

Improving The Microstructure And The Strength Of Alkali Activated Slag Mortar Under Ambient Temperature

A. Maher El-Tair, Ramez Bakheet, M.S. El-Feky, Mohamed Kohail, Mohamed I. Serag

Abstract: Alternative building materials for sustainable and eco-friendly development are now a major environmental issue in the construction industry. Over the last decades, Alkali Activated Material (AAM) has shown a remarkable development as an alternative material to ordinary cement-based concrete. The AAM uses an alumino-silicate powder as a total replacement of cement. The reaction between a source of alumino-silicate and alkaline solution, under thermal treatment, to form a binding material have the same product resulting from the reaction of ordinary cement and water in concrete. In this paper, the effect of different percentages of Na_2O and the activator modulus and water to binder ratio on the fresh and mechanical properties of mortar. Fresh properties were studied through studying the temperature, pH, and mortar flow test. While the mechanical properties were examined through compressive strength. Also, a microstructure analysis was examined through Scanning Electron Microscopy (SEM) and X-Ray Diffraction analysis (XRD). The results showed that increasing the percentage of Na_2O % and Ms and results in an increase in the compressive strength of Alkali Activated Slag (AAS) to a certain limit, exceeding these limits lead to undesirable results with respect to strength.

Index Terms: Alkali Activated Slag, Ground granulated blast furnace slag, GGBFS, Compressive Strength, pH, SEM, Temperature.

1. INTRODUCTION

Reducing the carbon dioxide emission has been a global environmental issue, recently and via the nano technology, the reduction of cement contents has been extensively studied [1-7]. but unfortunately, other costs have been added to the process due to the difficulty of the nano components utilization including but not limited to the dispersion techniques as the ultra sound sonicators, surfactants, and homogenizers [8-16]. Nowadays, Alkali Activated Materials (AAM) consider one of the sustainable cementing binding materials [17-19]. Some references named AAM as Geopolymers [20-23]. AAM can be produced from different aluminosilicate resources, which are differ in their availability, reactivity, and cost. The local availability of the sources of AAM is the main governing discussing point to use AAM. As, most of the elements used in the production of AAM are also used in the production of ordinary Portland cement [24-26]. On the other hand, AAM sometimes can be relatively inexpensive option compared to using ordinary Portland cement, especially in the sites which are relatively remote and requires Portland cement to be imported [21]. In addition to, the source of the activator can be also a challenging issue in some areas. Alkali Activated Materials (AAM), is produced as a result of the reaction of aluminosilicate in addition to the alkali activator, to produce a binder. There are two different methods to produce alkali-activated materials; the first method called a one-part mix, in which dry powder combined with water. While for the second method, which is the most commonly used in the market, can

be called two-part mix or liquid activator system [22]. Alkali activated materials, can be used with blast furnace slag, coal fly ash, calcined clays, and natural pozzolans to give good results [29-33]. Blast furnace slag is a by-product generated during manufacturing of pig iron and steel and may be defined according to ACI-116R [34] as “nonmetallic product consisting essentially of calcium silicates and other bases that is developed in a molten condition simultaneously with iron in a blast furnace”. It consists primarily of silicates, alumina-silicate and calcium-alumina-silicates. The production of different types of slag with different physical and pozzolanic characterizes, mainly, depends on the cooling system [35-37]. Granulated blast furnace slag is one of the supplementary materials for cement, in addition to, it can be also used as a cementitious material for different construction applications. Many researchers have shown the possibility of replacing 100 % of cement by the slag as a sole binder in mortar by activating them with an alkali [38-41]. The main function of the alkali activators is to provide the matrix with OH^- ions in the first stage of the reaction and to react later in the next stage [35]. Alkali had the same function in both activating slag and in blended GGBS-OPC cement, which provide the supply of OH^- anions in the system [36]. Researchers also reported that the hydrated calcium silicate gel, namely C-S-H, is the most abundant product in hardened alkali-activated slag pastes [24, 28, 35, 36]. It was found that the strength and the mechanical properties of alkali activated slag functions of the Na_2O percentage and silicate modulus [42-49].

This paper investigates the effect of Na_2O percentage, activator modulus (Ms), and water to binder ratio (W/B) on the production of Ground Granulated Blast Furnace Slag (GGBFS) mortar. Fresh properties (Temperature, pH and Flow) and mechanical properties (compressive strength) has been investigated. Scanning Electron Microscopy (SEM) and X-ray Diffraction analysis (XRD) was conducted to examine the changes in the microstructure of the paste.

