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INVESTIGATION INTO THE APPLICABILITY OF BREWERY SLUDGE RESIDUE-ASH AS A BASE MATERIAL FOR GEOPOLYMER CONCRETE

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Abstract: The environmental challenges such as high energy demand, large CO₂ emission, and exorbitant raw material consumption among others associated with ordinary Portland cement led researchers to the search for alternatives and thus the advent of geopolymer concrete. Fly ash, a waste product of the thermal generating power station, has been the base material commonly used in geopolymer production. However, Nigeria depends majorly on hydro-power and hence, fly ash is unavailable and this has contributed to the restricted application of geopolymer technology. Therefore, the suitability of brewery sludge residue (a waste byproduct generated in high quantity from brewing process) as an alternative base material in geopolymer concrete was investigated in this study. The physical and chemical properties of brewery sludge residue ash (BSA) were investigated to assess its suitability for use as a base material for geopolymer binder. Brewery sludge residue ash-based geopolymer concrete (BSAGC) specimens were produced by activating BSA with selected alkaline liquids (NaOH and Na₂SiO₂) used as activator. The BSAGC specimens were subjected to compressive strength to assess the strength development and consequently the effectiveness of the polymerization reaction that occurred. It was found that, amongst other factors, the BSA exhibits less satisfactory oxide characteristics at 425micron particle size utilised and consequently the compressive strength development was low at 28days curing duration at the 1:2:4 mix proportioning threshold adopted for the BSAGC mix. Given the marginal strength development of BSAGC, BSA could be reckon as having potentials for application as base material for geopolymer binder, however, more investigation is required to determine the optimum processing parameters for its usage as a base material for geopolymer binder and geopolymer concrete.

Keywords: Brewery sludge ash (BSA), Geopolymer concrete, Base material, Compressive strength, Oxide composition, Physical properties

1. INTRODUCTION

Besides economy, one of the major factors used in measuring the development of a nation is her infrastructural development. The construction industry is a major sector of concern when considering infrastructures. Concrete, which has for long been the most widely used construction material, is associated with Ordinary Portland Cement as a major raw component. The increasing standard of living and consequent growth of construction volume has made the demand for concrete to be increasing daily. Metha and Menterio [1] reported the average consumption of concrete in the world to be of the order of 11 billion metric tons per year.

However, there are some environmental challenges attached to both production and use of ordinary Portland cement as binder in concrete. These include the high CO₂ released during production, high raw material consumption and the high energy requirement for production. Global warming and climate changes have been a major problem to most developed and developing nations due to emission of greenhouse gases, which majorly is CO₂. Recent report from intergovernmental Panel Climate Change (IPCC) had warned that the world is headed for a 3°C temperature increase by the year 2100 if the existing carbon footprint is not substantially cut down. McCaffrey [2] reported that 65% of the global warming in the world is caused by CO₂. Therefore, researchers have been on the lookout for alternatives to Portland cement and this has led to the discovery of geopolymer binder as in

geopolymer concrete. Geopolymers are non-hydraulic binders developed as a sustainable alternative to Portland cement. Geopolymer binder consist of two main components, namely, the source materials (also known as base material) and the alkaline liquids (also known as activators). Geopolymer concrete is a type of concrete that is made using the concept of geopolymer binder technology. It involves reacting aluminate and silicate bearing materials (i.e. the base material) with a caustic activator (i.e. the alkaline liquid) to produce the geopolymer binder paste [3]. The geopolymer binder paste binds the aggregate components and other constituent of concrete mix together to create a hardened concrete known as geopolymer concrete [4]. It is an innovative and eco-friendly construction material and an alternative to Portland cement concrete. The most commonly used base materials for geopolymer binder are by-product materials such as fly ash, silica fume, slag, metakaoline, etc [5];[6];[7].

