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IN SITU IMMOBILIZATION OF COPPER, ZINC AND LEAD POLLUTED SOILS.

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ABSTRACT

In situ immobilization technique. Two rates (0.5 and 1.0%) of five immobilizing agents (hydrogel, polymer, zeolite, mud and geothite) were used for remediation of Cu, Zn and Pb polluted soils. Three soils containing various levels of Cu (66.9-82.9 ug/g) , Zn (150–328.0 ug/g) and Pb (59.7– 181 ug/g) were used. Incubation experiment was conducted to study the effect of these agents on soil available content of Cu, Zn and Pb. All immobilizing agents reduced the amount of DTPA available of these metals . The addition of 0.5 and 1% application rate of all agents was sufficient to decrease the DTPA extractable Cu by more than 50% compared to the untreated soils. The DTPA extractable Zn decreased by values ranged between 39.6-86.7% and 49.3 to 92.6% for soils treated with 0.5 and 1% , respectively compared to untreated soils. The available Pb values was decreased by 44.7-57.8 and 47.5-75.4% compared to untreated soils at application rate 0.5% and 1%, respectively. The ability of these agents in immobilizing Cu, Zn and Pb increased with increasing their rate of application and could be arranged as follows :

Zeolite> Polymer>Goethite>Mud>Hydrogel for Cu

Hydrogel > Mud > Goethite> Polymer> Zeolite for Zn

Hydrogel> Mud> Zeolite> Goethite> Polymer for Pb in the tested three soils.

Keywords: heavy metals, remediation, immobilization, Copper, Zinc and Lead

INTRODUCTION

The contamination of soils with toxic heavy metals is responsible for several environmental problems and risk to human health. Elevated concentration of heavy metals in soils can affect flora, fauna and human living. Metal contaminated soils could be remediated by chemical, physical and biological techniques Remediation strategies for metal contaminated sites may incorporate several distinct technology options assembled into a treatment train to attain specific site cleanup goals .These technologies could be grouped into two categories, ex-situ remediation techniques, and in situ fixation of heavy metal using exterior amendments which is a promising technology for cleaning up contaminated soils and wastes.

Stabilization and immobilization of metals in soil are very promising techniques because of their simplicity , high effectiveness, in situ applicability and low cost (Guo et al., 2006).

In situ chemical fixation, involves the use of specific chemical amendments to induce chemical reactions that provide for long-term immobilization of the contaminant without substantially altering the soil properties. On the other hand, in situ remediation approach creates a final solution that is protect human health and the environment. Stabilization is a remediation technology based on adding easily available amendments to polluted soil (e.g. cement, apatite, zeolites, lime), in order to reduce the mobility and bioavailability of metals in the soil without altering their total concentration (Friesl-Hanl et al., 2009; Lee et al., 2009).

Aboulroos et al., (2006) tested three rates (0.25, 0.5 and 1.0%) of seven immobilizing agents (cement, slag, phosphate rock, bitumen, Fe- and Al-gels, and δ-MnO_2) on three soils containing various levels of Pb ranged between (48–192.0 ug/g). The effectiveness of the various agents in immobilizing Pb followed the descending order: bitumen > cement > slag >Fe-gel > Al-gel > phosphate rock > δ-MnO_2. Cement and phosphate rock fixed Pb mainly in the carbonate form, whereas the slag, bitumen, Fe-gel, Al-gel andδ-MnO_2 fixed the metal mainly in the oxide form. Aikpokpodion et al., (2012) studied the potential of Sokoto rock phosphate for immobilization of Cu and Pb in contaminated soil. They showed that, bioavailable Cu in soil was reduced by 19, 35 and 42% due to application of 20, 40 and 60g phosphate per kg soil, respectively, while, Pb was reduced by 12, 23 and 25%, respectively.The application of 20g, 40g and 60g rock phosphate reduced foliar Cu by 80, 69 and 85% while foliar Pb was reduced by 88, 89 and 77%, respectively. Abdel-Hamid et al., (2012) used the immobilization technique for remediation of lead polluted soils. Two rates (0.5 and 1%) of five immobilizing agents (bentonite, barite, kaolinite, dowex and silica-gel) were tested on soils containing various levels of available Pb (24-77.3 mg kg^{-1}). The DTPA extractable Pb decreased by values ranged between (26.3-70.5)% and (35.4-95.7)% at the tested two rates (0.5 and 1%) ,respectively.

