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Efficiency of EDTA-Na$_2$ and Oxalic Acid Mixture in Removing Lead from Calcareous and Gypsum Soils

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ABSTRACT

The soil washing method is an effective approach for treating polluted soils with high heavy metals concentrations. Oxalic acid and EDTA are both chelating agents that can form stable complexes with heavy metals and increase their solubility and mobility in the soil solution. By mixing oxalic acid and EDTA, the synergistic effect of the two agents can be achieved. In a recent study, two types of soils, calcareous and gypsum, were selected from agricultural lands (depth 0-30 cm). The washing solution comprising an equal ratio (1:1) of EDTA-Na$_2$ (0.05 M) and oxalic acid (0.05 M) was used to remove lead (Pb) from the soil samples. Five factors were selected to evaluate their impact on lead removal efficiency, which include pH (2 - 4 - 6 - 8), washing time (15 min - 30 min - 60 min - 90 min), lead concentrations (0 mg.L$^-$ - 300 mg.L$^-$ - 400 mg.L$^-$ - 500 mg.L$^-$ - 600 mg.L$^-$), temperature (20°C - 40°C – 60°C – 80°C), and liquid/solid ratio (L/S) (5/1 ml/g - 10/1 ml/g - 15/1 ml/g - 30/1 ml/g). The results indicated that the pH level significantly influenced the removal of Pb, with higher removal at lower pH levels. Additionally, the removal percentage increased with higher temperature, Pb concentrations, washing time, and L/S ratio. The results of this study can be summarized, that is Pb removal efficiency of 60.19% at pH 2, 94.24% at 30 min of washing time, 78.25% at 0 mg.L$^-$ of Pb concentrations, 66.47% at 20°C, and 84.77% at 15/1 ml/g of liquid/solid ratio (L/S). However, the presence of used engine oil (UEO) reduced the removal of Pb. There is no clear difference in the efficiency of removing lead from both types of soil (gypsum and calcareous).

1. INTRODUCTION

Soil pollution is currently receiving global attention due to its threat to health and the environment. Therefore, it was necessary to find effective ways to get rid of this danger. The term soil pollution refers to the contamination of soil with anomalous concentrations of harmful or toxic substances. This can pose risks to both human health and the ecosystem [1]. Soil may be contaminated with heavy metals by natural sources, including rocks weathering, volcanic eruptions, etc., soil may be polluted by human sources, including agricultural applications and transportation [2, 3]. In addition, there are many other human activities that may increase heavy metals levels in soil such as municipal waste disposal, industrial liquid waste, waste from mining and irrigation soil with wastewater [4, 5]. Remediation methods of soil contaminated with heavy metals includes physical, chemical, physical–chemical processes, bioaugmentation and biostimulation. Each method with its advantages and limitations, the choice of method depends on site-specific factors, contamination levels, and environmental considerations [6]. Chemical methods (soil washing, electrochemical remediation, adsorption) that are very effective [7]. Soil washing is a commonly used technique that relies on the use of many washing solutions to remove target contaminants from the soil, including organic and inorganic acids, chelating agents, etc. [8]. It is considered an effective and economical method and requires less time to remove the contaminants [9, 10]. Soil washing process depends not only on the properties of the target metal, but also on various physical and chemical properties of soil such as texture, pH, organic matter (OM), and cation exchange capacity (CEC) [11]. The mixed soil washing technology aims to remove heavy metals effectively, at a low cost, and is environmentally friendly so it is considered an effective alternative to single removal [12]. Besides, washing with a mixed solution can achieve a high removal efficiency with low concentration compared with using a single solution, thus reducing secondary contamination [13]. Washing solutions should be carefully chosen and detailed information about their effect. Those solutions include chelating agents, organic acids, inorganic acids and surfactants [14]. Organic acids including oxalic acid is considered biodegradable [15, 16], so they may be less harmful to the soil environment [17]. The objectives of the current study are testing the efficiency of EDTA-Na$_2$ and Oxalic Acid Mixture for removing Pb from calcareous and gypsum soils contaminated with Cd and Ni in addition to used engine oil (UEO), determining of same optimal parameters for soil washing.

