



Strength of a Sandy Gypseous Soil Improved with Fly Ash Geopolymer

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ABSTRACT

This study investigates the strength performance and microstructural changes of a sandy gypseous soil improved with fly ash-based geopolymer, for shallow and deep applications. Different proportions of geopolymer were added to a natural gypseous soil having a gypsum content of 30% to 40% with different water contents. The fly ash was activated using sodium hydroxide with molar concentrations of 8 and 12 and sodium silicate. The ratios of the fly ash to the activator were 1 and 2. Specimens were cured for different ages at 30°C. To simulate the field conditions, a number of specimens were immersed in a salt-saturated solution. Materials performance was evaluated at the macro level by performing unconfined compression test and at micro level by performing scanning electron microscopy test. The study showed that an increase in the molar concentration of sodium hydroxide and of the binder ratio improved material's strength particularly at lower water contents of the soil. Increasing the binder content to about 30% improved the strength by enhancing the bonding between the soil particles. On the other hand, immersing the samples in the salt solution led, in most cases, to breakdown of the geopolymer network, as confirmed by the SEM images. It was concluded that the fly ash geopolymer-soil mixtures under investigation can provide as high as 8 MPa uniaxial strength under no sulfate attack. However, under sulfate attack condition, this strength can decrease to as low as 0.5 MPa. Even under the worst case, the later strength can be just enough to support shallow foundations rested on a saturated gypseous soil.

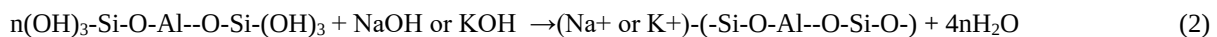
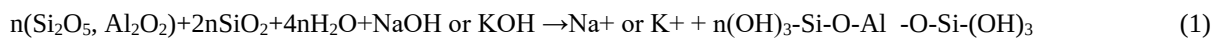
1.Introduction

Studies on gypseous soils' behavior appeared as early as the 1970s, due to their widespread in many arid or semi-arid regions of the world, such as North Africa, Southwest Asia, Russia, and the United States (Boyadgiev and Verheye, 1996). The problems of gypseous soils are typically associated with the presence of flowing water. These soils exhibit excessive volume changes in response to gypsum softening, dissolution and even leaching. Consequently, building constructed on these soils become susceptible to serviceability issues such as high total and differential settlement and depressions in roads and pavement (Sajedi et al., 2008; Chen and Liu, 2012). The depth of gypseous soil may extend to few meters below the ground. Deep replacement or treatment of these soils over the entire construction site is not always practically feasible due to the associated cost and unavailability

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of proper equipment. Instead, a less expensive yet effective improvement could be employed with in situ techniques such as deep soil mixing (Bouazza et al., 2004; Porbaha, 1998). In this technique, the in situ soil is locally mixed with the binder by means of an auger-mixing tool that digs through the soil while the binder is injected under high pressure. When the binder is hardened, a cylinder-like column with preferential stiffness and strength higher than those of the adjacent soil are achieved (Bruce et al., 2013).

Portland cement and lime are traditionally used in deep soil mixing to stabilize soils. However, the use of these materials is associated with some environmental and financial problems. The stabilization of gypseous soils using cement is not feasible given the expected internal sulfate attack. One of the main degradation mechanisms in sulfate attack is the formation of gypsum within the concrete structure resulting from the Ca^{+2} reaction in C-S-H and calcium hydroxide (OH) with SO_4^{2-} . The formation of ettringite from the interaction of the sulfate ions with C3A and its hydration products is another degradation mechanism (Irassar, 2009; Tian and Cohen, 2000). Both resulting compounds cause expansion and disintegration of the cement matrix (Schmidt et al., 2009; Neville, 2004). In addition, the use of cement in construction in general exposes the world to environmental risks, as the production of 1 ton of cement leads to the emission of 1 ton of CO_2 gas (Zhang et al., 2013). Therefore, it is of engineering and environmental interest to identify new soil remedial agents that are greener and more economical. The term geopolymer was initially coined by Davidovits in the 1970s. It is considered a cement alternative with numerous advantages, including for instance; good mechanical properties, low energy consumption, low cost, good volume stability, good impermeability and resistance to chemical attacks and low carbon footprint (Pham et al., 2021; Hassan et al., 2020). Geopolymer is formed by the reaction of silica- and alumina-rich materials with alkaline activator. Metakaolin, fly ash, palm oil and fuel ash are, generally industrial by-products, used as precursors in geopolymer binders which are mainly composed of SiO_2 , Al_2O_3 , Fe_2O_3 and CaO . The function of the activator is to dissolve the aluminum Al and silicon Si present in the precursor to facilitate the geopolymerization process by forming an aluminosilicate gel, as given in Eqs 1 and 2.



The dissolution could be related to the disintegration of -Si-O-Si or Si-O-Al- bonds by interaction with OH ions in the activator (Komnitsas, 2011). According to Cristelo et al. (2011), geopolymers have been widely used for soil stabilization. Arulrajah et al., (2018) in their work on geopolymer used two types of waste by-products; fly ash and sludge. The prepared geopolymer was used to treat a silty soil with moisture contents of (0.75, 1, and 1.25 of the liquid limit). The researchers concluded that the optimal precursor ratio was 20% binder consisting of 15% sludge and 5% fly ash. Rios et al., (2016) used fly ash and an alkaline solution made from sodium silicate and sodium hydroxide to stabilize soils for unpaved roads, it was found that the use of geopolymer differs from the ordinary cement in the curing rate, with alkali-activated specimens showing a more progressive and long-lasting strength increase. This was analyzed taking into account the chemical process responsible for the behavior of the mixtures. Laguros and Cokca, (2002) found that the compressive strength of expansive soil was improved by using 25% of fly ash class C. Gypseous soil is one type of collapsible soils. The most common definition of collapsible soils is any unsaturated soil of a metastable structure that goes through a radical rearrangement of particles and great loss of volume upon wetting (Clemence et al., 1981).

