

Clayey soil stabilization using geopolymer and Portland cement

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Abstract

This study compares the mechanical performance of clayey soil stabilization using volcanic ash (VA) based geopolymer and ordinary Portland cement (OPC). The effects of curing conditions and time, alkali activator/clay and alkali activator molarity, and VA/clay ratio are determined. The compressive strength of the untreated clayey soil specimens could be increased from 0.2 to 4 MPa and 2 to 12 MPa at the OC and DC conditions, respectively, when the soil partially replaced by 15 wt% of the binders. It is observed that geopolymer treatment is more efficient at the dry conditions (DC) while the Portland cement is superb at the wet environments (OC). This difference is associated with the role of water and pH in the kinetics of geopolymerization and the Portland cement hydration. Moreover, increasing the molarity of alkali activator and alkali activator/clay improve the compressive strength of the geopolymer treated soil. Besides, the higher energy absorption in all geopolymer specimens shows the superior ductility of this material in comparison with OPC.

Keywords: *Soil stabilization; Geopolymer; Cement; Volcanic ash; Clay; Natural pozzolan.*

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Introduction

Lack of consideration for building and infrastructure construction on weak or soft soils is highly risky due to their poor shear strength and high compressibility. These make them susceptible to differential settlements. Therefore, it is important to enhance the soil properties using stabilization techniques that can respond to increasingly demanding situations.

Currently, chemical stabilization of soft soils is a common method by which binders, such as ordinary Portland cement (OPC) and lime are incorporated into the soil to improve particle interfacial bonds [1]. In geotechnical engineering projects, OPC is the most favored material because of sufficient mechanical properties, availability and cost. Therefore, it is used in numerous stabilization techniques such as deep cement mixing and grouting [2, 3]. However, the overdependence on cement has given rise to several environmental concerns, including large CO₂ emission, natural resource depletion and dust generation. The OPC production is an extremely energy consuming process (5000 MJ/t PC) which causes a CO₂ emission of about 0.7–1.1 tonne per tonne of OPC [4-6]. Apart from the environmental drawbacks, OPC often shows a high plastic shrinkage and a reduction of mechanical strength due to the loss of water and incomplete hydration at early ages [7]. This is a big drawback for geotechnical applications, especially in torrid zones, as wet-curing of a big site is not applicable.

In order to reduce the environmental impacts and enhance the mechanical performance, OPC is partially replaced with pozzolanic materials such as fly ash (FA), ground granulated blast furnace slag (GGBS), palm oil fuel ash (POFA), volcanic ash (VA) and red gypsum [1, 8]. The partially replaced OPC examples exhibited enhanced mechanical properties, and durability in terms of moisture resistance, water sorptivity and shrinkage [8, 9]. However, the pozzolanic replacement is often limited to low quantities and the environmental impacts of OPC is still a concern.

Several attempts have been conducted to activate the pozzolanic wastes to produce a binder with a similar property as OPC gel. Na_2CO_3 was effectively used to activate GGBS to produce binder [10]. While, a similar result was not observed in another experiment [11]. This contradictory was attributed to the different pH environment of the experiments. The Na_2CO_3 hardly created a high pH environment in the clay-water-GGBS system while the rate of activating reactions depended on the pH of the starting solution [12]. Carbide slag, which is mainly composed of $\text{Ca}(\text{OH})_2$, was used to activate the GGBS to form a binder, however high water content used in that matrix had a significant impact on the early-age strength development [11].

Geopolymer has been emerging as a potential alternative to Portland cement by converting industrial aluminosilicate rich wastes into a value added binder [13, 14]. Apart from the environmental aspects geopolymer stabilized soils have been shown superior properties to meet the requirements engineered clayey soil through compact microstructures, improved mechanical properties and volume stability [15-18]. Different mixing designation have been investigated mechanical performance of geopolymer stabilized clayey soils. It was observed that shrinkage strain of metakaolin-geopolymer stabilized soil is much lower than those of the unstabilized or OPC incorporated ones. This low shrinkage was attributed to the slow evaporation of pore water from the compact structure of the stabilized soil using geopolymer [17]. Also, the compressive strength of lightweight GGBS-geopolymer stabilized clayey soil was improved by 200 to 350% compared to its corresponding lightweight OPC specimens [19]. Similarly, for the same percentage of binder content, the GGBS-geopolymer stabilized soil indicated 600% mechanical strength improvement compared to the OPC treated specimens over short-term curing time of 28 days [15]. However, FA-geopolymers often show a slower and long-lasting strength development compare with those of OPC at low curing temperatures. A comparison between FA-soil mixtures with and without

alkaline activator showed a remarkable increase in strength of alkali activated specimens from 0.3 to 2.8 MPa at 28 days and 5.2 MPa after 90 days [20]. Likewise, it was observed that with comparable 28th day mechanical properties, strength development of FA-geopolymer stabilized samples were in the range of 250-500 % after one year, while it was limited to 10-25 % in OPC specimens [21]. However, a higher early strength was observed when FA-geopolymer soil was cured at higher temperatures [22, 23]. The compressive strength of POFA-geopolymer soil which subjected to hot curing was increased by 112% in comparison with that of cured in the absence of the heating process [24].

In addition to the effects of curing condition and time, it was reported that increasing the binder to soil ratio improves the compressive strength of stabilized soils [22, 25]. Also, increasing the sodium silicate/sodium hydroxide ratio or higher alkali activator concentration increase the mechanical strength of the geopolymer soil treated specimens however they reduce the workability [21, 26]. In contrary, an increase in strength was reported by decreasing activator/ash ratio. When the Na₂O/fly ash ratio increased from 0.160 to 0.375, the mechanical strength decreased by 50% [21]. The variation in results are due to the type of mixture, curing time and condition of geopolymers. Therefore, further studies on the controlling variables are required.

This study compares the mechanical performance of clayey soil stabilized with VA-geopolymer and OPC and investigates the dominant factors of stabilization process, including: curing conditions, curing time and binder content. The incorporation of both VA and OPC replacement contents varied in the applicable range of 0 to 15 w.t.% of the soil. Furthermore, the geopolymer stabilization was optimized by considering the alkali concentration and binder to soil ratio. The experiment carried out at two curing conditions of oven dried and optimum water content to verify stabilizer function at different climates.

2. Materials and methods

2.1. Soil characterization

A locally low plastic available clayey soil was collected from Shiraz-Iran. It contained a trace of sand and fine gravel. Therefore, it was dried and sieved through No. 4 (opening of 4.75 mm) to remove the gravel fraction. The full particle size analysis of the soil used in this study can be observed in Fig. 1. The engineering properties of studied soil in terms of the Atterberg limits, grain fractions, and soil classification was according to the ASTM D 4318, the ASTM D 422 and the Unified Soil Classification System, respectively, as in Table 1. The grain size distribution was obtained by means of sieve analysis coupled with hydrometer testing as per ASTM D 2487.

