

TRANSPORT AND EXCHANGE BEHAVIOR OF IONS IN BENTONITE DURING ELECTRO-OSMOTIC CONSOLIDATION

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Abstract—Electro-osmotic consolidation is considered to be an efficient technique for dewatering and consolidation of soft soil. In the present study, four experiments were conducted on a Na-rich bentonite using two reactive electrodes (copper and iron) and two inert electrodes (graphite and stainless steel) to study the transport and exchange behavior of ions during electro-osmotic consolidation. The results showed that the changes in pH and ion contents were limited to the zone close to the electrode due to the buffering capacity of bentonite and the significant reduction in electric current density. The ion concentration profiles indicated that Na⁺ ions were largely responsible for carrying the pore water to the cathode. The reactive electrodes are better at transporting Na⁺ ions and therefore induce better drainage than inert electrodes. Ion-exchange reactions occurred between the Cu²⁺ and Fe²⁺/Fe³⁺ ions released and pre-existing Na⁺ ions in the electrical double layer, causing decreased water adsorption capacity and plasticity index. The swelling and shrinkage characteristics of the bentonite were thus reduced, and electro-osmotic consolidation may therefore provide a new way to improve the stability of expansive soils and slopes.

Key Words—Electro-osmotic Consolidation, Electrode Material, Geotechnical Property, Ion Content, Ion Exchange, Na-rich Bentonite.

INTRODUCTION

Clay particles are characterized by a net negative charge caused mainly by isomorphous substitution, crystal-lattice defects, and ionic dissolution. In a clay-water system, cations are adsorbed near the clay layers to retain electric neutrality, and an electrical double layer is formed. Under an applied electric field, the adsorbed cations are moved from the anode to cathode (electromigration), and the dipolar water molecules are driven along with the cations to the cathode during this process (electro-osmosis). Based on the electrically induced process, several techniques are developed for soil improvement and remediation. Electro-osmotic consolidation is proposed for the strengthening of soft soil with extremely low hydraulic conductivity (Casagrande, 1948; Bjerrum *et al.*, 1967; Esrig, 1968; Lorenz, 1969; Casagrande, 1983; Lo and Ho, 1991; Lefebvre and Burnotte, 2002; Burnotte *et al.*, 2004; Zhuang and Wang, 2007; Hu *et al.*, 2012; Wu and Hu, 2013; Zhou *et al.*, 2013; Hu and Wu, 2014). Previous studies indicated that the physical and chemical properties changed during electro-osmotic consolidation, including plasticity, zeta potential, cation exchange capacity, and microfabric (Bjerrum *et al.*, 1967; Lo and Ho, 1991; Micic *et al.*, 2001; Wu and Hu, 2014; Wu *et al.*, 2015). Electrochemical treatment takes advantage of electromigration to transport injected ions (Na⁺, Ca²⁺, Al³⁺, and

others) in the soil, and the shear strength of the treated soil can be improved greatly due to the change in ionic species and the production of cementing agents near the electrodes (Acar *et al.*, 1990; Otsuki *et al.*, 2007; Chien *et al.*, 2009; Ou *et al.*, 2009; Chang *et al.*, 2010; Abu Rabi-Stankovic *et al.*, 2012). Electro-kinetic remediation has been studied extensively in recent decades for the removal of heavy metals (lead, chromium, nickel, cadmium, arsenic, and others) as well as organic pollutants (oil and polycyclic aromatic hydrocarbons) (Acar and Alshawabkeh, 1993; Reddy and Saichek, 2002; Al-Hamdan and Reddy, 2008; Ricart *et al.*, 2008; Zhang *et al.*, 2010; Cameselle and Reddy, 2013; Tong *et al.*, 2014). Successful application of these techniques depends on the transport and exchange characteristics of ions in soil under an applied electric field.

Previous studies of electrochemical treatment and electro-kinetic remediation investigated the transient behavior and removal effect of ions in soil; however, literature on electro-osmotic consolidation has focused mainly on understanding drainage and consolidation characteristics including water discharge, voltage distribution, electric current, pore-water pressure, and settlement. In contrast, the transport and exchange behavior of ions in soil during this process have seldom been studied (Micic *et al.*, 2001; Glendinning *et al.*, 2005; Lamont-Black and Weltman, 2010).