The current work explores the use fly ash geopolymer as a binder to stabilize a sandy gypseous soil. The expected role of the binder is to bridge soil particles in such a way that gypsum dissolution, on exposure to water, will not cause substantial deformation or lose of strength.

2. Materials and methods

2.1 Materials

2.1.1. Soil

The soil used in this work was brought from a site within the main campus of the University of Anbar. This campus was constructed on a layer of sandy gypsums soil extending few meters below the ground surface. In this area, groundwater is usually encountered at 3 to 4 meters below the ground level. The top 0.5 m soil was removed

to avoid plant roots and other unwanted materials. Visual inspection indicated that the soil color was light brown to reddish brown and the soil consisted mainly of sand particles bonded by gypsum lumps. Salt leaching caused by water percolation and by capillary action was evident. The geotechnical index property tests were as shown in Table 1. The grain size distribution is presented in Fig.1.

Table 1- Soil properties

Test	Specification	Value
Gravel content (%)	ASTM D422-07	2.3
Sand content (%)		94.2
Fines content (%)		3.5
Liquid limit (%)	ASTM D4318-17	Non-plastic
Plastic limit (%)		
Specific gravity	ASTM D854-06	2.43
Field bulk unit weight (kN/m ³)	ASTM D1556-15	12.3
SO ₃ (%)	BS1377-3:1990	14.42-18.6
Gypsum content (%)		31-40
Classification (ASTM D 2487-17)	(ASTM D2478 2006)	SP
		Cu=2.7, Cc=0.9

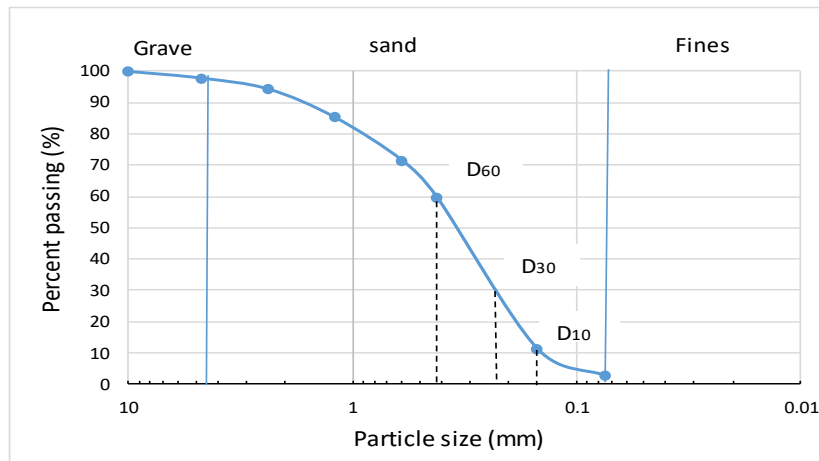


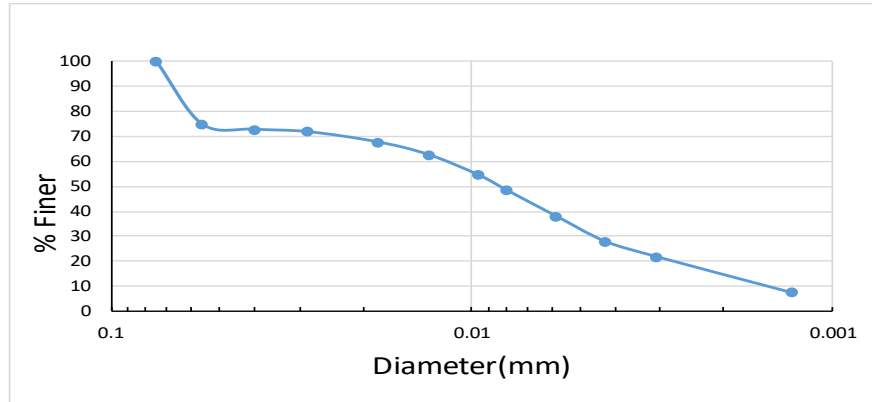
Fig.1 Soil sieve analysis

2.2. Fly ash

The fly ash used in this study was obtained from power plants as a by-product of electromagnetic induction processes. The main chemical compounds constituting the fly ash used in this study were determined by performing X-Ray Fluorescence (XRF) test and are given in Table 2. It can be considered a low activity fly ash containing slightly more than 10% calcium oxide (CaO). Physically, the fly ash had a brown color with a specific gravity of 2.55 as determined according to ASTM D854-06. The gradation analysis, by sieving and hydrometer, indicated that all granules passed through sieve #200 (0.075 mm), as shown in Fig.2.

Table 2- Chemical composition of the fly ash

Compound	SiO ₂	Al ₂ O ₃	K ₂ O	CaO	Fe ₂ O ₃	SO ₃	MgO
Mass (%)	11.1	2.33	1.015	10.37	10.16	0.324	0.27

**Fig.2 Fly ash gradation**

2.3. Alkali activator

Alkali activator was formed by mixing 8 Molar or 12 Molar sodium hydroxide (NaOH) with sodium silicate (Na₂SiO₃) solution. The sodium hydroxide was flake-like shells with 99% purity. The solution was prepared 24 hrs before use by dissolving the designated amount of NaOH in one liter of distilled water then mixing the solution for 10 minutes. Table 3. shows specifications of the sodium silicate solution used in this study.

