

ELECTRO-OSMOTIC CHEMICAL TREATMENTS: EFFECTS OF Ca^{2+} CONCENTRATION ON THE MECHANICAL STRENGTH AND pH OF KAOLIN

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Abstract—Electro-osmotic chemical treatment is an innovative method to improve the strength of soft clays for geotechnical engineering purposes; the effectiveness of the treatment may be related to treatment time, the concentration of the solutions injected, and to variation of pH in the clay. The objective of this study was to investigate the relationship between the above-mentioned factors and the improvement in strength when calcium chloride solution was used as an injection material. A series of tests was carried out by injecting different concentrations of calcium chloride solution into a kaolin suspension, for different treatment times, during electro-osmosis. After the tests, the pH, cone resistance, water content, and concentration of Ca^{2+} in the kaolin at different locations were measured and analyzed. The results show that the concentration of Ca^{2+} in the kaolin, the pH, and the strength were increased near the cathode with increases in concentration of CaCl_2 and treatment time. An insignificant increase in strength, due to ion exchange over the entire specimen, for short treatment times of 2 to 24 h, was observed because of a small increase in concentration of Ca^{2+} and in pH. During long-term treatment (120 h), a considerable increase in concentration of Ca^{2+} (137.0 mg/g) and pH (pH = 10) was observed near the cathode. This led to a pozzolanic reaction, which in turn caused a significant increase in the mechanical strength of the kaolin.

Key Words—Electro-osmotic Chemical Treatment, Ion Exchange, Pozzolanic Reaction, Calcium Chloride.

INTRODUCTION

Kaolinite is a common clay mineral found in sediments, soils, hydrothermal deposits, and sedimentary rocks. Electro-osmotic chemical treatment has been applied to improve the strength of soft clays, which consist mainly of kaolinite. Since 1930 electro-osmosis has been used as a tool in geotechnical engineering to improve the mechanical strength of soft clays. Casagrande and Casagrande (1986) attempted to improve the strength of sensitive silty clay by electro-osmosis. Fundamental aspects of electro-osmosis in soils were reported by Gray and Mitchell (1967). Coelho *et al.* (1996) reported electro-osmotic phenomena in porous media. Beddiar *et al.* (2005) reported the influence of pH on pore-water pressure.

Dewatering of soils and clays during electro-osmosis involves movement of fluid; as a result, pore water is pulled, along with ions, toward one of the electrodes. The dewatering process leads to consolidation and a consequent increase in strength of the system until

intergranular stresses are in equilibrium with the tension that develops in the pore water during electro-osmosis. Additional strength is gained by chemical reactions in the soil/clay matrix including cation exchange, cementation, and precipitation (Chen and Murdoch, 1999; Alshawabkeh and Sheahan, 2003; Mohamedelhassan and Shang, 2003; Alshawabkeh and Sheahan, 2004; Burnotte *et al.*, 2004; Asavadorndeja and Glawe, 2005; Paczkowska, 2005; Otsuki *et al.*, 2007; Angelica *et al.*, 2008; Chien *et al.*, 2009; Ou *et al.*, 2009). However, the chemical reactions usually occur when the pH is high, *e.g.* precipitation when the pH is >7. Therefore, pH plays an important role (Acar and Alshwabakeh 1993, Alshawabkeh and Acar 1996, Asavadorndeja and Glawe 2005, Beddiar *et al.* 2005, Otsuki *et al.* 2007). Understanding the relationship between ionic concentration, variations in pH, and the improvement in strength during electro-osmotic chemical treatment is critical. Using calcium chloride during the electro-osmotic chemical treatment has proven to be more effective than other types of solutions (Ou *et al.*, 2009), and so it was used in the present study, over different treatment times, to study the effects of Ca^{2+} concentration on the pH and mechanical strength of soil during electro-osmotic chemical treatment with a commercial kaolin serving as the analog for soil.

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METHODS AND MATERIALS

ICP-AES and pH

Electro-osmotic cell

A modified version of the electro-osmotic cell designed by Ozkan *et al.* (1999) was used in the present study. The cell was made up of a Plexiglas cylinder 160 mm in diameter and 470 mm long (Figure 1). The top plate, connected to the electrode (anode), had holes bored to accommodate an injection tube and three valves set to exhaust the gas. In order to measure the volume of injection solution and drain water during the experiment, two pressure meters were set at the bottom. Platinum (Pt)-coated titanium (Ti) meshes were used as electrodes which were connected to a power-supply device and a current meter in order to measure the variation of current during the experiment. Several O-rings were fastened on both ends of the cell to prevent leakage.

