Development of lightweight construction blocks by alkaline activation of BOF slag

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Abstract

Large quantities of basic oxygen furnace (BOFS) are dumped in landfills of which the available land for land-filling of large quantities of waste is reducing all over the world. It is therefore important to develop processes which beneficiates solid waste; BOF slag specifically. The present study attempts to investigate the potential to synthesize BOF slag based light weight construction blocks. The effects of several factors on the UCS of BOF slag based light weight construction blocks (LWCB) was 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 %); the sodium silicate (Na_2SiO_3) to NaOH ratio (0.5:1, 1:1, 1.5:1, 2:1, 2.5:1 and 3:1); the curing temperature (40°C, 80°C and 100°C). It was found that optimum synthesis conditions were 5M NaOH, 80°C and 1:1 Sodium Silicate: NaOH ratio. The LWCB composite met the minimum requirements for ASTM C34-13, C129-14a and South African standard (SANS227: 2007).

Keywords: Basic oxygen furnace slag, lightweight construction block, alkaline activation, geopolymer, porosity.

1.1. Introduction

Ordinary Portland Cement (OPC) is the most commonly used binder in the construction industry. The manufacturing of OPC presents so many problems. It consumes significant amount of natural materials 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 responsible for nearly half the CO_2 emissions for the entire continent of Africa. 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 by-products like fly ash, ground granulated blast furnace slag, steel slag, copper slag generated from various industries as an alternative to OPC. An estimate of about 2.4 million tons of BOF slag is produced annually in South Africa of which about 5 % is recycled the remainder being disposed in heaps (Sithole, 2016). Compared to other countries like China and USA which produces thrice the amount of BOF slag; only 22% is utilized. The utilization of BOF slag in Africa is still limited and it is still particularly low in South Africa. According to Kambole et al., 2017 one of the resulting factors for limited utilization of BOF slag is that there are very limited studies on the utilization of BOF slag in Africa, specifically in Southern Africa. This may partly explains why most Southern African construction specifications do not cater for slags, resulting in the limited use of this recyclable material resource in construction. There is a need for Southern Africa to consider increased use of BOF slag in construction (Kambole et al., 2017).

The use of BOF slag as a cementing material to replace Portland cement and as a hydraulic binder is well documented (Belhadj et al., 2014; Wang and Yang 2010; Brand et al., 2015; Ren et al., 2017). In addition the use of BOF slag aggregates for road construction is well documented in literature (Chen et al., 2016; Haritonovs et al., 2013; Motz and Geiseler, 2001, Egesi, 2012; Xue, 2006; Taherkhani, 2015; Ioannou et al., 2013). Some researchers (Belhadj et al., 2014; Brand et al., 2015; Taherkhani, 2015; Ren et al., 2017) reported that BOF slag is an expansive material due to the presence of free lime in BOF slag, and thus it cannot be used for construction works. However, it has also been reported that slag expansion could be avoided if the slag particle sizes of limited to 13.2 mm for construction work (Ameri et al., 2013; Yüksel, 2017). Furthermore BOF slag expansion might be dependent on the type of minerology and chemical nature. They would differ as per manufacturing processes and the source of the raw material. It is noted that also

investigations and studies regarding the production of BOF slag based lightweight geopolymers are scarce and limited. The present study attempts to fully beneficiate BOF slag by development of light weight construction blocks. The utilization of BOF slag in making LWCB is rare and not well documented in literature. LWCB are construction composites with a density of less than 1680 kg/m^3 and a minimum unconfined compressive strength (UCS) of 4.1 MPa (ASTM C129-14a). The advantage of LWCB is that it reduces the dead load of a building (Falayi et al., 2017).

2. Methodology

2.1. Materials and Methods

BOFS was obtained from ArcelorMittal South Africa. Sodium silicate solution was supplied by Sigma Aldrich South Africa. Sodium hydroxide was supplied by a chemical company South Africa. Table 1 shows the chemical and geotechnical properties of BOF slag.

