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Zeta potential of soils with surfactants and its relevance to electrokinetic remediation

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Abstract

There are numerous studies on the application of electrokinetic decontamination technique to remediate heavy metal contaminated fine-grained soils. In recent studies, surfactants have been used to increase the efficiency of contaminant removal. However, there is limited data available on how physicochemical parameters such as zeta potential (ζ) of soils changes in the presence of surfactants. Understanding the ζ potential variations of soils with surfactant addition is important because it controls the direction and magnitude of electro-osmotic permeability, which plays important role on the efficiency of electrokinetic remediation. In this study, ζ potentials of kaolinite, montmorillonite and quartz powder with Li^+, Ca^{+2}, Cu^{+2}, Pb^{+2} and Al^{+3} in the presence of anionic, cationic and non-ionic surfactants were determined. The results indicate that anionic surfactants produce negative ζ potentials. The other surfactants produce both positive and negative ζ potentials depending on soil type and ion present in the system. The results also indicate that the ζ potential of kaolinite and quartz powder with surfactants showed similar trends; however, the absolute magnitude of the ζ potential of quartz powder is higher than that of kaolinite. The ζ potential of montmorillonite commonly shows a different trend from those of kaolinite and quartz powder. Based on the test results, it is recommended that ζ potential of soils be determined before the electrokinetic decontamination in order to maximize the efficiency of the technique.
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1. Introduction

In recent years, electrokinetic remediation has been developed to remove organic and inorganic compounds from contaminated soils [1–3]. The electrokinetic remediation technique involves installing trenches and/or wells to encompass the contaminated zone, inserting electrodes into the trenches and/or wells. A voltage gradient is created across the electrodes or direct current (dc) is passed through the contaminated soil. As a result of the induced electric potential, the contaminants are transported towards either the cathode or the anodes depending on their charge (cationic or anionic) and the direction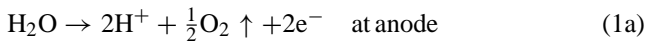 of the pore water flow. Contaminates collected at the electrodes are then extracted and subsequently treated. Using the electrokinetic technology, inorganic and organic contaminant compounds can be removed from fine-grained soils [4–6].

Although the principle of electrokinetic decontamination is fairly simple and electrokinetic remediation has been applied to many bench scale laboratory as well as several small-scale field tests, physicochemical interactions that occur simultaneously at soil–liquid–contaminant interface are not well understood. The simultaneous interactions taking place are rather complex and require a better understanding of electrokinetic properties of soil–contaminant interaction. This is especially so because the specific surface area of fine-grained soils is high and it provides a large number of active sites for soil–contaminant interaction [7]. Also more complication arise because, the soil-contaminant interaction and
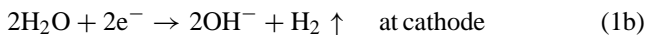
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changes in physicochemical properties at the soil–fluid interface are pH dependent.

The pH gradient during electrokinetic remediation between anode and cathode due to electrolysis of water is:

$$H_2O \rightarrow 2H^+ + \tfrac{1}{2}O_2 \uparrow + 2e^- \quad \text{at anode} \tag{1a}$$

and

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \uparrow \quad \text{at cathode} \tag{1b}$$

The reactions described in Eqs. (1a) and (1b), causes acidic pH at the anode and basic pH at the cathode. Such variations in pH activate changes in the physicochemical properties at the soil-liquid-contaminant interface and, may alter the electrokinetic properties of fine-grained soil. ζ potential is one of the physicochemical properties of fine-grained soils that shows variations with pH, which is the subject of this study.

Using the Helmholtz–Smoluchowski equation, the ζ potential of a particle in a suspension under an electric field is [8]:

$$\zeta = \frac{u\eta}{\varepsilon_0 \varepsilon E} \tag{2}$$

where u is the particle velocity, η the viscosity, ε the relative permittivity of the pore fluid, and ε_0 the permittivity of free space and E the field strength.

Researchers have indicated that ζ potential of fine-grained soils plays an important role in the efficiency of the electroremediation. This is due to the direction and rate of the electro-osmotic flow being ζ potential dependent as shown in Eq. (3).

$$k_{eo} = \frac{\varepsilon \varepsilon_o \zeta}{\eta} \frac{n_e}{\tau} \tag{3}$$

where k_{eo} is electro-osmotic permeability, which ranges between 10^{-9} to 10^{-8} m^2 V^{-1} s^{-1}, n_e the effective porosity and τ the tortuosity.