Well mixed and finely ground 0.5 g samples of kaolin were digested in 10 mL of aqua regia (1:3 ratio of HNO_3 and HCl) on a hot plate for 3 h at 110°C . After evaporation to near dryness, the residue was dissolved in 20 mL of 2% nitric acid and filtered using Whatman No. 42 filter paper. The filtrate was transferred to a 100 mL standard flask and diluted up to the mark with distilled water. A JY 2000-2 model inductively coupled plasma atomic emission spectrometer (ICP-AES) was used to analyze for Ca^{2+} . The instrument was set in accordance with the manufacturer's instructions. The concentrations of Ca^{2+} ions in the kaolin samples before electro-osmotic chemical treatment (untreated) and after injection of distilled water for 8 h (blank) were also analyzed. The relative coefficient of variation of Ca^{2+} was found to be

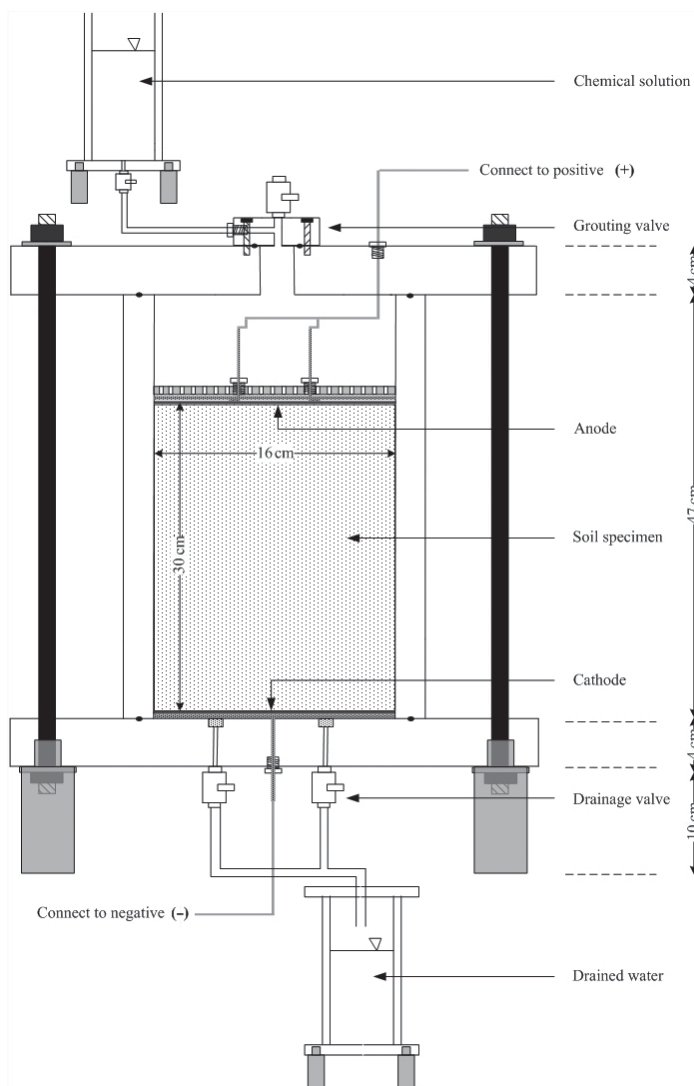


Figure 1. Schematic diagram of the electro-osmotic chemical grouting apparatus.

0.89% after six replicate determinations of 10 and 100 ppm.

The pH was measured using a portable pH meter (Mettler Toledo Seven Go SG2 – FK). The pH of the untreated kaolin sample was also measured.

Material and test procedure

The sample used in this study was kaolin from Warren County, Georgia, as supplied by U.S. Silica Co. (Berkeley, California) (Table 1). The supplier reported this kaolin to have a cation exchange capacity of 3.4 cmole/kg and a chemical composition of SiO₂ (44.50%), Al₂O₃ (39.50%), Fe₂O₃ (0.50%), CaO (0.5%), MgO (0.07%), (TiO₂ (1.00%), K₂O (0.04%), and Na₂O (0.52%). The kaolin was mixed with a sufficient amount of distilled deionized water (60%) in a mechanical mixer for 40 min. The kaolin suspension was then placed in the experimental cell in three layers and mixed simultaneously with a rod to avoid air bubbles. After placing the kaolin in the experimental cell, it was covered with a filter paper, the electrode was attached,

and the top porous plate added as shown in Figure 1. After setting up the specimen, the electro-osmotic chemical treatment was carried out by injecting different concentrations of CaCl₂ (0.5, 1.0, and 1.5 N) over different treatment times (2, 4, 8, 16, 24, and 120 h) with a constant voltage gradient (10 V/m) from the anode.

The samples were collected at five different positions, *i.e.* near the anode (NA), far from the anode (FA), in the middle (M), far from the cathode (FC), and near the cathode (NC), to measure different variables such as water content, pH, and Ca²⁺ ion concentration in the kaolin after electro-osmotic chemical treatment. At each position, three samples were collected and mixed well to obtain a typical sample from the location (Figure 3). The cone resistance from the anode to the cathode was also measured at C1, C2, and C3 before and after treatment as shown in Figure 3.