Table 3- Specifications of the sodium silicate (as given by the manufacture)

Properties	Measure
SiO ₂ %	32.5
Na ₂ O %	13.4
Ratio of SiO ₂ / Na ₂ O	2.4
Density – 20° Baume	51
Specific Gravity(g/cm ³)	1.54
Color	Cloudy

2.4. Experimental work

2.5. Mix design

A number of geopolymer-soil mixtures were prepared with variable ingredients proportions and tested to evaluate their mechanical performance. These mixtures were prepared with different magnitudes of; sodium silicate solution to sodium hydroxide solution denoted as activator (A), fly ash to alkaline activator (F), water content (w), sodium hydroxide solution molarity (M), binder content (Ac) defined as the ratio of blending material (i.e., fly ash, NaOH and Na₂SiO₃) to soil.

The activator (A) was fixed to 1, based on a suggestion by a number of researchers, which also helped reducing the number of tests. Three binder contents were investigated in this work; 17%, 23%, and 33%. Two fly ash to activator (F) values were selected; 1 and 2. To incorporate the effect of field water content on the mechanical properties of the materials, three water contents were selected; 10%, 20% and 30%.

2.6. Specimen preparation

For the mixtures with $A=1$ (by other words; 1:1), the required amount of NaOH and Na_2SiO_3 solutions were first mixed together and left for about 2 hours to cool down. The liquid geopolymer was added to the soil at a specific binder content (A_c). The mixing process continued for about 3 minutes until a homogeneous paste was formed inside the bowl, as shown in Fig.3. Then, the mixture was poured into cylindrical open-ended molds and PVC tubes with 50 mm in diameter and 100 mm in height. These molds were mounted vertically on a levelled plate. To get rid of entrapped air, the material inside each mold was rodded 25 times. The casted material was subsequently placed in an oven at a constant temperature of 30 °C for 24 hours. Next, the specimens were extracted from the molds. To replicate a field dry condition, a number of these specimens were wrapped with a bakery wrap plastic sheets and returned to the oven for curing until the time of testing. Another number of these specimens were soaked inside a container containing gypsum-saturated solution, as shown in Fig.4. The container was stored inside an oven at a constant temperature of 30 °C so that results are comparable with those stored at dry condition. The specimens were kept soaked until the day of the test.

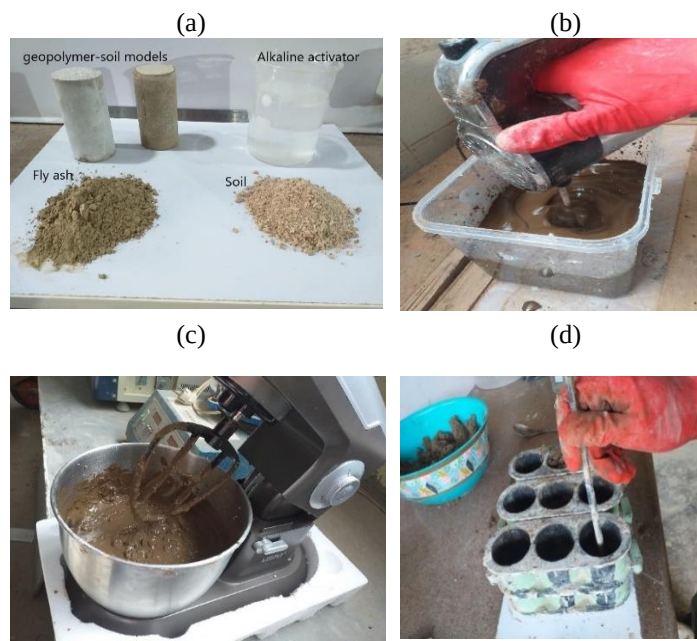


Fig.3 Preparation of geopolymer-soil mixtures (a) Mix materials (b, c) Mixing process (d) Rodding process



Fig.4 Curing process for durability

To investigate the effect of mix proportions on strength, stiffness, durability and before that mix workability, quality control, and cost, four test sets were designed and performed, see Table 4. Test Sets 1 and 2 were performed on the specimens prepared from geopolymer without soil. On contrary, Test Sets 3 and 4 were performed on the specimens prepared from geopolymer mixed with soil. Specimens of Sets 1 and 3 were wrapped with bakery wrap plastic sheets and stored in the oven whereas specimens of Sets 2 and 4 were soaked in gypsum-saturated solution

to evaluate the effect of sulfate attack on materials behavior. In all the test sets, specimen behavior was examined at age of either 3, 7, 14, 28 or 240 days.

Table 4- Details of the test sets

Test set	Mix code	M	F	A	Ac	Wc	Test age (day)	The mix contains soil	Curing conditions
1	M8F1A1	8	1	1	-	0	3,7,14 & 28	No	Wrapped in plastic sheets + constant temperature of 30°C
	M8F2A1		2						
	M12F1A1	12	1						
	M12F2A1		2						
2	M12F1A1	12	1	1	-	0	7,14, 28 & 240	No	Soaked in gypsum solution + constant temperature of 30°C
	M12F2A1		2						
3	M8F1A1	8	1	1	17, 23 & 33	10, 20 & 30	3,7,14& 28	Yes	Wrapped in plastic sheets + constant temperature of 30°C
	M8F2A1	8	2	1	17, 23 & 33	10, 20 & 30		Yes	
	M12F1A1	12	1	1	17, 23 & 33	10, 20 & 30		Yes	
	M12F2A1	12	2	1	17, 23 & 33	10, 20 & 30		Yes	
4	M8F1A1	8	1	1	17, 23 & 33	10, 20 & 30	3,7,14& 28	Yes	Soaked in gypsum solution + constant temperature of 30°C
	M8F2A1	8	2	1	17, 23 & 33	10, 20 & 30		Yes	
	M12F1A1	12	1	1	17, 23 & 33	10, 20 & 30		Yes	
	M12F2A1	12	2	1	17, 23 & 33	10, 20 & 30		Yes	

2.7. Testing

2.7.1 Workability

The workability of the mixtures was investigated according to American specification (ASTM C 124-39) based on how easy was to stir and mix and cast the specimens of a given mix. Figure 5 shows two specimens from different mixes where one of which was found difficult to prepare and cast. It is clear that low workability resulted in low quality specimens characterized with irregularity and disintegration. Table 5 shows the limits for workability of mixtures.

