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

Muhammad Nasir1, Megat Azmi Megat Johari2, Moruf Olalekan Yusuf3*, Mohammed Maslehuddin4 and Mamdouh A. Al-Harthi5,6

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).
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 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 AASBx. For example, AASB0.3 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

| Mix ID  | SMF (kg/m³) | BFS (kg/m³) | FA (kg/m³) | FW (kg/m³) | SH (kg/m³) | SS (kg/m³) | FA/PMs | SS/SH | AAs/PMs |
|--------|-------------|-------------|------------|------------|------------|------------|--------|-------|---------|
| AASB₀  | 723.0       | -           | 1301.4     | 14.46      | 103.2      | 258        | 1.8    | 2.5   | 0.5     |
| AASB₀.₁| 650.7       | 72.3        | 1301.4     | 14.46      | 103.2      | 258        | 1.8    | 2.5   | 0.5     |
| AASB₀.₂| 578.4       | 144.6       | 1301.4     | 14.46      | 103.2      | 258        | 1.8    | 2.5   | 0.5     |
| AASB₀.₃| 506.1       | 216.9       | 1301.4     | 14.46      | 103.2      | 258        | 1.8    | 2.5   | 0.5     |
| AASB₀.₄| 433.8       | 289.2       | 1301.4     | 14.46      | 103.2      | 258        | 1.8    | 2.5   | 0.5     |
| AASB₀.₅| 361.5       | 361.5       | 1301.4     | 14.46      | 103.2      | 258        | 1.8    | 2.5   | 0.5     |
(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, AASB 0 (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-Kα 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-

| PMs | Density (g/cm³) | Particle size (µm) | Specific surface area (m²/g) |
|-----|----------------|--------------------|----------------------------|
| SMF | 2.876          | 0.588 29.173 179.958 | 10.1                       |
| BFS | 2.933          | 1.541 13.585 39.211  | 2.5                        |

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 m²/g, respectively. Generally, BFS has relatively smaller particles as shown by the proportions of particle sizes corresponding to d₁₀, d₅₀, and d₉₀ (Table 2) while its size is more uniform as shown in the span ((d₉₀−d₁₀)/d₅₀) 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 d₁₀, d₅₀ and d₉₀ 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 K₂O in SMF is very high, whereas it is insignificant in the BFS. A reverse trend is noted in the quantity of Al₂O₃. Therefore, there is propensity that K₂O and Al₂O₃ together with
MnO, CaO, and SiO₂ in both materials could play a synergistic role in the setting and strength development of the AASB binder.

As shown in Fig. 3, SMF is crystalline in nature and it mainly consisted of quartz (1-SiO₂) (PDF#85-0796), glaucochroite (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 precipitation 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.

$$\text{CaO + CO}_2 \rightarrow \text{CaCO}_3 \ (1)$$

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 (AASBₐ₂₀) 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

| PMs | CaO | MnO | SiO₂ | K₂O | Al₂O₃ | SO₃ | MgO | Fe₂O₃ | Cl | ZnO | P₂O₅ | Ag₂O | TiO₂ | Total | LOI (%) |
|-----|-----|-----|------|-----|-------|-----|-----|-------|----|-----|------|------|-----|-------|--------|
| 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 | - | 95.53 | 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 |

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.

Fig. 5 Flow of the AASB mortar.
The 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 (AASB0.2), 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% (AASB0.3).

3.4 Compressive strength development

Figure 6 shows the compressive strength development in AASBx 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 replacement 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 strätlingite hydrate [4-Ca2Al2(SiO2)(OH)2.2H2O] (PDF#85-8414), nchwaningite [1-MnSiO4(OH)2.H2O] (PDF#83-1006) and rancieite minerals [2-(Mn,Ca)Mn4O9.3H2O] (PDF#085-8414), 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 AASBx 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 AASB0.3. Unlike specimen AASB0, the cracks are localized and disconnected. Further, there is fewer traces of un-reacted 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 AASB0.3, it is evident that the denser mi-
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 (KAlSi₃O₈) (32 and 45° 2θ; PDF#71-0897) is more pronounced in AASB₀ for a Ca/K ratio of 0.32, and upon inclusion of 30% BFS the peak disappeared and appeared apparently at ~50.4° 2θ as the Ca/K increased to 1.75 (Table 4) in the more stable and dense AASB₀.₃ binder. Thus, the presence of potassium oxide in SMF could only contribute to the strength development positively in the presence of CaO or SiO₂ just as the Si/K ratio (EDS) increased from 1.28 (AASB₀) to 1.85 in AASB₀.₃ 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 AASB₀.₃ unlike in the specimen AASB₀, as shown in the relevant X-ray diffractogram in Fig. 8.

The FTIR spectra in Fig. 10 exhibits an asymmetric stretching of CO₃²⁻ at 1400 cm⁻¹ in AASB₀ in contrast with its lower intensity in AASB₀.₃ corroborates less carbonation effects in the latter. There is more -OH precipitation due to the formation of portlandite or bonded water molecule as noted with a deep band of asymmetric stretching of -OH at wavenumber of 3375 cm⁻¹ in the AASB₀ system. This points to the weakness of its microstructure unlike the shallow band noticed in AASB₀.₃ system. Further, a pronounced band at a wavenumber of 1644 cm⁻¹ depicting H-O-H symmetrical bending in AASB₀ also supports the weaker microstructure (Fig. 9) of the specimen AASB₀ in comparison with a more stable AASB₀.₃ specimen. The possible attachment of Ca to Si-O to build Ca-aluminosilicate molecules (Ca-Si-O-T) in specimen AASB₀.₃ shows the asymmetric stretching of Si-O-T compared to specimen AASB₀ (972 cm⁻¹) with low calcium content. This suggests a low silicate re-organization in AASB₀ with the consequence of the poor strength due to the fragmented skeletal framework.

| Element | AASB₀ (Spectrum 11) | AASB₀.₃ (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                  |

Fig. 9 SEM and EDS of specimens AASB₀ (top) and AASB₀.₃ (bottom).
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/SiO$_2$ ratio. The increase in BFS content tends 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 nchwaningite/glaucochroite (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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