Cone resistance

The strength of the kaolin before and after treatment was measured using a specially designed laboratory cone-penetration device (Figure 2), which was attached

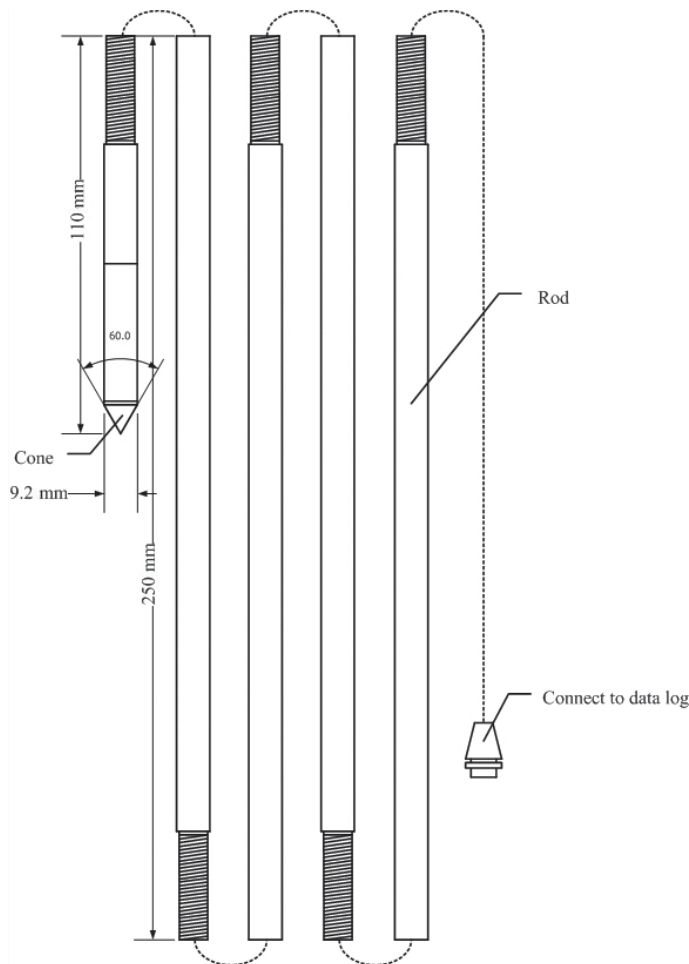


Figure 2. Laboratory cone-penetration apparatus.

Table 1. Basic properties of the kaolin used in this study.

Liquid limit (%)	46
Plastic limit (%)	25
Plastic index (%)	21
Specific gravity	2.61
Particle size (µm)	~1.0–2.0

Table 2. Ca²⁺ concentrations in untreated kaolin sample. (Injection of distilled water instead of CaCl₂, with 8 h of treatment time.)

Treatment time	Distance from anode	Concentration of Ca ²⁺ in kaolin (mg/g)
8 h	NA	12.782
	FA	12.776
	M	12.536
	NC	11.646
	FC	15.362

to an adjustable rod 9.2 mm in diameter. The apparatus was capable of measuring the cone resistance at different locations and depths.

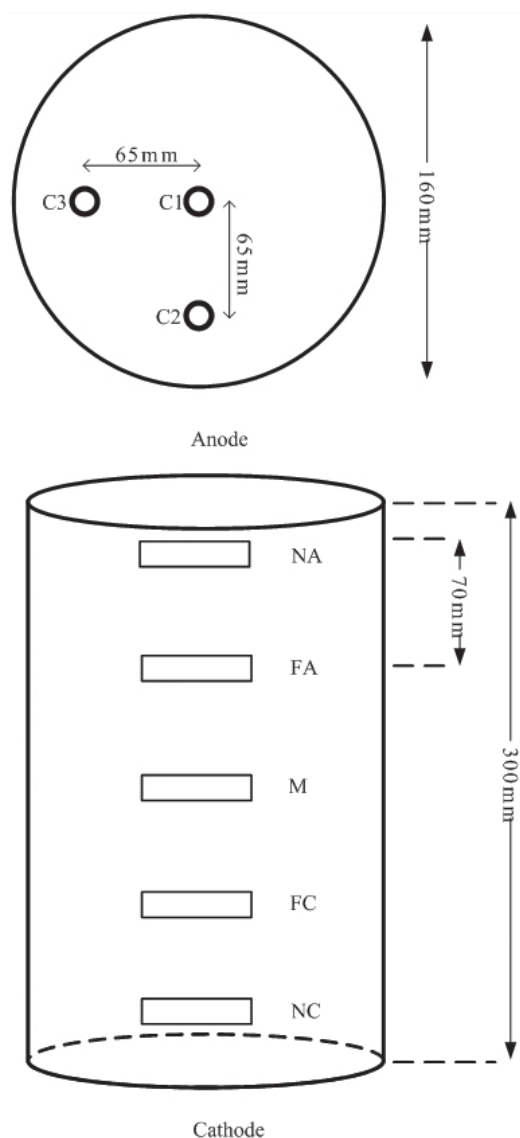


Figure 3. Schematic diagram of penetrated point (aerial view and side view).

RESULTS AND DISCUSSION

The results of cone resistance, concentration of Ca²⁺ ions, and pH of the kaolin samples are summarized in Tables 2, 3, and 4.

Water content

The distribution of the water content from the anode to the cathode when different concentrations of CaCl₂ were injected over different treatment times (Figure 4a–c) revealed that the water content was 60% before the electro-osmotic chemical treatment. The water content of samples injected with 0.5 N CaCl₂ and reacted for 2 and 4 h was greater than that of the untreated sample, and remained nearly constant for all locations in the specimen. When the reaction times were 8, 16, 24, and 120 h, initially, the water contents decreased to 58%, 56%, 54%, and 53%, respectively, at the NA positions, and finally, the water content increased to 61% at the NC position.