2.7.2 Initial and final setting time test

The time for initial and final setting is considered practically important. Up to the researchers' knowledge, no particular standard specification has been issued to measure the initial and final setting time for geopolymer binders. The American specification (ASTM C 191-04) was utilized to perform this test on geopolymer pastes

(i.e., without soil), although it has been originally specified for cement pastes. Vicat device was used in this test. Table 6, Show the limits for initial and final setting time test.



Fig.5 The effect of mix workability on specimen's quality

Table 5- Workability of the mixtures of Test Set 4

Molarity		8 Molar NaOH				
Fly ash /activator		F=1			F=2	
Binder content	Ac=17	Ac=23	Ac=33	Ac=17	Ac=23	Ac=33
Water content						
Wc=10%	Fail	Fail	Pass	Fail	Fail	Pass
Wc=20%	Pass	Pass	Pass	Fail	Pass	Pass
Wc=30%	Pass	Pass	Pass	Pass	Pass	Pass

Molarity		12 Molar NaOH				
Fly ash /activator		F=1			F=2	
Binder content	Ac=17	Ac=23	Ac=33	Ac=17	Ac=23	Ac=33
Water content						
Wc=10%	Fail	Fail	Pass	Fail	Fail	Pass
Wc=20%	Fail	Pass	Pass	Fail	Pass	Pass
Wc=30%	Pass	Pass	Pass	Pass	Pass	Pass

Table 6- Initial and final setting time

Mixture	Initial setting (min)	Final setting (min)
8 (NaOH molarity) mixture	30	60
12 (NaOH molarity) mixture	20	50

2.7.3 Unconfined compression test

This test was performed in accordance with the specification (ASTM D2166-06). Before commencing the test, specimen's weight and dimensions were taken and the specimen's top and bottom faces were levelled off. The test was performed by using a displacement-controlled load frame, equipped with a 50 kN load cell and a 0.01 mm precision digital dial gauge to measure axial displacement.

2.7.4 Microstructural tests

To gain clear understanding on the differences in the behavior of various geopolymer-soil mixes, a number of microstructural tests were performed by using Field Emission Scanning Electron Microscope (FE-SEM).

3. Results and discussion

3.1 Compressive strength of hardened geopolymer specimens (Test Set 1)

Figure 6 presents compressive strength values of Set 1 specimens, against specimen's age. The specimens prepared with 12M expressed significantly higher strength than those prepared at 8M, regardless the value of the fly ash to activator ratio (F). Interestingly, specimens prepared at F=1 showed always higher strength than those prepared at F=2. This may be attributed to increase in sodium hydroxide ion (OH⁻), which works on the continuous dissolution of oxides of silicon and aluminates. In addition, the increase in sodium silicate enhances this reaction (Komnitsas, 2011).

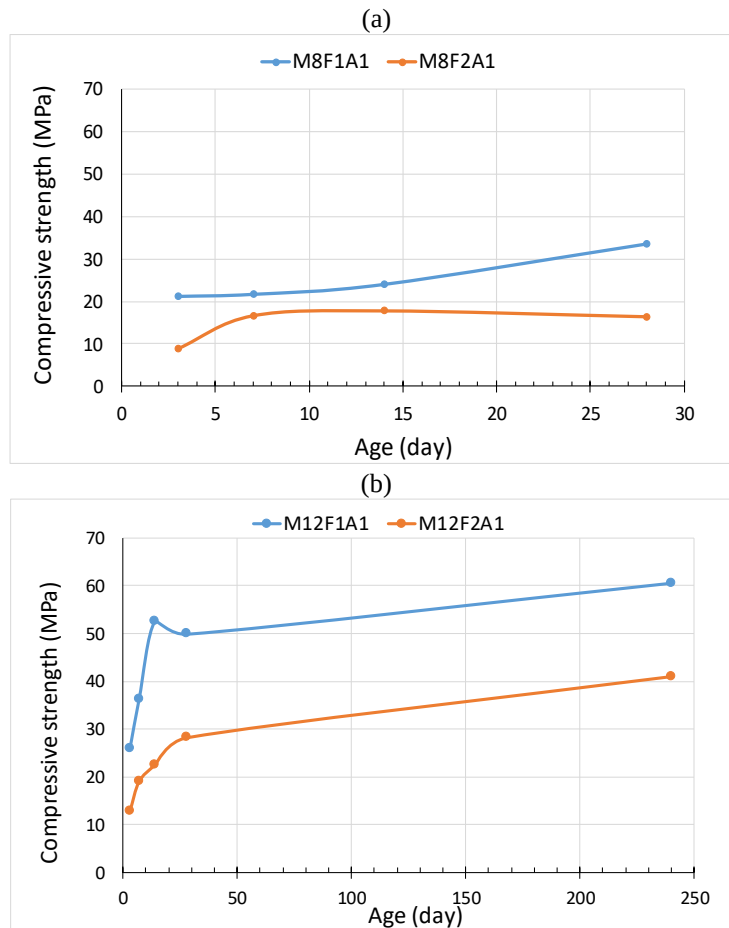


Fig.6 Compressive strength variation with specimens' age for Test Set 1 (a) 8M (b) 12M

