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Metals Removal from Contaminated Soil Using Electrokinetic Treatment – Effect of Different Permeable Reactive Barrier and Flushing Solution

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ABSTRACT

Metals pollution is often found in the immediate or neighboring areas of industrial or agricultural activities. This situation may significantly affect the environment, such as water, soil, and air pollution. Electrokinetic (EK) treatment is known to have higher efficiency for metals contaminated soil. However, the use of EK treatment is not widely as expected. This study employs EK treatment with different permeable reactive barriers and flushing solutions to remove Cd and Pb from agricultural soil. Soil pH, temperature, water content, electroosmotic flow, electric current, and metal concentration are calculated as responses to the EK treatment. Results showed that the EK treatments were effectively removed the metals from the contaminated soil. On the fifth day of the treatment, EK, which used activated carbon as PRB and citric acid as the chelating agent, removed a significant amount of Cd from the soil. Besides, the treatment using zeolite as PRB and citric agent as chelate can remove more than 90% of Pb after the sixth day of treatment. These results showed that PRB and chelating agents could effectively remove the metals from the contaminated soils.

Keywords: activated carbon, citric acid, electrokinetic treatment, permeable reactive barrier, soil flushing, zeolite.

INTRODUCTION

Soil contamination by heavy metals needs particular attention because of the high risk for living things [Moghadam et al., 2016]. Heavy metal pollution in soil comes from nature, industry, transportation, and agricultural activities [Yin et al., 2021]. Heavy metals have properties that are difficult to degrade but are easy to mobilize and dissolve [Moghadam et al., 2016;Wang et al., 2021]. Therefore, its presence in the soil can potentially enter the food chain and even accumulate in the long term in the bodies of living things [Wang et al., 2021]. Lead (Pb) and cadmium (Cd) are classified as non-essential elements in the human body, which can endanger health if the levels accumulate. Pb and Cd toxicity causes diseases such as kidney dysfunction, hemoglobin deficiency, neuropathy, bone demineralization, hypertension, and even cancer [Priyadi et al., 2013]. Cases of heavy metal contamination of Pb and Cd are commonly found in Indonesia, including on agricultural land in Denpasar [Siaka et al., 2016]; Jelegong rice fields, Rancaekek, Bandung Regency

[Komarawidjaja, 2017]; and many coastal areas in Indonesia [Arifin et al., 2012]. The two heavy metals are widely used in commercial activities [Purnamawati et al., 2015].

Various techniques have been developed to remove heavy metals in the soil, including physical separation, chemical washing, immobilization, phytoremediation, and electrokinetic remediation [Xie et al., 2021]. Electrokinetic remediation is one of the most developed techniques today. This method is effective for soils with low permeability and does not cause damage to the soil [Gong et al., 2018]. In its development, this method is combined with a permeable reactive barrier (PRB) which is expected to reduce and assist the degradation of heavy metal content in the soil [Wen et al., 2021]. Activated carbon is one of the most widely used PRB materials. This condition occurs because of its high efficiency at a reasonably affordable price [Mehdinia et al., 2012]. Activated carbon can help the migration of heavy metals by absorbing the heavy metals and their precipitation which can block the migration path of the desorbed heavy metals [Li et al., 2021]. Besides, zeolite could also be used as an adsorbent of some ionic compounds [Waqas et al., 2019]. Gill et al. reviewed that using zeolite as PRB effectively removed Cr^{6+} from contaminated soils, achieving 60% removal efficiency [Gill et al., 2014]. However, zeolites can also absorb some moisture which may increase the electrical resistance in the EK process [Conrardy et al., 2016; Zhou et al., 2020]. Some additional understanding of the use of activated carbon and zeolite as PRB materials is needed to achieve a better EK remediation process.

