Effect of NaOH Molarity on the Strength and Microstructure of Natural Pozzolan-Based AAC

Mohammed Ibrahim[1], Megat Azmi Johari[1], Mohammad Kalimur Rahman[2*], Mohammed Maslehuddin[2] , and Mohamed Hatim Dafalla[2]

[1]Universiti Sains Malaysia, Engineering Campus, 14300 Nibong Tebal, Pinang, Malaysia
[2]Center for Engineering Research, Research Institute, KFUPM, Dhahran 31261, Saudi Arabia

Abstract. Alarming levels of greenhouse gas emissions has triggered change in the mode of direction of building material research. In this pursuit, alkali activated binders (AAB), synthesized by activation of industrial by products and natural materials in the presence of highly alkaline solutions, have offered viable alternative to OPC. However, there are quite a number of variables which controls the properties of these binders. Among these parameters, SS/SH ratio and molarity of sodium hydroxide solution plays a very important role in the development of these binders. Therefore, this research investigates the effect of SS/SH ratio, subsequently, molarity of SH solution on the properties of natural pozzolan based AAB. The NP was activated with sodium silicate to sodium hydroxide ratio (SS/SH) between 2.0 to 2.75. Subsequently, the molarity of SH solution was varied between 8 to 14. The development in strength was monitored on the specimens cured at 60 °C. SEM and EDS techniques were used to determine the nature of the binder formed during alkali activation. The results have shown that SS/SH ratio of 2.5 and 14 molar SH solution resulted in higher strength and finer microstructure as compared to others. Also, it was understood that there exists a suitable silica modulus of combined activator which results in higher polymerization.

1 Introduction

Currently world is facing a challenging situation due to global warming triggered by increase in the greenhouse gas emissions. The stringent conditions are being imposed by the global community to limit the greenhouse gas emissions prompted the building material research to focus on developing new class of alternative binders to ordinary Portland cement (OPC). In this search, alternative binders such as, calcium sulfoaluminate cements [1], magnesium based-cement binders [2] and alkali activated binders (AAB) [3] have been developed. Among these, AAB are having promising future as they are synthesized utilizing industrial by-products and natural materials. By effective utilization of these source materials in developing AAB, the problems such as greenhouse gas emissions due to manufacturing of OPC and accumulation of industrial waste could be efficiently tackled.

Corresponding author: mkrahman@kfupm.edu.sa

Industrial by-products including; fly ash, ground granulated blast furnace slag (GGBFS) and palm oil fuel ash (POFA) have been extensively utilized to synthesize AAB [4-6]. The factors effecting the properties of these binders such as alkaline activator composition and concentration, curing conditions and duration and incorporation of mineral additives including; aluminum hydroxide, lime, and silica fume have been studied. Majority of AAB developed were cured at temperatures ranging from 40 °C to 80 °C [7-9]. Natural pozzolan, an aluminosilicate precursor material has great potential to be utilized as starting material in synthesizing AAB. The composition of alkaline activators and their concentration plays a very important role in synthesizing AAB, which is the subject of this research endeavor. Therefore, in this study the effect of parameters such as SS/SH ratio and their concentration on the mechanism of strength development, enhancement in microstructural characteristics as well as nature of the binder so formed was investigated in detail. It is envisaged that the results obtained would be beneficial in understanding the behavior and practical application of the end product.

2 Materials and Methods

Chemical composition of natural pozzolan is given in Table 1. The NP used in the study was ground form of basaltic rock from red sea coast of Saudi Arabia. The sustainability of the source material is good as it is abundantly available covering at least 90, 000 km^2 in the western region of Saudi Arabia. The locally procured NP confirms to the physical and chemical requirements of ASTM C618. The specific surface area and average particle size of NP used are 442 m^2/kg and 30 μm, respectively. The alkaline activators used were a combination of aqueous industrial grade sodium silicate (SS) solution in combination with 8 M, 12 M and 14 M sodium hydroxide (SH) solution. The silica modulus of sodium silicate was 3.3 and its composition includes; H_2O: 62.50%, SiO_2: 28.75% and Na_2O: 8.75%.