3.2 Compressive strength of geopolymer specimens (Test Set 2)

Figure 7, presents the variation of compressive strength against age of Set 2 specimens in parallel with the results of Test Set 1 for comparison. For ages up to 28 days, the specimens prepared with $F=1$ showed a continuous buildup of strength and yielded higher strength values than those prepared with $F=2$. At the age of 240 days the compressive strength of the specimen with $F=2$ was however higher than that with $F=1$. The effect of sulfates on the compressive strength was very clear, particularly on the specimens with $F=1$. These specimens developed much less strength compared to those cured under dry condition. For instance, for the mix M12F1A1 at the age of 28 days the unsoaked specimen yielded a compressive strength of 50000 kPa, as shown in Fig.7. On the other hand, the corresponding soaked specimen yielded a compressive strength of 36560 kPa. For the specimens with $F=2$, the situation was however different where both test groups demonstrated nearly the same strength. Rigorously, the reason of this observation is not clear to the researcher. This suggests that high F value are preferable as long as durability is major concern. Nevertheless, a compressive strength of not less than 30 MPa should be guaranteed. This suggests that fly ash geopolymer can evidently be used as a substitute to conventional concrete for underground applications with severe sulfate attack.

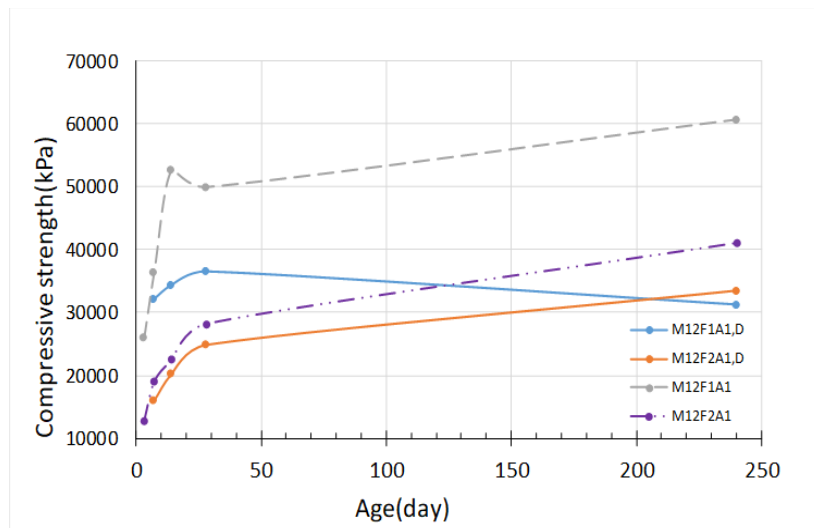


Fig.7 Effect of soaking in gypsum solution on the strength of hardened geopolymer specimens with $A=1$

3.3 Compressive strength of geopolymer-soil specimens (Test Set 3)

Figure 8, shows the effect of NaOH molarity (M) on compressive strength of specimens with $F=1$ and $F=2$, respectively. Inspection of these figures indicates that the strength of specimens with 12M is, in most cases, higher than that of specimens with 8M. The effect of 12M on the strength increase is much pronounced in the mixes with binder content of 33% and water content of 20%. On the other hand, only a little difference in the compressive strength can be noticed between the mixes prepared at 17% binder content and 30% moisture contents. This is, perhaps, due to the fact that increasing the mixing water leads to dilution of the activator, in a way that negatively affects the strength for all ages. This agrees well with the works of Risdanareni et al. (2014) and Bachtiar et al. (2020).