Table 1: Chemical and geotechnical properties of BOF slag

Parameter	BOF slag
pH	12.4
Specific gravity	3.25
% CaO	51.81
% Al_2O_3	3.5
% SiO_2	7.7
% MnO	4.188
% Fe_2O_3	27.58
% Gravel	0
% Sand	77.92
% Fine	11.12
% Silt	10.96
Liquid limit	76
Plastic limit	non plastic
Shrikange limit	non shrinking
MDD (kg/m3)	2265
OMC (%)	10.58

The chemical and geotechnical characterization was performed prior to alkaline activation to better understand the properties of BOF slag. The specific gravity of basic oxygen furnace slag was found to be 3.25. This specific gravity is greater than the specific gravity of natural inorganic soils which

typically ranges between of 2.6 to 2.9. This is attributed to the high Fe_2O_3 content in BOF slag. The main constituents of BOF slag are calcium oxide (CaO), iron oxide (Fe_2O_3), silicon dioxide (SiO_2), and magnesium oxide (MgO). The Fe_2O_3 content for this sample is higher than those of most BOF steel slags reported in literature (Yildirim et al., 2015). However, according to Shen et al. (2009), the Fe_2O_3 content of BOF steel slag can be as high as up to 38% depending on the amount of oxidized iron that cannot be recovered during the conversion process of molten iron to steel. BOFS has the lowest SiO_2, Al_2O_3, content compared to other aluminosilicate sources (fly ash, metakaolin, granulated blast furnace slag and mine tailings) which have been used to achieve an efficient geopolymer synthesis. SiO_2 and Al_2O_3, contents of BOF slag are respectively 5-7 times and 6-8 times lower than those of fly ash, metakaolin and GBFS. In this study the problem of low SiO_2 and Al_2O_3 in BOF slag is addressed through Na_2SiO_4 addition. The addition of sodium silicate provided Si^+ ions as a secondary source and the Na^+ ions played a vital role in the formation of geopolymer by acting as charge balancing ions; and also help to enhance strength development (Part el al., 2015). It is also noticeable that BOF slag compositions are similar to those of clinker. These compositions revealed that BOF slag exhibits cementitious properties. It has been reported by several researchers that BOF slag is a weak Portland cement clinker (Shi 2002), due to the fact that C_3S content in BOF slag is very low and sometimes the C_3S is not in the BOF slag sample at all (Tsakiridis et al., 2008). BOF slag sample used in this study reveals that C_3S is present since the CaO/Si ratio is greater than 2.7 (Shi 2002). The C_3S is well known to be responsible for hardening, initial setting and early strength development of Portland cement concrete (Reddy et al., 2006). Additionally the high Ca content in BOF slag played a role in forming C-H-S gel during alkaline activation. The formation of C–S–H gel may help to make a dense and homogeneous geopolymer paste (Kumar et al., 2009). Based on the properties of BOF slag revealed by XRF analysis; this study attempted to investigate the feasibility of synthesizing LWCB from BOF slag via alkaline activation. The low Atterberg limits could be classified as a sandy material with non-plastic and non-shrinking behavior.

2.2. Equipments

X-Ray fluorescence (XRF; Rigaku ZSX Primus II) was used to determine the chemical composition of the geopolymer. FTIR (Thermo scientific IS10) was used to characterize of BOF slag before and after the alkaline activation. XRD was used to identify the mineralogical phases on the BOFS before and after alkaline activation. The BOFS morphology was captured using a

Scanning Electron Microscopy (SEM; Tescan Vega 3 XMU 1) to investigate the micro-structure of the BOF slag before and after alkaline activation.

2.3. Geopolymer paste preparation

1100 kg BOFS and NaOH solution varied from 5 to 15 M as shown in Table 2 were mixed keeping the S/L ratio fixed. After forming a workable slurry, the slurry was poured into a 100 x 100 x 100 mm^3 mould. The cast sample was allowed to set and was removed from the mould when it was stiff enough for demolding. The hardened samples were then cured at 80ºC until they were dry. Molding was done in triplicate.

Table 2 Mix ratio of NaOH and BOFS

% BOFS	*NaOH Concentration (M)*	*Solid/Liquid ratio*
100	5	30
100	10	30
100	15	30

2.3.1. The effect of solid/liquid ratio

1100 kg of BOFS was mixed with 5 M NaOH and cured under different solid/ liquid ratios and curing regime as shown in Table 3.

Table 3 Solid/ liquid ratio variation

% BOFS	*NaOH Concentration (M)*	*Solid/Liquid ratio(%)*	*Na_2SiO_3/NaOH ratio*
100	5	20	2:1
100	5	25	2:1
100	5	30	2:1

2.3.2. The effect of temperature

The BOFS was mixed with 5M NaOH and cured under different temperatures and the curing regime as shown in Table 4

Table 4 Curing temperature variation

% BOFS	*NaOH Concentration (M)*	*Solid/Liquid ratio (%)*	*Na_2SiO_3/NaOH ratio*	*Temperature (°C)*
100	5	20	2:1	40
100	5	20	2:1	80
100	5	20	2:1	100

2.3.3. The effect of Na_2SiO_3/NaOH ratio

The BOFS was mixed with Na_2SiO_3 and NaOH under different ratios as shown in Table 5.