In order to find suitable SS/SH ratio, AAC mixes were prepared with SS/SH of 2.0, 2.25, 2.5 and 2.75 with constant 14 M sodium hydroxide. Table 2 gives the constituent materials used to prepare these concrete specimens. Subsequently, depending on the compressive strength results obtained, a suitable SS/SH was used to prepare two different concrete mixes, one with 8 M and other having 12 M sodium hydroxide solution in order to determine the effect of molarity on the strength and microstructure of concrete. Table 3 summarizes the constituent materials for preparing the AAC specimens with varying SH molarity. The coarse aggregate to total aggregate and fine aggregate to total aggregate ratios were 0.65 and 0.35, respectively. The concrete specimens prepared after demolding were placed in the plastic bags and kept in the oven maintained at 60 °C. Compressive strength was measured after ½, 1, 3, 7, 14 and 28 days of curing. Samples retrieved from middle portions of AAP after 7 days of curing were utilized to study the morphology and mineralogy of the developed binder. JEOL scanning electron microscope fitted with energy dispersive spectroscope (SEM+EDS) model 5800 LV was used to evaluate the morphology and elemental compositions of the AAP. Micrographs were collected at 20 kV using secondary mode. The XRD was conducted using a Bruker instrument model d2-Phaser. The test was conducted at a scan rate of 2.5 °/min using Cu-Kα radiation (40 kV, 40 mA) through continuous scanning within the angle 2-theta range of 0–80°.

Table 1: Chemical compositions of NP.

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	LOI
weight, %	40.48	12.90	17.62	11.83	8.33	1.67	3.60	1.37	1.6

Table 3. Constituent materials used to prepare AAC mixtures.

Mix#	Binder Content, kg/m^3	Sodium Silicate (SS), kg/m^3	Sodium Hydroxide (SH), kg/m^3	Total Water, kg/m^3	Total Alkaline Activator, kg/m^3	SS/SH Ratio	Fine Aggregate (FA), kg/m^3	Coarse Aggregate (CA), kg/m^3
M1	400	140	70	139.22	210	2.00	650	1206
M2	400	145	65	139.37	210	2.25	649	1207
M3	400	150	60	139.51	210	2.50	650	1206
M4	400	154	56	139.63	210	2.75	650	1207

Table 4. Constituent materials of AAC mixes prepared with varying SH molarity.

Trial Mix#	Natural pozzolan (NP), kg/m^3	Sodium Silicate (SS), kg/m^3	Sodium Hydroxide (SH), kg/m^3	Total Alkaline Activator, kg/m^3	SS/SH, Ratio	Fine Aggregate (FA), kg/m^3	Coarse Aggregate (CA), kg/m^3
M0-8M	400	150	60	210	2.50	643	1195
M0-12M	400	150	60	210	2.50	645	1198
M0-14M	400	150	60	210	2.50	650	1206

3 Results and Discussion

3.1 Compressive Strength

3.1.1 ***Effect of Alkaline Activator Composition on the Compressive Strength***

Evolution of compressive strength of concrete prepared by varying SS/SH ratio from 2.0 to 2.75 with an incremental value of 0.25 is given in Figure 1. Generally, there was steady increase in strength with the duration of curing up to 7 days in all the mixes prepared. Since the onset of curing higher strength was recorded in the concrete mix synthesized using SS/SH ratio of 2.5. For instance, at 12 hours of curing it was 9.59, 11.17, 12.57 and 9.68 MPa for the mixes prepared with SS/SH ratio of 2.0, 2.25, 2.5 and 2.75, respectively. The strength development was continuously higher, as the curing progressed, in the concrete mix prepared with SS/SH ratio of 2.5 in relation to other mixes. At the end of 7 days curing, compressive strength recorded in the mixes prepared with SS/SH ratio of 2.0, 2.25, 2.5 and 2.75 was, respectively, 35.22, 36.72, 37.52 and 36.48 MPa. After 7 days of curing there was marginal reduction in the compressive strength in all the mixes.

According to the results of compressive strength, it is evident that there exist a suitable composition of alkaline activator and combined silica modulus which results in better strength for a given binder content and type of precursor material as also demonstrated in Figure 2. Combined silica modulus of alkaline activator of about 1.35 in developing NP-based AAC appears to be suitable beyond which there could be reduction in strength. An increase in SS/SH ratio beyond 2.5, slowed down strength development and took lengthier period of curing to achieve ultimate strength. This is largely due to the excess silica modulus for a given binder content, which delayed the polymerization process [10].