During electrokinetic decontamination remediation, all parameters in Eq. (3) remains constant except ζ potential. Although the ζ potential of fine-grained soils is negative, it is pH dependent and subsequently may show positive values. The pH value at which soil surfaces have no net charge is called point zero charge, *pzc*. The variations in the ζ potential of fine-grained soils occur because positive ions balance the negative charges that occur as a result of isomorphous substitution and broken bonds present on soil particles.

Eykholt and Daniel [9] reported that the pH at anode can be as low as 2, and as high as 12 at the cathode. Such pH gradient during electrokinetic remediation creates a ζ potential gradient; which may ultimately control the efficiency of the electrokinetic treatment. Several researchers reported reverse electro-osmotic flow during heavy metal skipped kaolinite [9–12]. The observed reverse electro-osmotic flow was explained by citing to sign reverse in

the ζ potential of kaolinite. They verified their results using the data reported by Lorenz [13]. The offered explanation for reverse electro-osmosis flow may not hold true for all soils or contaminates with different ion species, since the data used to explain the observed phenomenon was for one type of kaolinite. This is because ζ potential of soils varies not only with type of soil and pH of the solution, but also type and concentration of ions present in the solution [14].

As previously noted, the interaction of soil surface–fluid–contaminant may produce numerous simultaneous reactions such as electrolysis, acidification, adsorption and precipitation. Electrically active and large specific surface areas of fine-grained soils further complicate the reactions that occur during electrokinetic treatments. Thus, understanding the reactions and interactions at the soil–liquid–contamination interface is required to improve the removal efficiency. For example, contaminants that are adsorbed on the soil or are present as precipitates or immiscible liquids cannot be effectively removed by an electrokinetic remediation technique unless they can be absorbed into the aqueous phase. The solubility of heavy metals may be considerably reduced at basic pH values. Surfactants are introduced to increase the solubility and mobility of heavy metals during electrokinetic remediation. Several researchers reported enhanced electrokinetic remediation efficiency by introducing surfactants [15–18]. This is because surfactants have an effect on the electrical double layer interactions and Van der Waals interactions. Ionic surfactants induce electrostatic interactions, but non-ionic surfactants are adsorbed by only steric interactions [19].

Previous researchers indicated the significance of ζ potential of soil on electrokinetic remediation efficiency, there are a large number of studies on effect of surfactants on zeta potential of soils [20–22], however, these studies either investigate one soil type (mostly kaolinite) with one type of surfactant and/or different surfactants with one type soils. None of these studies systematically compares the ζ potential of different soils with different surfactants and cations. The objective of this paper is to determine the ζ potential of kaolinite, montmorillonite, and quartz powder in the presence of alkali, alkaline earth, hydrolysable metal ions with anionic, cationic and non-anionic surfactants in a systematic manner. We believe that such data will help to understand the electrokinetic properties of soils with surfactants, and shed light on how surfactants enhance the efficiency of electrokinetic decontamination.

2. Materials and methods

Kaolinite, montmorillonite, and quartz powder were used in the experiments. Kaolinite was obtained from Evans Clay Company, Georgia, USA, and montmorillonite was obtained from Clay and Clay Mineral Repository, Missouri, USA. Quartz powder was obtained by crushing Ottawa sand. Cation

Table 1
ICP analysis of the kaolinite and montmorillonite

Sample	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	MgO (%)	CaO (%)	Na_2O (%)	K_2O (%)
Kaolinite	46.93	35.47	0.87	0.1	0.04	0.09	0.21
Montmorillonite	59.47	18.64	3.47	2.24	0.55	0.38	0.3

exchange capacities of kaolinite and montmorillonite were found to be 2.2 and 104.4 mequiv./100 g, respectively using the Na-method [23]. X-ray diffraction analysis showed the kaolinite consisted of 90–95% kaolin and 2–3% quartz and the montmorillonite consisted of 85–90% montmorillonite and 5–7% quartz. The ICP analysis test results of the kaolinite and montmorillonite are given in Table 1. The samples were diffused with lithium metaborith at 1000 °C. Entirely molten samples were poured 5% nitric acid and 1.5% hydrofluoric acid, with, samples shaken for about 30 min before analyzing chemical composition.