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2 EXPERIMENTAL

2.1 Material

The alkali-activated slag mortars used in this study were made of granulated blast furnace slag and a solution of Sodium silicate and Sodium Hydroxide. A locally produced Ground granulated blast furnace slag (GGBFS) was used in this research. Table 1 shows the chemical composition of slag. A solution of sodium hydroxide and sodium silicate was used to activate the slag. Sodium silicate was used as a solution, while sodium hydroxide as solid particles soluble in water. The mass ratio of SiO_2 to Na_2O in the sodium silicate solution was 2.58. Table 2 shows the chemical composition of both sodium silicate and sodium hydroxide. Natural clean sand with particles size smaller than 5 mm, with specific gravity 2.65 while the fineness modulus of 2.25 was used as fine aggregate.

2.2 Mix design, preparation of samples, and curing

Prior to the preparation of the mortar mixture, alkali solution of the desired concentrations was prepared at the room temperature. Three Na_2O concentration (8, 9.5, and 11 %) by weight of total binder and four activator modulus Ms ($\text{SiO}_2/\text{Na}_2\text{O}$) ratios (0.8, 1.0, 1.2, and 1.4) were prepared. Two water to binder ratios (0.25 and 0.30) were also prepared. All mixtures were prepared by mixing the liquid sodium silicate and sodium hydroxide flakes with water in a laboratory beaker until all NaOH flakes dissolved. The temperature and the pH of solution were measured immediately after the mixing, then the solution was added to the binder and mixed for 2 mins. $5 \times 5 \times 5$ cm cubes were used to cast the mortar. The mixes were prepared with replacement of total amount of cement by grinded ground granulated blast furnace slag. The specimens were demolded after one day and cured in water at a temperature of 20°C to test days.

3 RESULT AND DISCUSSION

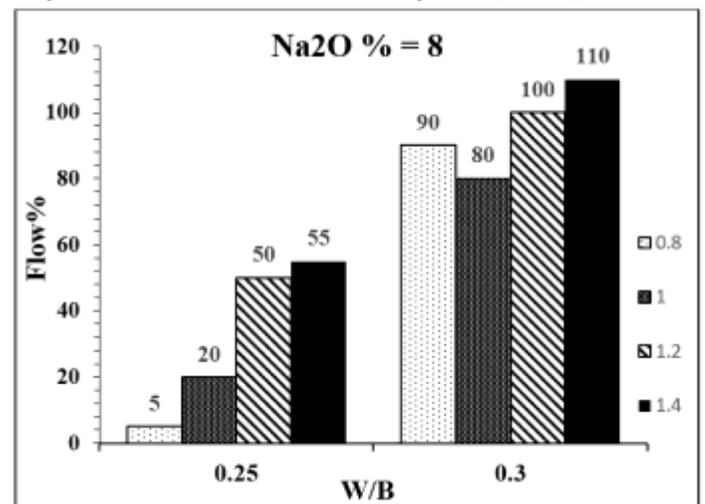
In the following part, fresh properties (flowability, temperature and pH), mechanical Properties (compressive strength) and microstructure properties (SEM and XRD) results will be discussed in order to investigate the effect of different percentages of Na_2O ($\text{Na}_2\text{O}\%$), different ratios of activator modulus ($M_s = \text{SiO}_2/\text{Na}_2\text{O}$), and different water to binder ratios (W/B) on the production of Cementless alkali activated slag.