This study was undertaken to evaluate the efficiency of five immobilizing agents (Hydrogel, Polymer, Zeolite, Mud and Geothiteat) at two rates (0.5 and 1%) to remediate Cu, Zn and Pb in contaminated agricultural soils.

MATERIALS AND METHODS

Soil

Three surface soil samples (0-30cm) were collected to represent different sources of Cu , Zn and Pb contamination.

1- Sludged contaminated soils:

El-Gabal El-Asfar area : the soil was settled under irrigation with sewage effluents for more than 75 years

2- Industrial contaminated soil:

Mostorod area : the soil is contaminated with the outputs of mining and smelting .

3- Industrial contaminated soil:

Helwan area : the soil is contaminated with industrial sewage of Iron and steal factories.

The collected soil samples were air-dried and ground to pass through a 2-mm sieve and preserved for the following analysis. Some of physical and chemical characteristics, available and total portions of Cu, Zn and Pb in the studied soils are presented in Table (1).

Immobilizing Agents

Five immobilizing agents were tested in the present study as follows:

(1) Hydrogel: obtained from the Egyptian starch and yeast company - Alexandria . The grain size ranges from 100 to 500 u .The used Hydrogel is characterized by its solubility in both water organic solvents.

(2) Polymer: obtained from Evonik stockhausen Germany . It is insoluble in water and organic solutions; swells to a gel from upon contact with aqueous fluids .

(3) Zeolite: It has the chemical formula $Na_2Al_2Si_3O_{10}$-$2H_2O$. Obtained from El- Ahram Company. It has a high CEC 216 (meq/100g) and the surface area is 31.1 m^2g^{-1}.

(4) Mud : obtained from the Egyptian Public Authority for Mineral Resources. And The major constituents are Quartz, Montmorillonite, with minor content of Kaolinite.

(5) Geothite: was prepared in the laboratory, according to Schwertmann, and Cornell, (1991). The molecular weight is 88.85gm, with the empirical formula: $Fe^{3+}O(OH)$.

Table1. General characteristics , total and available Cu,Zn and Pb contents of the studied soils

Location	El-gabal El-Asfar	Mostorod	Helwan
Source of pollutants	S*	I**	I**
PH (1:2.5)	6.4	7.5	7.6
EC (1:2.5) dS/m	1.83	1.59	5.18
OM%	2.8	1.3	1.4
Total carbonte content%	1.04	1.39	1.0
Sand %	69.8	17.6	33.8
Silt%	7.5	44.9	24.4
Clay%	22.7	37.5	41.8
Textural class	Sandy clay loam	Silty clay loam	Clay
Total Cu (ug/g)	66.9	82.9	60.5
Total Zn (ug/g)	328.0	199.0	150.0
Total Pb (ug/g)	181.0	62.2	59.7
DTPA-Cu (ug/g)	13.9	15.22	8.92
DTPA-Zn (ug/g)	47.30	22.18	4.06
DTPA-Pb (ug/g)	18.3	11.02	18.70

***S:Sewage wastes** ****I:Industrial wastes**

Immobilization Technique

Incubation experiment was conducted to evaluate the tested agents to stabilize Cu, Zn, and Pb in the studied soils. Each soil under study was amendment with each of the five immobilizing agents with two rates (0.5 and 1.0 %). The procedure was as follows: 20 g of each soil were transferred to 100 ml glass bottle, each bottle received 20ml of de-ionized water containing the appropriate amount of immobilizing agent. The treated soils were then dried in an oven, at 40C^o for 48 hrs . then alternatively wet, with 10 ml of de-ionized water. One wetting and one drying formed a cycle. Each soil was subjected to four wetting and drying cycles (for 28 days). Soil moisture content was maintained at 60% by weight of the water holding capacity during the experiment . with water added every two days. At the end of the incubation period, soils were crushed to pass through a 2mm sieve, then analyzed for total , DTPA extractable Cu, Zn and Pb.

