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Properties of One-Part Alkali Activated Ground Granulated Blast Furnace Slag Using Slaked Lime as Alkali Activator – A Comparative Study with Different Silicate Sources

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Abstract

The paper reports the results of investigating the prospect of using slaked lime as a solid activator to activate the ground granulated blast furnace slag (GGBFS) with micro-silica as an additive. The results of the above-mentioned one-part mixare compared to that of GGBFS activated with solid sodium silicate in combination with slaked lime and quick lime, discretely. The compressive strength is found to be comparable to that of slaked lime and sodium silicate activated GGBFS; the silica fume substitutionis recommended at 10% with 5% slaked lime. The main hydration products are C-S-H and C-A-S-H. Although the setting is delayed, strength development is acceptable with curing at ambient temperature. The microstructural analyses conducted through Field emission gun scanning electron microscopy (FEGSEM) and X-ray diffraction (XRD) are in good correlation with the results obtained for strength development.

Keywords: Alkali activation, one-part geopolymer, ground granulated blast furnace slag (GGBFS), micro-silica (MS), slaked lime, microstructure.

1. Introduction

With the trait of being easily moulded into any desired form and shape, concrete is essentially a reliable and robust construction material, that can sustain the mechanical and environmental impacts over the ages. However, concrete production consumes a large amount of virgin materials in manufacturing. Also, cement production liberates ample greenhouse gases. Driven by these ecological considerations, sustainable concrete production is a wide-ranged hot topic of research. Alkali-activated materials or geopolymers are an acknowledged alternative to cement as binders. Then again, the shortcomings are, the corrosive alkaline solution used in the production. To counter this drawback, one-part geopolymers are investigated; the "just add water to the binder-aggregate mix" practice, following the conventional concreting approach.

Ground granulated blast furnace slag (GGBFS) and fly ash are the most commonly used precursors in alkali-activated binders. However, the GGBFS blended mix has short setting times, which poses a major challenge in cast-in-situ applications. Also, sodium hydroxide activated GGBFS leads to fast setting and less workability due to a quicker deprotonation-hydrolysis reaction and a higher pH. This could resultin a porous mix. Sodium silicate activated slag is reported to undergo rapid carbonation. Micro-silica (MS) can prove to be effective in this case [1]. MS in combination with slag up to 15% replacement level, enhances strength; beyond which there is a decrease in strength. Even with micro-silica, the workability is reported to be significantly lower. This again is detrimental to the ease of mixing and placing the concrete. In this regard, activation of GGBFS by calcium hydroxide has proved to be effective.The presence of Ca^{+2} ions also has a positive impact on the mechanical strength owing to the formation of two different phases of C-S-H and the geopolymeric gel [2], bridging and densifying the microstructure. It also helps to accelerate the geopolymerization reaction in ambient temperature through the heat generated in the exothermic reaction of CaO, thereby improving the initial strength [3, 4].Despite the aforementioned studies, a gap remains in the usage of user-friendly solid activators. Previous studies have shown the application of anhydrous sodium aluminate[5] and sodium silicate[6] as activators. However, the toxicity of the anhydrous form of the activators still remains an issue. This study thus aims at providing a one-part geopolymer concrete, which besides utilizing the industrial waste, also uses an amiable and benign activator. The experimental program in this paper aims at contributing to a comparative study and the suitability of using micro-silica as an additive and slaked lime as an activator instead of industrial anhydrous sodium silicate. It is expected to provide an apt one-part contender in the industrial and structural application of geopolymer binder.

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2. Materials

For a suitable precursor for the alkali activation, GGBFS has to be either neutral or basic. This can be determined by checking the basicity coefficient of GGBFS. Based on the previous works of literature, the basicity (B) of GGBFS can be defined as the ratio between the total basic oxides to the totalacidic oxides. The acidic oxides (O^{2-} ionsacceptors) in GGBS are SiO_2 and P_2O_5, whereas CaO represents basic oxide (O^{2-} ions donators) and Al_2O_3, an amphoteric oxide. Hence, the basicity of GGBS can be expressed as $B = (CaO+MgO+Fe_2O_3+K_2O+Na_2O)/(SiO_2+Al_2O_3+P_2O_5)$. With the Fe_2O_3, K_2O, Na_2O, and P_2O_5present in a negligible amount, B becomes, $(CaO+MgO)/(SiO_2+Al_2O_3)$. The slag is grouped as- acid (B<1), neutral (B = 1), and basic (B>1). Also, the GGBFS requires a criterion of a CaO/SiO_2 ratio < 1.4 (as per BS: 6699) to act as a precursor material.

Ground granulated blast furnace slag with (GGBFS) from Rashmi Cement Limited, West Bengal is used as the precursor material with B = 1.14, CaO/SiO_2 = 1.36, satisfying the criteria to be a basic slag. The specific surface area and specific gravity obtained are 385 m^2/kg and 2.89 respectively. Slaked lime powder is bought from a local supplier with a specific surface area of 422 m^2/kg and specific gravity of 2.24. Micro-silica (MS) is obtained from Walter Enterprises is used as an additive. It has a specific surface area of 589 m^2/kg and a specific gravity of 2.17. The higher fineness of MS indicated the presence of finer particles as compared to GGBFS and quick lime. The chemical compositions of the materials are tabulated in Table 1. From the XRD patterns (Figure 1), industrial-grade anhydrous sodium meta silicate showed fully amorphous characteristics. The XRD pattern of GGBFS showed a major hump with its maximum around 31.5° 2θ and 2.84 Å d-spacing indicating almost amorphous silica with only minor crystalline impurities of akermenite-gehlenite.

