



EFFECTS OF LEAD NITRATES ON THE COMPACTION CHARACTERISTICS OF ELECTROKINETIC REMEDIATED LATERITIC SOIL

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ABSTRACT

The moisture-density relationship between Pb-contaminated soil and electrokinetically remediated treated soil is investigated in this study. Five of three kilograms of lateritic soil were contaminated with lead (II) nitrate salts with a maximum 10% lead nitrate concentration at a stepwise rate of 0.2% by weight of dry soil. The mixture was kept in a plastic container for 30 days. Soil samples were remediated using electrokinetic techniques to study the efficiency of this method to extract lead nitrate from soil. 0.1 M ethylenediamine tetracetic acid (EDTA) and 0.1 M acetic acid were used as a purging solution at the cathode and anode compartments,

Introduction

Construction must be constructed on, in, or through the earth. Low shear strength, excessive compression, collapsing behavior, and high swelling potential must not exist in the soil. Some soils in nature have one or two undesirable characteristics that must be corrected in order to increase their bearing capacity, improve shear strength and permeability, increase the stiffness density of the soil, and immobilize or stabilize contaminants in dredged soil in order to reduce and, if possible, eliminate their negative effects on the environment. Contaminants may be inorganic, organic, or radioactive species, and they may originate from fertilizers, pesticides, biosolids and manures, wastewater, leaded gasoline, paint, petrochemical spills, and atmospheric deposition (Wauna and Okielmen 2011). Due to their persistence and irreversibility, heavy metals such as arsenic, cadmium, chromium, mercury, lead, copper, nickel, zinc, and selenium have been major global environmental concerns. As a heavy metal, lead is insoluble, stable, and has a longer residence time and its presence of lead in soil poses a threat to the environment, microorganisms, plants, and human health. Bioremediation (Yaashukaa et al., 2022; Palas et al., 2022; Martinez et al., 2021), soil washing/flushing (Liu et al., 2021; Rosas et al., 2013; Lee et al., 2013); and other technologies have been studied to remediate contaminated soils, sediments, and water. Stabilization and solidification (Scanferia et al., 2009; Alpastari and Yukselen, 2002); vitrification (Sivers et al., 2019; Bathesteros et al., 2017). (Awa and Hadiburata 2020; Jadia and Fuleka 2009) Phytoremediation Electrokinetic (EK) remediation (Yan et al., 2022, Wang et al., 2022). Electrokinetic remediation is a green



respectively. After testing the removal efficiency of lead nitrate with X-ray fluorescence equipment ranging from - to -, compaction was performed on both natural and lead nitrate-contaminated soil and EKR-treated soil samples at British Standard Light (BSL), West African (WAS), and British Standard Heavy (BSH) compaction energies. The result obtained shows that as lead concentration increases, there is an increase in maximum dry density, with the peak at 0.6%, and thereafter there is a reduction for BSL, WAS, and BSH. For the electrokinetically treated soil, the dry density was greatly increased at all concentrations. Therefore, the electrokinetic method can be used to improve the engineering properties of problematic soils.

Keywords: Compactive Effort, Electro kinetic, Lateritic, Lead, optimum moisture content, maximum dry density.

technology used for dewatering, forced consolidation, stabilization of waste contaminants, control and/or removal, and as a grouting aid. Ek can be used ex-situ or in-situ, is effective on low-permeability and heterogeneous soil, is effective on both saturated and unsaturated soil, is effective on heavy metals, radionuclides, and organic contaminants, and is simple to integrate with existing technologies. Other methods contain one or more flaws.

Electrokinetic remediation involved inserting electrodes into contaminated soils and applying a low direct current voltage gradient to the electrodes (Acar and Alshawaber, 1993; Lagerman, 1993). During Ek, the applied current causes electrolysis of water at the electrodes, resulting in a series of electric effects, including electroosmosis, electromigration, and electrophoresis.

Electroosmosis is the movement of soil moisture or groundwater from the anode of an electrolytic cell to the cathode. Ions and ion clusters undergo electromigration when they migrate toward an electrode with a different charge. Colloids, or charged particles, move in an electric field. The term for this is electrophoresis.

Numerous researchers have discovered that when EK is used to eliminate contaminants, the shear strength of the soil increases. Ozkan et al. (1999) strengthened kaolinite by injecting it with phosphate and aluminum ions. According to their research, the atterberg limit and shear strength increased from 7 kPa to 30 kPa throughout the entire specimen. Asavadorndeja and Glave (2005) arrived at an identical conclusion. They also electrically injected stabilizing agents into the soil, which increased its strength by up to 170% immediately and by up to 570% after seven days.

Jayekanthan et al. (2012, Lefebvre and Burnette (2003), Alshawabeh and Shehan (2003), Alshawabeh et al. (2004 and Paczkowaka (2005) have investigated the injection of stabilizing agents to improve soil properties, as has soil compressibility (Ahmad et al. 2011).

Mohammed Hassan (2011) investigated the viability of reducing the water content and increasing the shear strength and axial load capacity of soft clays by applying electrokinetic treatment with fresh water in an experiment, and the researcher reported a high degree of effectiveness (Azhar et al. 2017). After EK treatment, bentonite clay had two orders of magnitude less conductivity than kaolinite clay, according to Jayasekera (2004). However, the electroosmotic permeability of both clays was $10^{-5} \text{cm}^2/\text{V/s}$.