The purpose of the study was to investigate the transport and exchange behavior of ions in clay soil during electro-osmotic consolidation. One-dimensional column experiments were conducted on a Na-rich bentonite by varying the electrode materials, including

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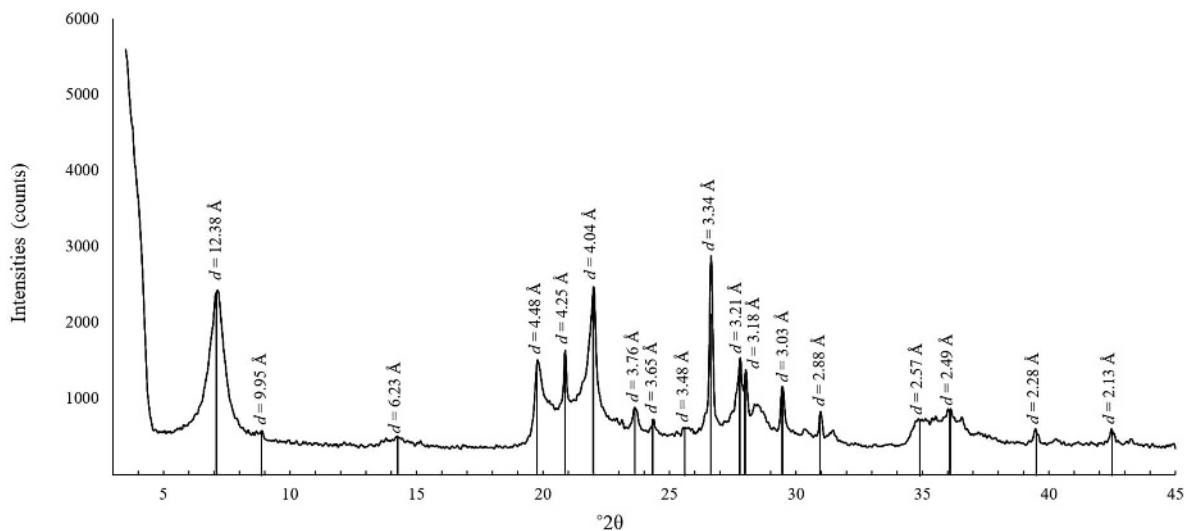


Figure 1. XRD pattern of the Na-bentonite (CuK α radiation).

copper and iron as reactive electrodes, and graphite and stainless steel as inert ones. The variation of pH and ion contents including pre-existing Na⁺ cations as well as Cu²⁺ and Fe²⁺/Fe³⁺ cations released from the anodes along the soil column were detected and analyzed. The ion concentration in the discharged water was measured to understand the drainage mechanism of electro-osmotic consolidation. The Atterberg limits of the bentonite were investigated to study the change of soil properties due to the transport and exchange of ions during electro-osmotic consolidation.

MATERIALS AND EXPERIMENTS

Materials

A Na-rich bentonite from the City of Zhangjiakou, Hebei Province, China, was used for electro-osmotic consolidation experiments. The mineralogical composition of the bentonite was studied using a Bruker D8 Advance X-ray diffractometer (Karlsruhe, Germany) equipped with a sample spinner and a diffracted-beam graphite monochromator using CuK α radiation generated at 1.6 kW. The diffractometer was operated at 40 kV and 40 mA with a step size of 0.02°2 θ using a 1° divergence slit, 1° scattering slit, 0.3 mm receiving slit, and a scan speed of 1°2 θ /min and a scan range of 3 to 60°2 θ . The random bentonite sample to be examined using XRD was first oven-dried at 105°C and then ground to pass a #200 mesh with an opening size of 0.075 mm. The X-ray diffraction (XRD) pattern (Figure 1) indicated the presence of clay minerals Na-rich smectite (Sme) and illite (Ilt) in addition to minor amounts of quartz, cristobalite, feldspars, and carbonates. Chemical analysis demonstrated that Na⁺ ions were the major exchangeable cations, as reflected by the 2.9 wt.% Na₂O, and this further induced a high plasticity index (124%) and free swelling ratio (540%) (Table 1).

The cation exchange capacity was measured by the barium chloride-magnesium sulfate method according to Chinese ‘Specifications of Soil Testing’ (SL237-1999), and the result was 40 cmol(+)/kg, indicating that strong ion exchange reactions may occur during the experiment. The pH of the bentonite sample after saturation with deionized water was 9.4.

Experimental apparatus

One-dimensional experiments were conducted in a vertical cylinder (Figure 2) and the testing samples were 9 cm in diameter and 20 cm in height. Four different electrode materials were employed to study their effect

Table 1. Geotechnical properties and chemical composition of the Na-rich bentonite.

Properties	Values
Geotechnical properties	
Specific gravity, G_s	2.625
Liquid limit, w_L (%)	155
Plastic limit, w_p (%)	31
Plasticity index, I_p (%)	124
Free swelling ratio, FSR (%)	540
Cation exchange capacity, CEC (cmol(+)/kg)	40.03
Chemical composition (weight proportion, %)	
SiO ₂	68.2
Al ₂ O ₃	15.1
CaO	4.2
MgO	3.8
Fe ₂ O ₃	3.1
Na ₂ O	2.9
K ₂ O	1.6
SO ₃	0.4
TiO ₂	0.4
Cl	0.08

