Technical report

Synthesis of Alkali-Activated Binary Blended Silico-Manganese Fume and Ground Blast Furnace Slag Mortar

Muhammad Nasir¹, Megat Azmi Megat Johari², Moruf Olalekan Yusuf^{3*}, Mohammed Maslehuddin⁴ and Mamdouh A. Al-Harthi^{5,6}

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Abstract

This study was aimed at developing novel and sustainable alkali-activated binder of silico-manganese fume (SMF) and ground blast furnace slag (BFS) at room temperature. The level of BFS substitution was varied from 0 - 50% and activation was done using 10 M NaOH and Na₂SiO₃ of an initial silica modulus of 3.3. The Na₂SiO₃/10MNaOH ratio was kept constant at 2.5. The setting time, flow and compressive strength were evaluated and complemented with the microstructural and bond characteristics of the product. A 28-day strength of 45 MPa was achieved with the base materials ratio (BFS/(BFS+SMF)) of 0.3. The activation of SMF resulted in the formation of glaucochroite and nchwaningite minerals (C-Mn-S-H) while the inclusion of BFS influenced the formation of potassium feldspar (K-A-S-H), gehlenite hydrate (C-A-S-H) and additional C-S-H due to later-age contribution of hydration of BFS. The Manganese in SMF contributed significantly to the consistency of the mixture while lime in BFS improved the microstructure thereby enhancing strength of the developed alkali-activated binder.

1. Introduction

The alkali-activation technology is gaining importance owing to its economic and ecological advantages that result in a reduction in the consumption of ordinary Portland cement (OPC) and hence a decrease in the greenhouse gas (CO₂) emission. The production of an alkali-activated binder (AAB) requires alkaline activation of aluminosilicate or calcium-aluminate based source material, such as fly ash, blast furnace slag or other wastes. An alkaline activator could be of low to high concentration alkalis or silicate of alkaline metal elements, such as sodium or potassium (Provis 2014). The resulting binder possesses excellent strength and durability together with the capability of immobilizing the toxic metals (Salami *et al.* 2017; Vu and Gowripalan 2018). The use of a combination of Na₂SiO₃ and NaOH promotes the activation of an AAB (Phoo-Ngernkham *et*

al. 2015, 2016). The molarity or concentration of NaOH that enhances the dissolution of base materials with high silica content is reported to be within the range of 8 – 12M (Yusuf *et al.* 2014a; Heah *et al.* 2013; Nagaraj and Babu 2018) while the optimum activator ratio (Na₂SiO₃/NaOH) varies from 2 – 3 (Ibrahim *et al.* 2019, 2017; Mohseni 2018).

The proliferation of silico-manganese fume (SMF) calls for its usage in large quantities. For instance, as per 2012 statistics of International Manganese Institute (IMnI), the world annual production of SM waste was 14.8 MMT. In the light of this, there is a need for utilizing this waste material (SMF) together with others with hydraulic property, such as blast furnace slag (BFS). It is hypothesized that these two materials may complement each other through alkali activation to provide a binder with good structural characteristics. The development of a binder with good properties will lead to the use of SMF in large quantities along with BFS whose hydraulic characteristic has been established (Puertas *et al.* 2000). Other researchers also established the contribution of BFS to the mechanical and microstructural characteristics of AAB developed with other precursors, such as fly ash, palm oil fuel ash, metakaolin and others (Rashad 2013; Yusuf *et al.* 2014b; Bernal *et al.* 2011). The higher quantity of Ca²⁺ in BFS could play a charge balancing role, contribute to carbonation effect or prompt primary or secondary hydration reaction when it reacts with silica to form calcium silicate hydrate (C-S-H) (Haha *et al.* 2011; Fernández-Jiménez *et al.* 2003; Zhang *et al.* 2008; Liu *et al.* 2018; Bonk *et al.* 2003).

Prior studies on silico-manganese slag (SMS) conducted by Kumar *et al.* (2013) already established mechanical treatment of the raw materials through ball milling processes (attritions and eccentric vibration).

¹Ph.D. Researcher, School of Civil Engineering, Universiti Sains Malaysia, Pulau Pinang, Malaysia.

²Professor, School of Civil Engineering, Universiti Sains Malaysia, Pulau Pinang, Malaysia.

³Assistant Professor, Department of Civil and Environmental Engineering, University of Hafr Al Batin, Hafr Al Batin 31991, Saudi Arabia.