3.1 Fresh Properties

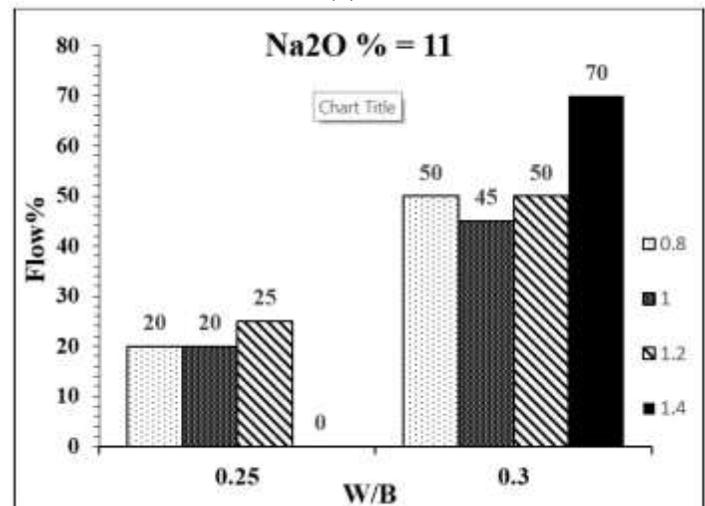
3.1.1 Flowability

The flowability test was performed according to ASTM C230-83 [50]. The results showed that increasing the M_s ratio from 0.8 to 1.4 results in increasing the workability of mixes regardless the percentage of $\text{Na}_2\text{O}\%$ due to the increase of the sodium silicate amount in alkaline activator solution which increases the workability and also decrease the sodium hydroxide amount [51]. The sodium silicate solution improves the dissolution of GGBFS in alkaline medium and more dissolved GGBFS results in higher workability. Increasing of sodium silicate to alkaline solution ratio results in the increase of the flow of GGBF slag-based geopolymer paste while keeping others parameters constant [52, 53]. On the other hand, increasing the Na_2O percentages will decrease the

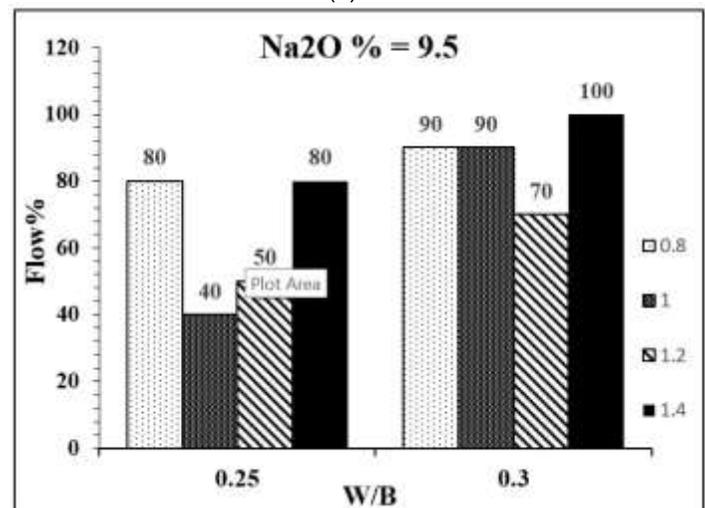
workability, and this can be attributed to decreasing the sodium silicate amount as shown in Fig (1) and this results was agreed with Memon et al. and Yang et al. [54, 55].



(a)



(b)



(c)

Figure 1. Effect of W/B ratio on the workability at different M_s ratio and at various percentage of $\text{Na}_2\text{O}\%$: (a) $\text{Na}_2\text{O} = 8$, (b) $\text{Na}_2\text{O} = 9.5$, (c) $\text{Na}_2\text{O} = 11$.

Table 1: Chemical Composition of GGBFS by XRF

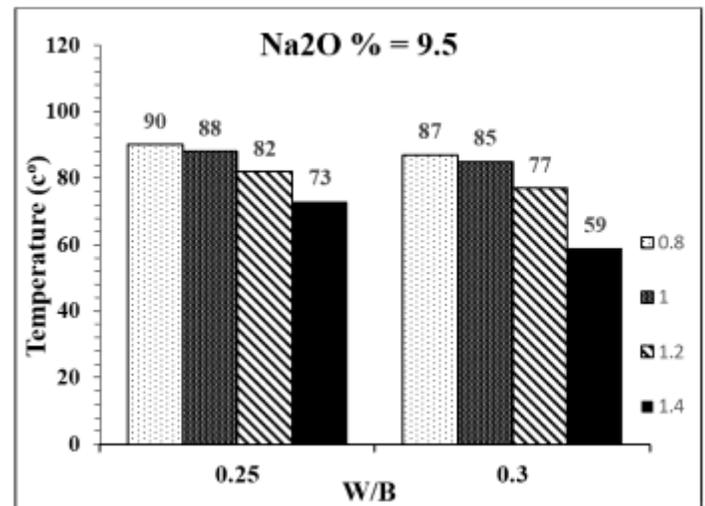
Constituents	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	SO ₂	TiO ₂	P ₂ O ₅	MnO	Cl	LOI
GGBFS	38.25	7.84	34.2	0.53	7.73	1.14	1.35	3.01	0.54	0.41	5.17	0.18	0.01