Analytical Methods

- DTPA extractable Cu, Zn and Pb were extracted as described by Lindsay and Norvell (1978).
- Total contents of Cu, Zn and Pb were extracted by aqua regia (HCl ,HNO3) according to the method described by Cottenie *et al*. (1982). Concentrations of Cu, Zn and Pb of the extracts were measured using Inductively Coupled Plasma (ICP).
- Mechanical analysis was performed according to the pipette method, organic matter by oxidation with dichromate, and total carbonate content gasometrically using a Collins calcimeter (Sparks, 1996). Soil pH was measured in a 1:2.5 soil: water ratio suspension using a glass electrode (Jackson, 1973). Electrical conductivity (EC) was measured in 1: 2.5 soil: water ratio extracts (Black, 1982).

RESULTS AND DISCUSSION

1. Total and DTPA extractable Cu, Zn and Pb in the studied soils.

The values of the studied heavy metals (Table 1) showed that Zn had the highest values (total or available) in all the studied samples. Data revealed that, soils of El Gabal El Asfar area showed the highest Zn and Pb contents of total and available. The total values were 328 and 181 ppm, Meanwhile the available values available was 47.3 and 18.3 ppm for Zn and Pb, respectively. As for Copper , the highest total amount was found in soils collected from Mostorod (82 ppm), followed in decreasing order by El Gabal El Asfar (66.9 ppm) , and Helwan (60.5 ppm). The available Cu content were 15.2, 13.9 and8.9 ppm for Mostorod, El Gabal El Asfar and Helwan, respectively.

2. In situ immobilization treatments of heavy metals:

In this study ,five immobilizing agents (Goethite, Polymer, Zeolite, Mud and hydrogel) at rates (0. 5 and 1%) were used to remediate Cu , Zn and Pb, in the selected tested polluted soils.

The tested agents showed their ability to reduce the mobile pool of the investigated metals in the studied soils. The magnitude of reduction varied widely according to the type of immobilizing agents and the rate of its addition.

The data presented in Table (2) showed that the effect of the different immobilizing agents on DTPA extractable Cu. The DTPA extractable Cu values after 0.5% application rate (Table 2), ranged between 4.8 – 5.65, 4.5 – 6.1 and 2.85 – 4.05 µg/g for soils of El Gabal El Asfar , Mostorod and Helwan, respectively. The values was 4.2 – 4.9, 4.0 – 5.4 and 1.6 – 2.22 µg/g, for application rate of 1%, respectively. It may be concluded that the addition of 0.5% and 1% application rate for all agents were sufficient to decrease the DTPA extractable Cu by more than 50% compared with the untreated treatment. It was noticed that using 1%. application rate of the studied agents slightly decreased the DTPA extractable Cu compared to the 0.5% application rate.

Table 2 . DTPA extractable Cu (µg/g) in the studied soil samples before and after treating the soils with five immobilizing agents.

Location	Initial concentration, µg/g	Soil Application rates %	DTPA extractable Cu µg/g in soil				
			Hydrogel	Polymer	Zeolite	Mud	Goethite
Al-Gabal Al-Asfar	13.92	0.5	5.17	5.18	5.02	5.65	4.80
		1	4.85	4.90	4.70	4.90	4.20
Mostorod	15.22	0.5	6.10	5.60	5.70	5.50	4.50
		1	5.40	4.00	4.20	4.80	4.00
Helwan	8.92	0.5	2.90	2.85	3.59	3.30	4.05
		1	2.10	1.70	1.60	2.10	2.22

All the tested amendments relatively decreased the mobility of Cu in the soils under study. The tested immobilizing agents varied in their effect on fixing Cu (Fig. 1). In general, and for all the studied soils and tested agents, DTPA extractable Cu decreased by values ranged between 54.6 and 70.4% for soils treated with application rate of 0.5% compared to the untreated soils. On the other hand, application of 1% was rather effective in reducing DTPA Cu by values ranged between 64.5 to 82% compared to the untreated treatment.