2. MATERIALS AND METHODS

2.1 Sampling and preparation

The study included choosing soil samples from two different agricultural areas, one with calcareous soil (CS) and the other with gypsum soil (GS), at a depth of 0-30 cm. The collected samples were air-dried at room temperature for several weeks, crushed into a powdered form, and filtered through a sieve with a 2 mm mesh size. Afterward, different physical and chemical characteristics of the soil samples were measured, as described in Tables 1 and 2.

Table 1. Physio-chemical properties of samples

Properties	Unit	GS	CS
EC	dS.m$^-$	4.17	2.29
pH	--	7.27	7.63
CEC	Cmol.Kg$^-$	6.42	9.095
$CaSO_4$		31.08	0.067
$CaCO_3$		22.74	34.11
O.M		0.134	0.164
HCO_3^-		9	10
CO_3^{-2}		Nil	Nil
Mg^{+2}	meq.L$^-$	5	5.5
Ca^{+2}		7.5	3.5
Cl^-		5.64	8.46
K^+		14.0	10.6
Na^+	mg.L$^-$	29.6	33.7
Pb^{+2}		40.0	32.4

Table 2. Particle size distribution

Soil Sample	The Distribution of Particle Size			
	Sand	Clay	Silt %	Texture
Gypsum	47.2	33.6	19.6	Sandy Clay Loam
Calcareous	49.2	35.6	15.2	Sandy Clay

2.2 Used engine oil (UEO)

UEO was used to contaminate the soil samples after determining some of its characteristics (Table 3) in Basra Oil Company.

2.3 Contaminating the soil with lead

The soil samples in the study were contaminated with various concentrations of Pb (0 mg.L$^-$ -300 mg.L$^-$ - 400 mg.L$^-$ - 500 mg.L$^-$ - 600 mg.L$^-$) individually and allowed to dry, (0 mg.L$^-$) was chosen to be used as control samples in lead removal experiments, The contaminated soil samples were divided into two equal portions. The first portion was contaminated with Pb only, while the second portion was contaminated with both Pb and UEO (50 ml. Kg) according to previous studies [18, 19], and the soil samples were mixed thoroughly with UEO. Afterward, the samples were kept in a humid environment for three weeks to simulate natural weathering processes, and the humid environment may enhance or reduce the bioavailability of lead, affecting its interaction with UEO, followed by air-drying, crushing, and sieving through a 2 mm sieve (Figure 1) [18].

Table 3. Properties of used engine oil (UEO)

Test	Standard Methods	Used Engine Oil
Kinematic Viscosity @40°C, cst	ASTM D-445	76.97
Water content by KARL FISCHER, vol. %	ASTM D-6304	0.08
Kinematic Viscosity @100°C, cst	ASTM D-445	11.65
Denticity (g/cm^3) 15.6°C	ASTM D-4052	0.8724
Viscosity Index	ASTM D-2270	145
Flash Point	IP-36	205
Pb (mg.L$^-$)	---	16.83

Figure 1. Soil samples after contamination with lead

2.4 Lead removal experiments

The study utilized a removal solution comprised of equal parts (1:1) of EDTA-Na$_2$ (0.05 M) and oxalic acid (0.05 M) to eliminate lead from the soil samples. Five factors were selected to evaluate their impact on lead removal efficiency, which include pH (2 - 4 - 6 - 8), washing time (15 min - 30 min - 60 min - 90 min), lead concentrations (0 mg.L$^-$ - 300 mg.L$^-$ - 400 mg.L$^-$ - 500 mg.L$^-$ - 600 mg.L$^-$), temperature (20°C - 40°C – 60°C – 80°C), and liquid/solid ratio (L/S) (5/1 ml/g - 10/1 ml/g - 15/1 ml/g - 30/1 ml/g). Throughout the experiments, the values of the remaining factors remained constant while one factor was altered: EDTA-Na$_2$ at pH=4 and oxalic acid at pH=2, washing time of 90 min, lead concentration of 400 mg.L$^-$, temperature of 20°C, and liquid/solid ratio (L/S) of 10/1 ml/g. All removal experiments were conducted at a shaking speed of 180 rpm. Leachate was collected using a Whatman No: 42 filter paper to determine the concentration of Pb eliminated from the soil samples [19] and analyzed using atomic absorption spectrometry (Figure 2).