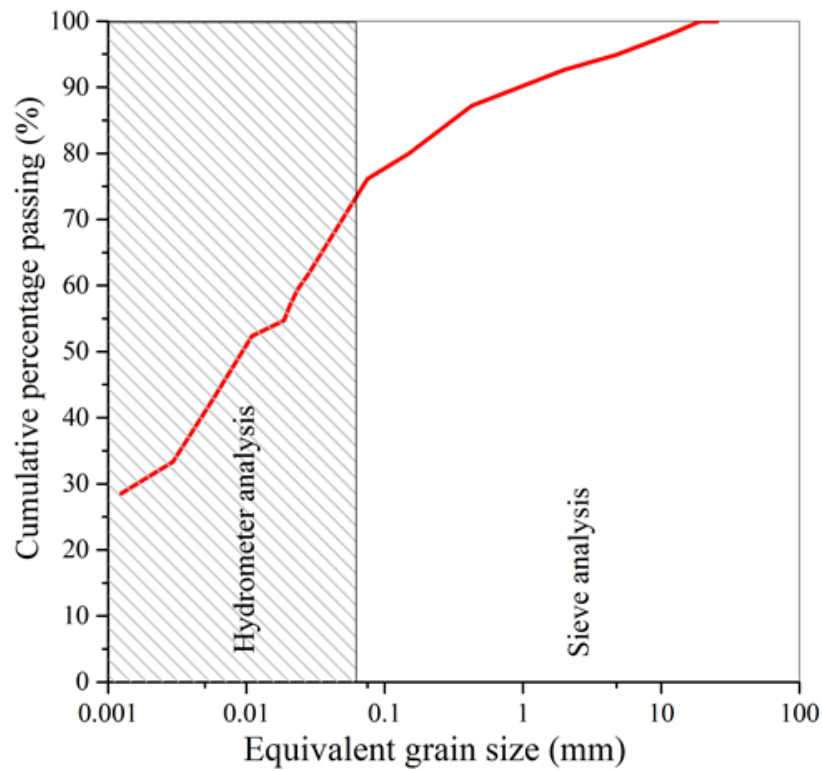


Fig 1. Particle size distribution of the Shiraz clayey soil.

Table 1. Engineering properties of clay soil.

Atterberg limits (%)	Grain fractions (%)	Soil classification
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LL	PL	PI	Gravel	Sand	Silt	Clay	
31	22	9	5	19	33	43	CL

To determine the maximum dry density (ρ_{\max}) and the optimum water content (OWC) of the soil the standard Proctor compaction test was conducted based on the ASTM D 698. The ρ_{\max} of 1.74 g/cm³ and OWC of 14% of untreated soil were determined for the stabilized specimens.

2.2. Binders characterization

The volcanic ash used in this research was collected from the Taftan Mountain, located in the south east of Iran. The as-received material was sieved to 74 μm to remove large particles and impurities. The ordinary Portland cement (OPC) type II was collected from Fars Cement Company. The X-ray fluorescence (XRF) using PANalytical Axios mAX instrument was used to determine the oxide composition of the VA and OPC, as listed in Table 2.

Table 2. Chemical composition of VA and OPC.

Oxide composition	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	Na ₂ O	MgO	TiO ₂	SrO	SO ₃	P ₂ O ₅	MnO
VA [wt.%]	46.8	19.1	13.5	8.5	4.3	4.1	1.7	0.9	0.3	0.3	0.2	0.2
OPC [wt.%]	11.78	69.62	3.23	8.425	1.45	0.36	2.36	0.295	0.121	1.92	0.049	0.168

Specific surface area of VA and OPC as measured by nitrogen adsorption according to the Brunauer, Emmett, and Teller (BET) method using NanoSORD92 instrument were 2.424 and 2.003 m²/g, respectively.

In order to evaluate the efficiency of geopolymer and cement on stabilization of clayey soil, three sets of predetermined concentrations of VA or cement were mixed with the activator for a constant time of 5 min. Sodium hydroxide solution and water were used as activators for the VA and OPC, respectively. Set 1 was determined to compare the different soil replacement by the VA or OPC in the range of 0, 5, 10, 15 w.t.%. The activator content was kept 14 w.t.% (as in the optimum water content of the untreated soil) for all the specimens. Sets 2 and 3 were performed only for

geopolymer treated soil. Set 2 was to investigate the effect of alkali activator molarity at 4, 8 and 12 M, while the VA replacement kept at 10 w.t.%. Set 3 focused on the influence of alkali activator/optimum water content which varied by 1, 1.2 and 1.4; and, the binder replacement and sodium hydroxide molarity kept constant at 15 w.t.% and 8 M.

3. Soil stabilization and characterization tests

3.1. Specimen preparation and mechanical characterization

The unconfined-uniaxial compressive test specimens were prepared using cylindrical molds with an inner diameter of 32 mm and a height/diameter ratio of 2.0. The soil was manually mixed with either geopolymer or cement solution for ten minutes to achieve a homogeneous mixture. The soil liquid fraction ratio was kept constant, 14%, to the optimum water content of the untreated sample. All samples were statically compacted in three equal layers with the aid of loading machine and extruded using a Harvard Miniature Compaction Apparatus once after the compaction. The specimens were cured at two conditions of optimum water content (OC) and dry (DC) and tested after 1st, 7th, and 28th day. The average of three samples tested for all the measurements. The specimens wrapped using plastic film and cured in plastic chamber with a nearly constant relative humidity of $80 \pm 2\%$ and temperature of $\sim 25 \pm 2$ °C using humidifier for OC condition curing. After the course of curing, the samples were unwrapped and UCS testing was performed. The DC curing was carried out by placing the specimens in oven of 40°C and relative humidity of $15 \pm 2\%$. Summary of the test plan is shown in table 3.

Table 3. Summary of the test schedule for the effects of geopolymer and OPC on soil stabilization.

	Binder type	Binder replacement [%]	Activator type	Alkali activator molarity [M]	curing time [day]	curing condition	Activator content/optimum water content
Set 1	VA	0,5,10,15	NaOH	8	1,7,28	OC*,DC [‡]	1 ^b
	OPC	0,5,10,15	H ₂ O	-	1,7,28	OC,DC	1
Set 2	VA	10	NaOH	4,8,12	1,28	OC,DC	1

Set 3	VA	15	NaOH	8	1,2,8	OC,DC	1,1.2,1.4
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* OC temperature=25°C, relative humidity 80%
 † DC temperature= 40°C, relative humidity 15%
 ‡ Optimum water content =14 w.t.%

UCS testing was performed with a universal testing machine (Digital Tritest 50/ELE). The axial strain rate was controlled at a rate of 0.5 mm/min. The maximum strength and the stress–strain curves were obtained by the average of three specimens. Energy absorption of the specimens was determined as the area under compression stress-strain of each specimen [27].

The actual moisture content of all specimens was measured immediately after the compressive testing by heating them at 110 °C for 24 h.

Optical images of treated and untreated samples were recorded by a Dino-Lite digital microscope (AM4113ZT(R4)) with magnification range of 10x~50x and 200x.

3.2. Microstructural characterization

Microstructural characterization was carried out 28 days cured specimens by Scanning Electron Microscopy (SEM), together with Energy Dispersive Spectroscopy (EDS) elemental analysis and X-ray diffraction (XRD). The SEM (TESCAN vega 3, Czech Republic) was operated in a high vacuum condition, at 20 kV accelerating voltage in the mode of backscattered electron (BSE). EDS at 20 KV fully embedded into the TESCAN system was used, to determine the elemental analysis of the specimens. The XRD patterns were measured on an DRON-8 (BOUREVESTNIK company, Russia) using Cu K α radiation ($\lambda = 1.54056 \text{ \AA}$), operated at 40 kV and 30 mA with a step size of 0.04 deg and a scanning rate of 0.15 deg s⁻¹ in the 2 θ range of 5 to 80 deg.

3.3. pH of soil

The pH values were measured by using a pH Meter and Probe, according to the ASTM D 4972. The samples were prepared by crushing and sieving of 10 g of soil specimens through a 2.0 mm (No. 10) sieve and mixing with 10 ml of water for 5 min. The samples were then stored in a curing room. The pH was recorded in one-hour duration.

3.4. Reactivity test

The reactive fraction of the VA was quantified by dissolving 1 g of ash in 100 mL NaOH solution for 24 hours at a temperature of 40 °C. The insoluble particles were separated from the solution using a centrifuge and washed with distilled water to a neutral pH and dried for 1 hour at 250°C and weighted [28].