Application of Zeolite at rate of 1%, decreased DTPA extractable Cu by values ranged between 66.2 – 82 % compared to the untreated soil. Zeolite appears to be an effective amendment to stabilize soil polluted with lead, copper and zinc, because of the negatively charged alumino-silicate structure within giving the Zeolite high cation exchange capacity (CEC), and have reduced the transfer of these metals from polluted soil into plants (Gadepalle et al., 2007).

Results indicated that application rate of 1% Goethite decreased DTPA extractable Cu by values ranged between 69.8 – 75.1 % compared to the untreated soil samples. The mechanism ascribed to the reductions rate indicating that the goethite surface plays an important role in controlling reduction by forming a monodentate innersphere Cu^{2}+/goethite surface complexes (Rickard, 1974).

The application rate of 1% polymer reduced the DTPA extractable Cu by values ranged between 64.5 – 80.9%. These polymers contain groups, such as carboxyl groups, that are capable of forming bonds with metallic cations, thereby decreasing their bioavailability in soils (De Varennes 2009).

The reduction in DTPA extractable Cu was 64.5 – 76.5% with application rate 1% of either hydrogel or mud. The Hydrogel is a water-swollen, and cross-linked polymeric network, it is a colloidal substance which can form viscous jellylike forms, and characterized by high surface area; therefore, hydrogel can adsorb heavy metals on its surface (Ahmed 2015). While for Mud , the high specific surface area, layered structure, high cation-exchange capacity, etc., have made it excellent, adsorbent materials(Gupta, and Bhattacharyya, 2006).

The reduction of DTPA extractable Cu with 0.5 addition rate was 59.75 – 62.54%, 62.78 – 68%, 59.9 – 67.48%, 59.41 – 63.86%, and 54.59 – 70.43% for Zeolite, Polymer, Hydrogel, Mud and Goethite, respectively.

The tested agents could be arranged according to their efficiency in immobilizing Cu as follows: Zeolite> Polymer >Goethite > Mud> Hydrogel.

The DTPA extractable Zn in the soil treated with 1% (Table 3) ranged between 11.5 – 24.0 , 7.1 – 10.0 and 0.3 – 0.46 µg/g for soils of El Gabal El Asfar , Mostorod and Helwan, respectively. The mean values at the application rate of 0.5% was 18.97 – 25.75, 10.0 – 13.4 and 0.54 – 0.72 µg/g, respectively

