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Effect of calcium chloride and kaolinite on shear strength and shrinkage of cement grout

Sina Kazemian¹*, Bujang B. K. Huat² and Maassoumeh Barghchi¹

¹Department of Civil Engineering, Bojnourd Branch, Islamic Azad University, Bojnourd, Iran.
²Department of Civil Engineering, University Putra Malaysia, Serdang, Selangor, Malaysia.

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Grouting is injecting a liquid (cement and additives) into voids within a structure or soft soils for displacing the gases and liquids from within these. The varieties of additives for different grouting purposes are using with cement. Calcium chloride is known as accelerator in the grout, it causes to decrease the set time of grout and it has a minor effect on fresh grout properties. Kaolinite or some other fine mineral soils are used as filler in the grout to get cost-effective grout. In this study, the different effects of calcium chloride and kaolinite were investigated on shear strength of soil treated by cement grout, and shrinkage of the grout. Samples were prepared by mixing specific amounts of cement, water, and organic soil with different ratios of calcium chloride and kaolinite. Unconfined compressive strength (UCS) of the soil treated was evaluated after curing for 3 and 30 days and the linear shrinkage of each grout were measured after 30 days. The results indicated that, kaolinite has positive effect on shear strength of soil treated and shrinkage of the cement grout. By increasing kaolinite, the shear strength of samples increased and the shrinkage of grout decreased respectively. On the other hand, calcium chloride seemed to have different effects on the shear strength of samples and shrinkage of grout. It did not have tangible effect on shrinkage of the grout except in the grout with 1% calcium chloride.

Key words: Grouting, kaolinite, calcium chloride, cementation, pozzolanic reactions.

INTRODUCTION

Soil improvement refers to any method or techniques that improve the engineering properties of soil, like shear strength, compressibility, stiffness and permeability. Raju (2009) classified the soil improvement methods to the following principles: (i) consolidation (e.g. prefabricated vertical drains and surcharge, vacuum consolidation, stone columns), (ii) chemical modification (e.g. deep soil mixing, jet grouting, injection grouting), (iii) densification (e.g. vibro-compaction, dynamic compaction, compaction grouting), and (iv) reinforcement (e.g. stone columns, geosynthetic reinforcement). The Deep Mixing Method (DMM) is today accepted world-wide as a soil improvement method which is performed to improve the strength, deformation properties and permeability of the soil. It is based on mixing binders, such as cement, lime, fly ash, chemical grouts and other additives, with the soil by the use of rotating mixing tools in order to form columns of a hardening material, since chemical reactions between the binder and the soil grains are developed (Costas and Maria, 2008).

The demand for improving and stabilizing land for different purposes is expected to increase in the future and the best way to fulfill it is by using DMM. The main advantage of these methods is long term increase in strength, especially for some of the binders used. Pozzolanic reaction can continue for months or even years after mixing, resulting in the increase in strength of cement stabilized clay with the increase in curing time (Bergado, 1996; Roslan and Shahidul, 2008). Majority of the companies which are working in these fields agree that, the DMM can be divided into three common techniques (Andromalos et al., 2000; Keller, 2005; Raito, 2008).

*Corresponding author. E-mail: sina.kazemian@gmail.com.
Shallow Soil Mixing (SSM), which uses a single mechanical mixing auger located at the end of the drilling tool (Kelly bar), (ii) Cement Deep Mixing System (CDM), which utilizes a series of overlapping augers and mechanical mixing shafts, (iii) Injection and grouting methods which can be considered a type of soil mixing. The Federal Highway Administration has suggested that, these techniques can be classified based on Elias et al. (1998): (i) method of additive injection (that is wet or dry injection), and (ii) method by which additive is mixed (that is rotary/ mechanical energy or by high pressure jet). Grouting or injection generally is used to fill voids in the ground (fissures and porous structures) with the aim to increase resistance against deformation, to supply cohesion, shear–strength and uniaxial compressive strength or finally (even more frequently), to reduce conductivity and interconnected porosity in an aquifer (Moseley and Kirsch, 2004). Gleize et al. (2003) stated that, pozzolanic materials and fillers (kaolinite and sand) can be added to the chemical grouts as these materials change the microstructure of mortars and consequently modify some of its properties. Chemical stabilization is the effective method to improve the soil properties by mixing additives to soils.

