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Enhancing Electrokinetic Environment to Improve Physicochemical Properties of Kaolinite Using Polyvinyl Alcohol and Cement Stabilizers

Hossein Moayedi[1,*], *Afshin Asadi*[2], *Bujang B K Huat*[3], *Fatemeh Moayedi*[4], *Sina Kazemian*[5]

1. Department of Civil Engineering, Estahban Branch, Islamic Azad University, Estahban, Iran
2. Department of Civil Engineering, Isfahan Science and Research Branch, Islamic Azad University, Isfahan, Iran.
3. Department of Civil Engineering, Universiti Putra Malaysia, Serdang, Selangor, Malaysia
4. Department of Biology, Arsanjan Branch, Islamic Azad University, Arsanjan, Iran.
5. Department of Civil Engineering, Bojnourd Branch, Islamic Azad University, Bojnourd, Iran.

[*]E-mail: hossein.moayedi@gmail.com

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Adsorption of polymers on clay and flocculation of clay by polymers in presence of cement were studied in order to understand clay polymer interactions. Also, findings into the stabilization effect of the kaolinite that was mixed with various binders to form a stabilized soil are presented. Special attention was focused on two types of PVA: fully (PVA-F) and partially (PVA-P) hydrolyzed with varying degrees of concentration. Although, increasing polymer concentration in both PVA-F and PVA-P samples enhanced physicochemical results, PVA-F showed higher improvement than PVA-P. As a result, Unconfined compressive strength (UCS) of stabilized kaolinite increased as high as 5 to 109 times comparing with untreated kaolinite. According to 28 days curing time, the optimum dose of PVA was also evaluated 3gr/L and 1gr/L for PVA-P and PVA-F, respectively. Although, pH at isoelectric point was between 3.1 and 3.2, isoelectric point of kaolinite immersed in PVA solution observed at pH between 1.9 and 2.1.

Keywords: Zeta potential, clay, polyvinyl alcohol, Electrokinetic, soil improvement, Cement.

1. INTRODUCTION

In recent years, concern about soft problematic soil and its difficulties from the geotechnical points of view such as high liquid limit, low hydraulic conductivity, low strength and high compressibility have led to the development of many new binders for soft clay stabilization [1-6]. Generally, discrete clay particles have a negative surface charge that influences and controls the particle environment. This surface electric charge can be developed in different ways, including the presence of broken bonds and due to isomorphous substitution1 [7]. The quantity of this surface charge varies from mineral to mineral and is affected by pore fluid environment [6, 8-9]. On the one hand, kaolinite is a clay mineral, part of the group of industrial minerals, with the chemical composition $Al_2Si_2O_5(OH)_4$. It is a layered silicate mineral, with one tetrahedral sheet linked through oxygen atoms to one octahedral sheet of alumina octahedral. Kaolinite consist basically repetitive layers of tetrahedral sheet (silica) and octahedral sheet (alumina or gibbisite). It is called as 1:1 clay mineral, as sheets are held in such a way that, tips of the silica sheet and one of the layers of octahedral sheet form a single layer of 0.72nm thickness [7, 10-12]. Moreover, kaolinite is a neutrally balanced and held by hydrogen bonding in between the successive layers of the basic layer (hydroxyls of octahedral sheet and the oxygens of tetrahedral sheet). So, there is no space in between Kaolinite minerals [3, 5, 11]. In the past sodium silicate and calcium chloride were often used as grouting material in sandy soils because of their noticeable advantages such as non-contamination, non-toxicity and inexpensiveness. However, injection of sodium silicate and calcium chloride solutions into clay soils by hydraulic pressure will cause hydraulic fracturing, which makes the improvement backfire. Since Ca^{2+} in the calcium chloride solution and SiO_3^{2-} in the sodium silicate solution may migrate in clay under the electric field, sodium silicate and calcium chloride solutions can be beneficial as injection material for clay soils, which induces as good a cementation between particles under the electric field as those used in sandy soil under hydraulic pressure [6, 12-13]. On the other hand, Water based polymers like polyvinyl alcohol (PVA), polyvinyl pyrrolidone (PVP) and carboxymethyl cellulose (CMC) are known to be eco-friendly polymers and are widely used as stabilizers in cosmetics, pharmaceuticals etc. This paper discusses the utility of polyvinyl alcohol (PVA) as polymeric binders in soft clays. The effects of added polymers on unconfined compressive strength, UCS, are reported [14-16]. This reported research involved a number of experiments using cement, sodium silicate and two types of PVA to stabilize kaolinite as soft clay, in order to provide a better understanding of the strength behavior of kaolinite after stabilization with various dosages of PVA.

2. MATERIALS

2.1. Soil samples and characteristic

Soil samples were collected from Serdang, Malaysia and sealed to maintain soil moisture in accordance with the British Standard Institution (BSI) methods [17].

Table 1. Physico-chemical properties of soil samples

Parameter	Kaolinite	Standard
Color	Yellow	
Moisture content,%	24	BS 1377-2 1990
Soil pH	5.2	BS 1377-3:1990
Specific surface area, m²/g	15	BET technique
CEC, meq/100 g soil	10	After Chapman, 1965
Organic content %	2	BS 1377-3: 1990
Zeta potential, mV	-28.4	ASTM D 4187
UCS (kPa)	35	ASTM D 4187

Table 1 provided the properties of the soil sample. Also, grain size distribution of the kaolinite soil presented in Figure 1. The shear strength of the undisturbed kaolinite soil and the stabilized kaolinite with cement and PVA were studied by UCS tests.