*Corresponding author, E-mail: moruf@uhb.edu.sa

⁴Senior Researcher (Professor), Center for Engineering Research, Research Institute, King Fahd University of Petroleum and Minerals.

⁵Professor, Department of Chemical Engineering, King Fahd University of Petroleum and Minerals.

⁶Professor, Center of Research Excellence in Nanotechnology, King Fahd University of Petroleum and Minerals.

Table 1 Mix design parameters.

Mix ID	Ingredient quantities (kg/m ³)						Constant ratios		
	SMF	BFS	FA	FW	SH	SS	FA/PMs	SS/SH	AAs/PMs
AASB ₀	723.0	-	1301.4	14.46	103.2	258	1.8	2.5	0.5
AASB _{0.1}	650.7	72.3	1301.4	14.46	103.2	258	1.8	2.5	0.5
AASB _{0.2}	578.4	144.6	1301.4	14.46	103.2	258	1.8	2.5	0.5
AASB _{0.3}	506.1	216.9	1301.4	14.46	103.2	258	1.8	2.5	0.5
AASB _{0.4}	433.8	289.2	1301.4	14.46	103.2	258	1.8	2.5	0.5
AASB _{0.5}	361.5	361.5	1301.4	14.46	103.2	258	1.8	2.5	0.5

This treatment is capable of reducing the setting time of an activated SMS. Nath and Kumar (2016) also reported that a part replacement of BFS with SMS in BFS/OPC paste could delay both initial and final setting of the binder. The inhibition in the setting was attributed to the presence of MnO in SMS (Frias and Rodríguez 2008; Frias *et al.* 2005; Péra *et al.* 1999). However, there is a distinctive difference between SMS and SMF by virtue of their oxide composition owing to the dearth of K₂O and MnO₂, and higher CaO and Al₂O₃ in the former than the latter, respectively (Frias and Rodríguez 2008).

The quantity of potassium in SMF could be higher than 15% due to the presence of potassium aluminosilicate (KAlSi₃O₈) (Najamuddin *et al.* 2019) or other impurities, such as potassium feldspar generally occurring in manganese oxide ore (Zhong *et al.* 2017). Abdulkareem *et al.* (2018) reported that high K₂O content could contribute to the strength in the presence of silica and lime in the OPC-based binder. However, the role of K₂O in AAB synthesis co-existed with MnO in SMF and their synergy with BFS, is not yet fully elucidated.

Hitherto, SMF and BFS have not been used towards developing AAB at room temperature. Similarly, the role of Mn-bearing particles on the fresh and strength properties of AAB with these two materials is not yet fully understood. The reported study was aimed at developing a sustainable AAB by utilizing high volume of SMF in combination with BFS. The findings are expected to promote the use of both industrial byproducts in the development of a green binder.

2. Materials and methods

2.1 Materials and sample preparation

(1) Precursor materials

Silico-manganese fume (SMF) was collected from a ferro-alloy manufacturing industry at Jubail, Saudi Arabia. For every ton of ferro-alloy production, about 10% of SMF waste is generated. As received SMF was then collected and sieved through 40 micron sieve to eliminate any lumps. Ground blast furnace slag (BFS) was procured from a local ready-mix concrete plant.

The particle size distribution of the precursor materials (PMs) was determined using Malvern's Mastersizer 2000 with a measuring range of 0.02 to 2000 microns. A gas pycnometer, AccuPyc II 1340, was used to determine the specific gravity and the bulk density of the PMs. An automatic gas sorption data acquisition and

reduction machine, Quantachrome ASiQwin, was used to determine the surface area of the PMs. X-ray fluorescence (XRF) spectrometer, PANalytic model EPSILON 3 XL, was used to determine the elemental composition of the PMs.

(2) Synthesis of alkaline activators

The alkaline activators (AAs) consisted of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) while their combinations with the addition of free water (FW) is referred to as combined alkaline activators (CAAs). One liter of 10 M NaOH (SH) solution was prepared by mixing approximately 400 g of SH pellet (98.6% purity) with distilled water and then allowed to cool for one day prior to its use. The composition of Na₂SiO₃ (SS) was as follows: H₂O = 62.50%, SiO₂ = 28.75% and Na₂O = 8.75% such that its initial silica modulus (Ms = SiO₂/Na₂O) was 3.3.