Today, commercially available chemical grouts cover a wide range of materials, properties and costs, and give the grouter the opportunity to select a grout for specific job requirements. These reagents bring an almost instant setting time and produce very low penetrability type grout, that are unsuitable for permeation treatments; but in the soils with high ground water level, it can be effective as the water causes the grout to be diluted (Rawlings et al., 2000). The function of the accelerator is to control gel time and impart strength to the gel. The effect of the accelerator is important at temperatures below 37°C and increases in importance as the temperature decreases. An excessive amount of accelerators may result in undesirable flocculation or formation of local hardening. This causes variations in both the gel and setting times that would tend to plug injection equipment or restrict penetration, resulting in poorly grouted area. Therefore, a retarder should be added in the mixture for delaying the setting time and formation of gelation (USACE, 1995).

Calcium chloride has been used in concrete since 1885 and finds application mainly in cold weather, when it allows the strength gain to approach that of concrete cured under normal curing temperatures (Rixom and Mailvaganam, 1986). Calcium chloride has the ability to accelerate cement hydration and reduce set time by as much as two thirds. Research has shown that, the important contributions of cold weather protection and early strength of concrete, calcium chloride provides other benefits as well: (i) improves workability, (ii) improves strength of air-entrained concrete, (iii) reduces bleeding, these advantages cause the production of better quality concrete faster. Concrete acceleration with calcium chloride greatly facilitates completing jobs as quickly and economically as possible (The Dow Chemical Company, 2006). This study was aiming to describe the effects of different ratios of calcium chloride and kaolinite on cementation and pozzolanic reactions process of cement grout in the soil, characterized by Unconfined Compressive Strength (UCS) and effect of these compounds in the shrinkage of cement grout.

**EXPERIMENTAL METHODS AND MATERIALS**

**Materials**

Cement used in this study (as binders) were sourced from Anuza Enterprise Company, Malaysia respectively. The chemical composition of cement as provided by the manufacturers, are summarized in Table 1. Furthermore, calcium chloride anhydrous powder (CaCl₂) and kaolinite were used as a reactor/accelerator and filler respectively. The kaolinite [Al₂Si₂O₅(OH)₄] structure is made up of silicate sheets (Si₂O₅) bonded to aluminum oxide/hydroxide layers [Al₂O₃(OH)₃] called gibbsite layers. Tables 2 and 3 show the chemical composition of calcium chloride and kaolinite respectively.

**Table 1. Chemical composition of cement.**

<table>
<thead>
<tr>
<th>Constituent</th>
<th>(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>21</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>5.3</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>3.3</td>
</tr>
<tr>
<td>CaO</td>
<td>65.6</td>
</tr>
<tr>
<td>MgO</td>
<td>1.1</td>
</tr>
<tr>
<td>SO₃</td>
<td>2.7</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>0.9</td>
</tr>
<tr>
<td>Fineness (% passing 45 µm)</td>
<td>90.5</td>
</tr>
</tbody>
</table>

**Table 2. Chemical composition of calcium chloride.**

<table>
<thead>
<tr>
<th>Constituent</th>
<th>(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minimum assay</td>
<td>96</td>
</tr>
<tr>
<td>SiO₂</td>
<td>0.02</td>
</tr>
<tr>
<td>Ca(OH)₂</td>
<td>0.04</td>
</tr>
<tr>
<td>Mg²⁺</td>
<td>0.6</td>
</tr>
<tr>
<td>alkalis (sulfate)</td>
<td>0.6</td>
</tr>
</tbody>
</table>