Table 2: Chemical Composition of Na₂SiO₃ and NaOH

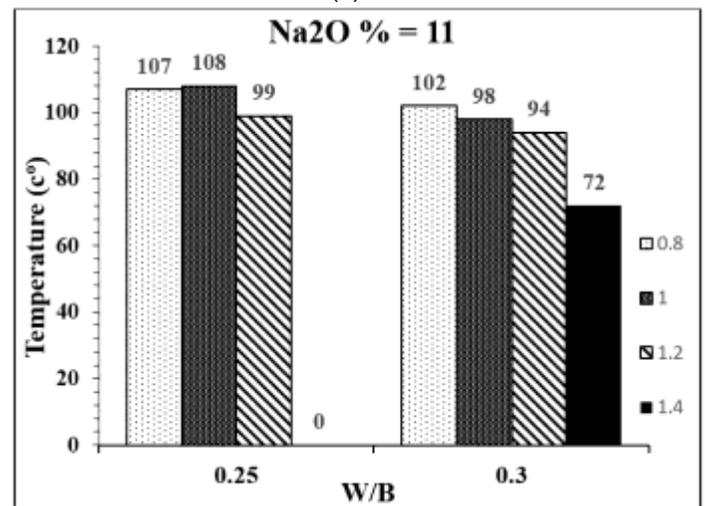
	SiO ₂	Na ₂ O	H ₂ O
Sodium Hydroxide (NaOH)	----	60.25%	39.75%
Sodium Silicate (Na ₂ SiO ₃)	31%	11.98%	57%

3.1.2 Temperature

The temperature was immediately measured after mixing both sodium hydroxide and sodium silicate with water and it was observed that increasing the w/b ratio from 0.25 to 0.30 a decrease in the temperature of all mixes was observed. This can be attributed to the increase of extra water which lower the solution temperature. Increasing Ms ratio from 0.8 to 1.4 results in decreasing the temperature of the alkaline solution, this could be due to increasing the Ms which results in increasing the sodium silicate amount in the alkaline solution in order to diluted the solution. While increasing the Na₂O percentage from 8% to 11% increases the temperature of the alkaline solution and this is due to the increasing of sodium hydroxide amount [56]. As shown in Fig (2).

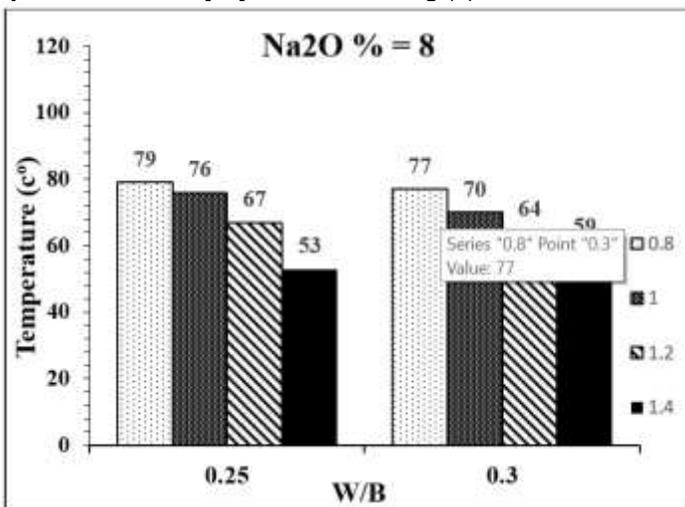


(b)



(c)

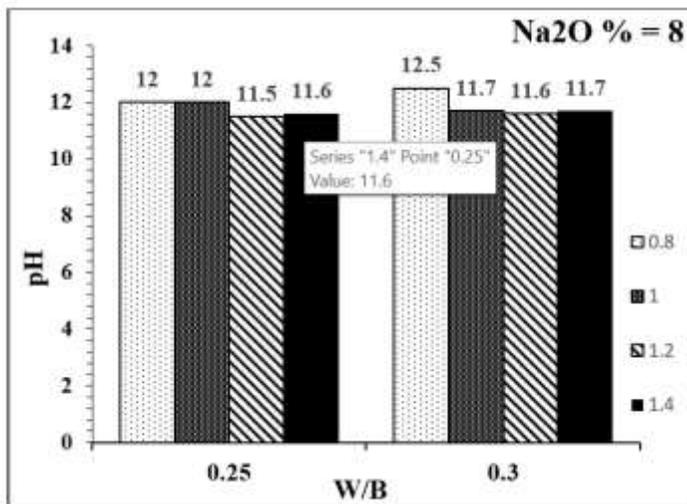
Figure 2. Effect of W/B on the solution temperature at different Ms ratio and at various percentage of Na₂O%: (a) Na₂O =8, (b) Na₂O= 9.5, (c) Na₂O =11.



