ELECTROOSMOTIC FLOWS AND ELECTROMIGRATIONS DURING ELECTROKINETIC PROCESSING OF TROPICAL RESIDUAL SOIL

Kamarudin Ahmad^{1,*}, Khairul Anuar Kassim¹, Mohd Raihan Taha²

 ¹Faculty of Civil Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia
 ² Department of Civil and Structural Engineering, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor Darul Ehsan Malaysia

*Corresponding author: kamarahmad@utm.my

Abstract: Saturated soft soils can be strengthened by several methods such as dewatering or adding suitable chemicals. Introduction of chemicals through electrokinetic (EK) process to strengthen a residual soil was investigated. The results of chemical tests on an elecrokinetically-treated tropical residual soil are presented. The chemical substances used as electrolytes in the experiments were aluminum chloride (Al), calcium chloride (Ca) and phosphoric acid (PA) in addition to distilled water (DW). Four different opened-anode and opened-cathode electrokinetic systems were employed in this study, namely DW-DW, Al-DW, Ca-DW and DW-PA. A constant voltage of 30 V was applied for 168 hours to each of the opened-anode and opened-cathode configuration system. It was observed that the quantities and directions of electroosmotic flows were dependent upon the types of electrolyte. The chemical contents of the treated samples were position dependent and influenced by both the electrolyte types and electroosmotic flows.

Keywords: Residual Soil; Electrokinetics; Electroosmosis; Electromigration

Abstrak: Tanah tepu lembut boleh diperkuatkan melalui beberapa kaedah seperti menyahair atau mencampur bahan kimia yang sesuai. Penggunaan proses elektrokinetik (EK) untuk memasukkan bahan kimia ke dalam tanah bagi menguatkan satu tanah baki telah diselidiki. Keputusan-keputusan ujian kimia ke atas tanah baki tropika yang dirawat secara elektrokinetik dibentangkan. Larutan-larutan kimia yang digunakan sebagai elektrolit dalam ujikaji elektrokinetik adalah aluminum klorida, (Al), kalsium klorida (Ca) dan asid fosforik (PA) di samping air suling (DW). Empat sistem anod terbuka dan katod terbuka yang dinamakan DW-DW, Al-DW, Ca-DW dan DW-PA diaplikasikan dalam kajian ini. Setiap sistem dikenakan satu voltan tetap 30 V selama 168 jam. Kuantiti dan arah aliran elektroosmosis didapati bergantung kepada jenis elektrolit yang digunakan. Kandungan kimia tanah yang dirawat pula bergantung kepada kedudukan, jenis elektrolit dan arah aliran elektroosmosis.

Katakunci: Tanah Baki; Elektrokinetik; Elektroosmosis; Elektromigras

1.0 Introduction

Various chemicals have been utilised to strengthen soft soils particularly clays. These chemicals are normally introduced into the soils by mechanical means such as pumping and ploughing. In such methods, the migration of the ions from the additives is restricted to a limited distance, thus inefficient. A greater challenge is to diffuse chemicals into soils with low hydraulic conductivity such as clay.

In addition to using additives, dewatering and consolidating by electrokinetics (EK) processing have been employed to strengthen and to reduce compressibility of saturated soft soils. Besides dewatering, the ions produced from the corroded anode material especially iron react with clay minerals in the treated soil to form hard compounds, which in turn increase the strength of the soil (Bell, 1975; Hausmann, 1990).

EK processing has also been employed with combination of chemical solutions to increase the strength of cohesive soil. The commonly used chemicals include calcium chloride (Harton et al, 1967), aluminium chloride (Gray, 1970), and aluminium sulphate and phosphoric acid (Ozkan et al, 1999). These chemical solutions can be fed at the anode or the cathode depending on the ions to be transferred into the soil. Considering the successful application of this technique to temperate soils, the feasibility of employing this method on tropical residual soils was investigated. The emphasis of this paper is to present the electroosmotic flows and the migration of electrokinetically-injected ions in a tropical residual soil. The selection of chemical substances was based upon the types of chemicals that have been utilised successfully on temperate soils, particularly kaolinite.