3. Experimental Methods

a) Mixing

Paste and mortar samples are prepared in groups by variating the percentages of the precursor and activators and are shown in Table 2. The mixes are identified as- (i) L5NS series indicating 5% lime (L) with % variation of sodium silicate (NS), from 5 to 20% with an increment of 5%, (ii) Q5NS series indicating 5% quicklime (L) with a similar % variation of sodium silicate (NS), (iii) L5MS series indicating the addition of micro-silica (MS), with % variation of micro-silica, from 5 to 20% with an increment of 5%.

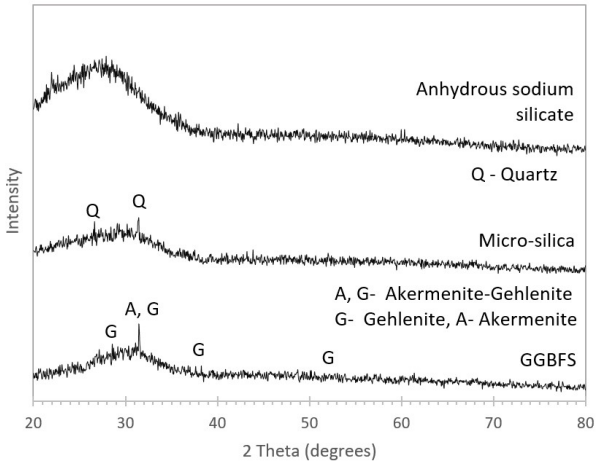


Figure 1: XRD of (a) anhydrous sodium silicate, (b) micro-silica, (c) GGBFS

Table 1: Chemical composition of the raw materials

	CaO	SiO2	Al2O3	MgO	MnO	K2O	Na2O	Fe2O3	TiO2	P2O5	SO3	LOI
GGBS	43.78	32.08	11.20	5.82	0.84	0.42	0.03	0.75	0.87	1.33	1.65	0.97
SF	3.81	84.12	0.15	1.43	1.15	2.70	0.02	2.64	0.40	0.72	0.33	2.53
SL	72.8	1.47	0.87	1.53	0.06	0.04	<0.01	0.36	0.02	-	0.15	22.7

Table 2: Weight fractions of the mix blends of L5NS, Q5NS, and L5MS series. The variations in the molar ratio are also calculated. The w/b is taken as 0.45 and the aggregate/binder is taken as 3.0.

Mix ID	% weight of the binder					Calculated molar ratios					
	GGBS	Slaked lime	Quick Lime	Sodium silicate	Micro silica	SiO_2/Al_2O_3	Na_2O/SiO_2	Na_2O/Al_2O_3	CaO/SiO_2	H_2O/Na_2O	H_2O/Al_2O_3
L5NS5	90	5	-	5	-	5.30	0.07	0.38	1.46	0.66	0.25
L5NS10	85	5	-	10	-	5.81	0.14	0.80	1.34	0.33	0.27
L5NS15	80	5	-	15	-	6.37	0.20	1.27	1.22	0.22	0.28
L5NS20	75	5	-	20	-	7.02	0.26	1.80	1.12	0.17	0.30
Q5NS5	90	-	5	5	-	5.30	0.07	0.38	1.46	0.66	0.25
Q5NS10	85	-	5	10	-	5.81	0.14	0.80	1.34	0.33	0.27
Q5NS15	80	-	5	15	-	6.37	0.20	1.27	1.22	0.22	0.28
Q5NS20	75	-	5	20	-	7.02	0.26	1.80	1.12	0.17	0.30
L5MS5	90	5	-	-	5	5.55	-	-	1.40	0.03	0.25
L5MS10	85	5	-	-	10	6.33	-	-	1.24	0.03	0.27
L5MS15	80	5	-	-	15	7.21	-	-	1.10	0.04	0.28
L5MS20	75	5	-	-	20	8.20	-	-	0.97	0.04	0.30

The water/binder for all mixes is 0.45. For the casting of the mortar samples, the aggregate/binder ratio is kept constant at 3.0. GGBFS, micro-silica, and slaked lime/quicklime were dry-mixed in a pan,for about a minute, so that they are initially mixed thoroughly. The standard sand mix was then added to the dry blend followed by the addition of tap water. The blend was hand-mixed for 4 minutes. Following this, casting was done in sextuplets of each mixing sample in cubes of 70.4 cm in a vibrating machine for 2 minutes.