Citric acid belongs to the group of organic acids with properties that are easier to degrade and are environmentally friendly [Wang et al., 2021]. This chemical solution is most often used in electrokinetic remediation processes. According to research by Zhou et al. and Silva et al., 0.1 M citric acid as an electrolyte was proven to have the most optimal heavy metal removal efficiency [Zhou et al., 2020; Silva et al., 2018]. The same thing is shown by research conducted by Li et al. that 0.07 M citric acid effectively removed Cd heavy metal contamination in the soil [Li et al., 2020]. Other studies, such as Xu et al., used 0.1 M citric acid as a buffer (buffer solution) as a pH control that can help remove heavy metals during the electrokinetic remediation process [Xu et al., 2020]. The need for the development of EK technology is increasing every year. Some improvements are needed to get a good combination of EK-PRB and chelating agents for treating metals contaminated soil.

This study aims to analyze the effect of activated carbon and zeolite as PRB and citric acid as the electrolyte in the electrokinetic remediation process. This study will also see the changes in the soil pH, temperature, water content, electroosmotic flow, and electric current due to the effect of the electrical field. The removal efficiency of Cd and Pb are evaluated to get the best configuration of the EK treatment. This study is expected to contribute significantly to the development of EK technology, especially to removing metals from contaminated soils.

METHODOLOGY

Experimental design

The reactor was constructed using acrylic material with 40×9.5×12 cm of dimension. This reactor consists of 4 chambers: an overflow chamber, electrolyte chamber, soil chamber, and permeable reactive barrier chamber. The overflow chamber is used as a reservoir for taking the overflow of the electrolyte due to electroosmotic flow. The PRB zone consists of PRB material and deionized water (DW) in a ratio of 1:1 w/w. The soil chamber was divided into three consecutive areas, namely near cathode (NC), middle (M), and near anode (NA). The electrolyte chamber was used to preserve the electrolyte (citric acid and deionized water). The permeable reactive barrier (PRB) was placed near the cathode to enhance the removal of heavy metals. The electrodes were made from carbon which has dimensions of 8×8×0.3 cm^3. The carbon electrode was connected to a DC power supply (Zhaoxin RPS-3005DB). Whatman filter paper and perforated acrylic (0.4 cm of pore diameter) separated the electrolyte and soil chamber. This study employs an electric voltage of 20 V (1 V/cm) and a moisture content of ± 30% with a running time of 6 days. The detail of the reactor design can be seen in Figure 1.

The soil used in each variable was 1.5 kg with 0.1 M citric acid as the electrolyte. The soil samples were taken from the textile contaminated rice field in Surakarta City, Java Province, Indonesia. The physical test was conducted in the soil mechanics laboratory, Civil Engineering Department, Faculty of Engineering, Diponegoro