The formation of a residual soil profile is extremely complex, difficult to understand and to generalise. Residual soils differ from sedimentary soils in that they have formed in place in response to the local parent material, climate, topography and drainage conditions (Mitchell, 1993). In tropical regions, weathering of primary minerals is more intense and occurs to greater depths than elsewhere. The base-forming cations such as K^+ , Na⁺, Ca²⁺ and Mg²⁺ were leached away by running water (Taha et al, 1997), resulting in high concentration of iron and aluminium oxides. As a consequence, the exchange complex is dominated by Al³⁺ and H⁺ ions (Brady and Weil, 1996) that are responsible for soil acidity. Most soils in Malaysia are high in iron and aluminium contents resulting in positively charged soils (Taha et al, 1997).

2.0 Electrokinetics

The EK phenomena in porous medium are based on the relative motion between a charged surface and the bulk solution at its interface. The formation of electric double layer at the charged surface of clay particles is responsible for EK phenomena of interest, namely electrophoresis, electromigration and electroossosis. Only electromigration and electroossosis will be examined in this paper.

Electromigration is the movement of charged ions towards the oppositely charged electrodes relative to solution. In a dilute system or a porous medium with moderately concentrated aqueous solution of electrolytes, electromigration of ions is the major cause of current conduction.

When a DC electric potential is applied to a clay-water system in an electroosmosis (EO) process, the surface or particle is fixed. However, the mobile diffused layer moves and drags the solution with it. According to Gray and Mitchell (1967), the fundamental importance in EO phenomena is the cation-anion distribution and the water-ion distribution in the soil. The EO flow rate is described by the Helmholtz-Smoluchowski's equation in which

$$q_{eo} = \left(\frac{-\zeta D}{\eta}n\right) \left(\frac{\Delta E}{\Delta L}\right) A \tag{1}$$

where ζ is zeta potential, D is dielectric constant of the pore fluid, n is porosity, η is viscosity of pore fluid, ΔE is potential difference, ΔL is length of soil sample, and A is cross-sectional area of sample.

Based on Eq. (1), negative zeta potential results in a positive flow rate indicating flow toward the cathode and vice versa. Eq. (1) can be related to Darcy's law and written as

$$q_e = k_e i_e A \tag{2}$$

where k_e is electroosmotic hydraulic conductivity, i_e is voltage gradient and A is cross-sectional area. Eq. (1) and Eq. (2) are indeed similar as the first and second terms in the parentheses of Eq. (1) are equivalent to k_e and i_e in Eq. (2), respectively. Electrolysis of water at the anode and the cathode produces oxygen and hydrogen, respectively, which can be represented by the following equations;

$$2H_2O - 4e^- \to 4H^+ + O_2 \uparrow \qquad \text{(anode)} \tag{3}$$

$$2H_2O + 2e^- \rightarrow 2(OH)^- + H_2 \uparrow \qquad \text{(cathode)} \tag{4}$$

During electrochemical (EC) treatment, EO is supplemented by the actions of chemicals introduced at the anode or cathode, either in solution or by decomposition of the electrodes. In a study conducted by Ozkan et al (1999), it was observed that soil gained up to 600% increase in strength when treated by EK process utilising $Al_2(SO_4)_3$ as the anolyte and H_3PO_4 as the catholyte. However, the strength increases were not homogeneous throughout the sample.

EK treatment process on contaminated porous media is most effective when the transported substances are ionic, surface charged or in the form of small micelles with

little drag resistance (Hamed et al 1991; Acar et al 1994; Taha *et al* 1994). As contaminants transport continues, considerable precipitation within a narrow zone (very close to the cathode) may happen. This results in the reduction of soil porosity in that region, blocking the flow path, and subsequently decreases the electroosmotic flow and efficiency.

3.0 Experimental Procedures

3.1 Sample Preparation

Samples of granite residual soil were obtained from depths between 2m to 3m within the compound of Universiti Teknologi Malaysia, Skudai. The soil was first air-dried in laboratory before subjected to laboratory testing such as sieve analysis, compaction and EK processes. Figure 1 illustrates the soil particles distribution. Only soil passing 2.00 mm sieve was used in the compaction tests and EK experiments.