For treatment times of 2 and 8 h, the water content at NA increased initially, when 1.0 N CaCl₂ was injected, and then remained stable with increasing distance from the anode (Figure 4b). When the treatment times were 4, 16, 24, and 120 h, the water contents decreased to 58%, 55%, 57%, and 52%, respectively, at the NA positions. The decrease in water content in the NA region was consistent with the longer treatment time. The water contents at the NA position were initially small and increased slightly at the FC position, and remained stable at the NC position when 1.5 N of CaCl₂ was injected for different treatment times, *i.e.* 4, 16, and 24 h, but not for 2 h of treatment time (Figure 4c). The water contents decreased and reached a minimum of 54% at the NC position with 1.5 N CaCl₂ and 120 h of treatment. As described above, the decrease in water content at the NA position was consistent with the longer treatment time.

pH

The pH profile after electro-osmotic chemical treatment (Figure 5) revealed significant variation in the pH from the anode to the cathode in samples treated for 24 and 120 h and with different concentrations: 0.5, 1.0, and

Table 3. Effects of treatment time and solution concentration on the Ca²⁺ concentration in kaolin.

Treatment time	Distance from anode	Concentration of CaCl ₂ solution (N)		
		0.5	1.0	1.5
		Concentration of Ca ²⁺ in kaolin (mg/g)		
2 h	NA	13.340	9.968	11.886
	FA	12.636	9.224	11.304
	M	12.286	9.728	10.248
	FC	12.176	9.792	10.816
	NC	12.122	9.384	11.976
4 h	NA	14.918	16.332	11.338
	FA	11.876	9.420	9.260
	M	12.926	8.838	9.288
	FC	12.540	9.080	9.354
	NC	11.464	9.040	9.372
8 h	NA	19.932	10.276	13.296
	FA	13.770	13.756	12.652
	M	12.064	8.898	12.354
	FC	11.872	8.772	10.352
	NC	12.960	9.102	18.614
16 h	NA	13.880	27.482	18.812
	FA	17.530	29.554	14.948
	M	12.448	20.342	15.644
	FC	12.802	14.320	14.382
	NC	22.272	25.250	31.904
24 h	NA	15.868	21.146	51.306
	FA	19.706	45.708	65.128
	M	14.650	19.406	22.376
	FC	13.320	16.702	22.878
	NC	23.044	21.896	50.940
120 h	NA	35.294	61.062	101.426
	FA	17.994	38.890	95.952
	M	20.926	71.784	79.108
	FC	41.858	64.758	100.816
	NC	117.812	133.636	137.084

1.5 N CaCl₂. The pH was almost neutral for the samples treated for 2, 4, 8, and 16 h (with concentrations of 0.5, 1.0, and 1.5 N CaCl₂). When the specimen was treated for 24 h with 0.5, 1.0, or 1.5 N CaCl₂, significant changes in pH were observed at the NC position. One third of the specimens were consistently alkaline (pH>7), *i.e.* from the NC to the FC positions. The neutral point (pH = 7) was ~20 cm from the anode.

Significant change in pH, which reached a maximum of 10.21, was observed from the M to the NC positions for samples treated for 120 h with 0.5, 1.0, or 1.5 N CaCl₂. Half of the specimens, *i.e.* from the NC to the M positions, were completely alkaline (pH>7). The neutral point (pH = 7) was ~10 cm from the anode. These data indicate that the increase in the alkalinity depended on treatment time and on the concentration of the injection solutions.

Table 4. pH of kaolin samples after treatment with different Ca²⁺ solution concentrations for varying time intervals.

Distance from anode	2 h treatment time			24 h treatment time			120 h treatment		
	0.5 N	1.0 N	1.5 N	0.5 N	1.0 N	1.5 N	0.5 N	1.0 N	1.5 N
NA	6.47	6.66	6.75	6.68	7.13	6.23	4.75	5.04	5.02
FA	6.63	6.55	6.86	6.52	6.75	6.5	4.45	4.97	5.55
M	6.61	6.66	6.86	6.83	6.92	6.8	6.59	9.61	9.77
FC	6.65	6.67	6.84	7.22	6.97	6.74	9.43	9.75	10.09
NC	6.69	6.65	6.91	8.96	8.44	9.21	10.57	9.94	10.21