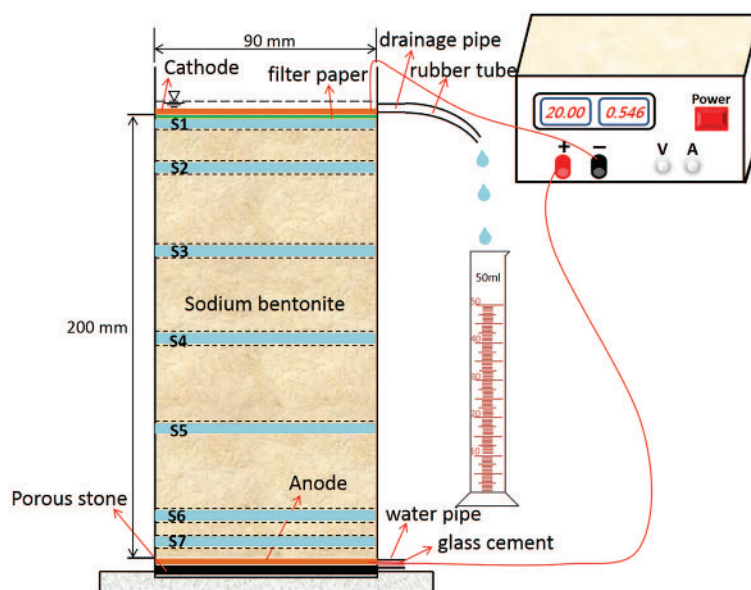


Figure 2. One-dimensional apparatus for electro-osmotic consolidation (S1–S7 denote the sampling points after the tests).

on the ion transport and exchange behavior, including two reactive materials (copper and iron) and two inert materials (graphite and stainless steel). The anode platen was placed at the bottom of the sample column, with the cathode platen on the top. Small holes were drilled in the cathode platen for the discharge of pore water. Two pipes were inserted in the wall for inflow and outflow of water. The upper pipe was used for the outflow of discharged water during electro-osmotic consolidation, and the lower pipe for the inflow of deionized water during the saturation period after which it was sealed with glass cement to form an impervious bottom boundary. The bentonite powder was first mixed with deionized water at a pre-determined water content (10 wt.%) and then compacted in the column by five layers. After that, the compacted sample was saturated by a vacuum method to achieve an initial water content of 150 wt.%. A constant voltage gradient of 100 V/m was applied to the bentonite samples, and the experiment lasted 24 h until electro-osmosis was stopped.

Tests after electro-osmotic consolidation

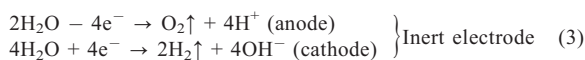
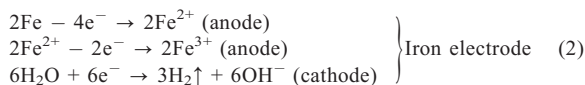
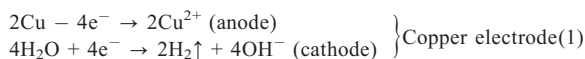
During the electro-osmotic consolidation process, the discharged water was collected at the cathode and diluted 40 times with deionized water in order to detect the ion concentrations with a Thermo Iris Induced Coupled Plasma-Atomic Emission Spectrograph (ICP-AES, Thermo Fisher Scientific Inc., Waltham, Massachusetts, USA). After the experiment, soil pH along the sample column was measured by a penetrating pH probe (IQ150-77 Meter and Probe System, Hach Company, Loveland, Colorado, USA) at different depths in the sample. Soils from different positions (marked S1 to S7 in Figure 2) at distances of 10 mm, 20 mm, 60 mm,

100 mm, 140 mm, 180 mm, and 200 mm from the anode were prepared for chemical analysis by X-ray fluorescence (an XRF-1800, Shimadzu Corporation, Kyoto, Japan). The soil samples for the XRF tests were first dried at 105°C and then ground to pass a #200 mesh with openings of 0.075 mm size.

RESULTS AND DISCUSSION

Chemical reactions

During electro-osmotic consolidation, the primary electrode reactions were different for reactive (copper and iron) and inert (graphite and stainless steel) electrodes, as listed below



When reactive electrodes (copper and iron) are used, the primary chemical reaction at the anode is oxidation of the metal electrodes, accompanied by a small amount of water electrolysis. Cu^{2+} , $\text{Fe}^{2+}/\text{Fe}^{3+}$, and H^+ are released into the soil during this process, causing the change in soil chemistry and pH. For the inert electrodes (graphite and stainless steel), the chemical reaction at the anode is mainly the electrolysis of water and only H^+ is produced. At the cathode, the main chemical reaction is the same for the four different electrodes – electrolysis of water.

Electrolysis of water at the anode not only produces H^+ but also generates oxygen which has significant impact on the behavior of the soil–anode interface during electro-osmotic consolidation. For the same electric current, more H^+ and O_2 gas are generated at the anode for the inert electrodes than the reactive ones.