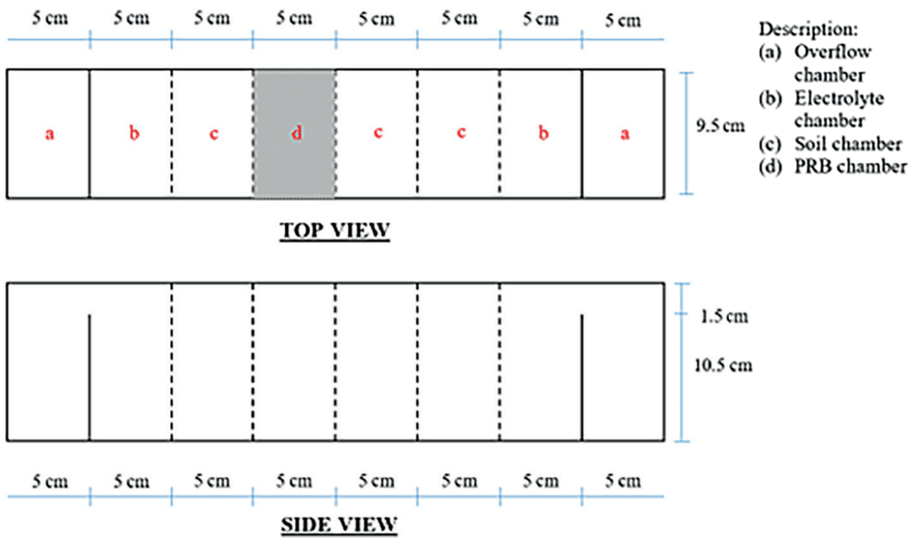

Figure 1. Reactor design

University. Soil chemical properties were analyzed in the Environmental Laboratory, Department of Environmental Engineering, Faculty of Engineering, Universitas Diponegoro. The soil was air-dried and crushed before it was used for the study. After the drying process, the soil was sieved in a 2 mm pore and mixed using cone and quartering methods to homogenize the soil samples [Ramadan et al., 2018]. There are 2 types of PRB materials used, namely activated carbon and zeolite. 3 different treatments are prepared in this study, which includes (1) the use of activated carbon – PRB (AC-PRB) with 0.1 M citric acid electrolyte; (2) zeolite – PRB (Z-PRB) with 0.1 M citric acid electrolyte; (3) activated carbon – PRB with the use of deionized water as the electrolyte. The dependent variable consists of soil pH, temperature, water content, electroosmotic flux, and heavy metal contents. The experimental procedures are detailed in Table 1.

Data sampling and analysis

As mentioned earlier, the soil chamber is divided into three sections used to see the pH, temperature, moisture content, and heavy metals dynamic concentration between the sections. All of the parameters are taken twice. At the same time, the electric current and electroosmotic flow (EOF) was recorded once a day. The pH and temperature were measured using an automatic measuring instrument, namely a soil meter (B1905). Soil samples from the reactor were taken daily in the three sections (NC, M, NA) to measure the water content and heavy metals concentration. Moisture content was measured using a moisture analyzer (Shimadzu MOC63u). Soil samples whose moisture content had been measured were reused for heavy metal content measurements using the spectrophotometric method (SNI 6989.8:2009 for Pb and SNI 6989.16:2009 for Cd) using the Atomic Absorption Spectrophotometer (AAS). The soil samples were weighed and digested using 9 mL concentrated HCl (37%) and 3 mL concentrated HNO_3 (65%), then heated using a magnetic stirrer for 30 minutes at 150°C. Furthermore, the digestion results were diluted in a 50 mL volumetric flask, filtered, and the final metal content was measured using AAS. In contrast, the EOF is measured based on the decrease in the anolyte volume in the anode chamber.

RESULTS AND DISCUSSION

Soil characteristic

The physical and chemical characteristics of the contaminated soil are shown in Table 2. The soil is dominated with silt as much as 52.46% and fine sand as much as 30.25%. The soil has an initial moisture content of 6.82% with a porosity of 41.94%. Soil moisture is one of the essential factors in conducting electrokinetic remediation because it can form the electromigration of contaminants through soil pores [Wang et al., 2021]. According to Zhou et al., the optimum soil moisture content in the electrokinetic remediation process is 30%, while according to Xu et al., the optimum soil moisture content is 40% [Zhou et

Table 1. Experimental procedure

Treatment	PRB Composition	Electrolyte
A	Activated carbon	Citric acid 0.1 M
B	Zeolite	Citric acid 0.1 M
C	Activated carbon	Deionized water

Table 2. Soil characteristics

Physical characteristics		Chemical characteristics
Water content (w)	6.82 %	Electrical conductivity
The specific gravity of solid (Gs)	2.6796	Cd concentration
Unit weight (ɣ)	1.6618 g/cm^3	Pb concentration
Dry unit weight ($ɣ_d$)	1.5558 g/cm^3	
Porosity (n)	41.94 %	
Void ratio (e)	0.72223	
Gravel	11.31 %	
Rough sands	5.98 %	
Fine sands	30.25 %	
Silt	52.46 %	
Clay	0.00 %	

al., 2020; Xu et al., 2016]. Therefore, this study set the initial soil moisture content to ± 30%, and citric acid was used as an electrolyte to maintain soil moisture. The electrical conductivity of the soil used was 135.4 Ω^{-1}, with an initial content of Pb in the range of 0.855–1.508 mg/kg and 60.6859–162.1582 mg/kg for Cd. The national limit of heavy metal contamination level of Pb in the soil is 100 mg/kg, while Cd is 0.5 mg/kg. The Cd and Pb concentration in the sample soil was detected higher than the national limit.