Table 5. Variation of Na_2SiO_3/ NaOH ratio

% BOFS	*NaOH Concentration (M)*	*Solid/Liquid ratio (%)*	*Na_2SiO_3/NaOH ratio*	*Temperature (°C)*
100	5	20	0.5:1	80
100	5	20	1:1	80
100	5	20	1.5:1	80
100	5	20	2:1	80
100	5	20	2.5:1	80

2.4. Open porosity

The specimens were weighed after curing then they were immersed 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 specimens were removed from water and were wiped using a soft cloth to remove any visible water. The wet specimens 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):

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

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 discussion

3.1. Effect of using different particle size

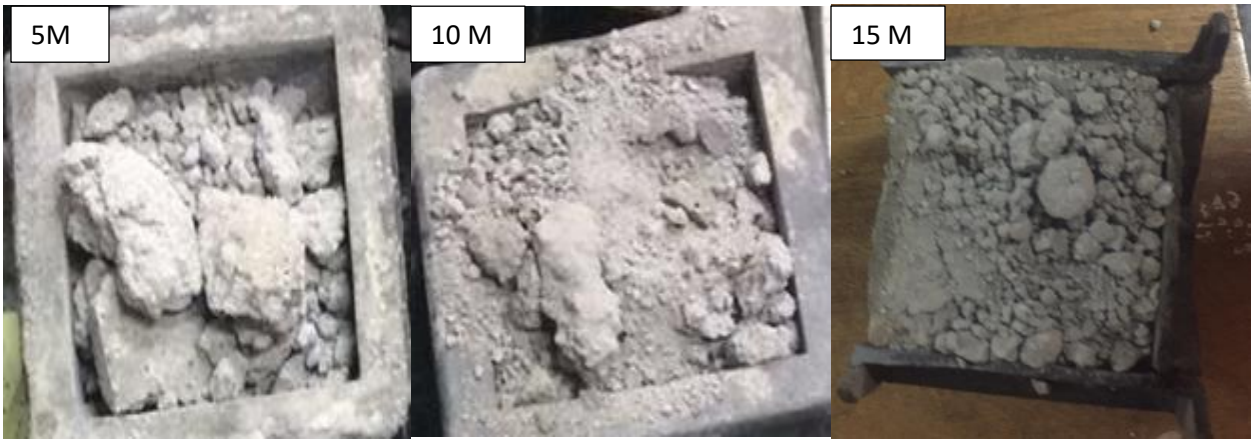

Fig. 1. Picture of casting at different particle size with variation in NaOH concentration

Fig 1 shows the resulting product after attempting to make a geopolymer using different sizes of BOFS ranging from gravel to sand. The geopolymer did not form, the dried paste crumbled during demolding process. The BOFS was then milled to a finer particles size, it has been reported by several authors that uniform particle sizes enhances and increases the mechanical properties of geopolymer gel (Duxson et al., 2005; Part et al., 2015;Issabella et al., 2003; Ryu et al., 2013).