b) Testing methods

The standard consistency of each paste was checked to determine the water demand of the pastes. Vicat apparatus was used with the specified plunger for the same. The initial setting time of the samples wasalso tested with theVicat apparatus with the specific needle. For both consistency and initial setting time, the water content and time are noted down in respective order, when the needle penetrates 5 mm to 7 mm above the bottom of the mould. Compressive strengths of the mortar samples were determined after curing at ambient temperature and 100% RH, at 7 and 28 days respectively. The scanning electron micrographs of both the raw materials and the activated mortar samples were obtained to have a semi-quantitative chemical composition. As for the hardened samples, the ones with the highest compressive strength are taken up for the microstructural study and XRD. Microstructural image analysis is done by a FEGSEM-EDS (field emission-gun scanning electron microscope-energy dispersive spectrometer), ZEISS Merlin Scanning Electron Microscope with Oxford EDS Detector was used. All analyses were done at 10 kV acceleration voltage. Before testing, the samples were gold coated to have the measurements smoothly.

Identification and quantification of crystalline phases for both raw materials and the hydrated products are done. It was performed with XRD using X'PertPROPANalytical diffractometer. The sample was scanned in the angular range of 20-80° at a step of 2θ. XRD analyses were performed on mortar sample powders at the age of 28 days. To stop the hydration reaction thereafter, the solvent exchange method is adopted. The crushed mortar powder was collected from post-compression test samples after 28 days and dipped in acetone. This was followed by drying at a temperature of 40°C for a few minutes to remove the solvent, before testing[7]. The crystalline phases were then quantified using the Rietveld refinement method in X'PertHighscore Plus software. The observed X-ray pattern is then close fitted to the peak intensities of the crystalline phases from the ICDD database by the least-squares method.

4. Results and Discussions

a) Consistency and Initial Setting Time

The initial setting time for all the mixes was recorded(Figure 2). The trend followed as- L5MS series > L5NS series >Q5NS series. Initial setting times were also observed to be dependent on the molar ratios, (see Figure 3). GGBS particles are irregular shaped with sharp-edged angularity and rough surface texture resulting in high internal friction.This necessitates more alkaline activators to achieve normal consistency [8]. The specific surface area of GGBFS is almost equal to OPC. So a decrease in the GGBFS content reduced the water demand and hence consistency was also reduced. Given the reduced specific surface area, a mix containing a reduced fraction of GGBFS will require less amount of activators to nucleate on the precursor particle surface, justifying the reduced consistency. Simultaneously increasing the amount of solid alkali activators, for a constant percentage of slaked lime, an increased percentage of sodium silicate will decrease the H_2O/Na_2O. H_2O/Na_2O depicts the alkali concentration on which the rate and degree of dissolution are dependent [9]. Higher the activator concentrations, lower the H_2O/Na_2O ratio, and stronger the geopolymer gel while lower concentrations tend to form zeolites. As H_2O/Na_2O is decreased, the water content is dropped, liberating more cations that are fused into the medium, causing a reduction in the viscosity. This also decreased the setting time which is obvious as the pH is increased. The increased pH of the system enhances the rate of dissolution of the Al^{+3} and Si^{+4} ions. With quicker dissolution, the setting time shortens. Rees et al.[10] reported that the induction period reduces with the increasing alkalinity but beyond a certain limit (in their case, Na_2O/Al_2O_3=0.63), there is a fall in the rate. The same trend is observed for the L5NS mixes and Q5NS mixes. Beyond a ratio of Na_2O/Al_2O_3 = 1.27, there is a sharp fall in the setting time(Figure 3. (c)). This might be due to the repelling action of the highly charged ionic system hindering the condensation reactions. The setting of the one-part geopolymers can also be altered by adjusting the Na_2O/SiO_2ratio[11]. Arnoult et. al[12] showed the increment in ionic conductivity with the increase in Na_2O/ SiO_2 due to decreased negative charge, owing to the presence of the non-bridging oxygen. The ionic conductivity however can be reduced by decreasing the H_2O/Na_2O and the SiO_2 content. The L5NS mixes and Q5NS mixes also showed accelerated setting time with an increase in Na_2O/ SiO_2.

When compared to the L5NS series, the Q5NS series showed an increased water demand and reduced setting time. This can be attributed to the higher heat evolution, locally, of quick lime causing a significant loss of water [3, 4]. It is also notable that in the L5MS mixes, increasing the amount of micro-silica increased the water demand of the mix, owing to its fineness, hence an increased specific surface area. Also, the addition of micro-silica delayed the initial setting time of the mixes. Higher the Al_2O_3 content i.e. lower SiO_2/Al_2O_3 would reduce the setting time [13]. Although the H_2O/Al_2O_3 ratio, which plays a major role in the setting time, remains the same for all three series of blends, the H_2O/CaO ratio influences the initial setting in the case of the L5MS series, as the additional silica also tends to participate in a secondary reaction to form C-S-H gel along with the C-A-S-H gel.