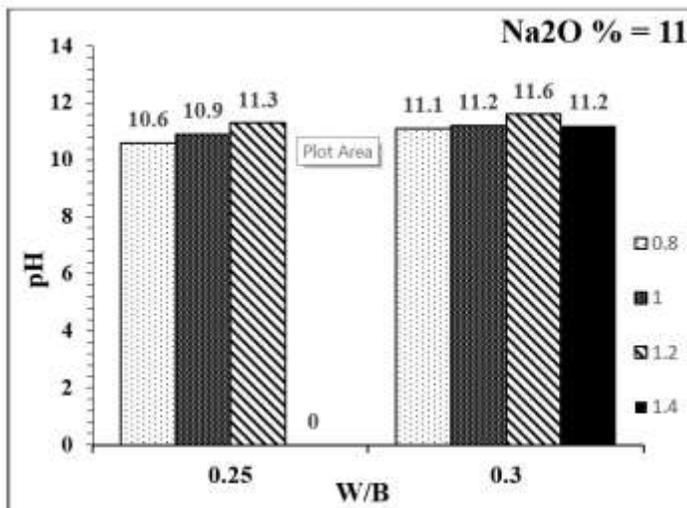
(a)

3.1.3 pH

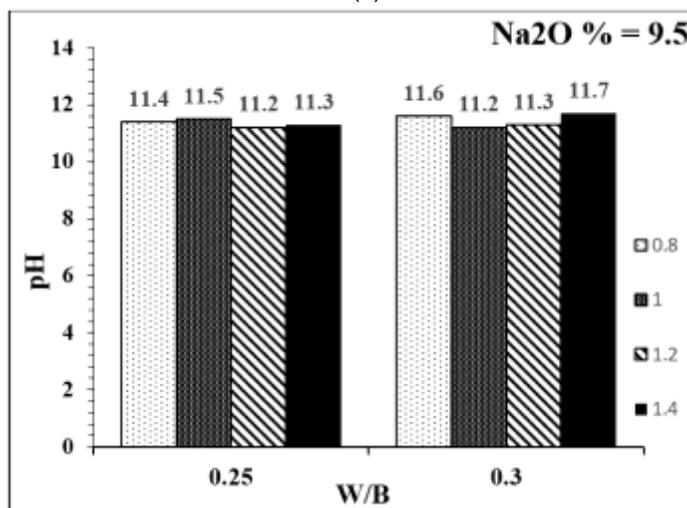
The pH values of solution showed that pH values of all mixes are relatively high, ranging between 10.6 and 12.5. For using Na₂O with a percentage 8% and 9.5%, by increasing the Ms ratios a decrease in the pH values for both w/b ratios was observed. On the other hand, when using Na₂O with a percentage of 11%, increasing the Ms ratios the pH value will increase directly. Where exceeding the percentage of Na₂O more than 9.5%, pH value will reduce to a value less than 11 and the temperature of the solution will exceed 100°C. The decrease in the pH value can be attributed to the raise of the temperature to more than 100 °C, and this was agreed with Delory et al. and Ashton et al. [57, 58]. From the data shown in Figure (3), W/B ratio had a minor effect on the pH values. This could be due to that the measurement of pH was immediately after mixing and there wasn't enough time to the dilution of solution.



(a)



(b)



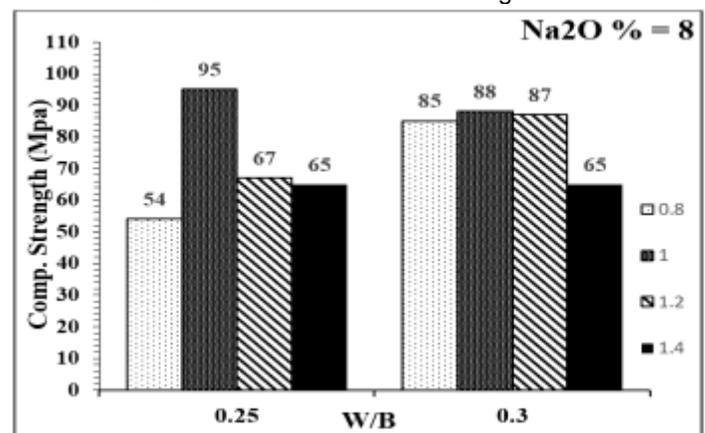
(c)

Figure 3. Effect of W/B on the solution pH at different Ms ratio and at various percentage of Na₂O%: (a) Na₂O =8, (b) Na₂O= 9.5, (c) Na₂O =11.

