

# RESISTANCE OF ALKALI ACTIVATED WATER- COOLED SLAG GEOPOLYMER TO SULPHATE ATTACK

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*Ground granulated blast furnace slag is a finely ground, rapidly chilled aluminosilicate melt material that is separated from molten iron in the blast furnace as a by-product. Rapid cooling results in an amorphous or a glassy phase known as GGBFS or water cooled slag (WCS). Alkaline activation of latent hydraulic WCS by sodium hydroxide and/or sodium silicate in different ratios was studied. Curing was performed under 100 % relative humidity and at a temperature of 38°C. The results showed that mixing of both sodium hydroxide and sodium silicate in ratio of 3:3 wt.% is the optimum one giving better mechanical as well as microstructural characteristics as compared with cement mortar that has various cement content (cement : sand were 1:3 and 1:2). Durability of the water cooled slag in 5 % MgSO<sub>4</sub> as revealed by better microstructure and high resistivity-clarifying that activation by 3:3 sodium hydroxide and sodium silicate, respectively is better than using 2 and 6 % of sodium hydroxide.*

## INTRODUCTION

Geopolymers are a class of cementitious materials that do not depend on the presence of calcium. Geopolymers are formed by mixing aluminosilicate materials (e.g. metakaolin, fly ash or slag) with alkali-silicate solutions produces high strength binder under highly alkaline conditions (pH = 12-14.5). This binder has great potential as an environmentally favourable alternative to Portland cement. It could produce a binder with the advantages of Portland cement but at a lower cost, with a large reduction in CO<sub>2</sub> emissions and will recycle an industrial waste material as a raw material. Examples of this type of binder include Class F fly ash (low calcium fly ash) based geopolymer (FFG) [1-3] and alkali-activated slag cement (AAS) [4-8].

Geopolymers can be thought of as a new generation binder as a substitute for the calcium silicate hydrate which are essential components of Portland cement. Ordinary Portland Cement (OPC) is the main ingredient used in the production of concrete- the most widely used construction material in the world. In the past, concrete was simply a composite of OPC paste with aggregates, however, modern-day concrete incorporates other cementitious materials, which act as partial replacements of OPC. The manufacturing of OPC requires the burning of large quantities of fuel, and decomposition of limestone. Both, burning of fuel and decomposition of limestone, result in significant emissions of carbon dioxide. For every ton of OPC manufactured, nearly

one ton of CO<sub>2</sub> is produced depending on the production process adopted [9]. Cement plants are reported to emit up to 1.5 billion tons of CO<sub>2</sub> into the atmosphere annually [10,11]. Hence, environmental preservation has become a driving force behind the search for new sustainable and environmentally friendly composites to replace conventional concrete produced from OPC. In 1978, Davidovits [12] introduced the word 'geopolymer' to describe an alternative cementitious material, which has ceramic-like properties. As opposed to OPC, the manufacture of aluminosilicate-based geopolymer does not consume high levels of energy, as water cooled slag (WCS), known also as ground granulated blast furnace slag, is already an industrial by-product. This geopolymer technology has the potential to reduce emissions by 80% [9] because high temperature calcining is not required. It also exhibits ceramic-like properties with superior resistance to fire at elevated temperatures. Geopolymer can be produced by combining a pozzolanic compound or aluminosilicate source material with highly alkaline solutions [13].

The addition of alkali-silicate solution (e.g. water glass, Na<sub>2</sub>SiO<sub>3</sub> or K<sub>2</sub>SiO<sub>3</sub>) can raise the rate of strength development as well as largely enhance the final strength by promoting the hydrolysis of the siliceous and aluminum species of the raw materials as well as providing additional silicate species, sodium, or potassium ions, which alkaline ions are required for geopolymerization [2,3,14]. The effect of adding the water glass to the reaction mixture on the final phases present

in the reaction product has not yet been cleared. Many studies have concluded that geopolymer binder may be viewed as an amorphous analogue of zeolites or zeolitic precursors [1,15,16]. The analogue of hydrothermal zeolite synthesis [17-20] to geopolymerization has been proposed based on similarities such as raw materials and activating solutions [2,15]. However, geopolymerization uses a relatively lower solution/binder ratio, less than 0.6, and lower reaction temperature, under 90 °C. This leads to the geopolymer products being nearly amorphous [1-3]. Geopolymer binder is generally synthesized in a temperature range of 20-90 °C [2]. Geopolymers formed at room temperature are amorphous. With increasing temperature, crystalline phases begin to appear. However, there have not been any detailed studies of the effect of curing temperature on the properties of the geopolymer.

This paper provides an investigation of alkaline activation of water cooled slag using separately sodium hydroxide and sodium silicate in different ratios and mixing of both so as to elucidate the optimum mix composition and comparing the mechanical properties of the resulted mix along with the mortar compositions.