Figure 1: Grain size distribution of soil utilised in the study

Figure 2 illustrates the compaction curve with the maximum dry density and optimum moisture content of 1415 kgm⁻³ and 33%, respectively. These values were used as references for preparing samples. The detailed procedures are discussed elsewhere by Ahmad et al (2005). Table 1 shows the types and amount of major elements present in the granite residual soil used in this study. Those values were determined using X-Ray Fluorescence (XRF) and will be used as reference to changes in the soil contents after EK treatments.



Figure 2: Compaction curve to determine the maximum dry density and optimum moisture content

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Elements	%
SiO ₂	31.08
TiO_2	0.35
Al_2O_3	30.73
Fe_2O_3 (total)	21.81
MnO	0.04
MgO	0.02
CaO	0.03
Na ₂ O	0.15
P_2O_5	0.04
LOI	15.65

3.2 Electrokinetic Experiments

The EK experiments were performed using EK cells (Figure 3). The details of the experimental program are explained elsewhere by Ahmad et al (2005). In addition to distilled water, the electrolytes used in this study were 1.0 M aluminium chloride, 1.0 M calcium chloride and 1.0 M phosphoric acid. The selection of these chemicals was based

on their high solubility to be injected into the soil via EK processing and the capability of the ions to improve the strength of clayey soils due to ion exchanges and formation of cementing materials (Harton et al, 1967; Gray, 1970; Bros et al, 1983; Ozkan et al, 1999). Each anolytes and catholytes used for EK processing was given different abbreviations or codes (Table 2). The left and right terms of each code represent the anolyte and catholyte, respectively.

This study used titanium meshes as electrodes due to its high resistance to corrosion (Berkeley and Pathmanaban, 1990), and a constant voltage of 30 V was applied to each system for 168 hours. This voltage was selected after several trials. It was found that applying voltages below 30 V to soil with distilled water as the anolyte and catholyte has failed to produce electroosmotic flow. The electrodes were placed in the electrolyte compartments instead of on both ends of soil sample in order to maintain a maximum contact between the electrodes and electrolytes. This was to ensure effective current density during EK processing. The treatment period was chosen based on soil decontamination experiments performed by Thenavayagam and Wang (1994) and Taha *et al* (1994). No attempts were made to control the current density (A cm⁻²), voltage gradient (V cm⁻¹), or the pH of the electrolytes.



Figure 3: Schematic diagram of the electrokinetic experiment.

Table 2 :	Codes	for EK	treatment	system
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Code	Anolyte	Catholyte
DW-DW	Distilled water	Distilled water
Al-DW	1.0 M aluminium chloride	Distilled water
Ca-DW	1.0 M calcium chloride	Distilled water
DW-PA	Distilled water	1.0 M phosphoric acid

To meet stringent procedures of sample preparation and EK experiments, only two samples and two EK experiments were performed at a time. Two DC power supplies capable of supplying up to 60 V and 3 A were used to carry out the EK experiments. Upon completion of the EK experiments, each of the treated soils was carefully removed from the soil compartment and cut into five equivalent slices. Each slice represented its respective position in the soil compartment. The chemical contents of the untreated and EK-treated samples were determined by XRF method.

4.0 **Results and discussion**

The results of this study are classified into two main categories, i.e. (i) EK flows and EK hydraulic conductivity and (ii) electromigrations.

4.1 EK flows and EK hydraulic conductivity

The Helmholtz-Smoluchowski's expression in Eq. (1) is referred to in explaining the quantities and directions of the net electroosmotic flows. The cumulative net electroosmotic flows in the tested residual soils, i.e. DW-DW, Al-DW and DW-PA, were toward the anode and continued to increase to maximum values of -22 ml, -80 ml and -158 ml, respectively (Figure 4). The net flow toward the anode resulting from the DW-DW systems agreed with the findings by Araruna et al (2002) for residual soil originating from gneiss. Their results indicate positive zeta potentials.