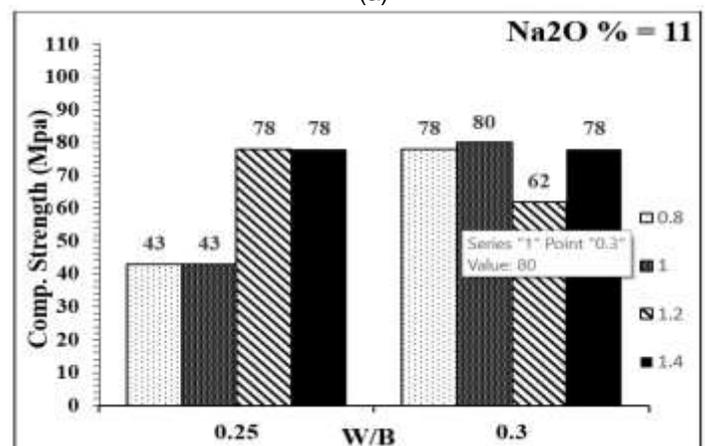
3.2 Compressive Strength

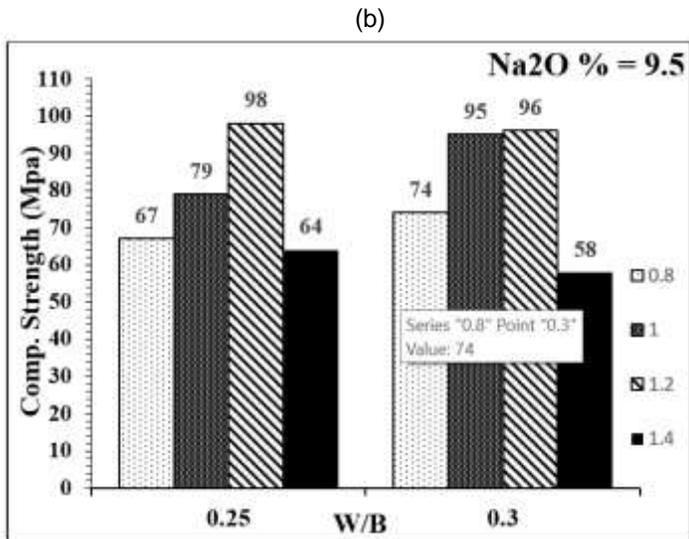
Generally, increasing Na₂O percentages increases the 28 days' compressive strength. Otherwise when Na₂O

percentages reach 11%, the compressive strength reduces than the other percentages. The same behavior observed by studying the effect of Ms ratio, where the 28 days' compressive strength increase by increasing the Ms ratio just to a certain and then it reduces, at both W/B ratios; 0.25 and 0.30, as shown in Figure 4. These results agreed with the results obtained by Aydin et al. and other researchers [51, 54, 59]. At Na₂O 8%, the maximum 28 days' compressive strength was obtained at Ms =1 at both W/B ratios, as shown in Figure (4-a). At which increasing W/B ratio the compressive strength decreased. This can be attributed to the fact that increasing the water content decreased the mix temperature, and consequently the temperature needed for sodium hydroxide to leach SiO₂ and CaO particles from the slag is lowered, these conclusions is agreed with Kang et al. [56]. While at Na₂O 9.5%, increasing Na₂O percentage from 8% to 9.5% increased the 28 days' compressive strength to reach its optimum strength 98 MPa and 96 MPa at W/B ratios 0.25 and 0.30, respectively with Ms ratio 1.2. This could be due to increasing the amount of Na₂O content leads to more leaching of CaO from slag particles and therefore, more silica ions produced. This conclusion was also agreed with kang et al. [60]. On the other hand, increasing Na₂O percentage to reach 9.5 leads to a reduction in the 28 days compressive strength and this was due to excess sodium oxide in the solution and this was also listed by Allahverdi et al. [61]. It was also noticed that increasing W/B ratio increase the 28-day compressive strength. This can be due to the high temperature occurred with W/B=0.25 which exceeded the 100 C° and this could lead to evaporate some water needed for activation process and also cause micro cracks due to heat shrinkage.



(a)





(c)
Figure 4. Effect of W/B on 28 days' compressive strength at different Ms ratio and at various percentage of Na₂O%: (a) Na₂O =8, (b) Na₂O= 9.5, (c) Na₂O =11.