## EXPERIMENTAL

### Materials

The materials used in this investigation is water cooled slag or what is known as ground granulate blast furnace slag (GGBFS) sourced from Iron and Steel Factory- Helwan, Egypt. Two different alkali activators, sodium silicate solution and sodium hydroxide. Sodium hydroxide (NaOH) is produced by SHIDO Company with purity 99 % and liquid sodium silicate (LSS) from Fisher Company consists of 32 % SiO<sub>2</sub> and 17 % Na<sub>2</sub>O with Silica modulus SiO<sub>2</sub>/Na<sub>2</sub>O equal 1.88 and density 1.46 g/cm<sup>3</sup> (Na<sub>2</sub>SiO<sub>3</sub>, 9H<sub>2</sub>O). Ordinary Portland cement from Helwan Company, Egypt and fine sand (< 1 mm) from Oases (Wahat)-Road, Egypt, both for mortar formation as a method for a comparison with the produced geopolymer composite. The chemical composition of the starting raw materials was illustrated in Table 1.

### Geopolymerization and curing

Geopolymer was made by hand-mixing raw materials of each mixture passing a sieve of 90 µm with the alkaline solution for 10 min. and a further 5 min. with a mixer. All investigations involved using of Na<sub>2</sub>SiO<sub>3</sub>

and NaOH in different ratios and a combination of Na<sub>2</sub>SiO<sub>3</sub> and NaOH in the ratio of 1:1, 2:2 and 3:3 (wt.%) of dry mixes, respectively. The water-binder material ratio (w/b) was 0.25 by mass. The paste mixture was cast into 25×25×25 mm cubic-shaped moulds, vibrated for compaction and sealed with a lid to minimize any loss of evaporable water.

All mixes were left to cure undisturbed under ambient temperature for 24 h, and then subjected to curing temperature of 38°C with a 100% relative humidity. At the end of the curing regime, the specimens were subjected to the compressive strength measurements and then the resulted crushed specimens were subjected to stopping of the hydration process using solution of alcohol/acetone (1:1), followed by washing with acetone as recommended by different investigators [21] to prevent further hydration and for further analysis followed by drying of the crushed specimens for 24 hours at 80°C, then preserved in a well tight container until the time of testing. On the other hand mortar specimens were prepared as the last mention mixing and curing condition using different sand (< 1 mm) to cement ratios of 2:1 and 3:1, respectively.

On the other hand, for sulfate attack measurements for optimum mixes is recorded according to ASTM C1012 [22]. Where, specimens were cast, cured in a fog room for 28 days, and then immersed in a solution containing 0.352 M of MgSO<sub>4</sub> (about 5 % MgSO<sub>4</sub> solution). The solution in the container was replaced every 2 weeks up to 6 months. Compressive strength was measured periodically (monthly) over 6 months.

### Methods of investigation

Chemical analysis was carried out using Axios, WD-XRF *Sequential* Spectrometer (Panalytical, Netherland, 2009). Compressive strength tests were carried out using five tones German Bruf pressing machine with a loading rate of 100kg/min determined according to the DIN EN 197-1/09 [23]. The microstructure of the hardened alkali activated water cooled slag was studied using SEM Inspect S (FEI Company, Netherland) equipped with an energy dispersive X-ray analyzer (EDX). The removal of free water was accomplished by using alcohol/acetone method as recommended by different investigators [21]. Basicity of the medium was recorded on pH-meter. Setting time of the mixes used in this investigation were done using Vicat instrument according to ESS 2421-1993 [24].

Table 1. Chemical composition of starting materials (wt.%).

Oxide	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	MnO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	Cl <sup>-</sup>	L.O.I.	Total
Water-cooled slag (GGBFS)	37.95	11.01	1.48	33.07	6.43	3.52	0.74	1.39	0.52	3.44	0.10	0.05	0.00	99.70
Fine sand	89.90	2.00	1.45	1.56	1.91	0.87	0.37	0.06	0.03	0.04	0.03	0.12	1.65	99.99
(Sand dunes)	21.92	4.62	3.39	64.43	1.52	1.14	0.06	0.18	–	–	–	0.06	2.20	99.52

## RESULTS AND DISCUSSION

## pH-measurement of the used activators and setting time of geopolymer mixes

Measurements of pH the used activators illustrated in Figure 1, indicate the higher pH value of the mix that has 3:3 (wt.%) sodium hydroxide and sodium silicate, respectively, followed by lower doses of the last mentioned mix. While, sodium hydroxide has a lower value but exceed those of separate dose of sodium silicate. Geopolymers are formed by mixing aluminosilicate materials (e.g.: metakaolin, fly ash or slag) with alkali-silicate solutions produces high strength binder under highly alkaline conditions (pH = 12-14.5).

