized with 3 replications.

Samples of 200g of each studied soil material were put in plastic cups, watered every week with of each organic material to reach the water saturation capacity of each soil material and incubated at Lab temperature for 8 weeks. Then, the soil samples in the plastic cups were continued in the incubation for another 8 weeks with adjusting the water content of each soil material at 60% of the saturation capacity by the daily addition of ionized water during this incubation period. At the end of the experiment (16 weeks), samples from the incubated soil materials were taken, air-dried, sieved through a 2 mm sieve and kept for Pb and Cu fractionation using sequential extractions.

**Pb and Cu Fractionation**

Two grams of each soil material were weighed and placed in a 50 ml polyacarbnate centrifuge tube. Sequential extractions of soluble and exchangeable (S+EXCH, Kabala and Singh, 2001), Carbonate specifically bound (C-bound, Ahnstrom and Parker, 1999), Mn oxide bound (MnO-bound, Sims et al., 1986), Organic bound (O-bound, Shuman, 1979), poorly crystalline Fe oxide bound (PCFeO-bound, Shuman, 1979) and crystalline Fe oxide bound (CFeO-bound, Kittric and Hope,

1963) forms of Pb and Cu were carried out. The total amount of soil Pb and Cu in the studied soil materials was estimated using the digestion with concentrated acids of HF, $HNO_3$ and HCl (Shuman, 1979). The Residual form of each metal was estimated by the difference between the total amount of a metal in the soil samples and sum of the six extracted forms of this metal.

Pb and Cu in soil material digests were determined using a model 906 of GBC atomic absorption spectrophotometry. Data were subjected to statistical analysis according Sendcor and Cochrana (1980). The means of treatments were compared using the least significant difference (LSD) values at a 5% level of significance.

**Results and Discussion**

**1. Lead**

Table 3 shows the concentrations of various Pb forms in Helwan, El-Gabal El-Asfar and El-Madabeg soils incubated with the studied organic materials for 8 weeks. All organic materials significantly increased the S+EXCH form in all studied soils. The increments varied from soil to another due to its physico-chemical properties and its content of lead as well as the type of organic material

The increase in the S+EXCH Pb form induced by EDTA, PLE and HA in Helwan soil was mainly on the expense of the carbonate, poorly crystalline Fe oxide and crystalline Fe oxide forms as well as the residual form of Pb. The respective decreases in these forms for Helwan soil were 32.2, 1.1, 2.2 and 11.6% with EDTA, 7.3, 8.9, 11.7 and 2.2% with PLE, and 1.0, 5.6, 7.5 and 3.8% with HA. Meanwhile, the increase in the S+EXCH Pb form in Helwan soil with vinase was mainly on the charge of the carbonate, crystalline Fe oxide and the residual forms of Pb. The corresponding decreases of Pb in these forms were 27.9, 10.0 and 3.0 %. On the other hand, Mn oxide and organically bound Pb forms in Helwan soil increased after the incubation with all investigated organic materials. These increases may be on the cost of the decreases in the poorly crystalline as well as crystalline Fe oxide and the residual forms of Pb in this soil.

Increases in the S+EXCH Pb form were also recorded in El-. Gabal El-Asfar soil treated with all organic materials except HA. The increase in Pb of this form induced by EDTA in this soil as well as in El-Madabeg one was very high. The increases in this form of Pb of El-Gabal El-Asfar soil due to EDTA and PLE applications were mainly on the cost of decreases in Pb of the Mn oxide, poorly crystalline and crystalline Fe oxide as well as residual forms. The relatively respective reductions in Pb of these forms were 23.2, 30.8, 8.0 and 2.5% with EDTA and 14.2, 1.4, 40.2 and 1.0% with PLE. Meanwhile, the increases in the S+EXCH Pb form induced by vinase in this soil were mainly on the charge of decreases in the Mn oxide, organically bound, poorly crystalline and crystalline Fe oxide as well as the residual forms of Pb. The corresponding reductions in these forms of Pb in this soil were 10.5, 10.8, 2.4, 27.6 and 1.6%. It was noticed in this soil that the carbonate bound form of Pb increased after incubation with all investigated organic materials. In presence of HA, Pb released from the residual and organically bound forms of this soil might transform to the carbonate, Mn oxide, poorly crystalline and crystalline Fe oxide bound forms. El-Gabal El-Asfar soil has the highest content of organic matter so that $CO_2$ released from organic matter degradation may participate in increasing carbonate bound form in this soil. This soil also contains high levels of Mn and Fe that under the high pH of HA, new Mn and Fe oxide forms containing some released Pb from the organically bound and residual forms may be formed.

