DESORPTION OF LEAD, COPPER AND ZINC FROM IRAQI SILTY CONTAMINATED SOIL

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ABSTRACT

This study presents an experimental investigations undertaken to evaluate the effect of two extractant solutions (EDTA- Na₂ and/or citric acid) and different conditions (pH, extractant concentration, and contact time) on the removal efficiency. Iraqi silty soil are studied, the soil was artificially contaminated with lead (Pb), copper (Cu) and zinc (Zn) to achieve higher levels of contamination.

A series of batch experiments were conducted to determine the removal efficiency of lead, copper, and zinc using EDTA- Na₂ and/or citric acid. The influence of solution pH, extraction time, and extractant concentration were studied in order to evaluate the optimum condition for soil washing process.

The feasibility of soil washing method for decontamination of soil was evaluated in laboratory-scale batch experiments. Soil samples containing 500 mg/kg of Pb, Cu, or Zn were subjected to ethelenediaminetetraacitic acid disodium salt (EDTA- Na₂) and/or citric acid (CA) treatment. Soil washing experiments performed on 2.0 g portions of the spiked soil using (0.001, 0.005, 0.01, 0.05, and 0.1M) chelating agents at a solid:liquid ratio of 1:10 showed that extraction efficiency of EDTA-Na₂ was much higher than that of citric acid, and higher concentrations of complexing agent lead to better extraction. Results show that EDTA-Na₂ was better in releasing soil-bound metals from the soil compared to citric acid where 67.13% of lead was extracted using 0.1M of EDTA-Na₂ while only 36.5% was extracted using the same concentration of CA. Up to 51.48% of copper was extracted using EDTA-Na₂ while only 24.86% was extracted using CA. similar results appear for zinc, where 44.2% was extracted using EDTA-Na₂ and 16.73% was extracted using CA.

The highest extraction values of lead can be observed at pH values from 4 to 5, copper at pH= 8 and zinc at pH = 9.

Key words: contaminated soil, heavy metals, soil washing, extraction.

انتشار الرصاص والنحاس والزنك من تربة عراقية غرينية ملوثة

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الخلاصة

هدف الدراسة هو إجراء تحريات عملية لتقييم تأثير محلولين للاستخلاص هما ملح صوديوم اثيلين دايامين ثلاثي حامض الخليك (Na₂EDTA) و حامض الستريك على كفاءة الازالة عند ظروف مختلفة للرقم الهيدروجيني وتركيز المستخلص وزمن التماس. ان الدراسة تمت على تربة غرينية عراقية تم تلويثها بالرصاص والنحاس والزنك لغرض الحصول على مستوى عالي لتلوث في التربة. اجريت سلسلة من تجارب الدفعة على التربة الملوثة لتحقيق كفاءة ازالة الرصاص والنحاس والزنك باستخدام Na₂EDTA و EDTA. اجريت سلسلة من تجارب الدفعة على التربة الملوثة لتحقيق كفاءة ازالة الرصاص والنحاس والزنك باستخدام Na₂EDTA و EDTA. جرحت سلسلة من تجارب الدفعة على التربة الملوثة لتحقيق كفاءة ازالة الرصاص والنحاس والزنك باستخدام Na₂EDTA و EDTA. تمت دراسة تأثير الرقم الهيدروجيني وزمن التماس وتركيز المستخلص لغرض تحديد الظروف المثلى لعملية غسل التربة. ان دراسة جدوى طريقة غسل التربة لازالة الملوثات من التربة تمت من خلال تجارب الدفعة المختبرية. ان نماذج التربة الملوثة التي تحتوي 500 ملغم/كغم رصاص او نحاس او زنك تم معالجتها باستخدام المستخلصين Na₂EDTA و حامض الستريك. ان تجارب غسل التربة اجريت من خلال اخذ 2 غم من التربة الملوثة واستخدام المستخلصات بتراكيز 0.001، 0.005، 0.01، 0.05، 0.1 مولاري و نسبة صلب: سائل تساوي 10:1. اوضحت نتائج التجارب ان استخدام Na₂EDTA أعطى أعلى كفاءة ازالة مقارنة مع حامض الستريك، كذلك ان استخدام تراكيز عالية من المستخلصات تؤدي إلى اعلان كفاءة ازالة. ان كان أفضل في ازالة المعادن الثقيلة (الرصاص والنحاس والزنك) من واوتش كروم التربة، حيث تم ازالة 67.13% من الرصاص باستخدام Na₂EDTA و 36.5% من الرصاص باستخدام حامض الستريك. اما باستعمال Na₂EDTA في ازالة الزنك فان Na₂EDTA أعطى اعلى كفاءة ازالة للرصاص كانت عند رقم هيدروجيني يتراوح بين 4 و 5 والرخص عند رقم هيدروجيني 8 والزنك عند رقم هيدروجيني 9.