Soil pH

The chemical reactions cause a decrease in pH near the anode and an increase in pH near the cathode. The ion-transport process under an electric field consisted of mass fluxes generated by diffusion, electromigration, and electro-osmosis (Acar and Alshawabkeh, 1993); among these electro-migration was the primary component. H^+ ions moved gradually from the anode toward the cathode, leading to the movement of the net-acidic front toward the cathode. The OH^- ions moved in the opposite direction from the cathode to anode and resulted in the movement of the net-alkaline front toward the anode.

‘Normalized distance’, defined as the ratio between the distance to the anode and the height of the soil sample, was used to illustrate the distributions of pH before and after electro-osmotic consolidation along the soil column (Figure 3). The results indicated that the pH decreased from 9.3 to ~ 7.0 , 6.9, 8.0, and 8.4 at the anode, and increased to ~ 12.8 , 13.1, 11.4, and 11.7 at the cathode for the copper, iron, graphite, and stainless steel electrodes respectively; the change in pH was limited to a zone close to the electrodes, however. The net-acidic front was located at ~ 0.38 and 0.22 normalized distances from the anode for the reactive (copper and iron) and inert (graphite and stainless steel) electrodes, respectively.

Most of the previous studies showed an increased distribution of pH from the anode toward the cathode in the soil under the effect of the electric field (Acar *et al.*, 1990; Acar and Alshawabkeh, 1993; Reddy and Saichek, 2002; Al-Hamdan and Reddy, 2008; Chien *et al.*, 2012).

Acar *et al.* (1990) and Chien *et al.* (2012) performed electro-osmosis experiments using kaolinite and obtained pH distributions after experiments (Figure 3). The comparison indicated that most of the soil sample was subject to acidic conditions and the pH at the anode decreased to ~ 3 . The net-acidic front was located at ~ 0.73 and 0.60 normalized distances from the anode in the two experiments. Compared to kaolinite, bentonite had a greater buffering capacity (Svensson and Hansen, 2013); the increase in H^+ concentration near the anode and the migration of H^+ ions was less complete in bentonite, therefore. The generation of gases and cracking of soil near the anode also influenced the pH changes by affecting the electric current through the soil.

During electro-osmotic consolidation, soil near the anode lost water, and volume shrinkage occurred at the bottom of the soil column which was further accompanied by increasing gas pressure at the soil–anode interface due to the impervious bottom boundary. For the Na-rich bentonite used in the present study, the shrinkage was more significant than for kaolinite used in previous studies (Acar *et al.*, 1990; Chien *et al.*, 2012). As a result, cracks formed in the soil near the anode (Figure 4, which contains a set of images of the soil–anode interface after 20 min and 1.8 h of electro-osmosis). For reactive electrodes, vertical cracks formed in the soil at the anode, while for inert electrodes a horizontal crack formed at the soil–anode interface because the gas pressure was much larger than that for reactive electrodes. These cracks, especially the horizontal crack, caused a significant loss in voltage immediately in front of the electrodes, and led to further decrease in the electric current density through the samples (Figure 5). The electric current density decreased from ~ 48.0 – 55.0 A/m^2 to 37.0, 37.0, 9.2, and 9.2 A after 1 h, and decreased to 5.5, 3.0, 1.0, and 1.0 A after 10 h for the copper, iron, graphite, and

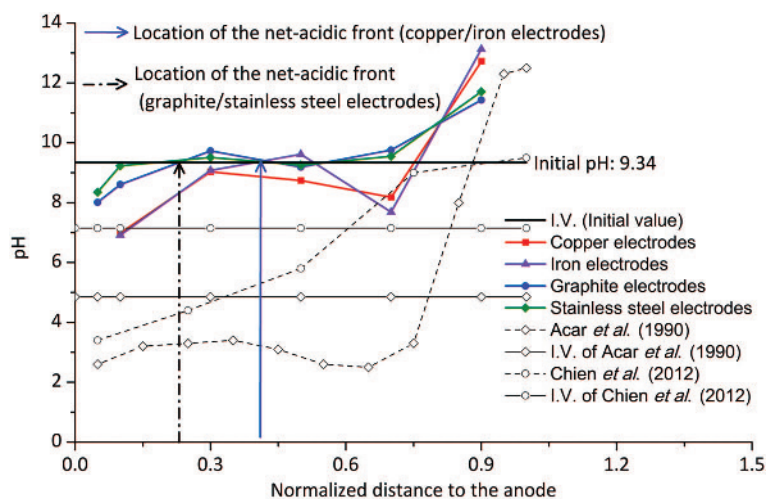


Figure 3. pH distribution along the soil column after electro-osmotic consolidation.