Changes in soil pH

Changes in soil pH in each treatment can be seen in Figure 1. Treatment A and B have a soil pH range between 4.5 to 5.5, while treatment C has a 5 to 5.5. Treatment C has insignificant pH

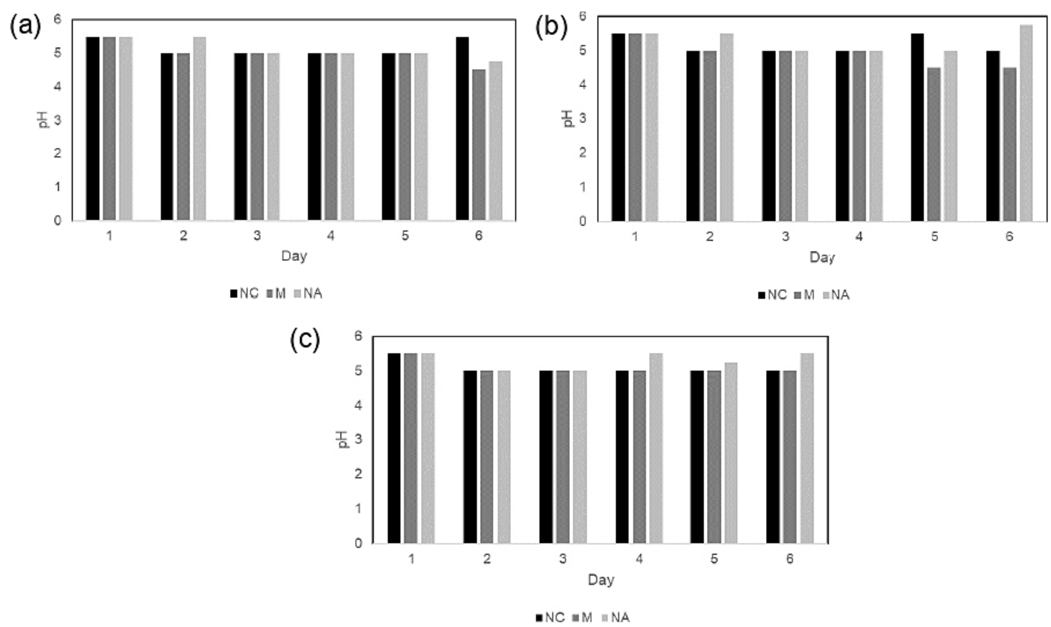

Figure 2. Changes in soil pH of (a) AC-PRB citric acid, (b) Z-PRB citric acid, (c) AC-PRB deionized water

changes because the electrolyte used is distilled water with a neutral pH. The soil pH decreased significantly on the first day and increased on the fifth day in the three treatments. The increase in pH generally occurs in the near cathode section. The mobility of H^+ ions caused a decrease in pH on the first day from the anode to the cathode [Xie et al., 2021]. The increase in pH was caused by a decrease in the volume of electrolytes in the anolyte chamber. This condition may be due to the EOF effect from the anode to the cathode. Thus, the chemical reaction at the anode side that produces H^+ ions (which are acidic) has decreased. At the same time, the chemical reaction on the cathode side that produces OH^- (alkaline) has increased [X. Li et al., 2020].