1. INTRODUCTION

Heavy metal contamination of soil is one of the most important environmental problems throughout the world (Doumett et al., 2008; Nouri et al., 2006; Wuana et al., 2010). The ability of heavy metals to accumulate and cause toxicity in biological systems - humans, animals, microorganisms and plants has been reported (Nouri, 1980; D’amore et al., 2005; Wuana et al., 2010). As chemical hazards, heavy metals are non-biodegradable and can remain almost indefinitely in the soil environment. However, their availability to biota can change considerably depending on their chemical speciation in the soil. The adequate protection and restoration of the soil ecosystems, therefore, require the characterization and remediation of soils that are contaminated with heavy metals (Nouri et al., 2008; Nwachukwu et al., 2010; Wuana et al., 2010).

The most common heavy metals found in the contaminated sites are Cadmium, chromium, copper, lead, mercury, nickel and zinc. These heavy metals are one of the main sources of environmental pollution and affect the human health, life of animals. The main sources of heavy metals are industrial discharge, energy production, chemicals used in agricultural production, construction, vehicular exhaust and particulate emission, coal and fossil fuel combustion. Metals are relatively immobile in subsurface systems due to precipitation or adsorption reactions. For this reason, remediation activities at metals-contaminated sites mainly focused on the solid-phase sources of metals, i.e., contaminated soils, sludges, wastes, or debris (Evanko, and Dzombak, 1997).

Heavy metals cannot be destroyed by biogeochemical processes and site restoration relies on their removal (Kirpichtchikova et al., 2006; Uwumarongie and Okieimen, 2010). Numerous in-situ and ex-situ soil remediation technologies such as incineration, disposal in landfill, flotation, electro-remediation, bioleaching, phytoremediation and chemical soil washing. Developed for the treatment of heavy metal contaminated soils are based on two principles: immobilization, by increasing the retention of metal on soil or decreasing the mass transfer rate of metal; and mobilization, by the removal of metal from the soil matrix (Kirpichtchikova et al., 2006; Mulligan et al., 2001; Benschoten et al., 1997; Peters, 1999; Irene and Yang, 1999; Khodados et al., 2005; Uwumarongie and Okieimen, 2010).
Soil washing is represented one of the most suitable in-situ (on-site)/ex-situ physical/chemical treatment technologies. Soil washing method has been successfully used for the treatment of soils contaminated with heavy metals, hydrocarbons and semi-volatile organic compounds (SVOCs) (Peters, 1999). Soil washing usually employs different extractants such as acids, bases, chelating agents, electrolytes, oxidizing agents and surfactants (Hong et al., 1995; Schramel et al., 2000; Reddy and Chinthamreddy, 2000; Sun et al., 2001). Chelating agent has a high affinity for heavy metals, and may be used as remedial chemical treatment or in adjunct to another process. The efficiency of chelating agent in metal solubilization is high with less undesirable effects on the soil physicochemical properties. In addition, extraction agents are thought to enhance the bioavailability of metals in soil and thereby influence the iruptake by plants (Kirpichtchikova et al., 2006; Peters, 1999).

2. MATERIALS
2.1 Soils Preparation and Characterization

In the experiments silty soil was used, the soil collected from a non-contaminated abandoned farmland located in Aldujali in Wasit governorate. After collecting soil from surface (0-20 cm), this was air dried at room temperature, sieved by using 2mm sieve to remove stones and large particles, and then thoroughly mixed with hand to ensure uniformity. The samples stored in a plastic bags at room temperature for further experiments.