All investigated organic materials induced increments in Pb exchangeable form of El-Madabeg soil. EDTA in this soil also showed the highest increase in the Pb exchangeable form. It was followed by vinase and then PLE and HA. The increase in the Pb exchangeable form of this soil induced by EDTA was mainly on the expense of decreases in Pb of the Mn oxide, poorly crystalline and crystalline Fe oxide bound as well as the residual forms. The respective reductions in Pb of these forms induced by EDTA were 47.4, 53.0, 21.4 and 21.3%. Vinase caused an increase in the Pb exchangeable form of this soil on the charge of decreasing the Mn oxide, organically, poorly crystalline Fe oxide and residual bound forms of Pb accounted by 52.3, 5.4, 31.0 and 8.8%, respectively. The increases in Pb of the exchangeable form of El-Madabeg soil induced by PLE and HA were on the cost of decreases of the carbonate, Mn oxide, organically and residual bound forms accounted by 7.1, 46.0, 67.2 and 1.2% for PLE, and 36.9, 52.6, 45.7 and 1.4% for HA. This soil showed also Pb increases in the carbonate and organically bound forms induced by EDTA and Pb increases in the carbonate and crystalline Fe oxide bound forms induced by vinase. Humic acid also gave increases in the poorly crystalline as well as crystalline Fe oxide bound forms of Pb of this soil.

Since the ability of synthetic materials such as EDTA to chelate metals is much higher than other natural organic materials (PLE, vinase and humic acid), EDTA was the most efficient organic material in transforming Pb from Mn and Fe oxide as well as residual bound forms to the S+EXCH form. The most accepted mechanism that involved in the transformation of Pb among various forms could be the release of some Pb levels of some soil forms, such as oxide and residual forms, by complexation and/or chelation by soluble organics and various functional groups of the investigated organic materials followed by distributing these Pb levels on other soil forms, such as exchangeable and organically bound ones. Naidu and Harter (1998) suggested that the metal is released into soil solution as metal-organic complexes at high pH values. Humic acids are organic matters that contain various kinds of functional groups such as carboxylic or phenyl groups that can complex metal ions (Ali and Dzombak, 1996; Ho-Geong et al., 2005). Organic complexation of metals in soils and waters is one of the most important factors governing solubility and bioavailability of metals in the environment. (Berden and Berggren, 1990).

Concerning the different forms of Pb regardless the residual one of the studied soils, Table 3 also showed that the crystalline Fe oxide bound form (CFeO) was the dominant form of Pb in Helwan soil. The poorly crystalline Fe oxide bound one (PCFeO) was dominant in El-Gabal El-Asfar and El-Madabeg soil. The results also indicated that the Fe oxide bound forms of Pb in these soils are more important compared to the Mn oxide bound one. Higher levels of Pb are bound to Fe oxides than to Mn oxides, indicating that Fe oxides might play a greater role in the adsorption of metals such as Pb in these soils. El-Gabal El-Asfar and El-Madabeg soils contain higher Pb levels in the poorly crystalline Fe oxide form than in the crystalline Fe oxide one. The high organic matter content of these two soils (Table 1) may delay and inhibit the crystallization of Fe oxides resulting in high amounts of the poorly crystalline Fe oxides (Schwertmann et al., 1986). Sequential extraction results also show that most of Pb in these soils is present in the residual form (Table 3 and Figure 1).

The redistribution of Pb forms (as percentages of the total Pb) in the studied soils, induced by the investigated organic materials is illustrated in Figure 1. Incubating Helwan soil with EDTA, the exchangeable, Mn oxide and organically bound forms of Pb increased from 2.32 to 9.91%, from 0.88 to 2.39% and from 1.20 to 2.39%, respectively, of the total content, while the residual form of Pb decreased from 69.79 to 61.73% of the total content. For El-Gabal El-Asfar soil, EDTA caused the exchangeable, and carbonate bound forms of Pb to increase from 0.90 to 9.00% and from 1.56 to 4.27%, respectively, of the total content, while the poorly crystalline Fe oxide and residual bound forms of Pb decreased from 29.08 to 20.14% and from 65.85 to 64.24%, respectively, of the total Pb content. Moreover, EDTA induced the Pb exchangeable form of El-Madabeg soil to increase from 1.07 to 25.21% of the total Pb. However, the poorly crystalline Fe oxide and residual bound Pb forms of this soil decreased from 16.36 to 7.70% and from 72.34 to 56.92%, respectively, of the total Pb.

It is obvious that Pb increments in the exchangeable form induced by EDTA were transformed mainly from the residual form in Helwan soil, from poorly crystalline Fe oxide bound form in El-Gabal El-Asfar soil, and from the residual and poorly crystalline Fe oxide bound forms in El-Madabeg soil. Li and Shuman (1997a) reported that leaching the soil with EDTA transformed Pb from exchangeable, O-bound and MnO to water soluble form. Moreover, Shen et al. (2002) found that EDTA treatments significantly, decreased Pb in both carbonate and Fe-Mn oxide bound forms.