However, the unavailability of fly ash and other applicable byproducts has restricted the application and implementation of geopolymer concrete technology in Nigeria and neighboring developing countries. Brewery sludge residue is a waste generated by brewery industry in large quantities, typically; 14-20kg from every 1 hectolitre of beer produced [8]. Brewery sludge residue is a byproduct from brewing process of beer and it is being generated in large quantities by brewery industries in Nigeria owing to the country's annual beer production size estimated at 15million hectoliter [9]. Recent research by Fariás *et al.* [10]

had shown that the ashes of brewery sludge residue is rich in silicon oxide and possess moderate concentration of other oxides (including; Aluminium oxides and Iron oxides) which are indicators of aluminosilicate characteristic of a raw material. Hence, this study investigates the applicability of the ashes of brewery sludge residue as a base material for geopolymer concrete. The prospect of utilizing brewery sludge residue ashes as aluminosilicate base component of geopolymer binder may open-up holistic opportunities for production and utilisation of ecofriendly concrete in the creation of built environment in developing countries.

2. Experimental Details

The materials used for the production of the brewery sludge ash based geopolymer concrete (BSAGC) investigated in this study includes; brewery sludge residue ash (BSA), sodium hydroxide (NaOH), sodium silicate (Na_2SiO_2), sand (as fine aggregate), granite (as coarse aggregate) and water. The physical and chemical properties of BSA were investigated to assess its suitability for use as a base material for geopolymer binder. The BSAGC specimens were produced by activating BSA with the selected alkaline liquids (NaOH and Na_2SiO_2) used as activator. The BSAGC specimens produced were subjected to compressive strength to assess the strength development and consequently the effectiveness of the polymerization reaction that occurred.

2.1 Materials

2.1.1 Brewery Sludge Residue and Preparation of Brewery Sludge Residue Ash (BSA)

Just like any other waste, brewery sludge residue (Fig 1) management is an issue of concern for most breweries in Nigeria as large quantity is being generated continuously. It has an offensive smell and hence normally carted away to dump sites far away from industrial and residential settings. The brewery sludge residue used for this work was obtained from Lapite dump site, Akinyele Local Government, Ibadan, Oyo State Nigeria.



Fig 1: Brewery Sludge Residue

Samples of the brewery sludge residue obtained were sun dried for about 24 hours, sieved and calcined using a gas-powered furnace at the Foundry Workshop, Department of Mechanical Engineering, University of Ibadan at a constant rate up to a maximum temperature of 800°C. This temperature was maintained for another two hours while the combustion continued to form the brewery sludge residue ash (BSA) (Fig 2). The ash obtained was brought out of the system and allowed to cool at room temperature.



Fig 2: Brewery Sludge Residue Ash

2.1.2 Alkaline Solution

The alkaline solution was made from a combination of sodium hydroxide (NaOH) pellets and sodium silicate (Na_2SiO_2) solutions. The sodium silicate and the sodium hydroxide were both purchased from Elis Oye Ventures, a commercial store at Oke Ado, Ibadan. The NaOH pellet sample exhibited 5mm average particle size, specific gravity of 2.13 and 97% purity. The Na_2SiO_2 solution exhibited average density of 1.5gm/mL at room temperature, specific gravity of 1.5 and $\text{SiO}_2:\text{Na}_2\text{O}$ weight ratio of 2.5 (i.e. $\text{SiO}_2 = 30\%$, $\text{Na}_2\text{O} = 12\%$, water = 57%). The NaOH pellets were dissolved in distilled water to make 8 molar concentration solution. 320g of NaOH pellets were weighed and dissolved in one litre of distilled water to prepare the NaOH alkaline solution of 8 molar concentration.

2.1.3 Aggregates

The aggregate used is a combination of sand as fine aggregate and granite as coarse aggregates. The granite used as coarse aggregate was obtained from a construction site within the campus of University of Ibadan, and the particle size is of the range 12.5mm to 20mm as recommended by BS 882: 1992 [11] to enhance the strength of the produced concrete. The sand used as fine aggregate also passes through sieve size 4.75mm.