4. Results and analysis

4.1. Compressive strength

Figure 2 and 3 compares the unconfined compressive strength and moisture content, respectively, for the geopolymer and OPC treated soil specimens which cured in different conditions of OC and DC at 1, 7 and 28 days. Independent of binder concentration, treatment of the soil specimens with either geopolymer or OPC increases the compressive strength of the specimen over time. However, the mechanical performance of the treatment methods is highly depends on humidity and temperature. The geopolymer treatment shows superb performance at the DC curing. This is attributed to the role of water and temperature on the geopolymerization. The structural water, what is essential and participates in geopolymerization, provides a medium for dissolution and hydrolysis of aluminosilicates and supplies ions transfer. The structural water is not evaporable at low temperatures [29, 30]. However, there is another water portion, the residual water, which is

unfavorable for the strength development as it reduces the alkalinity of the matrix through dilution effects, and slows the rate of geopolymerization at dissolution and hydrolysis stages [24, 31, 32]. The residual water is only used to provide sufficient workability [13]. As observed, high-water content retards the strength development of geopolymer treated specimens at OC, Figure 2. Unlike at the DC curing, the free water is evaporated gradually and alkalinity of the mixture increased. On the other hand, the reactivity of VA is slow at low temperatures because the reaction is based on the dissolution of the particles which increases at higher temperatures [33]. Therefore, heat curing accelerates the dissolution, hydrolysis and polycondensation of the geopolymers during the early age. Unlike, in OPC reaction water is essential for the formation of C-S-H gel and completion of the reaction. Therefore, maintaining the specimen moisture results in a gradual strength development at OC, Figure 2. However, at dry condition the lack of sufficient water for the hydration causes a cessation on the reaction and suppresses the soil treatment. Noteworthy, the rise which observed in the strength of the OPC based specimens at DC is associated with the increase in soil strength itself due to the volumetric shrinkage that occurs during the air drying process which produces a denser soil structure [34, 35]. The compressive strength development of the geopolymer treated specimens is less changed once the moisture content of the specimens balanced at a constant level of 1%, Figure 3. The moisture content of the specimens is almost independent of material type but curing conditions.

Independent of binder types, the increase in the binder/clay improves the compressive strength of the matrix at the OC but not at DC. This difference is attributed to the constant activator content, 14 w.t.%, and the rate of water evaporation. Increasing the rate of water evaporation accelerates the dissolution and hydrolysis of higher numbers of reactive particles while, slow down the further activation of partially reacted particles once the water is evaporated. However, activation of higher

number of particles is possible at OC as there is enough water for further binder gel formation and therefore, higher compressive strength is achieved. A similar trend is observed at the energy absorption of the materials at DC. However, a reduction in energy absorption of 28 days cured OPC specimens at OC is associated with the brittle structure of OPC specimens over time, Figure 4.

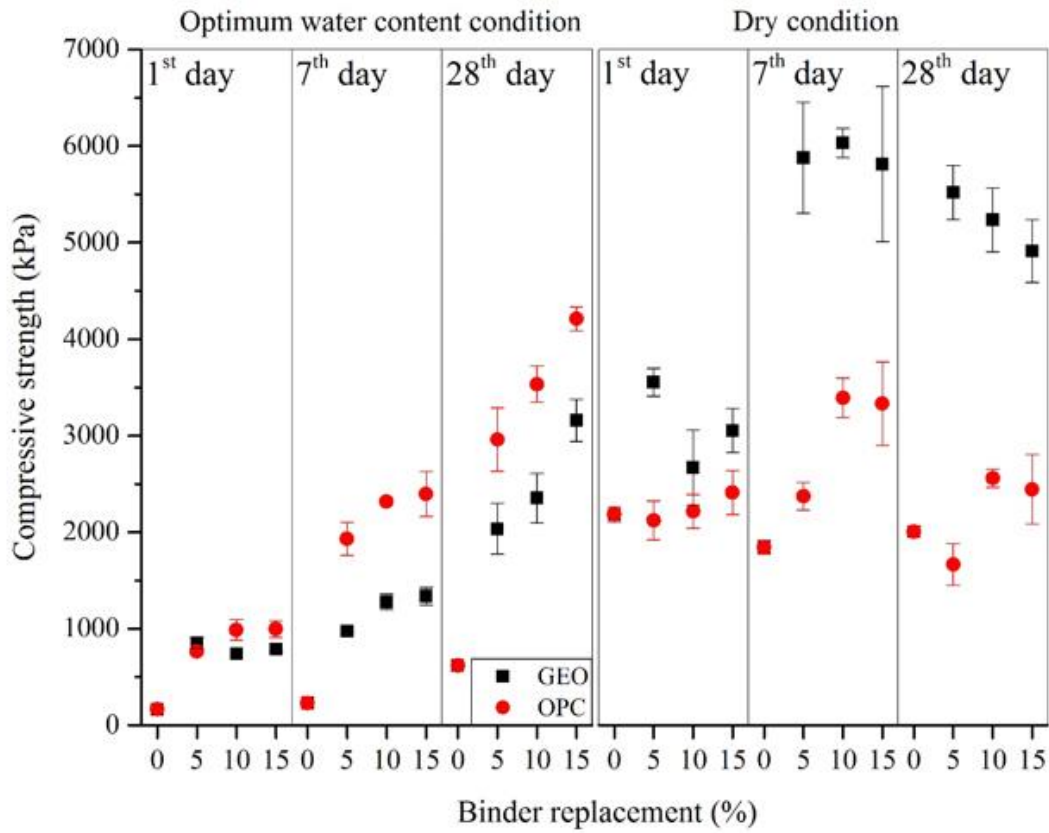


Fig 2. Compressive strength of stabilized soils using geopolymer and OPC at optimum water content and dry conditions with different binder content at different ages.

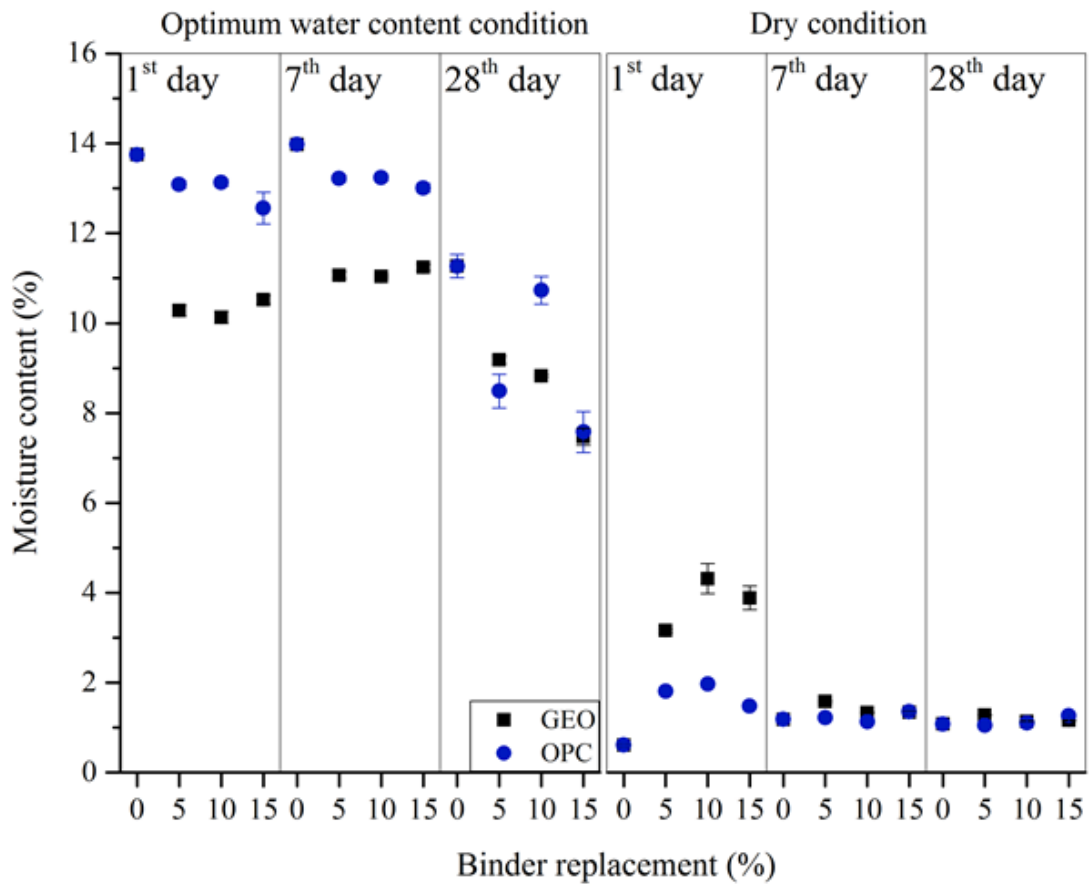


Fig 3. Moisture content of stabilized soils using geopolymer and OPC at optimum water content and dry conditions with different binder content at different ages.