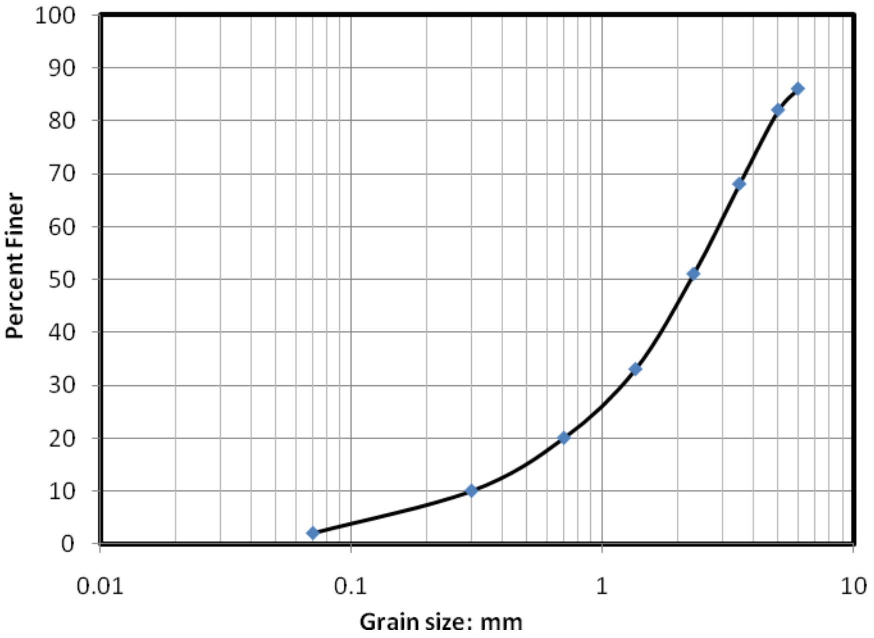


Figure 1. Grain size distribution curve of kaolinite

2.2. Polyvinyl alcohol properties

PVA, a water-soluble biodegradable polymer, available with different degrees of hydrolysis has immense potential as membrane material due to its film forming characteristics and hydrophilicity [10, 18]. It has excellent film forming, emulsifying, and adhesive properties. It is also resistant to grease, oil, and solvent.

It is nontoxic and odorless. It has high flexibility and tensile strength, as well as high oxygen and aroma barrier properties [19-22]. However these characteristics are dependent on humidity, in other words, with higher humidity more water is absorbed. Although, the absorbed water, which acts as a plasticizer, will then reduce its tensile strength value, increases its tear strength and elongation. PVA is also a fully degradable and quick dissolver. PVA, with molecular formula of $(C_2H_4O)_x$, in its pure state, is a solid which likes to absorb water from the air [10, 15, 23-25]. It is one of the few polymers that are freely dissolvable in water, and when the water of a PVA solution dries, a solid thin clear film is left behind; unless some other additives are present, such as glycerin, which can keep the PVA film from turning hard [26-27]. Since in kaolinite, organic matters as the main content prevent suitable hydration of binders using such water adsorbent polymer is worth being advised. Two different PVA species were investigated, fully hydrolyzed (PVA-F).and partially hydrolyzed (PVA-P)

3. METHODS

3.1. Zeta Potential Measurements

To determine the zeta potential of kaolinite, electrophoretic mobility measurements were conducted using a Zeta Meter Model 3.0 (Zeta Meter Inc., USA). The instrument, using Smoluchowski's equation, determines the electrophoretic mobility of the particles automatically and converts it to the zeta potential.

The zeta potential was measured as a function of pH, and adsorbed amount of PVA on the surface of colloidal particles. After adsorption period, we measured the zeta potential of particles which were obtained by centrifuging the suspension. For the zeta potential measurements, a 50mg sample was transferred into aqueous solution and the soil particles mixed homogenously with a magnetic stirrer. All the measurements were carried out therefore at 100 mg/L solid concentration. The voltage to be applied is determined based on the specific conductivity of the solution. The particle movement is observed through a microscope and the time taken for a particle to travel a particular micrometer distance is measured. To minimize reading error, a minimum of 10 particles are tracked and their average time is calculated. The pH measurements were carried out using a Mettler–Toledo Model MP220 pH meter combined with a Mettler–Toledo Model InLab 413 pH electrode. Boiled and double-distilled water was used to prepare aqueous solutions for all experiments to measuring zeta potential.

3.2. Unconfined Compressive Strength Tests

UCS tests were carried out on the undisturbed kaolinite soil samples, and on kaolinite soil samples stabilized with cement and PVA, in accordance with ASTM D2166-6. The samples were 76 mm long and 38 mm in diameter. UCS was conducted at different curing times: 14 and 28 days. Samples were removed from the water tank, and tested for unconfined compression.

3.3. Test procedure and sampling

In order to reduce the differences in soil sample properties during sample preparation, soil was blended until the mass was relatively homogeneous. The stabilising agent and kaolinite soil were mixed to a homogeneous mass by means of a household mixer and then compacted and stored in plastic tubes of inside diameter 38 mm. Each layer was given 10 constant full thumb pressures of about 10s to compact the kaolinite samples mixed with cement and PVA (Axelsson et al., 2002). The unit weigth were almost considered constant about 11.5 to 12.5 kN/m^3 for whole specimens.

According to Eurosoilstab (2002), during curing time period kaolinite samples were subjected to a vertical load of 18kPa. The test procedure is described as following (See also Figure 2): (a). A textile was taped over the bottom of the plastic tube to permit the soil specimen to take up water during curing time; (b). The kaolinite soil was blended with binders to form a homogeneous mass and then compacted into the plastic tubes by hand. Also, the specimens were subjected to a dispersed pressure of 18 kPa. As for such pressure a total load of 2kg was required for each sample (Fig. 2). The specimens were then placed in a unique design sample box which consist of a plastic tray positioned in order to store specimens vertically. Since the initial moisture content of kaolinite was 23%, it was blended in this rate with different binders. The tray was then filled with water up to 5 cm to simulate the insitu condition; (c) After 14 and 28 days at room temperature the specimens were extracted from the plastic tubes and UCS tests performed (Fig. 2). The mixing procedure and curing system used in this study project was that adopted in the EuroSoilStab project (Design Guide: Soft Soil Stabilisation; EuroSoilStab, 2002). The essential requirements were in accordance with BS-1377-7-5-2 1990 methods of test for soils. Disturbed specimens were also prepared and compacted in accordance with BSI-1377-7-5-4 1990.