3.2. Effect of NaOH concentration on UCS

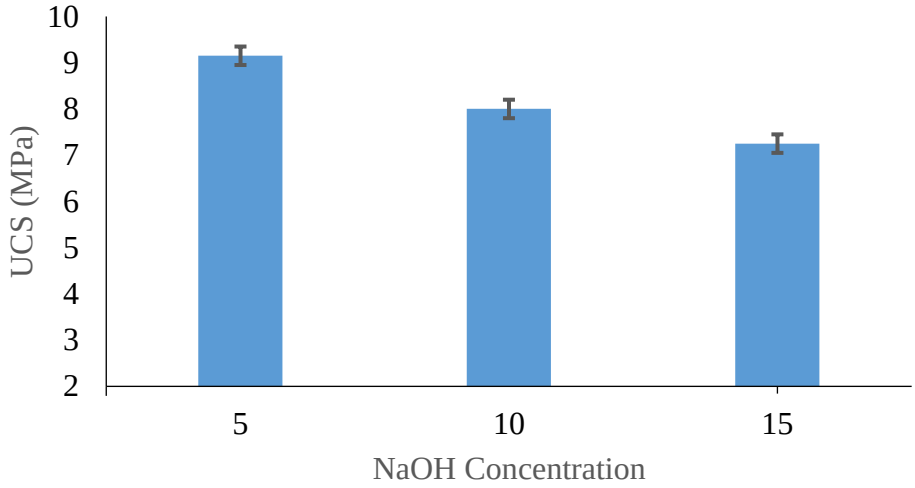

Fig 2. Effect of NaOH concentration on UCS

Fig 2 shows that indeed a uniform particle sizes ranging from 0.425-0.075 micron of BOF slag can be synthesized a BOF slag based geopolymer with low content of silica to alumina. The highest strength achieved under NaOH activation is 9.2 MPa. There was a decrease in UCS with increase in NaOH concentration. This was due to reduced workability of the paste formed. The geopolymer

paste hardened faster at 15 M than 5 M and 10 M reducing the time for dissolution of aluminosilicate species. It was also observed that as the concentration of NaOH was increased the paste tend to be less workable meaning that the paste needed more water to increase the workability which might lead to an increase in the UCS.

3.3. XRD Analysis

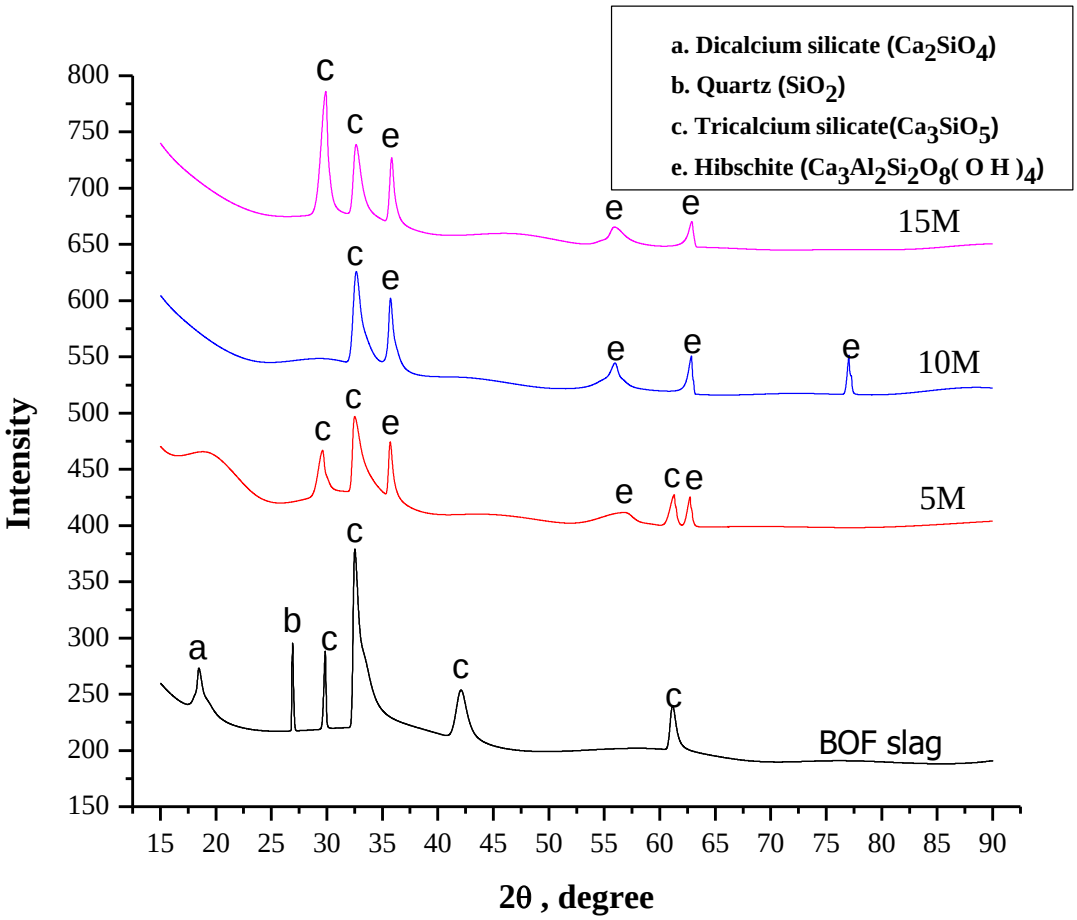

Fig 3 XRD patterns before and after alkaline activation

Fig 3 shows the diffactograms of BOF slag before and after geopolymerisation. Upon alkaline activation by NaOH (5M, 10M and 15M) the quartz (SiO_2) peak at 26.9°and dicalcium silicate (Ca_2SiO_4) at 18.4° disappeared. The disappearance of these peaks show a rapid reactivity of BOF slag. The tricalcium silicate was slightly consumed during alkaline activation; as its intensity at 32.5 decrease with a decrease in NaOH concentration. The high strength at 5 M might be due to more tricalcium silicate being consumed and reacting resulting in hardening, initial setting and early strength development of BOF slag geopolymeric paste. As the peak intensity of C_3S, C_2S