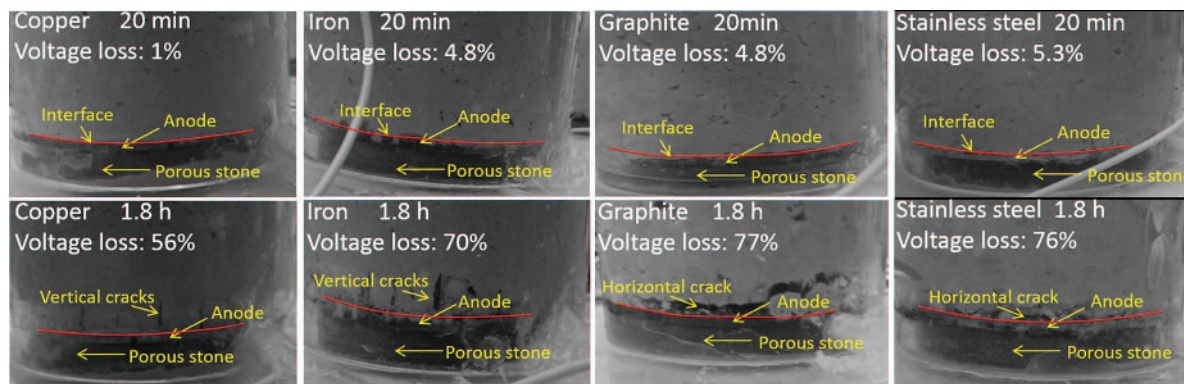


Figure 4. Images of cracks at the soil–anode interface.

stainless steel electrodes, respectively. The cracks also precluded the migration of H^+ ions (especially the horizontal crack); therefore, the movement of the acidic front was greatly restricted and the pH only decreased near the anode, resulting in different pH distributions from those observed in previous studies (Figure 3). These results demonstrated that expansive soil (bentonite) exhibited quite different behavior from other clay soil during electro-osmotic consolidation. Gas pressure and soil cracking need to be suppressed by methods such as a pervious anode boundary or combination with surcharge preloading before the successful application of this technique to expansive soil.

The distribution patterns of pH value in the bentonite samples after electro-osmotic consolidation were different for reactive and inert electrodes (Figure 3). For the inert electrodes, more gas was produced at the beginning with almost the same electric current density as that for the reactive electrodes; a horizontal crack instead of vertical cracks formed, therefore, at the soil–anode interface. The horizontal crack caused a greater decrease in electric current density than the vertical cracks (Figure 5) and the migration of H^+ then became extremely difficult. As a result, for the reactive

electrodes, the net acidic front moved a greater distance from the anode and the pH changed more dramatically.

Ion content along the soil column

As mentioned above, the migration of ions under the electric field played a significant role in electro-osmotic consolidation, electrochemical treatment, and electrokinetic remediation. The main ions in the Na-rich bentonite include Al^{3+} , Ca^{2+} , Mg^{2+} , Fe^{3+} , Na^+ , and K^+ . During the electro-osmotic consolidation process, Cu^{2+} and Fe^{2+}/Fe^{3+} ions were released from the anode into the soil due to oxidation reactions. The contents of both the pre-existing and the released ions in the samples after electro-osmotic consolidation were measured using XRF to understand the transport and exchange behavior of ions during this process.

Pre-existing ions. In the present study, the XRF results showed that the contents of Si, Al, Ca, Mg, and K in the soil were nearly the same before and after the electro-osmotic consolidation, indicating that these ions made no contribution to the movement of pore water; only the change in the Na^+ , Cu^{2+} , and Fe^{2+}/Fe^{3+} contents was analyzed, therefore (Figure 6). The Na^+ content decreased

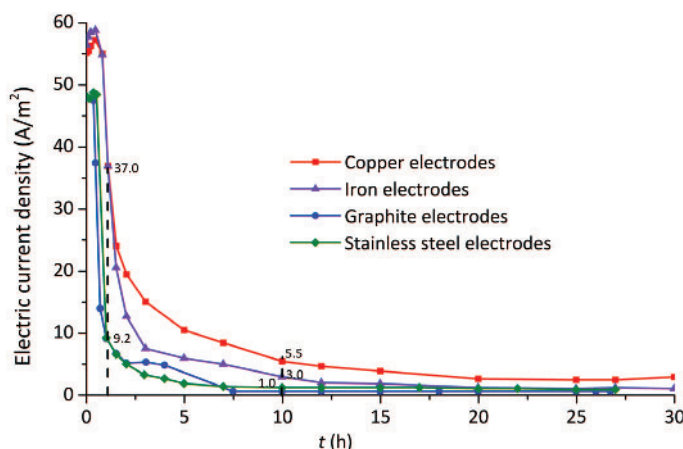


Figure 5. Electric current density variation with time during electro-osmotic consolidation.

near the anode and increased near the cathode, and the variation was greater in terms of quantity and range for the reactive electrodes than the inert ones. At the anode, the Na^+ content decreased from the initial value of 1.06% to ~0.03%, 0.17%, 0.59%, and 0.64%, while at the