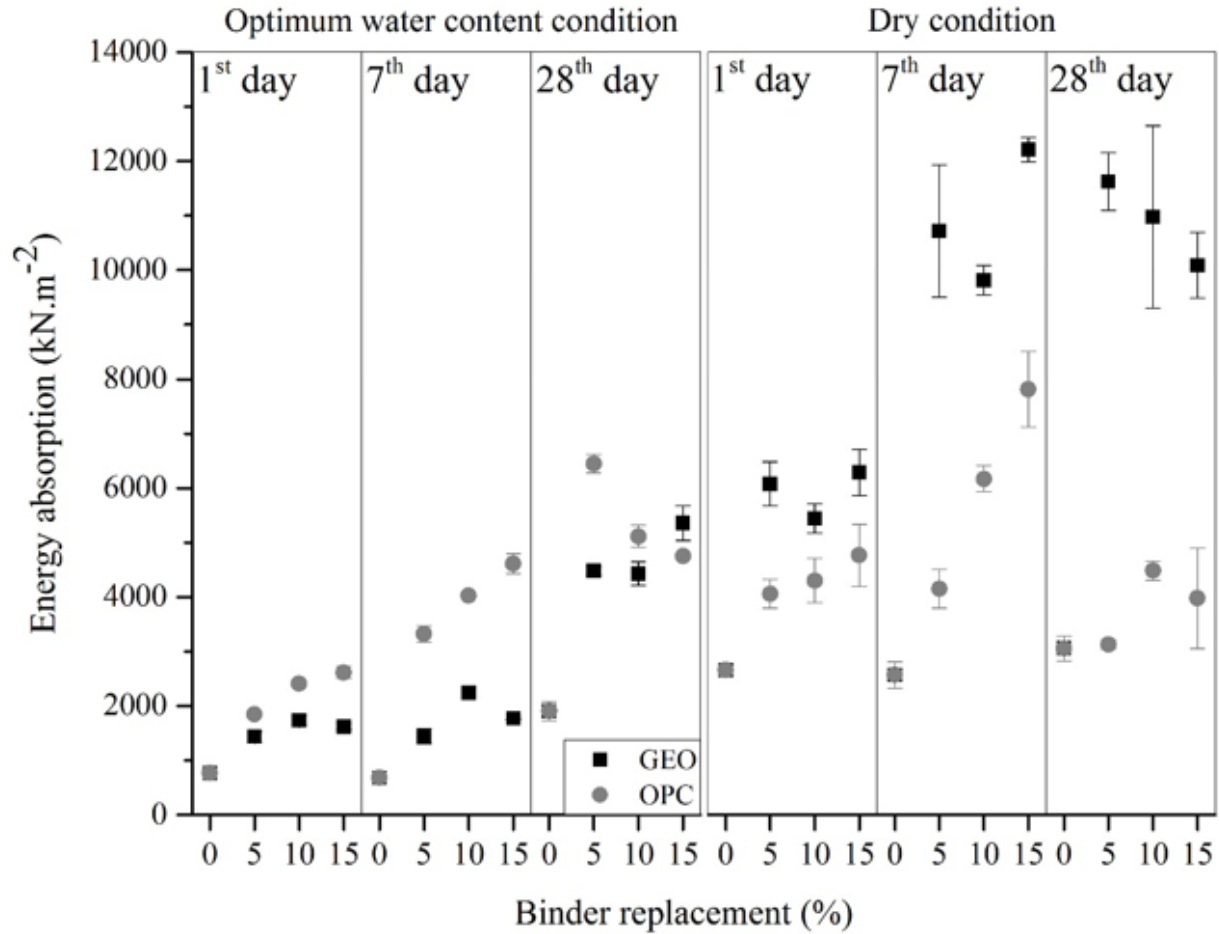


Fig 4. Energy absorption of stabilized soils using geopolymer and OPC at optimum water content and dry conditions with different binder content at different ages.

4.2. XRD analysis

The XRD patterns of untreated soil, stabilized soil using 15 w.t.% geopolymer and OPC at 28th day of curing are shown in Figure 5. The main crystalline phases of the as received clayey soil are calcium carbonate and quartz [36]. Considering the small fraction of soil replacement with the binders, the aforementioned crystalline phases are still dominant in the treated soils. A trace of anorthite is observed in the geopolymer treated specimens which attributed to the crystalline structure of the VA and VA based geopolymer [37].

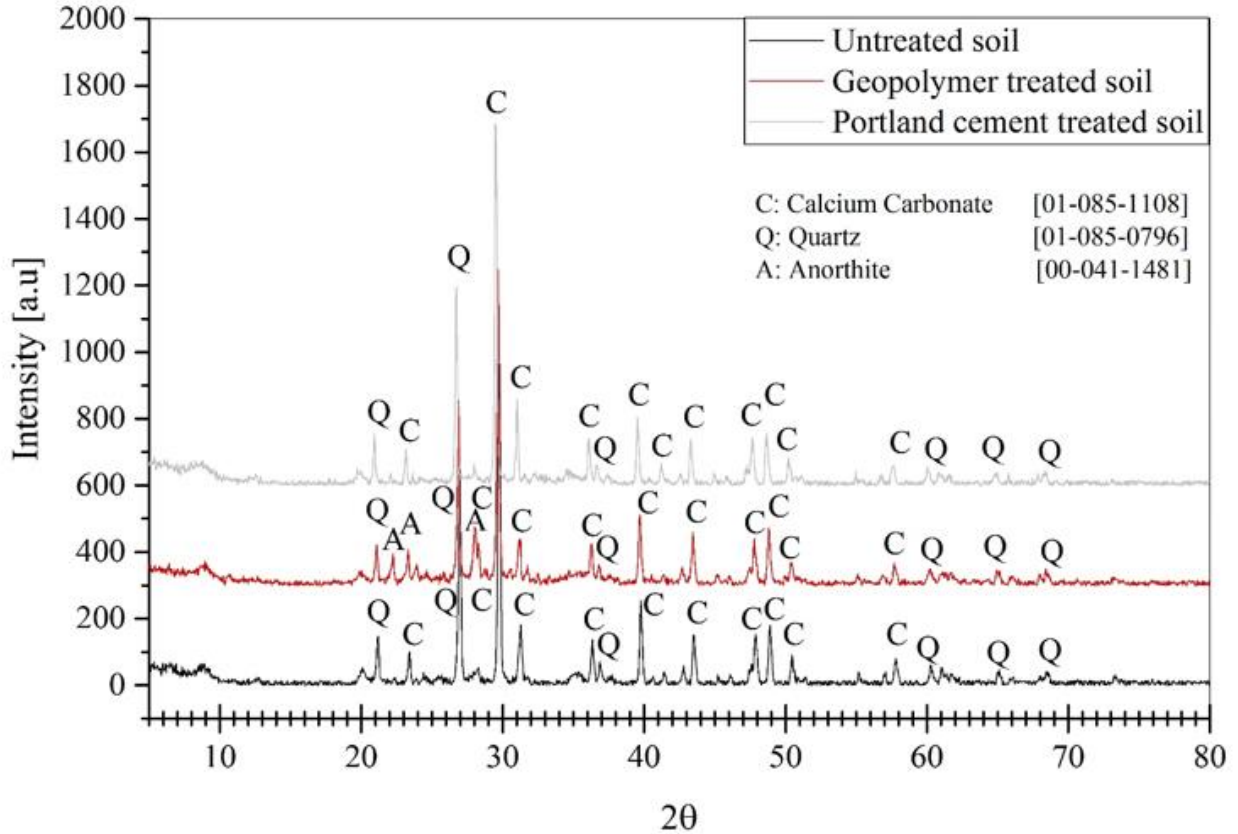


Fig 5. XRD patterns of clayey soil, geopolymer and Portland cement treated clayey soil specimens.