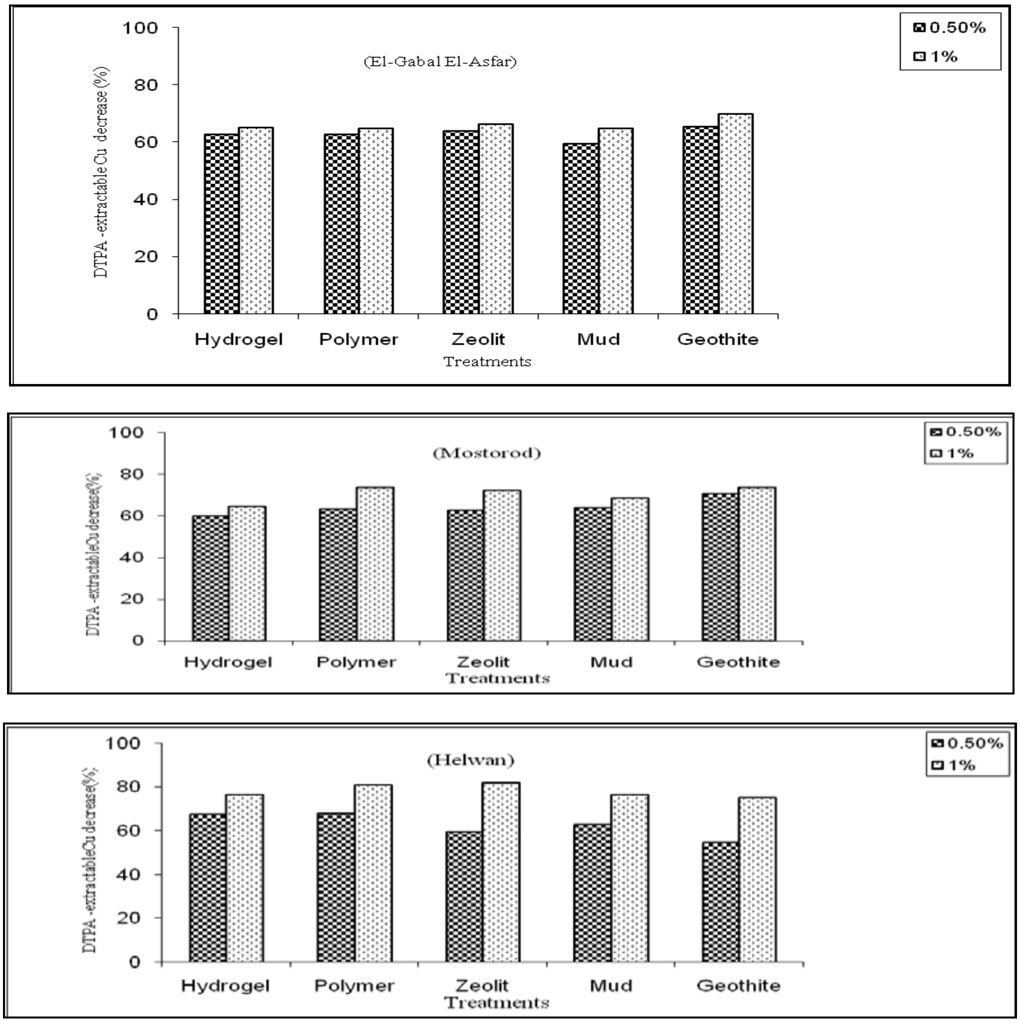

Fig. (1) Effect of different immobilizing agents on DTPA extractable Cu as a percentage of the initial level.

Table 3 . DTPA extractable Zn (µg/g) of the studied soil samples before and after treating the soils with five immobilizing agents.

Location	Initial concentration, µg/g	Soil Application rates %	DTPA extractable Zn µg/g in soil				
			Hydrogel	Polymer	Zeolite	Mud	Goethite
Al-Gabal Al-Asfar	47.34	0.5	18.97	25.75	25.50	18.90	25.20
		1	11.50	20.50	24.0	17.80	22.10
Mostorod	22.18	0.5	10.0	11.20	11.0	13.40	10.30
		1	7.90	10.0	9.10	8.00	7.10
Helwan	4.06	0.5	0.72	0.61	0.54	0.61	0.57
		1	0.46	0.40	0.30	0.40	0.32

All the amendments relatively decreased the mobility of Zn in the soils under study. DTPA extractable Zn decreased by values ranged between 39.6 to 86.7% for soils treated with application rate of 0.5% compared to the control treatment. On the other hand, the application rate of 1% decreased DTPA extractable Zn by values ranged between 49.3 to 92.6 % compared to the untreated soils. The effect of the tested agents at application rate of 0.5% and 1% in reducing DTPA extractable Zn was 82.3 – 86.7% and 85.7 – 92.6% respectively in soils of Helwan. These values were 45.6 – 60, and 49.3 – 75.7%, respectively for soils of El Gabal El Asfar, and 39.5 – 54.9% and 54.9 – 67.9% , respectively for soil of Mostorod.