(3) Specimen preparation

Alkaline activated mortar (50 mm cube) and paste (25 mm cube) specimens were prepared by casting six mixes by varying the ratio of BFS/(SMF + BFS) as 0, 0.1, 0.2, 0.3, 0.4 and 0.5. The mixtures were designated as AASB_x. For example, AASB_{0.3} means the AAB comprises 30% and 70% of BFS and SMF, respectively. The proportion of materials in the AASB mortar, as shown in **Table 1**, was selected based on the trial mixtures.

(4) Mixing, placing and curing

The mixture constituents was mixed in a Hobart bench top planetary mixer. The PMs were first mixed to remove the air-pocket followed by the addition of CAAs. The prepared AAB was placed in the molds in two layers by consolidating each layer for a duration of 12 s. The surface of the specimens was levelled using a trowel and wrapped with plastic sheets to prevent moisture loss before being placed at ambient laboratory temperature (25 ± 2°C) for setting purpose. The specimens were demoulded after 24 h, and then placed in polythene bags for continuous curing till the age of testing.

2.2 Evaluation

(1) Setting time

The initial and final setting time of AASB paste were measured according to ASTM C 191 using a Vicat apparatus. The initial setting time was measured after every 5 - 10 min interval until a penetration of 25 mm or less

is achieved while the final setting time was recorded at every 3 - 5 min until the circular needle left no indentation or impression on the surface of the specimen.

(2) Flow

The flow of the developed AAB was evaluated as per ASTM C1437 and ASTM C230. This flow was measured after 10 and 20 min intervals in order to evaluate the flow retention. The resulting values were classified based on the diameter of spread as either fluid (> 200 mm), plastic ($140 - 200$ mm) or dry (< 140 mm), as per BS EN 1015.

(3) Compressive strength

Compressive strength was determined on 50 mm cube specimens, according to ASTM C109. Mortar specimens were retrieved from plastic bags after 3, 7 and 28 days and compressive load was applied using a compression testing machine, model ELE-ADR-Auto, at a loading rate of 0.9 kN/s. Three specimens were tested from each batch at each age and their average readings were reported.

(4) Microstructure and composition

In order to study the contribution of SMF and BFS on the hydration products, AASB₀ (BFS-free) paste specimens were compared with the specimens exhibiting optimum strength (AASB_{0.3}) after 7-day curing. The selected specimens were ground and sieved through 100 μm sieve to be used in X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy. A Rigaku MiniFlex II – Benchtop X-ray diffractometer was utilized to obtain mineralogy of the products. The X-ray analysis parameters were Cu-K α 1 as the radiation source with the relevant voltage (30 kV) and current (15 mA) while the scan speed was 2.5 deg./min. Thermo Fisher Scientific Nicolet FT-IR Spectrometer was used to detect the functional groups or bond characteristics. The IR spectra were generated between the wavenumbers 400 – 4000 cm^{-1} . The morphology and elemental composition of the specimens were evaluated using JEOL model JSM-6610-LV scanning electron microscope (SEM) equipped with an energy dispersive spectrophotometer (EDS). The SEM-EDS analysis was performed on fragmented cube specimens, coated with gold and affixed on a brass holder using a double-sided cop-

Table 2 Physical properties of SMF and BFS.

PMs	Density (g/cm^3)	Particle size (μm)				Specific surface area (m^2/g)
		d_{10}	d_{50}	d_{90}	Span $(d_{90}-d_{10})/d_{50}$	
SMF	2.876	0.588	29.173	179.958	6.15	10.1
BFS	2.933	1.541	13.585	39.211	2.78	2.5

per tape, under the current supply of 20 mA.

3. Results and discussion

3.1 Physical, chemical and mineralogical characteristics of raw PMs

The BFS particles were irregular to polygonal in shape while the SMF particles were round and spherical (**Fig. 1**). The specific surface area of SMF and BFS was 10.1 and 2.5 m^2/g , respectively. Generally, BFS has relatively smaller particles as shown by the proportions of particle sizes corresponding to d_{10} , d_{50} , and d_{90} (**Table 2**) while its size is more uniform as shown in the span $((d_{90}-d_{10})/d_{50})$ value in comparison with SMF particles which also has a bigger surface area. The d-values represent the particle size distribution of a material, such that d_{10} , d_{50} and d_{90} indicate the particle size relevant to 10, 50 and 90% of cumulative mass, respectively. The disparity in the grain morphology, surface area and particle size distribution of SMF and BFS indicate that these materials could contribute to the rate of reaction in synergy while the difference in the texture could also influence the workability through particle interlocking, and frictional or lubricating effects. These attributes are expected to significantly affect the rheology of the developed binder. Further, BFS is denser than SMF, as shown in **Table 2**. This could significantly affect the density of the developed AAB. **Figure 2** shows the particle size distribution curves of both precursors.

