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REMEDIATION OF LEAD-CONTAMINATED SOILS

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by

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Remediation of Lead-Contaminated Soils*

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ABSTRACT

Excavation and transport of soil contaminated with heavy metals has generally been the standard remediation technique for treatment of heavy-metal-contaminated soils. This approach is not a permanent solution; moreover, off-site shipment and disposal of contaminated soil involves high expense, liability, and appropriate regulatory approval. Recently, a number of other techniques have been investigated for treating such contaminated sites, including flotation, solidification/stabilization, vitrification, and chemical extraction. This paper reports the results of a laboratory investigation determining the efficiency of using chelating agents to extract lead from contaminated soils. Lead concentrations in the soils ranged from 500 to 10,000 mg/kg. Ethylenediaminetetraacetic acid (EDTA) and nitrilotriacetic acid (NTA) were examined for their potential extractive capabilities. Concentrations of the chelating agents ranged from 0.01 to 0.10 M. The pH of the suspensions in which the extractions were performed ranged from 4 to 12. Results showed that the removal of lead using NTA and water was pH-dependent, whereas the removal of lead using EDTA was pH-insensitive. Maximum removals of lead were 68.7%, 19.1%, and 7.3% using EDTA, NTA, and water, respectively (as compared with initial Pb concentrations).

INTRODUCTION

Heavy-metal contamination of subsurface soils and groundwaters results from a number of activities, including application of industrial waste, application of fertilizers and pesticides, mining operations, smelting operations, automobile battery production, metal plating/metal finishing operations, vehicle emissions, and fly-ash from combustion/incineration processes. Ranges of heavy metal concentrations reported in the technical literature for soils contaminated with various heavy metals are summarized in Table 1.

Metal concentrations in groundwater are largely governed by interactions with surrounding soils and geological materials. Many different mechanisms influence the partitioning of metals between the solid and solution phases (thereby affecting the leachability of metals from contaminated soils), such as dissolution and precipitation, sorption and exchange, complexation, and biological fixation [1]. The major effect of complexation is the dramatic increase in solubility of heavy metal ions, especially for such strong complexing agents as ethylenediaminetetraacetic acid (EDTA) and nitrilotriacetic acid (NTA) [2-5]. Due to this increased solubility of metal ions in solution, complexing agents offer the potential to be effective extractants of heavy metals from contaminated soil.

BACKGROUND

Excavation and transport of heavy-metal-contaminated soil has been the standard remedial technique. This technique can hardly be viewed as a permanent solution. Moreover, off-site shipment and disposal of the contaminated soil involves high expense, liability, and appropriate regulatory approval. Furthermore, recent U.S. Environmental Protection Agency regulations require pretreatment prior to landfilling [15]. This has resulted in increased interest in technologies to treat contaminated soils either on-site or *in situ* [1].

For soils contaminated with organic pollutants, a number of techniques can be considered for remediation of a particular site, including thermal treatment, steam and air stripping, microbial degradation, and chemical oxidation. Fewer treatment techniques exist for remediation of metal-laden soils. Metals can be removed by either flotation or extraction. Process parameters affecting extraction technologies for cleaning up soils have been summarized in ref. 16. Migration of metals can also be minimized by solidifying or vitrifying the soil and fixing the metals in a nonleachable form. In solidification processes, lime, fly-ash, cement-kiln dust, or other additives are added to bind the soil into a cement-like mass and immobilize the metallic compounds. In vitrification processes, the soil is formed into a glassy matrix by applying

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current across embedded electrodes. Solidification is very expensive because the waste must be thoroughly characterized to determine compatibility with the specific treatment processes. Many existing technologies result in a solid with less-than-optimal long-term stability. Solidification/stabilization also results in an increase in the waste volume. Following remediation, site reuse is limited and long-term monitoring is generally required. For these reasons, solidification/stabilization is generally limited to radioactive or highly toxic wastes [17].

Pickering [18] identified four approaches whereby metals could be mobilized in soils: (1) changing the acidity, (2) changing the system ionic strength, (3) changing the oxidation/reduction (redox) potential, and (4) forming complexes. In the last technique, addition of complexing ligands can convert solid-bound heavy metal ions into soluble metal complexes. The effectiveness of complexing ligands in promoting metal release depends on the strength of bonding to the soil surface, the stability and adsorbability of the complexes formed, and the pH of the suspension [1]. From an application viewpoint, the type and concentration of the complexing ligand and the system pH are the operational parameters that can be controlled.