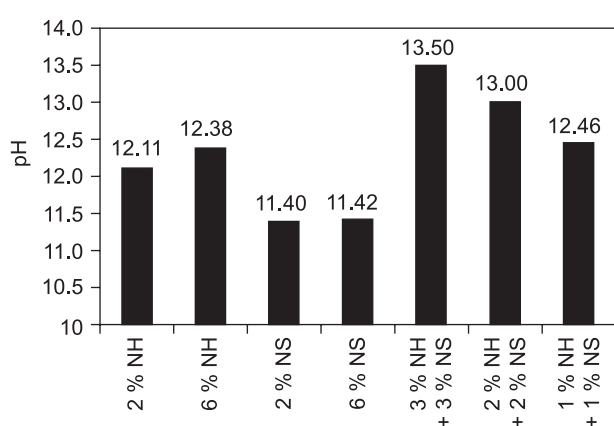


Figure 1. pH-measurements of the used activators.

The initial and final setting times of the water cooled mixes along with sodium hydroxide and mix of sodium hydroxide and sodium silicate are shown in Figure 2. Where, the mix specimens that activated using 6 wt.% sodium hydroxide have the lowest initial and final setting time (near to setting time of Portland cement) as compared with the lower dose of sodium hydroxide. Also, it can be seen that mixing of 3:3 (wt.%) of sodium, hydroxide and sodium silicate, respectively, requires longer time for hardening than 6 wt.% NaOH.

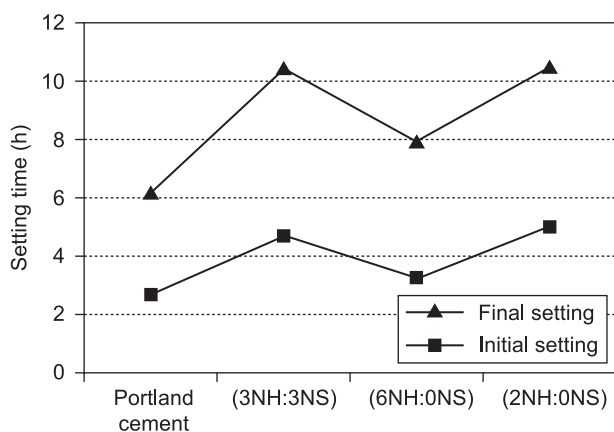


Figure 2. Initial and final setting time of alkali activated slag pastes (NH: Sodium hydroxide, NS: Sodium silicate).

## Effect of sodium hydroxide and sodium silicate on mechanical properties

The results of compressive strength for hardened geopolymer mixes along with various activator and cured at 100 % relative humidity and at 38°C for 90 days are shown in Figure 3. The results show the increase of strength in all activator mixes along with hydration age which is attributed to the continuing pozzolanic reaction in slag pastes. Also, mix with a dosage of 6 % NaOH (NH) achieved higher gain in compressive strength in comparison with all the other mixes. Where, it provides a high pH (more than 12) as mentioned latter, suitable for WCS activation and enhancing better geopolymeric structure as compared with other mixes. Where, 90 day compressive strength for 6%NH was 84.57 MPa while it was 67.23 MPa for 2 NH. This clearly shows the effect of the NaOH dosage on the early compressive strength gain and on the degree of geopolymerisation. In a similar manner, the 6 % NS (sodium silicate) mix had lower compressive strength gain (12.15 MPa at 90 days) in comparison to 2 % NS (9.31 MPa at 90 days) which is lower than the sodium hydroxide mixes. On the other hand, mortar strength is higher than that of the last mentioned mixes. These results of NS mixes alone are far from the NH mixes, reflecting the higher efficiency of NH than NS in the geopolymer formation.

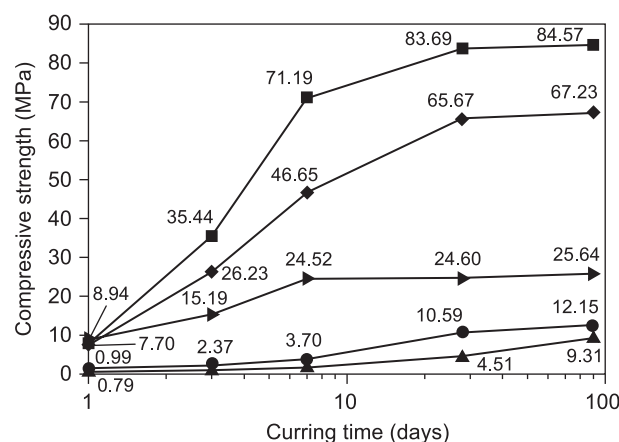


Figure 3. Compressive strength of the WCS mixes using different activators at different hydration ages (water/binder of 0.25 %); ♦ - 2 % NH, ■ - 6 % NH, ▲ - 2 % NS, ● - 6 % NS, ► - Mortar, 1Cement:3Sand.