4.3. SEM-EDS characterization

Fig 6a-c and insets show optical images of untreated soil, stabilized soil using 15 w.t.% geopolymer and OPC at 28th day of curing. The insets, Figure 6d-f, shows the optical images of pure soil, VA-geopolymer and hydrated OPC. As observed in the Figure 6b and c both materials, OPC and geopolymer, has been distributed uniformly through the soil particles. This is obtained by proper mixing of a diluted form of the activator with a low viscosity. Furthermore, the BSE images show a higher degree of compaction in the treated samples, particularly at geopolymer treated specimens. This is due to the high alkalinity of the binder which disconnects the soil particles and makes the

rearrangement of the particles possible. Besides, the slippery binders lubricate the surface of particles to roll over each other during the compaction.

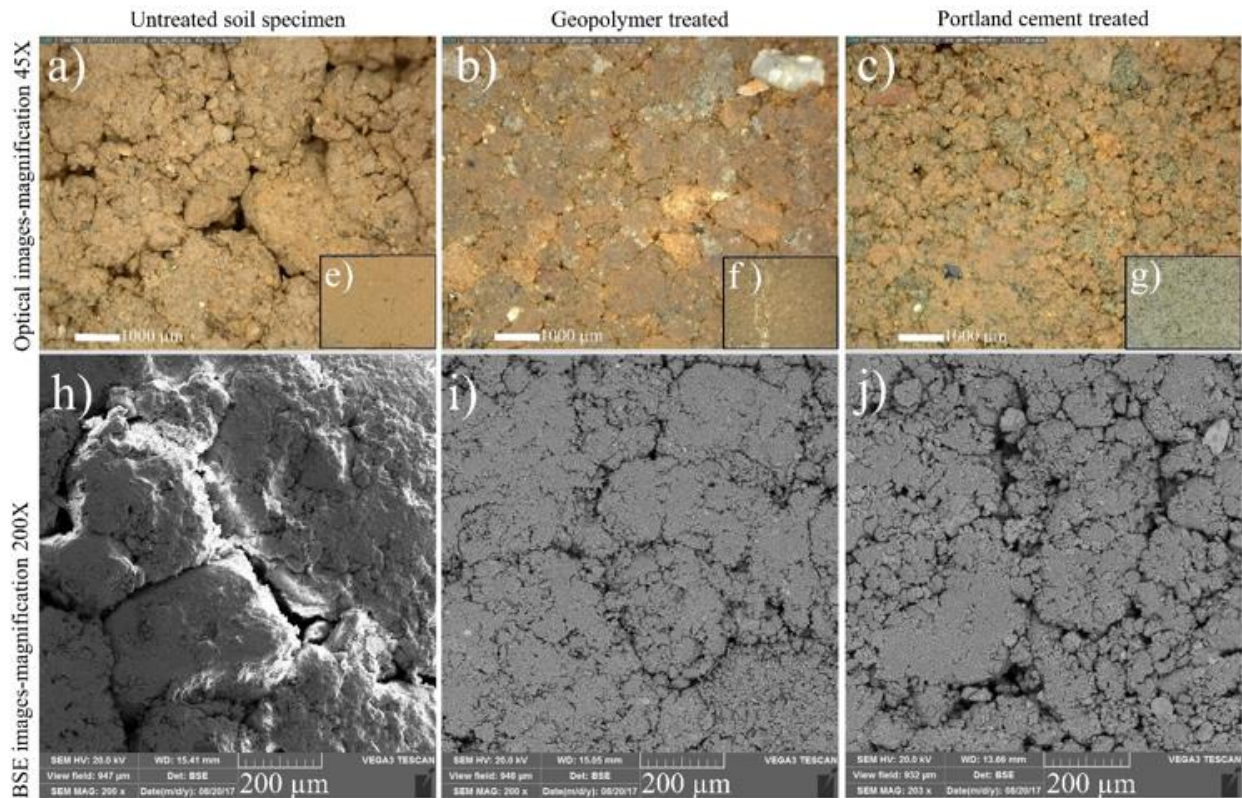


Figure 6. Optical and SEM images of a, e, h) untreated soil, b, f, i) stabilized soil using geopolymer (15% replacement at dry condition), c, g, j) stabilized soil using OPC (15% replacement at optimum water content condition) at 28th day of curing.

Figure 7a and b shows the influences of alkali activator concentration on the compressive strength of geopolymer treated specimens. The increase in molarity of the sodium hydroxide from 4 to 12 M resulted in higher dissolution of aluminosilicate precursors and, therefore, a higher mechanical strength of the geopolymer specimens at both OC and DC. This is attributed to a higher reactivity of volcanic ash particles at higher alkalinities, Figure 7c [38, 39]. Previously, it was shown that the

unreactive particles remain inert and should be considered in geopolymer mixing designation to overcome the overestimation of aluminosilicate performance [28, 40].

The moisture variation of the specimens is shown in Figure 8. It is observed that change in molarity of NaOH solution almost doesn't influence on the final moisture content of the specimens. Depending on the curing condition the moisture content balanced to about 8.5 and 0.5% at OC and DC, respectively.

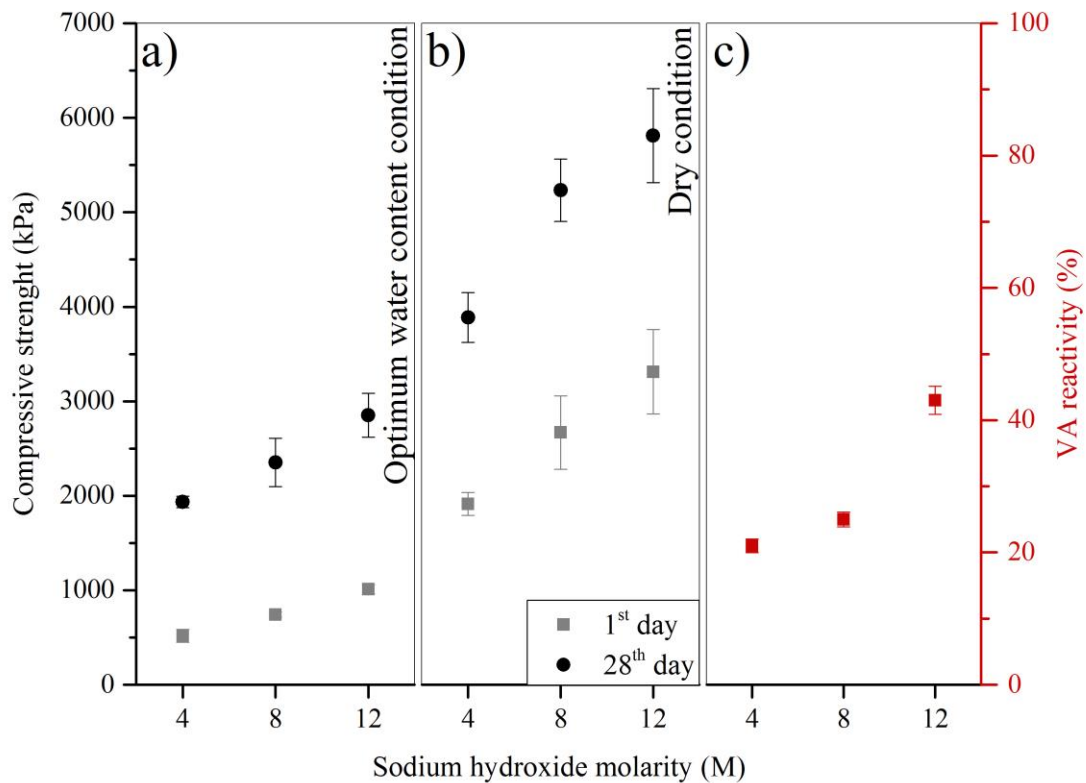


Fig 7. Compressive strength of stabilized soils using VA-geopolymer at a) OC and b) DC with different alkali activator molarity at different ages, c) VA reactivity at different alkali activator molarity.