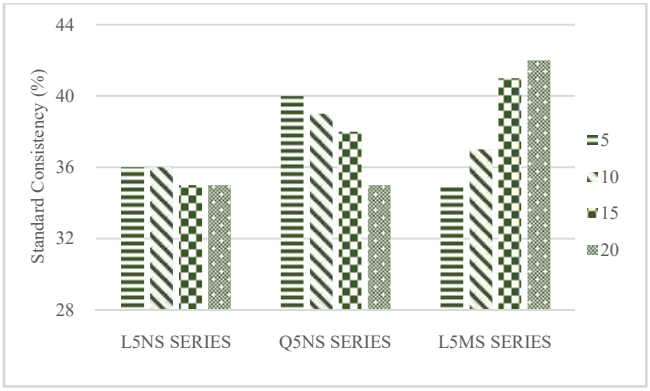
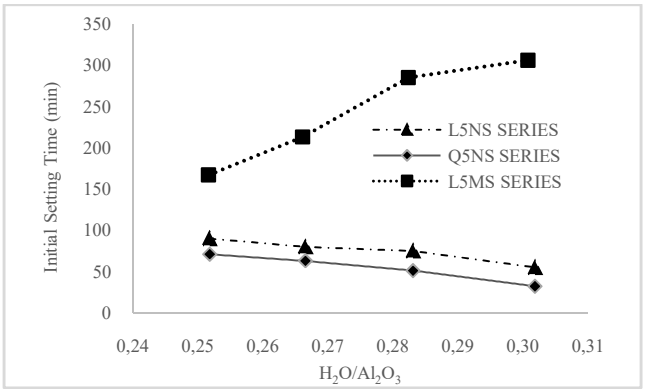


Figure 2: Standard consistency of one-part alkali-activated GGBFS mortars



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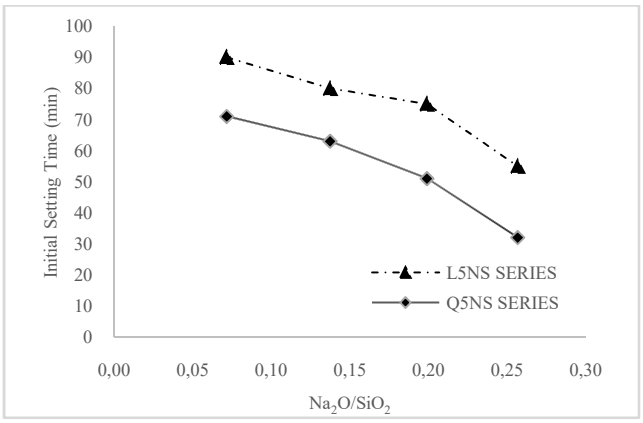

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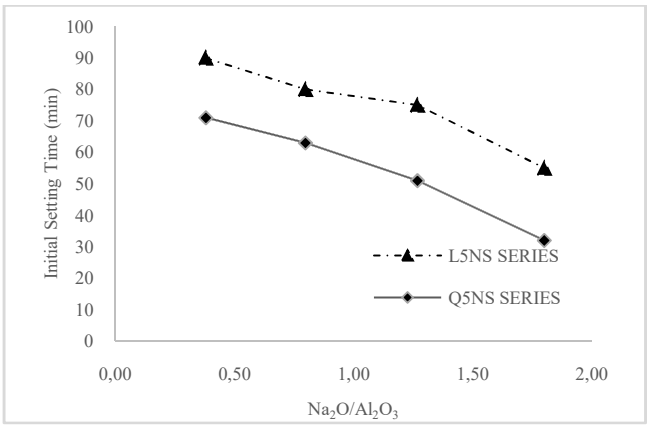

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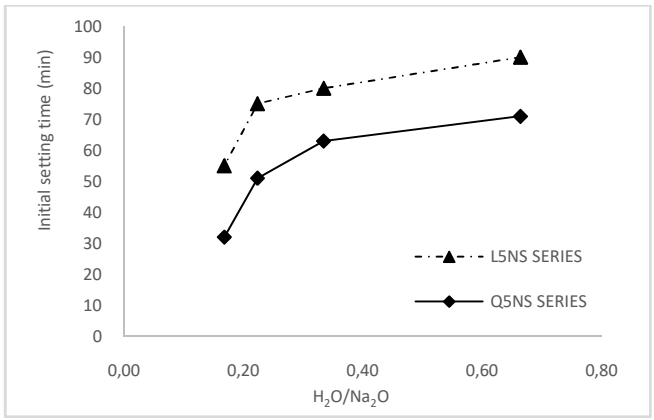

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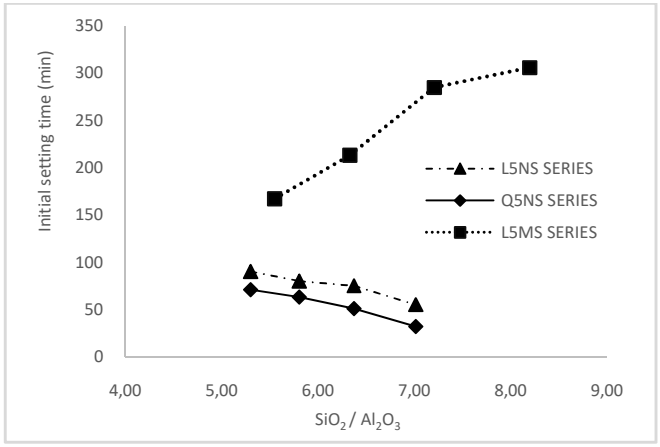