**Table 3. Physico-chemical parameters of kaolinite.**

<table>
<thead>
<tr>
<th>Constituent</th>
<th>(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>45.80</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>39.55</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>0.57</td>
</tr>
<tr>
<td>CaO</td>
<td>0.41</td>
</tr>
<tr>
<td>MgO</td>
<td>0.14</td>
</tr>
<tr>
<td>FeO</td>
<td>0.18</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.03</td>
</tr>
</tbody>
</table>

**EXPERIMENTAL METHODS AND MATERIALS**

**Materials**

Cement used in this study (as binders) were sourced from Anuza Enterprise Company, Malaysia respectively. The chemical composition of cement as provided by the manufacturers, are summarized in Table 1. Furthermore, calcium chloride anhydrous powder (CaCl₂) and kaolinite were used as a reactor/accelerator and filler respectively. The kaolinite [Al₂Si₂O₅(OH)₄] structure is made up of silicate sheets (Si₂O₅) bonded to aluminum oxide/hydroxide layers [Al₂O₃(OH)₃] called gibbsite layers. Tables 2 and 3 show the chemical composition of calcium chloride and kaolinite respectively.
Table 4. Grout formulae with their notations and group numbers.

<table>
<thead>
<tr>
<th>Grout no.</th>
<th>Grout formula (%)</th>
<th>Grout no.</th>
<th>Grout formula (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>K (%) Ca (%) Ce (%)</td>
<td></td>
<td>K (%) Ca (%) Ce (%)</td>
</tr>
<tr>
<td>1</td>
<td>20 0 20 20 0 30</td>
<td>2</td>
<td>20 0.1 20 0.1 30</td>
</tr>
<tr>
<td>2</td>
<td>20 0.25 20 0.25 30</td>
<td>3</td>
<td>20 1 20 1 30</td>
</tr>
<tr>
<td>3</td>
<td>20 2.5 20 2.5 30</td>
<td>4</td>
<td>20 5 20 5 30</td>
</tr>
<tr>
<td>4</td>
<td>20 10 20 10 30</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

K: Kaolinite, Ca: calcium chloride and Ce: Cement.

Experimental methods

In order to investigate the influence of calcium chloride and kaolinite on shrinkage of cement grout, different quantities of calcium chloride (1 mol/L) and kaolinite were admixed with specific amounts of cement as shown in Table 4. For preparing the samples to investigate the effect of calcium chloride and kaolinite on shear strength of cement grout, different ratios of cement were first thoroughly homogenized with the water in the ratio of 1:1 by a household mixer and then the grout were mixed with organic soil (750 g) and the desired amount of kaolinite, and calcium chloride were added to it (Table 4). After mixing thoroughly, the confection was then transferred to a cylindrical container of the same height and inner diameter as that used in the UCS test (38 mm in diameter and 76 mm high). Curing of the samples was accomplished by keeping the container with the confection submerged in distilled water in a large tray for 3 and 30 days in the constant temperature (22±2°C). For repeatability and reproducibility of the tests, six samples were prepared for each grout formula as mentioned before; and the variation in the shear strength of the six samples, during any curing period (3 and 30 days) was not more than 5%. The values of the shear strength reported here are the average values. The combinations of the chemicals used in preparing the grout are arranged according to the increasing component in the confection for easy readability and are shown in Table 4. UCS tests were performed according to BS 1377-7-7(1990) (e.g. British Standard Code: Ref. No. 1377, part 7, clause 7) on the samples at the end of 3 and 90 days of curing.

In the second part of this study, the linear volume shrinkage index (LVSI) was performed to evaluate the shrinkage of grouts cured in air. The linear shrinkage of grouts was determined according to BS 1377-7-7(1990). In this test, the standard dimensions of the semi–circular mould were 140 mm long and 25 mm in diameter. The fresh grouts (Figure 1) were poured in the molds and were air cured for 4 weeks. The dimensions of the samples were recorded everyday and the LVSI was calculated using Equation 1.
Percentage of shrinkage = \( \frac{L_0 - L_d}{L_0} \times 100\% \) \hspace{1cm} (1)

Where, \( L_d \) is the length after shrinkage, and \( L_0 \) is the original length of the sample.