The major oxides in SMF include Mn, Si, and K which constitute more than 70% of the total material oxide composition. The minor oxides, such as Ca and Mg constituted about 13%. In contrast, 90% of BFS is composed of oxides of Ca, Si and Al (**Table 3**). It is important to note that the quantity of K_2O in SMF is very high, whereas it is insignificant in the BFS. A reverse trend is noted in the quantity of Al_2O_3 . Therefore, there is propensity that K_2O and Al_2O_3 together with

Fig. 1 Morphology of: (a) SMF and (b) BFS.

Fig. 2 Particle size distribution of PMs.

Table 3 Oxide composition of SMF and BFS.

PMs	Oxide (%)														LOI (%)
	CaO	MnO	SiO ₂	K ₂ O	Al ₂ O ₃	SO ₃	MgO	Fe ₂ O ₃	Cl	ZnO	P ₂ O ₅	Ag ₂ O	TiO ₂	Total	
SMF	7.24	35.42	19.50	16.62	1.46	6.24	5.71	3.35	2.73	0.74	0.37	0.15	-	99.53	5.82
BFS	49.02	0.40	29.32	0.41	12.19	2.03	4.50	0.83	-	-	0.34	0.11	0.66	99.81	1.25

MnO, CaO, and SiO₂ in both materials could play a synergistic role in the setting and strength development of the AASB_x binder.

As shown in **Fig. 3**, SMF is crystalline in nature and it mainly consisted of quartz (1-SiO₂) (PDF#85-0796), glaucocroite (2-CaMnSiO₄) (PDF#14-376), sylvite (3-KCl) (PDF#04-0587) and wollastonite (4-CaSiO₃) (PDF#72-2284). The amorphous nature of BFS is indicated by peaks gehlenite [5-Ca₂Al(AlSiO₇)] (PDF#89-1489) and akermanite [6-Ca₂Mg(Si₂O₇)] (PDF#77-1149) within the range of 27-33° 2θ.

3.2 Setting time

As shown in **Fig. 4**, the inclusion of BFS in AASB binders decreases the initial and final setting times. This may be attributed to the higher specific gravity and polygonal shape of BFS thereby enhancing the precipita-

Fig. 3 X-ray diffractogram of PMs: SMF (top spectrum) and BFS (bottom spectrum).

Fig. 4 Initial and final setting time of the AASB paste.

tion of the oligomer within a short period of time in comparison with the BFS-free mixture (AASB₀). This is due to the formation of calcium aluminosilicate hydrate (CASH) that increases the density of the binder due to polycondensation process. The presence of CaO in the BFS increases the viscosity of the mixture (Fernández-Jiménez and Puertas 2001; Puertas *et al.* 2014) due to the formation of calcium carbonate upon reaction with atmospheric carbon dioxide, as shown in Eqn. 1.

BFS could also play a significant role of charge balancing in the condensation of aluminosilicate oligomers after initial dissolution by the alkaline activators thereby leading to the formation of additional C-S-H. It was noted that SMF alone takes a longer time of about 470 minutes for setting (initial) due to the paucity of CaO. Consequently, the setting time decreases with an increase in the quantity of BFS, as shown in **Fig. 4**. Specifically, the initial and final setting time of AASB with 10% BFS (AASB_{0.1}) decreased by 72.3% and 71.4%, respectively. This reduction increased to 76% and 75.23%, respectively, as the quantity of BFS was increased to 20%. Therefore, the extended setting recorded in the alkaline silico-manganese fume paste is attributed to the low CaO/SiO₂ ratio.

3.3 Flow

Figure 5 shows the flow of the developed AASB mortars. The flow generally decreases with BFS inclusion up to 50%. The decrease in flow in the AASB with the incorporation of BFS may be attributed to the formation of C-S-H and the irregular shape of BFS having a non-lubricating effect on SMF particles due to an increase in the inter-particle frictional resistance. The excessive presence of MnO in the precursor materials is expected to influence the rheology of the binder. Therefore, the

Fig. 5 Flow of the AASB mortar.

rheology of the binder is controlled by MnO/CaO, as it appears that MnO in SMF dissolves faster in the alkaline activators than CaO contributed by BFS. An increase in the MnO/CaO ratio increases the immediate flow from 176 to 184 mm up to 20% BFS addition (AASB_{0.2}), but any further addition of BFS decreases the flow from 184 to 175 mm, as shown in Fig. 5. As the mixing continues, further CaO is formed from the complexes of BFS - depending on the concentration of the activators (Puertas *et al.* 2014) - thereby further decreasing the MnO/CaO ratio. This causes decrease in flow of AASB mortar regardless of the level of BFS substitution, as indicated by the flow measurements taken after 10 and 20 mins of mixing (Fig. 5). Further, the mixture loses its consistency significantly as the BFS substitution level goes beyond 30% (AASB_{0.3}).