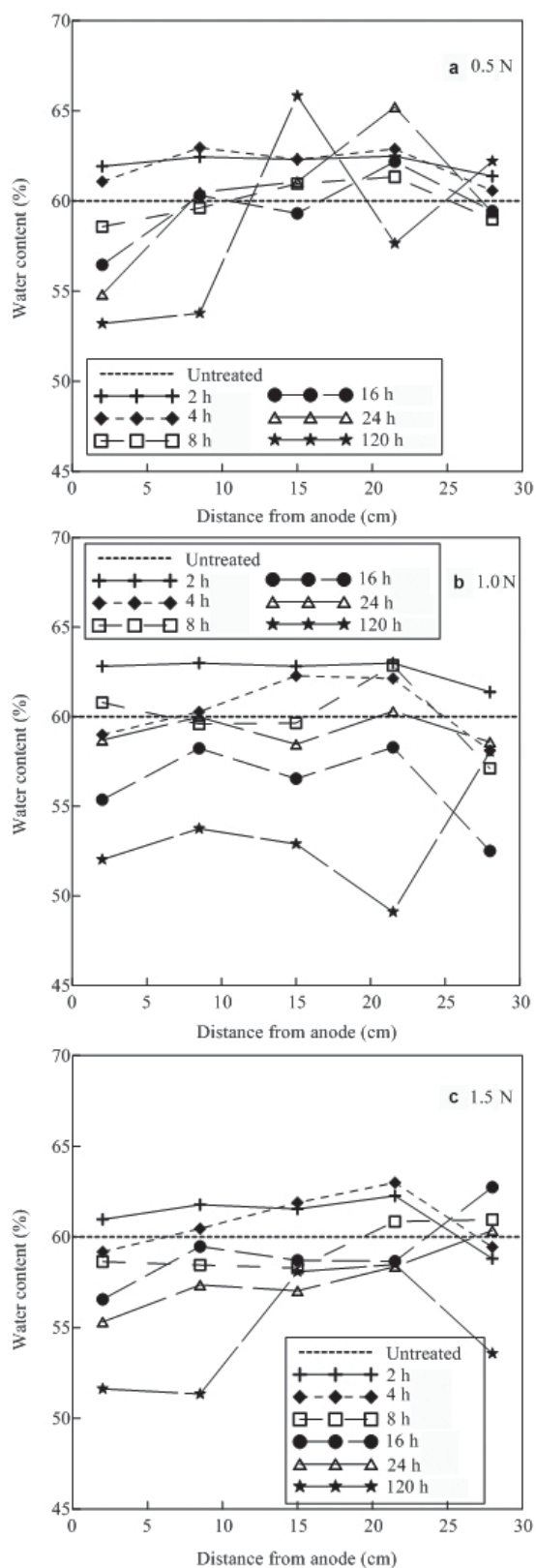


Figure 4. Variation of water content for 0.5, 1.0, and 1.5 N CaCl_2 with different treatment times.

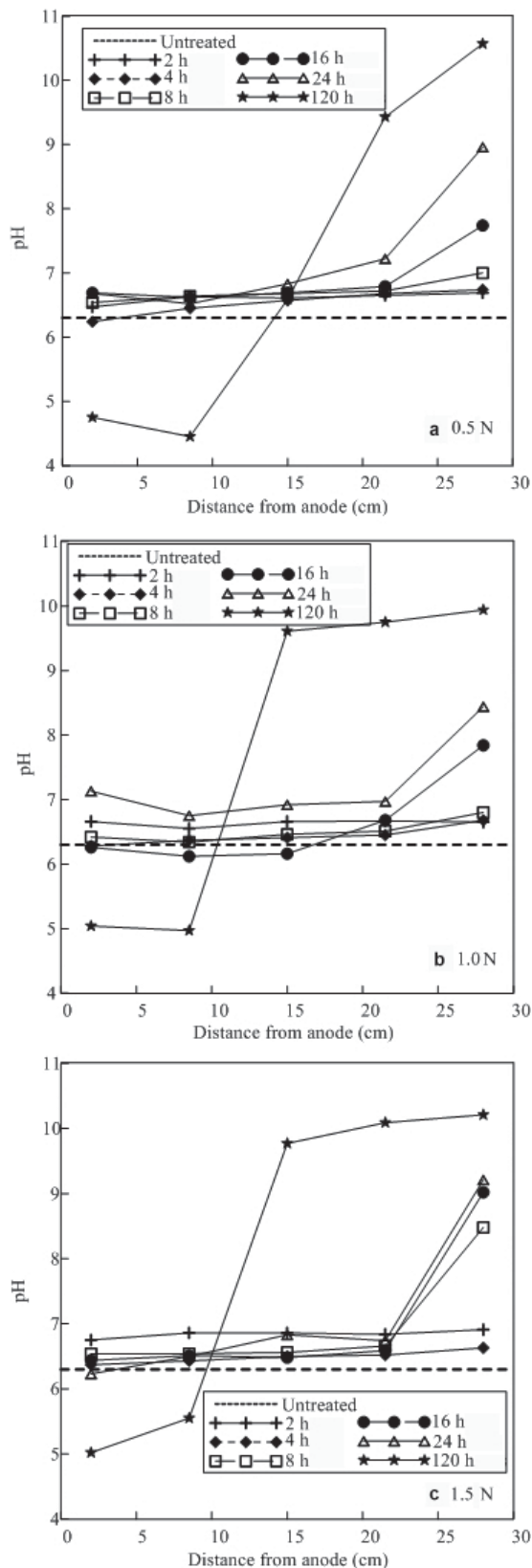


Figure 5. Effect on pH of increasing concentration of CaCl_2 and treatment times.