Figure 4: Variation of cumulative net flows with time

There were instances where zero net flows were observed for the DW-DW, Al-DW and DW-PA systems. For these instances the zeta potential was zero, when the pH of the soil reached zero mobility ($\zeta = 0$) or the isoelectric point (IEP) (Hunter, 1981; Fletcher and Walsh, 1990). Above this IEP, the zeta potential is negative and theoretically the flow reverses toward the cathode. Vane and Zhang (1997) suggest that the zeta potential might be highly position dependent due to the pH gradients resulted from the EK processing.

It is interesting to note that a lowered or reversed surface charge of the soil does not simultaneously cause the current and EO flows to stop. Instead, a complicated set of reaction between pore fluids and solids is likely to conspire the EO to stop flowing (Grundl and Reese, 1997). Ion exchange reactions, dissolution and subsequent precipitation of carbonate minerals and metal oxide coatings might decrease the ability of the soil to transmit water due to the decreasing magnitude of zeta potential, and changes in fluid chemistry and viscosity.

Unlike the three systems explained previously, the direction of cumulative net flow of the Ca-DW system shifted from toward the anode initially to toward the cathode. The cumulative net flow for this system at the end of the experiment was 55 ml. This finding is in contrast to observations by Yeung et al (1997) and Eykholt (1997) who reported small reverse flows. The change in flow directions also indicates that the sign of zeta potential changed with time during the experiments due to changes in soil pH as shown by Hunter (1981) and Vane and Zang (1997). In addition, the pH at which polarity reversal of zeta potential takes place depends on the physico-chemical properties of the soil and pore fluid (Yeung et al, 1997). According to Gurtu (1977), the sign of charge in most cases can be changed by adding trace quantities of certain active electrolytes in water.

In this study the net electroosmotic flows were enhanced by adding salt and acid in the systems. These results agree with Lockhart (1983a), who investigated the effects of salts, acids and flocculants on electroosmotic dewatering of clays. Lockhart (1983a) also found that the extra current due to the increased ionic concentration contributed to the increase in net EO flows. In addition, a study on the influence of current density and pH on electrokinetics by Hamed and Bhadra (1997) showed that higher current densities resulted in higher flow rates. However, these observations are contrary to the traditional theory and observations by Gray and Mitchell (1967). The increased in net flows might be due to a combination of several factors. As demonstrated in Eq. (1), with the assumption that the porosity was unchanged, the flow rates were influenced by the changes in the zeta potential, dielectric constant, viscosity and voltage gradient due to dynamic electrochemical processes.

The variations in electroosmotic hydraulic conductivity (k_e) are influenced by the chemistry of the soil-water system. In general, the k_e values were observed to vary with time resulted from the changes in pore water chemistry during the EK processing. The values of k_e of the DW-DW system fluctuated between 0 and -8 x 10⁻⁶ cm² s⁻¹ V⁻¹. There were instances where $k_e = 0$, indicating zero net flow corresponded to IEP when $\zeta = 0$. On the other hand, the k_e of the DW-PA decreased and fluctuated with time from zero to -5.26 x 10⁻⁶ cm² s⁻¹ V⁻¹. In the meantime, it is interesting to observe that the k_e of the

Al-DW changed from zero to negative values, followed by zero to negative value again and so forth. In their study, Hamed *et al* (1991) observed that k_e is not constant but decreases with changing chemistry across the soil during the EK processing.

The k_e values of the DW-DW, Al-DW and DW-PA systems were lower than the typical reported k_e value of 5 x 10⁻⁵ cm² s⁻¹ V⁻¹ (Hausmann, 1990; Mitchell, 1993; Hamed and Bhadra, 1997). In addition, the net flows were toward the anodes (negative k_e). Araruna *et al.* (2002) also reported that EO flowed toward the anode with k_e ranging between 1 x 10⁻⁴ and 1 x 10⁻⁶ cm² s⁻¹ V⁻¹. Such low values might be associated with the mineral type as EK is more effective in silty clays of moderate plasticity than in high plastic clays (Lockhart, 1983b). In addition, the low soil pH might be responsible for the low k_e values. Mise (1961) observed that electroosmotic flow for kaolinite at pH 10 was nearly five times greater than at pH near 2.0, whereas Shang and Lo (1997) found very effective electrokinetic process in a high pH environment (pH > 9.0) with fairly high salinity.