The ability of chelating agents to form stable metal complexes makes materials such as ethylenediaminetetraacetic acid (EDTA) and nitrilotriacetic acid (NTA) promising extractants for treatment of heavy metal-polluted soils [19]. Elliott and Peters [1] have noted that although complexation is the major mechanism responsible for the metal solubilization, the overall release process depends on the proton concentration and the system ionic strength. Because hydrous oxides of iron and manganese can coprecipitate and adsorb heavy metals, they are believed to play an important role in the fixation of heavy metals in polluted soils [20]. Their dissolution under reducing conditions may weaken the solid-heavy metal bond and thereby promote solubilization of the metal ions.

Elliott and Peters [1] noted that there are five major considerations in the selection of complexing agents for soil remediation: (1) Reagents should be able to form highly stable complexes over a wide pH range at a 1:1 ligand-to-metal molar ratio. (2) Biodegradability of the complexing agents and metal complexes should be low (especially if the complexing agent is to be recycled for reuse in the process). (3) The metal complexes that are formed should be nonadsorbable on soil surfaces. (4) The chelating agent should have low toxicity and potential for environmental harm. (5) The reagents should be cost-effective. Elliott and Peters note that, although no compounds ideally satisfy all these criteria, there are several aminocarboxylic acids which form remarkably stable complexes with numerous metal ions.

Extraction of heavy metals from contaminated soils can be performed either using *in situ* techniques or on-site extraction (following excavation). For the case of *in situ* soil flooding, the aqueous extractant is allowed to percolate through the soil to promote metal mobilization. For the case of on-site extraction following excavation, the operation can be performed on a batch basis, a semi-batch basis, or continuously. The contaminated soil is pretreated for size reduction and classification and contacted with the extractant. Then the soil is separated from the spent extractant. The eluant is recycled to decomplex and precipitate the metals from solution or, in the alternative, treated using electrodeposition techniques to recover the metals. The major components for a general soil-washing process are presented schematically in Elliott and Peters [1].

GOALS AND OBJECTIVES

The primary goals of this project were to determine and compare the performance of several chelating agent for their ability to extract lead from contaminated soil. The objective of this study was to determine the removal efficiencies of the various chelating agents (and water alone) as functions of solution pH and chelating agent concentration.

EXPERIMENTAL PROCEDURE

The experiments were performed using a batch shaker technique following the procedures summarized below.

Soil from a previous study was prepared by grinding with a ceramic mortar and pestle until the soil could pass through an 850- μm sieve (ASTM mesh #20). The characteristics of this soil are summarized in Table 2. Four batches (~200 g each) of uncontaminated soil were then spiked, or artificially contaminated, by soaking the soil with a solution of lead nitrate and then left to air dry. Each batch of soil was "contaminated" with a different concentration of lead nitrate (nominally 500, 1000, 5000, and 10,000 mg/kg soil). The contaminated soils were stored in glass jars (with screw-on covers) for approximately three months to age the soil prior to conducting the screening studies. The nominal experimental conditions are listed in Table 3.

Soil was weighed using a top loading balance into 5-gm portions and placed in plastic, lidded shaker containers. To these containers, 45-mL of one of the following solutions were added: (1) 0.01 M, 0.05 M, or 0.1 M EDTA- Na_2 (disodium salt); (2) 0.01 M, 0.05 M, or 0.1 M NTA- Na_3 (trisodium salt); (3) 0.01 M, 0.05 M, or 0.1 M hydrochloric acid — HCl; or

(4) deionized water. These conditions were employed to create a matrix of samples; each combination of lead-spiked soil and type and concentration of chelating agent was tested. Blanks of uncontaminated soil were also combined with each concentration of the various solutions of chelating agents and deionized water to detect any interferences in the analyses. Replicate experiments were performed in 25% of the experiments conducted.

The soil samples to which the extractant had been added were shaken for a period of nominally 3 hours at a low setting on an Eberbach shaker table. This time requirement was determined to be sufficient for chemical equilibrium to be achieved from preliminary experiments where the residual concentration of lead was monitored as a function of time on the batch shaker table. Following this agitation, the samples were centrifuged in plastic Nalgene centrifuge tubes equipped with snap-on caps, filtered using No. 42 Whatman filter paper, stored in glass vials, and maintained at $\text{pH} < 2$ (using HNO_3) prior to atomic absorption spectroscopy (AAS) analysis. The spectrophotometer was calibrated using AAS lead standards, and the analyses were performed in accordance with the procedures described in *Standard Methods* [21].