and SiO_2 decrease; in parallel a hydrated phase; hibschite is formed. There are more hibschite peaks formed at 10 M and 15 M; their peak intensity are even more pronounced as compared to those at 5 M. This might be due to excess hydroxide ions concentration which caused the geopolymeric gel to precipitate, hindering the poly-condensation process; hence the decrease in strength at 15 and 10M. Table 2 shows the quantitative phase amounts from XRD.

Table 2: Quantitative phase amount

	BOF slag	5 M	10 M	15 M
Dicalcium silicate Ca_2SiO_4	14.9	2.5	3.8	4.7
Tricalcium silicate (Ca_3SiO_5)	14.4	4.6	6.5	7.5
Quartz (SiO_5)	7.18	0.51	1.56	2.76
Calcium Oxide (CaO)	13.9	3.2	4.4	7.5
Brownmillerite ($Ca_2(Al,Fe)_2O_5$	38.3	5.3	7.56	8.6
Hematite Fe_2O_3	11.3	2.9	2.7	2.7
Hibschite [$Ca_3Al_3Si_2O_8(OH)_4$]	0	70.63	77.01	80.2
Zeolite Y, Na ($Na_2Al_2Si_{4.5}O_3$* H_2O)	0	10.37	1.7	3.8

Table 2 supports the XRD diffactogram results; that as the concentration of NaOH decrease the more the constituents namely; tricalcium silicate, dicalcium silicate, silica and Brownmillerite were consumed. The phenomena reveals the reactivity of BOF slag in forming the two zeolites; zeolite Y, Na and Hibschite which are responsible for strength development.