The physical and chemical characteristics of the soils are shown in Table (1). The particle size distribution was performed by Hydrometer method. Soil water content was determined by direct method (Soil Moisture Percent). Soil pH and Electrical Conductivity were determined with help of HANNA type pH meter after standardizing it with buffer solutions of pH 7.01 and 9.20. Cation exchange capacity (CEC) was determined following Na2-acetate method. Soil organic matter (OM) was determined following Walkley and Black rapid titration method by oxidizing organic carbon in the soil with K2Cr2O7 and back titrating the unutilized K2Cr2O7 against FeSO4. Background concentration of heavy metals was determined by acid digestion (HCl+HNO3). All heavy metal analyses were performed by using Atomic Absorption spectrophotometer (AAS) (novAA 300 (Germany)).

2.2. Contaminants

Since the concentration of heavy metals in soil samples is less than 100 mg kg⁻¹, the soil was spiked with Pb, Cu and Zn to simulate a representative contamination. Lead nitrate, copper nitrate, and zinc nitrate are used as sources of the Pb, Cu and Zn respectively. A solutions of Zn(NO3)2.6H2O, Pb(NO3)2.6H2O and Cu(NO3)2.3H2O respectively (which are manufactured by Scharlau Company/Spain) were prepared and added to the soil. The soil was spiked by adding these chemicals in distilled water to the air dried soil. The contaminated samples were equilibrated and dried by exposure at room temperature for at least two weeks and stored in plastic containers. The contaminant concentrations were measured according to acid digestion procedure followed by analysis with Atomic Absorption spectrophotometer (AAS) (novAA 300(Germany)).
2.3. Extractants

Two chelating agents, ethylenediaminetetraacetic acid (EDTA) [CH\(_2\)N(CH\(_2\)COOH)CH\(_2\)COONa]\(_2\).2H\(_2\)O with purity of 99.9% provided by Panreac, (Espana) and citric acid C\(_6\)H\(_8\)O\(_7\).H\(_2\)O was obtained from Panreac, (Espana) with purity of 99.9%, were selected as chelating agents to examine the solubility of metals in complex forms. The efficiency of synthetic chelant (EDTA) was compared with a natural organic complexing chelant (citric acid). Both EDTA and citric acid were used at concentrations of 0.001M, 0.005M, 0.01M, 0.05M, and 0.1M.

3. METHODS

3.1 Soil Contamination Procedure

The required amount of the Zn(NO\(_3\))\(_2\).12H\(_2\)O or Pb(NO\(_3\))\(_2\).6H\(_2\)O or Cu(NO\(_3\))\(_2\).3H\(_2\)O that would yield the desired concentration was weighed and then dissolved individually in distilled water. The contaminant solution was then added to the 1kg of the dry soil and mixed thoroughly with a stainless steel spatula in polyethylene container.

3.2 Chemical Analysis

Contaminant in different soil sections were extracted by performing acid digestion in accordance with the (Haswell, 1991). Total concentrations of lead, copper and zinc were determined using this extraction procedure. For this procedure, the soil samples were crushed and approximately of 1 g of a representative sample was weighed accurately in a beaker (capacity of 100 ml) and then mixed with 15 ml of concentrated hydrochloric acid (HCl) and 5 ml of concentrated nitric acid (HNO\(_3\)), the mixture was then heated in the heating sandy bath until the brown evaporation was disappeared and the sample arrived to dry state, this step takes about (45-60) minutes. Then cooling the beaker to lab. temperature and then add 5 ml of concentrated hydrochloric acid (HCl) and then heated in the heating sandy bath, this step takes about (5-10) minutes. Then cooling the beaker and add 5 ml of concentrated hydrochloric acid and 50 ml of heated distilled water to wash the sides of beaker from remains dissolved sample. After that heating the mixture to the boiling points to (2-3) minutes. Then filtrate the sample with filtration paper No. 42, and then keep it in volumetric flask capacity of 100 ml, then, washing the precipitation with distilled water and addition of the previous washed water to filtration and complete the size to 100 ml. Finally, The metal concentration was determined by Atomic Absorption Spectrophotometer (AAS).