The SEM microstructure of 90 days cured WCS specimens using 2 % NaOH as activator is shown in Figure 4. The micrograph indicates that the structure is nearly homogeneous with dispersing of massive geopolymer structure over the surface. Whereas, upon using EDX analysis for the geopolymeric structure, emphasize the composition of geopolymer structure that composes mostly of Si, Al, Na, Ca and O. On the other hand, using of 6 % NaOH results in a more homogeneous microstructure as represented in Figure 5,

where it reflects that the last mentioned dose is enough for mostly dissolving slag specimens but the EDX analysis ensure that there is much sodium content which can consume the surface species leading to lowering in oligomer structure and its stability upon time [25]. It is known that, the compressive strength of geopolymers is related to the degree of polymerization, which is strongly affected by the soluble silicate and aluminates of the geopolymeric system. The higher the degree of polymerization in the geopolymeric structures, the higher the obtained compressive strength. The increase of NaOH concentration in the aqueous phase of the geopolymeric system has as a result the direct increase of the dissolution rate of Si and Si–Al phases improving the effectiveness of the geopolymerisation process.

Increased Si and Al contents in the aqueous phase of geopolymeric systems are essential for the initiation of the formation of oligomeric precursors and therefore, of the polycondensation that is the most important process of strength development in geopolymeric materials. Considering this analysis, it could be concluded that the continuous increase of NaOH concentration in the aqueous phase of the geopolymeric system would affect positively the mechanical properties of the obtained materials. Although the formation of oligomeric precursors is enhanced by the increased contents of Si and Al in the aqueous phase caused by the increased dissolution rates, is inhibited under extremely high NaOH concentrations. Moreover, sodium cations, which are normally presented at high concentrations in the

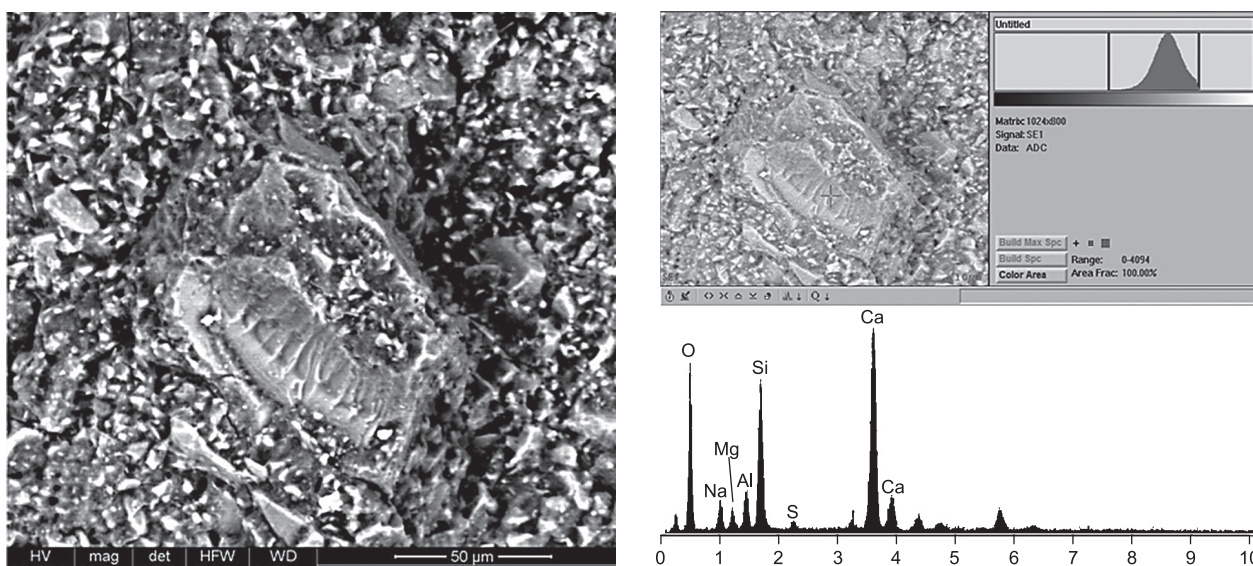


Figure 4. SEM micrograph of the 90 days cured WCS mixes having 2 % NH activator (water/binder of 0.25 %).

