ON THE IMPROVEMENT THROUGH THE MIDDLE AREA OF KAOLINITE WITH ELECTROOSMOTIC CHEMICAL TREATMENT

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ABSTRACT

Injection of the calcium chloride solution and sodium silicate solution during electroosmosis has been found to increase the clay strength effectively. However, the improved area is often limited to near the anode or the cathode and the middle area between the electrodes is unable to be improved. This study conducted the injection of the calcium chloride solution for shorter treatment duration and then injected an alkaline solution to neutralize any trace of acidity produced at the anode because the sodium solution gels rapidly under the acidic environment. The sodium silicate solution was then injected. Results show that when an alkaline environment was created near the anode, the sodium silicate solution can effectively flow into the middle region, promoting the formation of the polymeric network structure and thus the improvement of the clay strength from the anode through the middle area.

Key words: Electroosmotic chemical treatment, sodium silicate, calcium chloride, potassium hydroxide, middle area.

1. INTRODUCTION

Electroosmosis is a phenomenon that the water in the pores of soil particles moves from the anode to the cathode as a moderate electrical potential gradient is applied to a saturated soil. This technique can be used in the field in order to drain pore water off saturated soil and consolidate the soil such that the soil strength is increased. To enhance the increase of the soil strength with electroosmosis, several investigators have tried to add some chemical solutions into the treated soil either through the anode or cathode via several physicochemical behavior with electrical and hydraulic flows coupling across the soil such as electroosmosis flow, electrophoresis, and electromigration. This method, called as electroosmotic chemical treatment (ECT), is capable of generating cementation because of chemical reactions between injected solutions and soil particles, thereby increase the clay strength significantly. The ECT technique is particularly suitable for soft clay, in which its low permeability restricts the injection of chemical agents by hydraulic pressure.

The ECT technique and its application have been studied by many investigators (*e.g.*, Barker *et al.* 2004; Asavadorndeja and Glawe 2005; Ahmad *et al.* 2011; Nordin *et al.* 2013). More recently, Ou *et al.* (2009) presented the effectiveness of ECT process applied in the field by using the CaCl₂ solution and the sodium silicate solution. A significant improvement of soil surrounding the anode was recorded due to the effect of generating cementation between chemical ions and soil particles. Ou *et al.* (2013) further performed ECT tests with injecting different concentrations of sodium silicate solution into kaolinite from the anode. Results show that the strength of kaolinite can be improved at least 3 MPa near the anode as indicated by the cone penetration test resistance. In addition, the entire clay environment after treatment was almost subjected to highly alkaline condition.

Based on the studies as mentioned above, it indicated that the injection of the CaCl₂ solution, followed by the injection of W.G. solution, during electroosmosis is an effective method for improving the strength of soft clay near the cathode and the anode, respectively. The improving effects of the injection of the CaCl₂ solution are the ion exchange and pozzolanic reaction of clay near the cathode (Barker *et al.* 2004, Asavadorndeja and Glawe 2005; Ou *et al.* 2015b). In addition, the improving effect of the injection of the W.G. solution are due to the polymerization of silicic acid under different pH environments so as to improve the clay strength near the anode (Iler 1979).

However, it has been found from the above literatures that the improvement is only limited either near the anodic or cathodic region. In other words, it cannot increase soil strength at the middle part of the test sample. Therefore, the objective of this study was to find a way to increase the improved range through the middle part of the treated sample by using appropriate combinations of different chemical solutions and different treatment durations under a constant electrical gradient.

2. MATERIALS AND METHODS

2.1 Experimental Cell

An ECT cell as designed by Ou *et al.* (2013) was used in this study. The test container was made of Plexiglas plate and was 230 mm wide, 440 mm long and 320 mm deep. Two platinum (Pt) coated titanium (Ti) meshed plates were 350 mm in span, connected to a power supply, and served as the anode and

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the cathode, respectively. A small compartment connected to the anode was 20 mm wide and 150 mm long and was used to store a very small amount of injected chemical solutions, allowing the chemical solutions uniformly injected into the soil under the electric field. Another small compartment with the same dimension was connected to the cathode and used to store drained water, which would be discharged through a draining tube. The top plate was also served as a bearing plate, on which the designated consolidation pressure was provided by air. Several O-rings were fastened on both ends of the cell to prevent leakage.