Data collected during the experiments included: operating temperature ($\sim 23.5^\circ\text{C}$), extractant type and concentration, lead concentration on the soil (before and after treatment), lead concentration in the extractant after treatment, pH of the solution before and after treatment, and batch shaking time.

RESULTS AND DISCUSSION

1. Preliminary Experiments

Prior to performing the more comprehensive experiments, preliminary experiments were conducted using soil which had been contaminated with lead at a level of 1000 mg/kg soil. The purpose of these preliminary experiments was to determine the contact time required to reach pseudoequilibrium conditions for the particular contaminated soil. The extractants studied included EDTA, NTA, and HCl (all at a concentration of 0.2 M). The pH quickly reached a constant value for each system, with the pH corresponding to 4.2, 10.9, and 5.75 for the extractants of HCl, NTA, and EDTA, respectively. The approximate times required for the HCl, NTA, and EDTA systems to reach pseudoequilibrium conditions were 0.5, 3, and 2 hours, respectively. To facilitate the treatment with chelating agents, a contact time of 3.0 hours was selected to provide uniform treatment conditions. Based upon the very low solubilization of lead using hydrochloric acid as the extractant, this system was not studied further.

2. Batch Extraction Experiments

A series of batch extractions were conducted using water, NTA, and EDTA as the extractants. The lead concentrations on the soil were 500, 1000, 5000, and 10,000 mg/kg soil. The concentrations of EDTA and NTA ranged from 0.01 to 0.1 M. The extractions were performed over the nominal pH range of 4 to 12.

Figure 1 presents the results for the extraction of lead using water alone for the 10,000 mg Pb/kg soil. The results for the other lead concentrations behaved in a similar fashion. The concentration of lead solubilized by the water extraction was pH sensitive; a significant increase in lead concentration in solution is observed for $\text{pH} < 4$. Generally, the amount of lead extracted from the soil by water alone was minimal — less than 40 mg/kg soil. The maximum concentration extracted using water alone represented only 7.3% of the lead being solubilized.

Figure 2 presents the results for the extraction of lead from the 10,000 mg/kg soil using 0.1 M NTA. Again the results for the other lead concentrations behaved in a similar manner. The amount of lead extracted from the soil using water as the extractant had been as high as 730 mg/kg soil. However, the lead extracted using NTA as the extractant was significantly higher, with the amount of lead extracted as high as 1910 mg/kg soil at the lower pH range. The lead concentration in solution is observed to be much more pH sensitive than for the water system. Fairly small amounts of lead were extracted (indicating little lead solubilization) over the pH range of 6 to 9. Significant increases are observed on both sides of this pH range. The maximum amount of lead solubilized over the pH range of 4 to 12 was 19.1%.

Figure 3 presents the analogous set of data for the extraction of lead from the 10,000 mg/kg soil using 0.1 M EDTA. Once again, the results for the other lead concentrations behaved in a similar manner. In striking contrast to the other two extraction systems (employing water and NTA), which exhibited a significant pH dependency, the EDTA extraction system was strongly pH insensitive. The amount of lead extracted from the soil ranged from 5730 to 6420 mg/kg soil over the pH range of 4.9 to 11.3, corresponding to lead removals ranging from 57.3% to 64.2%.

Figure 4 compares these three extractants in terms of the amount of lead extracted from the soil (expressed as a percentage of the initial lead concentration of 10,000 mg/kg). The NTA extraction shows the largest pH dependency, whereas the EDTA extractant is strongly pH insensitive. For the EDTA extraction system, the removal of lead remained relatively constant at approximately 61% over the entire pH range.

Figure 5 shows the results of the lead extraction removal efficiency plotted a function of pH for various initial lead concentrations on the soil. For these experiments, the EDTA concentration was 0.10 M. The results indicate that the removal efficiency of lead using EDTA as the extractant was fairly insensitive to the initial lead concentration applied to the soil over the range of 500 to 10,000 mg/kg soil. The lead removal efficiency ranged from a low of 56.2% to a high of 68.7%.