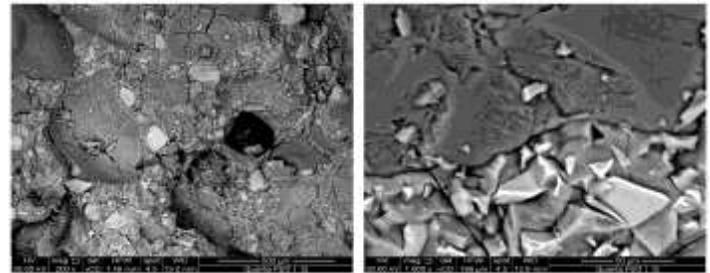


Figure 6. SEM micrograph of mix contain Na₂O= 8%, Ms=1.2 and w/b= 0.25.

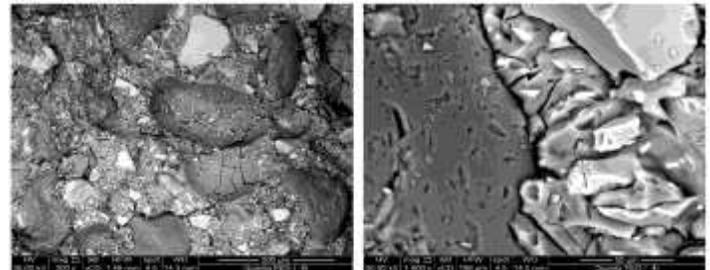


Figure 7. SEM micrograph of mix contain Na₂O= 9.5%, Ms=1 and w/b= 0.30.

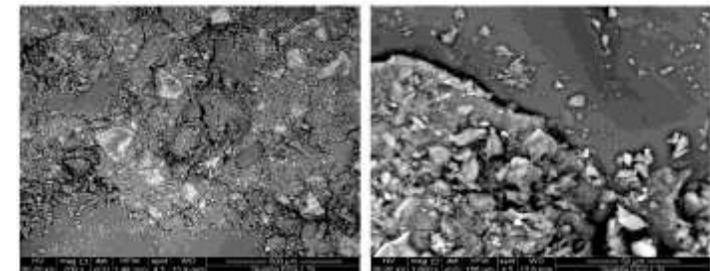


Figure 8. SEM micrograph of mix contain Na₂O= 9.5%, Ms=1.2 and w/b= 0.30.

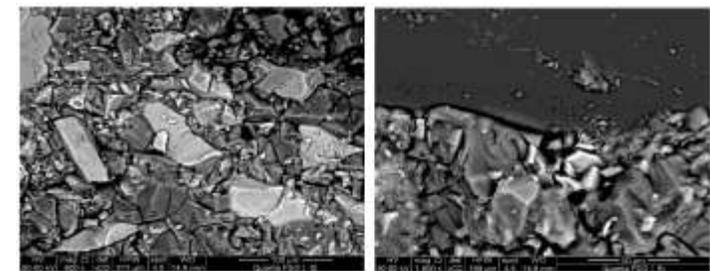


Figure 9. SEM micrograph of mix contain Na₂O= 9.5%, Ms=1.2 and w/b= 0.25.

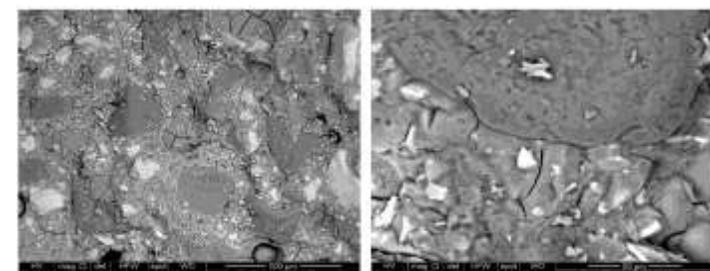


Figure 10. SEM micrograph of mix contain Na₂O= 11%, Ms=1 and w/b= 0.25.

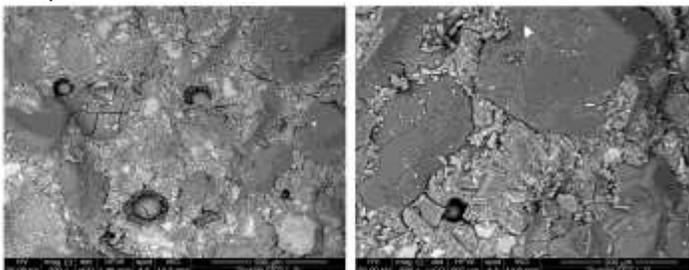


Figure 5. SEM micrograph of mix contain Na₂O= 8%, Ms=1 and w/b= 0.25.