2.2 Properties of Test Clay

The test clay used in this study was kaolinite. It was selected due to its high efficiency of conducting electroosmotic water transport and low activity as compared with other clays, as demonstrated by previous studies (Ou *et al.* 2015a; Mitchell and Soga 2005). The chemical compositions and engineering properties of this clay are shown in Table 1.

2.3 Test Procedures

The clay sample was prepared by thoroughly blending 12.8 kg of kaolinite and 7.7 kg of deionized water in a mechanical mixer for 50 minutes to produce a homogeneous sample with 60% water content, about 1.5 times greater than its liquid limit (Chien *et al.* 2010). The clay sample was then poured into the main compartment in three layers. After that, the top of the ECT cell and pneumatic jack was put at the top of the tank and fixed tightly with 8 screws.

The clay sample in the ECT cell was then subjected to consolidation to simulate the clay in the field condition. The axial loading pressure was set firstly at 15 kPa for about 1 day and then 30 kPa for 3 days subsequently. The applied axial loading stress remained at 30 kPa during the ECT test.

After consolidation, the supplying cylinder and the measuring cylinder were installed at the anode and the cathode, respectively. Then the monitoring system including voltage sensors and piezometers was connected to the cylinders to record the voltage, the electrical resistance, and the amount of the inflows and outflows. Finally, the DC power supply will be connected on both sides of the electrodes.

Three main phases of the ECT test was conducted, as shown in Table 2. The symbol C represents the CaCl₂ solution, K denotes the alkaline solution (KOH), N represents W.G. solution, and "xx" is the duration of injection of the KOH solution. The procedure was described as follows:

Phase 1 of the ECT test was performed by injecting the calcium chloride solution (CaCl₂) for 168 hrs in order to estimate the so-called turning point (Ou *et al.* 2013), the most suitable duration of supplying CaCl₂ solution in ECT test, designated as C168. The 168 hrs was selected based on the past experiences that the turning point for kaolinite was less than 168 hrs.

Phase 2 of the ECT test was performed by firstly injecting the CaCl₂ solution until the turning point was reached, and then the sodium silicate solution (W.G.) for another period of time. The turning point for the CaCl₂ solution was obtained from Phase 1, which was 72 hrs. When the sodium silicate solution was injected more than 72 hrs, the injection rate became small. Therefore, 72 hrs of the injection time for the sodium silicate solution was selected. This phase of tests was thus designated as C72N72.

Table 1Chemical compositions and engineering properties of
the kaolinite used in this study

Chemical analysis	Values		
SiO ₂ (%)	44.5		
Al ₂ O ₃ (%)	39.5		
TiO ₂ (%)	1.0		
MgO (%)	0.07		
Na ₂ O (%)	0.52		
Fe ₂ O ₃ (%)	0.5		
CaO (%)	0.05		
K ₂ O (%)	0.04		
Loss on ignition (%)	13.6		
Liquid limit (%)	46		
Plastic limit (%)	25		
Plastic index (%)	21		
Specific gravity, G_s	2.61		

Table 2 Main ECT test program

Test	ECT duration (hours)			No. of samples
	CaCl ₂	КОН	W.G.	for physical and chemical tests
Phase 1				
C168	168	-	-	-
Phase 2				
C72N72	72	-	72	10
Phase 3				
C48K12N72	48	12	72	10
C48K24N72	48	24	72	10
C48K48N72	48	48	72	10

Phase 3 of the ECT tests was performed by firstly injecting the $CaCl_2$ solution for 48 hrs instead of 72 hrs, then the KOH solution, and lastly the W.G. solution for another 72 hrs, designated as C48KxxN72.

