Mechanical properties and structure of Fly ash modified Basic Oxygen Furnace Slag based geopolymer masonry blocks

N.T. Sithole[1], F Okonta[2], F. Ntuli[1]

[1] University of Johannesburg, Department of Chemical Engineering, P.O. Box 17011, Doornfontein 2088, South Africa, email address: _fntuli@uj.ac.za_ * corresponding author email address: nastassias@uj.ac.za

[2] Department of Civil Engineering Science, University of Johannesburg, P O Box 524, Auckland Park, 2006, Johannesburg, South Africa, email address:fnokonta@uj.ac.za

Abstract

The objective of this research was to investigate the effect of Fly ash as a source of Si and Na on the mechanical performance of the Basic Oxygen Furnace Slag based (BOFS) geopolymer. BOFS has the lowest SiO_2, Al_2O_3, Na_2O content compared to other aluminosilicate sources (fly ash, metakaolin, granulated blast furnace slag and mine tailings); which have been used to effectively in geopolymer synthesis. SiO_2 and Al_2O_3, contents of BOFS are respectively 5-7 times and 6-8 times lower than those of fly ash, metakaolin and granulated furnace slag (GBFS). This study evaluated the potential use of fly ash as a source of Si and Al to improve the mechanical performance of a BOFS based geopolymer. The influence of varying amount of Coal Fly Ash (FA) (10–50%) on UCS was studied. The effects of several factors on the UCS of BOFS geopolymer were also investigated. The test variables were molarities of sodium hydroxide (NaOH) (5 M, 10 M and 15 M); the solid to liquid ratio (20 %, 25 % and 30 %); and the curing temperature (20°C, 40°C, 80°C and 100°C). It was established that most favorable conditions for the geopolymer synthesis were FA was 10% of the mixture, 5M NaOH and 80°C curing temperature. Attempts has been made to relate the microstructure of BOFS/FA based composite with properties of geopolymer. Aggregation was identified as the main particulate process as established by the evolution of the particle size distribution (PSD) and its derived moments during the geopolymerisation process. The morphology of the particles appeared flaky and fluffy. The developed composite met the minimum requirement of ASTM C34-13 for a structural clay load bearing non exposed masonry and load bearing exposed side construction masonry.

1. Introduction

Ordinary Portland Cement (OPC) is the most usually utilized binder in the construction industry. The manufacturing of OPC presents so many problems. It consumes substantial quantity of natural resources and energy; however Africa's energy crisis continues to grow. It was reported that the production of 1 ton of OPC consumes about 1.5 tons of limestone which approximately emits 1 ton of carbon dioxide (CO_2) to the atmosphere (Pereira et al., 2015 and Zhang et al., 2011). The South African cement industry is one of the major consumers of energy (thermal and electricity) in the country and accounts for 8 % of the total CO_2 emission from industries (Ohanyere, 2013; Pelser, 2017). South Africa is accountable for approximately half the CO_2 emissions in the whole African continent. Worldwide, the cement industry accounts for 7% of all CO_2 generated (Bernard et al., 2017; Bar and Azam, 2017). In line with South African government commitment to find a sustainable carbon low path in the construction of infrastructure to mitigate greenhouse gas emissions, especially CO_2; it is therefore of great national importance to find an alternative binder to reduce the utilization of Portland cement (Falayi et al., 2017).

A feasible alternative is alkaline activation of industrial solid waste like fly ash, steel slags, copper slags produced from numerous industries as an alternative to OPC. The utilization of industrial waste as the supplementary cementitious materials (SCMs) is well documented in literature; specifically on the article entitle "An assessment of microcracks in the Interfacial Transition Zone of durable concrete composites with fly ash additives", Composite Structures, 2018. An estimate of about 36 million tons of coal fly ash is generated annually in Southern Africa of which 95% is usually disposed of in landfills (Petrik, 2003). On the other hand approximately 2.4 million tons of BOFS is produced annually in South Africa of which about 50 % is recycled the remainder being disposed in heaps (Sithole, 2015). The rapid growth of industrial development has resulted in the generation of large amounts of BOFS and fly ash (FA) of which the available land for land-filling of extensive amounts of waste is diminishing everywhere throughout the world (Reutter et al., 2004). The land loaded with industrial waste has turn out to be a major source of air pollution, water and soil, and more adversely affects the human well-being, and the growth of vegetation. At the same time, dumping of industrial waste