Figure 2. Using atomic absorption spectrometry to estimate lead removed from soil samples

3. RESULTS AND DISCUSSION

3.1 Effect of pH solution

The study's findings suggest that the pH level has a notable impact on the elimination of Pb. The greatest removal of Pb was observed at pH 2 or pH 4, while the removal decreased at pH 6 or pH 8 (Figure 3). At lower pH levels, the rise in H^+ ions might lead to the movement of numerous ions that were previously adsorbed onto soil particles (such as clay minerals or organic matter) become desorbed to soil solution due to competition with H^+ ions. The increased H^+ concentration promotes the dissolution of soil salts like carbonates (CO_3^{2-}) may form bicarbonates (HCO_3^-) and sulfates (SO_4^{2-}) may release sulfate ions (SO_4^{2-}) into the soil solution [20, 21].

The pH can also influence how lead is distributed in the soil. Solid phases, lead may be attached to oxides, and these metal oxides dissolve more easily in lower pH levels [22]. However, an alkaline condition hinders the adsorption of lead ions, reducing the efficiency of extraction, higher pH promotes the formation of less soluble lead compounds, and lead remains attached to soil particles [23]. The impact of pH on the effectiveness of soil washing has been documented in previous research. The study also indicated that EDTA was superior to other substances like citric acid, malic acid, HCl, and HNO_3 in removing lead (Pb) from polluted soil. The results of this research showed that after 24 hours of washing, lead removal using EDTA-Na_2 reached 27.4%, and only 1.5% using oxalate [24].