The sequence of injection includes 5 different tests as described above. The concentration of the CaCl₂ solution used in this test was 0.75 M, based on the study by Chang *et al.* (2010). The concentration of KOH solution used in this test was 1.5 M. On the other hand, the concentration of the W.G. solution adopted for ECT test was the volume ratio of 1:1 (sodium silicate: deionized water), according to Ou *et al.* (2013). All the chemical solutions were injected at the anode only, and the injection procedure was conducted under a constant applied voltage of 17.5 V (0.05V/mm), as suggested by Mitchell and Soga (2005) for an optimum voltage gradient for geotechnical purposes.

2.4 Laboratory Test of Treated Clay

In addition to the ECT test, the other laboratory tests/measurements for the evaluation of the characteristics of the treated samples include cone penetration tests (CPT), water content measurement, pH measurement, inductively coupled plasma atomic emission spectroscopy (ICP-AES), X-ray diffraction (XRD) and nuclear magnetic resonance (NMR).

The CPT was used to measure the clay strength before and after treatment following ASTM D5778-12. The cone is a

specially designed laboratory cone penetration apparatus and it is attached to an adjustable rod of 0.0092 m in diameter, capable of measuring the cone resistance at different locations and depths. The penetration rate was 2 cm/sec. The treated clay was tested at 5 locations, named as NA (near the anode), FA (far the anode), M (middle part), FC (far the cathode), and NC (near the cathode) from the anode to the cathode, as shown in Fig. 1.

The measurement of water content for treated clays was tested at 10 locations from the anode to the cathode, as shown in Fig. 1, after the completion of the CPT test. It is determined according to ASTM D2216-10.

According to ASTM D4972-13, soil pH was measured using a portable pH meter (Mettler Toledo Inc., Model Seven Go SG2-FK). It was tested on the dried clay samples used for water content measurement. 10 g of dried clay sample (smaller than 2 mm in diameter) is firstly blended with 10 mL deionized water thoroughly in the centrifuge tube. Then the liquid from slurry clay sample was drained off by using centrifuge and the pH was measured by the pH meter.

The ICA-AES analytical technique was used for the detection of calcium ions (Ca^{2+}) concentration. The tested sample, according to the ISO 11466 (1995), should be digested with 10 mL aqua regia solution (a volume ratio of 3:1 by mixing solutions of 35% HCL and 69% HNO₃) for 24 hours in order to get suspension for analysis.

The XRD patterns were used to analyze the phase compositions of the specimen by comparing the patterns in the Joint Committee on Powder Diffraction Standards (JCPDS) database system. The required diffraction angle (2 θ) is between 15° to 70°, with an increment of 0.05 deg/step and a scan speed of 1 step/sec.

The variation of silicon with isotope 29 can be analyzed to examine the bonding condition of silicon inside the treated clay using NMR. The analysis is then called as ²⁹Si NMR. The different chemical structure of the silicon is denoted by Q_m , where m ranges from 0 to 4 and is an indication of the molecular bonding condition from the weakest to the strongest.

3. RESULTS AND DISCUSSION

3.1 Turning Point

Figure 2 shows the variation of the volume of drained water with treatment time for phase 1 of the ECT test (C168). As shown in this figure, the amount of water drained from the cathode increased with time, approximately linearly during the first 72 hours of treatment. After 72 hours of injection time, the rate of water discharge decreased with time. Nearly no water was drained from the cathode after 168 hours of treatment. This is because the soil particles were cemented near the cathode under the alkaline condition due to the formation of C-S-H, thereby forming an impermeable layer around the cathode. Therefore, according to the early study by Ou *et al.* (2013), the treatment time of the 72nd hour can be considered as a turning point, which was the timing for changing another chemical solution.



Fig. 1 Locations for CPT test and water content measurement (side view)



Fig. 2 Variation of total volume of drained water and duration



3.2 CPT Resistance

Figure 3 shows the clay strength as indicated by the CPT resistance (q_c) after the treatment with CaCl₂, KOH, and W.G. solutions for different durations. Results indicate that a significant strength improvement with the highest strength occurring in the anodic region was found after the treatment and the improvement decreased with the increase of the distance away from the anode. The strength of the clay, about 2 to 3 MPa, treated with the addition of KOH solution was much higher than that of untreated soil, about 0.15 MPa and also larger than those treated with pure electroosmosis only, about 0.35 MPa where no chemical reaction was involved between clay particles (Ou et al. 2009). Moreover, the improvement by injecting additional KOH solution had a greater increase, ranging from 1.2 to 2 MPa, in the anode and middle regions than the test results of C72N72, no injection of KOH solution, expect the middle area for the test of C48K48N72.