has become more problematic and costly owing to the increasing strict environmental rules and regulations and shortages of appropriate close disposal sites (Mahmoud, 2011). The utilization of both FA and BOFS is still very low compared to other countries producing even more quantities like China and India. This therefore calls for the beneficiation of BOFS and FA in order to reduce its environmental impact and make it a natural resource for other valuable products. The utilization of FA as a source of silica and alumina in other waste material such as tailings, granulated furnace slag, metakaolin is well known and reported in literature however; the use of FA as a secondary source of silica in BOFS for production of lightweight masonry is scarce and limited. Studies by (Golewski 2017 and Golewski 2018) reported that it is possible to make green composites by replacing cement with FA additive in the amount of 20 and 30% of mass of cement significantly increases the compressive strength and fracture toughness. However, FA additive in the amount > 30% of mass of cement drastically reduces the compressive strength and fracture toughness. This paper attempts to utilize two waste materials (FA and BOFS) to produce construction block and at the same time investigating whether supplementing BOFS with SiO_2 and Al_2O_3 from fly ash without addition of silicates will enhance the UCS of the BOFS based geopolymer; as fly ash has high content of SiO_2 and some Al_2O_3. This study also attempts to synthesize fly ash /BOFS based lightweight masonry by just alkaline activation. The influence of varying amounts of fly ash (10–50%) on the UCS has also been investigated. This paper therefore presents a possible beneficiation and utilization of FA/BOFS in order to reduce their environmental impact and make it a natural resource for other valuable products.

2. Methodology

2.1. Materials and Methods

BOFS was obtained from ArcelorMittal (South Africa). Fly ash was supplied by Camden power station (South Africa). Sodium hydroxide was supplied by Rochelle Chemicals South Africa. Sodium-based solutions were chosen because they are cheaper and they were reported to have a greater capacity to liberate SiO_2 and Al_2O_3 monomers (Zhang 2003). It is also reported that Na^+ cations have better zeolitization capabilities in geopolymerisation process (Duxson 2007). The sodium hydroxide solution was prepared using de ionized water. The mass of NaOH pellets in a solution were varied from 5 M to 15 M. Table 1 shows the chemical and geotechnical properties of BOFS and FA.

BOFS has the lowest SiO_2, Al_2O_3, content compared to which has been used to achieve an efficient geopolymer synthesis. SiO_2 and Al_2O_3, contents of BOFS are both 5 times lower than those of FA. However, BOFS CaO content is 4 times higher to that of FA. This high Ca content in BOFS will play a role in the development of C -S-H gel during alkaline activation. The development of C–S–H gel could aid to make a uniform and thick geopolymer paste (Kumar et al., 2009). This study took advantage of the Si/Al in FA to supplement BOFS to address the issue of low SiO_2 and Al_2O_3 in BOFS. FA is readily available and has been used as a source of silica in other waste material and it's well documented except in BOFS based geopolymers. FA has been used to provide Si^+ ions as a secondary source of silica and the Na^+ ions in it and in BOFS plays a vital role in the formation of geopolymer by acting as charge balancing ions; and also helps to enhance strength development (Part el al., 2015). It is also noticeable that BOFS compositions are similar to those of clinker. These compositions revealed that BOFS exhibits cementitious properties. It has been reported by several researchers that BOFS is a weak Portland cement clinker (Shi 2002), due to the fact that C_3S content in BOFS is very low and sometimes the C_3S is not in the BOFS sample at all (Tsakiridis et al., 2008). Based on the properties of BOFS and FA revealed by XRF analysis; this study attempted to investigate the feasibility of synthesizing Lightweight masonry from BOFS/FA via alkaline activation (NaOH solution).