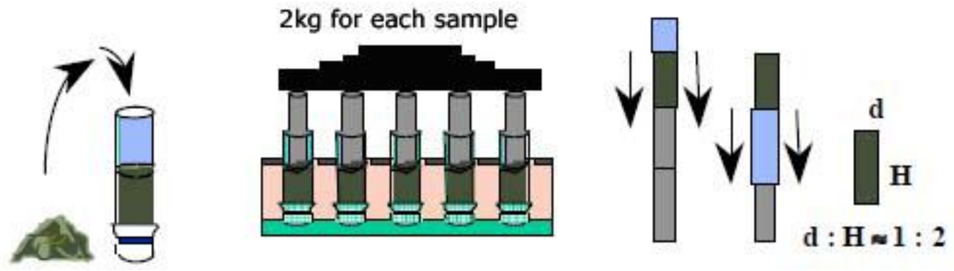


Figure 2. Sample preparation, (1) Mixing kaolinite with binders, (2) Curing soil samples “satuarating under 18 kPa loading”, (3) Extracting samples from the shelby.

3.4. Design of binders and Additives dosage rates

The study comprised an investigation of the increase in UCS over time achieved using different PVA dosage and types through the kaolinite.

Table 2. Composition of the mixtures

Binder no.	PVA-F[a] (gr/L.)	PVA-P[b] (gr/L.)	Sodium Silicate (mol/L.)	Cement (%)
1	1, 3, 5	---	1	10
2	1, 3, 5	---	5	10
3	1, 3, 5	---	1	30
4	1, 3, 5	---	5	30
5	---	1, 3, 5	1	10
6	---	1, 3, 5	5	10
7	---	1, 3, 5	1	30
8	---	1, 3, 5	5	30
a: Fully hydrolyzed polyvinyl alcohol				
b: Partially hydrolyzed polyvinyl alcohol				

As stated before, two different PVA species were investigated, partially hydrolyzed (PVA-P) and fully hydrolyzed (PVA-F) in conjunction with sodium silicate. Composition of the mixtures showed in Table 2. In addition, ordinary Portland cement (henceforth referred to cement) was used as the binding material, and PVA was used as the additive for stabilising the kaolinite soil.

4. RESULTS AND DISCUSSION

4.1. Interactions in double layer of kaolinite affected by reagents

The zeta potential of pour kaolinite soils varied from +32.9 mV at pH 2.03 to -41.9 mV at pH 12.2. The zeta potential was almost zero at pH 3.2 to 3.4 (Fig. 3). The variations in zeta potential with pH were probably related to the nature of electrical energy field in kaolinite soils. The natural pH of used kaolinite soils were 5.2. The sign of the natural zeta potential in kaolinite soils was negative (Fig. 3). The source of negative charges on the broken edges of kaolinite, as well as other clays, is belived to arise from dissociation of a proton (H^+) from an exposed OH group. This is possible because oxygen atoms at the edges are in contact with one rather than two Si of Al atoms [7, 28-32]. The hydrogens of tetrahedral OHs (those associated with Si) are presumed to be more likely to dissiciate than those of octahedral OH (those associated with Al) [33-36].

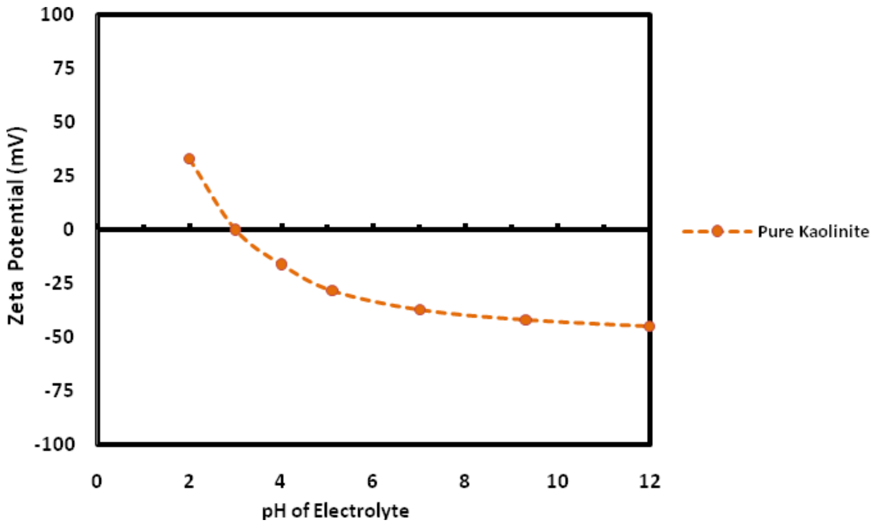
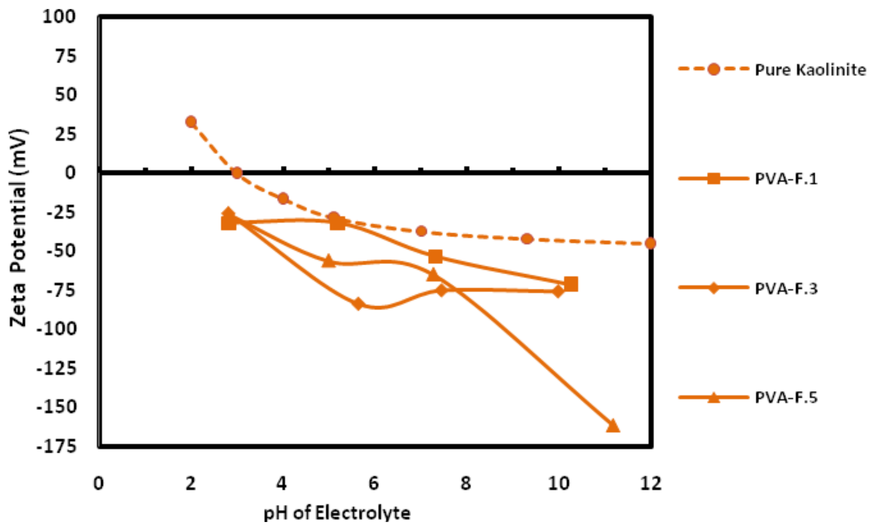


Figure 3. Zeta potential versus pH for kaolinite

4.2. Effect of polyvinyl alcohol on interactions in electric double layer of kaolinite

Figure 4 show the zeta potential of kaolinite soil particles as a function of pH for various concentrations of PVA-P as well as PVA-F. Observed results clearly show that PVA concentration has significant influence in the increasing zeta potential value in acidic part and decreasing in alkaline part. Immersing kaolinite particles in both PVA types caused increasing in EDL (Fig. 4.a). On the other hand, zeta potential for kaolinite soil suspended in 1, 3, and 5 gr/L of PVA-P at pH~7 were equal to -60.3, -72.1, and -79.35mV. It means the more concentration of PVA led to longer EDL length. The rest of specimens also showed a similar trend (Fig. 4).