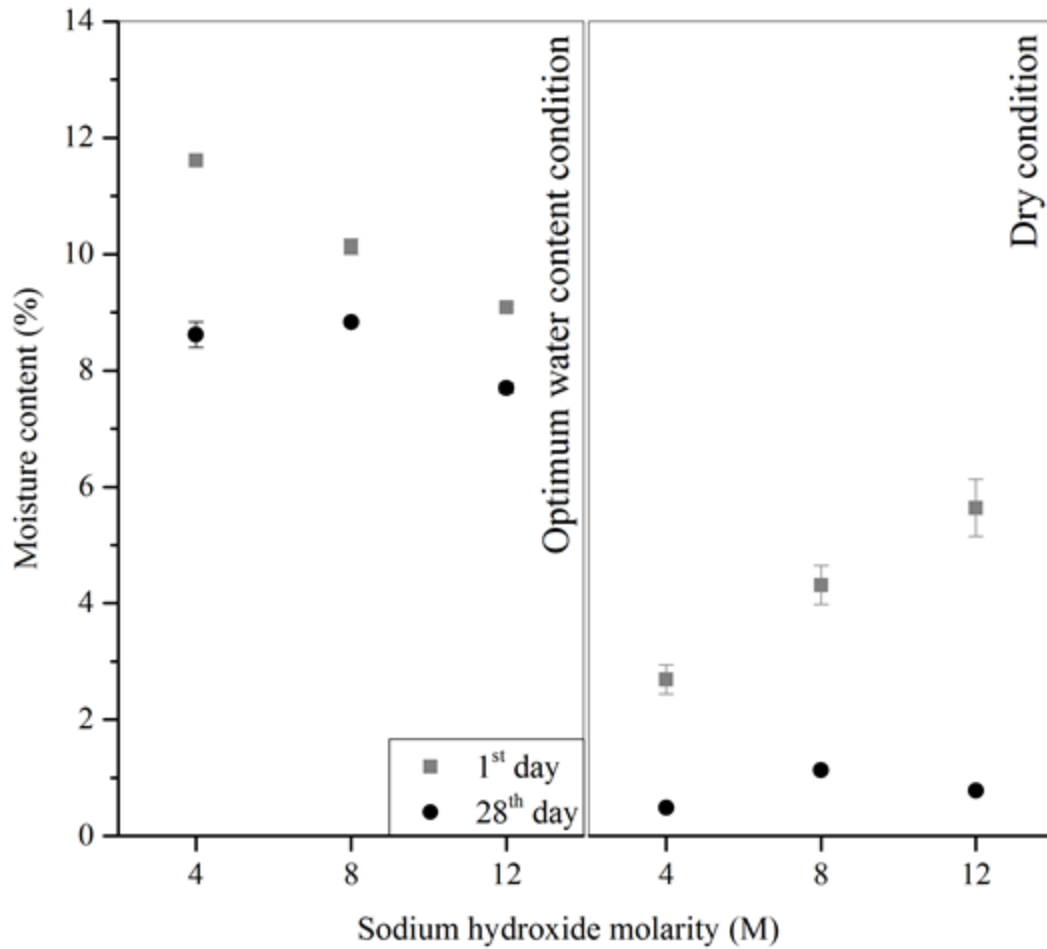


Fig 8. Moisture content of stabilized soils using geopolymer at OC and DC with different alkali activator molarity at different ages.

Figure 9 shows the compressive strength of the set 3 where the alkali activator to optimum water content ratio was changed in 1, 1.2 and 1.4. At the DC curing, the increase in alkali activator content from 1 to 1.4 resulted in 1.7 and 2.3 times rise in compressive strength of the treated soil at 1st and 28th day, respectively. This mechanical improvement is due to the activation of more particle through the higher alkali activator concentration in the matrix [41]. Despite of great efficiency at DC curing, the increase in alkali activator was ineffective at the OC, Figure 10. This is due to the dilution of alkali activator at the wet condition. As observed, the increase in alkali activator rises

the moisture content in early and long-term curing at OC, while it reduces to about 1% in DC curing at 28th day.

Figure 11 shows the energy absorption of the set 2 and set 3 where the alkali activator concentration and the alkali activator/optimum water content of the geopolymer treated soil specimens are investigated. Similar to the trends of compressive strength, increasing the alkali activator/clay had more influence on the energy absorption compared with the increase in molarity of the solution. However, higher energy absorption in all the geopolymer specimens shows the more ductility of this material compare with OPC. A similar conclusion was derived by comparison of flexural performance of reinforced geopolymer and OPC concrete beams [42].

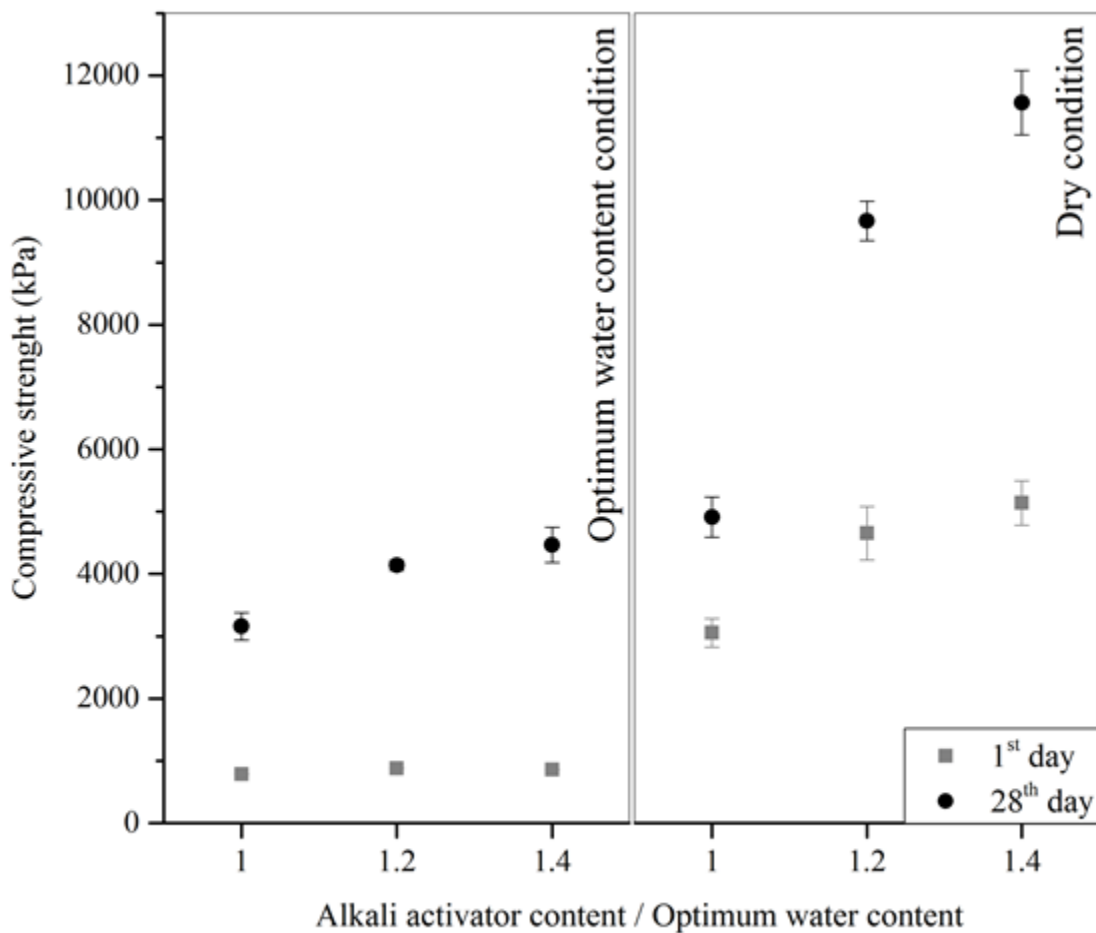


Fig 9. Compressive strength of stabilized soils using geopolymers at optimum water content and dry conditions with different alkali activator content at different ages.