2.2 Chemical and Physical Characterisation of Brewery Sludge Residue Ash

The chemical composition of the sludge ash was determined by X-ray fluorescence (XRF) using dispersive energy spectrophotometer. The silica and alumina present in the ash were principal criterion for a good geopolymer base material. Loss on ignition (LOI), which is a measure of percentage of organic content (unburnt carbon) in the material was also determined in line with ASTM C204. The physical properties of the brewery sludge residue ash obtained are; particle size, bulk density and specific gravity. The bulk density and specific gravity were carried out in accordance with BS EN. 1097-8:2009. The particle size was determined in line with BS EN. 933-1:1997 recommended procedures.

2.3 Production and Testing of Brewery Sludge Ash Based Geopolymer Concrete (BSAGC) Specimens

The mix proportioning thresholds of concrete grade with mix composition 1:2:4 proportions of binder, fine aggregate and coarse aggregate were adopted for the mix proportioning of materials for the BSAGC. Unlike cement used as binder in conventional concrete mixes, the binder in the BSAGC consist of the mixture of NaOH and Na₂SiO₂ (i.e. the alkaline activator) and BSA (i.e. the aluminosilicate base material). For the alkaline activator, the ratio of Na₂SiO₂ solution to NaOH solution was fixed at a 1.5 ratio, by mass. To make the geopolymer binder, the ratio of the alkaline activator to BSA was fixed at a 0.5 ratio, by mass. The sand sample used as fine aggregate and the granite sample used as coarse aggregate were added in the proportion appropriate for the adopted mix composition. BSAGC cube specimen of sizes 100mm by 100mm by 100mm were produced in accordance with procedures recommended by BS EN 12390-2:2009 [12]. The specimens were oven cured for 24 hours at 90°C temperature and subjected to ambient curing condition for 7 days and 28 days duration. A total of eight (8) specimens were produced with four (4) specimens allocated for testing each of the 7 days and 28 days curing ages. The 7 days and 28 days cured BSAGC specimens were subjected to compressive strength test in line with BS EN 12390-3:2009 [13].

3. RESULTS AND DISCUSSION

3.1 Chemical and Physical properties of BSA

The chemical oxide composition and physical properties of the brewery sludge residue ash are presented in Table 1.

Table 1: Chemical and physical Properties of Brewery Sludge Ash (BSA)

Chemical properties of BSA	
Oxides	% composition
SiO ₂	61.04
Al ₂ O ₃	8.17
Fe ₂ O ₃	6.04
CaO	0.28
Na ₂ O	2.56
K ₂ O	6.02
MgO	0.99
CuO	9.50
Fe	5.40
Loss on Ignition (LOI)	11.20
Physical Properties	
Specific gravity	1.59
Bulk Density	295.4 kg/m ³
Particle size	Passing 425 micron

3.1.1 Chemical Composition of BSA

The oxide composition of the BSAGC shows that it is rich in silicon oxide. Silica is the most important oxide that is expected to be present in a base material of geopolymer as it plays a major role in the alkaline activation of ash [14]. The main reaction product of the geopolymeric synthesis is an aluminosilicate gel. Thus, the high SiO₂ present in the BSAGC composition implies that the high silica content may be involved in the formation of alkaline aluminosilicate gel. However, the alumina content in the composition of BSA appears to be very low compared to the recommended percentage. As such the silica to aluminium (Si/Al) ratio for BSA is 7.47 (i.e. 61.04/8.17 = 7.47 see Table 1) and it is comparatively less satisfactory with regards to the recommended categories of Si/Al ratios (which includes; poly(sialate) for Si/Al=1, poly(sialate-siloxo) for Si/Al=2, and poly(sialate-disiloxo) for Si/Al=3) as reported by Wattimena *et al.* [15]. Calcium oxide (CaO) is usually required in low amount in the composition of geopolymer base material because its presence in high amount often interferes with the geopolymerisation reaction [16]. Thus, the very low amount of CaO (typically 0.28%) in the oxide composition of BSA could be an added advantage for its effectiveness in the polymerization reaction with the alkaline activator. The fineness of the particles of a base material of geopolymer contribute to high reactivity during the activation process and indirectly produces less carbon content [17], thus, the carbon content as indicated by the 11.02% LOI recorded for BSA may be regarded as

an indication of its coarse particle which is coarser than the recommended 45micron particle size.