Ca²⁺ concentration

The concentration of Ca²⁺ ions in the kaolin after treatment is shown in Figure 6. When different concentrations of CaCl₂ (0.5, 1.0 and 1.5 N) were injected, in conjunction with 2, 4, 8, and 16 h treatment times, no significant change in concentration of Ca²⁺ was observed from anode to cathode except for the 16 h samples at the NC position (Figure 6a–c). A significant increase in concentration of Ca²⁺ was observed for the samples treated for 24 and 120 h. With the 24 h treatment, the concentration of Ca²⁺ in kaolin was increased at the NC position (to 23.04 mg/g, 21.89 mg/g, and 50.94 mg/g) when concentrations of 0.5, 1.0, and 1.5 N were used, respectively. With the 120 h treatment, a prominent change in concentration of Ca²⁺ was observed at the M, FC, and NC positions and reached a maximum of 117.8 mg/g, 133.6 mg/g, and 137.0 mg/g when concentrations of 0.5, 1.0, and 1.5 N were used, respectively.

Cone resistance

The effect of the 0.5 N, 1.0 N, and 1.5 N concentrations with 2, 4, 8, and 16 h treatment times on the cone resistance (Figure 7a–c) was that the injection of different concentrations of CaCl₂ for 2, 4, 8, or 16 h produced no significant increase in cone resistance from anode to cathode, especially from the NA to the FC positions. At the NA position, the cone resistance was increased slightly due to cation exchange and decreased water content (effect of electro-osmosis) for the 2-, 4-, and 8-h experiments. The entire samples' cone resistance decreased and became zero at the M position and remained at zero up to the NC position. When the treatment time was 16 h, the cone resistance was increased slightly at the NC position due to a small amount of precipitation of Ca²⁺. The variation in pH and concentration of Ca²⁺ near the cathode (Figures 5 and 6) confirms the cone resistance behavior above.

The different concentrations with the 120-h treatment time produced a remarkable increase in the cone resistance (Figure 7d–f) compared with the 24 h samples. The cone resistances in the NC region for clay which underwent 120 h of treatment at all three concentrations of CaCl₂ were greater than those for other treatment times and reached 1007 kPa, 1514 kPa, and 2909 kPa, for concentrations of 0.5 N, 1.0 N, and 1.5 N, respectively.

DISCUSSION

In electro-osmotic chemical treatment with injection of saline solutions, the formation of cementing agents depends primarily on kaolin pH and on the availability of free Ca²⁺ ions in the system (Asavadorndeja and Glawe, 2005).

When Ca²⁺ ions are injected during electro-osmotic chemical treatment, two reaction mechanisms, ion

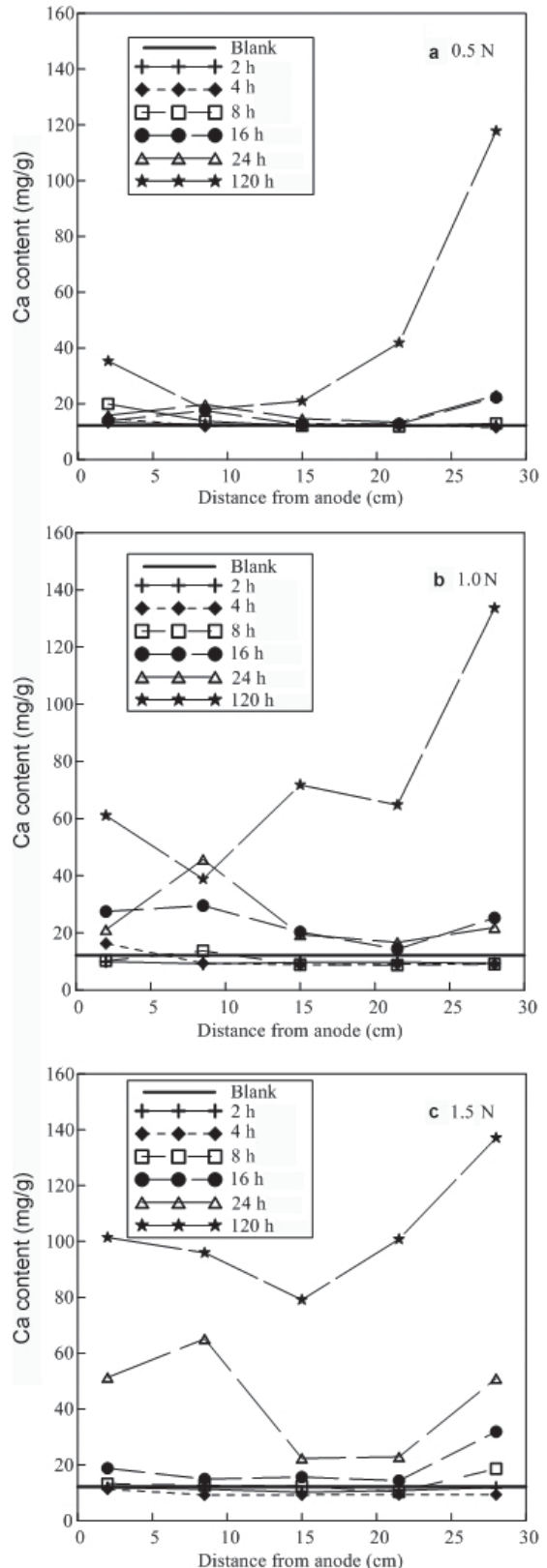


Figure 6. Effect of different time intervals on kaolin concentrations of Ca²⁺ with injection of 0.5, 1.0, and 1.5 N CaCl₂.