The plastic limit and liquid limit have been increased, resulting in a decrease in the plasticity index. This indicates that the soil's compressive strength has increased by more than 100 per cent with EKR alone, and by nearly 200 per cent with the addition of lime (Jayeseka and Hall, 2007).

Numerous studies have focused on removing contaminants and cleaning contaminated soil up until this point. However, according to Nasim et al. (2012), very few studies have investigated the use of electrokinetic technology to enhance the mechanical and engineering properties of problematic soil. The aim of this study is to determine the relationship between the moisture-density of Pb-contaminated and Electrokinetic treated soil.

MATERIALS AND METHODS

Materials

The lateritic soil sample used in this study was obtained from a non-contaminated borrowed pit in the Shika local government area of Kaduna State at 110.45°N and 70.08°E.

The soil sample was bagged and transported to the laboratory for soil mechanics. The soil sample was air-dried in the laboratory and then passed through a 2-mm sieve. Then, it was bagged and stored for later use. XRF spectroscopy was used to determine the chemical composition of the soil at the Nigerian Rolling Steel Council in Kaduna.

Tabularized in Tables 1, and 2, respectively, are the analytical-grade geotechnical properties of uncontaminated soil, and the chemical composition of the soil used in the test.

Table1: Geotechnical properties of the natural soil

| Properties | Value/Description |
|--------------------------------------------------|--------------------------|
| %Passing B.S Sieve No 200 | 82.15 |
| Liquid limit (LL)% | 43.5 |
| Plastic limit (PL)% | 15.38 |
| Liner Shrinkage% | 5.6 |
| AASH to classification | A-7-6 |
| Unified soil classification System (USCS) | CL |
| Plastic index | 28.12 |
| Group index | 15 |
| Specific gravity | 2.67 |
| Natural moisture content % | 22.7 |
| Colour | Reddish brown |
| Sand (0.0075mm-4.75mm) | 25.45 |
| Clay (< 0.002mm) | 82.18 |
| pH | 6.8 |

Table2: Oxide composition of Natural Lateritic soil.

| Oxide | Concentration % |
|------------------------------------|------------------------|
| SiO₂ | 52.309 |
| V₂O₅ | 0.081 |
| Cr₂O₃ | 0.004 |
| MnO | 0.084 |
| Fe₂O₃ | 11.499 |
| CO₃V₄ | 0.054 |
| CU | 0.043 |
| Nb₂O₃ | 0.017 |



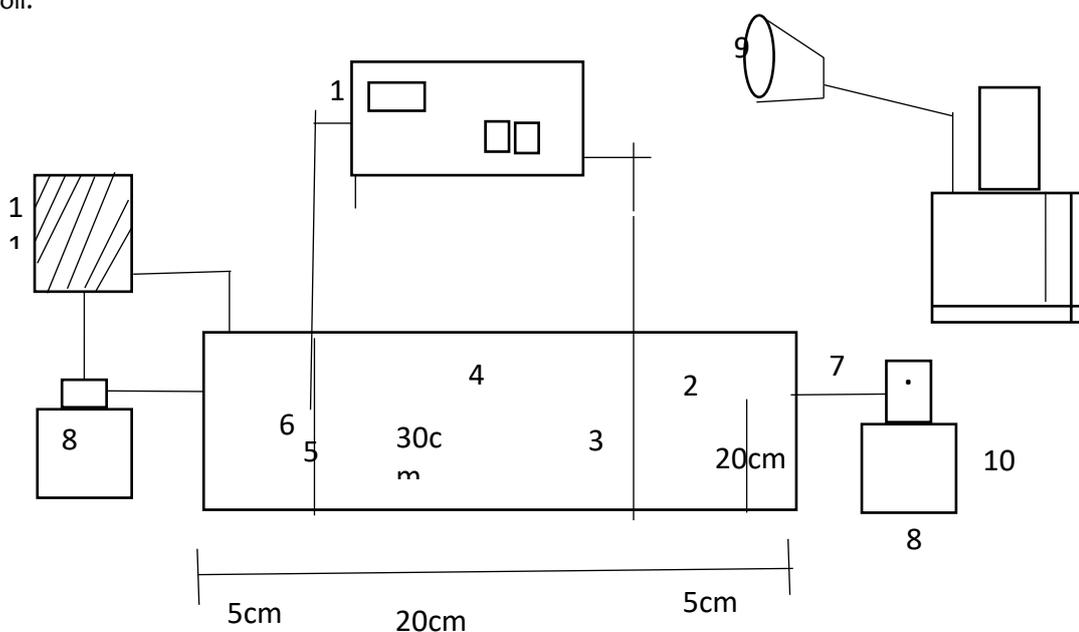
| | |
|-------------------------|-------|
| Moo_3 | 0.004 |
| Cao | 0.049 |
| K_2O | 0.527 |
| B_2O | 0.014 |
| Al_2O_3 | 32.99 |
| Ta_2O_5 | 0.028 |
| TiO_2 | 1.502 |
| ZnO | 0.002 |
| Ag_2O | 0.014 |
| CL | 0.65 |
| ZrO_2 | 0.157 |

Reagents:

The Lead nitrate ($\text{Pb}(\text{NO}_3)_2$) is 99% in purity, obtained from Loba Chemie PVT. Ltd. It has a density of $4.53\text{g}/\text{cm}^3$, solubility in water of $565\text{g}/\text{l}$ and a molar weight of $331.2\text{g}/\text{mol}$. Standard ASTM Type 1 deionized water was used throughout this experiment. Acetic acid glacial was obtained from Guangdong Guanghua Sci-Tech co., Ltd Shantou, Guangdong, China and the Ethylendiamintetraacetic (EDTA) from Merck KGaA Germany with a molar mass of $410.31\text{g}/\text{mol}$.