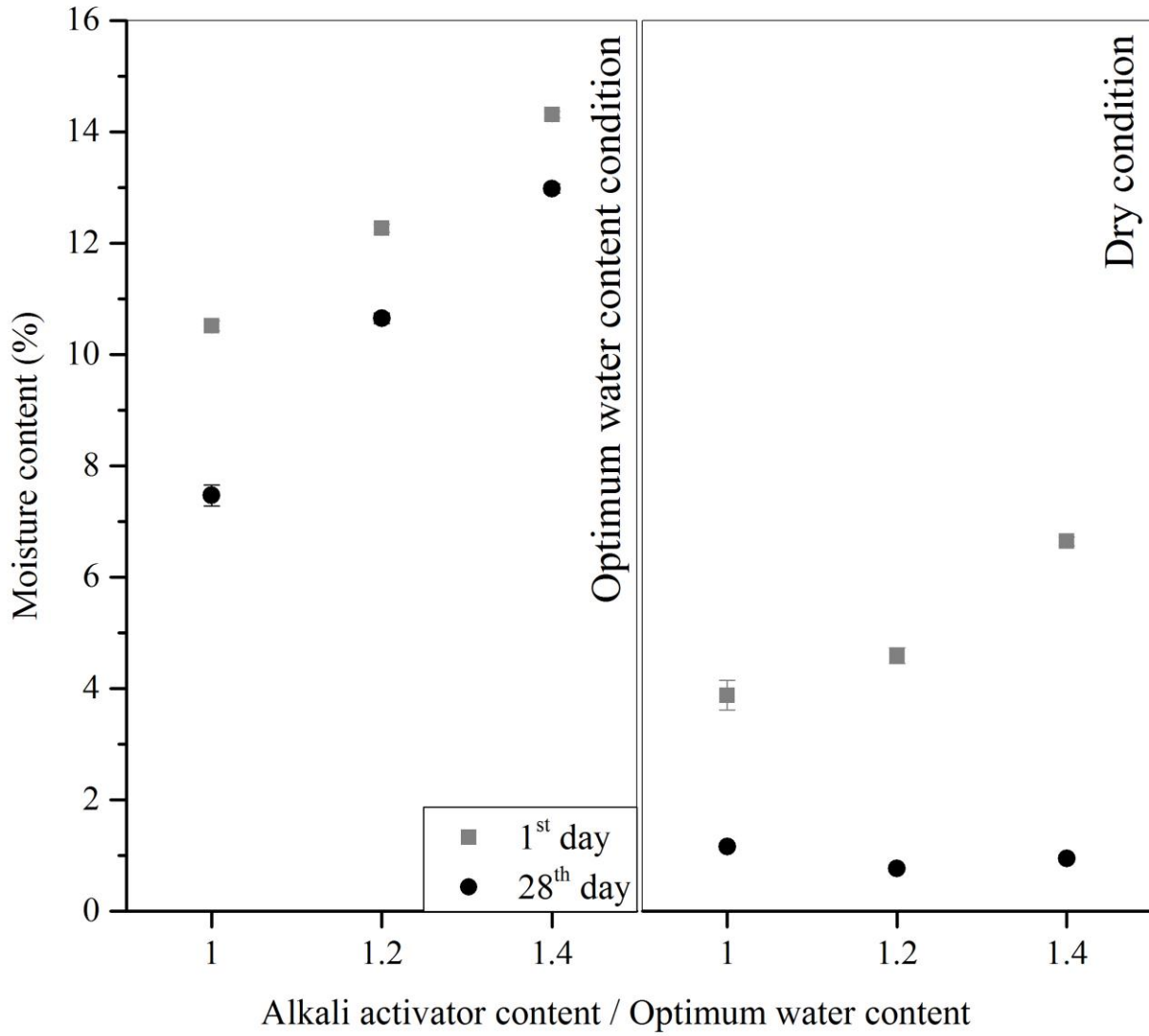


Fig 10. Moisture content of stabilized soils using geopolymers at optimum water content and dry conditions with different alkali activator content at different ages.

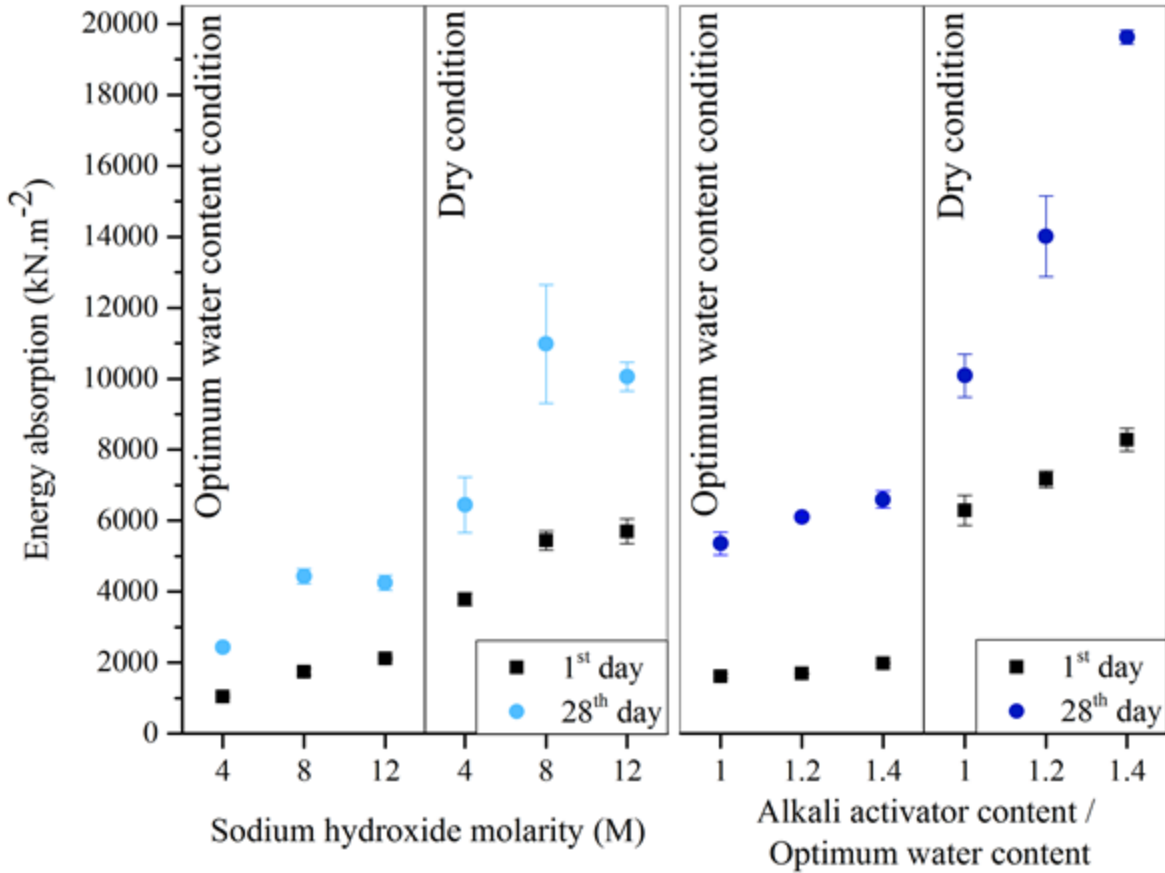


Fig 11. Energy absorption of stabilized soils using geopolymer at optimum water content and dry conditions with different alkali activator content and concentration at different ages.