Figure 4 shows the average increased strength in the NA region with injecting KOH solution were almost the same for different treatment durations, all higher than that without injection of KOH. On the other hand, as shown in Fig 5, the improvement of clay strength in the M region gradually increased with the related injected volume of KOH solution, except for the test of C48K48N72.



Fig. 4 Average CPT resistance and injected volume of KOH and W.G. solutions in the NA region



Fig. 5 Average CPT resistance and injected volume of KOH and W.G. solutions in the M region

3.3 Water Content

Figure 6 shows the water content from the anode to the cathode across the clay sample for different treatment durations with CaCl₂, KOH, and W.G. solutions. Results indicate that the water content after treatment mostly decreased and was below the untreated condition (*i.e.*, 51%), except for the NC region. The maximum decrease, ranging from 34 to 38%, occurred near the NA region, and then the water content gradually increases from the anode to the cathode, causing the cathode to have higher water content than that before treatment (from 50 to 58%). This trend may be due to the electroosmosis flow induced by the electrical gradient that causes the decrease in water content near the anode and accumulation of drained water near the cathode. In addition, the electrochemical process and the polymerization of silicic acid provided by W.G. solution during the ECT test may change the clay properties with time and lower the water content near NA region (Iler 1979; Ou et al. 2013).

3.4 pH Measurement

Figure 7 shows the distribution of pH across the clay samples for different treatment durations with $CaCl_2$, KOH, and W.G. solutions. Results indicate that the pH value of the C72N72 near the anode decreased significantly to pH 4 after treatment, implying an acidic condition being created. However, the pH value near the anode for the tests with the addition of the KOH solution significantly increased to pH 11, implying the creation



Fig. 6 Variation of water content with distance from anode



Fig. 7 Variation of pH values with distance from anode

of a highly alkaline environment. The increase in the NA region may be due to the strongly alkaline KOH solution injected through the anode, providing a sufficient amount of the hydroxyl ions so as to effectively neutralize the hydrogen ions generated from electrolysis process. In addition, a long period of treatment by the W.G. solution afterwards also makes the environment at NA more alkaline. On the other hand, in the vicinity of the cathode, there was a considerable increase in pH by about $10 \sim 11$ after treatment, implying a generation of a highly alkaline environment. This phenomenon is due to the hydroxyl ions generated near the cathode via the electrolysis process. However, the middle part of the treated clay was found to be neutral. It may be because the hydrogen ions neutralized the hydroxyl ions.

3.5 Concentration of Calcium Ions

Figure 8 shows the variation of calcium ions concentration in clay sample of C72N72 and C48K24N72, two representative cases used to realize the spreading of Ca²⁺ ions in the clay sample after the ECT. Results show that the Ca²⁺ ions gathered near the anode $(10 \sim 20 \text{ ppm})$ and the cathode $(13 \sim 15 \text{ ppm})$, respectively. The accumulation of Ca²⁺ ions near the anode may be attributed to the CaCl₂ solution injected from the anode directly and so a large amount of Ca²⁺ ions remained here during the test. On the other hand, the accumulation near the cathode may be attributed to the effects of electroosmosis flow and electromigration, thereby causing an increase of Ca²⁺ ions concentration associated with the totally longer duration of treatment. Such great amount of Ca²⁺ ions in the cathodic region (15 ppm) can explain for the increase of clay strength near the cathode by the possible formation of C-S-H gel (Ou et al. 2015a), despite of less treatment duration of CaCl₂ solution. It is also found that the concentration of Ca²⁺ ions detected from the middle region of the treated kaolinite of C48K24N72 is found to be around 7 ppm, higher than that of the clay sample C72N72, which contained the amount of Ca^{2+} ions almost the same as in the untreated clay.