2.2. Equipment

The chemical composition of the geopolymer was determined using X-Ray fluorescence (XRF; Rigaku ZSX Primus II). FTIR (Thermo scientific IS10) was used, to characterize of BOFS before and after the alkaline activation. XRD was used to identify the mineralogical phases on the BOFS before and after alkaline activation. Finely grounded BOFS and FA was carbon coated and mounted onto a Scanning Electron Microscope (SEM; Tescan Vega 3 XMU 1) at 7.5 kx to investigate the microstructure of the BOFS/FA before and after alkaline activation. The geopolymer particle size distribution was obtained using a Laser diffraction method (Malvern mastersizer 2000) and the data generated was used to determine the external surface area, the evolution by particle number and volume distribution.

2.3. Geopolymer paste preparation

283 g of (Fly ash and BOFS) were mixed in different proportions with NaOH solution to prepare a paste as illustrated in Fig. 1. The Fly ash quantity was varied from 0% to 50%. The solid/liquid ratio was varied from 20 to 30 %; this was necessitated by the need to have a workable slurry. The NaOH concentration was varied from 5 to 15 M and temperature from 40 to 100°C. The optimum blending ratio FA/ BOFS was used to investigate the optimum solid/liquid ratio as shown in Table 2, thereafter the optimum NaOH concentration was investigated and was used to find the optimum temperature. The paste was then transferred into a 50 mm ×50 mm × 50 mm mold.

2.3.1. Curing

The prepared pastes were allowed to set and to harden for 24 hours before they were removed from the mould. The hardened samples were then cured at 80°C until they were dry. The solid/ liquid ratio was kept at 20%. This S/L was optimum as the paste was more workable.

2.3.2. UCS testing

To determine the UCS of the hardened samples; the geopolymer samples were placed within the compression machine, and the load of the compression machine was applied until the geopolymer sample fizzled.

2.4. Open porosity

The geopolymer samples were weighed after curing then they were soaked in a water bath for 24 h. 24 h had been determined as the time when an increase in mass of wet specimen was less than 1%. After 24 h the geopolymer samples were removed from water and were wiped using a soft cloth to get rid of any visible water. The wet geopolymer sample were weighed within 5 min after being removed from the water. Open porosity, f, was then calculated using equation (1) as follows (ASTM C373 – 14):

$$f = \frac{Ws - Wd}{V\alpha}$$

(1)

where *Ws* is the mass of the soaked specimen, *Wd* was the mass of the dry specimen, *V* was the volume of the specimen and α was the density of water.

2.5. Wet compressive strength

The specimen was weighed after curing then, it was soaked in water for 24 h. After 24 h the specimen was removed from the water and wiped with a soft cloth then weighed. Immediately after weighing the wet specimen was then tested for UCS. A similar procedure was also used to assess whether the LWCB is expansive.

2.6. Water absorption tests

The specimen was immersed in water for 24 h. After 24 h the specimen was removed from water and wiped with soft cloth. The wet specimen was weighed within 3 minutes of being removed.

3. Results and discussions

3.1. Effect of variation of Fly ash content

Fig.2. shows the variation of UCS of the BOF slag based geopolymer with 10 to 50 % FA content in the mixture after 7 days curing. UCS was found to decrease with an increase in FA content. The decrease in FA content resulted in an increase in UCS. The optimum FA content which gave the highest UCS was at 10 % FA giving UCS of 4.4 MPa. However, higher strength than 4.4 MPa were expected but that was not the case. This is due to the fact that high content of calcium; in this case in BOFS has been reported to hinder Fly ash geopolymerisation process; as it result in high viscosity and also contribute to the rapid setting time, low workability; which in turn results in low UCS (Rahman 2015). Fig.3. shows the XRD pattern of the BOFS based geopolymer at different FA blending ratios.