3.3 Microstructure Analysis

3.3.1 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) images were taken to investigate the micro-structure for the materials. The tested specimens used were taken from the crushed specimens used in compressive strength test. From the figures 5 to 10, it found that a dense matrix consists of aggregate fine gel-like particles, could be CSH, NASH or CASH, which is can't be recognized clearly for their small size, different composition, disorder structure and close aggregation as was reported previously by [62]. The interfacial zone had a vital role in the strength of mixes. the stronger the interfacial zone between the aggregate and the gel matrix the higher the compressive strength. Generally, the images indicated that the products for all types of alkali activated slag samples are Na-substituted C-S-H. Increasing the Ms from 1 to 1.2 where the Na₂O% and W/B where constant it was observed that the ITZ was enhanced and the voids were reduced and this was thought to be the reason for the increase in the compressive strength as shown from Fig 5 to fig 10. It was also noticed a lot of micro cracks widespread all over the mixes and this could be due to the high temperature of activator solution which cause a shrinkage cracks as for mix Na₂O=11%, Ms=1 and W/B= 0.25. It could be also due to the crushing of the specimen in the compressive test for the rest of mixes.

3.3.2 X-Ray Diffraction (XRD)

Figure 11 show the XRD test results, which performed to detect, changes in the hydration products due to using different percentages of Na_2O , activator modulus and w/b ratios. from the figures shown, it is found that ground blast furnace slag particles are mainly amorphous with some crystalline particles such as akermanite, gehlenite and quartz, as stated by [52]. A large diffuse diffraction Peaks about 20-40 is obvious which indicate that slag specimen is almost amorphous. As shown in Fig. 11 the peak around 20 degrees in mix with Na_2O % =8%, Ms =1 and w/b = 0.25 with a compressive strength 95 MPa is higher than the same mix with w/b = 0.30 with 88 Mpa. The same trend was observed with mix Na_2O % = 9.5%, Ms = 1.2 and w/b =0.25 is higher than with w/b = 0.30.

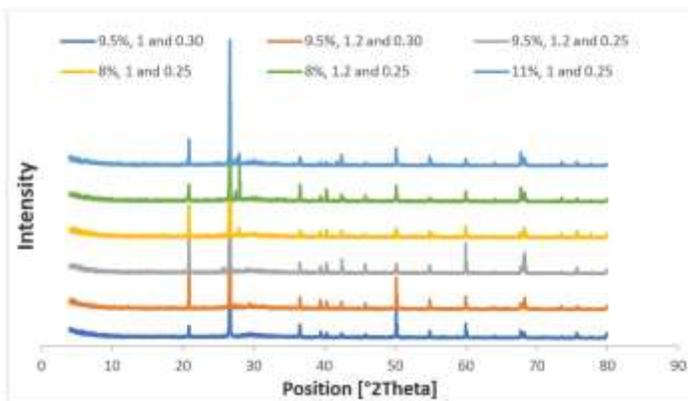


Figure 11. X-Ray Diffraction (XRD) analysis on Alkali Activated paste with different Na_2O %, Ms and W/B Ratio

4 CONCLUSION

This study examines the effect of Na_2O percentage (Na_2O %), activator modulus (Ms) and water to binder ratio (W/B) on the production of alkali activated slag mortar. The main conclusions are summarized as follows:

1. Changing the Ms ratios in study rang (0.8 - 1.4) showed slight effect for the flowability results of all mixes, while Increasing the Na_2O % by weight within the study rang (8% - 11%) decreases the flowability of all mixes.
2. Increasing the Ms decreases the Temperature of activator solution. While increasing the Na_2O % increases the Temperature. Increasing the temperature up to 90 °C increases the compressive strength, while over 90 °C the compressive strength decreases.
3. Increasing the Ms within study rang (0.8-1.4) increased the compressive strength up to a certain level (1-1.2) then the strength decreased. While Increasing the Na_2O % by weight for the study rang (8%-11%) increases the compressive strength up to Na_2O = 9.5% then the strength decreases. The maximum compressive strength reaches (98 Mpa) which is obtained with Ms =1.2, Na_2O % =9.5% and w/b = 0.25.
4. From the SEM analysis, a dense structure was obtained consists of aggregate fine like particles, which is very difficult to be recognized may be CSH, NASH or CASH, due to their small size, different composition, disorder structure and close aggregation.

5. XRD analysis shows that C-S-H of GGBS is the main phase with amorphous particle characterize

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