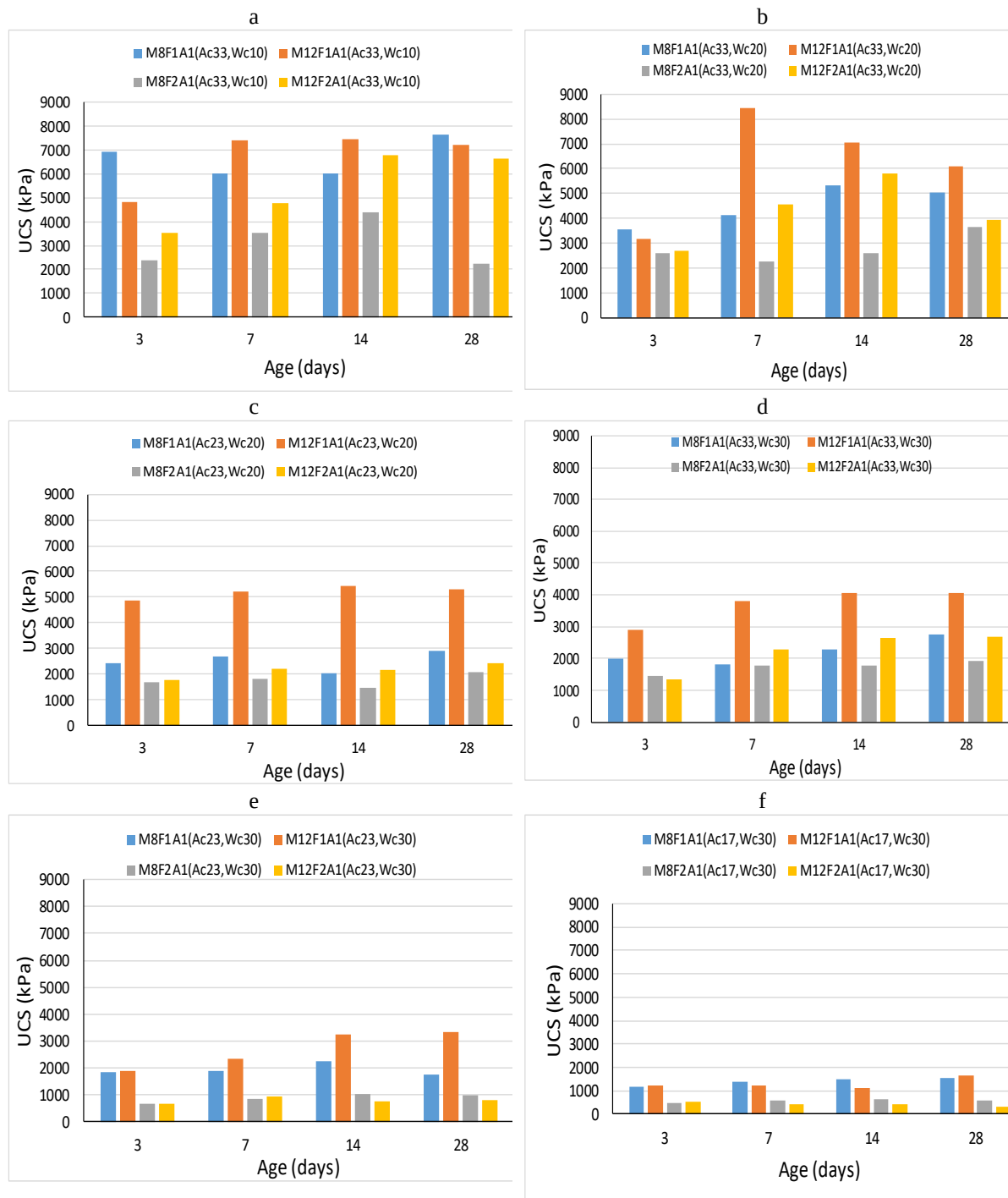


Fig.8. The effect of NaOH molarity (M) and (F) values on the UCS of the specimens (Test Set 3)

Figure 9, demonstrates the effect of increasing binder content on compressive strength of specimens prepared with 8M and 12M. The increase of binder content from 17% to 33% resulted in an approximately linear increase of 50% to 150% in the compressive strength. It was observed that specimens with lower water contents exhibit higher compressive strength values (not shown in the figure).

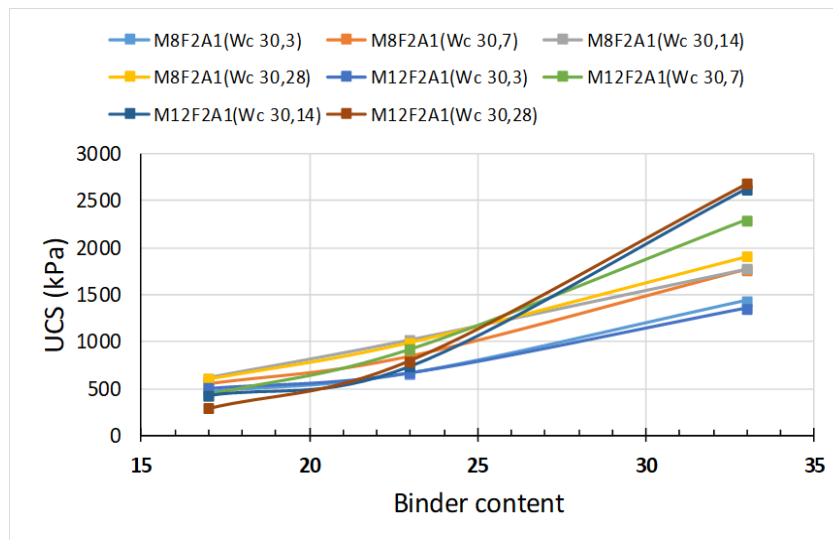


Fig.9 The effect of increasing binder content on compressive strength

3.4 Compressive strength of geopolymer-soil specimens (Test Set 4)

Most of the specimens with $F=1$ experienced a severe deterioration while immersed in the salt solution, see Fig.10. On the contrary, the majority of the specimens with $F=2$ maintained their integrity and showed a reasonable resistance to sulfate attack.



Fig. 10 Immersed specimens in solution

Figure 11, compares compressive strength of specimens of Set 3 in which the specimens were subjected to dry curing, and the corresponding specimens of Set 4 in which the specimens were subjected to wet curing with sulfate attack. In all the cases presented in the figure, the compressive strength exhibited a reduction of 10% to 80%, as a result of soaking in gypsum solution. Unlike in Set 3, the compressive strength of Set 4 was always decreasing with increasing time of soaking. Visual inspection of the soaked specimens indicated that these specimens tended to become softer with increase soaking time.