The effect of varying the EDTA concentration on the removal of lead from contaminated soil is shown in Figure 6. The pH for this EDTA extractant ranged from 5.9 to 6.9. The removal efficiency is shown for four separate lead concentrations on the soil, ranging from 500 to 10,000 mg/kg soil. The removal efficiency was greatest for the case of a lead contamination level of 5000 mg/kg, although all the removal efficiencies were fairly constant, in the range of 54% to 69%.

Figure 7 shows the effect of varying the EDTA concentration for treating the four lead contamination levels (ranging from 500 to 10,000 mg/kg soil). The EDTA concentration ranged from 0.01 to 0.1 M and the solution pH ranged from 5.9 to 6.9. As shown in Figure 7, nearly all the EDTA concentrations studied resulted in removal efficiencies in the 60% range.

SUMMARY AND CONCLUSIONS

The results from other research studies [13,14] indicate that removal of lead from contaminated soil can exceed 80%. This study, which involved extraction of lead from a soil with a very high clay and silt content (~71%), indicated that lead removal exceeds 60% when using EDTA concentrations in the range of 0.01 to 0.1 M for lead concentrations on the soil ranging from 500 to 10,000 mg/kg. Extraction of lead using water alone removed a maximum of 7.3% for pH ~4. The removal of lead using water and NTA as extractants were both pH dependent, whereas the removal of lead using EDTA was strongly pH insensitive. When using EDTA, the removal of lead ranged from 54% to 69% over the entire range of initial lead concentrations in the soil for the pH range of 4.9 to 11.3. The initial lead content had very little effect on the removal efficiency of lead for the EDTA system (for initial lead concentrations in the range of 500 to 10,000 mg/kg soil). The applied EDTA concentration over the range of 0.01 to 0.1 M also had little effect on the removal efficiency of lead from the soil.

Extraction of lead with EDTA was rapid, reaching equilibrium within a contact time of 1.0 hour. Extraction of lead with NTA was slower; a contact time of approximately 3.0 hours was required to reach equilibrium. The order of lead removal efficiency for the various extractants was EDTA >> NTA >> water. The maximum lead removals observed for this high clay and silt soil were 68.7%, 19.1%, and 7.3%, for the extractants EDTA, NTA, and water, respectively.

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Table 1 Reported Heavy Metal Concentrations

Heavy Metal	Site Type	Reported Concentration Range (ppm)	Reference
Zn	Smelting	26,000 - 80,000	[6]
Cd	Smelting	900 - 1,500	[6]
Cr	Chromium Production	500 - 70,000	[7,8]
Pb	Battery Reclamation		
	Soil	2 - 135,000	[9,10]
	On-Site Sediment	2.16 - 42,700	[9,10]
	Surface Water	0 - 140 mg/L	[11]
Pb	Battery Recycling		
	Soil	210 - 75,950 mg/kg	[12]
	Soil	0 - 211,300 mg/kg	[13,14]

Table 2 Analysis of the Uncontaminated Soil Employed in Study

pH	7.75
Electrical Conductivity (mmho/cm)	5.46
Cations (meq/L)	
Na ⁺	20.26
K ⁺	0.62
Ca ⁺⁺	37.88
Mg ⁺⁺	24.32
Anions (ppm)	
NO ₃ -N (1:5 H ₂ O extrac.)	2.7
P (mg/L)	10.3
Total Kjeldahl Nitrogen (TKN) (mg/L)	471
Fe (DTPA) (mg/L)	18.69
Sodium Absorption Ratio (SAR)	3.63
Calculated ESP	3.9
Percent H ₂ O at saturation	75.63
Cation Exchange Capacity (CEC) (meq/100 g)	13.27
Percent Organic Matter (OM)	2.02
Texture	Clay Loam
Size Fractions (%)	
Sand	29.2
Silt	35.0
Clay	35.8

Table 3 Nominal Experimental Conditions

Parameter	Experimental Range
Heavy Metal (Lead) Concentration (mg/kg soil)	500 - 10,000
Extractant Type and Concentration (M)	
EDTA	0.01 - 0.10
NTA	0.01 - 0.10
HCl	0.01 - 0.10
Solution pH	4 - 12
Operating Temperature (°C)	23.5
Preliminary Contact Time (h)	0.5 - 6.0