3.6 XRD Analysis

Figures 9 and 10 show that the XRD patterns taken from the samples in the NA and M region, respectively, with different treatment durations with CaCl₂, KOH, and W.G. solutions and the overall comparison between these three different patterns and



Fig. 8 Variation of concentration of calcium ions with distance from anode

the untreated clay. In addition, a pure chemical sample made by directly mixing $CaCl_2$ and W.G. solutions to characterize a condition that the pure chemical solutions were used exclusively without inclusion of the clay, was also detected using XRD. The result is also shown in the same figure as indicated by pattern (a). The reason for this comparison of pattern (a) was that this series of ECT test might possibly induce a chemical reaction between $CaCl_2$ and W.G. solutions near the anode because these two chemical solutions were all injected from the anode. This comparison enables us to correctly deduce the newly formed products during the ECT.

Pattern (a) in Figs. 9 and 10 show that only the peaks located at 32° and 46° (2 θ) in the pure sample were detected by XRD, indicating the possible formation of new products due to the chemical reaction. By comparing pattern (a) with that in the JCPDS database system, the new product can be identified as the crystalline phase of NaCl.

Results at NA region indicate that a new peak in C72N72 was located at around 32°, and the peaks located at 26° and 46° have higher intensity than those shown in the original kaolinite pattern. After carrying out the mineralogical analysis by comparing the XRD patterns with those in the JCPDS database, it is found that the peak located at 26° represents the crystalline phase of quartz (SiO₂). The peaks located at 32° and 46° represent the





Fig. 10 XRD patterns in the M region

crystalline phase of sodium chloride (NaCl), which is comparable with the pattern (a). Therefore, it is important to note that there are some new crystalline phases formed during ECT progress near the anode, especially NaCl. However, there are no new peaks found in the XRD patterns of the tests with injecting additional KOH solution. This can be demonstrated that neutralization of environment near the anode by using alkaline before injecting W.G. solution can reduce the formation of some additional crystalline.

On the other hand, results in the M region showed no additional peaks as compared with the original kaolinite pattern. It can also be presumed that there is no new crystalline phase formed during the ECT progress in the M region.

According to the studies (Iler 1979; Wang *et al.* 2012), the sodium silicate (W.G.) may occur polymerization by forming the silica gel at an acidic environment. In addition, silicates will also react with polyvalent metalic ions to produce insoluble metalic silicate precipitates. Therefore, calcium salts will possibly react with sodium silicate to form calcium silicate hydrates (C-S-H) precipitates. However, the expected gel phases of silica gel or C-S-H gel were not identified in any XRD patterns because of their amorphous characteristics.

3.7 NMR Analysis

Figures 11 and 12 show the NMR patterns near the anode and middle part of the clay samples with different treatment duration with CaCl₂, KOH, and W.G. solutions. Results indicate that there is an individually high peak located at -91 ppm, which means that the chemical shift ranges from -86 to -100 ppm. Hence, the chemical structure can be determined as the Q₂ structure (*i.e.*, linear silicate) according to Davidovits (2008). This implies that the structure intensity all remains within the same range under the current injected chemical solutions.

3.8 Discussion

Results of the CPT resistance demonstrate that the injection of the CaCl₂, KOH and W.G. solutions can not only significantly improved clay strength near the anode but also raised the clay strength at middle part of the sample. The results from XRD and NMR analyses show that the newly formed products due to the injection of chemical solutions were insufficient to enhance such a large increase in the clay strength.

According to Iler (1979), when the silicic acid is provided from the W.G. solution under acidic environment (pH = $4 \sim 7$), it will speed up the effect of polymerization to reach the gel point, causing free silicic acid to polymerize and link together into branched silica gel network between the clay particles, leading to increase the strength of the soil. On the other hand, as the pH value ranges from 7 to 11, the silica molecules will begin to dissolve into silicate ones, forming discrete particles which are negatively charged and repel each other. Therefore, they do not form branched silica gel network between the clay particles. Since the environment in the anodic region is acid (pH = 4) as observed from pH measurement of the test of C72N72, the free units of silicic acid provided from the injection of W.G. solution thereby polymerize and increase clay strength of the C72N72 near the anode.