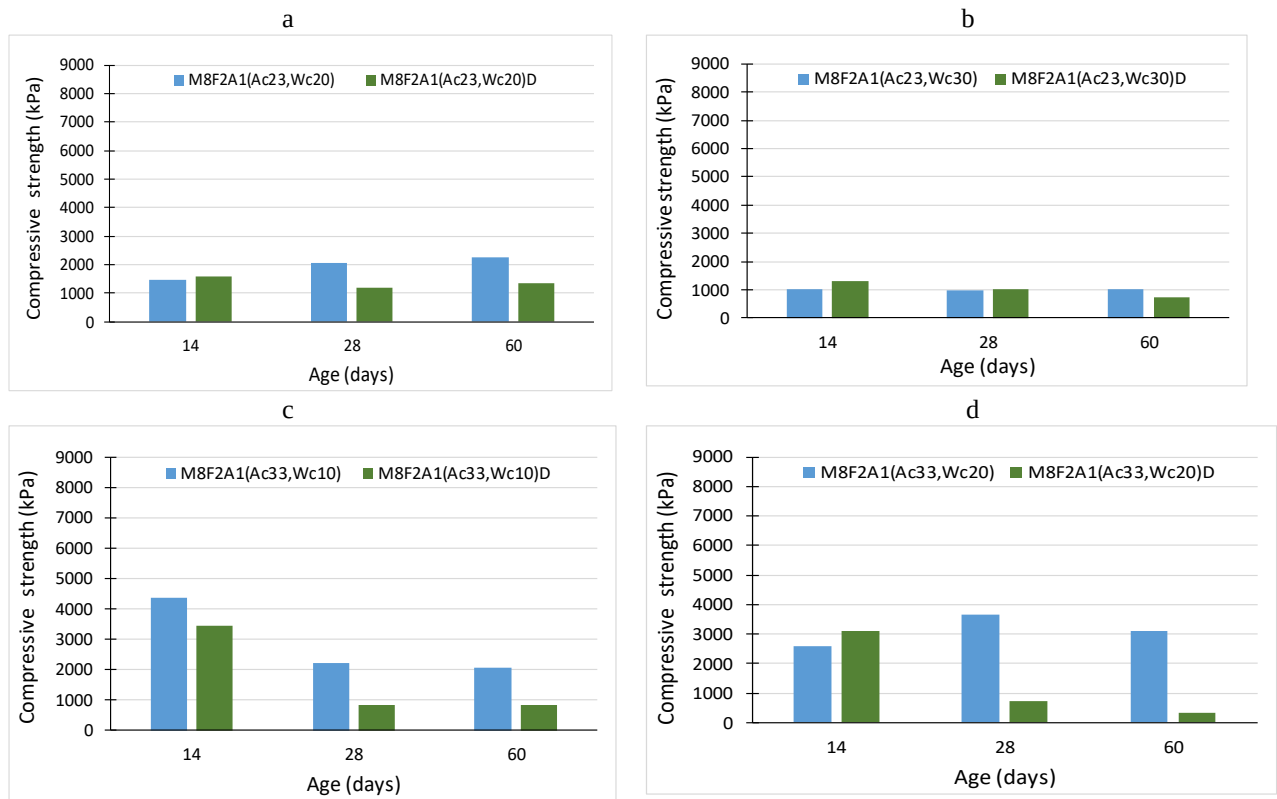


Fig. 11 Compressive strength of Set 3 (dry curing) vs Set 4 (wet curing with sulfate attack). The soaked specimens are identified with the letter “D”

3.5 Microstructure of geopolymer – soil mixtures

Figure 12, shows SEM images taken on a specimen made from geopolymer-soil mixture, cured to 28 days. Fig. 12(a) shows geopolymer gel coating both soil particles and gypsum crystals. Fig. 12(b) shows the forms of pores and cracks in the internal structure that occur as a result of the increase in mixing water or through shrinkage of the material with water evaporation. In addition, individual unreacted fly ash particles can be clearly seen. Inspection of Fig. 12(c) indicates an in-progress geopolymerization process occurring at the age of 28 days. Evidently, geopolymer acts as a binder between solid materials.

Figure 13, shows SEM images taken on a specimen after 28 days of immersion in the salt solution. In Figure 13(a) a number of soil particles appear on the surface and geopolymer gel does not provide a complete encapsulation of soil particles and gypsum crystals, where the geopolymer chains are weak and disconnected. Figure 13(b) shows a number of non-interacting fly ash particles. This is probably due to the cessation of interaction between fly ash and the active substance. Inspection of Figure 13(c) shows uneven distribution of pores where the produced geopolymer is not sufficient to bridge the soil particles.