Test Equipment

The test equipment consists of an acrylic plastic electrokinetic remediation (EKR) cell, a DC power supply, a peristaltic pump, an overflow collection system, and a data acquisition system. The EKR cell measures 40 cm (length), 20 cm (width), and 30 cm (height) (depth). The dimensions of the soil compartment ($30\text{ cm} \times 20\text{ cm} \times 30\text{ cm}$) and the electrolyte chambers ($5\text{ cm} \times 20\text{ cm} \times 30\text{ cm}$). The graphite electrode measured 8 millimeters in diameter and 30 centimeters in length. There were a total of six electrodes. Porous Plexiglas separated the soil compartment from the electrolyte compartment. On the porous plate, the holes were 5 mm in diameter and 2 mm apart. In order to prevent soil particles from entering the electrolyte chamber during the test, 40 cm of filter paper was placed between the porous plate and the soil.



The top and bottom of the electrolyte chamber were equipped with two control valves. The



top valve was designed to collect the electromotive flow fluid, while the bottom valve pumped the electrolyte into the electrolyte chamber. The entire setup is depicted in figure 1.

Systematic Diagram of Experimental Setup:

1. DC Power supply; 2. Cathode chamber; 3. Cathode; 4. Soil chamber; 5. Anode
6. Anode chamber; 7. Overflow port; 8. Graduated cylinder; 9. Camera; 10. Acquisition collection system; 12. Primastic pump

PREPARATION OF SAMPLE

Artificial contamination of soil samples

Five separate plastic containers were filled with soil weighing a total of 117 kilograms each. The soil sample in each bucket was thoroughly mixed with lead nitrate at concentrations of 0.2% ranging from 0% to 1% based on the sample's dry unit weight. In accordance with Resmi et al. (2011) and Karkush et al., the mixture was left for a month to achieve a homogeneous saturation of lead nitrate (2013). After 30 days, the contaminated soil was stirred for 10 minutes to evenly distribute the lead nitrate throughout the soil (Gang et al., 2021). The soil was then air-dried at room temperature, pulverized, and stored in polyethene bags after passing through a 2mm sieve.

Soil sample preparation for electrokinetic remediation

Three 15-kg samples of contaminated soil were collected for each of the three cells that were constructed. The soil was mixed with liquid-limit water and then loaded into electrokinetic cells. A voltage of 30 volts was applied across the electrode to achieve 1 volt per centimetre. Both the anolyte and the catholyte consisted of 0.1 M acetic acid. After the test, four thin metal sheets were inserted with a 6-centimetre gap between them into the chamber containing the soil sample, and the soil was divided into five equal parts. The soil was excavated with a spatula, the areas from the cathode to the anode were sequentially labeled S1–S5, and the soil was dried for testing.

ANALYTICAL METHOD

Measurement of current, pH, and electrical conductivity

The current was measured by the digital DC power supply.

Measurement of pH

During the pH measurement of the soil sample, 5.0 g of the dried soil after the test was placed in a beaker and added to 25 ml of distilled water. A magnetic stirrer was used to mix the soil and water well for 15 minutes. After that, the mixture was left to settle for an hour before the pH meter's probe was put in to measure the soil's pH.

The electrical conductivity (EC) was monitored with a digital multimeter, and the cumulative electroosmosis flow (EOF) was also measured by monitoring the liquid spillage overflowing the electrode compartment once a day. The graduated bottle was covered and kept for further analysis.

Determination of Pb(II) nitrates concentration

The XRF spectrometer was used to measure the concentration of lead in the soil. The following equation was used to calculate the pH removal efficiency:



$$[(M_o - M_e) / M_o] * 100 = R$$

Where R is the removal efficiency percentage%

M_o is the initial mass of pollutants in mg.

M_e denotes the total mass of remaining pollutants in milligrams. **Compaction**

Three compaction efforts, namely; British Standard Light (BSL), the British Standard Heavy (BSH), and the West Africa Standard (WAS) or Intermediate, were used. The BS compacting is equivalent to the standard proctor, while the BSH compaction is the equivalent of modified proctor compaction. The WAS, or intermediate compaction, is the usual energy level used in the area (Osinubi, 1998).

The comparison of the three-compaction effort is given in Table 3

Table 3: Comparison of compaction test procedures (Source Osinubi 1998)

| Test Procedure | Width of rammer(kg) | Height of fall (m) | Number of blows per layer | Number of layers | Compaction energy KNm/m ³ |
|----------------------------------|---------------------|--------------------|---------------------------|------------------|--------------------------------------|
| BS Light compaction | 2.5 | 0.3048 | 27 | 3 | 605.9 |
| West African Standard Compaction | 4.5 | 0.457 | 10 | 5 | 1008.7 |
| B.S Heavy Compaction | 4.5 | 0.457 | 27 | 5 | 2723.5 |

Result and Discussion

Compaction Characteristics for lead Contaminated soil

The results of compaction tests for Pb-contaminated soil are presented in Figure 2. Values of optimum water content vary from 19-18.8%, 16.8-15.8%, and 14-14.8% for BSL, WAS, and BSH compactive effort respectively.