In addition, Sun et al. (2001) showed that the exchangeable Pb increased after leaching with EDTA in most soils. In general, the Pb levels that were transformed by EDTA in Helwan, El-Gabal El-Asfar and El-Madabeg soils were 10.12, 10.82 and 25.88%, respectively, of the total content. Meanwhile, the studied soils may be ranked according to the progress degree in Pb transformation inducaed by EDTA as: El-Madabeg soil > El-Gabal El-Asfar soil ≈ Helwan soil.

Vinase increased the exchangeable, Mn oxide and organically bound Pb in Helwan soil from 2.32 to 5.42%, from 0.88 to 1.71% and from 1.20 to 2.57%, respectively, of the total Pb content. These increments were on the charge of decreasing carbonate, crystalline Fe oxide and residual bound forms of Pb in this soil from 5.25 to 3.75%, from 12.66 to 11.29% and from 69.79 to 67.14%, respectively, of the total content. In El-Gabal El-Asfar soil treated with vinase, low Pb increments were recorded in the exchangeable and carbonate bound forms on the expense of decreasing the residual form. However, vinase in El-Madabeg soil induced a high Pb increments the exchangeable form (from 1.07 to 13.32% of the total content) on the cost of decreasing both the poorly crystalline Fe oxide and the residual bound forms (from 16.36 to 11.24% and from 72.34 to 65.72%, respectively). In general, the Pb increment in the exchangeable form induced by vinase was transformed mainly from both the residual and poorly crystalline Fe oxide forms in these soils. The levels of Pb that were transformed by vinase in Helwan, El-Gabal El-Asfar and El-Madabeg soils were 5.52, 2.41 and 13.52%, respectively, of the total Pb content. Depending upon the progress of Pb transformation induced by vinase, the soil can be ordered as: El-Madabeg soil > Helwan soil > El-Gabal El-Asfar soil. Very low relative increases in the Pb exchangeable form of the studied soils were recorded with the poultry litter extract and humic acid.

**Table(3):** Effect of some organic materials on the redistribution of lead (Pb) among various forms of the studied soils.

| Soil | Treatment | Pb forms (mg/kg) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | S+EXCH | C-bound | MnO | O-bound | PCFeO | CFeO | Sum | Residual | Total |
| Helwan | control | 1.32 | 2.98 | 0.50 | 0.68 | 4.49 | 7.19 | 17.16 | 39.64 | 56.80 |
| | EDTA | 5.63 | 2.02 | 1.26 | 1.36 | 4.44 | 7.03 | 21.74 | 35.06 | 56.80 |
| | PLE | 2.44 | 2.79 | 1.57 | 1.44 | 4.09 | 6.35 | 18.68 | 38.76 | 57.44 |
| | Vinase | 3.10 | 2.15 | 0.98 | 1.47 | 4.65 | 6.47 | 18.82 | 38.47 | 57.29 |
| | HA | 1.38 | 2.95 | 2.44 | 1.00 | 4.24 | 6.65 | 18.66 | 38.14 | 56.80 |
| | LSD0.05 | 0.45 | 0.73 | 0.44 | 0.35 | 0.70 | 0.61 | - | 0.75 | - |
| El-Gabal El-Asfar | control | 2.36 | 4.07 | 1.72 | 1.57 | 75.91 | 3.51 | 89.13 | 171.87 | 261.00 |
| | EDTA | 23.50 | 11.15 | 1.32 | 1.58 | 52.55 | 3.23 | 93.34 | 167.66 | 261.00 |
| | PLE | 2.61 | 8.42 | 1.39 | 1.62 | 74.85 | 2.10 | 90.99 | 170.71 | 261.70 |
| | Vinase | 3.66 | 9.26 | 1.54 | 1.40 | 74.08 | 2.54 | 92.49 | 169.05 | 261.54 |
| | HA | 2.32 | 4.52 | 2.08 | 1.41 | 76.73 | 4.67 | 91.72 | 169.30 | 261.01 |
| | LSD0.05 | 2.24 | 1.16 | 0.37 | 0.48 | 3.24 | 0.26 | - | 0.95 | - |
| El-Madabeg | control | 1.02 | 3.52 | 3.02 | 1.86 | 15.54 | 1.31 | 26.28 | 68.72 | 95.00 |
| | EDTA | 23.95 | 4.36 | 1.59 | 2.68 | 7.31 | 1.03 | 40.92 | 54.08 | 95.00 |
| | PLE | 1.83 | 3.27 | 1.63 | 0.61 | 18.35 | 1.70 | 27.40 | 67.87 | 95.27 |
| | Vinase | 12.70 | 3.64 | 1.44 | 1.76 | 10.72 | 2.42 | 32.68 | 62.67 | 95.35 |
| | HA | 1.53 | 2.22 | 1.43 | 1.01 | 18.66 | 2.38 | 27.23 | 67.78 | 95.01 |
| | LSD0.05 | 2.20 | 1.13 | 0.56 | 0.16 | 5.67 | 0.65 | - | 2.13 | - |