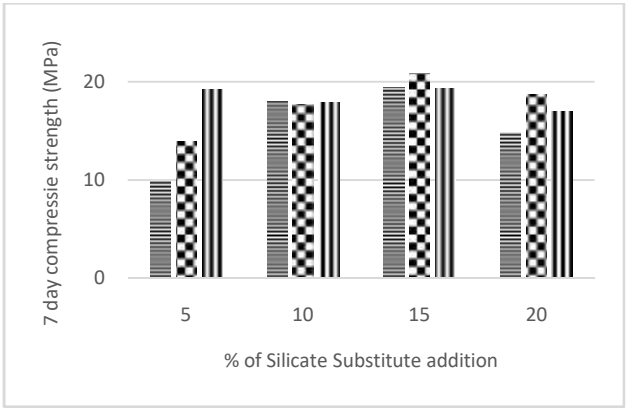
(e)

Figure 3: The variations of initial setting time as a function of the calculated molar ratios (a)H_2O/Al_2O_3 (b) Na_2O/SiO_2 (c) Na_2O/Al_2O_3 (d) H_2O/Na_2O (e) SiO_2/Al_2O_3.

b) Compressive strength

The 7-day and 28-day compressive strength of all mixes are shown in Figure 4. The development of compressive strength at 7 and 28 days, as a function of the molar ratios,is presented in Figure 5. For the sodium silicate activated mixes, both L5NS and Q5NS series showed a maximum strength at H_2O/Na_2O = 0.22 (Figure 5. (c)). This can be attributed to the higher pH of the mortar samples, which enhances the dissolution phenomenon of the aluminosilicates. It has been studied that, in a sodium silicate activated mix, the activator content influences the formation of the hydration products and thereby contributes to the strength gain by influencing the zeta potential of the GGBFS particles[14]. The higher content of activators resulted in the positive zeta potential of the GGBFS particle. However, beyond this, there is a notable decrease in the strength, which suggest the possibility of repelling action.

Slag activated by slaked lime solely, however, showed a comparable maximum strength at CaO/SiO_2 of 1.24. The divalent calcium ion reacted with the hydroxyl groups to form the precipitate which increases the pH and thus provided sites for the nucleation and polymerization [15]. The continuous formation of the binding gels of C-S-H and C-A-S-H however depends highly on the availability of the calcium ions and the pH of the mix. The extent of calcium ions should be such as to facilitate the reaction between the silicate and aluminate tetrahedrons[16].



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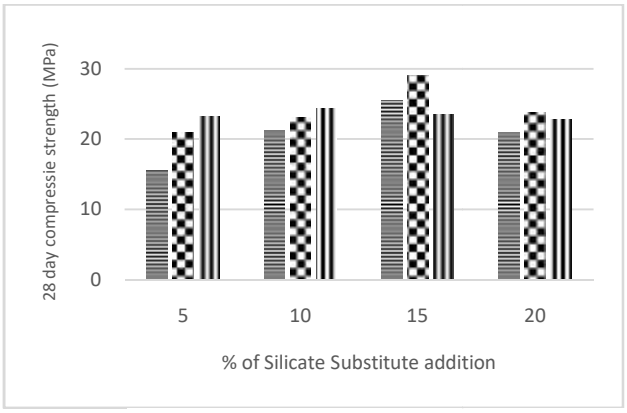
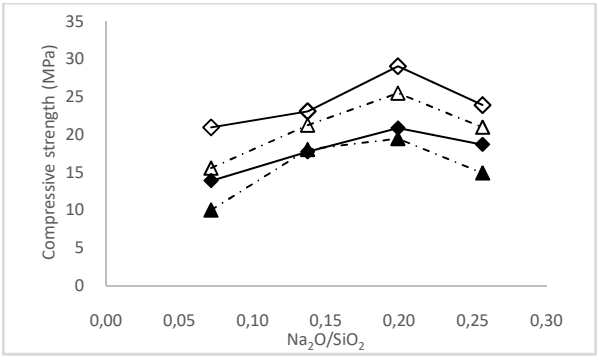


(b)

Figure 4: 7-day and 28-day strength development of the L5NS, Q5NS, and L5MS series, with the % variation in the silicate substitute (sodium silicate and micro-silica) addition



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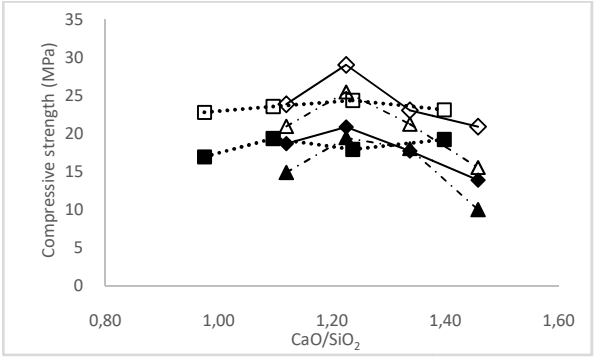