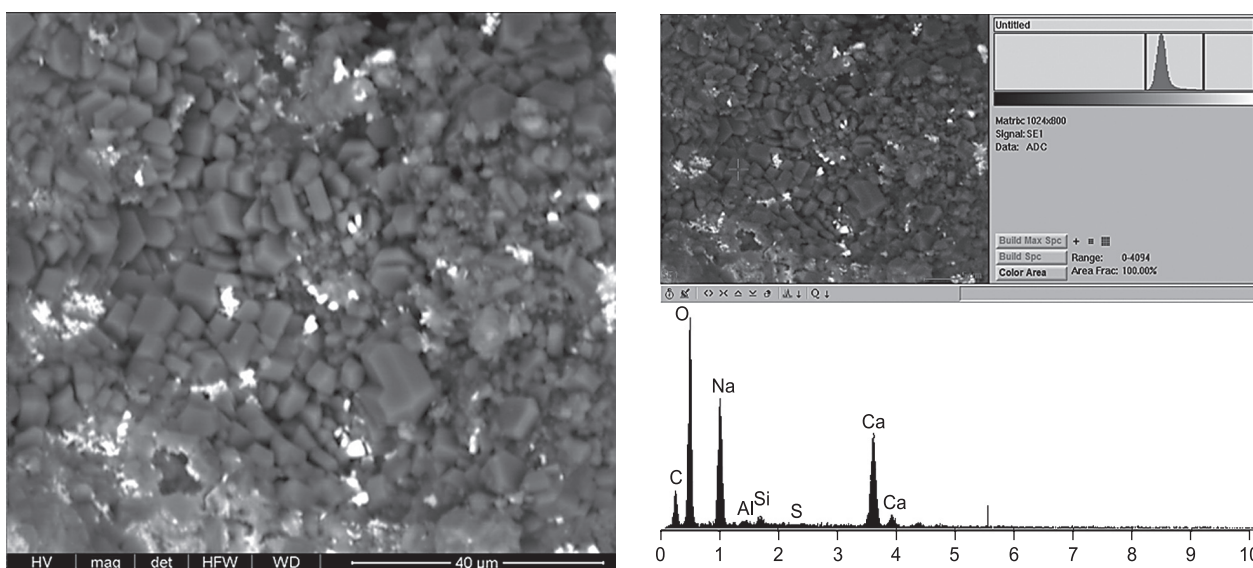
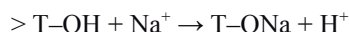


Figure 5. SEM micrograph of the 90 days cured WCS mixes having 6 % NH activator (water/binder of 0.25 %).

geopolymeric systems, are specifically adsorbed ions on the surface of WCS particles changing the surface speciation according to the chemical equation:



The stability area of  $>T-ONa$  surface species is pH dependent and is located at extremely high pH values, that is referred to the aluminol groups [26]. The sodium cations adsorption in highly alkaline conditions consumes the surface species ( $>T-OH$  and  $>T-O^-$ ), on which the chemical bonding between the insoluble solid particles and the geopolymeric framework takes place in the final stage of the geopolymerization process. Thus, the resulted geopolymeric materials have low mechanical strength.

The results of compressive strength for hardened geopolymer mixes along with variation in activators ratio from NH: NS, cured at 100 % relative humidity and at 38°C for 90 days are shown in Figure 6. Where, the strength increases with age due to slag hydration as mentioned latter. Whilst, the strength increases with the activator ratio from 3:3 to 1:1 sodium hydroxide to sodium silicate, respectively. Where 3:3 gives a compressive strength of 84.79 MPa at 90 days which is almost near that of 6 % NH. Reflecting that, the highest degree of geopolymerization can be achieved by using either 6% NH or equal ratio from both sodium hydroxides to sodium silicate (3:3). Also, latter strength is mostly be doubled that reference cement mortar even the cement content was increased to be 2:1 (sand: cement) as indicated in Figure 7.

An increase in  $Na_2SiO_3$  ratio results in an increase of Na content in the mixtures which in turn produces more stable geopolymer structure with high strength properties. Rapid strength development occurs within geopolymer with higher sodium concentrations especially within the  $Na_2SiO_3$  [27]. However, the compressive strength is expected to decrease as more silicate is

added into the system. This is because excess sodium silicate hinders water evaporation and structure formation [28].

Soluble silicate is an essential factor of geopolymerisation process. It provides the aqueous phase of the geopolymeric system with soluble silicate species, which are necessary for the initiation of oligomers (monomers, dimmers, trimers and tetramers) formation and accordingly, for the polycondensation. Increase of  $SiO_2$  to  $Na_2O$  ratio caused by the addition of soluble sodium silicates in the aqueous phase of the geopolymeric system has as a result the gradually shift of the chemical system from the monosilicate, chains and cyclic trimers to species with larger rings and then to complex structures and polymers giving rise to the three dimensional polymeric framework and consequently, increasing the mechanical properties of the resulted geopolymeric materials[29].