**RESULTS AND DISCUSSION**

The influence of different compounds (calcium chloride and kaolinite) on cement grout has been investigated and the results are presented in Figures 2 and 3.

**Influence of calcium chloride on shear strength of cement grout**

The influence of calcium chloride was studied by preparing samples with different concentrations of calcium chloride \((0, 0.25, 0.5, 1, 2.5, 5\) and \(10\)% by weight of soil) as mentioned in grouts numbers 1 and 2. The effect of calcium chloride on the shear strength and moisture content of the samples after 3 and 30 days of curing are presented in Figure 2 for grout number 1. It was observed that (Figure 2) by increasing the calcium chloride concentration in grout number 1 from 0 to 1%, the shear strength of the samples increased from 208 to 264 kPa after 3 days of curing. Furthermore, by increasing the calcium chloride up to 10% the trend of the shear strength showed a reversal, that is, the shear strength decreased from 264 to 138 kPa. Similarly, the shear strength of the samples varied with an increase in curing time from 3 to 30 days for grout number 1, it was increased from 248 to 278 kPa by increasing calcium chloride from 0 to 1%, then the trend was reversed and the shear strength decreased from 278 to 149 kPa, when the concentration of calcium chloride increased from 1 to 10%, and the trend was similar for grout number 2 also. Kazemian et al. (2009) and Huat et al. (2011) stated that, when water comes in contact with cement, three phenomena take place; (i) cement reacts with water, this is called hydration, (ii) there are pozzolanic reactions between Ca(OH)\(_2\) from burnt cement and pozzolanic minerals in the soil, and (iii) there is ion exchange between calcium ions (from cement and additives) with ions present in the kaolinite, which leads to an improvement in the strength of the mixture. First step (when the calcium chloride concentration is less than 1%), the confection particles present in grout tend to go towards the particles with a zero net charge and will not repel each other. This is the reason for an increase in the shear strength the samples.

Conversely, due to the addition of extra calcium cations, the charge balances in the confection, giving a positive charge to the confection and thereby leading to the re-stabilization of the colloidal fraction and deflocculating of the large size particles. ACI Committee 212 (1963) and Transportation Research Board (1990) emphasized that, according to laboratory tests, most increases in compressive strength of concrete, resulting from the use of 2% of calcium chloride by weight of cement range from 400 to 1,000 psi (2.8 to 6.9 MPa) at 1 through 7 days, for 70°F (21°C) curing and long-term strength is usually unaffected and is sometimes reduced, especially at high temperatures.

**Influence of kaolinite on shear strength of cement grout**

The effect of kaolinite on samples was investigated by preparing different samples based on grout numbers 3
Figure 3. Influence of different kaolinite concentrations for grout number 3.

and 4 (Table 2) and the results are presented in Figure 3. The grout numbers 3 and 4 consisted of different amounts of kaolinite (0, 5, 10, 20, and 30% by weight of soil) while the amounts of other additives were kept constant. Firstly, the shear strength of the samples with grout numbers 3 and 4, after 3 days of curing, was observed to increase with an increase in the kaolinite content. Furthermore, with an increase in the curing time, the shear strength increased as well. For grout number 3, the shear strength increased from 238 to 275 kPa and from 254 to 284 kPa, respectively, after 3 and 30 days of curing (Figure 3). The trends of variations in shear strength for the samples with grout number 4 were similar to grout number 3.