Results (Fig 2) indicated that application of Zeolite at rate of 1% decreased DTPA extractable Zn by values ranged between 49.3 – 92.6 % compared to the untreated soil. The application rate of 1% Goethite decreased DTPA extractable Zn by values ranged between 53.3 – 92.1 % compared to the untreated soil. The corresponding results of adding polymer, hydrogel and mud were: 54.9 – 90.1% , 64.4 – 88.7% and 62.4 – 90.1%, respectively. The reduction of DTPA extractable Zn with 0.5 application rate was 46.1 – 86.7%, 53.6 – 85.9%, 49.5 – 84.9%, 54.9 – 82.3% and 39.6 – 84.9% compared to the untreated soil for Zeolite, goethite , polymer, hydrogel and mud, respectively.

The tested agents could be arranged according to their efficiency in immobilizing Zn as follows:
Hydrogel > Mud > Goethite> Polymer> Zeolited

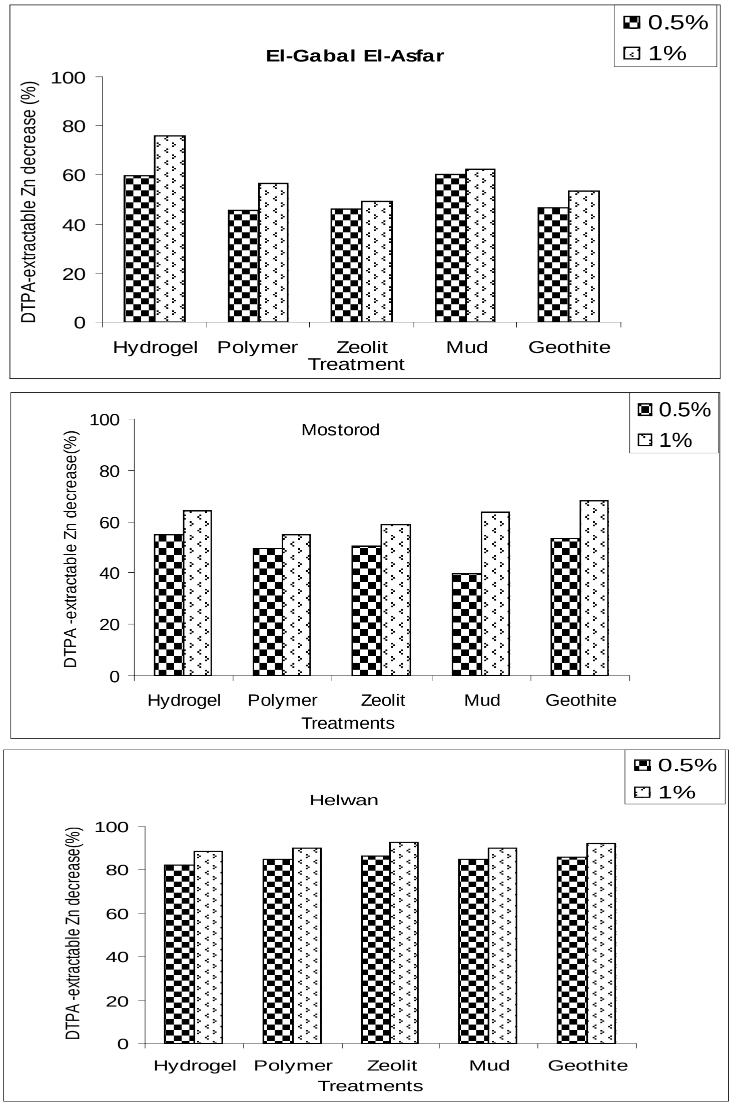

Fig. (2) Effect of different immobilizing agents on DTPA extractable Zn as a percentage of the initial level.

Data presented in Table (4) show DTPA extractable Pb values after treated with immobilizing agents. The DTPA extractable Pb after treated soils with 0.5% ranged between 8.4 – 10.12 , 5.0 – 5.8 and 7.9 – 9.55 µg/g for soils of El Gabal El Asfar , Mostorod and Helwan , respectively. The corresponding values at the rate of 1% was 4.5 – 9.6, 4.1 – 4.5 and 5.0 – 7.3 µg/g , respectively. It was clear that, using 1% application rate of the different agents was more effective in decreasing the DTPA extractable Pb compared to 0.5% application rate. It could be noticed that the tested amendments decreased the mobility of Pb in the studied soils and varied in their effect on fixing Pb. In general, and for all soils and all tested agents, DTPA extractable Pb decreased by values ranged between 44.7 – 57.8% for soils treated with application rate of 0.5% compared to the untreated soils. While the corresponding values at application rate of 1% recorded reduction ranged between 47.5 and 75.4% .