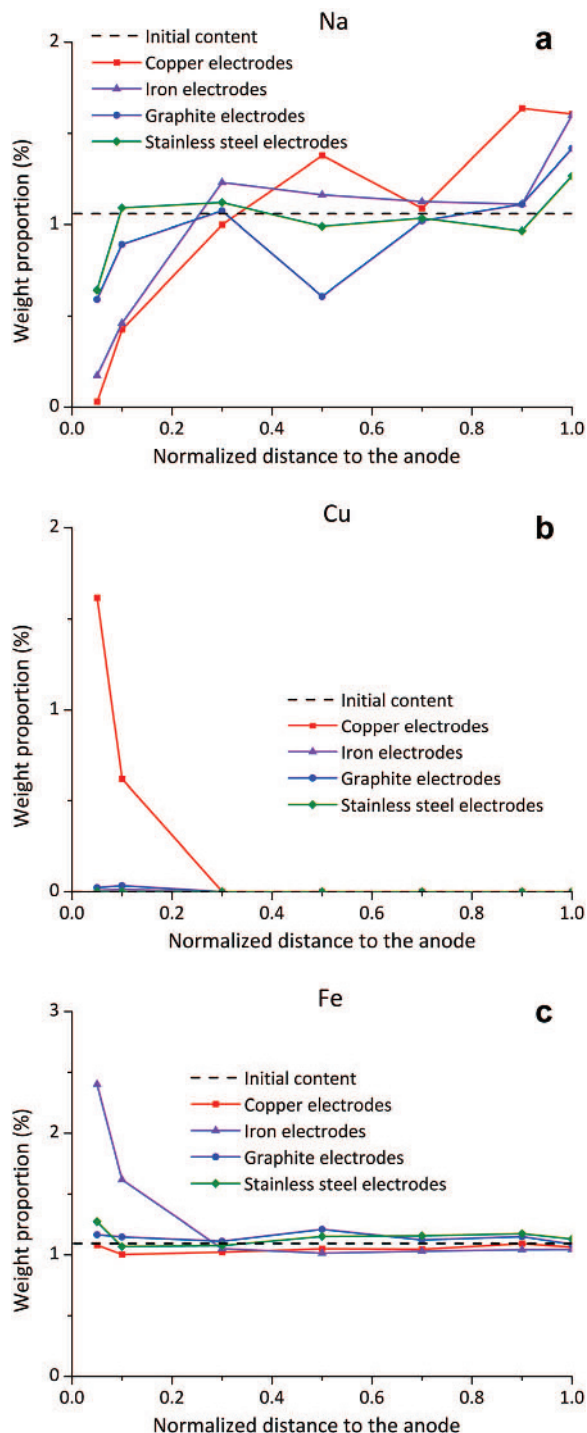


Figure 6. Distributions of different ions along the soil column after electro-osmotic consolidation: (a) Na, (b) Cu, (c) Fe.

cathode, it increased to ~1.61%, 1.6%, 1.42%, and 1.27% for the copper, iron, graphite, and stainless steel electrodes, respectively. The Na^+ content was the same as the initial value at ~0.3 and 0.1 normalized distances from the anode for the reactive and inert electrodes.

Released ions. As mentioned above, Cu^{2+} and $\text{Fe}^{2+}/\text{Fe}^{3+}$ ions were released from the anode into the soil during the experiments and the amounts of them present in the soil sample changed. Significant increases in the Cu^{2+} and $\text{Fe}^{2+}/\text{Fe}^{3+}$ contents near the anode, from 0 to 1.62% and from 1.09 to 2.40%, were observed for the copper and iron electrodes, respectively (Figure 6). Unlike Na^+ and H^+ , the migration of Cu^{2+} and $\text{Fe}^{2+}/\text{Fe}^{3+}$ was restricted by the decreased electric current density and the alkaline condition, and only moved as far as a normalized distance of ~0.1–0.3 from the anode.

Ion concentration in the discharged water. Water discharge and corresponding ion concentration in the discharged water were further analyzed to study the electro-osmosis process (Figure 7). Because the Na^+ concentration was about two to three orders of magnitude greater than those of other ions, only the Na^+ result is shown. The ultimate water discharges were 69, 47.5, 30, and 26 mL for the copper, iron, graphite, and stainless steel electrodes, respectively, indicating that the drainage effect varied from the best to the worst as follows: copper > iron > graphite/stainless steel. A similar result was found for the Na^+ concentration. For the copper and iron electrodes, the Na^+ concentrations increased rapidly from 2000 ppm (mg/L) to maxima of ~7000 and 6200 ppm, respectively. For the graphite and stainless steel electrodes, water discharge almost ceased after 10 h, and the maximum concentrations of Na^+ for the two electrodes were 3800 and 3000 ppm, which were much smaller than those for the copper and iron electrodes.