Alkaline activation and addition of FA from 20-30% resulted in an increase in intensity of the tricalcium silicate hydrate which is seen in the XRD diffractogram at around 27°. Tricalcium silicate hydrate is largely responsible for strength development in the cementation process. The crystalline peak (dicalcium silicate hydrate) appearing on the 10% FA diffactogram at around 42 ° disappeared as the FA content was increased from 20%-50%. This peak presents Calcium silicate hydrate (CSH). Though the CSH peak is normally at 29° and 50°, the peak at 46° can also be attributed to CSH (Elena. and Lucia, 2012). Basal shift of calcium hydrates might be due to low reactivity of FA which is hindered by high calcium content in the BOFS. As the FA content is increased to 40-50% the UCS was further impaired which might be due to the reduction and

disappearance of peaks at 42 ° and 51 °. This results supports the results obtained in Fig.2. FTIR analysis is presented in Fig. 4. to check whether any further information could be obtained to understand the reason for declining UCS with an increase of FA content.

Fig.4. shows the IR spectra of BOFS when blended with FA variation from 10% to 50%. The IR spectra do not clearly show any significant spectral differences which can be correlated to declining UCS when fly ash content is increased. However there was a minor shift to higher frequencies at 876 cm^{-1} and a minor shift to lower frequencies at 1445 cm^{-1} as the FA content was increased from 10% to 50%. Fig.5. shows the SEM of the geopolymer composites with variation of FA %.

Fig. 5. Shows the SEM Micrographs for blending BOFS with variable content of FA. 10 % FA composite resulted in the formation of a prism flaked shaped, with a flattened dense and a very little visibility of spherical particles. This therefore accounts for the high UCS at 10% FA. At 20% to 30% FA the particles appeared spherical with a less dense, crumbled, fragmented structure formed which accounts for reduction in UCS at 20 % and 30%. At 40% the spherical, crumbled and fragmentation of the structure became more pronounced than on the 20-30% which explains the further impairment of strength. At 50% the particles appears to be agglomerated yet fragmented; there were micro-cracks observed which explains why the UCS was at its lowest compared to other mixed designs. The micro cracks might be due to the high content of FA which reduces the workability of the paste. It was observed that this paste hardened faster as compared to other pastes.

3.2. Effect of NaOH concentration on UCS

Fig. 6. Shows the influence of NaOH on the 7 days UCS. The results shows that an increase in NaOH concentration from 5 M to 15 M resulted in a decrease in UCS. The increase in UCS with increasing NaOH concentration is mainly due to lower degree of silica and alumina leaching (Part el at., 2015). Fig.7. shows the FTIR spectra of BOFS/FA geopolymer composites with increasing concentration of the alkaline activator.

A broad shallow component appearing at 2359-3560 cm^{-1} represents to stretching and deformation vibrations of hydroxyl functional group (OH/OHO). These bands reveal that water molecules are trapped between the rings of the geopolymer network. The peaks of bands at 1437 cm^{-1}- 1456 cm^{-1} were due to stretching vibrations of O-C-O due to carbonation reaction. The carbonation reaction occurred due to high alkaline NaOH aqueous phase where it reacted with CO_2 from the atmosphere (Yahya et al., 2015). In addition it can be seen that the intensity of the bands at 1437 cm^{-1}- 1456 cm^{-1} increased with increasing NaOH concentration. The existence of Si-O-Si symmetric vibration bands were found at area 872cm^{-1} to 993 cm^{-1}. There was a shift of this band to lower frequencies attributed to dissolution of the alumina silica gel which might be caused by low reactivity of FA due to the high calcium content of BOFS in the BOFS/ FA system which ideally was supposed to supplement Si and Al to the BOFS but due to high calcium hindered the reactivity of FA. Fig. 8. shows the SEM Micrographs of the geopolymer composites at different molar concentration.

It can be seen that at 5 M, a flat, dense matrix and complete geopolymerisation was formed as compared to 10 M and 15 M micrographs. However, incomplete geopolymerisation was still observed. The 5 M micrograph reveals that BOFS/FA reacts homogenously with the alkaline activator which resulted in an increase in UCS. An increase of the alkaline activator concentration from 5 M to 10M /15M resulted an incomplete geopolymerisation and a wide structure formed. This is due to excess Na which obstructs water evaporation and structure

formation thus in this case a loose/ wide structure was formed which accounted for lower UCS (Liew et al.,2011).