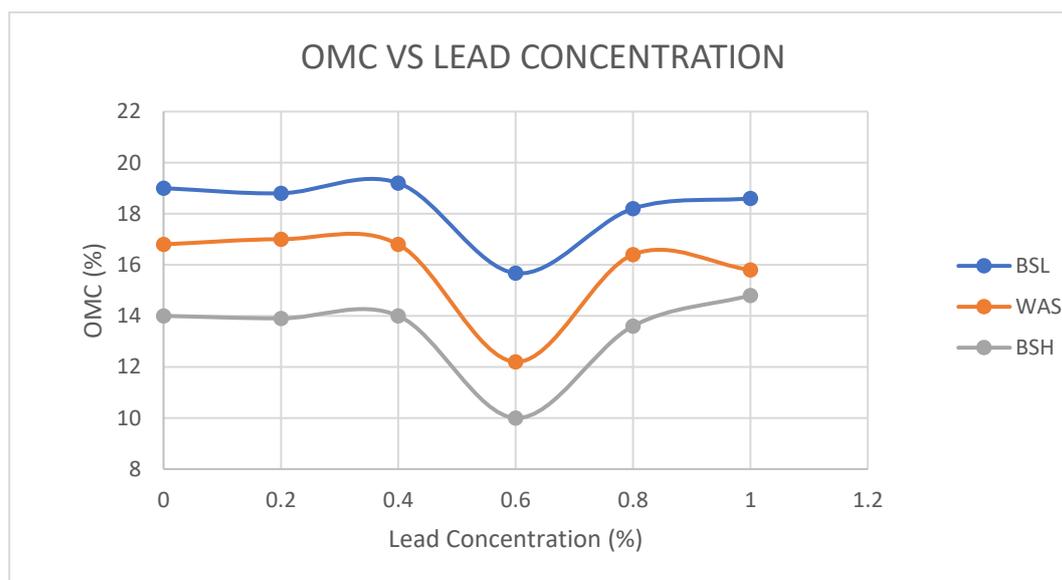


Figure 2: Variation of lead concentration with optimum moisture content at different compactive efforts for lead-contaminated soil



The maximum dry unit weight value ranges from 1.68 – 1.70KN/m² for BSL, 1.76 – 1.84 KN/m² for WAS and 1.84 – 1.95KN/m for BSH compacting effort as shown in Figure 2.

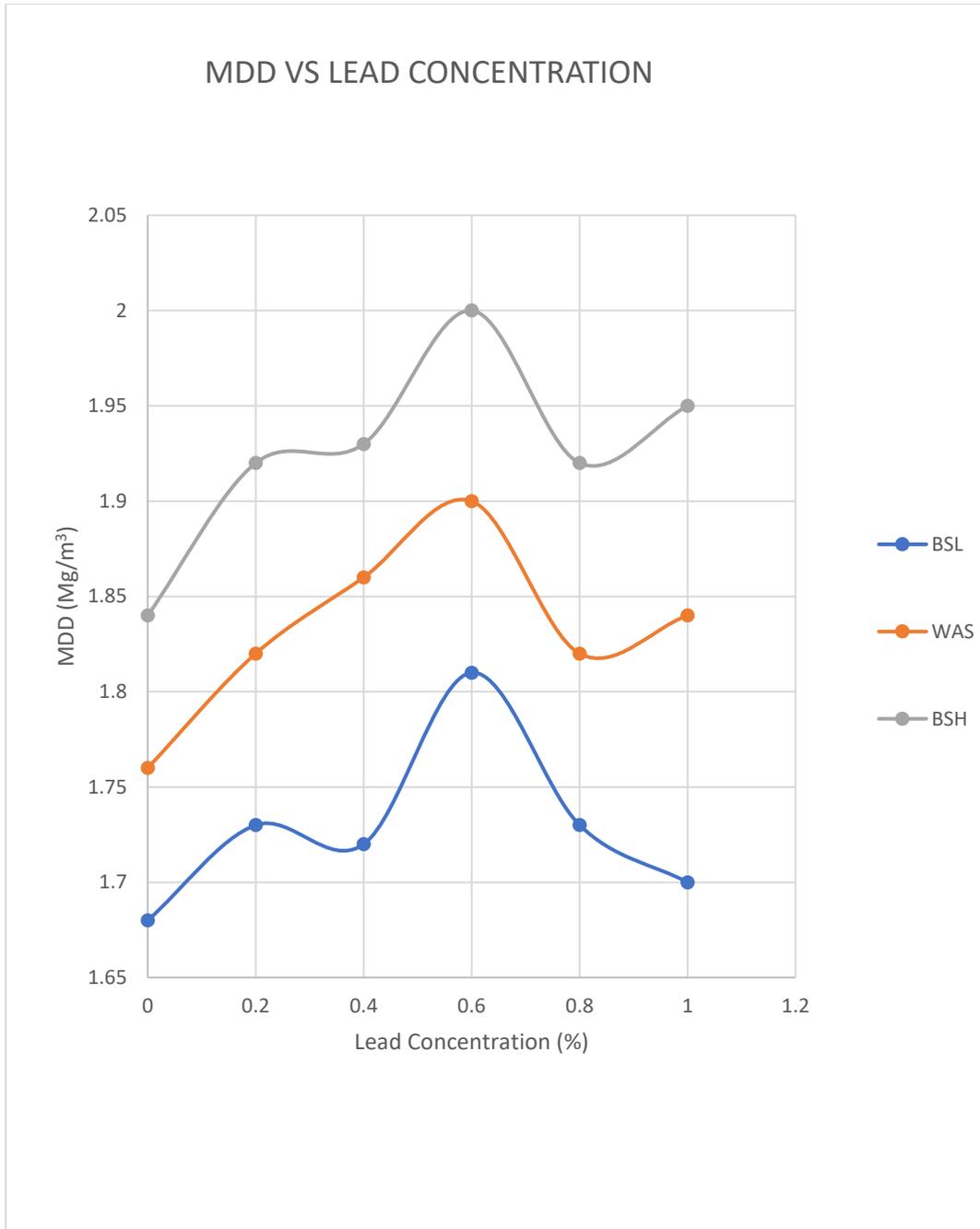


Figure 3: Variation of lead concentration with maximum – dry density at different compactive efforts for lead-contaminated soil