The samples were purified by washing ammonium acetate ($CH_3CO_2NH_4$) several times before any ζ potential measurements were conducted. The washing process was as follows: as-received sample was prepared by mixing 2 M ammonium acetate for 15 min at a solid to liquid ratio of 1:2, diluting with distilled water to 2–3% solids, allowing it to settle, and discarding the supernatant. Measuring the electrical conductivity in which until negligible changes occurred in the supernatant controlled the quality of the purified samples. The washed sample dried at 80 °C for over 48 h. All samples were passed through 75 μm sieve.

Alkali salt ions and hydrolysable metal ions used in the present work were LiCl, $CaCl_2 \cdot 2H_2O$, $CuCl_2$, $AlCl_3$, and $Pb(NO_3)_2$, all from Merck Chemical. The surfactants used were anionic surfactant, sodium dodecyl sulfate, (SDS), ($C_{12}H_{25}NaO_4S$) (MW = 288.4, from Fluka), cationic surfactant, dodecylamine hydrocloride, (DAH), ($CH_3(CH_2)_{11}NH_2 \cdot HCl$) (MW = 221.8, from Kodak) and non-ionic surfactant polyethleyene oxides (Brij 30) ($C_{12}H_{25}(OCH_2CH_2)_4OH$) (MW = 362.6, from Aldrich Chemie).

Selection of these surfactants was based on the fact that these surfactants are extensively used as model surfactants in research studies and are also the main ingredients of many typical surfactant products. For example, SDS is a main ingredient used in detergents and shampoos, DAH is a primary amine extensively used in flotation, and PEO is a typical wetting agent used for cleaning and emulsifying. Brij 30 is a well-known non-ionic surfactant used as an emulsifier and wetting agent.

The ζ potential of the soil particles were measured using an automated electrophoresis instrument (Zeta Meter 3.0+, Zeta Meter Inc., New York), equipped with a microprocessor unit, which enabled all statistical calculations to be made on the sample. The ζ potential was measured in accordance with Eq. (2).

For the ζ potential measurements, a 50 mg sample was transferred into aqueous solution and the soil particles mixed homogenously with a magnetic stirrer. The measurement of ζ potential of soils with different solid concentrations showed that the ζ potential changes slightly up to a concentration of 100 mg/L, and then remains constant. All the measurements were conducted therefore at 100 mg/L concentration. Furthermore, the time versus pH results indicated that the pH of the solution stabilizes after 10 min. The pH of the solution was adjusted by a dropwise addition of HCl and NaCl solutions. The solution was stirred using a magnet until the pH of the solution became constant. The pH of the solution was measured before and after each measurement. If changes occurred in pH, the last one was recorded as the pH of the solution. The reliability of the ζ potential measurements was determined using the standard deviation of the readings. Standard deviations are calculated by the zeta-meter, obtained from the readings. The standard deviation of each measurement was <2 mV, which is automatically calculated by the instrument. The ζ potential of at least six particles for each sample was determined and their average was taken. Temperature of the room was 22.5 ± 2.5 °C. Further details of zeta potential measurements are described by Yukselen and Kaya [24].

3. Results

The ζ potentials of soils are presented without ions, ions and surfactants that can be used for comparing the effect of surfactants. Throughout the discussion of the results effects of surfactants are always compared with zeta potential of soils with 1×10^{-3} M respective ion concentration.

### 3.1. Effect of surfactants on the ζ potential of soils

The ζ potentials of the soils were determined in several cationic, anionic and non-ionic surfactant concentrations to evaluate the effect of surfactant concentration on ζ potential (Fig. 1a–c). As can be seen from Fig. 1, the marked variations in the ζ potentials with surfactant type occur at 1×10^{-3} M concentration. All measurements were conducted at 10^{-3} M surfactant concentration with 1×10^{-3} M alkali and hydrolysable metals for consistency and to facilitate comparison of the effects of different cationic types.

We also determined the pH of surfactant solutions as a function of surfactant concentrations. Table 2 lists the pH of the surfactants when they were added into DDI water and soil. Note that pH of the surfactant, as expected, slightly varies with surfactant and soil types and surfactant concentration.