3.3. Effect of solid to liquid ratio on UCS

Fig.9. shows that the UCS of the geopolymer composites decreased with the S/L ratios. The highest UCS was obtained when the S/L ratio was 20%, and the lowest UCS was obtained when the S/L ratio was 30%. These results indicate that a decrease in the S/L ratio promotes higher UCS. The 20% S/L ratio gave the most favorable workability and as a result optimal UCS was attained. The S/L less than 20% reduces the workability of the mixture. Optimum workability resulted in homogeneity of the paste. The UCS measured confirms that the activator content was optimum at 20% S/L ratio that permits dissolution of BOFS/FA while not hindering the polycondensation rate during the geopolymer synthesis ((Liew et al., 2011). The increase of S/L ratio from 20% to 30% resulted in the mixture having very low gelation to permit molding and was of much reduced workability for good compaction, respectively (Yahya et al., 2015).

3.4. Effect of curing temperature

A change of temperature from 20°C to 80° resulted in 68% increase in UCS of BOFS: FA based geopolymer composite showing that; the initial temperature increase accelerated the hydration reaction. At lower curing temperature (20°C to 40°C), the dissolution of FA/BOFS particles was slow, the geopolymer gels developed slowly and geopolymer samples were still gelatinous and tacky (Mo et al., 2014). Based on experimental observation the specimens cured at 20°C and 40°C took longer to set and harden as compared to the specimens cured at 80°C and 100°C which set hardened within 24 h. This observation indicate that elevating the curing temperature increases the extent of dissolution of aluminosilicate species and accelerates the formation of a hardened specimen particularly in the initial-stage of geopolymerisation process. This proved

that heat treatment is required to expedite the rate of development of the UCS of the geopolymer matrix. The increase in temperature from 80°C to 100°C brought about a slight decrease of 5.6% in UCS, this is due to the viscosity increasing rapidly at the onset of polycondensation. The aluminate species released by dissolution of the aluminosilicate source was immediately consumed and then reacted (Mo et al., 2014). Therefore, the setting rate of the geopolymer paste in the initial and final stages was very fast and short. Furthermore, dehydration at higher curing temperature resulted in micro- crack and contraction of geopolymer gels as shown on the SEM micrograph (Mo et al 2014). Fig. 11. Shows the XRD diffactogram of the geopolymer composite cured at different temperatures.

As the curing temperature is increased from 20 °C to 100°C the intensity of the Ca_3SiO_4 peak became more crystalline at around 18°. This peak is responsible for hardening and setting of the geopolymer paste. Hence the increase in temperature resulted in an increase in UCS. However, at 100° C there was a slight decrease of 5.6% in UCS due to dehydration which resulted in micro-crack and contraction of geopolymer gels as shown on the SEM micrograph (Fig. 18.) (Mo et al 2014). It was observed that the intensity of the $CaSi_2$ peak at around 29° for geopolymer cured at 80°C was more crystalline compared to other curing temperatures. This peak presents the CSH gel which is responsible for strength development which explains the high UCS curing at 80°C. Fig.12. displays the FTIR analysis of the geopolymer composite cured at different curing temperatures.

When curing temperature is increased there was a shift of stretching vibrations Si-O-Si which are found at 870-900 cm^{-1} to higher frequencies. This indicates the formation of a new product (the aluminosilicate gel phase) due to dissolution of BOFS/FA mainly BOFS in alkaline activator. The broad component at 3300-2351 cm^{-1} for 20°C spectra is sharp compared to the elevated temperatures around the same wavenumber. The existence of this broad component represent the hydroxyl functional group which reveal that there were water molecules that were adsorbed on the surface or trapped in the geopolymeric matrix. In addition, the vibrations bands O-C-O were located at 1410 -721 cm^{-1}. There was a shift of bands from lower 869 to 873-877 cm^{-1}

frequencies attributed to dissolution of the alumina silica gel. Fig. 13. shows SEM images of the geopolymer composites cured at different temperatures.