3.4 Compressive strength development

Figure 6 shows the compressive strength development in AASB_x mortars. The mortar specimens prepared with more than 30% BFS content were discarded due to dry consistency and quick setting which resulted in air voids and honeycombs in the specimen (Fig. 7). Generally, the strength of room-cured specimens increased with age. It is evident that the controlling factor governing the compressive strength is the ratio of CaO/SiO₂. This ratio increases as the level of substitution of BFS in the binary blending increases. Since BFS serves as a source of CaO supplement in the mixture, it is quite evident that the strength development follows the same pattern. The early strength increased by 122.2% due to the replace-

ment of 30% BFS while it was only 40.7% for 20% BFS, in comparison with the BFS-free specimens. However, there was a significant gain in strength after 28 days. This suggests that there could be a delayed hydration process leading to the formation of C-S-H (PDF#72-2396, 40-0513, 11-0507), in parallel to the formation of condensed gehlenite (Ca₂Al₂SiO₇) (PDF#89-1489) or stratlingite hydrate [4-Ca₂Al₂(SiO₂)(OH)₁₀.2.5H₂O] (PDF#085-8414), nchwangingite [1-Mn₂SiO₃(OH)₂.H₂O] (PDF#83-1006) and rancieite minerals [2-(Mn,Ca)Mn₄O₉.3H₂O] (PDF#058-0466), as indicated by the X-ray peaks in Fig. 8. This dual product formation is more significant in the binders with 30% BFS. The 28-day compressive strength was 45, 34, 23, and 19 MPa in binders with 30, 20, 10, and 0% BFS, respectively.

The morphology (Fig. 9) of AASB₀ shows significant quantities of un-reacted/un-hydrated SMF particles due to lower CaO/SiO₂ with prolonged or interconnected cracks indicative of a weak and less dense microstructure. The weak microstructure of AASB₀ suggests that there will be more ingress of atmospheric carbon dioxide to the binder resulting in the formation of CaCO₃. This is evident by the presence of C and O peaks in the EDS profile.

A relatively dense microstructure is indicated by the SEM for specimen AASB_{0.3}. Unlike specimen AASB₀, the cracks are localized and disconnected. Further, there is fewer traces of unreacted SMF particles, this is also indicated by a decreased quantity of Mn in the EDS for this specimen. By comparing the EDS spectra (Table 4) of AASB₀ and AASB_{0.3}, it is evident that the denser mi-

Fig. 6 Compressive strength development in AASB mortar.

Fig. 8 X-ray diffractogram of AASB₀ (top spectrum) and AASB_{0.3} (bottom spectrum).

Fig. 7 Visual appearance of AASB specimens: (a) discarded (BFS > 30%) (b) properly consolidated (BFS = 0-30%).

crostructure of the latter (**Fig. 9**) has Ca/Si ratio of 0.95 compared to 0.25 of the weaker microstructures of the former. More importantly, is the ratio of Si/Mn which was 0.66 and 1.30, respectively, while Ca/Mn was also 0.16 and 1.23, respectively. Therefore, for the synthesis of silico-manganese fume alkaline binder, the ratio of Ca/Si, Si/Mn and Ca/Mn ratios could be the relevant indicators to determine the performance of the binder in terms of the strength characteristics. This further strengthens the presence of C-Mn-H, C-S-H and C-A-S-H phases in the X-ray diffractograms of **Fig. 8**. The peak for potassium feldspar (KAlSi_3O_8) (32 and $45^\circ 2\theta$; PDF#71-0897) is more pronounced in AASB_0 for a Ca/K ratio of 0.32, and upon inclusion of 30% BFS the peak disappeared and appeared apparently at $\sim 50.4^\circ 2\theta$ as the Ca/K increased to 1.75 (**Table 4**) in the more stable and dense $\text{AASB}_{0.3}$ binder. Thus, the presence of potassium oxide in SMF could only contribute to the strength development positively in the presence of CaO or SiO_2 just as the Si/K ratio (EDS) increased from 1.28 (AASB_0) to 1.85 in $\text{AASB}_{0.3}$ binder.