3.3 Soil Washing Procedure

Batch extractions of heavy metal contaminants using a various extractant concentrations and liquid/solid ratio of 10:1 were conducted in 250 mL conical flask. The flask contained 2 g of soil and 20 mL of EDTA or citric acid was agitated using an orbital shaker at a speed of 200 rpm at room temperature for 1 hr. The suspensions were centrifuged at 3000 rpm for 10 min and the supernatants were then filtered through 0.45μm Whatman filter paper for heavy metal analysis. The concentrations of metals were measured by flame atomic absorption spectrometry (AAS).

In the experiment different operating variables for removal of heavy metals from soil using EDTA or citric acid, including the different extractant concentration, solution pH and operation time. In experiments, five different concentrations (0.001,0.005,0.01, 0.05, and 0.1) of Na\(_2\)EDTA or citric
acid were chosen. The pH from 4-9 was adjusted with diluted HNO₃ and NaOH. The kinetic study was performed for 1, 2, 3, 4, 5 h. Finally, the removal of each contaminant was calculated using the following equation (Lim et al., 2005; Reddy and Chinthamreddy, 2000; Gzar et al., 2014):

\[
\text{Contaminant removal} = \frac{\text{contaminant mass in supernatant} (C_L V_L)}{\text{initial contaminant mass in soil} (C_s M_s)} \times 100\%
\]

where \(C_L\) and \(C_s\) are the concentration of heavy metals in supernatant (in mg/L) and soil (in mg/kg), respectively. \(V_L\) is the volume of supernatant (in L), and \(M_s\) is the dry mass of the soil (in kg).

4. RESULTS AND DISCUSSION

4.1. Extraction with EDTA

4.1.1. Effect of pH

Solution pH is an important factor determining the efficiency of soil washing because it can influence the soil retention of metals by adsorption. Experiments were performed in pH of 4, 5, 7, 8, and 9. Nitric acid (HNO₃) and sodium hydroxide (NaOH) were used for pH adjustment.

As for most metals, the amount of extracted Pb increase with lowering pH values as shown in figure (1). The highest extraction of lead can be observed at pH values from 4 to 5. At pH = 4, the removal efficiency of lead was 69.66%.

For copper, at the lower pH values the removal efficiencies follow the predictable trend but unexpected increase occurs at pH 8 to 9. The maximum removal efficiencies for copper was 58.24% and this percentage achieved at pH = 8. At higher pH values a significant decrease occurs abruptly.

The curves of the extraction of zinc as a function of pH are not smooth. Additionally some results do not seem to be logical. The general trend is the decrease of extracted zinc at pH = 5 but increase after that with increasing pH value. For lower pH value the removal efficiency of Zn was 46.6% while it reaches to 60.2 at pH = 9.

The results in figure (1) is accepted with Mohanty and Mahindrakar (2011), which used EDTA to remove lead and cadmium from soil and the maximum removal of lead occurred at pH = 4. The results also accepted with Mahvi et. al. (2005), which found that the recovery or desorption of lead from silt- clay- loam soil was generally greatest under acidic condition and decrease modestly as the pH become alkaline, accurately, they found that the remediation efficiency for lead in pH = 3 was approximately 10% more than in pH =9, but they observed a decrease of 4% in removal efficiency of zinc at pH =3 and that is may be due to distribution of metals in the different solid phase of the contaminated soil. Niinae et. al. (2008) report that the removal of lead with EDTA is very high in the pH range from 4 to 10. However, it is considered that acid also contributes to the extraction of lead at pH= 4.

4.1.2. Kinetics of Extraction

Extraction time plays a very important role in soil washing. In order to comprehend the washing process and determined the optimum contact time for contaminants removal, a kinetic study was performed at different contact times (1, 2, 3, 4, and 5 hr) and different concentration of Na₂EDTA (0.001, 0.005, 0.01, 0.05, and 0.1M) and the result shown in figure (2).
The kinetic of lead extraction occurs within the first two hours then the curves stay nearly constant. At highest concentration, 63% of lead was extracted within the first two hours while increasing the reaction time for five hours will rise the removal efficiency of lead to 67.13% only.

Copper and zinc exhibit the same behavior observed for lead. The removal efficiencies of copper varied from 47.92% after 2 hours of extraction to 51.48% after 5 hours. The removal efficiencies of zinc varied from 58.01% after 2 hours of extraction to 60.2% after 5 hours at pH =9.