Therefore, a SS/SH ratio of 2.5 by weight and 7 days curing was found to be suitable for achieving superior strength.

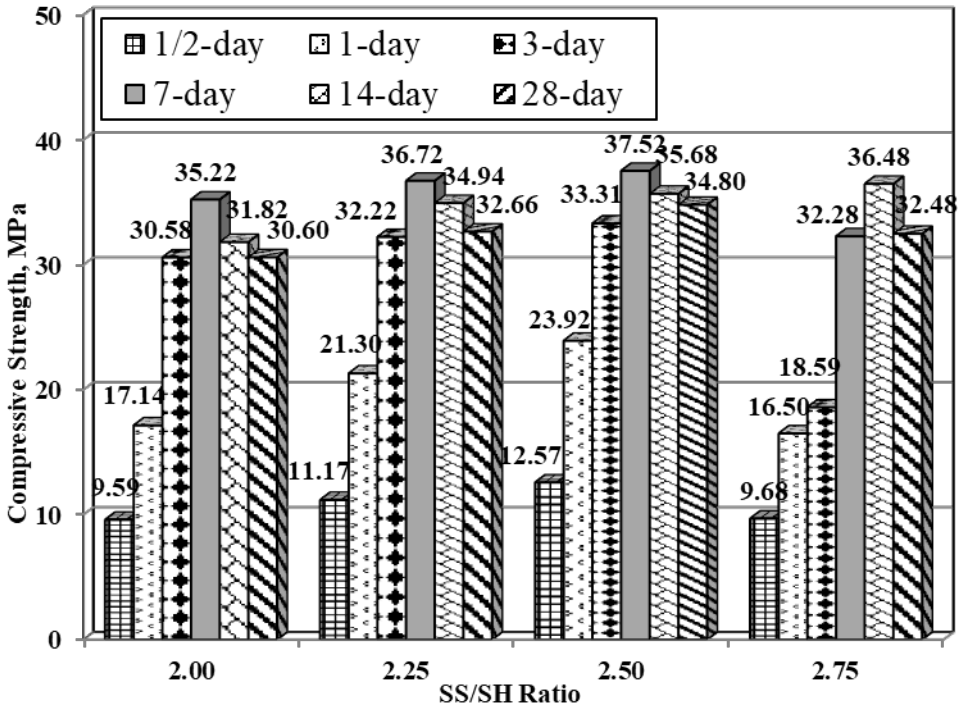


Fig. 1. Compressive strength of concrete prepared with varying SS/SH ratio.

3.1.2 Effect of NaOH molarity on the Compressive Strength

Figure 3 shows the compressive strength development in the concrete specimens prepared with different concentrations of NaOH solution having same SS/SH ratio of 2.5. Compressive strength development was steady as the curing progressed up to 7 days of curing in all the concrete mixes. The strength gain at the onset of curing in all the mixes was more or less similar. However, as the curing continued strength development was more in the specimens prepared with higher molarity of SH solution as compared to the lower ones. For instance, at the completion of 7 days of curing, compressive strength recorded in the specimens having 14 molar SH solution was 37.52 MPa as compared to that of 30.45 MPa in 8 molar. Further, concentration of 12 molar solution resulted in moderate results. The increase in molarity of NaOH solution increases solubility of Si from the precursor materials due to the presence of higher hydroxyl ions for a given binder content. Also, it is expected that the solubility of Al in four-fold reaction that was required to accommodate Si would have accelerated thereby permitting the formation of Si-O-Al which perhaps resulted in increase in strength at higher NaOH molarity due to the formation of C-A-S-H together with the pure polymeric gel as the reaction products [11-13].