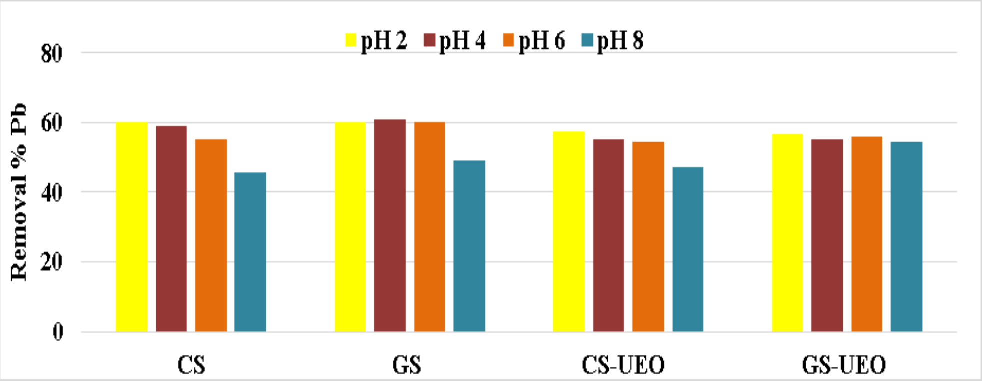

Figure 3. Effect of pH

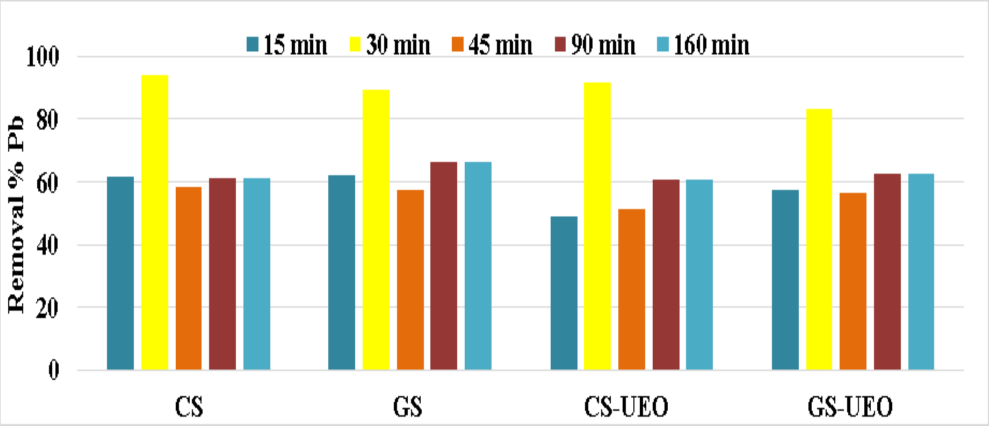

Figure 4. Effect of washing time

3.2 Effect of removal time

The duration of washing is a critical factor that influences the adsorption between soil and heavy metal ions [25]. The removal of Pb is impacted by the washing time, with the removal percentage increasing as the washing time rises until it reaches the maximum removal, after which it gradually decreases (Figure 4). Initially, the removal rises rapidly due to the release of weakly bonded ions with soil particles, followed by a gradual decrease, consistent with previous reports [26, 27]. This pattern could be attributed to two phases of lead release: the rapid release of weakly bonded lead ions to the soil surface, followed by the slow release of strongly bonded lead ions onto soil particles [28-30]. It is also possible that the extracted lead is re-adsorbed onto soil particles with a relatively long washing time [31].

3.3 Effect of lead concentration

Lead concentrations affect removal efficiency (Figure 5). The reason may be that when the soil is contaminated with lead, the lead ions increase on the soil surface and form one adsorption layer followed by the formation of adsorption layers where the formation of adsorption layer ions away from the adsorption surface becomes weak compared to the adjacent layer. That is, an increase in the concentration of lead means an increase in the adsorption layers [19]. The gradual decrease in lead removal from 78.25% at mg.L$^-$ to 3.05% at 300 mg.L$^-$ and then the gradual increase to 66.47% at 400 mg.L$^-$ may be attributed to an increase in lead concentrations, causing saturation of the washing solution with lead ions, because when the wash solution comes into contact with the soil, it binds to lead ions present in the soil particles. Initially, the solution can effectively remove lead ions. However, over time, if the concentration of lead ions in the solution reaches its saturation point, the solution becomes unable to receive additional lead ions. This saturation occurs because the available binding sites are already occupied by lead ions [32].