Irrespective of the lead concentration the maximum dry density increases with the various compactive effort, consequently as the lead concentration increases there is an increase in maximum dry density with the peak at 0.6% and thereafter there was a reduction, and the maximum dry density value increases from a natural value of 1.68, 1.76, and 1.84 Mg/m³ to 1.89, 1.90, and 2.0 Mg/m³ at 6% lead concentration for BSL, WAS and BSH respectively. The increase



is a result of the lead having a higher specific gravity of 4.53g/cm^3 than the natural soil of 2.67mg/m^3 and also the contact points on the clay molecules when covered by lead nitrate salt, engineering performance of the soil and the ability of clay to dissociating water molecules is decreased.

Increasing contamination causes a clear change in the compaction curves shape, so as the contamination increase the optimum moisture content reduces this could be a result of a reduction of the voids among the particles due to sedimentation of salts in pores.

The reduction in optimum moisture content causes an increase in the maximum dry density. This finding is similar to the work of (Mahdised and Shadach 2020) and (Sunit et al., 2008) and also confirms the classical theory of soil mechanics the higher the optimum moisture content the lower the maximum dry density.

The contamination of lateritic soil with heavy metal will need more compaction effort to get the desired maximum density if to be used as a hydraulic barrier (Ugwu et al, 2018).

Compaction characteristics for EKR treated soil

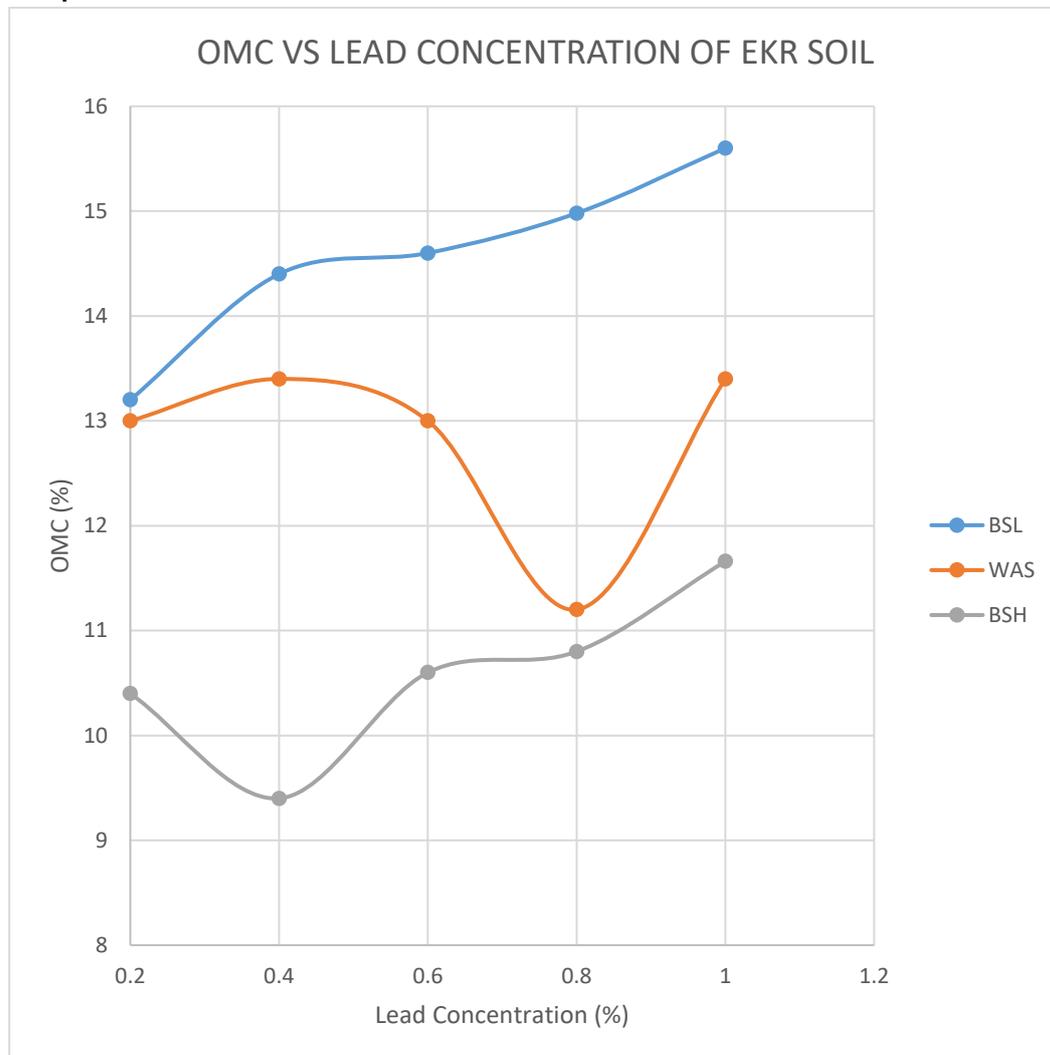


Figure 4: Variation of Lead Concentration with maximum dry density at different compactive efforts for electrokinetic treated soil.