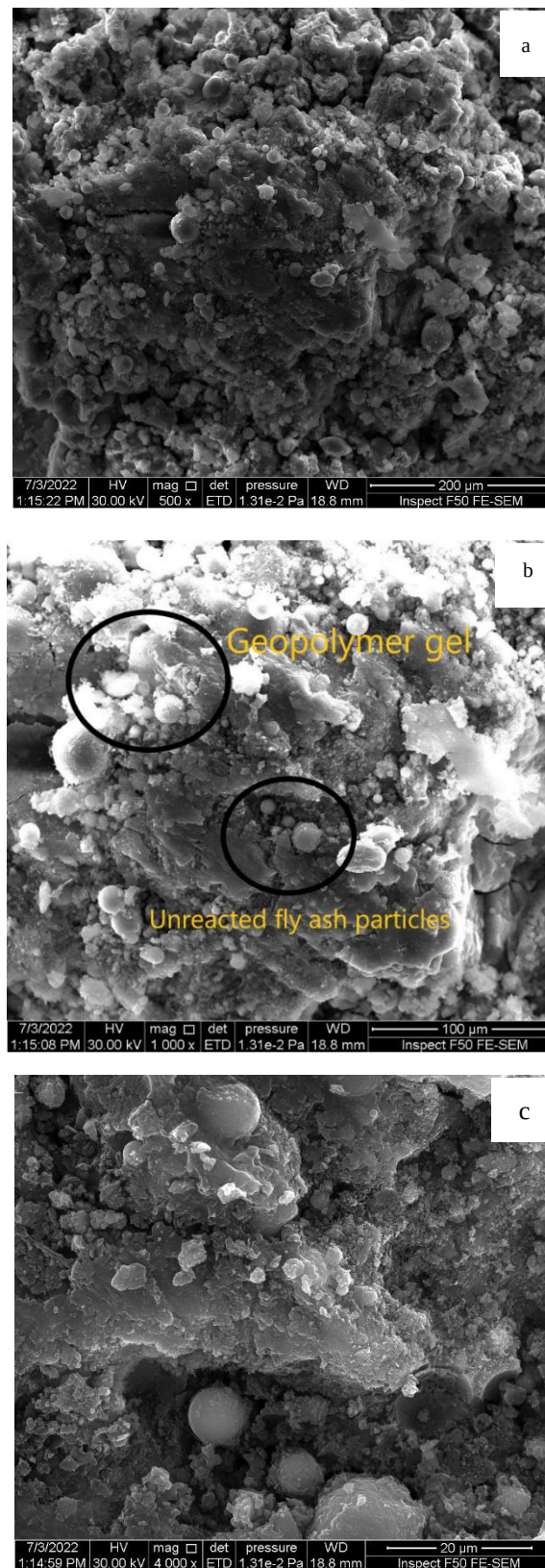


Fig.12 SEM images of a geopolymer-soil mixture (a) 500 x magnification (b) 1k x magnification (c) 4k x magnification

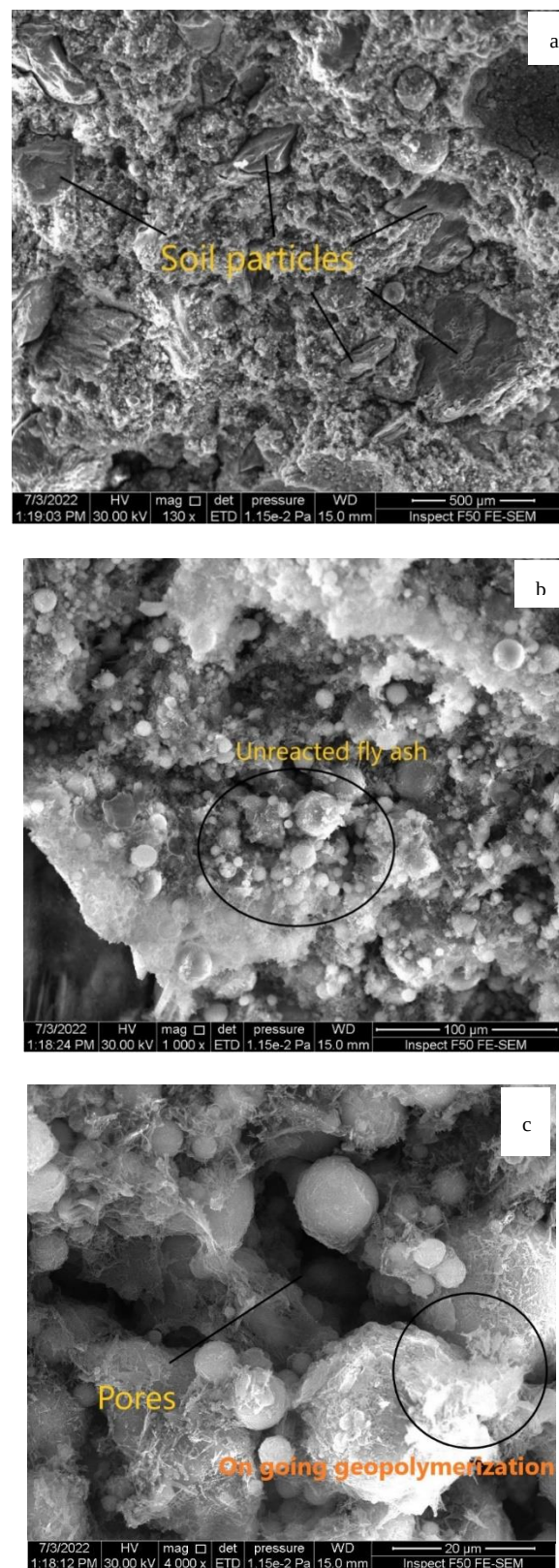


Fig.13 SEM images of a geopolymer-soil mixture subjected to sulfate attack (a) 130 x magnification (b) 1 kx magnification (c) 4 kx magnification

4. Conclusions

This study investigated the possibility of using fly ash geopolymer to stabilize a sandy gypseous soil for shallow and deep applications. The main conclusions are summarized below.

- The initial and final setting time tests showed that the geopolymer needed a relatively short time (in the order of few hours) to solidify at ambient temperature.
- Mixing of geopolymer with gypseous soil produced mixtures with an unconfined compressive strength of 0.5 to 8 MPa. This range of strength gives geopolymer the potential to be used for soil improvement. Geopolymer pastes gained about 80% of their ultimate strength during the first 3 to 7 days of casting.
- The geopolymer-soil mixtures containing fly ash / activator equal to 1 showed higher strength values, by about 20% to 150%, than those with fly ash/activator equal to 2. Increasing the molar concentration of sodium hydroxide to 12M improved the properties of the geopolymer thus soil. For example, the strength of the 12M samples was higher than that of corresponding samples with 8M by 50%. Although the decrease in the amount of the mixing water led to an increase in the strength, it negatively affected the workability. This phenomenon is normal, where with increasing water content, more voids generated in the specimens caused by evaporation and resulted in strength decrease.
- Increasing the proportion of binder in the mixture led to an improvement in its strength and workability in general. For example, the strength increased by about 100% in response to an increase of 95 % in binder content. However, under sulfate attack conditions, strength of the geopolymer-soil mixtures degraded by different amounts in response to that water saline stops spreading of geopolymer mesh to cover whole soil particles. The microstructural investigation by the SEM technique showed that geopolymer chains of the specimens with higher strength tended to be more interconnected than those of specimens with lower strength.

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