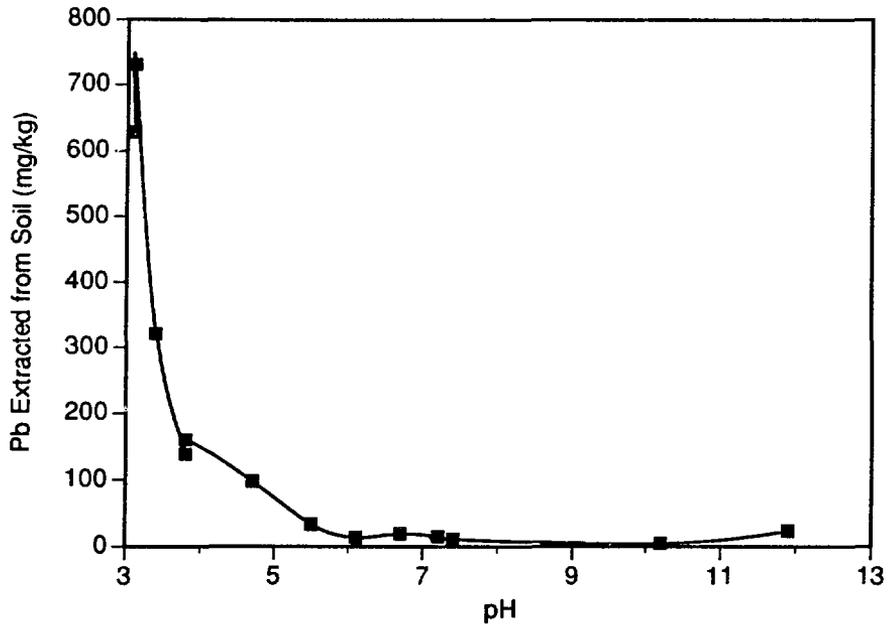


Figure 1 Residual concentration of lead after extraction with water for a soil sample contaminated with 10,000 mg lead/ kg soil as a function of pH

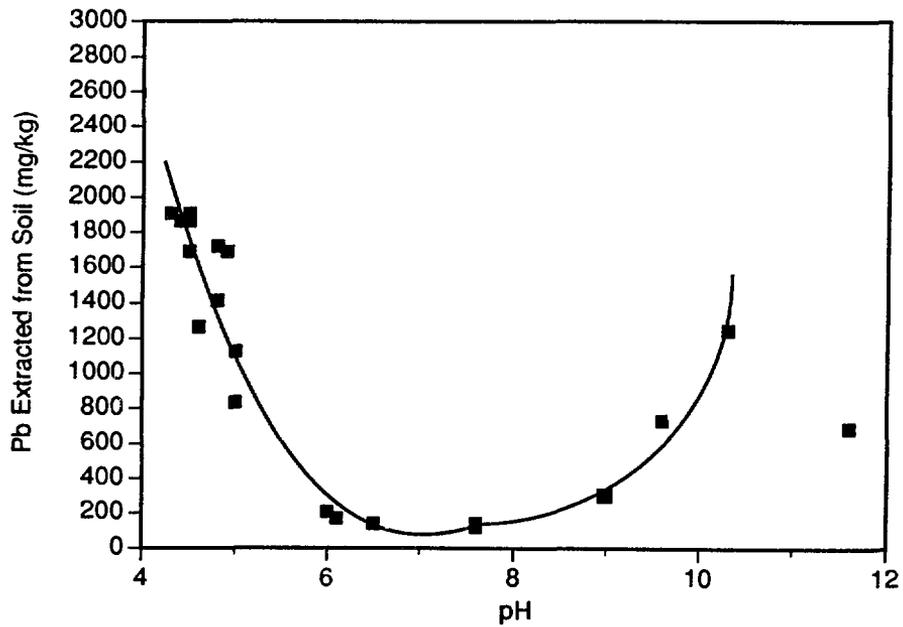


Figure 2 Residual concentration of lead after extraction with 0.1 M NTA for a soil sample contaminated with 10,000 mg lead/ kg soil as a function of pH