3.4. SEM analysis

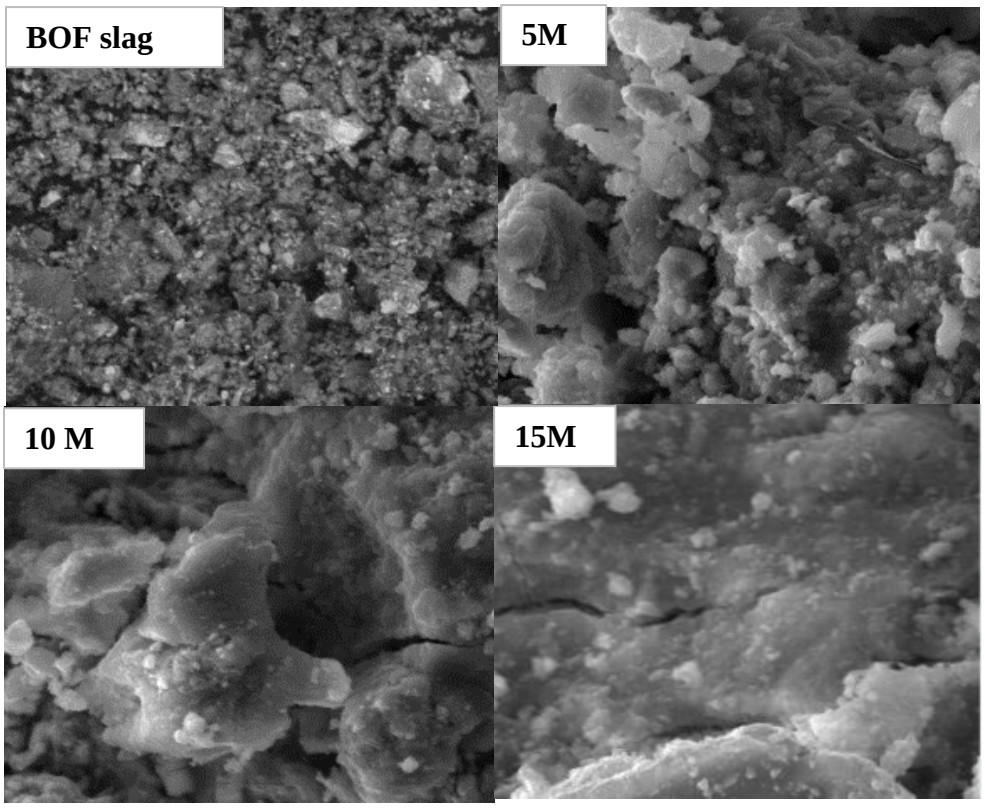

Fig 4 SEM micrographs before and after alkaline activation

Fig 4 shows the micrographs of BOF slag before and after alkaline activation. Upon alkaline activation the particles appear closed packed flaky and fluffly which reveals that geopolymerisation took place. The 5 M micrograph shows there was formation of a lightly whitish colored particles which represent zeolite structure (Zeolite Y, Na); this is supported by the quantitative analysis using XRD (Falayi et al., 2017). The formation of the zeolite might have been incorporated with the hibschite on the XRD. The increase in strength upon 5 M alkaline activation might be as result of the formation of this zeolite as presence on zeolitic material increases the strength of a geopolymer matrix (Falayi, 2017). The decrease of strength at 10 M and 15 M was a result of cracks as shown on the micrographs. Both the cracks are related to the substantial loss of moisture from the geopolymer matrix which resulted in excessive shrinkage during drying and subsequent loss of structural integrity of the BOF slag geopolymer matrix.

3.5. FTIR analysis

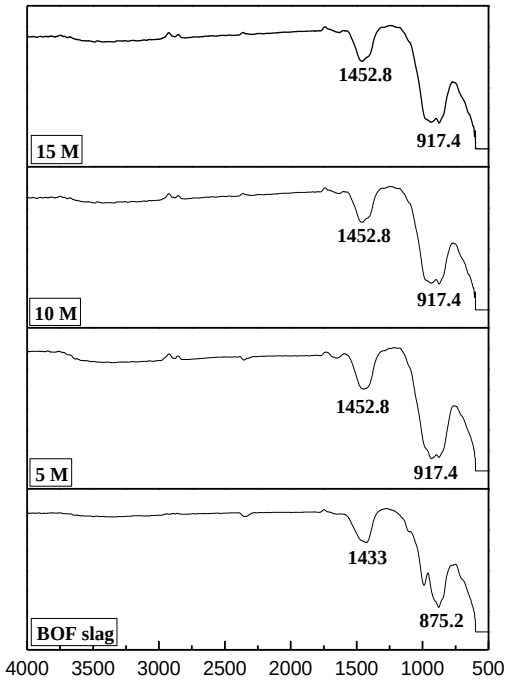

Fig 5 FTIR analysis of BOF slag before and after alkaline activation

Fig. 5. shows the IR bands of BOF slag before and after alkaline activation. The IR spectrum of BOF slag reveals the main absorption bands at 1433 and 875.2 cm^{-1}. The intense band at 875.2 cm^{-1} and 1433 cm^{-1} are associated with Si–O–Si and Si–O–Al asymmetric stretching vibration. These intense bands become sharper and move towards higher frequencies of 917.4 cm^{-1} and 1452.8 cm^{-1} upon alkaline activation (Abdullah et al 2012; Somna et al., 2011). This indicates the formation of a new product (the aluminosilicate gel phase) due to dissolution of BOF slag in the alkaline activator.