Whilst, upon microscopic scan of 90 days cured alkali activated slag specimens using 3:3 sodium hydroxide: sodium silicate, respectively, as represented in Figure 8, there is a high efficiency of the used mix in dissolving the slag network forming a homogeneous structure with high geopolymer content as compared with other dosage of the activators. While the sodium content is not too much as that of 6 % NH from EDX analysis which has a negative effect in consuming the surface as mentioned before.

#### Sulfate attack on water cooled slag geopolymer

The results of magnesium sulfate attack for WCS pastes using different alkali activators are represented in Figure 9. Where, the strength decreases with immersion time up to 6 month. Also, the pattern of compressive strength reflects the higher efficiency of sodium hydroxide and sodium silicate (3:3) than other dosages

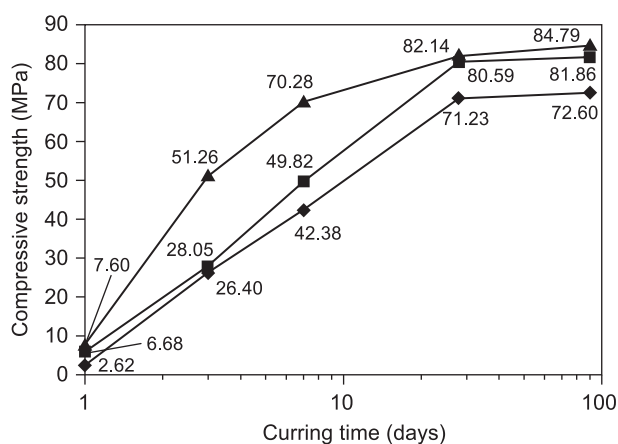


Figure 6. Compressive strength of the WCS mixes using different ratios of NH:NS, at different hydration ages (water/binder of 0.25 %); ◆ - 1 % NH + 1 % NS, ■ - 2 % NH + 2 % NS, ▲ - 3 % NH + 3 % NS.

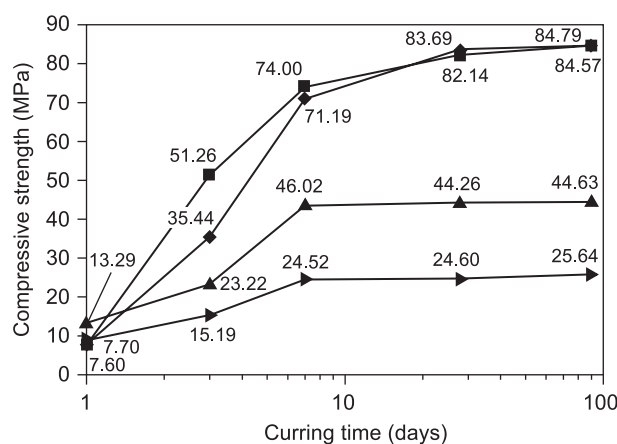
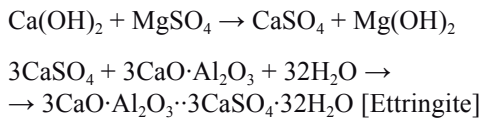


Figure 7. Compressive strength of the alkali activated WCS and cement mortar pastes at different hydration ages (WCS: ◆ - 6 % NH, ■ - 3 % NH + 3 % NS; Mortar: ▲ - 1Cement:2Sand, ▼ - 1Cement:3Sand).

of sodium hydroxide in resisting the effect of sulfate solution. Even as mentioned latter, the strength of 6 % NH that cured at normal curing condition (38°C, 100 % R.H.) almost near that of mixed ratio of both especially at later age.

It is known that the effect of sulfate species lies on the formation of ettringite by interaction of liberated lime from hydration reaction which in turn can increase the internal stress by increasing the volume of the slag structure leading to an expansion of tricalcium aluminates by 227% from its original volume of aluminates structure [30] as represented from the following equations:



Moreover, Mg(OH)<sub>2</sub> can on the other hand cause destabilization of hydration materials by the lower alkalinity provided by brucite formation during exposure to a high concentration of magnesium sulfate solution and will eventually decompose to gypsum.

The strength reduction ratios after 6 month of immersion relative to the strength of 28 days cured specimens are 52, 49 and 21 % upon using 2 % NH, 6 % NH and sodium hydroxide: sodium silicate (3:3), respectively, which reflects the lower reduction in strength for the mixed activator as compared with other dosages and so indicating the higher limit of geopolymer condensation and stabilization for the latter mix.