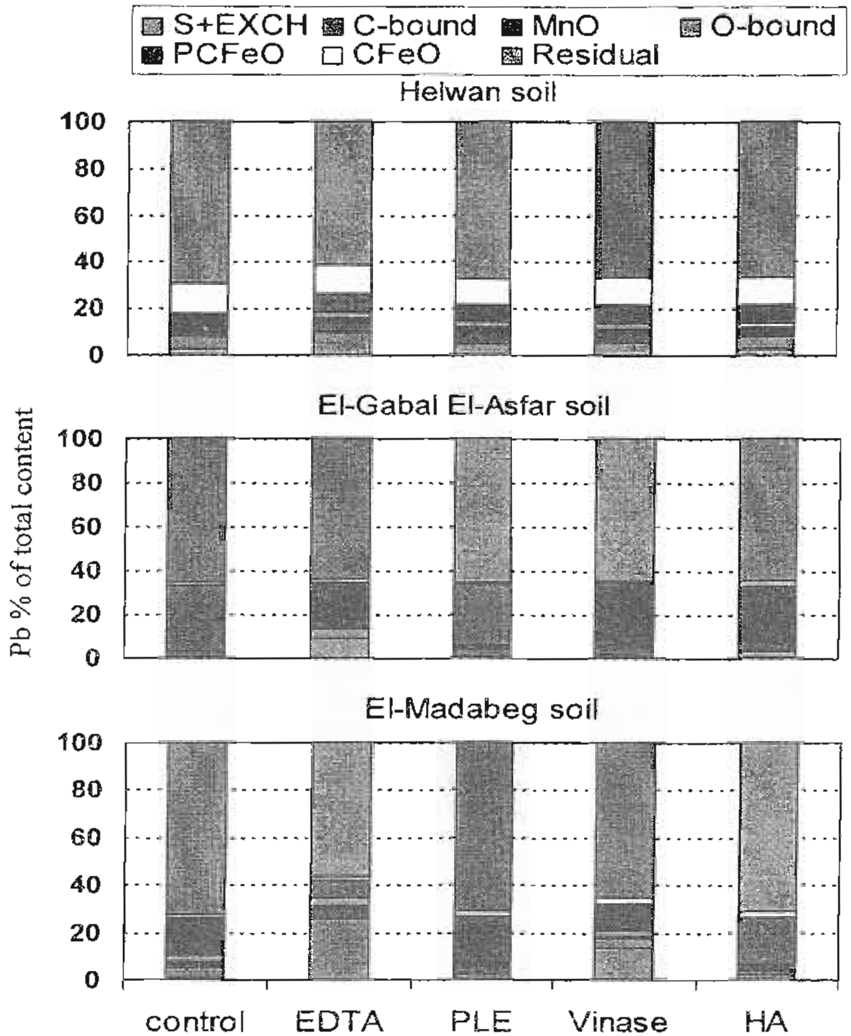


**Fig.(1):** Redistribution of Pb (as percentages of the total content) among its chemical forms of the studied soils treated with organic materials.

Most of Pb transformations induced by these two materials were from the residual and crystalline Fe oxide forms to Mn oxide and organically bound forms in Helwan soil and to the carbonate bound one in El-Gabal El-Asfar soil, and from the Mn oxide and organically bound forms to the poorly crystalline and crystalline Fe oxide ones in El-Madabeg soil. In general, the percentages of Pb that were redistributed by PLE in Helwan, El-Gabal El-Asfar and El-Madabeg soils were 5.09, 1.78 and 4.15%, respectively, of the total content. However, the respective Pb ones induced by HA were 4.09, 1.08 and 4.66% of the total content

**2. Copper**

The distribution of Cu among its various forms of the studied soils treated with the investigated organic materials is shown in Table 4.

The increase in the S+EXCH Cu form caused by EDTA in Helwan and El-Madabeg soils was on the expense of decreasing the organically-bound, poorly crystalline and crystalline Fe oxide forms as well as the residual form of Cu. The respective decreases in these forms induced by EDTA were 56.7, 4.1, 35.9 and 14.5% for Helwan soil and 54.6, 57.9, 37.4 and 21.7% for El-Madabeg soil. Sadovnikova et al. (1996) found that incubating soils with farmyard manure or sewage sludge increased Cu availability, but the increases were quantitatively small. It was reported that the addition of free EDTA or the existence of other metal–EDTA complexes could result in the partial remobilization of adsorbed metals from metal oxides and in the simultaneous dissolution of minerals (iron and aluminum oxides) and remobilization of adsorbed metals (Van Devivere et al., 2001).