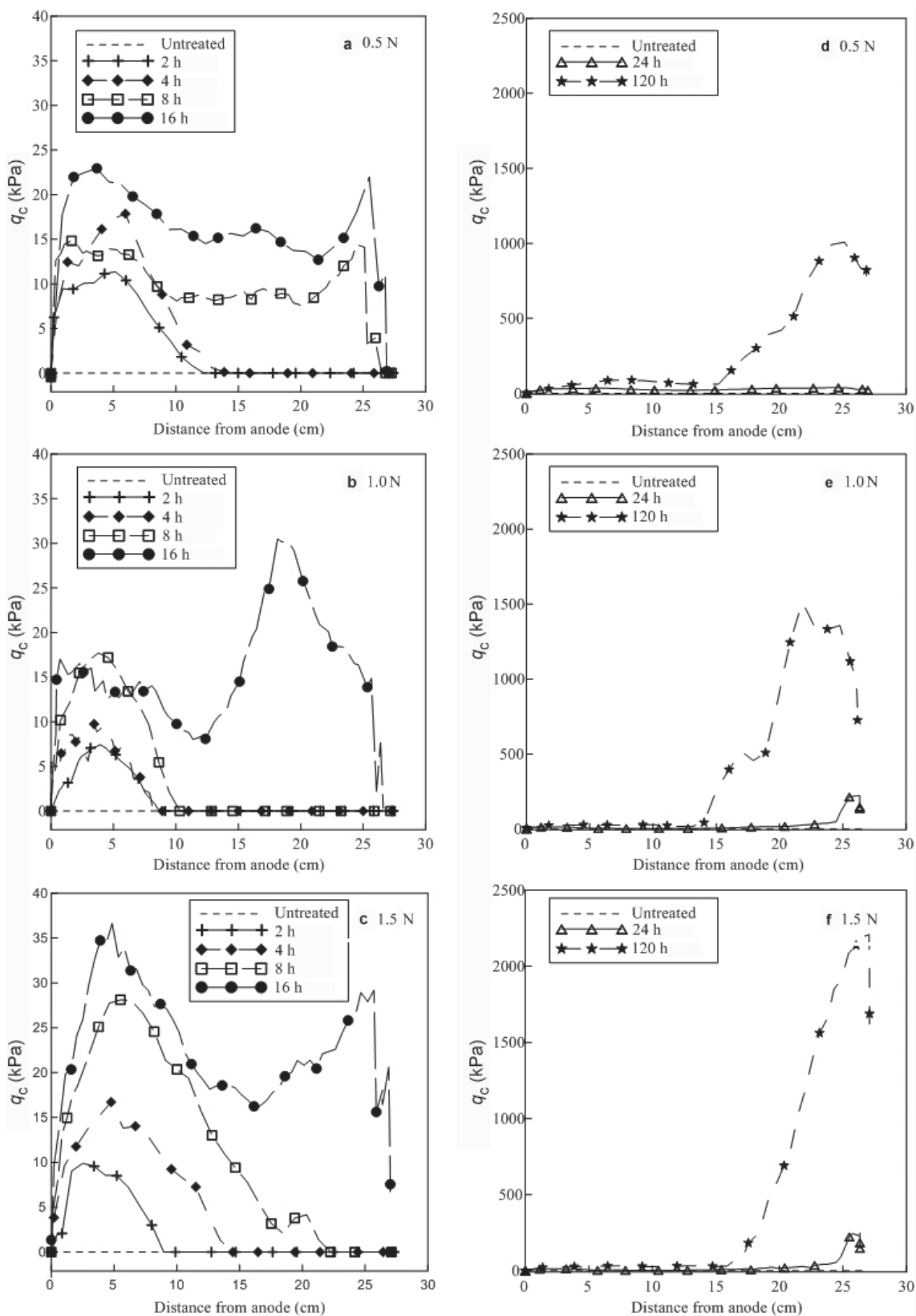


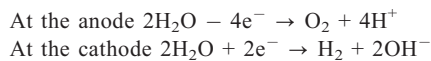
Figure 7. Variation of q_c for 0.5, 1.0, and 1.5 N CaCl_2 with different treatment times.

exchange and pozzolanic reaction, are responsible for the increase in mechanical strength. The OH sites in kaolin-group minerals in soils are important in terms of ion-exchange reactions during electro-osmotic chemical treatment. Cliff *et al.* (2008) characterized the OH groups of kaolin-group minerals with the help of low-temperature Fourier-transform infrared spectroscopy, analyzing samples from different parts of the world. The ion-exchange reaction of Ca^{2+} and monovalent ions leads to flocculation and coagulation of kaolin particles into large colloids. The pozzolanic reaction occurs primarily between siliceous materials and slaked lime (calcium hydroxide) or between siliceous material and aluminates, forming calcium silicate hydrates (CSH) or calcium aluminate hydrates (CAH) (Diamond and Kinter, 1965), which are cementing agents that bind the kaolin particles together. The ion-exchange reaction is relatively fast but provides only small increases in strength; the pozzolanic reaction is comparatively slow, but causes significant increases in strength (Assarson *et al.* 1974).