The scanning electron microscope images of 2 month immersed alkali activated WCS specimens are shown in Figure 10. In case of 2 % NH, the available hydroxide ions are not enough for the ettringite formation

by interaction with calcium sulfate as represented in Figure 10a where the ettringite needle is so little but the geopolymeric structure is not homogeneous and dense as the pH is not enough for completely dissolve the slag network. On the other hand, using of 6 % NH leads to the formation of excess hydroxide ions that in turn form excess ettringite that may increase with time leading to higher internal stress (Figure 10). While, using of mixed sodium hydroxide: sodium silicate (3:3) showed the higher resistance to ettringite formation and stability of geopolymer structure as compared with other mixes (Figure 10c). As an increase in Na<sub>2</sub>SiO<sub>3</sub> ratio results in an increase of Na content in the mixtures which in turn produces more stable with high strength properties.

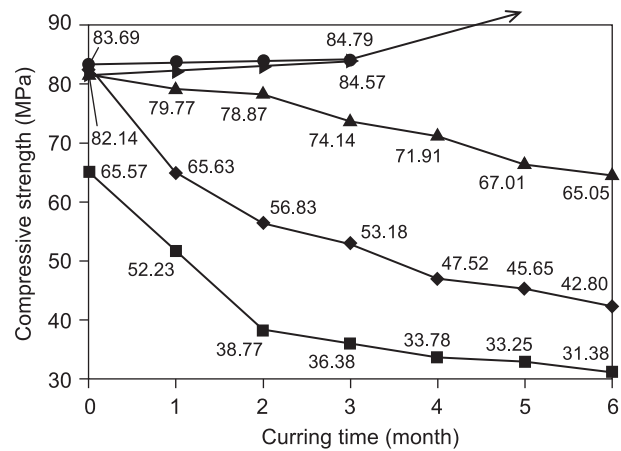


Figure 9. Compressive strength of the WCS mixes immersed in 5 % MgSO<sub>4</sub> solution using different ratios of NH:NS, at different hydration ages (water/binder of 0.25 %); S.A.\*WCS: ◆ - 6 % NH, ■ - 2 % NH, ▲ - 3 % NH + 3 % NS; WCS: ● - 6 % NS, ▴ - 3 % NH + 3 % NS.

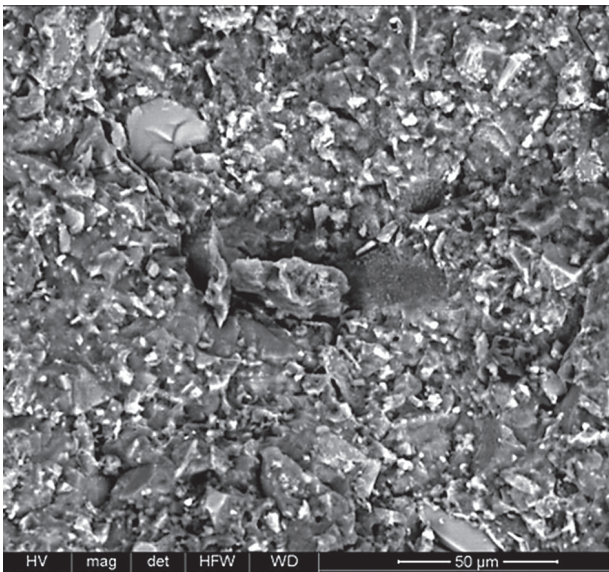
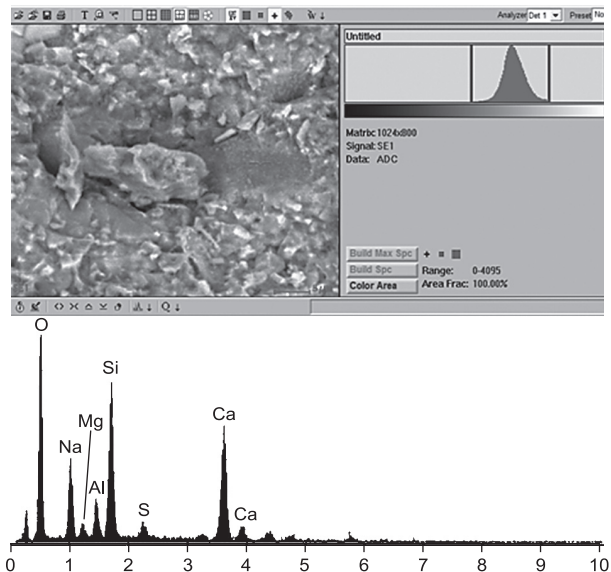
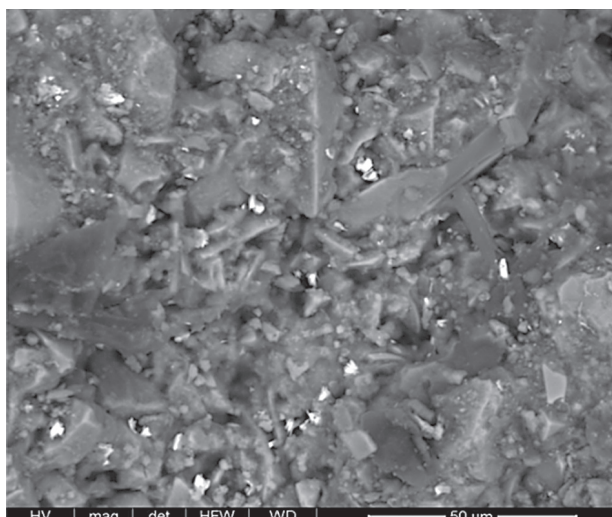
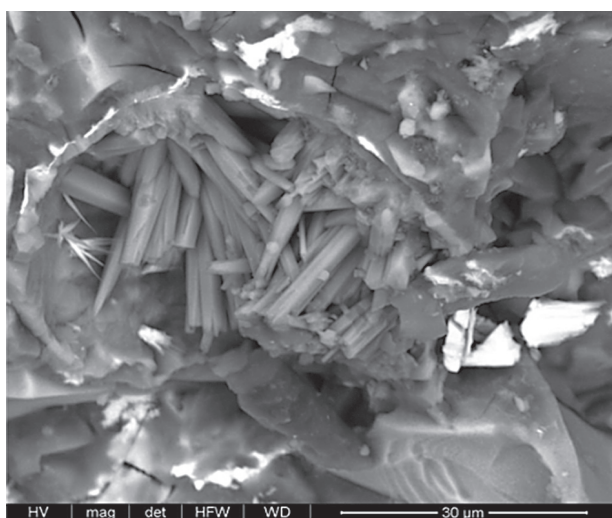