It is also important to note that the presence of more pores accelerates the carbonation of SMF binders (**Fig. 9**). The inclusion of BFS improved the microstructural density thereby causing pore refinement. This is indicated by the disappearance of calcite in specimen $\text{AASB}_{0.3}$ unlike in the specimen AASB_0 , as shown in the relevant X-ray diffractogram in **Fig. 8**.

The FTIR spectra in **Fig. 10** exhibits an asymmetric stretching of CO_3^{2-} at 1400 cm^{-1} in AASB_0 in contrast with its lower intensity in $\text{AASB}_{0.3}$ corroborates less carbonation effects in the latter. There is more -OH precipitation due to the formation of portlandite or bonded

Table 4 Composition of AASB specimens with and without BFS.

Element Wt.%	AASB_0 (Spectrum 11)	$\text{AASB}_{0.3}$ (Spectrum 18)
O	37.1	34.1
C	21.6	19.3
Mn	12.58	7.98
Si	8.34	10.34
K	6.5	5.59
Ca	2.06	9.79
Na	5.42	5.33
Al	0.5	2.04
Ca/Si	0.25	0.95
Ca/Mn	0.16	1.23
Si/Mn	0.66	1.30
Ca/K	0.32	1.75
Si/K	1.28	1.85

water molecule as noted with a deep band of asymmetric stretching of -OH at wavenumber of 3375 cm^{-1} in the AASB_0 system. This points to the weakness of its microstructure unlike the shallow band noticed in $\text{AASB}_{0.3}$ system. Further, a pronounced band at a wavenumber of 1644 cm^{-1} depicting H-O-H symmetrical bending in AASB_0 also supports the weaker microstructure (**Fig. 9**) of the specimen AASB_0 in comparison with a more stable $\text{AASB}_{0.3}$ specimen. The possible attachment of Ca to Si-O- to build Ca-aluminosilicate molecules (Ca-Si-O-T) in specimen $\text{AASB}_{0.3}$ shows the asymmetric stretching of Si-O-T compared to specimen AASB_0 (972 cm^{-1}) with low calcium content. This suggests a low silicate re-organization in AASB_0 with the consequence of the poor strength due to the fragmented skeletal framework.

Fig. 9 SEM and EDS of specimens AASB_0 (top) and $\text{AASB}_{0.3}$ (bottom).

Fig. 10 FT-IR spectra for raw PMs and AASB specimens.

4. Conclusions

In this study, low temperature cured alkaline activated binary binders (AASB) were developed with the synergy of silico-manganese fume (SMF) and blast furnace slag (BFS). The following conclusions can be drawn based on the experimental data obtained in the reported study.

- (1) The spherical nature of SMF improved the workability of the mixture while the workability of the AASB system mainly increased with increasing MnO/CaO ratio. The inclusion of BFS decreased this ratio and hence negatively affected the consistency of the mixture. Extended mixing time also lowered this ratio due to the dissolution of more CaO and the consequent decrease in the consistency.
- (2) The setting time of AASB system depends on the CaO/SiO₂ ratio. The increase in BFS content tend to reduce setting time due to accelerated product formation. The effect of BFS in the product formation is more pronounced at the later age.
- (3) The compressive strength of the developed binders and microstructural density was influenced by the Ca/Si, Ca/Mn, Si/Mn ratios.
- (4) The strength gain in the developed binders is attributed to the dual presence of calcium silicate hydrate (C-S-H) and the formation of stratlingite/gehlenite hydrate (C-A-S-H), and nchwanningite/glaucocroite (C-Mn-S-H) phases.
- (5) The compressive strength increased up to 30% BFS (AASB). A maximum compressive strength of about 45 MPa was noted after 28 days when the specimen (AASB_{0.3}) is cured under the condition of ambient temperature (25°C).
- (6) The inclusion of BFS in AASB_{0.3} system enhanced the reactivity of potassium oxide leading to the formation of potassium feldspar (K-A-S-H) and an increase in K/Ca ratio.
- (7) Excessive carbonation effect and weak hydroxyl compound of alkaline activated silico-manganese fume

could be reduced by the synergistic-interaction of BFS and SMF due to increase in the microstructural density and the reduction in microcracks by pore filling effects.

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