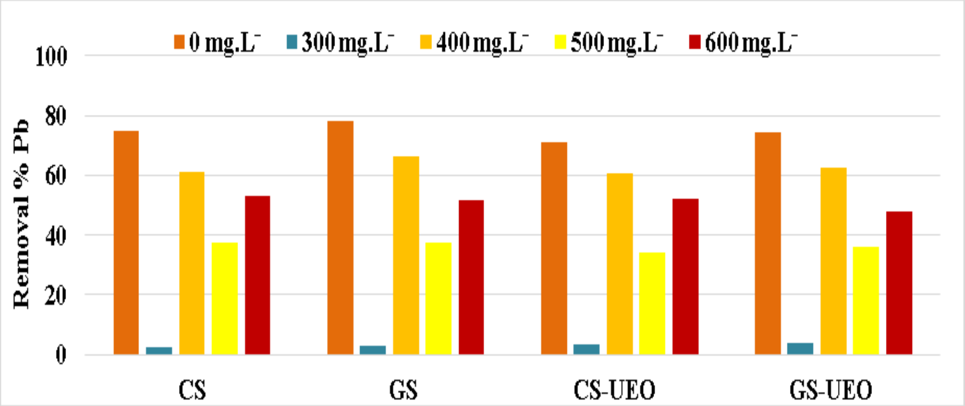

Figure 5. Effect of lead concentration

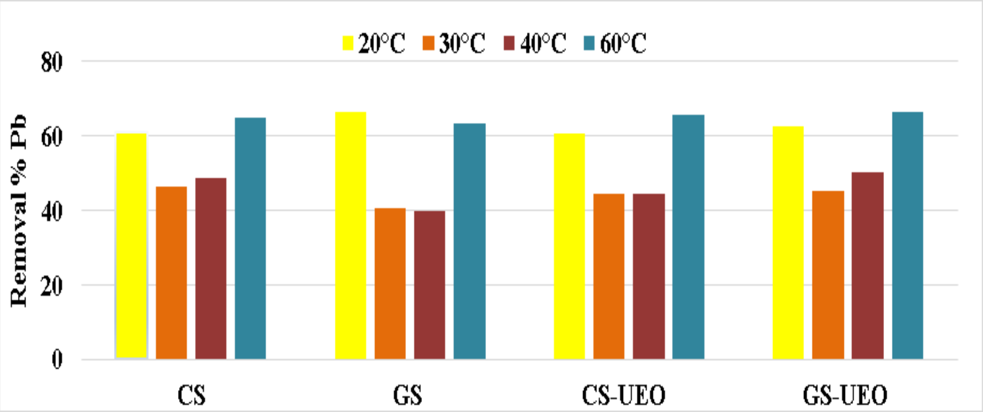

Figure 6. Effect of solution temperature

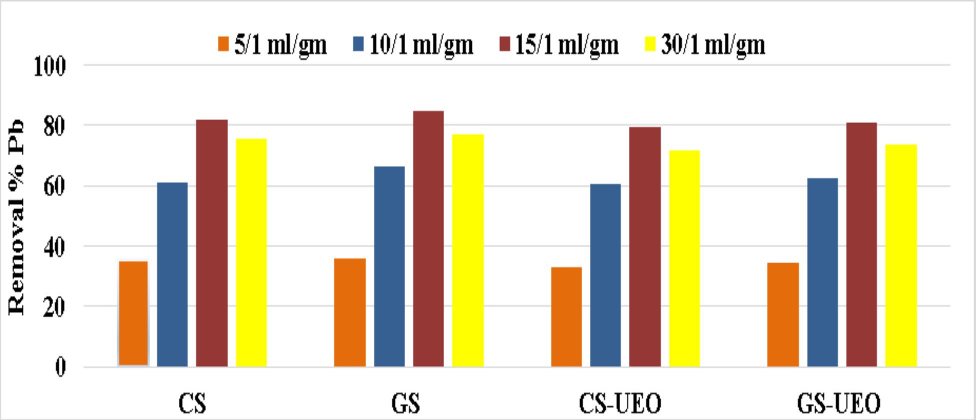

Figure 7. Effect of liquid/solid (L/S) ratio

3.4 Effect of solution temperature

According to Figure 6, the lead removal decreases and then gradually increases with increasing temperature. Increasing the temperature leads to an increase in the reaction kinetics and thus an increase in the removal efficiency [33]. Temperature affects adsorption and solubility and thus affects the removal [34]. Temperature affects the viscosity and density of UEO. At lower temperatures, its difficulty of reaching locations of the soil surface. High viscosity hinders the diffusion of lead ions through the oil. Used engine oil can experience changes in density due to temperature fluctuations [35]. Decreased density (due to temperature increase) affects lead ions, the partitioning behavior of lead ions between oil and soil depends on their relative densities. The physical properties of used engine oil—specifically its viscosity and density—play a crucial role in determining the behavior of lead ions. These properties influence the diffusion, mobility, retention, buoyancy, and partitioning of lead ions, ultimately affecting their release into the environment [36].

3.5 Effect of liquid/solid (L/S) ratio

According to the results of Figure 7, the removal of lead increases with an increase of the ratio until it reaches the maximum limit and then begins to decrease. The reason for this may be that an increase where the increase of this ratio indicates an increase in the ability of the removal solution to release Pb ions. The decrease in removal may have occurred due to the solution released most of the lead ions adsorbed to the soil [35]. However, higher this ratio will result in a solution of more residual metals for post-processing [37].

4. CONCLUSION

The study concludes that the soil washing technique is an effective method for eliminating heavy metals from heavily polluted soils, particularly when other remediation methods face difficulties. The use of a mixed washing solution (EDTA-Na_2 and oxalic acid) can result in high efficiency in removing Pb at a lower concentration, thereby reducing costs. The pH parameter plays a significant role in removing Pb from soil samples, as removal increases with a decrease in pH. The presence of UEO contamination had little impact on Pb removal compared to uncontaminated samples. Additionally, the efficiency of removing lead from gypsum soil remained unaffected when compared to calcareous soil.

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