Geotechnical properties

The bentonite samples at the bottom of the soil columns were prepared for geotechnical property analy-

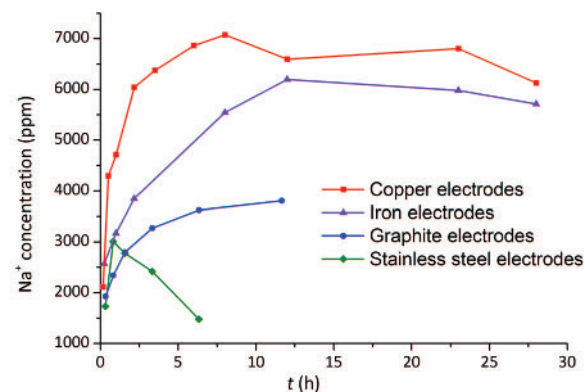


Figure 7. Na^+ concentration in the discharged water vs. treatment time.

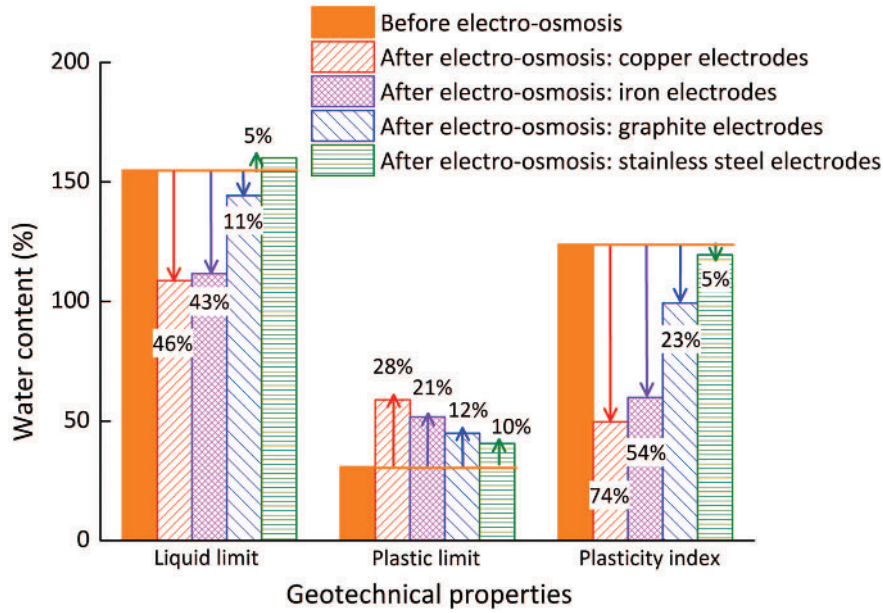


Figure 8. Change in the geotechnical properties of the Na-bentonite near the anode.

sis (Figure 8). The liquid limit decreased for the copper, iron, and graphite electrodes and increased slightly for the stainless steel electrodes, while the plastic limit increased in all four experiments, resulting in a decrease in plasticity index. The change was greatest for the copper electrodes, somewhat smaller for the iron electrodes, and much smaller for the graphite and stainless steel electrodes. A Casagrande plasticity chart was used to further analyze the plasticity property of the soil (Figure 9). The reactive electrodes (copper and iron) caused a greater change than the inert ones (graphite and stainless steel). For both reactive electrodes, the soil classification changed from high-plasticity clay to high-plasticity silt (*i.e.* the soil plasticity moved from above the A-line to below).

DISCUSSION

The main cause of ion transport during electro-osmotic consolidation is electromigration; a straightforward relationship exists, therefore, between the transport of electric current and the mass transport of dissolved ions. Thus, greater electric current (copper and iron electrodes) leads to a greater mass flux and more changes in ion content along the samples, both for H^+ and Na^+ . Because the electric current density decreased in the sequence of copper > iron > graphite/stainless steel, H^+ moved a greater distance from the anode and the pH changed more dramatically for the two reactive electrodes. Moreover, the amount of Na^+ transported in this process decreased in the same sequence, and the Na^+ abundance in the soil and the discharged water decreased similarly.

During electro-osmotic consolidation, pore water was transported from anode to cathode by the cations which were mobilized in the pore fluid and discharged with these cations at the cathode. The abundances of different cations along the soil column after electro-osmotic consolidation indicated that only Na^+ was transported to the cathode (Figure 6). The water discharge and the high Na^+ concentration in it demonstrated that the movement of pore water was accompanied by the transport of Na^+ ; Na^+ was, therefore, the primary cation contributing to the electro-osmosis. As a result, the total water discharge decreased in the sequence of copper > iron > graphite/stainless steel (the same as the electric current density and Na^+ transport). Reactive electrodes can transport Na^+ cations from the anode to cathode better and therefore induce a better drainage effect during the electro-osmotic consolidation.