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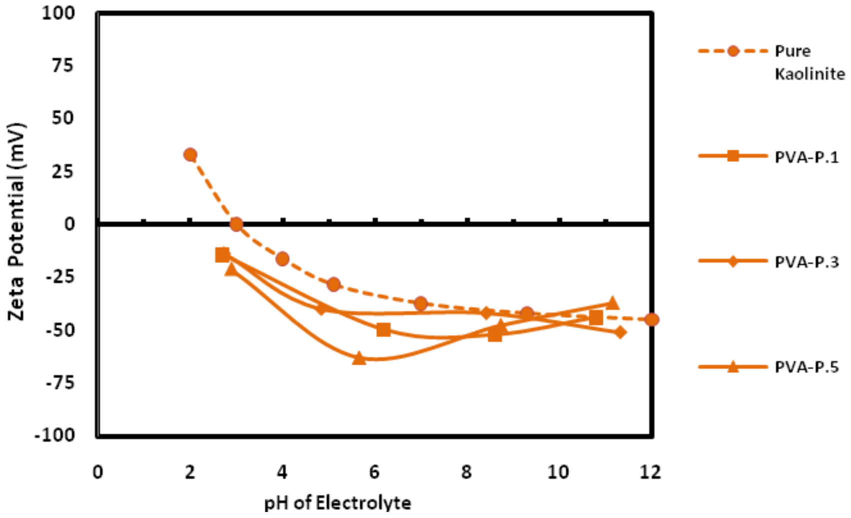


(b)

Figure 4. Effect of PVA concentration on zeta potential, (a) PVA-F, (b) PVA-P

4.3. Effect of polyvinyl alcohol on isoelectric point and electroosmosis in kaolinite

The EO flow can virtually be eliminated at the IEP point. Therefore, elimination of EO flow in the kaolinite suspended in water can be expected at pH 3 to 3.2, however adding PVA caused a shift in pH at IEP towards to the acidic part at pH~1.9 to 2.1 (Fig. 4. a, and b). On the other hand, negative surface charge of particles (negative zeta potential) causes EO to occur from anode to cathode, while positive surface charge causes EO to occur from cathode to anode [7-8, 25, 32, 35]. When the net charge is zero, soil particles in soil water suspension will not repel each other but will tend to aggregate and form larger particles [7, 36-39]. This effect in turn will contribute to an increase in soil permeability through the soils. In contrast, negatively charged soil particles repel each other, resulting in dispersion and decrease in soil permeability [28, 31, 39-40]. Figure 5 shows the values of IEP of some minerals in comparison with kaolinite soils immersed in PVA from this study. It is important to note that all charge in humus is strongly pH-dependent, the sensitivity of the highly organic soils such as peat to pH changes in EK phenomena is more than mineral soils like kaolinite [39-41]. Despite this high sensitivity, the IEP point of kaolinite soils immersed in PVA as depicted in Figure 5 was less than amorphous iron, gibbsite, as well as pure kaolinite. Thus, the consistency of flow direction in highly organic matter soil immersed in PVA may be more probable than that in mineral soils (Fig. 5).

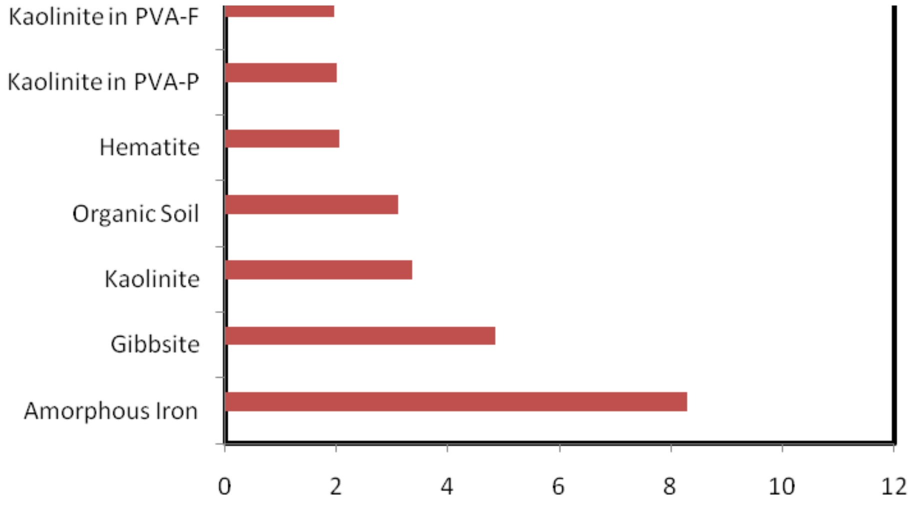
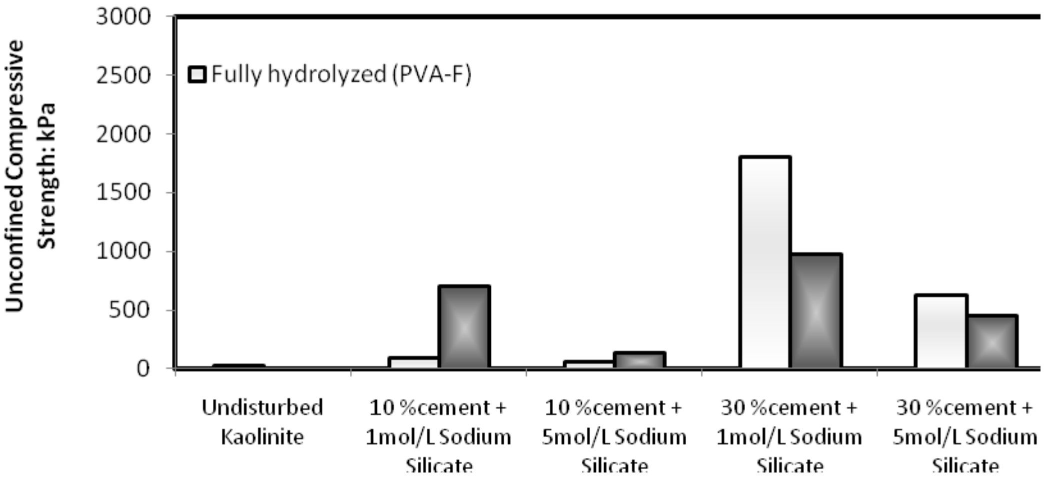