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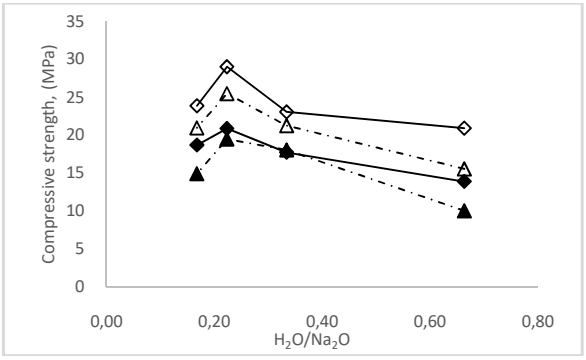

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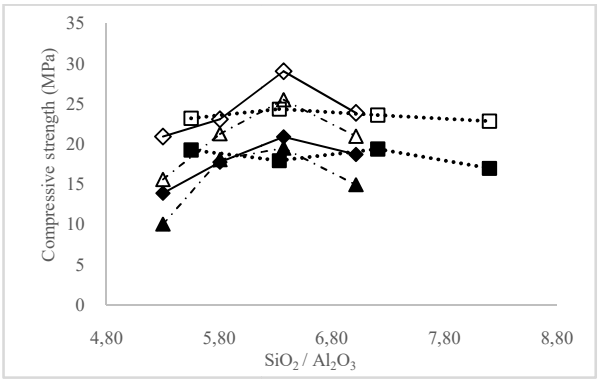


(d)

Figure 5:The development of compressive strength at 7 and 28 days,as a function of the molar ratios (a)H_2O/Al_2O_3 (b) Na_2O/SiO_2 (c) Na_2O/Al_2O_3 (d) H_2O/Na_2O (e) SiO_2/Al_2O_3.

It is notable that with reduced Al_2O_3 content and increased SiO_2/Na_2O and Na_2O/Al_2O_3 content, the strength increased. This is because Al_2O_3 mainly acts as a network modifier; and with more SiO_2, more of it could be consumed by the network to form C-A-S-H. The compressive strength increased with the addition of sodium silicate and posed a maximum at $Na_2O/SiO_2 = 0.2$, beyond which it dropped.

For the L5MS series, the strength development is steady at all replacement levels of micro-silica for both 7 and 28 days, Figure 4. This, again, establishes micro-silica's role as an additive in the mix. The maximum strength reached is 17.94 MPa and 24.35 MPa at 7 and 28 days respectively. Although previous works of literature showed the activator amount restricted to 10% for slaked lime[17], any further addition is subjected to investigation. For 28 days, Q5NS15 showed the highest strength of 29.02 MPa.

c) Microstructure

Scanning electron micrographs of L5NS15 (Figure 7) and L5MS10 (

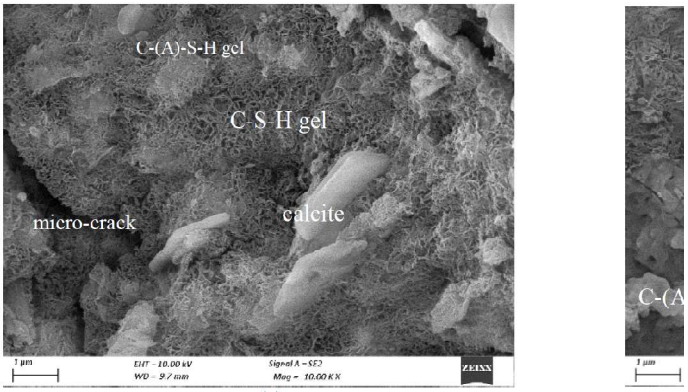


Figure 8) are studied to better understand the differences in the application of the different silica sources. For L5NS15, the phases are distinguishable as a layered N-A-S-H gel formation, for the 7-day sample. Some unreacted GGBFS particles are observed, resulting from incomplete geopolymerization. Also, there are micro-cracks which might be due to local temperature alterations. However, the unreacted GGBFS is not noticed in the 28-day micrograph, establishing the likelihood of the secondary reactions taking place in due course of time, supporting the formation of NASH. The 28-day micrograph also showed the formation of zeolitic crystals. For L5MS10, however, wide formations of C-S-H are seen than the C-A-S-H, along with calcite. This, again, establishes the fact that the silica fume is rather acting as an additive and the secondary reactions possibly have taken place simultaneously with the primary alkali activation of GGBFS. The dissolution of GGBFS particles, on the other hand, solely takes place due to activation by slaked lime. The pH of the L5MS series is apparently less than the L5NS series, which institutes the formation of more C-S-H gel like structures. Hence, in this case, pozzolanic reaction prevails over geopolymerization.In high calcium phases, however, previous literatures reported the formation of C-S-H gel along with N-A-S-H, C-A-S-H, and C-N-A-S-H gels. C-S-H is reported to have the typical dreierketten sheet structure of tobermorite[18] with two paired Si tetrahedra sharing two oxygen atoms with the centeredCaO membrane and a bridging tetrahedron sharing only one. However, L5NS15 or Q5NS15 did not show any C-S-H structures in SEM. With the addition of Ca^{+2}, the gel structure of N-A-S-H is modified; this is due to the polarizing effect of Ca^{+2}, and the Si-O-Al linkages tend to form Si-O-Ca[19].