Table 2
pH of soils at different concentration of surfactants

Surfactant concentration (M)	pH								
	Kaolinite			Montmorillonite			Quartz		
	Cationic	Anionic	Non-ionic	Cationic	Anionic	Non-ionic	Cationic	Anionic	Non-ionic
10^{-2}	5.56	6.46	4.45	5.70	6.78	5.92	6.00	6.50	4.36
10^{-3}	5.82	5.84	5.71	6.3	6.64	6.85	5.95	5.81	5.20
10^{-4}	5.96	5.79	5.85	6.85	6.54	6.86	6.03	5.53	5.60
10^{-5}	6.22	5.82	6.00	6.88	6.52	6.68	5.88	5.60	5.68

Table 2 indicates that the pHs of three soils are slightly to moderately acidic in the presence of surfactants. The pHs of the soils are more acidic with non-ionic surfactants than those with anionic and cationic surfactants. Note also that, in general, increase in surfactant concentration slightly alters the pH of the solutions.
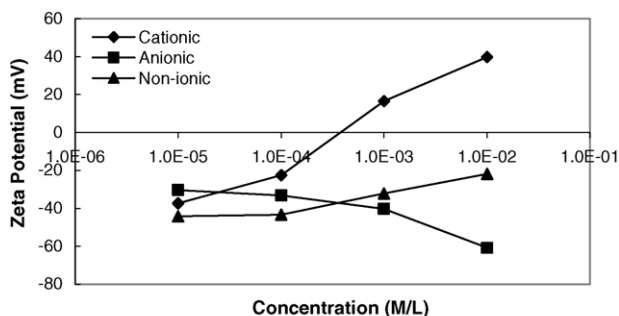
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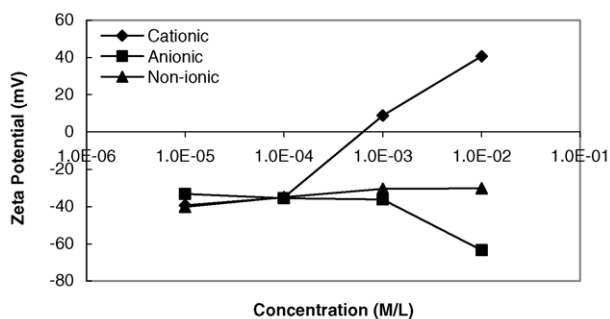
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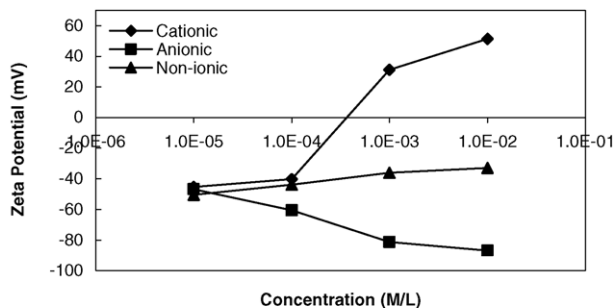
(c)

Fig. 1. Variation of the ζ potential of kaolinite and montmorillonite with surfactants concentrations: (a) kaolinite, (b) montmorillonite and (c) quartz powder.

Fig. 1a–c also show that kaolinite, montmorillonite and quartz, have negative ζ potential values with anionic and non-ionic surfactants at all concentrations. The soils in the presence of cationic surfactant up to 1×10^{-4} M concentrations have negative ζ potential values. Above 1×10^{-4} M concentration, the ζ potential becomes positive, indicating that soil surfaces are completely covered by positive ions. As expected, as the concentration of anionic surfactant increases, the ζ potential of soils become more negative. The results also reveal that non-ionic surfactants cause the soils to have a slightly less negative ζ potential. The decrease in ζ potential becomes more pronounced with the increase in surfactant concentration. However, changes in the ζ potential magnitude with non-ionic surfactant are less than those of cationic and anionic surfactants.

3.2. The effect of alkalis

The ζ potential of soils with LiCl at 1×10^{-3} M concentration as a function of pH in the presence of surfactants are shown on Fig. 2a–c. In Fig. 2, the ζ potential values of soils in water are also plotted for comparison purposes. In general, the ζ potentials of soils follow a similar trend although the magnitude varies, except when montmorillonite with cationic surfactant is present. When a cationic surfactant is present in the system, both kaolinite and quartz have positive ζ potential, which seems to remain constant for all pHs where measurements were conducted. The reason for the almost constant ζ potential is that surface is saturated by cationic surfactants. The ζ potential of montmorillonite is only positive in an acidic environment (pH 4) and becomes increasingly negative as pH becomes basic.