The results of the present study indicated that a reaction time of 2 hr was considered suitable since the curves illustrate that no appreciable amount of metals was extracted beyond this time and this is agreed with Mahvi et. al. (2005) which reported that the vacillation of remediation process results usually occurs in initial hours and then the efficiency will follow a constant rate, therefore the optimum time period which is used as a bench mark for comparison is 2 hr. Bermond and Ghestem (2001) reported a rapid initial release of lead and cadmium from contaminated soil into the EDTA extraction solution. Khalkhaliani et al. (2006) also reported that 2 hr is the suitable for metal extraction from contaminated soil.

4.1.3. Effect of Extractant Concentration

Chelating agents modify metal concentration in soil solution by forming various soluble complexes, thus enhance metal removal (Reddy and Chinthamreddy 2000). Different concentrations (0.001, 0.005, 0.01, 0.05, 0.1M) of EDTA was used in the present study. The solid- liquid ratio was 1:10 with shaking speed of 200 rpm and shaking time (1, 2, 3, 4, and 5 hr). No pH adjustment was made in these experiments.

In figure (3), no appreciable extraction observed for lead at the lower concentrations of EDTA-Na2. EDTA-Na2 solution of 0.1M extract up to 67% of lead from the soil after 5 hr of extraction.

For copper and zinc, no extraction occurs when low concentrations of EDTA-Na2 (0.001 and 0.005) M were used. At concentrations of (0.01 and 0.05) M, no appreciable removal appear for these metals. For copper, the results show that at concentration of 0.1M 51.48% was extracted after 5 hr of extraction. 44.2% of zinc was extracted using 0.1M of extractant shaked for 5 hr.

According to these results the maximum and minimum metal removal efficiencies achieved with 0.1M and 0.005M of EDTA-Na2 and this is agreed with Mahvi et. al (2005) and khalkhaliani et. al. (2006) reported that there was a cardinal effect of EDTA on lead removal; meanwhile the increase of the EDTA concentration in zinc has not a considerable effect on the remediation efficiency.

4.2 Extraction with Citric Acid

Citric acid was selected as a natural organic complexing chelant to examine the solubility of metals in complex form. Batch extraction experiments were conducted using citric acid solution at various concentrations (0.001, 0.005, 0.01, 0.05, and 0.1) M and contact times to determine appropriate range of concentration and dosage of the washing solution to achieve high heavy metals extraction efficiencies.

4.2.1. Effect of pH

To examine the effect of pH value on the efficiency of extraction, 0.1M of CA was added to the soil. Experiments were performed in pH of 4, 5, 7, 8, and 9. Nitric acid (HNO3) and sodium hydroxide (NaOH) were used for pH adjustment.
Copper and zinc follow the predictable trend (figure 4). The highest removals occur at the lower pH values. Up to 38.5% of lead was extracted at pH = 4 after 5 hours of extraction. The amount of extracted Pb decreases with increasing pH value steadily until reaches to 9.04% only at pH = 9.

Copper and zinc exhibit similar behavior. 26.6% of copper extracted at pH = 4 while only 10.1% extracted at pH = 9. Removal efficiencies of zinc varies from 20.9% at pH 4 to 8.33% at pH = 9. The drop in extraction for copper and zinc was about 30.5% and 31.2% respectively when pH value rises to 9.

4.2.2. Kinetics of Extraction

Figure (5) show that the significant extraction also occur within the first two hours and the increase in the removal efficiency after that time is not impressive. No removals occur for all metals at all times of extraction when 0.001M of CA was used (figure 5a). 33.15%, 24.1%, and 13.81%, of lead, copper, and zinc respectively were extracted when 0.1M of CA was used with contact time of 2 hours (figure 5e), but after 5 hours of extraction only 36.5%, 24.86%, and 16.73% of lead, copper, and zinc respectively were extracted using the same concentration of CA.

4.2.3. Effect of Extractant Concentration

EDTA and citric acid (CA) are chelating agents modify metal concentration in soil solution by forming various soluble complexes. Different concentrations (0.001, 0.005, 0.01, 0.05, 0.1M) of CA was used in this study with solid-liquid ratio of 1:10 with shaking speed of 200 rpm for (1, 2, 3, 4, and 5 hr). No pH adjustment was made in these experiments.