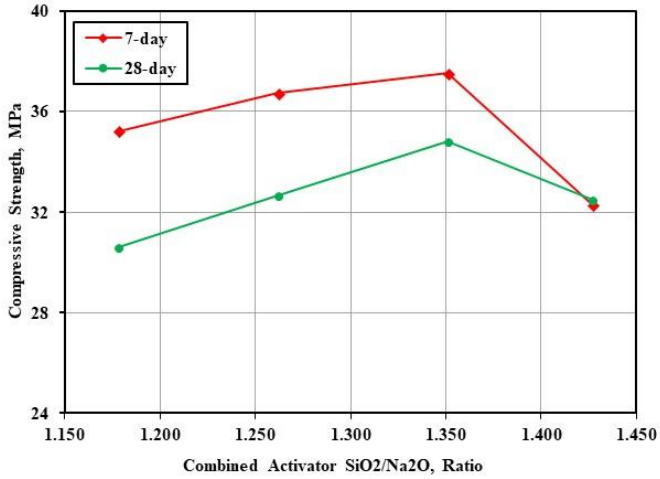


Fig. 2. Variation in compressive strength of concrete prepared with variable silica modulus.

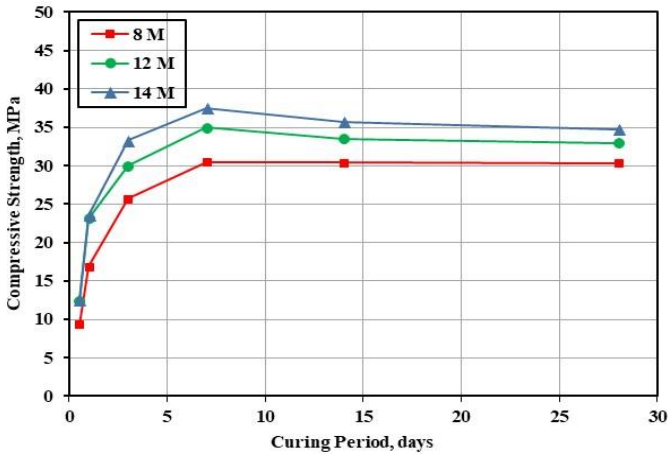
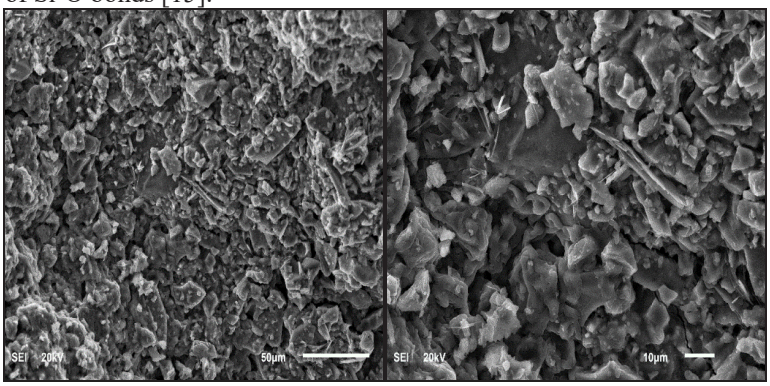


Fig. 3. Evolution of compressive strength of AAC with varying SH molarity.

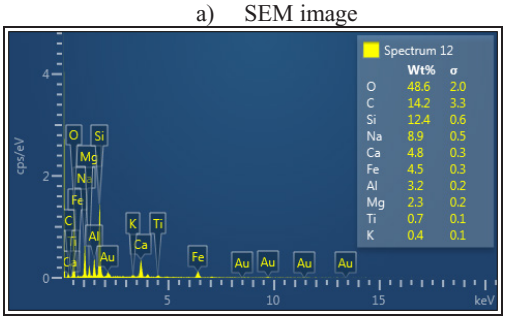
3.3 Microstructural Characteristics

Figure 4, 5 and 6 shows the SEM/EDS of AAP prepared with 8 M, 12 M and 14 M NaOH, respectively. The microstructure of 14 M NaOH (Figure 6) was homogenous, relatively compact and partially denser as compared to the one prepared with lower concentrations of NaOH solution. In the micrograph of 8 M NaOH AAP (Figure 4), it is clearly appearing that there were unreacted binder particles in the matrix because of which the structure looks coarser and non-homogenous. However, the microstructure of AAP having 12 M NaOH (Figure 5) was moderately compact without excessive unreacted binder particles. These results corroborate the higher degree of polymerization in the concentrated alkali activators than the lower ones. The EDS shows the presence of Al and greater proportion of Ca in the AAP having 12 and 14 molar NaOH. These are the backbone of any alkali activated binders. Because of the presence of these elements, there is strong possibility that the structure of the gel consists of C-A-S-H or C/N-A-S-H as the reaction product [14]. However, in the AAP with 8 molar NaOH, Ca was found to be marginal because of which

formation of C-A-S-H products was also minimal. Apart from this, Ca/Si and Ca/Na ratios were high in the pastes prepared with 12 and 14 molar NaOH solutions which enhances formation of Si-O bonds [15].