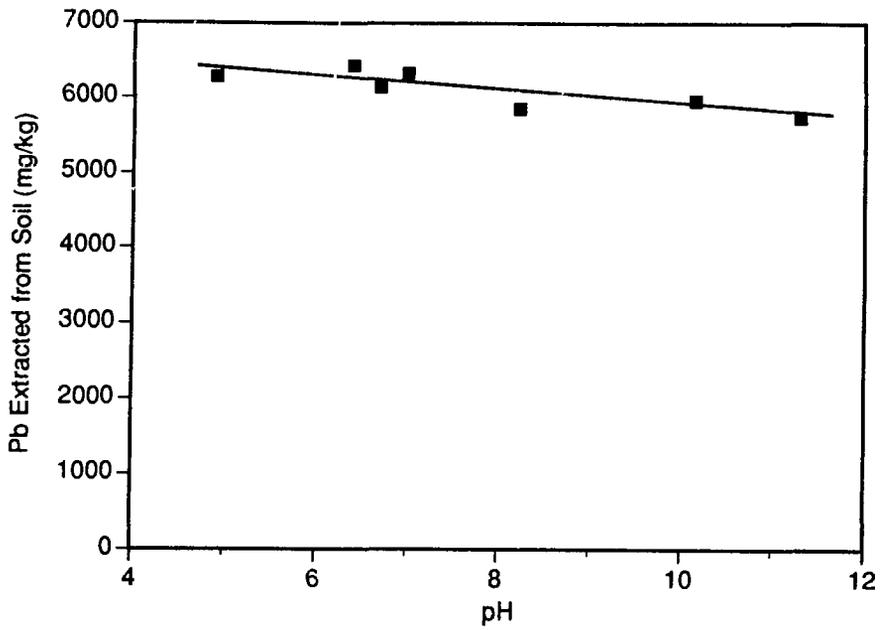


Figure 3 Residual concentration of lead after extraction with 0.1 M EDTA for a soil sample contaminated with 10,000 mg lead/ kg soil as a function of pH

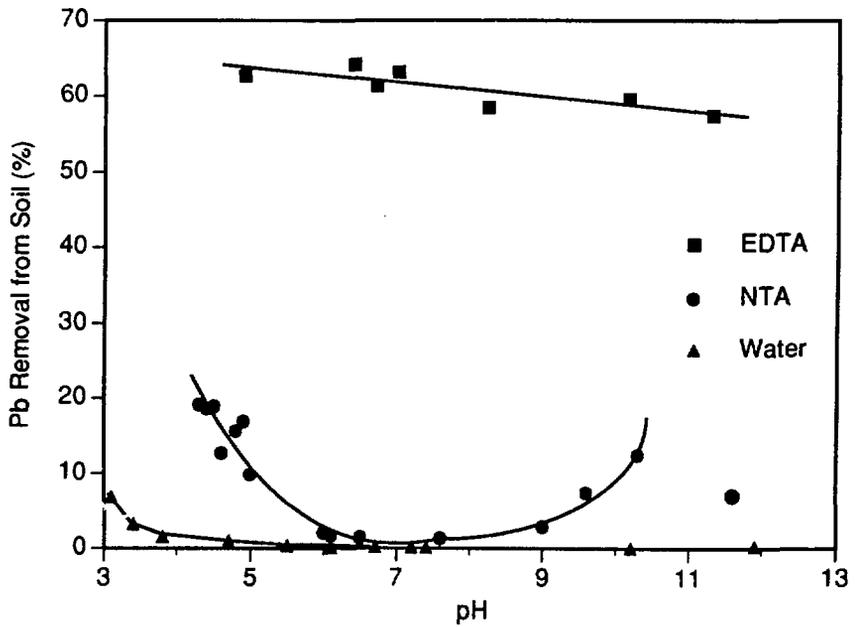


Figure 4 Comparison of the percent lead removal from contaminated soil using different extractants