Chemical shift (ppm)





Fig. 12 NMR patterns in the M region

However, the polymerized silica gels and those additional products, *i.e.*, quartz (SiO₂) and sodium chloride (NaCl) as detected from the XRD analysis of the test of C72N72, would occupy the space in the NA region. This will reduce the injected volume of free silicic acid to flow into the middle part of the treated sample. As a result, polymerization of silicic acid cannot be relied upon to increase the clay strength in the middle region.

As observed in the pH measurement after treatment, injecting CaCl₂ solution for shorter period of time and using the alkaline solution (KOH) before injecting W.G. solution will effectively reduce the acidity in the NA region, as expected. Once under alkaline condition, it will therefore: (1) lower the ability of forming additional compounds while injecting W.G. solution, as observed in the XRD analysis (2) cause the silica molecules to begin to form discrete particles, which are negatively charged and repel each other under the alkaline condition (Iler 1979) (3) make the diffuse double layer become thinner due to a high electrolyte concentration of the KOH solution and increase the permeability of clay (Abdullah *et al.* 1997; Mitchell and Soga 2005; Chien *et al.* 2009).

Due to those above mentioned three effects in the clay sample, injection of the KOH solution will possibly lead the W.G. solution to flow smoothly into the middle region of the treated sample rather than to gel rapidly near the anode. Appropriate supply of silicic acid can thereby cause polymerization reaction of silicic acid and then increase the clay strength in the middle. On the other hand, the free Ca²⁺ ions present in insufficient amounts (less than 10 ppm) in the middle region within these cases, and the environment in the M region is too neutral. Hence, we can similarly demonstrate that it will be very difficult to induce cementation by the formation of C-S-H gels at the middle region and then to increase clay strength (Barker et al. 2004; Asavadorndeja and Glawe 2005, Ou et al. 2015a, Ou et al. 2015b). As a result, the possible mechanism of clay improvement in the M region in these tests should be attributed to polymerization reaction of silicic acid provided by W.G. solution.

However, the test C48K48N72, with a longer treatment of the KOH solution, indicated a less improvement in the M region. Such behavior can be explained by the fact that the injection of a strongly alkaline solution before injecting W.G. solution will result in a much higher alkaline condition in the NA region. It causes free Ca²⁺ ions deposits herein more easily to occur pozzolanic reaction or to form amorphous C-S-H gels before injecting the W.G. solution. Therefore, it will block the flow path in the clay sample, leading to reduce the injected volume of W.G. solution afterwards. Due to a relatively small amount of silicic acid flowing inside, it cannot induce a significant improvement in the middle part of the clay sample. However, as the possible formation of C-S-H gels presented, it is still good for increasing clay strength in the NA region. In conclusion, among all of the tests, the test C48K24N72 was the best result of clay improvement through the whole test sample.

4. CONCLUSIONS

In this study, an attempt has been made to improve the clay strength from the anode through the middle area of the treated kaolinite when the CaCl₂ solution was injected followed by the injection of the W.G. during the ECT. It was found that the injection of the CaCl₂ solution with a short period of duration, then the alkaline solution (KOH), and lastly the W.G. solution can obtain improvement through middle area of the treated kaolinite. Use of appropriate amount of alkaline solution and reduction of treatment duration of the CaCl₂ solution before the injection of the W.G. solution can slow down the polymerization effect of silicic acid in the NA region as compared with the effect at acidic environment. Moreover, a high concentration of KOH solution will make the diffuse double layer become thinner and increase the permeability of clay. Therefore, it will cause sufficient the W.G. solution to flow from the anode into middle part of the treated kaolinite, causing polymerization reaction of silicic acid to take place and then to increase clay strength. This explanation will be the possible mechanism of clay improvement in the M region. However, once a highly alkaline environment is created near the anode, it will cause the pozzolanic reaction to occur before injecting W.G., forming additional C-S-H gels to block the flow path in the NA region. Therefore, it will be difficult to get improvement in the M region due to a relatively small amount of silicic acid flowing inside.

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