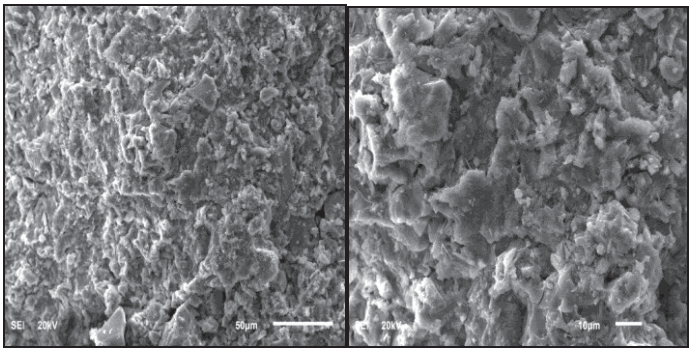


a) SEM image

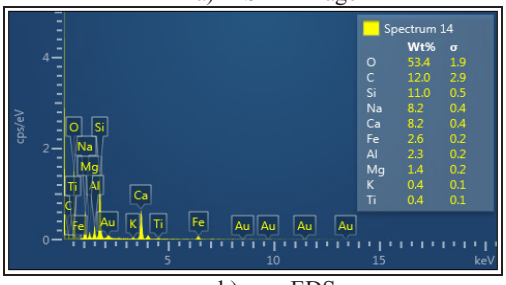


b) EDS

Fig. 4. SEM/EDS of AAP prepared with SS/SH: 2.5 and 8 M NaOH solution.

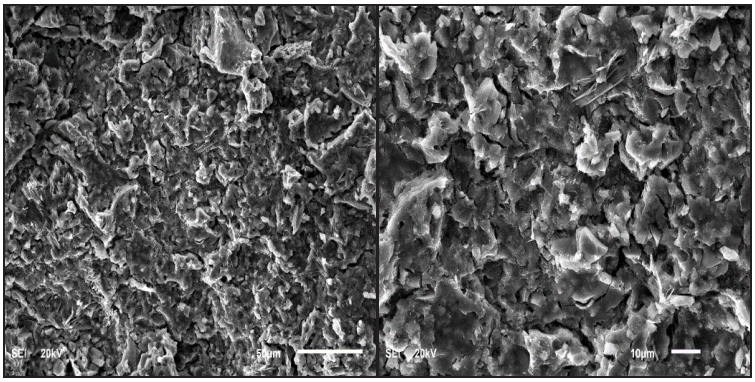


a) SEM image

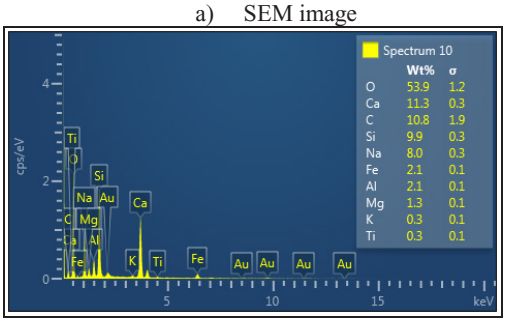


b) EDS

Fig. 5. SEM/EDS of AAP prepared with SS/SH: 2.5 and 12 M NaOH solution.



a) SEM image



b) EDS

Fig. 6. SEM/EDS of AAP prepared with SS/SH: 2.5 and 14 M NaOH solution.

4 Conclusions

The aim of the study was to find out the suitable alkaline activator composition and concentration in synthesizing AAC utilizing natural pozzolan as the precursor material for a particular binder content. The results of the study have shown that about SS/SH ratio of 2.5 and 14 molar NaOH solution is suitable in developing AAC. The higher NaOH concentration triggers the formation of additional C-A-S-H which enhances the strength and microstructure of the binder.

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