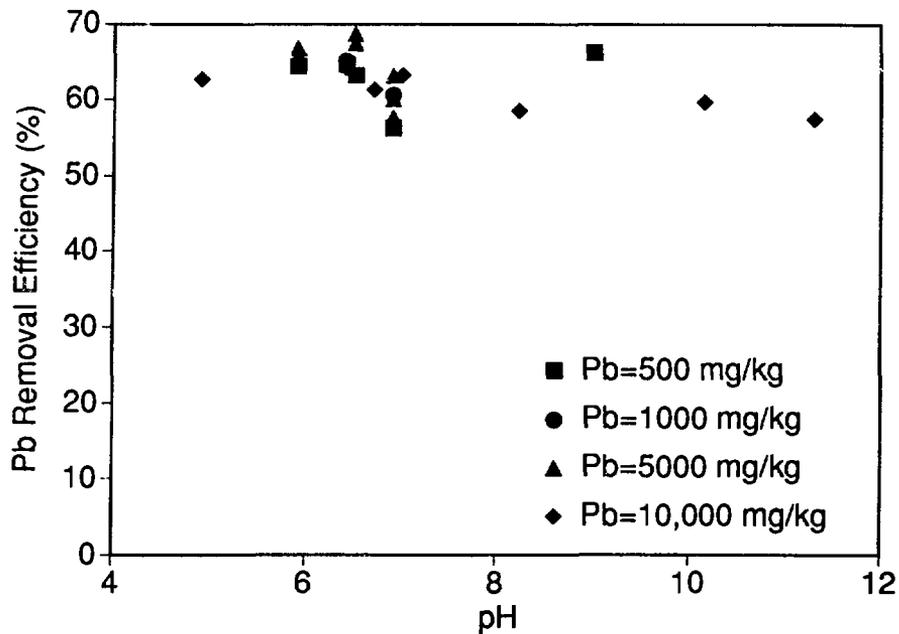


Figure 5 Lead extraction removal efficiency using EDTA as the extractant as a function of pH for different initial lead concentrations

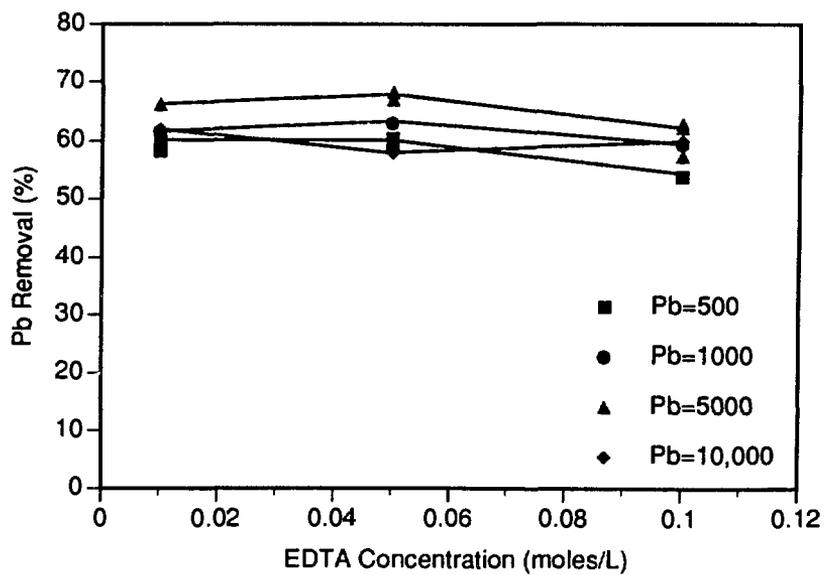


Figure 6 Comparison of lead removal efficiencies using EDTA as a function of initial lead concentration

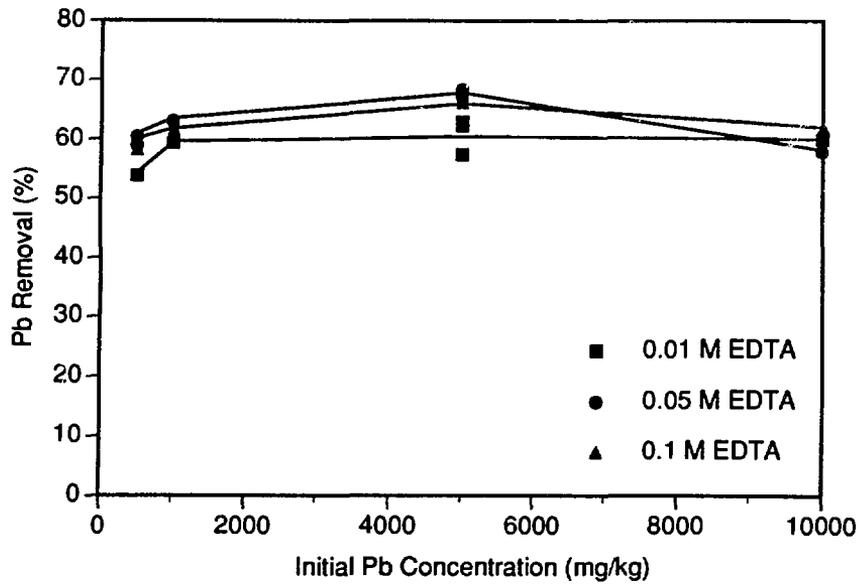


Figure 7 Comparison of lead removal efficiencies as a function of EDTA concentration