The geopolymer composite cured at 20°C shows more incomplete geopolymerisation which made sense as at this temperature geopolymerisation starts slowly to form a geopolymer matrix. The morphology of the particles appeared flaky, fluffy and wide. As the curing temperature is increased to 40° C a prism shaped, with a spherical closely packed dense structure is formed; this accounted for the UCS increase compared to when the temperature was 20°C. At 80°C SEM image reveals a more distinct closely packed and denser geopolymer matrix as compared to the 40°C image, almost a complete geopolymerisation reaction occurred which could be the reason for development of high strength. The SEM image at 80°C shows a spherical closely packed and dense geopolymer matrix formed with some micro cracks which might be caused by curing at elevated temperatures resulting in a rapid setting rate and hardening process thus leading to micro cracks, thereby impairing the strength of the geopolymer. Fig.14 shows the evolution of PSD by volume distribution and Fig.15 shows the evolution by number distribution of the composites cured at different temperatures.

Fig. 14. Shows the PSD by volume distribution of the geopolymer composite cured at different temperatures. The PSD at all different curing temperatures was uni-modal with modal sizes of approximately 100 μm and 1000 μm. There was a gradual shift to the left indicating a generation of smaller sized particles while the decrease in volume % in relation with the 20°C curve indicating particle growth of the geopolymer composites forming bigger particles was more prominent at lower curing temperature.

Fig 15 was used to determine whether curing at low or elevated temperature would result in production of smaller particles or that of larger sized particles. It can be seen that composites became less aggregated with increasing curing temperature. This might be due to rapid dehydration and excessive shrinkage from the geopolymer matrix which resulted in subsequent loss of structural integrity of the BOFS/FA geopolymer matrix.

3.5. Durability using wet compressive strength

Fig. 16. shows the UCS of the geopolymer after soaking for 24 h. There was an 18%, 19% and 19% reduction in UCS for 5 M, 10 M and 15 M respectively. The results revealed that 5 M Fly ash/NaOH BOFS geopolymer is more durable compared to the 10 and 15 M composites. Table 4 shows the geopolymer composites properties at different concentration of the alkaline activator.

The 15 M geopolymer composite had the lowest water absorption rate (13.3%) and the least reduction in UCS after soaking (2.3 MPa) as compared to the 5M and 10 M geopolymer composite. The lowest water absorption rate is due to high bulk density and low porosity related to such composites (Freidin and Erell, 1995). The low porosity is also related to high contacts between particles hence less water can be absorbed. The 5M geopolymer composite outperformed the 15M and 10 M by still maintaining high UCS. The 5M geopolymer composite satisfies the minimum requirement of ASTM C34-13 for a structural clay load bearing non exposed masonry and load bearing exposed side construction masonry (Zhang, 2013). The geopolymer composites also satisfy the minimum requirements for the South African building council standards for non-facing plastered brick (SANS 227:2007).

4. Conclusion

Camden Power Station FA can be used as a secondary source of silica to supplement BOFS. It was found that the optimum mix design to successfully alkali activate BOFS/FLY ASH were 5 M of NaOH, L/S ratio of 20%, BOFS: FA ratio 90:10 and the curing temperature 80 ºC. Based on the results obtained the following conclusion can be drawn;

1. BOFS/FLY ASH can be used as raw material for making alkaline activated composites
2. The developed composite met the minimum requirement of ASTM C34-13 for a structural clay load bearing non exposed masonry and load bearing exposed side construction masonry with average UCS of 4.4 MPa.

3. This paper therefore provides an opportunity for the use of BOFS/FLY ASH as a structural clay load bearing masonry and load bearing side construction mansory although there is still need to allow the curing to take place at room temperature to reduce energy consumption associated with the alkaline activation process.

4. This research also demonstrates that it is possible to use large amounts of hazardous and non-hazardous wastes (FA and BOFS) to develop new products that satisfies ASTM C34-13, C129-14a and South African standard (SANS227: 2007) as well as to minimize environmental impact.

5. Based on the evolution of the particle size distribution (PSD) and its derived moments the dominant particulate process identified during the alkaline process was aggregation. The morphology of the particles appeared flaky and fluffy.

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