In El-Gabal El-Asfar soil the increase in the S+EXCH Cu form induced by EDTA was mainly on the charge of the decreases in the organically bound, poorly crystalline Fe oxide and residual forms. These decreases were 62.2, 25.3 and 11.3%, respectively. Higher Cu increases in the S+EXCH form of El-Gabal. El-Asfar soil were induced by all investigated organic materials, especially EDTA and vinase compared to those of the other two soils. Moreover, EDTA caused significant increases in the carbonate bound and Mn oxide forms of Cu in Helwan soil, in the carbonate bound and crystalline Fe oxide forms in El-Gabal El-Asfar soil, and in the carbonate bound form in El-Madabeg soil. These increases may be on the charge of the decreases in the organically bound, Fe oxide and residual forms of Cu.

**Table(4):** Effect of some organic materials on the redistribution of copper (Cu) among various forms of the studied soils.

| Soil | Treatment | Cu forms (mg/kg) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | S+EXCH | C-bound | MnO | O-bound | PCFeO | CFeO | Sum | Residual | Total |
| Helwan | control | 1.32 | 2.66 | 0.97 | 2.24 | 6.80 | 5.54 | 19.53 | 35.47 | 55.00 |
| | EDTA | 9.02 | 3.26 | 1.37 | 0.97 | 6.52 | 3.55 | 24.69 | 30.31 | 55.00 |
| | PLE | 1.85 | 2.68 | 0.70 | 1.50 | 11.40 | 4.29 | 22.43 | 32.75 | 55.18 |
| | Vinase | 5.39 | 2.48 | 1.77 | 2.16 | 4.83 | 5.33 | 21.96 | 34.48 | 56.44 |
| | HA | 1.44 | 2.50 | 0.51 | 1.82 | 14.87 | 2.28 | 23.42 | 31.59 | 55.01 |
| | $LSD_{0.05}$ | 0.56 | 0.51 | 0.24 | 0.39 | 0.78 | 0.72 | - | 0.97 | - |
| El-Gabal El-Asfar | control | 2.55 | 4.25 | 0.99 | 70.65 | 35.31 | 3.39 | 117.14 | 77.86 | 195.00 |
| | EDTA | 51.23 | 14.96 | 1.26 | 26.69 | 26.39 | 5.38 | 125.91 | 69.09 | 195.00 |
| | PLE | 5.65 | 6.32 | 1.60 | 55.67 | 51.80 | 3.61 | 124.65 | 70.56 | 195.21 |
| | Vinase | 22.35 | 22.83 | 10.22 | 34.23 | 40.09 | 4.55 | 134.28 | 62.31 | 196.59 |
| | HA | 3.38 | 6.50 | 1.23 | 84.16 | 35.00 | 5.18 | 135.45 | 59.56 | 195.01 |
| | $LSD_{0.05}$ | 0.94 | 1.36 | 1.19 | 2.74 | 1.58 | 0.65 | - | 1.2 | - |
| El-Madabeg | control | 1.05 | 1.45 | 1.20 | 5.13 | 15.35 | 5.38 | 29.56 | 15.44 | 45.00 |
| | EDTA | 16.27 | 3.56 | 0.92 | 2.33 | 6.47 | 3.37 | 32.91 | 12.09 | 45.00 |
| | PLE | 2.66 | 2.57 | 0.72 | 4.50 | 15.38 | 4.65 | 30.48 | 14.62 | 45.10 |
| | Vinase | 7.88 | 3.88 | 1.57 | 5.10 | 8.29 | 3.46 | 30.18 | 15.61 | 45.79 |
| | HA | 1.77 | 1.89 | 0.92 | 5.20 | 20.10 | 2.70 | 32.57 | 12.43 | 45.00 |
| | $LSD_{0.05}$ | 0.55 | 0.38 | 0.39 | 0.98 | 1.90 | 0.57 | - | 0.79 | - |

Vinase came in the second order after EDTA followed by PLE and HA in increasing the S+EXCH form of Cu in the studied soils. The increments in this form existed by vinase were on the expense of decreasing the poorly crystalline Fe oxide and residual forms (29.0 and 2.8%, respectively) in Helwan soil, the organically bound and residual forms (51.6 and 20.0%, respectively) in El-Gabal El-Asfar soil, and the poorly crystalline and crystalline Fe oxide forms (46.0 and 35.7%, respectively) in El-Madabeg soil. Moreover, vinase induced significant Cu increases in the Mn oxide form in Helwan soil, the carbonate bound, Mn oxide, poorly crystalline and crystalline Fe oxide forms in El-Gabal El-Asfar soil, and the carbonate bound in El-Madabeg soil.