Changes in soil temperature

Soil temperature did not change significantly. In general, the soil temperature in the three treatments is influenced by room temperature. Soil temperature during the electrokinetic remediation process ranged from 27–30 °C. Based on the analysis results shown in Figure 3, the soil temperature increased insignificantly in the three sections on the three treatments on the third day of treatment. Soil temperature is affected by energy consumption and electrical conductivity. According to Nasiri et al., the precipitation reaction between hydroxide and carbonate during the electrokinetic remediation process causes electrical resistance in the soil, increasing energy consumption and soil temperature [Nasiri et al., 2020]. However, as shown in Figure 2b, citric acid increases the pH by about 1.5 °C on the 4th to the sixth day of the treatment. While on the other treatment, the average temperature of the soil was found to be steady or insignificantly changed compared to the initial temperature. Although the use of citric acid is not positively changing the soil temperature. Fu et al. said that citric acid might lead to soil heating and excessive use of energy [Fu et al., 2017]. This problem should be concerned when scaling up the treatment at the pilot or field scale.

Changes in water content

In this study, the water content did not reduce as the temperature did. The only reason why there are some differences between the anode and cathode sides is the electroosmotic flow. The application of a direct electric field produces water and colloidal movement from the cathode to the anode [Mena et al., 2016]. It is assumed that there is no significant contribution of the water reduction by the evaporation process as the temperature is not changing significantly [Ramadan et al., 2018]. Figure 4 showed that the water content in the Z-PRB (citric acid) treatment gets the highest reduction of water content in the near anode area. Therefore, all treatments showed the same trends where water in

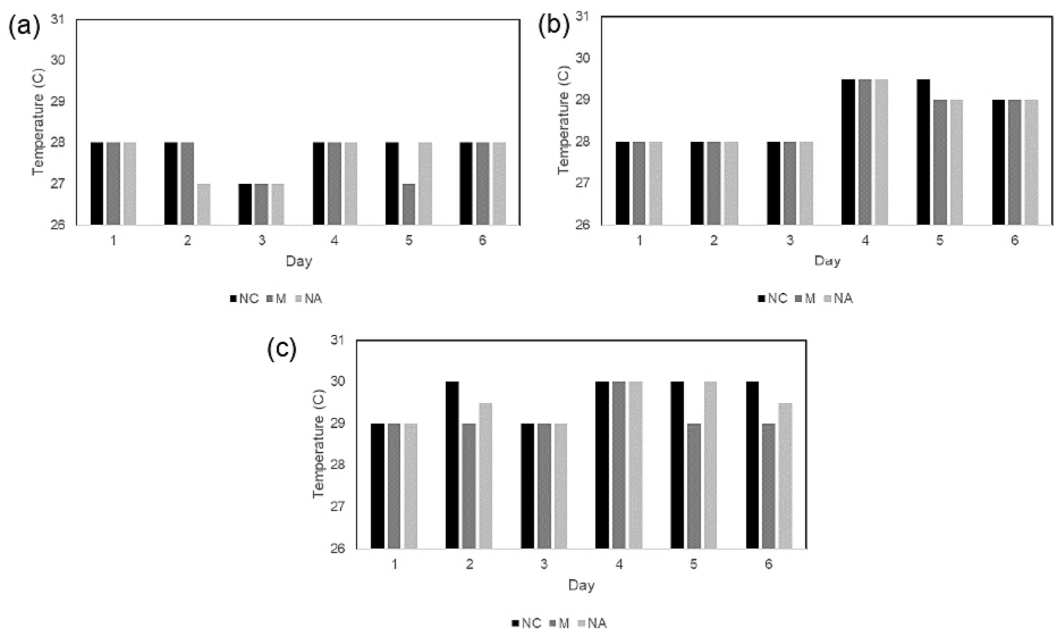

Figure 3. Changes in soil temperature of (a) AC-PRB citric acid, (b) Z-PRB citric acid, (c) AC-PRB deionized water