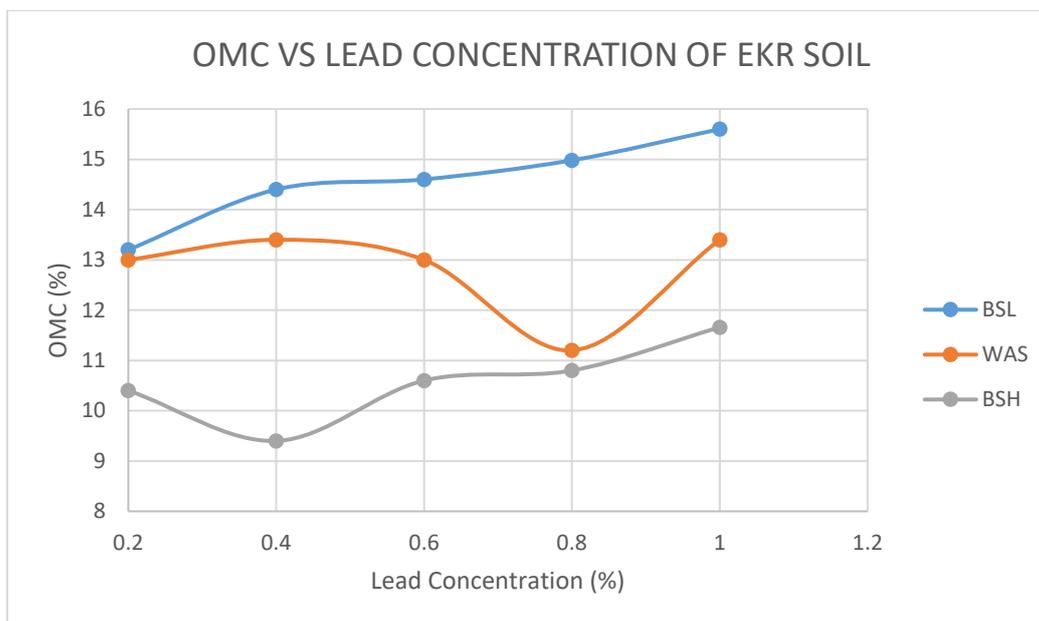


Figure 5: Variation of lead concentration with optimum moisture content at different compactive efforts for electrokinetic treated soil

The Figure 4 shows the relationship between dry density and moisture content concerning different levels level and different percentage of lead contaminates.

The result of compaction tests are presented in Figure 5 values of optimum water content vary from 13.20 – 15.60, 13.00 – 13.40, 10.40 – 11.60 for BSL, WAS, and BSH compactive effort respectively. The maximum dry unit weight ranges from 1.81 – 1.86 for BSL, 1.87 – 1.86 for WAS and 1.96 – 1.94 for BSH compacting effort shown shown in figure 4 above.

In accordance with the findings of Osinubi et al. (2005), as compactive effort increases, maximum dry density also increases, while optimum water content decreases. Moreover, relative changes in MDD and OMC will be influenced by the resistance offered by soil particles during compaction. With the use of EDTA salt as a catholyte electrolyte and acetic acid as the anolyte, the diffuse double layer tends to depress and allow particles to come closer under the same amount of compaction energy, resulting in the maximum dry density increase (Singh and Prasad, 2007).

In addition, a change in the soil's pH results in changes in the chemistry of the pore fluid, which leads to a change in the soil's structure or mineralogy, particularly in the anode and cathode regions. It has been demonstrated by the increase in MDD, which is consistent with the conclusions of Saufil Azhar's 2012 study.

In addition, when the pH of clays becomes extreme (extremely high or low), soil components such as iron, aluminum, and sodium are dispersed. Liaki et al. (2010) reported that soil is strengthened by the dispersion and migration of aluminum ions when the pH is less than 5.

With the addition of an alkaline solution, such as lime, the increase in soil compressive strength is significantly greater (Malekzedehe et al., 2016). In this study, alkaline EDTA salt was used to maintain a higher pH, thereby increasing the basic environment and soil precipitation, which resulted in improved efficiency and a higher dry density, which invariably led to greater soil strength.



Result of Electrokinetic Remediation

Electrical Current

The result for electrical current with time is depicted in Figure 6 it is observed that 0.2% lead contamination had a maximum current of 0.09 mA, which dropped to 0.07 mA after 72 hours and remained constant for the remaining 42 hours of the experiment. At 0.4%, its initial current was 0.18 mA and after 144 hours, it dropped to 0.09 mA. The electromotive flow ceases, indicating the cessation of ion movement. The starting current increased by 0.6% to 0.21 mA. After 120 hours, the current dropped to 0.06 mA, where it remained until the conclusion of the experiment. At 288 hours, both 0.8% and 1.0% lead contamination began with a higher initial current of 0.41 mA and 0.64 mA, respectively. For 0.8%, the current remained constant at 240 h for the next 72 hours, before falling to 0.07 mA for the following 216 hours. While there was lead contamination of 1.0%, the current remained constant after 312 hours, dropped to 0.07 mA for the next 24 hours, and then dropped to 0.06 mA until the conclusion of the experiment.