The Cu increments in the S+EXCH form of the studied soils induced by PLE and HA were very low but they were on the charge of the decreases in the Mn oxide, organically bound, crystalline Fe oxide and residual forms in the Helwan soil, and the crystalline Fe oxide and residual forms in El-Madabeg soil. In El-Gabal El-Asfar soil, Cu increments in the exchangeable, carbonate bounded and poorly crystalline Fe oxide forms induced by PLE were on the expense of the organically bounded and residual forms (21.2 and 9.4%, respectively). Copper released from the residual form in this soil due to HA treatment transformed to exchangeable, carbonate bounded, organically bounded and crystalline Fe oxide forms. Increases in the carbonate bound form of Cu also were recorded in El-Gabal El-Asfar soil, and El-Madabeg soils induced by all organic materials, especially with vinase and EDTA. High pressure of $CO_2$ released from the degredation of organic matter that is very high may correspond in increasing the carbonate bound form of Cu in these soils. The addition of tested organic materials significantly decreased Cu levels in the residual form of all investigated soils.

Data also indicate that the dominant fraction in the non-residual Cu forms was the poorly crystalline Fe oxide in Helwan and El-Madabeg soils (Table 4). It may be attributed to the high stability constants of the Cu-oxide as reported by (Agbenin and Olojo, 2004). On the other hand, the organically bounded Cu was the dominant form in El-Gabal El-ElAsfar. However, both the organically bound and the poorly crystalline Fe oxide forms dominated in this soil after adding organic materials. Karaca (2004) reported that the organic form of Cu could be expected to be the dominant form in soils that contain large values of organic matter as well as in soils treated with sewage sludge (Sposito et al, 1982).

The results, clearly, show that the addition of humic acid to El-Gabal El-Asfar soil resulted in increasing Cu of the organically bounded form (Table 4). The high values of both pH of HA and organic matter may also participate in increasing the organically bounded Cu form (19.1%) of El-Gabal El-Asfar soil with humic acid. Copper has a very high affinity to organic compounds as indicated by Almas and Singh (2001). They reported that the stability of metal-organo complexes is, however, attributed to the heavy metals affinity for organic mater. They added that Cu and Pb form stable complexes with dissolved organic matter, and only a very small fraction of these metals exists as free hydrated metal ions when soil pH is suitable. Humification of sewage sludge and organic matter during incubation experiments conducted by Iakimenko et al. (1996) tended to promote the formation of fulvic acids that have a high capacity for Cu2+ complexation (Tipping and Hurley, 1992). Copper in the soil solution and exchangeable, specifically adsorbed, and organically-bound Cu are considered in equilibrium and represent the available forms for plant uptake whereas Cu in the oxide-bound and residual forms is relatively unavailable to plants (Adriano, 1986).

The results indicated that vinase caused Cu increases in the Mn oxide form in all studied soils (Table 4). Very high levels of Cu were present in this form of El-Gabal El-Asfar soil treated with vinase due to the high Mn content of this soil as well as to the higher Mn content and the low pH of vinase. The distribution of Cu between the main soil constituents was reported to be influenced considerably by the presence of free manganese oxides (Mclaren and Crawford, 1973).

The redistribution of various forms of Cu as percentages of the total Cu in the studied soils treated with the investigated organic materials is illustrated in Figure 2. The exchangeable, carbonate bound and Mn oxide forms of Cu in Helwan soil treated with EDTA increased from 2.41 to 16.39%, from 4.84 and 5.93 % and from 1.76 to 2.50%, respectively, of the total Cu, while the organically bound, crystalline Fe oxide and residual forms decreased from 4.07 to 1.76%, from 10.07 to 6.46% and from 64.50 to 55.11%, respectively, of the total Cu. In El-Gabal El-Asfar soil, EDTA caused the organically bound, poorly crystalline Fe oxide and residual forms of Cu to decrease from 36.23 to 13.69%, from 18.11 to 13.53% and from 39.93 to 35.43%, respectively, of the total Cu. The released Cu from these forms of this soil transformed to the exchangeable, carbonate bound and crystalline Fe oxide forms resulting in Cu increases in these forms from 1.31 to 26.27%, from 2.18 to 7.67% and from 1.74 to 2.76%, respectively, of the total. Moreover, Cu levels were released form the organically bound, crystalline and crystalline forms as well as the residual form of the El-Madabeg soil treated with EDTA and changed from 11.41to 5.18%, from 34.12 to 14.37%, and from 11.95 to 7.48%, respectively, of the total content and were transformed to the exchangeable, and carbonate bound forms which their Cu levels increased from 2.33 to36.16% and from 3.22 to 7.90%, respectively, of the total content.