The results (Fig. 3) indicated that application rate of 0.5% of Hydrogel, Polymer, Zeolite, Mud and Goethite reduced DTPA extractable Pb by 51 – 54, 44.7 – 52.4, 47.4 – 57.8, 50.1 – 54 and 8.9 – 51.9%, respectively. While at the application rate of 1%, the reduction percentage were 60.9 – 75.4, 47.5 – 73.2, 57.9 – 72.2 , 57.9 – 73.8 and 55.7 – 70.1, respectively.

Table 4 : DTPA extractable Pb (µg/g) of the soil samples before and after treating the soils with five immobilizing agents .

Location	Initial concentration, µg/g	Soil Application rates %	DTPA extractable Pb µg/g in soil				
			Hydrogel	Polymer	Zeolite	Mud	Goethite
Al-Gabal Al-Asfar	18.30	0.5	8.40	10.12	9.20	8.80	8.80
		1	4.50	9.60	7.70	7.70	8.10
Mostorod	11.02	0.5	5.40	5.70	5.80	5.50	5.00
		1	4.10	4.50	4.40	4.10	4.40
Helwan	18.70	0.5	9.01	8.90	7.90	8.60	9.55
		1	7.30	5.01	5.20	4.90	5.60

The tested agents could be arranged according to their efficiency in immobilizing Pb as follows:

Hydrogel> Mud> Zeolite> Goethite> Polymer

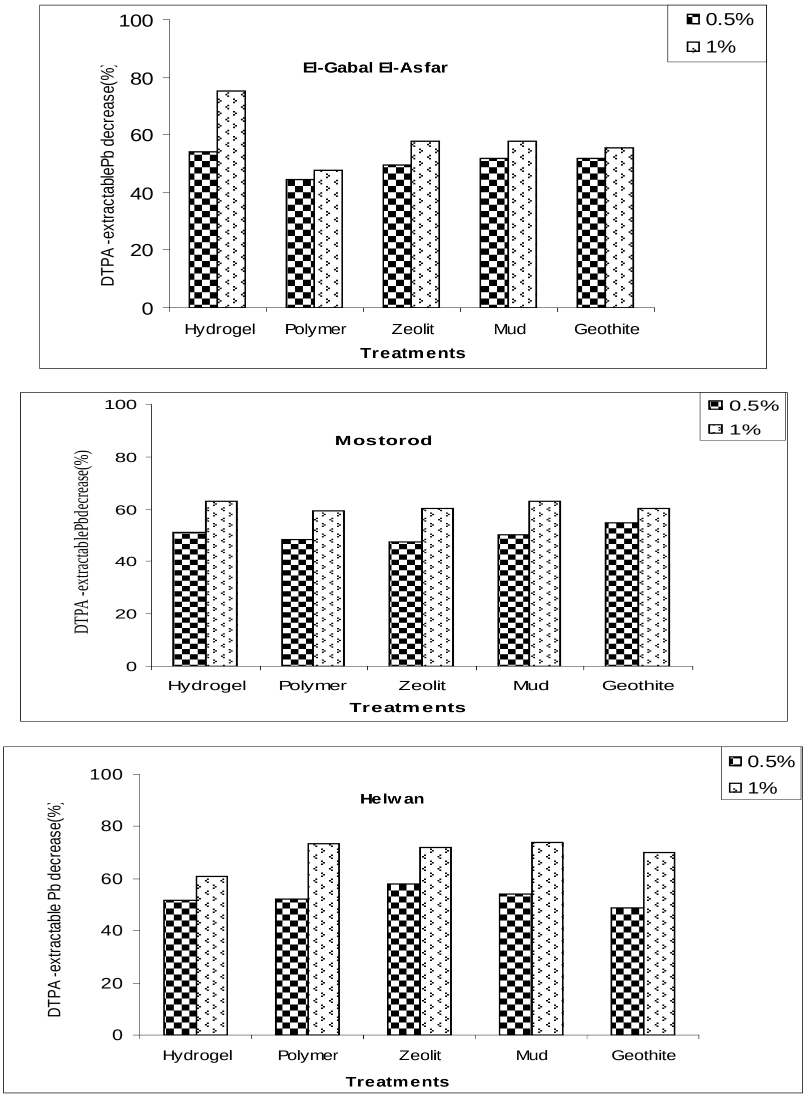

Fig. (3) Effect of different immobilizing agents on DTPA extractable Pb as a percentage of the initial level.

CONCLUSION

Application Hydrogel, Polymer, Zeolite , Mud and Goethite of decreased the mobility of Cu,Zn and Pb in the soils under study. Zeolite appears to be an effective amendment to stabilize soil polluted with lead, copper and zinc . The addition of 0.5% and 1% application rate for all agents were sufficient to decrease the DTPA extractable Cu by more than 50% compared with the untreated treatment. The addition of 1% application rate of the different agents was more effective in decreasing the DTPA extractable Zn and Pb compared to 0.5% application rate. The tested agents could be arranged according to their efficiency in immobilizing the tested metals as follows.

Zeolite> Polymer>Goethite>Mud>Hydrogel for Cu
Hydrogel > Mud> Goethite> Polymer> Zeolite for Zn
Hydrogel> Mud> Zeolite> Goethite> Polymer for Pb

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تثبيت النحاس والزنك والرصاص فى الأراضى الملوثه

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تهدف هذه الدراسه الى استخدام طريقه تثبيت الفلزات لمعالجه الأراضى الملوثه بالنحاس و الزنك و الرصاص و لقد تم إستخدام خمسه مواد مختلفه (الهيدروجيل ، البوليمر، الزيوليت، الطين والجيوثيت) بمعدلين (٠,٥ ، ١.٠ %).و تم استخدام ثلاث انواع من الترب تحتوى على نسب مختلفه من النحاس (٣٢,٩ – ٦٦,٩ ميكروجرام / جرام) ، الزنك (١٥٠ – ٣٢٨ ميكروجرام / جرام) ، الرصاص (٥٩،٧ – ١٨١ ميكروجرام / جرام) . أجريت تجربة تحضين لدراسة تأثير هذه المواد المثبته على المحتوى الميسر فى التربه من النحاس والزنك والرصاص. أدت المواد المثبته الى خفض الكميه الميسره من كل من النحاس والزنك والرصاص المستخلصه بال DTPA . وكان لإضافه معدلين (٠.٥ و ١%) من المواد المثبته كافى لخفض النحاس المستخلص بنسبه اكبر من ٥٠% مقارنه بالتربه الغير معامله. انخفض تركيز الزنك المستخلص بال DTPA بقيم تراوحت بين ٣٩,٦ – ٨٦,٧ % و ٤٩,٣ – ٩٢,٦ % للترب المعامله بكل من ٠,٥ و ١% على التوالى مقارنه بالتربه الغير معامله. إنخفضت قيم الرصاص بحوالى ٤٤,٧ – ٥٧,٨ و ٤٧,٥ – ٧٥,٤ % مقارنه بالتربه الغير معامله عند معدلى الإضافه ٠,٥ و ١% على التوالى. زادت قدرة هذه المواد في تثبيت النحاس والزنك والرصاص بزياده معدل الإضافه و أمكن ترتيب كفاءه المواد كالتالى:

الزيوليت > البوليمر>الجيوثيت > الطين > الهيدروجيل للنحاس

و الهيدروجيل > الطين > الجيوثيت > البوليمر > الزيوليت للزنك

و الهيدروجل > الطين > الزيوليت > الجيوثيت > البوليمر للرصاص

وذلك فى الثلاث ترب المختبره .