The stated findings, agree well with the fact that (as mentioned earlier), cement initiates a chemical reaction with water called hydration. When tricalcium silicate (C₃S) and dicalcium silicate (C₂S) are mixed with water, calcium ions are quickly released into the solution with the formation of hydroxide ions. When the concentration of calcium and hydroxide ions reaches a certain threshold value, calcium hydroxides crystallize out of solutions and this finally leads to the production of calcium silicate hydrate (C–S–H) and thus, an increase in the shear strength of the treated samples. The pozzolanic reaction takes place with the mineral parts of the soil; since there is kaolinite in the mixture, it contains aluminous and siliceous minerals. Some of these possess pozzolanic properties, that is under certain conditions they can react with Ca(OH)₂ to form calcium aluminate silicate hydrate (C–A–S–H), which leads to an increase in the shear strength together (Janz and Johansson, 2002; Huat et al., 2011). As it mentioned earlier, the third reaction in this confection is ion exchange between calcium ions (from cement and calcium chloride) with ions present in the kaolinite which cause to increase, the shear strength by increasing the kaolinite and this was also evident in the confections (Figures 2 and 3).

Influence of calcium chloride and kaolinite on shrinkage of cement grout

The effect of calcium chloride and kaolinite were studied on shrinkage of cement grout. They were investigated by adding the same ratio of those compounds in the last section. The linear shrinkage of cement grout with different ratios of calcium chloride after 4 weeks is shown in Figure 4 for grout numbers 1 and 2. The shrinkage of grouts did not have a lot of effect by changing the ratio of calcium chloride in grout number 1. It indicates that, in samples with calcium chloride around 1%, the shrinkage is more than the other ratios. This trend is the same shrinkage of grout number 2 as well. These findings agree well with Shideler (1952) and Ramachandran (1984) findings, that stated that by adding calcium chloride in the concrete (around 2% of cement weight), the shrinkage will be increased in initial times of mixing due to at earlier periods may be attributed mainly to more hydration. It means that, in the specific ratio of calcium chloride that cause highest shear strength (in this study around 1% by the weight of soil) the highest shrinkage occurs because of more hydration.

The effect of kaolinite in the shrinkage of grout was studied as well. Figure 5 shows the effect of different ratios of kaolinite on the shrinkage of grout numbers 3 and 4. It was observed that, by increasing the ratio of kaolinite from 0 to 30%, the shrinkage of grout decreased
Figure 4. Influence of different ratios of calcium chloride on the shrinkage of grout numbers 1 and 2.

Figure 5. Influence of different ratios of kaolinite on the shrinkage of grout numbers 3 and 4.

from 6 to 1.7% for grout number 3. Furthermore, shrinkage of grout number 4 increased from 1.63 to 5.2% by decreasing kaolinite ratio from 30 to 0%. This is due to the fact that, kaolinite cause to observe water because of its high specific surface area and fill the voids in the mixture. CIRIA (2000) suggested that, the grout should have final shrinkage less than 25% and according to the above findings, adding kaolinite helps to improve the shrinkage, but the effect of kaolinite on viscosity of grout should be considered (Kazemian et al., 2010).

Conclusions
This study was carried out to investigate the effects of the calcium chloride and kaolinite on shear strength of treated soil and shrinkage of cement grout. It was observed that, by increasing calcium chloride within 1% by the weight of soil, shear strength of grout increased, after that it decreased due to an increased positive charge on the surface of particles, thereby leading to the re-stabilization of the particles and defloculating of the
large size particles.

The shear strength increased by increasing the kaolinite in the grout, due to increasing of pozzolanic reactions and ion exchange between calcium ions (from cement and additives) with ions present in the kaolinite and mineral part of soil. The effect of calcium chloride and kaolinite was investigated on shrinkage of grout as well. It was found that by increasing the kaolinite the shrinkage of grout decreased due to usage of water by kaolinite and calcium chloride had no tangible effect on shrinkage of grout, except in the samples with around 1% calcium chloride which had the highest shrinkage, due to more cementation and pozzolanic reaction in this sample.

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REFERENCES