Dissolution of sodium silicate and disintegration of GGBS released the Ca^{+2}, Na^{+}, SiO_2^{-} and AlO_4^{-} ions simultaneously. For Q5NS15, the initial exothermic reaction accelerated the polycondensation reaction. Consequently, the ion exchange period gets shortened. Both the alkaline cations get bounded to the SiO_4 and AlO_4, balancing the charge disparity due to the replacement of the Si^{+4} by Al^{+3}, thereby forming the 3D C-N-A-S-H structure. Now, due to a stronger polarising effect of Ca^{+2} over Na^{+}, the C-N-A-S-H tends to form C-A-S-H. The C-N-A-S-H however retains its 3D structure until the calcium replaces all of the sodium content[20]. The dissolution was reported [21] to have been delayed and so is the formation of the polymer network owing to the competition between the alkali cations Ca^{+2} and Na^{+} due to Lewis acidity. The same is noted in the L5NS15. Without the initial heat, there is delayed condensation due to the competition between the ions. The ion exchange is thus prolonged allowing for the formation of C-N-A-S-H, C-A-S-H, and N-A-S-H gel. With time, the Ca^{+2} ions are drained out, and C-N-A-S-H, C-A-S-H, and N-A-S-H will co-exist.

The same is observed in the XRD patterns, Figure 6, of the L5NS15 and Q5NS15. With an accelerated dissolution and quicker setting, it showed the simultaneous formation of C-A-S-H and C-N-A-S-H. A strong peak of zeolite A is seen at 35.62 (ICDD #98-602-8832), cubic crystals of which are visible in the SEM images too. For the L5NS15, however, with delayed condensation, the formation of N-A-S-H gel is distinctly visible, along with calcite, C-A-S-H, and boggsite. Distinct patterns of C-S-H (#98-011-5657) and C-A-S-H (#98-007-7872 and #98-002-8980) are seen with amounts of calcite for the L5MS15 sample.The coexistence of N-A-S-H and C-A-S-H exhibits a lesser compressive strength.

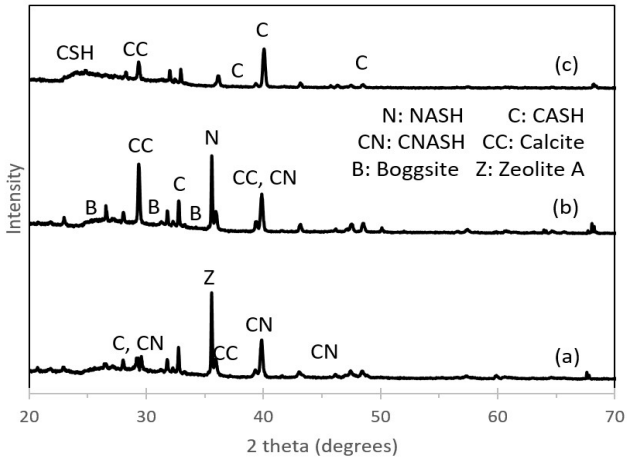


Figure 6: XRD of the hydration phases formed after 28days of curing (a) Q5NS15, (b) L5NS15, (c) L5MS10

Table 3shows the ratios of the oxides, calculated from the EDS data. These results are sensitive to the selection of the sample surface points and hence a mean of 20 points is taken for the experimental analysis. As a source, GGBFS solely contributes to the Al_2O_3 content. So a higher Na_2O/SiO_2 would suggest lower silicate content or lower silica incorporation. This simply suggests that GGBFS has not participated completely.

Table 3: Comparison of the oxide ratios as observed in the experiment and calculated.

	SiO_2/Al_2O_3		Na_2O/SiO_2		Na_2O/Al_2O_3		CaO/SiO_2	
	expt.	calc.	expt.	calc.	expt.	calc.	expt.	calc.
L5NS15, 7days	3.62	6.37	0.38	0.20	1.39	1.27	1.37	1.22
L5NS15, 28 days	4.00		0.15		0.61		1.07	
Q5NS16, 7 days	3.98	6.37	0.33	0.20	1.31	1.27	1.29	1.22
Q5NS16, 28 days	4.66		0.16		0.74		1.19	
L5M10, 7 days	4.08	6.33	-	-	-	-	1.67	1.24
L5M10, 28 days	4.28						1.10	

It is interesting to note that the experimental values of the Na_2O/SiO_2 ratio and the Na_2O/Al_2O_3 ratio at 7 days for L5NS15 are higher than the calculated value. This supports the SEM images for the same, indicative of incomplete dissolution of the GGBFS particles (Figure 7); which otherwise is no longer visible for the 28 days. L5NS15 has a comparatively delayed dissolution-condensation than

Q5NS15, which is also reflected in the EDS reading, as $[Na_2O/SiO_2]_{Q5NS15} < [Na_2O/SiO_2]_{L5NS15}$ at 7 days. however, at 28 days, the values are almost equal. Again, higher $[Na_2O/Al_2O_3]_{Q5NS15}$ can be contributed to the local temperature rise. Also lesser margin than L5NS15 is observed in 7 days' experimental value as the dissolution is accelerated. It is to be noted that the CaO/SiO2 value for L5MS10 is maintained close enough to both L5NS15 and Q5NS15 to support the compressive strength results of the same. The SiO_2/Al_2O_3 values follow the trend $[SiO2/Al2O3]_{Q5NS15} > [SiO2/Al2O3]_{L5MS10} > [SiO2/Al2O3]_{L5NS15}$, emphasising that the rate of dissolution follows the same trend.