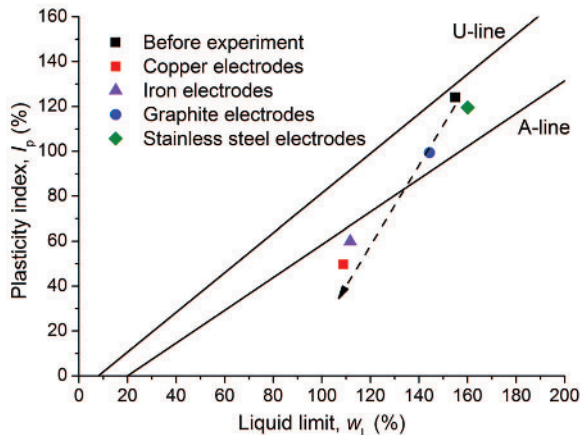


Figure 9. Effects of electro-osmotic consolidation on Atterberg limits of the Na-bentonite (A-line: $I_p = 0.73 (\omega - 20)$; U-line: $I_p = 0.9 (\omega - 8)$).

Along with the release of Cu^{2+} and $\text{Fe}^{2+}/\text{Fe}^{3+}$ ions into the bentonite, ion-exchange reactions occurred between the cations released and the pre-existing cations in the electrical double layer. The ease of exchange depends mainly on the valence, ion size, and relative abundance of different ions (Mitchell and Soga, 2005). Divalent cations were bound more strongly to clay surfaces than monovalent cations, following a typical exchange series such as: $\text{Na}^+ < \text{Li}^+ < \text{K}^+ < \text{Rb}^+ < \text{Cs}^+ < \text{Mg}^{2+} < \text{Ca}^{2+} < \text{Ba}^{2+} < \text{Cu}^{2+} < \text{Al}^{3+} < \text{Fe}^{3+} < \text{Th}^{4+}$. According to the exchange abilities of different cations, Na^+ cations in the electrical double layer were exchanged preferentially by the Cu^{2+} and $\text{Fe}^{2+}/\text{Fe}^{3+}$ cations released. The exchanged Na^+ cations were then transported to the cathode under the influence of electric field, carrying with them the pore water in the form of sodium ions' waters of hydration.

The exchange reactions between Na^+ cations and Cu^{2+} and $\text{Fe}^{2+}/\text{Fe}^{3+}$ cations changed the ion species and concentration in the electrical double layer, leading to the change of soil geotechnical properties (Stawinski *et al.*, 1990; Zhang *et al.*, 2003, 2004; Katz *et al.*, 2013; Heuser *et al.*, 2014). Cu^{2+} and $\text{Fe}^{2+}/\text{Fe}^{3+}$ cations possess greater valence than Na^+ cations; according to double layer theory (Van Olphen, 1977; Shang, 1993; Vane and Zang, 1997), the thickness of the double layer decreased after the ion-exchange reactions, therefore. As a result, the water-adsorption capacity of the clay decreased and the plasticity index was reduced. Because the graphite and stainless steel electrodes were inert, no ion was released into the bentonite to conduct an ion-exchange reaction. The change in soil geotechnical properties was therefore much less than that for the copper and iron electrodes. The influence of electro-osmotic consolidation on the plasticity index indicated that this technique was able not only to consolidate the soil but also to limit the swelling and shrinkage characteristics of expansive soil, especially when reactive electrodes (copper and iron) were used. This may provide a new approach to improve the stability of expansive soils and slopes.

CONCLUSIONS

One-dimensional electro-osmotic consolidation experiments were conducted on a Na-rich bentonite using different electrode materials to study the transport and exchange behavior of ions during this process. The distribution of pH and the amount of different ions along the soil column were monitored. Concentrations of ions in discharged water were also measured. In addition, the Atterberg limits of the bentonite before and after electro-osmotic consolidation were analyzed. The following conclusions can be drawn from these results:

The movement of H^+ and OH^- ions under an electric field led to change in the pH; the change was limited by the large buffering capacity of the bentonite, and the significant reduction of electric current density caused

by the generation of gases and soil cracking. Because the electric current density was greater for the reactive electrodes than the inert ones, the net acidic front moved further from the anode and the change in pH was greater for the copper and iron electrodes.

Electro-migration of Na^+ ions was the main reason for the movement of pore water from the anode toward the cathode under an electric field. The Na^+ content decreased near the anode and increased near the cathode, and the greater electric current density for reactive electrodes (copper and iron) induced greater changes in the Na^+ content, both in the soil and in the discharged water. Reactive electrodes can achieve greater transport of Na^+ ions and therefore induce a better drainage effect during the electro-osmotic consolidation on Na-rich bentonite.

In the case of the reactive electrodes, Cu^{2+} and $\text{Fe}^{2+}/\text{Fe}^{3+}$ cations were released from the anode into the bentonite causing ion-exchange reactions with the pre-existing Na^+ cations in the electrical double layer, leading to a decrease in the water-adsorption capacity and plasticity index. Electro-osmotic consolidation may provide a new approach to improving the stability of expansive soils and slopes.

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