Figure 5.pH at iso-electric point of variable charge soil by comparison with the some minerals

A difference in the IEP between different samples having the same chemical formula has been often attributed to the differences in their crystallographic structure and degree of hydration [42-44]. In general, multivalent ions, polymers, and surfactants tend to adsorb specifically and shift the IEP. The magnitude of the shift depends on the solid to liquid ratio. The shift in the IEP is commonly negligible when the amount of the solute in the system is small compared with the proton charge [44-46]. This explains the discussed above difference in the IEP obtained by means of electrophoresis on the one hand and electroacoustics on the other, namely, in electrophoresis (small solid to liquid ratio) traces of the impurities in solution, e.g. silicates leached out of the glassware, may induce a substantial surface coverage and thus a shift in the IEP [24, 39, 41]. In other words, the pristine IEP obtained in the absence of specific adsorption are also valid for sufficiently low concentration (compared with the proton charge) of specifically adsorbing ions. When the concentration of the solute is high enough, its specific adsorption may (but not necessarily does) induce a shift in the IEP. Also, specific adsorption of anions induces a shift in the IEP to low pH for materials having a high pristine IEP (e.g. iron and aluminum oxides and hydroxides) [7, 24]. Specific adsorption of cations changes the EK curves of materials having a low pristine IEP [46].

4.4. Optimum percentage of polyvinyl alcohol

UCS test Results are presented in Figures 6 to 8. As showed, various concentration of PVA-F dosage had a remarkable influence over increasing the UCS values. However, the effect of low PVA-P dosage was almost negligible in changing the UCS. Within 30% cement content, having 1gr/L dose of PVA provides more UCS results than with 3 or 5gr/L of PVA dosage (Fig. 6 to Fig. 8). Moreover, UCS results of 28 days showed higher values than those in 14 days curing time. Within 28 days curing, 3gr/L PVA concentration provided highest UCS values among the various concentrations. The UCS of treated kaolinite with PVA-F dosage was also higher that with PVA-P. Using PVA-F dosage within 14days curing, the UCS results for 30% cement and 1mol/L sodium silicate concentration were 1808.14, 1486.7, and 1305.88 kPa at PVA concentration of 1, 3, and 5gr/L, respectively. However, having PVA-P, the UCS results for same condition were 984.44, 602.72, and 753.4 kPa at PVA concentration of 1, 3, and 5gr/L, respectively. This is due to hydration process of cement content which could make a better bonding along with time. Besides, this could probably because of available silicate minerals in clay portion of the kaolinite. The silica surface charge from kaolinite minerals is negative practically in the whole range of studied pH and its absolute value increases when solution pH is more alkaline. It means that pH increase induces stronger electrostatic repulsion between the negatively charged surface and dissociated acetate groups in PVA macromolecules [2, 5, 7, 35]. Despite this fact, the adsorption of PVA on the silica surface takes place, which is testified by obtained changes of the solid surface density in the presence of polymer. Therefore, different non-electrostatic forces have to be responsible for PVA adsorption process on the SiO_2 surface. The more PVA adsorption leads to more hydration cement especially in longer period.



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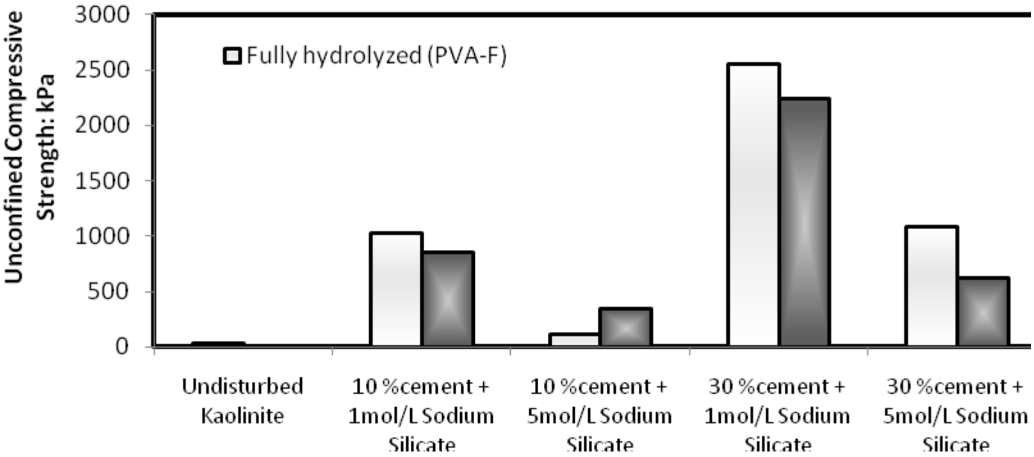
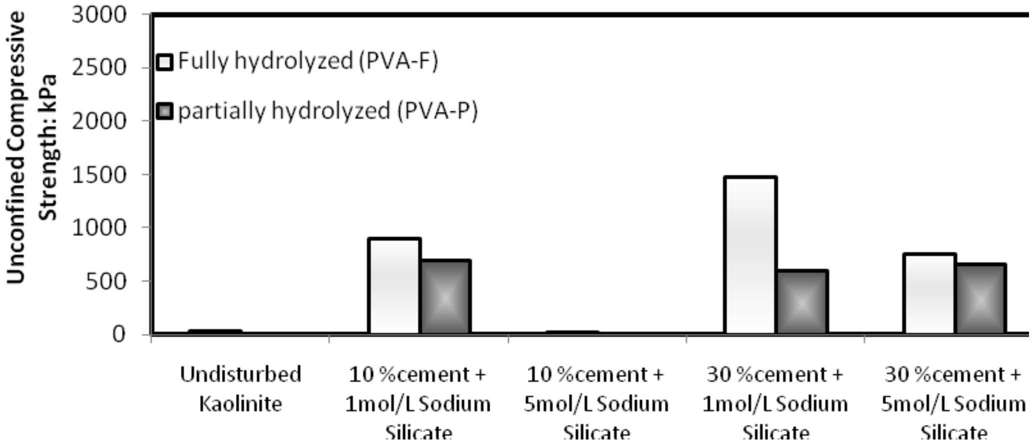


(b)

Figure 6. UCS values for different percentages of cement and Sodium Silicate for 1gr/L PVA samples;(a) after 14 days curing, (b) after 28 days curing.