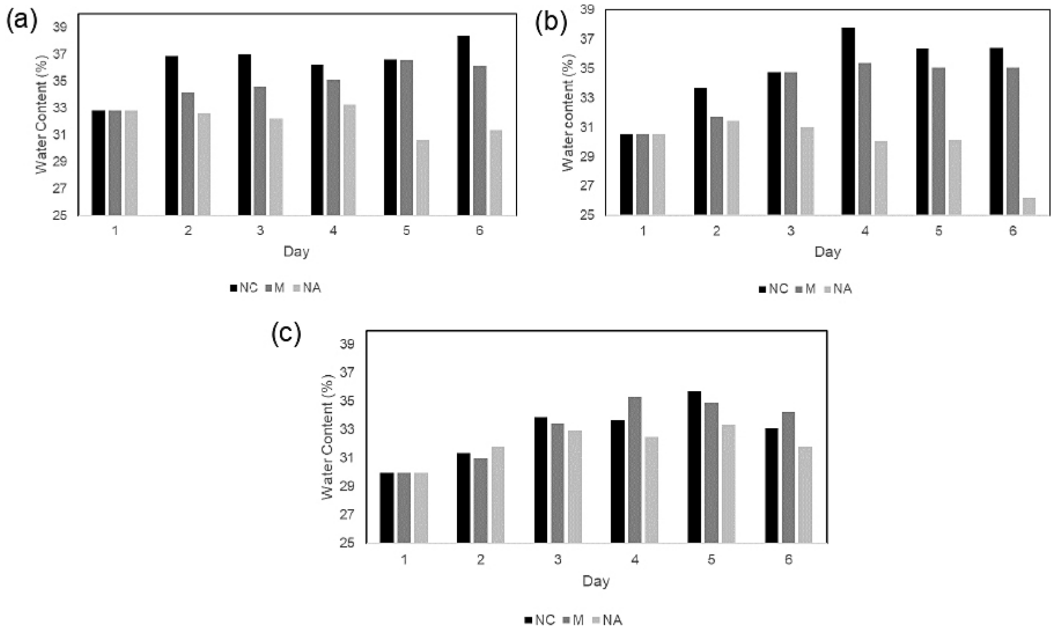

Figure 4. Changes in water content of (a) AC-PRB citric acid, (b) Z-PRB citric acid, (c) AC-PRB deionized water

the near cathode areas was reduced. Zeolite can absorb the water molecules, which may explain the significant reduction of water content in the Z-PRB reactor [Eroglu et al., 2017].

Changes in current and electroosmotic flow

Figure 5 showed high differences between the electroosmotic flow (EOF) and current density measured in the soil. Treatment A and B have a high current density which treatment C has a low current density. It seems that the use of citric acid and deionized water has a high implication on the current density produced. The use of citric acid proves to give a higher current which may imply to the EOF. However, there is a unique situation where the EOF A has the lowest electroosmotic flow. This condition may be due to activated carbon, which prevents the possibility of reverse EOF, which leads to a lower EOF rate [Kamal et al., 2021]. This result should be confirmed in future studies since this study cannot predict the exact reason behind this phenomenon. Even though using zeolite as PRB significantly reduces the water content, the electroosmotic flow is higher than the others, which may be responsible for the highest reduction of the metalloid component in the contaminated soil.