The results showed that the soil did not respond to the treatment with the lowest concentration of citric acid (0.001M) (Figure 6). The maximum removal efficiencies achieved at the highest concentration (0.1M) at contact time 5 hours, where 36.5%, 24.86%, and 16.73% of lead, copper, and zinc respectively were extracted the soil (Figure 6e).

4.3. Comparison between EDTA and Citric Acid Extraction

EDTA could form strong complexes with heavy metals (Manouchehri et al., 2006), however, citric acid is relatively a weaker complexant than EDTA. A comparison between EDTA and CA for removing of heavy metals, as a function of extractant concentration and contact time are shown in figures (7) and (8) respectively.

Results of the present study show that EDTA was better in releasing soil-bound metals from the soil compared to citric acid where 67.13% of lead was extracted using 0.1M of EDTA while only 36.5% was extracted using the same concentration of CA (Figure 7a). Up to 51.48% of copper was extracted using EDTA while only 24.86% was extracted using CA (Figure 7b). Similar results appear for zinc, where 44.2% was extracted using EDTA while 16.73% was extracted using CA (Figure 7c).

The ability of EDTA to enhance the release of metals from insoluble or sparingly soluble compounds compared to other chelating agents has been attributed to its higher binding capacity for metals as observed in other studies (Blaylock et al., 1997; Huang et al., 1997; Wu et al., 1999).

A study by Nascimento et al. (2006) investigated the release of Pb in soil solution after application of chelating agents viz. EDTA, DTPA, oxalic acid, citric acid, vanillic acid and gallic acid. Authors concluded that EDTA was one of the most efficient chelating agents as it significantly increased the concentration of Pb in soil solution after 24 h of application.
5. CONCLUSIONS

1. Results of the present study show that EDTA was better in releasing soil-bound metals from the silty soil compared to citric acid where 67.13% of lead was extracted using 0.1M of EDTA while only 36.5% was extracted using the same concentration of CA. Up to 51.48% of copper was extracted using EDTA while only 24.86% was extracted using CA. similar results appear for zinc, where 44.2% was extracted using EDTA while 16.73% was extracted using CA.

2. The highest extraction of lead can be observed at pH values from 4 to 5.

3. The maximum removal efficiency for copper was 58.24% and this percentage achieved at pH =8.

4. The maximum removal efficiency for zinc achieved at pH =9.

5. The results of the present study indicated that a reaction time of 2 hr was considered suitable since the curves illustrate that no appreciable amount of metals was extracted beyond this time.

6. The results showed that the silty soil did not respond to the treatment with the lowest concentration of citric acid (0.001M), while the maximum removal efficiencies achieved at the highest concentration (0.1M) at contact time 5 hours.

7. Compared with citric acid, EDTA caused more extraction of metals at all the levels of application.

Overall the results from this study demonstrated that EDTA could potentially be used as an amendment to enhance metals mobility in soils and thus its availability to plants for removal from contaminated soils.

REFERENCES


### Table 1: Composition and properties of the studied soil.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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<tr>
<td>Silt (%)</td>
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<td>Clay (%)</td>
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<td>Soil classification</td>
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Figure (1): Effect of pH on the removal efficiency of lead, copper, and Zinc using 0.1M of EDTA at different contact times.
Figure (2): Effect of contact time on the removal efficiency of lead, copper, and Zinc using EDTA at different concentrations.
Figure (3): Effect of concentration on the removal efficiency of lead, copper, and Zinc using EDTA at different contact time.
Figure (4): Effect of pH on the removal efficiency of lead, copper, and Zinc using 0.1M of CA at different contact time.
Figure (5): Effect of contact time on the removal efficiency of lead, copper, and zinc using CA at different concentrations.
Figure (6): Effect of concentration on the removal efficiency of lead, copper, and zinc using CA at different contact time.
Figure (7): comparison between EDTA and CA for removing of heavy metals as a function of extractant concentration.

Figure (8): comparison between EDTA and CA for removing of heavy metals as a function of contact time.