Addition of anionic surfactant makes the ζ potential of both kaolinite and quartz more negative than those with water and water with 1×10^{-3} mol/L LiCl. Changes in zeta potential can be explained by mechanisms taking place between the clay particle and anionic surfactant. Clay surfaces OH^{-} ions can exchange anionic part of the surfactant, second H-bonds can form between clay particles and surfactant molecules. Third, present cation (here Li^{+}) can establish electrostatic bridges between the anionic part of the surfactants and the surface of clay [19]. Here, in the presence of Li^{+} cations it is expected that third mechanism occurs. As a result, ζ potential becomes more negative in the presence of anionic surfactant.
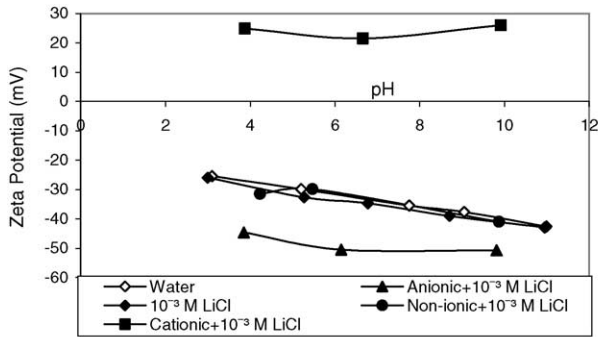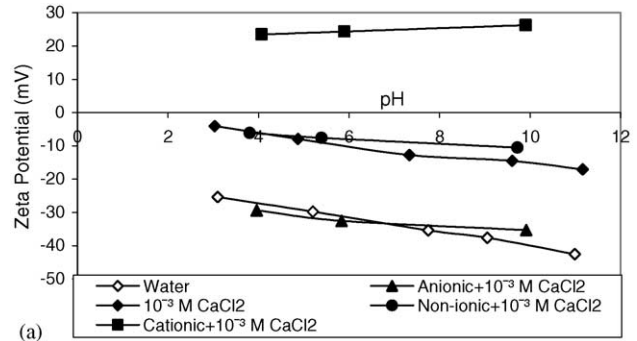
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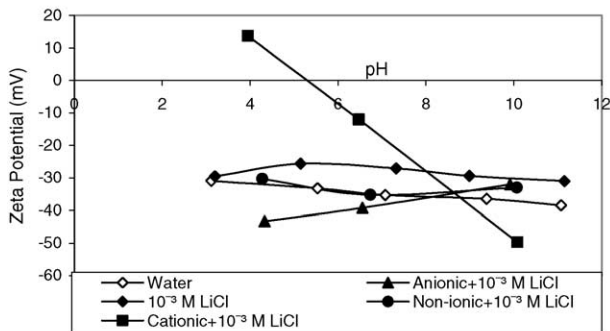
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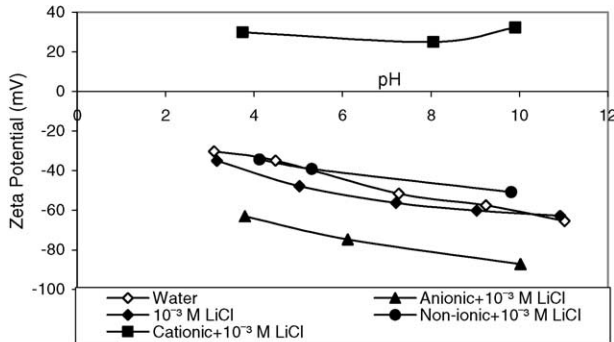
(c)

Fig. 2. Effect of pH on ζ potential of soils with 10^{-3} M LiCl in the presence of 10^{-3} M surfactant concentrations: (a) kaolinite, (b) montmorillonite, and (c) quartz powder.
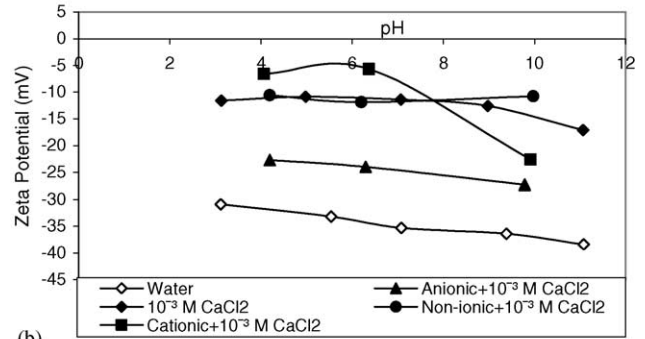

(a)

(b)

(c)

Fig. 3. Effect of pH on ζ potential of soils with 10^{-3} M CaCl$_2$ in the presence of 10^{-3} M surfactant concentrations: (a) kaolinite, (b) montmorillonite, and (c) quartz powder.