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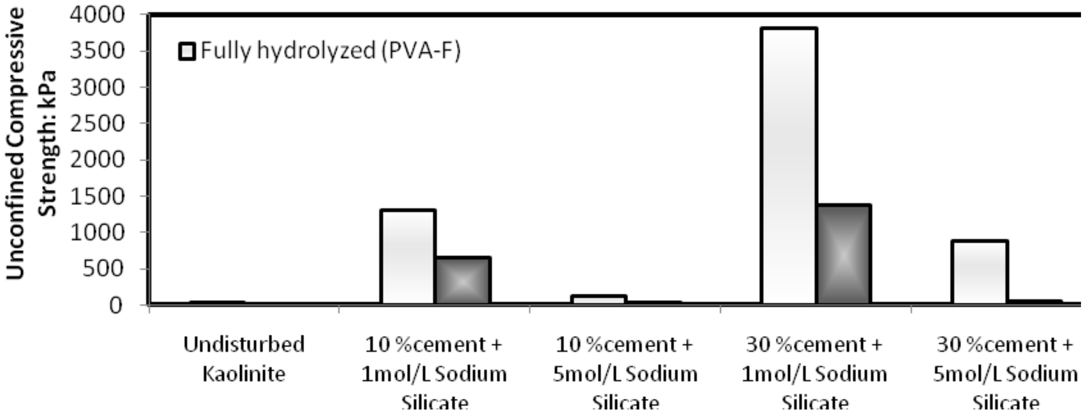
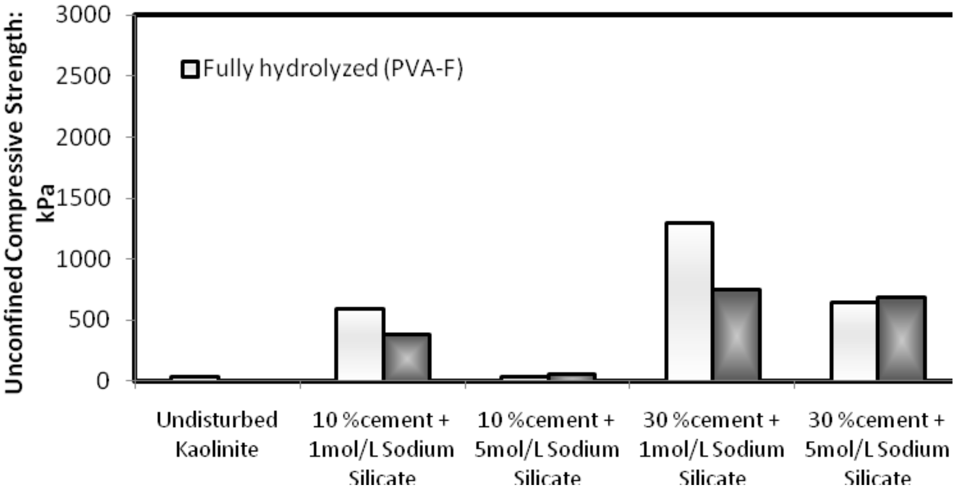


(b)

Figure 7. UCS values for different percentages of cement and Sodium Silicate for 3gr/L PVA samples;(a) after 14 days curing, (b) after 28 days curing.



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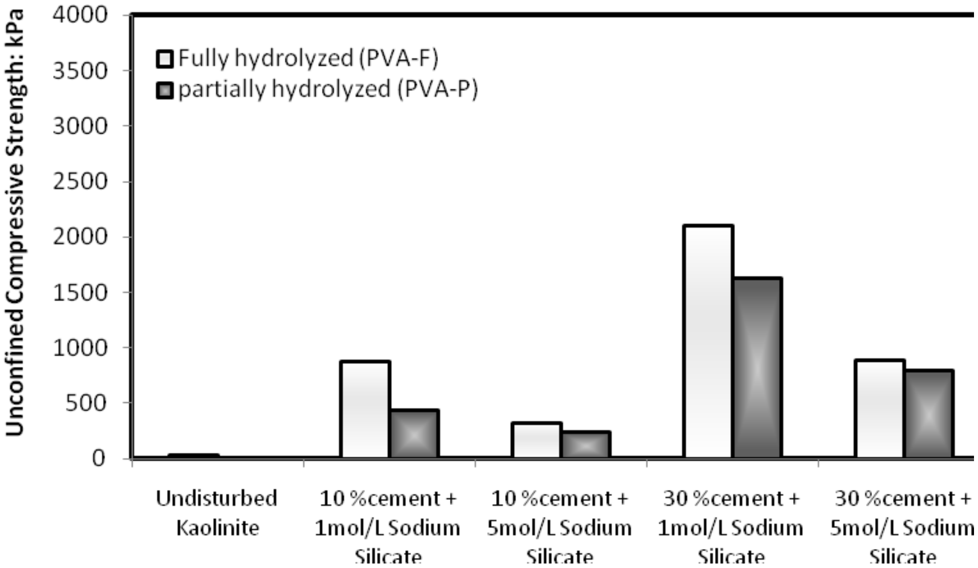


(b)

Figure 8. UCS values for different percentages of cement and Sodium Silicate for 5gr/L PVA samples;(a) after 14 days curing, (b) after 28 days curing.