Figure 7 SEM images of L5NS15 mortar containing 5% slaked lime and 10% sodium silicate at (a) 7 days (b) 28 days

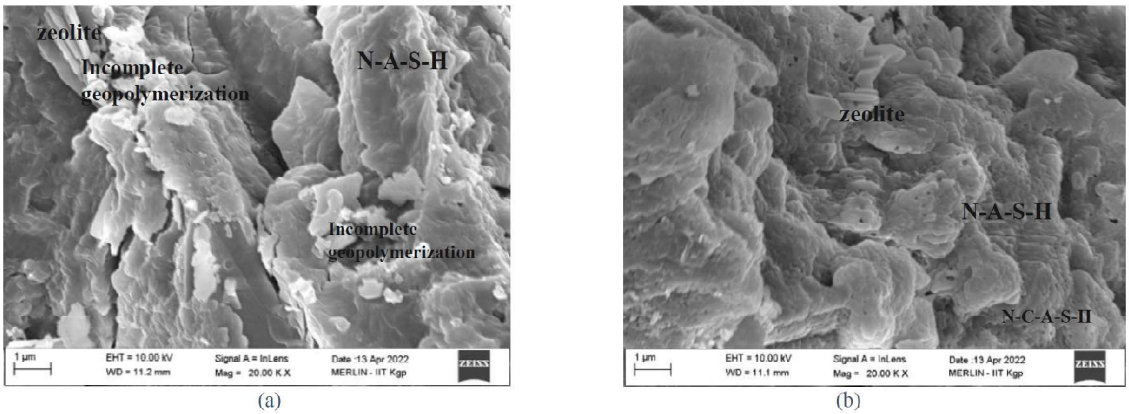
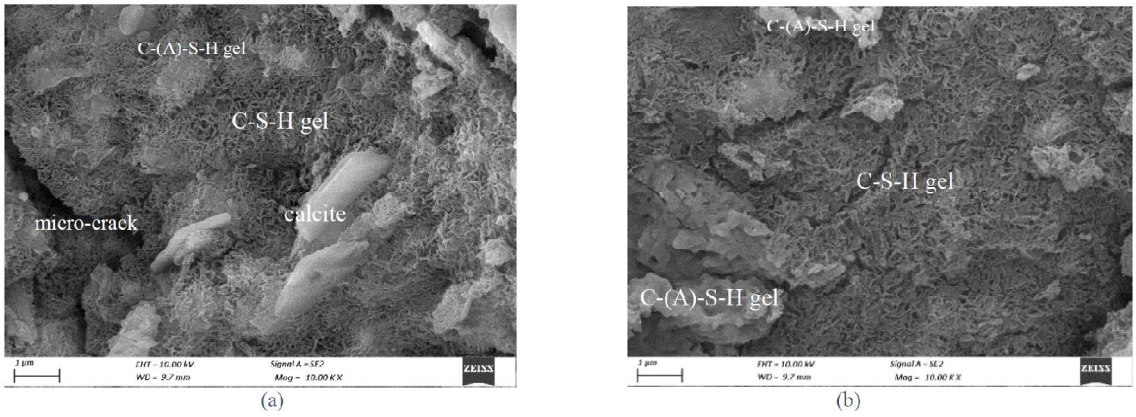


Figure 8 SEM images of L5MS10 mortar containing 5% slaked lime and 10% silica fume at (a) 7 days and (c) 28 days. The formation of Al- substituted tobermorite at 28 days is widely visible.



5. Conclusions

In this article, the properties of ground granulated blast furnace slag activated by different solid activators were investigated. The activators are- (i) slaked lime and solid sodium silicate, (ii) quick lime and solid sodium silicate, and (iii) slaked lime with micro-silica as an additive. The following conclusions were drawn-

(a) The sodium silicate replacement level can be varied up to 15% for both slaked lime and quick lime (5%) for maximum strength, beyond which there is a strength reduction. The corresponding addition of micro-silica gives a maximum strength at a 10% replacement level. In all cases, the CaO/SiO2 ≈ 1.24.
(b) The setting time can be tailored with the choice of activator. Using micro-silica can undoubtedly increase the water demand and initial setting time.
(c) The compressive strength of all three mixes is comparable. The 28-day strength is 25.48 MPa, 29.02 MPa, and 24.35MPa for L5NS15, Q5NS15, and L5MS10 respectively.
(d) Both C-S-H and C-A-S-H govern the microstructural gel for the combination of GGBFS and micro-silica activated by slaked lime.
(e) Further investigation is needed to determine the engineering properties of GGBFS and MS activated by slaked lime.

Disclosures

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