Fig. 3a–c shows the ζ potential of soils with CaCl$_2$ and surfactants. In general, the ζ potentials of soils in the presence of 1×10^{-3} M CaCl$_2$ with surfactants are similar to those with 1×10^{-3} mol/L LiCl. However, the magnitude of zeta potential of soils with Ca^{+2} is less than that of Li^{+}.

3.3. The effect of heavy metals

Since most of electrokinetic decontamination is applied to remove heavy metals such as Pb^{+2}, Cu^{+2}, Zn^{+2}, etc., it is essential to determine how zeta potential of soils with these hydrolysable metal ions and surfactants changes. By determining the effect of surfactants on the ζ potentials of soils with hydrolysable metals, it is possible to enhance heavy metal removal during electrokinetic decontamination by introducing surfactants to the system. The ζ potentials of soils with heavy metals in cationic, anionic and non-ionic surfactants were determined and presented in Figs. 4–6. It was not possible to determine the ζ potential of the soils at pH ~ 7 because surfactants precipitated with metal ions. Only the ζ potentials of soils in acidic pHs (pH < $\sim$6) and basic pHs (pH $\sim$ 10) could be determined.

Fig. 4a and b shows the ζ potential variations of soils with anionic, cationic and non-ionic surfactants with 1×10^{-3} M CuCl$_2$ for kaolinite, and montmorillonite, respectively. Cationic surfactant produces positive ζ potential
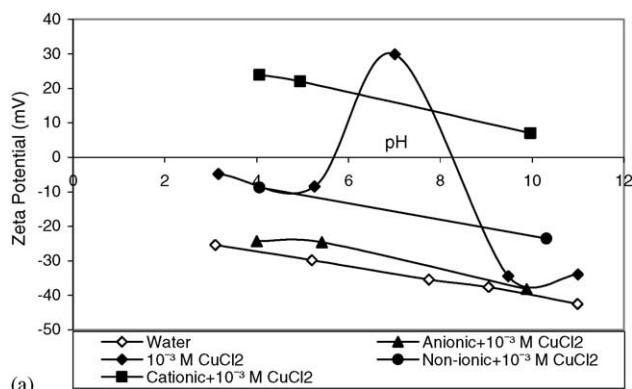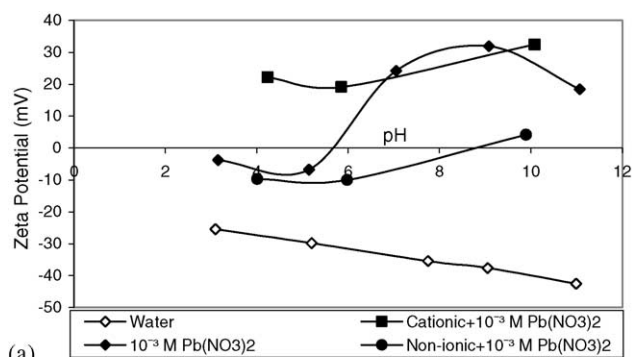
(a)
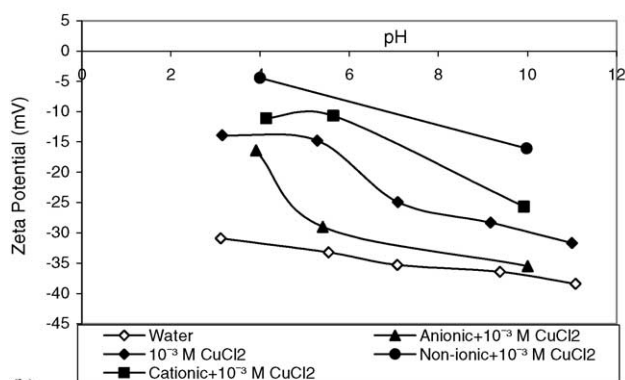
(b)