3.1.2 Physical Properties of BSA

As shown in Table 1, at particle size passing 425micron, BSA exhibits an average specific gravity of 1.59, average bulk density of 294.5kg/m³. In contrast to fly ash which is a popularly used geopolymer base material, the 1.59 average specific gravity of BSA is less than the 2.0 to 3.0 reported in the literature for fly ash. Similarly, the 294.5 kg/m³ average bulk density reported for BSA is lower compared to 540 kg/m³ to 840 kg/m³ reported for fly ash in the literature. This indicates that the particles of BSA are coarse and are less closely packed together compared to those of fly ash. This may affect the ability of the BSA to dissolve in the alkaline liquid during the geopolymer activation process.

3.2 Compressive Strength of BSAGC

As presented in Table 2, the brewery sludge ash based geopolymer concrete (BSAGC) developed a low average compressive strength at 7 and 28 days curing durations. However, the initial strength development appeared to be rapid being over 100% higher than those recorded at 28 days curing age. Comparatively, the 0.53N/mm² average compressive strength developed by the BSAGC is low with reference to the 17.3N/mm² average compressive strength reported by Hardjito *et al.* [3] for conventional fly ash based geopolymer concrete cylinder specimen at 7 days curing age (though using different processing parameters).

Adequate strength development in geopolymer binder and geopolymer concrete depends majorly on the efficiency of the polymerization reaction that occur between the source material and the alkaline liquid. However, polymerisation may be altered or interfered with when either the base material or the alkaline liquid does not meet certain requirements.

Table 2: Compressive Strength of Brewery Sludge Ash Based Geopolymer Concrete Specimen

Curing duration (days)	BSAGC Samples	Density (kg/m ³)	Compressive (N/mm ²)
7	1	1754.50	1.09
	2	1670.70	1.10
	3	1759.34	1.06
	4	1853.70	1.05

	Average	1759.56	1.08
28	1	1585.90	0.50
	2	1680.98	0.70
	3	1561.24	0.40
	4	1584.97	0.50
	Average	1603.27	0.53

Evidence from previous research had shown that physical and chemical characteristics including; particle size, oxide composition, and loss on ignition (LOI) properties play a major role in the efficiency of a geopolymer base material. In contrast to the recommended particle size (less than 45micron [14], Si/Al ratio (ranging from 1 to 3 [15] and LOI (less than 5%), the BSA used as source material in this study exhibited coarser particle size of 425micron, less satisfactory Si/Al ratio of 7.47 and higher LOI of 11.20%. Thus, amongst other factors (including low molarity of activator, mix proportion in terms of; ratio of activator to BSA, ratio of geopolymer binder to aggregates, etc), the low strength development of BSAGC may be attributed to the less satisfactory; particle size, Si/Al ratio and LOI of BSA as a geopolymer base material. This low average compressive strength obtained for BSAGC at 28 days curing age is an indication that BSA exhibits weak polymerization reactivity under the processing parameters employed in this study. The weak polymerisation reaction implies two possible phenomena; either the inefficiency of the activator to fully dissolve the Silicon and Aluminum phases present in the BSA at the 8 molar concentration utilised or the partial dissolution of the BSA due to the 425micron particle size utilised. Either of these phenomenon might have prevented the transport of the ions into monomers and consequently alter polymer formation. Unlike conventional concrete, the compressive strength of geopolymer normally does not grow with the age of the concrete because the mechanism of reaction of geo-polymerization is fast [3]. Thus, the higher strength development recorded at 7 days compared to that of 28 days may be attributed to the fast geo-polymerisation reaction mechanism that might have taken place.

However, the 0.53 N/mm² average compressive strength obtained for the BSAGC specimen tested at 28 days indicated an unusual drop in the compressive strength compared to the 1.08N/mm² average compressive strength recorded at 7 days curing age. Amongst other possible factors, the drop in compressive strength of BSAGC specimen at 28 days may be attributed to the unusual physical deterioration in the form of surface scaling/disintegration (Fig 3) that were noticed on the surface of the specimens at 28 days curing age.