It is clear that the EDTA induced Cu increases in the S+EXCH form were transformed mainly from the residual form and partially from the crystalline and organically bound forms in Helwan soil, mainly from the organically bound and partially from the poorly crystalline Fe oxide and residual forms in El-Gabal El-Asfar soil, and mainly from the poorly crystalline Fe oxide form and partially, from the organically bound and crystalline Fe oxide forms in El-Madabeg soil. Generally, the levels of Cu that soils were 15.81, 30.6 and 38.51%, respectively, of the total. The studied soils may be ranked with respect the progress of Cu transformation induced by EDTA as: El-Madabeg soil > El-Gabal El-Asfar soil > Helwan soil.

Copper levels were released from the poorly crystalline Fe oxide and residual forms of Helwan soil induced by vinase from 12.36 to 8.56% and from 64.50 to 61.09%, respectively, of the total Cu and were redistributed to the exchangeable and Mn oxide forms which their Cu levels were raised from 2.41 to 9.56% and from 1.76 to 3.14%, respectively, of the total content. In El-Gabal El-Asfar soil, vinase caused Cu levels to release from 36.23 to 17.41% and from 39.93 to 31.70% of the total content from the organically bounded and residual forms, respectively, and to be transformed to the exchangeable, carbonate bound, Mn oxide and poorly crystalline Fe oxide forms. as their Cu increased from 1.31 to 11.37%, from 2.18 to 11.61, from 0.51 to 5.20% and from 18.11 to 20.39%, respectively, of the total content. Meanwhile, Cu levels, mainly from the poorly crystalline Fe oxide form (from 34.12 to 18.11% of the total) and partially from the crystalline Fe oxide (from 11.95to 7.56% of the total), were transformed to the exchangeable form (from 2.33 to 17.21% of the total) and the carbonate bound form (from 3.22 to 8.47% of the total). Generally, the vinase induced Cu increases in the exchangeable form transformed mainly from the

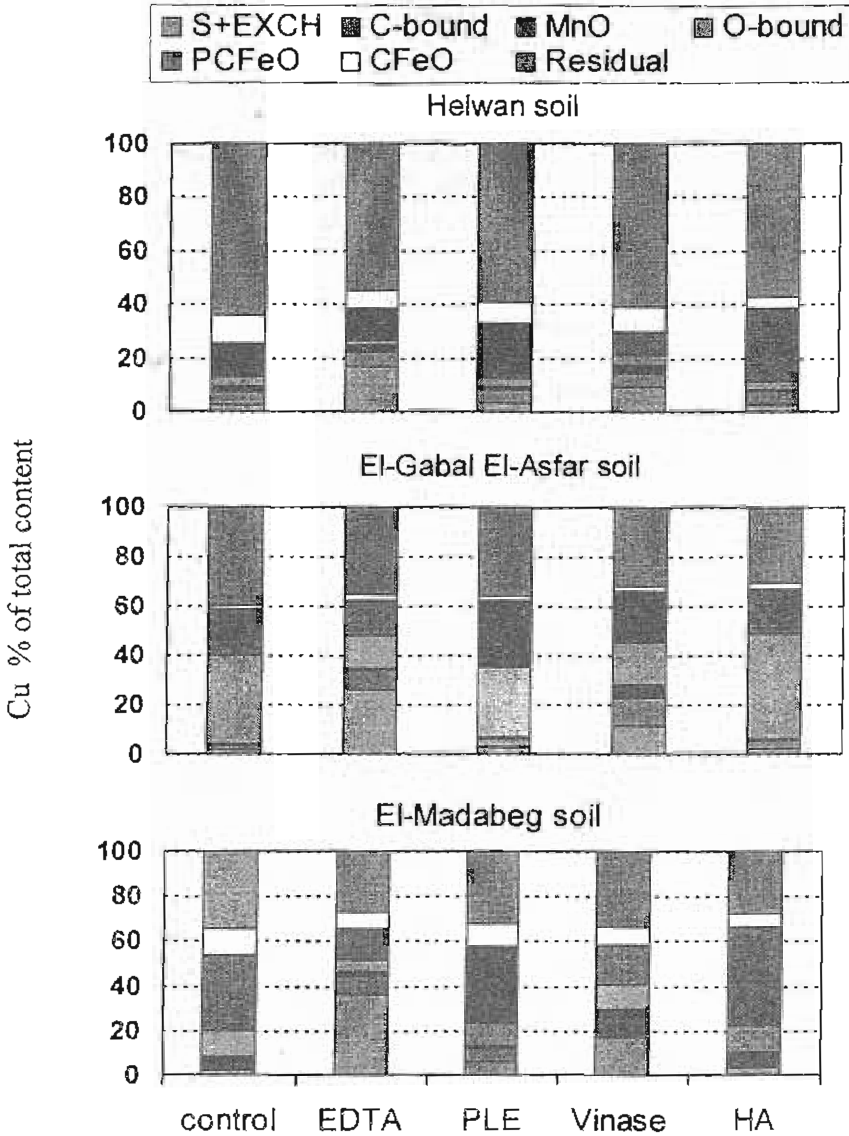


**Fig.(2):** Redistribution of Cu (as percentages of the total content) among its chemical forms of the studied soils treated with organic materials.