3.6. The effect of solid/ liquid on UCS

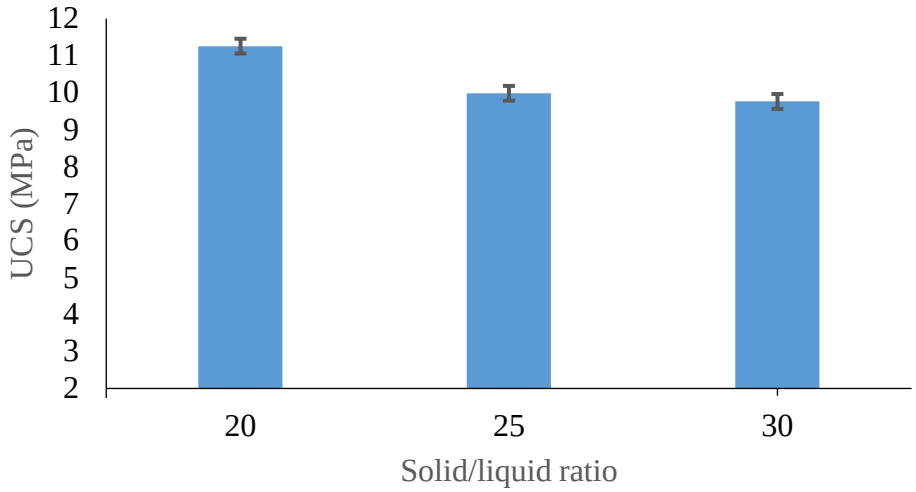

Fig. 6. The effect of solid to liquid ratio on UCS

Fig. 6 shows the UCS of the BOF slag based geopolymer at various S/L ratios with constant Na_2SiO_3/NaOH ratio at 2:1. The addition of sodium silicate was to enhance the process of geopolymerisation which resulted in 23.3% increase in UCS as compared to when NaOH alone was used as the activator (Morsy et al., 2014). There was an increase in UCS with a decrease in solid/liquid ratio. The results suggest that the workability of BOF slag based geopolymer decreased with increasing S/L ratio. Workability decreased as result of the highly viscous property of sodium silicate than the NaOH solution (Heah et al., 2012). By experimental observation at 30 % S/L ratio, the geopolymer paste was less workable and exhibits segregation (Suresh and Manojkumar 2013). In addition the dried paste appeared peeled off with cracks on one side (see Fig.4.4) which might be due to the high amount of activating solution which hinders the geopolymerisation process. However, at 25 % S/L ratio, the dried paste had less pronounced cracks as compared to 30 % S/L ratio and there was no peeling observed. At 20 % S/L ratio the paste was the most optimum in terms of workability and strength, and was thus used to investigate the other variables.

3.7. The effect of curing temperature

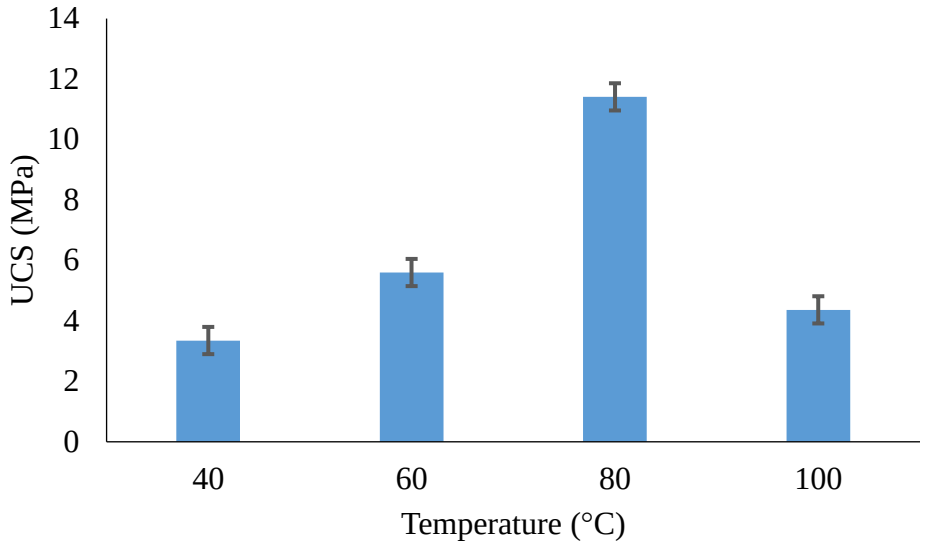

Fig 8. Effect of temperature on UCS

Fig. 8 shows the UCS of the BOF slag based geopolymer at different curing regimes. There was an increase in UCS with curing temperature increase from 40°C to 80°C. Basically, the initial curing at an elevated temperature (80°C) activates and favors the geopolymerisation process, hence there was 70% increase in UCS. An increase in temperature from 80°C to 100°C resulted in (56.7 %) substantial decrease in UCS. When curing at 100°C, the viscosity increases rapidly at the onset of poly-condensation. Therefore, the geopolymer paste hardened faster. This can also be attributed to rapid moisture loss at 100°C which results in the paste hardening faster and in turn shortening the geopolymerisation process. A similar trend has been reported by (Falayi et al., 2017)