Fig 3: Surface scaling/disintegration of BSAGC Specimen at 28 days curing age

Surface scaling defect occurs in concrete due to freeze-thaw phenomenon. In cold environment, concrete experiences freeze-thaw when the hydraulic pressure from water freezing within the concrete microstructure exceeds the tensile strength of the concrete [18];[19]. Similarly, disintegration defect is a form of surface scaling that could be caused by de-icing chemicals, sulphates, chlorides or by frost action[18];[19]. Considering the hot humid environment (27°C average temperature and 85% relative humidity) in which the BSAGC specimen studied was produced and cured, the surface scaling/disintegration defect observed on the BSAGC specimen at 28 days curing age may not be attributed to either freeze-thaw or frost action. Rather, amongst other possible causes, the high carbon content of the BSA (recorded at 11.20% instead of the recommended maximum of 5%) may have contributed to the defect. Carbon particles may trap excess water in the microstructure of concrete specimen (which in the case of geopolymer should be expelled and vaporise during curing) thereby weakening the bond in the concrete matrix. Also, alkali oxides such as Na_2O and K_2O are known for their potential formation of alkali silica gel upon reaction with silica aggregates of concrete and thereby result in expansion and disintegration of concrete. Consequently, the recommended maximum alkali oxides in an aluminosilicate raw material is 0.3 wt % to 1.2 wt % [20]. The studied BSA contains 8.58 wt % of alkalis (i.e. Na_2O =2.56 wt% and K_2O = 6.02 wt%) and hence the resultant geopolymeric binder and geopolymer concrete may be susceptible to alkali related disintegration. Above all, more investigation is needed to determine the main cause(s) of the surface scaling /disintegration defects that were observed on the surface of BSAGC specimens at 28days curing age.

3.3 Future Research Needs

The findings from this investigation show that BSA exhibits weak polymerization reaction under the processing parameters utilised. Future research may investigate suitability of BSA for this purpose when applied at finer particle size granulation exploring varying mix proportioning threshold to determine the optimum processing parameters. BSA may also be combined with other known materials of geological origin (such as Fly ash, metakaoline etc). Possibility of combining BSA with other mineral materials may provide opportunities for low cost application of geopolymer binder in developing countries. Additionally, research investigation should also be conducted to identify the main cause(s) of the observed surface scaling /disintegration defects in BSAGC at 28days curing age.

4. CONCLUSION

The most commonly used aluminosilicate base materials for geopolymer binder includes fly ash, metakaolin, GGBFS etc., nevertheless other locally available byproducts rich in alumina and silica can serve the same purpose. Brewery sludge residue is a major waste from the brewing process which has not been explored for engineering applications. It has though found uses in agriculture as a compost material, plant nutrients and in neutralizing soil acidity. This study assessed the applicability of brewery sludge residue ash (BSA) as a base material for geopolymer concrete production. Based on the results obtained, the following conclusions were made.

- At particle size passing 425micron, brewery sludge residue ash (BSA) contains appreciable amount of silica oxide, moderate quantities of alumina and iron oxide, and very low amount of calcium oxide.
- Brewery sludge-ash based geopolymer concrete (BSAGC) displayed rapid early strength at 7 days curing age, typically over 100% higher than the strength developed at 28 days curing age.
- BSA undergo weak polymerization reaction when activated with 8 molar concentration of NaOH and Na_2SiO_2 due to its less satisfactory characteristics in terms particle size of 425micron, Si/AL ratio of 7.47 and LOI of 11.20%.
- At 1:2:4 mix proportioning of BSA geopolymer binder, sand and aggregate; BSAGC developed low compressive strength at both 7 and 28 days curing age.
- At 28days curing duration BSAGC displayed physical deterioration in the form of surface scaling/disintegration and consequent drop in compressive strength.

- BSA exhibit potential for application as a base material of geopolymer binder if more satisfactory and optimum processing parameters are investigated and employed.

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