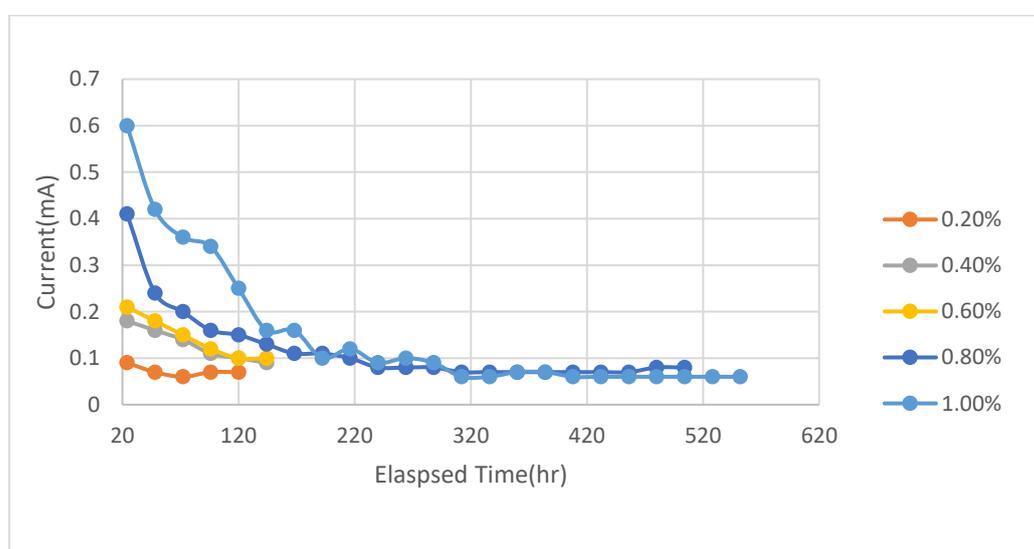


Figure 6: Variations of Current with time

First, electrolysis reactions between the anode and the cathode produced bubbles, such as O_2 and H_2 , which may have adhered to the electrode surface, thereby increasing the resistance of the electrode surface and reducing the current. Second, the electric field caused H^+ and OH^- to migrate toward the opposite electrode, forming an acidic migration zone and an alkaline migration zone until they were neutralized. As a result of this process, the amount of current was reduced.

Finally, as the soil near the cathode became slightly alkaline, Pb^{2+} and OH^- combined to form $Pb(OH)_2$. The soil pores were obstructed by the precipitate, which was not conducive to the migration of charged ions; consequently, the resistance was measured and the current decreased. (Zang et al 2019).

Rate of electro-osmosis flow (EOF)

Figure 7 illustrates the accumulation of EOF over time. After 24 hours, a flow of 267 mL was recorded for the 0.2% lead contamination, and after 120 hours, a cumulative electro-osmosis flow of 497 mL was recorded. While the 0.4% had an EOF of 112 mL after 24 hours, 1113 mL were recorded at the conclusion of the experiment after 144 hours.



After 24 hours, 0.6% had a flow of 246 ml and a cumulative EOF of 1288 ml was recorded after 144 hours. 0.8% and 1.0% have an initial EOF of 170 mL after 24 hours, which increases to 6309 mL after 504 hours and 5013 mL after 552 hours, respectively. During the experimental procedure, the observed direction of EOF was from anode to cathode, and the accumulation of EOF in each group increased gradually. Researchers have demonstrated that the size of the EOF is primarily dependent on the Reddy, and Cameselle (2009).

As shown in Figure 7. The EOF trend is consistent with the current trend. The greater the current, the greater the EOF, so the greater the accumulation of EOF between 0.8% and 1.0%. Since the direction of the EOF was consistent with this electromigration, it also affected the Pb removal rate.

Pb distribution in the soil following remediation.

Figure 1 depicts the soil Pb concentrations as a function of the normalized distance from the anode for 0.2%, 0.4%, 0.6%, 0.8%, and 1.0% EK experiments.