poorly crystalline Fe oxide and residual forms in Helwan soil, mainly from the organically bound form and partially from the residual one, and from the poorly crystalline and crystalline Fe oxides forms in El-Madabeg soil. Copper percentages that were redistribed by vinase among various forms of Helwan, El-Gabal El-Asfar and El-Madabeg soils were 8.53, 26.76 and 20.9%, respectively, of the total. The soil can be ordered according to Cu increases induced by vinase as: El-Gabal El-Asfar soil > El-Madabeg soil > Helwan soil. Martınez et al. (2003) found a paralleled behaviour between dissolved organic-C and total soluble metal concentrations in OM-rich soil.

Low Cu transformations among different forms of the studied soils were induced by PLE and HA (Figure 2). The studied soils can be ordered according to the transformed Cu levels by PLE as: El-Gabal El-Asfar soil > Helwan soil >El-Madabeg soil.

The levels of Cu that were transformed by HA among different forms of Helwan, El-Gabal El-Asfar soils and El-Madabeg soils were 14.88, 8.62 and 13.27%, respectively, of the total. Usman and Gallab (2006) indicated that Cu transformed from the carbonate bound form towards the exchangeable, organically bound and residual forms due the irrigation with sewage water for 4 years.

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# تحولات الرصاص والنحاس بين صورها الكيميائية فى بعض الأراضى المصرية الملوثة والمعاملة بالمواد العضوية.*

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تم إجراء تجربة تحضين في المعمل أستخدم فيها ثلاث أنواع من الترب المصرية الملوثة هي حلوان، الجبل الأصفر (محافظة القاهرة) ، وعرب المدابغ (محافظة أسيوط) لمدة 16 أسبوعا وذلك لتقييم أثر إضافة المواد العضوية وهى EDTA ، كمادة عضوية صناعية وكذلك مستخلص فرشة الدواجن (PLE) والفيناس وحامض الهيوميك كمواد عضوية طبيعية وذلك مقارنة بالماء المقطر لدراسة تأثير إضافة هذه المواد العضوية على تحولات وإعادة توزيع العناصر الثقيلة المدروسة(الرصاص والنحاس) فيما بين صورها الكيميائية لإظهار الصور الكيميائية للعناصر الثقيلة في التربة التي تشارك في هذه الحركة في هذه الترب الملوثة. تصميم التجربة كامل العشوائية بثلاث مكررات. والصور هي المتبادل ، والمرتبط بالكربونات ، المرتبط بأكاسيد المنجنيز ، المرتبط بالمادة العضوية ، المرتبط بأكاسيد الحديد ضعيفة التبلور ، المرتبط بأكاسيد الحديد المتبلورة ثم المتبقي .

وضعت عينات من الترب المستخدمة فى أصيص بلاستك وعوملت بهذه المواد العضوية عند مستوى 6 ميللمول EDTA / كجم ، 75 جم / لتر من PLE ، للفيناس النقي ؛ 0.025% حامض هيوميك 8 مرات بمحاليل هذه المواد(مرة واحدة / أسبوع) مقارنة بالماء المقطر ، وتم إجراء استخلاص تتابعي على العينات بعد انتهاء فترة التحضين (8 أسابيع أخرى) لدراسة الصور الكيميائية المختلفة لهذه العناصر الثقيلة جميع المواد العضوية تحت الدراسة أدت إلى زيادة معنوية في مستوى الصورة المتبادلة من كل عنصر في كل الترب المستخدمة مقارنة بالكنترول ، وقد اعتمدت قيمة هذه الزيادة على نوع التربة ومحتواها من العنصر الثقيل ونوع المادة العضوية . كان EDTA أكثر المواد العضوية فاعلية في زيادة مستوى الصورة المتبادلة لهذه العناصر الثقيلة في كل الترب المستخدمة ، تلاه الفيناس ثم مستخلص فرشة الدواجن ، على الجانب الآخر ، فقد كان حامض الهيوميك أقل المواد العضوية فاعلية في زيادة هذه الصورة من العناصر الثقيلة في كل الترب المستخدمة ، وقد يرجع انخفاض كفاءة كل من مستخلص فرشة الدواجن وحامض الهيوميك في تحريك العناصر الثقيلة وتحولاتها إلى قصر الفترة الزمنية المستخدمة في هذه التجربة وانخفاض مستويات إضافتها .

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