Fig. 4. Effect of pH on ζ potential of soils with 10^{-3} M CuCl$_2$ in the presence of 10^{-3} surfactant concentrations: (a) kaolinite and (b) montmorillonite.
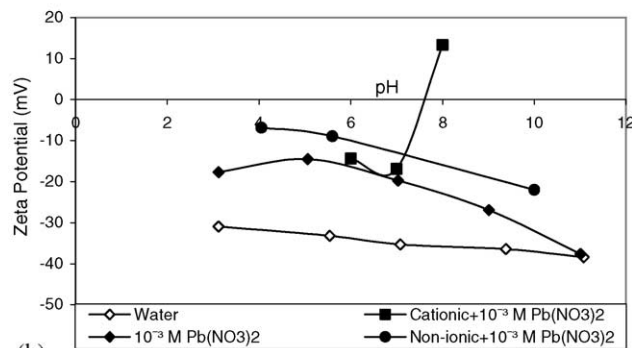

(a)

(b)

(c)

Fig. 5. Effect of pH on ζ potential of soils with 10^{-3} M Pb(NO$_3$)$_2$ in the presence of 10^{-3} M surfactant concentrations: (a) kaolinite, (b) montmorillonite, and (c) quartz powder.

with kaolinite for both acidic and basic pHs, whereas it produce negative ζ potential with montmorillonite. Addition of 1×10^{-3} M anionic surfactant produces almost the same ζ potential as water, whereas addition of non-ionic surfactant produces negative ζ potentials for both kaolinite and montmorillonite. However, the ζ potential of montmorillonite with non-ionic surfactant becomes less negative.

Fig. 5a and b shows the ζ potential of kaolinite and montmorillonite in the presence of cationic and non-ionic surfactants and 1×10^{-3} M Pb(NO$_3$)$_2$. It was not possible to determine the ζ potential of all samples with anionic surfactant due to precipitation (which occurred even in the absence of soils). Cationic surfactant produces positive ζ potentials for kaolinite and quartz at all pH values for which measurements are taken, whereas for montmorillonite it produces negative ζ potentials for acidic pH (pH < 6) and positive ζ potentials for pH ∼ 8. The ζ potential of kaolinite with non-ionic surfactant with 1×10^{-3} M Pb(NO$_3$)$_2$ increases as the pH of the system becomes neutral and finally reaches positive values, whereas the ζ potential of montmorillonite with non-ionic surfactant becomes more negative as the pH of the system increases.
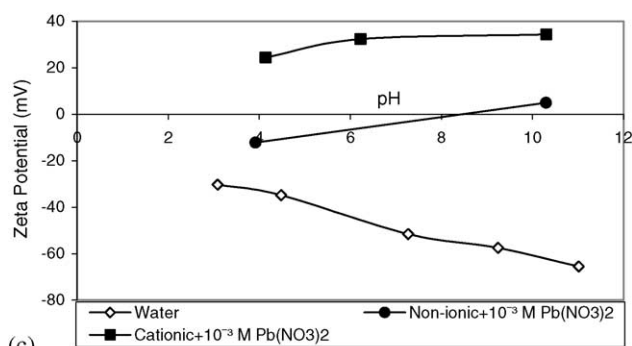
Fig. 6a–c shows the ζ potential of soils with surfactants in the presence of 1×10^{-3} M AlCl$_3$. As can be seen from the