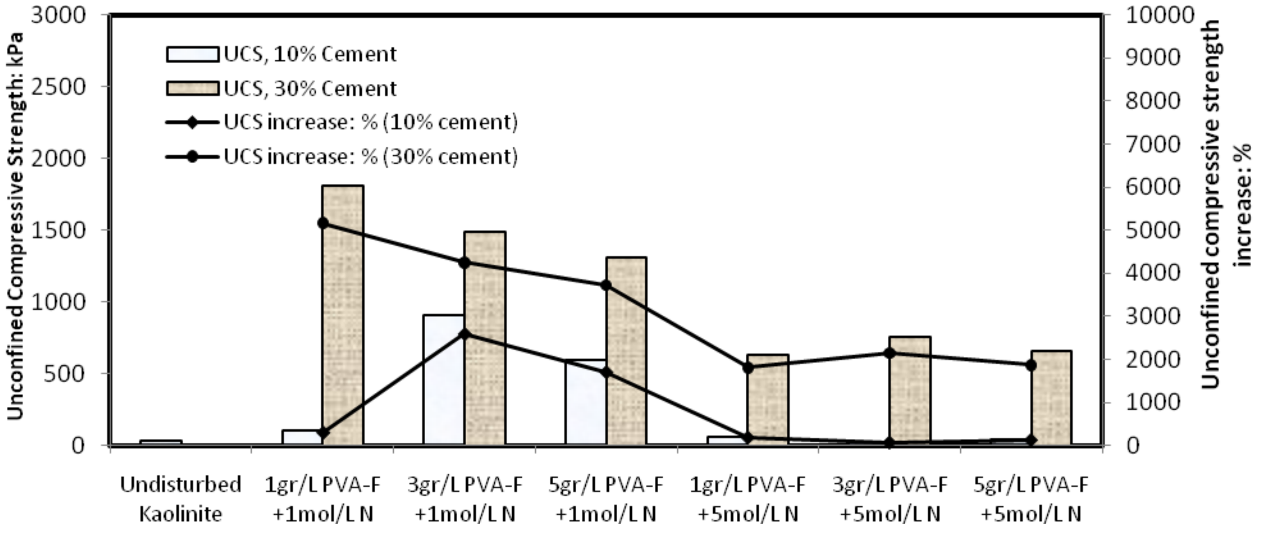
4.5. Effect of sodium silicate on UCS results

Sodium silicate is a white powder or colorless solution that is readily soluble in water, producing an alkaline solution. Silicate which is from sodium silicate enhances electrostatic repulsion between colloids [7]. Also, due to its high pH it makes suitable environment for kaolinite to stabilize under cement and PVA dosages. Moreover, sodium silicate can increase the UCS of the kaolinite soils due to injecting silicate minerals. The more sodium silicate used, the lower UCS results observed for kaolinite. This is probably due to using various concentration of PVA. For instance, the UCS values of kaolinite having 30% cement and 3gr/L of PVA-F dosage, with 1 and 5mol/L sodium silicate were

1486.7 and 753.4kPa, respectively (Fig. 7.a). Furthermore, among the UCS results of treated kaolinite, mixture of 30% cement, 1gr/L PVA-F with 1mol/L led to the highest UCS values (Fig. 9.a). However, having 3gr/L of PVA-F as in 28 days curing showed the highest improvement with 109 times increasing the UCS values comparing with the baseline kaolinite soil strength.

4.6. Effect of curing time

As showed in Figure 6 to Figure 8, the higher UCS results for kaolinite observed when longer curing time considered (Fig. 6 to Fig. 8).