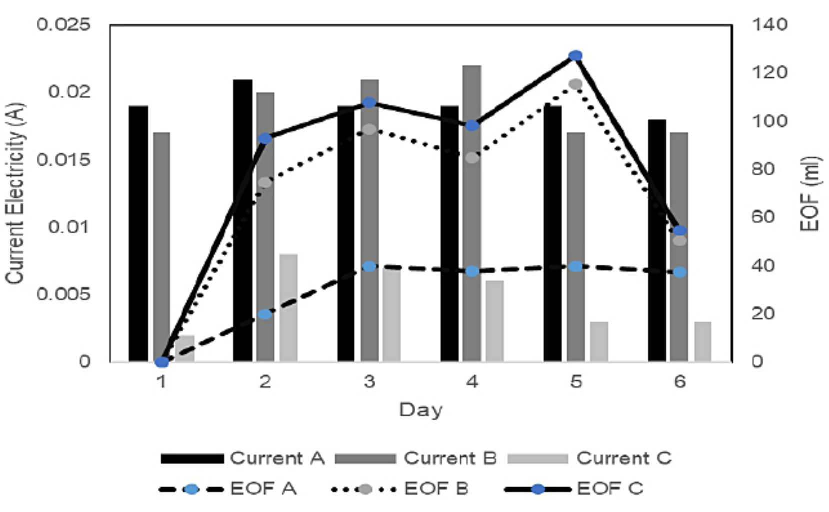

Figure 5. Changes in current and electroosmotic flow

Changes in heavy metals content

The treatments have different responses to the removal efficiency. As shown in Figure 6, the highest Cd removal efficiency was seen in the A treatment then followed by the B and C treatment. In this case, the activated carbon has successfully removed the Cd after the fifth-day treatment. Besides, the zeolite can also remove the Cd for about 88% of the initial average concentration. The DW can still remove the Cd but remain 30% of Cd in the soil after treatment. Thus, the combination of activated carbon and citric acid could be the best treatment so far.

Even though the A treatment gives a good result than the other treatment for removing the Cd, the B treatment took the highest removal efficiency of Pb. The B treatment has 90% removal

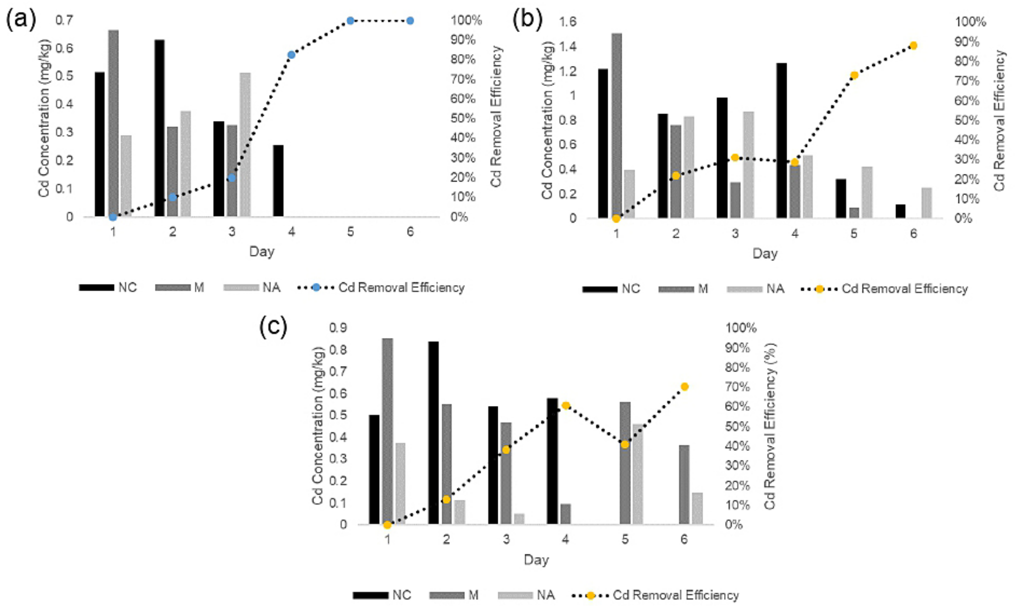

Figure 6. Changes in Cd concentration of (a) AC-PRB citric acid, (b) Z-PRB citric acid, (c) AC-PRB deionized water