Unlike the other three systems, the Ca-DW system produced fluctuating values of k_e in addition to a change in the sign of k_e from negative to positive. The change in the sign of k_e indicated change in the sign of zeta potential due to changes in pH as mentioned previously. The k_e values changed from -2.8 x 10⁻⁵ cm² s⁻¹ V⁻¹ initially to a maximum value of 2 x 10⁻⁵ cm² s⁻¹ V⁻¹ and dropped back to 5 x 10⁻⁶ cm² s⁻¹ V⁻¹ toward the end of the experiment. It is also interesting to note that unlike the other three systems, no zero net flow was observed for this Ca-DW system, indicating that IEP was not achieved.

4.2 Electromigrations

Soil samples that were treated using the Ca-DW system resulted in significant increases in Ca²⁺ contents of the soil due to electromigration and advection. The distribution of the Ca²⁺ contents with respects to the locations along the treated sample is shown in Table 3. The increases in Ca²⁺ contents are high at both ends of the sample. Such a variation could be associated with the distance from the Ca²⁺ source (the anode compartment) and the alkaline environment near the cathode compartment which lead to precipitation of Ca(OH)₂.

Dist. from anode, cm	% Ca ²⁺	% increase
0.5	1.44	4700
1.5	0.79	2500
2.5	0.72	2300
3.5	0.81	2600
4.5	1.84	6000

Ta	ble	3:	Ca	Content after	EK	treatment	using	Ca-DW	system
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Note: Initial Ca^{2+} content was 0.03%

Figures 5(a) and 5(b) show the images of the soil fabric near the anode and cathode, respectively resulted from the Ca-DW treatment. Figure 5(a) shows stacks of large platy shape soil particles with large voids after being treated using the Ca-DW system. It is believed that the coating materials (oxides of iron or aluminium) were dissolved due to the acidic environment or other chemical reactions that took place near the anode, thus exposed the platy shape particles. On the other hand, Figure 5(b) shows large inter-connected soil particles on the background besides randomly arranged small nodules on the foreground of the Ca-DW treated sample near the cathode. Ion exchanges that altered the property of the material as found by Esrig and Gemeinhardt (1967) might lead to such fabric or particle arrangements. In addition, dissolution of coating materials might not occur within the cathode zone in the slightly alkaline environment (pH = 7.53).



(a)

(b)

Figure 5: Image of soil fabric treated using Ca-DW system; (a) near anode and (b) near cathode

As the net EO flow was toward the anode, the movement of Al^{3+} from the anode compartment into the soil was fully governed by electromigration. Thus advection of Al^{3+} did not take place during the EK processing. Table 4 shows the distribution of the Al^{3+} contents in different segments of the soil based on Energy Dispersive X-ray analyser (EDX), an accessory to the Scanning Electron Microscopy (SEM) after EK treatment. The increase in Al^{3+} contents was highest near the anolyte compartment from which the Al^{3+} originated, and the contents generally decreased toward the cathode. Such trend might be due to the net EO flow toward the anode. The electromigration of the Al^{3+} towards the cathode might be restricted or reduced by the EO flow towards the anode. In addition, such negative net flow could have drifted the Al^{3+} back toward the anode zone.

EK processing decreased the soil pH near the anode from 5.30 to 3.29, whereas near the cathode decreased to pH 3.55. The reduction in the soil pH at the cathode

indicated that the amount of H^+ migrated across the soil exceeded the quantity of the OH^- migrated toward the anode, even though the net flow direction was toward the anode.

Murayama and Mise (1953) found that the electrokinetically-injected Al^{3+} would precipitate as $Al(OH)_3$ and dehydrate to become bauxite (AlO(OH)) and alumina (Al₂O₃). According to these authors, the reactions that took place during dehydration to form bauxite (AlO(OH)) and alumina (Al₂O₃) could be described as follows;

$$Al(OH)_{3} \longrightarrow AlO(OH) \longrightarrow Al_{2}O_{3}$$
(5)
$$-H_{2}O \qquad -H_{2}O$$

Dist. from anode, cm	% Al ³⁺	% increase
0.5	23.69	37
1.5	19.53	13
2.5	20.81	20
3.5	19.95	15
4.5	20.09	16