figures, addition of cationic surfactant causes soils to have positive ζ potential values at acidic pHs; however, the magnitude of the ζ potentials slightly decrease when the soils are kaolinite and montmorillonite. Addition of cationic surfactant to kaolinite and quartz in the presence of AlCl$_3$ keeps the ζ potential positive in both acidic and basic pHs, whereas its addition to montmorillonite makes the ζ potential positive in an acidic environment and negative in a basic environment. Furthermore, the addition of both anionic and non-ionic surfactants decreases the ζ potential in acidic environment; however, the magnitude of ζ potential is still higher than that of water. All soils produce negative ζ potential with both anionic and non-ionic surfactants in a basic environment.
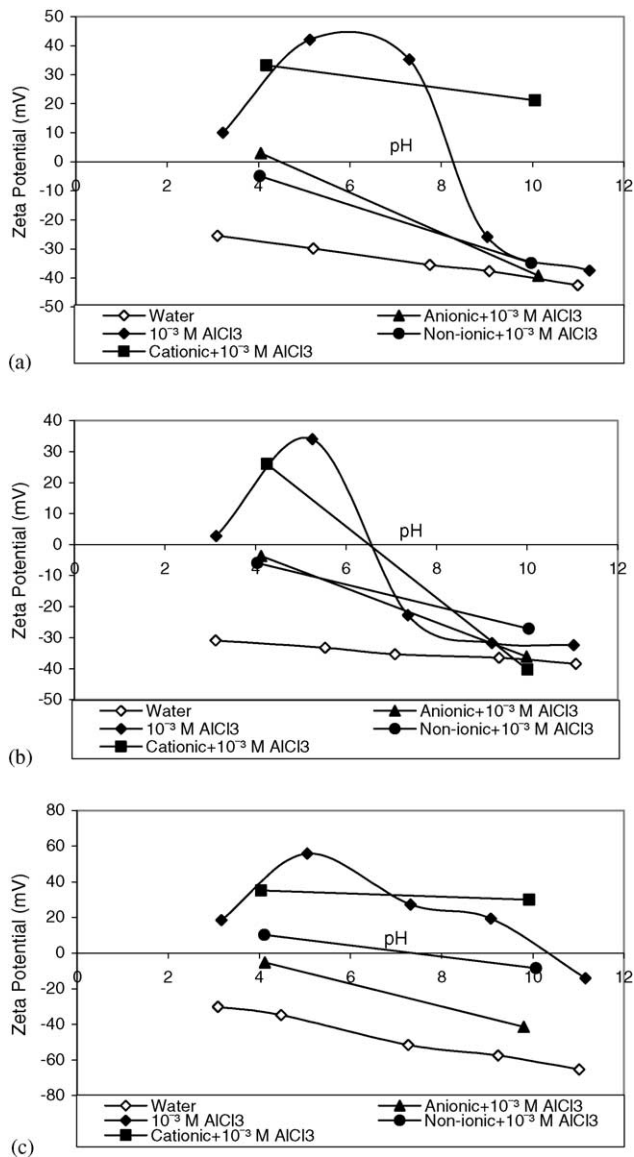
Fig. 6. Effect of pH on ζ potential of soils with 10^{-3} M AlCl₃ in the present of 10^{-3} M surfactant concentrations: (a) kaolinite, (b) montmorillonite, and (c) quartz powder.

4. Summary

Electrokinetic remediation is a promising technology for efficient clean up of contaminated soils. The technology becomes efficient especially when pump-and-treat technology is impractical for fine-grained soils. Early application of electrokinetic remediation used acetic acid to regulate the pH gradient across the electrodes [25,26]. However, recently researchers used surfactants to improve the contaminant removal efficiency [10,18]. As previously noted, several electro physicochemical reactions take place during electrokinetic remediation. ζ potential is a parameter used to asses the electro physicochemical reactions taking place at the soil–liquid–contaminant and surfactant interface. The

results of this study, based on ζ potential measurements, indicate that complex interactions are taking place at the interface of soil–liquid–ion–surfactant such as precipitation of both heavy metals and surfactants. These interactions depend on soil, surfactant, heavy metal ion types and the pH of the system.

In general, it can be stated that the presence of cationic surfactant significantly decreases (makes more positive) the ζ potential of soils in an acidic environment (pH ∼ 4). The presence of the anionic surfactant makes the ζ potential of the soils more negative in comparison with the measurements in water + alkali and alkali earth metal ions. However, the non-ionic surfactant has little effect on the ζ potential of the soil. Similar observations were made by Watson and Tuzinski [27] who found that the anionic additives produced more negative ζ potential than water without attaining a *pzc* condition. Non-ionic additives produced either no effect on the ζ potential or produced a more negative potential depending on ion and soil type. For example, the non-ionic surfactant increased the ζ potential of kaolinite with Pb(NO₃)₂, whereas it decreased that of montmorillonite. On the other hand, the non-ionic surfactant decreased the ζ potential of kaolinite with AlCl₃. In general, the pH of the solution affects the ζ potential of soils with surfactants, but not as much as when there is no surfactant in the solution. Furthermore, the ζ potential of kaolinite and quartz in the presence of surfactant and ions show similar trends although the magnitude may vary depending on soil surface charges.

The practical implications of ζ potential measurements with various ions and surfactants in electroremediation of contaminant soils are that ζ potential of the soils should be determined using the contaminants targeted to be removed and the surfactants to be employed. Zeta potential measurements with targeted ions and surfactants will help to not only understand the electro physicochemical reactions at the interface but also to manipulate such interaction to improve the efficiency of the electrokinetic remediation. This is important to prevent precipitation of heavy metals and surfactants during the treatment. It is also essential to keep ζ potential of soils as low as possible with ions and surfactants without allowing the ions to precipitate near by electrodes in order to increase the electro-osmosis permeability; thus, efficiency of the electrokinetic remediation according to Eq. (3).

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