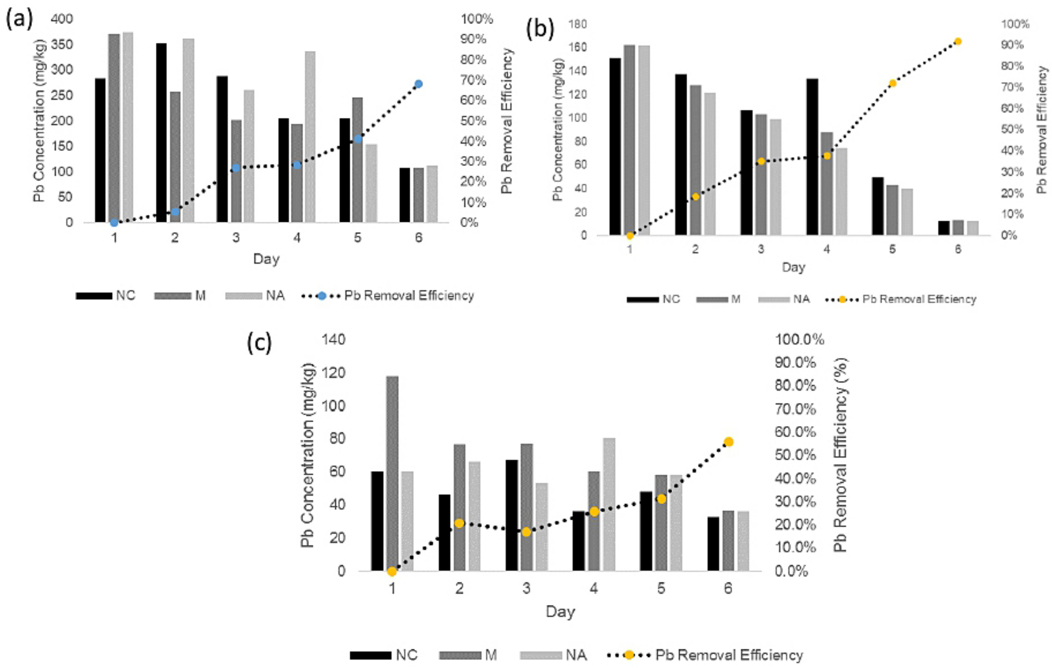

Figure 7. Changes in Pb concentration of (a) AC-PRB citric acid, (b) Z-PRB citric acid, (c) AC-PRB deionized water

efficiency, while the A and C treatment has only 68% and 56% removal efficiency. These results showed that the zeolite is likely worked better for Pb than Cd removal. Figures 6 and 7 show that the lowest metals component in the treatments is conducted in the cathode section. These results confirmed that electroosmosis and electromigration play an essential role in transporting chelating agents and ionic contaminants [Peng and Tian, 2010]. The electroosmosis transports the chelating agents through the soil pores. Then, the chelate detaches the metal's ion and brings it through the electroosmotic flow. The electromigration enhances the rate of transport, and the permeable reactive barrier entrapped and adsorbed the metals. Thus, the chelating agents can be recirculated for a sustainable remediation strategy [Nasiri et al., 2020]. Besides, citric acid as the chelating agent has been proved to work better than deionized water. The DW can only remove half of the initial concentration of Pb and Cd. The PRB can effectively be used for removing heavy metals contaminated soils. The DW treatments cannot produce higher efficiency because the electromigration limitation in DW treatments will hinder the possibility of the metals detaching from soil particles [Wen et al., 2021]. The use of both activated carbon and zeolite may produce higher efficiency of heavy metals removal. Citric acid and other chelating agents are also better solubilize the metals' compound to the PRB and electrolyte area [Zhao et al., 2016].

CONCLUSIONS

This study found that the use of activated carbon and zeolite as a permeable reactive barrier (PRB) and citric acid as chelating agents have been proved to remove heavy metals from naturally contaminated soil. The treatments do not give any significant changes in the soil pH, temperature, and water content. The soil pH is not affected significantly. This situation may be because the use of PRB can prevent drastic pH changes in the contaminated soil. The electrical resistance is not higher enough to induce temperature changes. Therefore, on a larger scale, these factors should be considered significant factors of overall performance. Using a chelating agent produces a higher EOF than deionized water since the chelating agent can increase the current density. Those results also imply the higher removal efficiency of heavy metals concentration in the soil. On the fifth day, the concentration of Cd is under detection of our instruments, and the concentration of Pb only remains 8% from the initial concentration. This result showed that the electrokinetic remediation using chelating agents and permeable reactive barrier could effectively remove heavy metals from contaminated soil. Future research should be focused on the development of pilot-scale studies which employ a PRB combination of zeolite and activated carbon to remove the heavy metals.

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