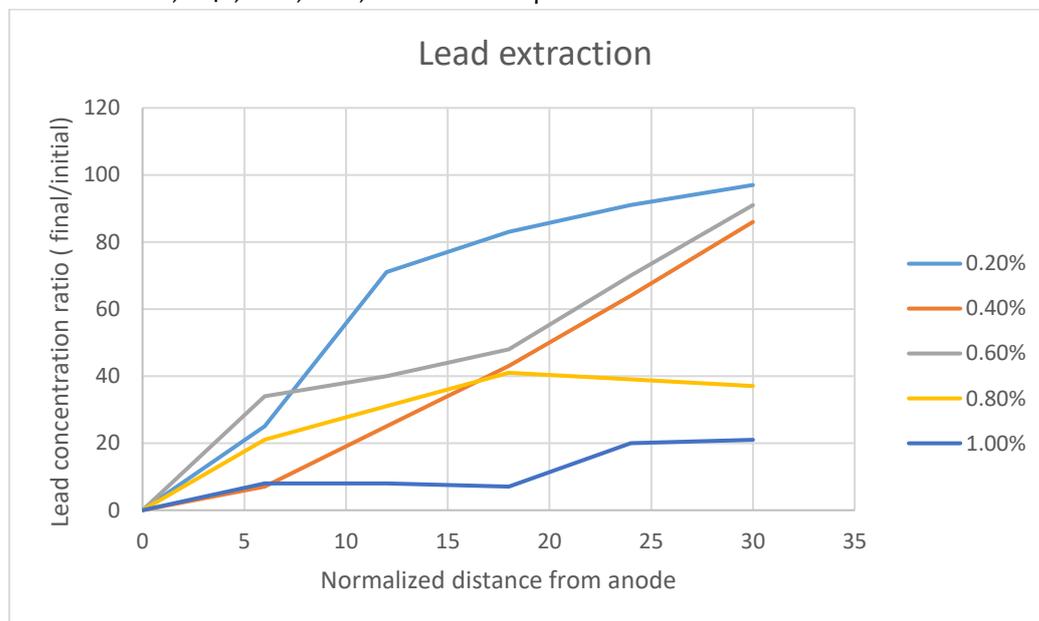


Figure 8: Variation of Lead Concentration with normalize distance from Anode

Observations indicate that Pb^{2+} migrated effectively through the soil towards the cathode, resulting in the majority of Pb^{2+} precipitating at regions S4 and S5 due to the alkaline soil condition. Variations in concentration indicate that Pb^{2+} migrates to the cathode and accumulates there. Similar findings were found in earlier studies, which demonstrated that Cr^{2+} migrates toward the cathode and accumulates as precipitates or adsorbates in the pH-high regions closest to the cathode (Zong-Ping et al., 2015; Al-Hamdan and Reddy, 2008; Alt Ahmed et al., 2015; Kamal et al., 2021).

Amrate et al. (2005), Wang et al. (1997), and Kim et al. (2005) all agree that the molar amount of EDTA required to extract lead from contaminated soil should equal the molar amount of lead in the soil. In this study, higher EDTA concentrations than lead concentrations are used to extract the most metal from contaminated soil. All lead concentrations were treated with 0.1M EDTA; a higher molarity of EDTA could have been used for higher lead concentrations. The removal efficiencies were 86% and 91% for lead concentrations of 0.4% and 0.6%, but only 41%



and 9% for concentrations of 0.8% and 1.0%. The low removal rate could be due to the formation of an insoluble compound on the soil surface or in the pore liquid.

pH VALUE

The pH of the soil is one of the most influential factors on the movement and transport of pollutants. Maintaining the pH level of the soil appears crucial. The changes in soil pH following remediation are depicted in Figure... From the anode to the cathode, the pH of the soil increased.

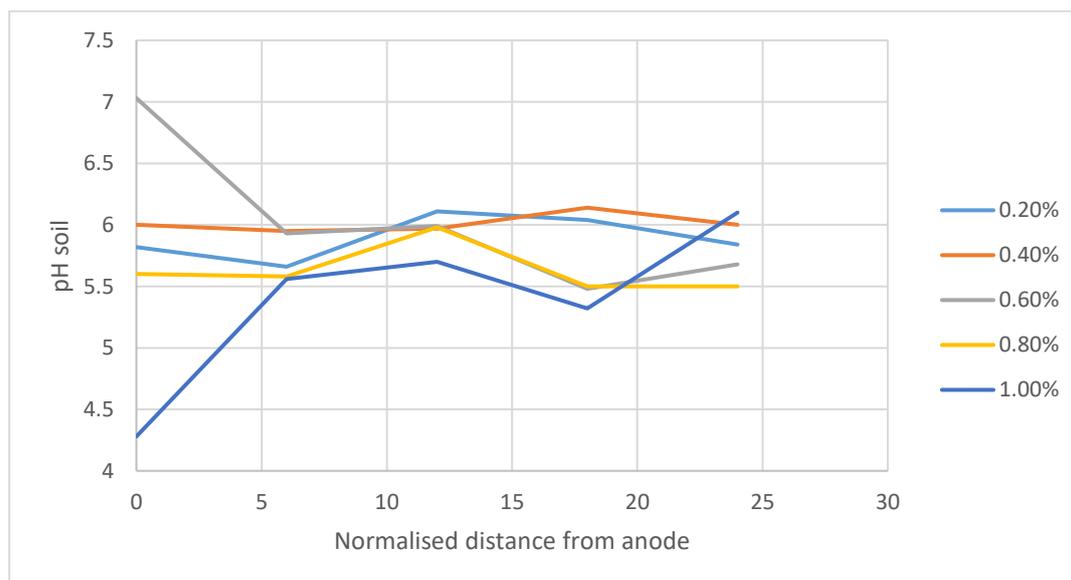


Figure 9: Soil Ph with normalized distance from the Anode

The anolyte produced H^+ , which acidified the soil surrounding the anode, while the catholyte produced OH^- , which alkalinized the soil surrounding the cathode. The pH values of the soil were kept between 6.5 and 7.0. However, the final pH values were greater than the soil's initial pH value prior to remediation. During the remediation process, H^+ produced by the anode migrated from the anode to the cathode as a result of electromigration. This process resulted in a gradual decrease in soil pH levels. It aided in the desorption and elimination of heavy metals. Kamal et al., 2021.

Conclusion

According to the findings of the studies that were conducted, there is a correlation between an increase in the lead concentration and an increase in the maximum dry density. The maximum dry density reaches its highest point at 0.6%, and after that, there is a reduction in the BSL, WAS, and BSH values. The electrokinetically treated soil exhibited a significant increase in dry density across the board, regardless of concentration. Because of this, electrokinetic technologies are a viable option for enhancing the engineering features of problematic soils.

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