5. Discussion

Dissolution and precipitation reactions of silicates, aluminates and calcium sources and hence the stable equilibrium phase assemblages of the oxide compositions are mainly depending on the pH of the medium and oxide concentration. Figure 12 shows that the pH of the geopolymer treated soil is about 11.5 to 12.5 which, depending on the activator concentration and binder content. A higher mechanical strength was obtained at higher pH where alkali activator concentration is higher [43, 44]. This is due to the predominance of smaller chain oligomers and monomeric silicate available to react with soluble aluminum in the presence of higher concentration of hydrogen ions during

dissolution and hydrolysis stages. This mechanism can be incorporated by calcium precursors available in VA [45]. Therefore, higher binder gel is produced. Possible dissolution (equations 1 and 2) and precipitation (equations 3 and 4) reactions occur during setting and hardening processes of high calcium based systems are [46]:

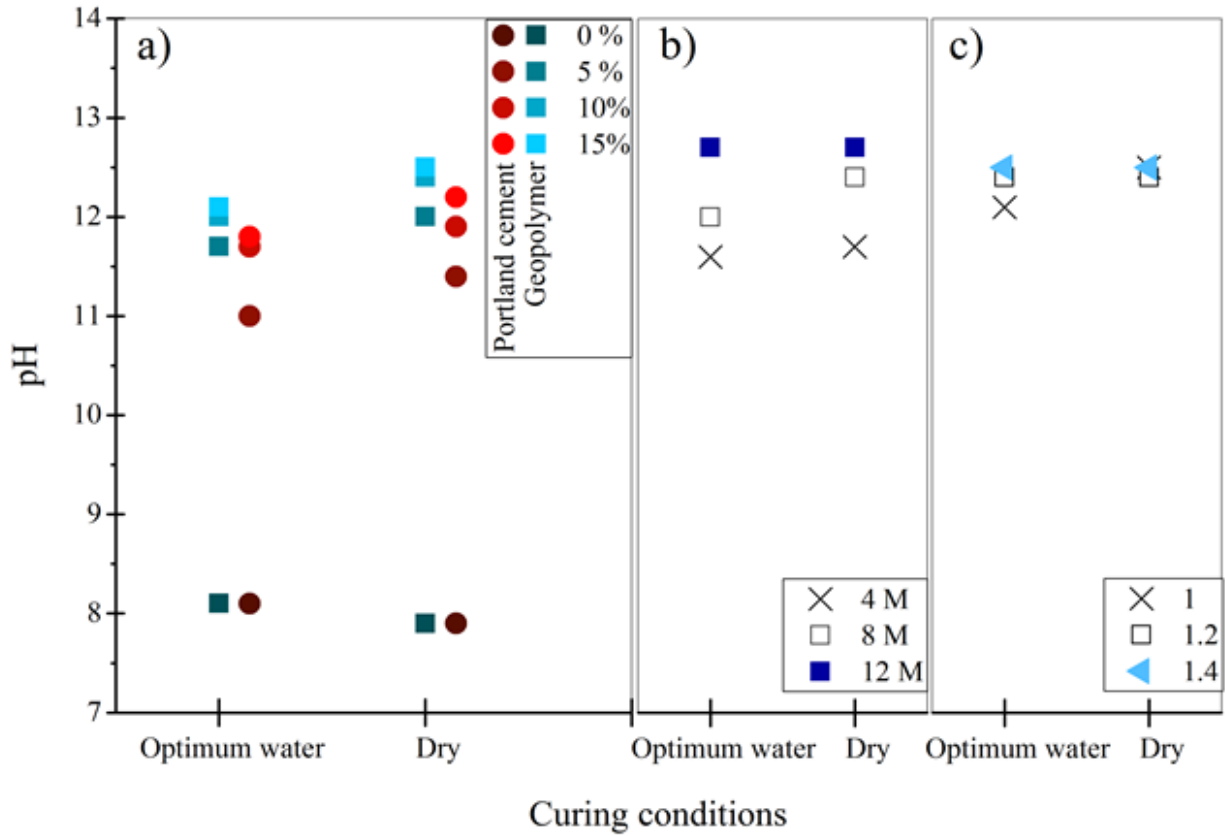
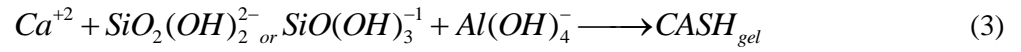
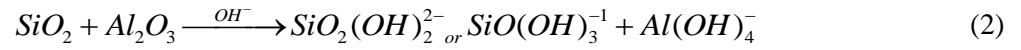
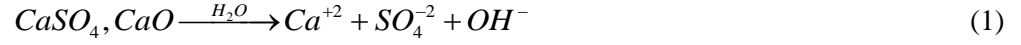
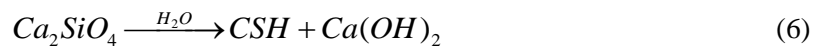
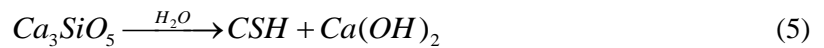


Figure 12. pH values of stabilized soils using geopolymer and OPC at optimum water content and dry condition with different binder content at 28th day of curing.

Similarly, upon the addition of water to OPC, each of the compounds undergoes hydration. However, mainly calcium silicates contribute in further mechanical strength development. In particular, tricalcium silicate initiates to release calcium ions, hydroxide ions. This reaction continuous till the system gets saturated and followed by crystallization of calcium hydroxide and formation of calcium silicate hydrate, equation 5. Simultaneously, dicalcium silicate reacts with water in a similar manner but with a slower rate, equation 6.



This gel formation can be explored through the evaluation of pH value. As observed in Figure 12, the pH value rises in the range of 11 to 12.1 when the OPC concentrations increased from 5 to 15 w.t. %. However, it was reported that the pH value increases significantly at lower cement content, but it stabled at the cement content of about 20 and above [47, 48].

The formation of the calcium hydroxide and calcium silicate hydrate crystals provide "seeds" upon which more calcium silicate hydrate can form. The calcium silicate hydrate crystals grow thicker, making it more difficult for water molecules to reach the unhydrated tricalcium silicate. Therefore, the strength development is reduced over time [49]. This is compounded when the water is not sufficient in the system or evaporated because of exothermic reactions and curing at hot and dry environments [50-52]. This loses the functionality of OPC stabilization at torrid zones.

6. Conclusions

This study compares the potential of geopolymer as an alternative environmental friendly clayey soil stabilizer to the conventional OPC in dry (DC) and wet (OC) curing conditions. The compressive strength of the untreated clayey soil specimens could be increased from 0.2 to 4 MPa and 2 to 12 MPa at the OC and DC conditions, respectively, when the soil partially replaced with 15 w.t.% of the binders. This increase is significantly depending on curing humidity and temperature. The VA-geopolymer stabilized soil indicated 200% improvement in compressive strength compares with the corresponding OPC specimens at the DC condition. While, OPC stabilized soil was superb at OC and shows 33% higher strength compare to the geopolymer specimens. Furthermore, high energy absorption in all the geopolymer specimens shows the ductile behavior of this material compare with OPC.

In addition to the curing condition, the increase in molarity of the sodium hydroxide from 4 to 12 M resulted in higher dissolution of aluminosilicate precursors and a consequent higher binder gel production. This leads to a higher mechanical strength (up to 50%) of the geopolymer specimens at both OC and DC. Furthermore, the increase in alkali activator content/optimum water content from 1 to 1.4 improves the DC compressive strength up to about 70% and 130% at 1st and 28th day, respectively. Whereas, the increase in alkali activator was ineffective at the OC, which is associated with the dilution of alkali activator at the wet condition.

Conflict of interest

The authors declare no conflict of interest.

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