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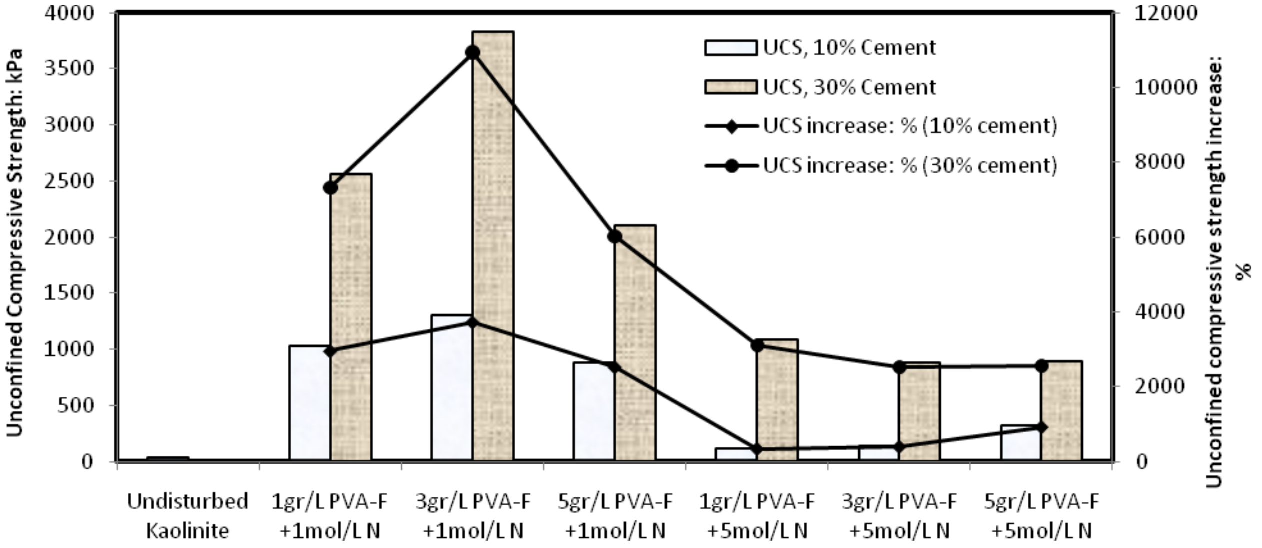
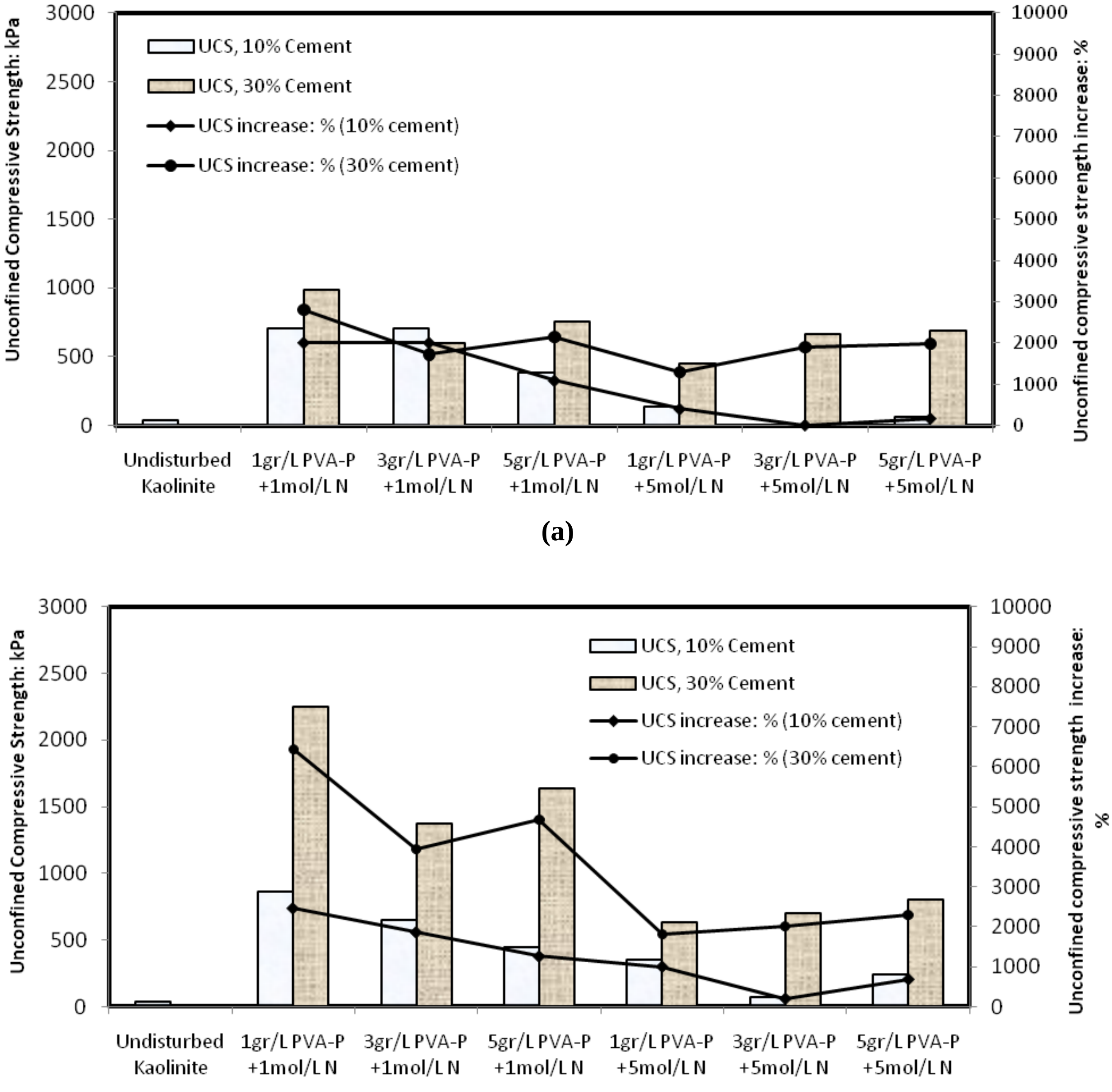


(b)

Figure 9. Percentage increase in UCS values for different percentages of cement and PVA-F;(a) after 14 days curing, (b) after 28 days curing.(The term N is used for sodium silicate)



(a)

(b)

Figure 10. Percentage increase in UCS values for different percentages of cement and PVA-P; (a) after 14 days curing, (b) after 28 days curing.(The term N is used for sodium silicate)

This is due to better hydration process which carried out from cement and organic soil composition in presence of PVA. It was apparent that for 14 days treatment curing, the addition of 30% cement and 1gr/L PVA-F gave the maximum values for the stabilized kaolinite soil samples among all those in the study (Fig. 9 and Fig. 10).

However, for 28 days, using 3gr/L concentration of PVA-F caused USC results as high as 3827.25 kPa. UCS results for 14 and 28 days in similar dosage of 1mol/L sodium silicate for 30% cement was 1808.15 and 2561.54kPa, respectively. Also, binders mixed with PVA-F dosage showed an increasing trend on their UCS with curing time. The longer curing time caused higher differences between UCS result of treated and untreated stabilized kaolinite (Fig. 9 and Fig. 10).

5. CONCLUSIONS

In the present research, kaolinite soil was treated with cement as main binding agent and PVA and sodium silicate as additives. Based on the results of this study, the following conclusions may be drawn:

- From the results of the UCS test and percentage increase, UCS values of the samples increases with increase in cement content. For example, in the presence of dosages 3gr/L of PVA-F as well as 5mol/L sodium silicate, it increases from 35 kPa for the untreated soil to 1305.89 and 592.67 kPa with 10% and 30% cement, respectively. Comparing with the untreated results, UCS increased by a factor as high as 5 and 109 as compared with untreated kaolinite soil with only UCS equal to 35kPa.
- As for 14 days treatment curing, the optimum dose of PVA-F was 1gr/L for binders with 30% cement content and 3gr/L for those binders with 10% cement content. However, within 28 days curing, the optimum dose of PVA-F was 3gr/L for both binders with 10% and 30% cement content. For PVA-P, 1gr/L concentration with both 10% as well as 30%cement content can be mentioned as the optimized UCS value (Fig. 9). This can be attributed to the increasing molecular attraction between the kaolinite particles and PVA-F with increasing degree of saponification due to the formation of hydrogen bonds. PVA-P shows inferior compressive and flexural strength to PVA-F due to the presence of ester group which interrupts the formation of hydrogen bond. Also, this could probably due to specific behavior of tropical area soil (i.e. variable charge soils) to adsorb ions (Fig. 10).
- Further, the use of PVA reduces the requirement for cement and sodium silicate. This implies part of the cement can be replaced with PVA, making it more economical.

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