3.8. The effect of Na_2SiO_3/NaOH ratio on UCS

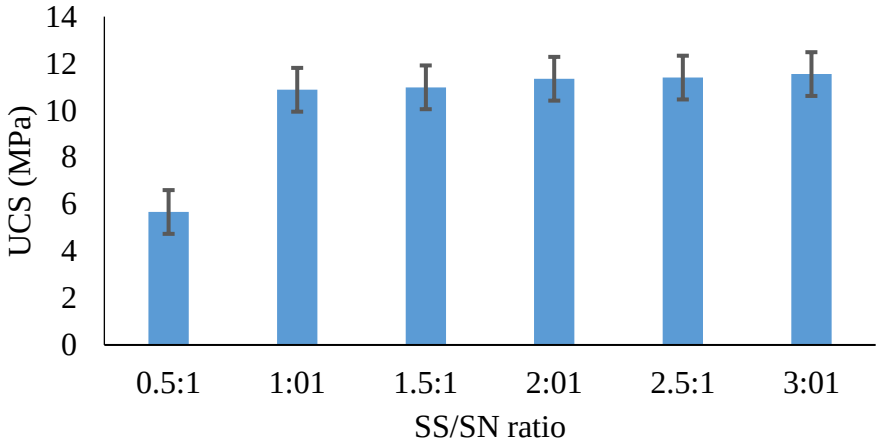

Fig 9. The effect of S/N ratio on UCS

Fig. 9 shows that there was an increase in UCS with increase in Na_2SiO_3/NaOH (S/N). However, the difference in UCS from 1:1 to 3:1 was statistically insignificant as $F < F_{crit}$ as shown in Table 3. The insignificant difference could have resulted from the fact that at 1:1 ratio the dissolution of silica and alumina was high leading to improvement in compressive strength of the BOF slag geopolymer (Morsy et al., 2014). This reveals that at 1:1 ratio most of the silica and alumina in BOF slag are dissolved on the first contact with alkaline activator, accelerating the geopolymerisation process, hence thereafter beyond S/N =1:1 the strength was insignificantly different. This phenomena consumes the silica and alumina in raw BOF slag material. Basically, the increase in the Na_2SiO_3/NaOH ratio resulted in the increase of sodium content in the mixture. Sodium is important for the formation of geopolymers as it acts as charge balancing ions. However, excess sodium/ sodium silicate might hinder water evaporation and structure formation; which in turn in this case resulted in insignificant difference in the UCS (Škvára et al., 2006). By experimental observation the increase in S/N= 1:1 ratio resulted in the geopolymer paste becoming very sticky as a result of the viscous nature of the water glass. This resulted in the geopolymer hardening faster, hence there was a very slight acceleration of geopolymerisation from 1:1 to 3:1 ratio which in turn resulted in insignificant change in UCS. The results obtained show that the addition of Na_2SiO_3 increased the strength of the geopolymer by 23% as compared to when only NaOH was used as the activator. This means Na_2SiO_3 can be added to the NaOH/BOF slag based paste to enhance the geopolymerisation process which; in turn improves the strength of the geopolymer.

Table 3 Statistical Anova test

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.04096	1	0.04096	0.34535	0.57297	5.31766
Within Groups	0.94884	8	0.11861			
Total	0.9898	9				

3.9. Wet compressive strength of the LWCB

Table 4 shows the 24 h soak properties of the LWCBs cured under different conditions.

Table 4: 24 h soak LWCB properties

BOFS Sample		
Mass of cured sample (kg)	1310	1330
Mass of cured sample after 24hr soak (kg)	1441	1489
UCS before soak (Mpa)	10.4	10.31
UCS after soak (Mpa)	9.496	8.954
% water absorption	10.01	11.95
% reduction in UCS	8.69	13.15
Open porosity	0.131	0.159
Mass after 5 h boil (kg)	1508	1531
Saturation coefficient	0.859	0.794

The synthesized LWCB cured satisfied the requirements of ASTM C34-13 with an average bulk density of 1320 kg/m^3.

4. Conclusions

South African BOF slag can be used as a low cost building material. The best geopolymerisation parameters were obtained at 5 M of NaOH, L/S ratio 20%, SS: SN ratio 1:1 and the curing temperature 80 ^{o}C. The particle size, concentration and quantity of NaOH and curing regime have a direct effect on the LWCB strength. BOF slag could be alkali activated using NaOH and SS to produce LWCB that met the minimum requirements for ASTM C34-13, C129-14a and South African standard (SANS227: 2007). The zeolite Hibschite was responsible for the strength development of the LWCB. The addition of SS increased the strength of the LWCB by 25% as compared to when only NaOH is used as the alkaline activator. The BOF slag characterization revealed that it's a sandy material with non-shrinking behavior and non-expansive as shown in the wet compressive strength results.

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