The pozzolanic reaction is as follows:



In general, during electrolysis H^{+} and OH^{-} ions will be generated at the anode and cathode, respectively:



Hence, the pH drops near the anode and increases near the cathode during electrolysis. As a result, acidic and alkaline conditions will be developed near the anode and cathode, respectively, during electro-osmosis with injection.

In the present study, the electro-osmotic chemical treatment could be divided into two phases: short term (2–24 h) and long term (120 h). In general, the ionic mobility of hydrogen ions is ~ 1.8 times faster than that of hydroxide ions and the electro-osmotic flow is toward the cathode. Almost the entire specimen (from the NA to the FC position) was, therefore, subject to acidic conditions ($\text{pH} < 7$) except at the NC position. During the short-term treatment experiments, the ion exchange reaction and the short-term treatment promoted the general phenomenon described above and, at the same time, the pozzolanic reaction was prevented in areas of acidic conditions. No significant variation in the concentration of Ca^{2+} or pH was observed due to short treatment times. The results prove, therefore, that no marked increase in the strength (cone resistance) of the test samples was observed from the M to the FC position (Figure 7). By contrast, a small increase in strength was observed with a slight increase in concentration of Ca^{2+} in kaolin under alkaline conditions near the cathode (NC) position for 16 and 24 h of treatment. Pozzolanic reaction did not occur at the NC position, in spite of the

slightly alkaline conditions, because of the short treatment time.

During the long-term treatment experiment, OH^{-} ions still move slowly toward the anode, compared with H^{+} ions. During the movement of ions, H^{+} may react with OH^{-} ions leading to the formation of H_2O , or it may react with Ca^{2+} ions and lead to the formation of $\text{Ca}(\text{OH})_2$ as a precipitate which will neutralize the acidic front formed during short-term treatment. After neutralization, the alkaline front would have formed slowly and the pH would increase gradually from the NC to the FA position, causing the alkaline region to increase. On the other hand, for high-pH, long-term treatment, and high concentrations of Ca^{2+} , the formation of a pozzolanic reaction is promoted, *i.e.* the formation of CSH under alkaline conditions. Significant increases in the strength (cone resistance) with increasing concentration of Ca^{2+} and pH (Figures 5 and 6) and treatment time in test samples was, therefore, observed (Figure 7) from the FA to the NC positions. The cone resistance of treated kaolin was very consistent with concentration of Ca^{2+} at the cathode region (FC and NC) under alkaline conditions (Figure 8).

CONCLUSIONS

The purpose of this study was to investigate the movement and accumulation of Ca^{2+} ions and change in pH during electro-osmotic chemical treatment with different concentrations of CaCl_2 and treatment times and their role in improving the strength of kaolin. Variables such as water content, pH, Ca^{2+} concentration, and cone resistance of the kaolin samples were measured after electro-osmotic chemical treatment. The results

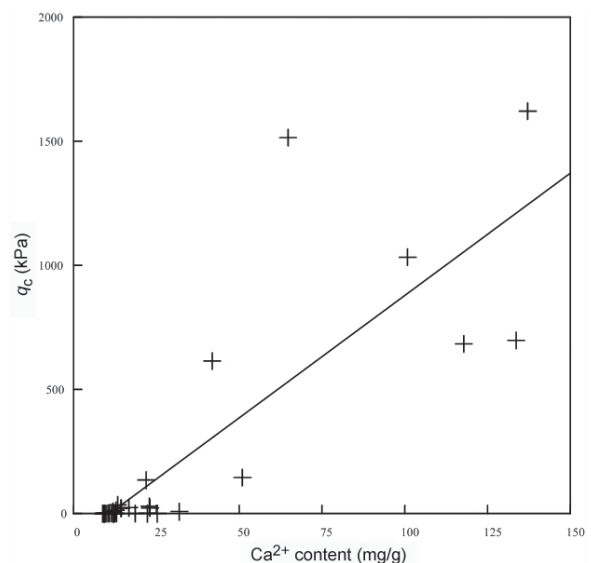


Figure 8. Relationship between q_c and the concentration of Ca^{2+} in the kaolin.

show that with short-term treatment and with different concentrations of CaCl_2 , the concentrations of Ca^{2+} ions from the anode to the cathode were almost identical, with the exception of 16- and 24-h samples at the NC position. An insignificant increase in strength was observed for short treatment times due to ion exchange over the entire sample. The Ca^{2+} ions move toward and accumulate near the cathode during long-term treatment. The pH (pH = 10), Ca^{2+} ion concentration (137.0 mg/g), and cone resistance (maximum 2209 kPa) increased significantly in the samples collected at the NC and FC positions after 120 h of treatment. The results confirm that the improvement in kaolin mechanical strength was mainly due to the precipitation and pozzolanic reactions of Ca^{2+} ions at the NC position of the cathode during electro-osmotic chemical treatment.

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