Figure 8. SEM micrograph of the 90 days cured WCS mixes having 3 % NH: 3 %NS activator (water/binder of 0.25 %).

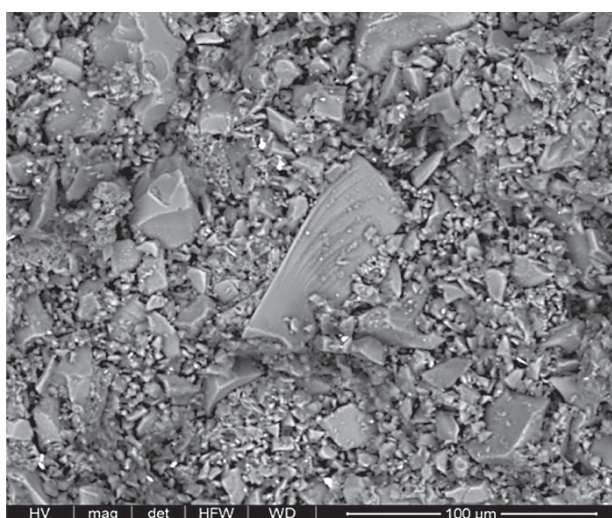




a)



b)



c)

Figure 10. SEM micrograph of the WCS mixes immersed in 5 %  $MgSO_4$  cured for 2 month (water/binder of 0.25 %) using: a) 2 % NaOH, b) 6 % NaOH and c) NH: NS (3:3).

## CONCLUSIONS

1. The increased NaOH concentration in the aqueous phase of geopolymeric systems causes positive effects on the mechanical properties of the geopolymeric materials. The reduced concentrations have negative impact. Thus, a compromise of the NaOH concentration in the aqueous phase is claimed.
2. Sodium silicate has lower efficiency in dissolving alumino-silicate network resulting in low mechanical properties.
3. Activation using sodium hydroxide and sodium silicate together has better performance as compared to other activator mixes, thus increasing the mechanical properties of the resulted geopolymeric materials. The ratio of NaOH to  $Na_2SiO_3$  of 3:3 by weight gives a compressive strength of 84.79 MPa at 90 days which is almost near that of 6 % NH.
4. Sodium hydroxide: sodium silicate (3:3 by weight) activator showed superior resistance to sulfate attack as compared with other mixes. Strength reduction ratios after 6 month of immersion relative to the strength of 28 days cured specimens are 52, 49 and 21 % upon using 2 % NH, 6 % NH and sodium hydroxide: sodium silicate (3:3).
5. Other durability testing (e.g., acid resistance, fire resistance, alkali-aggregate reaction) are currently going for assuring that WCS 3:3 (NH:NS) activator is optimum geopolymer mix.

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