Table 4 : Al³⁺ contents in different segments of Al-DW treated sample

Note: Initial Al³⁺ content based on XRF was 17.29%

Figures 6(a) and 6(b) show the sample fabric near the anode and cathode after the Al-DW treatment. At the anode, the soil particles are well inter-connected and encapsulated which resembles to aggregates-cement matrix. Stacks of well-coated platy materials are also visible within the matrix. Such coating materials might be compounds containing AI^{3+} , which originated from the anode compartment. On the other hand, randomly arranged and stacked flaky shaped particles with sharp edges are observed at the cathode together with irregularly shaped nodules Dissolution or ionisation of the coating materials and other compounds or precipitates might be the reason for the exposed flaky particles.

For the DW-PA treated sample, the formation of phosphate compounds as coating and cementing agent was due to electromigration and advection of phosphate ions from the cathode compartment. The distribution of phosphate contents in different segments of the DW-PA treated sample obtained from the XRF analyses is tabulated in Table 5. The magnitudes of increases in phosphate contents in the treated soil decrease toward the anode. The phosphate compounds were formed as the phosphate ions migrate from the cathode compartment through the soil to the anode, thus decreasing the available phosphate ions toward the anode.



Figure 6: Images of sample fabric after Al-DW treatment; (a) near anode and (b) near cathode

Dist. from anode, cm	% phosphate	% increase
0.5	0.07	75
1.5	0.58	1350
2.5	0.99	2375
3.5	2.82	6950
4.5	3.72	9200

Table 5 : Percentage of phosphate after EK treatment using DW-PA system

Note: Initial phosphate content is 0.04%

The reactions that fix phosphorus differ from soil to soil and are closely related to soil pH. The greatest degree of phosphorus fixation for aluminium and iron occurs at very low pH, whereas for calcium at very high soil pH (Hesse, 1972; Brady and Weil, 1996). Since the soil used in this study contained aluminium and iron (Table 2) and was acidic, aluminium or iron phosphates must have been formed after the DW-PA treatment. The process and reaction that form phosphate compounds can be expressed as

$$Al^{3+} + H_2PO_4^- + 2H_2O \leftrightarrow 2H^+ + Al(OH)_2H_2PO_4$$
(6)
(soluble)
(insoluble)

Attempts to identify the formation of new minerals or compounds in the DW-PA treated samples using X-Ray Diffraction method (XRD) immediately after the completion of electrokinetic processing were unsuccessful. Hence, it is hypothesised that the new and fresh cementing compounds are in a non-crystalline or an amorphous form.

Figures 7(a) and 7(b) illustrate the fabric of the DW-PA treated samples near the anode and cathode, respectively. At the anode, the soil on the background is tightly packed with some voids as shown by the dark spots, whereas various sizes of nodules with irregular shapes are visible on the foreground. At the cathode, the soil particles are inter-connected and coated by phosphate compounds to form large particles with some large voids and nodules of various sizes.



(a)

(b)

Figure 7: Images of fabric of DW-PA treated samples; (a) near anode and (b) near cathode

5.0 Conclusions

Experiments were performed to investigate the electroosmotic flows and electromigrations during electrokinetics (EK) processing with injection of different chemicals on a tropical residual soil. Several findings can be drawn specifically for the tropical residual soil used in this study.

- i. The net electroosmotic flows for tropical residual soil, namely DW-DW, Al-DW and DW-PA systems were toward the anodes. This was due to positive sign of zeta potential during EK processing.
- ii. The direction of net electroosmotic flows of the Ca-DW system shifted from toward the anode initially to toward the cathode. Such observation was associated with changes in the sign of zeta potential from positive to negative as the pH of the soil-pore fluid system change.
- iii. The average electroosmotic hydraulic conductivity (k_e) of the tropical residual soil used in this study is lower than that of temperate clayey soils. There were instances where $k_e = 0$ indicating no flow due to the achievement of isoelectric point.
- iv. The EK processing using different types of electrolyte resulted in increases of ion species within the treated soil due to electromigration and advection. The magnitudes of increases were position and system dependent.

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