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Production of Alumino-Silicate Clay-Bonded Bagasse Ash Composite Crucible by Slip Casting

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Abstract

This research work developed a new material for the production of crucible using alumino-silicate clay (Kankara) and bagasse ash. The bagasse ash serves as a source of graphite to the crucible, the clay was used to bind the materials together and to make the slip more plastic and the grog was used to strengthen the crucible. The physical properties such as the bulk density, porosity, crushing strength, thermal shock resistance and actual melting of aluminium with the crucible were determined. The results obtained showed that density, porosity, crushing strength and thermal shock resistance are within the recommendation standard. The developed crucible has a high stability to the contamination of crucible materials (Al_2O_3 and SiO_2) on the cast aluminium. Hence the developed crucible can be use in the melting of non-ferrous alloy of good microstructures.

Keywords: Bagasse ash, Kankara clay, Crucible and Aluminium alloy.

1. Introduction

A crucible is a high refractory open mouth vessel that can withstand very high temperature, they are used for melting iron and steel, glasses, various metals and alloys [1], while crucible historically were usually made from clay, they can be made from any material that withstands temperature high enough to melt or otherwise alter its contents [2]. There are various types of crucibles depending on the constituent materials, these include graphite crucibles, silicon carbide crucible, clay crucible [3]. High temperature operations are involved in almost all the industries dealing with the treatment of ores and other materials for the manufacture of metallurgical, chemical and ceramic product such equipment used for treatment of these materials must sustain the operating temperature and other working conditions such as corrosive, erosive and load conditions [4-5]. In the production of metallurgical crucibles, it is important for one to know their component raw materials, their important properties and correlation between these properties and the actual use of the crucible [6].The important raw materials required for the manufacture of crucibles are: binders, clay which are available in large quantities. There is good investment opportunity for the production of crucible locally. Sugar cane waste (bagasse) is a plentiful lignocellulosis waste typically found in topical countries that process sugar cane, such as Brazil, Indian, Cuba, China and Nigeria [7]. The bagasse fiber (BF) Bundles are usually coarse and stiff. It is used either as a fuel for

boilers by the sugar factory or as a raw material for the manufacture of pulp and paper products, various type of building boards and certain chemicals[8]. Recently Aigbodion et al [7] found out that bagasse can be used in the production of low cost metal matrix composites. Clays are natural argillaceous matters formed by decomposition of felspathic rock as a result of weathering action of wind and rain, and are of general chemical formula mAl_2O_3 $nSiO_2.pH_2O$. Clays belong to the Alumino-silicate group of refractory materials and are the most readily available sources of fireclay refractory bricks used in furnaces [9]. Grog is a term used for fired clay particles. It is added mainly as an anti-shrinkage element in the form of angular particles of various sizes to achieve better interlocking of grains [10]. As per the literature above, no investigation has been recorded on the production of graphite-clay crucibles using bagasse ash as the source of graphite. Hence, an attempt has been made in this paper to develop a bagasse ash-clay crucible.

2. Experimental

2.1 Materials

The raw materials used in this research work includes: bagasse, Kankara clay, grog, quartz, feldspar and silicon carbide, Silicon and aluminium.

2.2 Equipment

The equipment used in the course of this research includes the following: muffle electric Furnace, Wire mesh sieve (420 µm), Plaster mould, Electric Oven (model: OV – 420), Ball milling machine (push and crank BS 2406), Weighing balance, X-ray spectrometer (XRF), X-ray diffractometry (XRD) machine and optical microscope.

2.3 Method

The bagasse (sugar cane waste) was packed in the graphite crucible air tight, and placed inside muffle electric control furnace and burned at a temperature of 2200^{o}C for 5 hours to obtain a black color ash [7]. The raw Kankara clay(alumino-silicate) was soaked in water for three days and dried in open air for a week, this treatment was necessary to remove dead organic matters. The dried clay was then crushed and ground into powder using jaw crushers and pulverized. The ground clay was sieved to pass through sieve 200µm aperture and the bagasse ash was sieve to 100µm. The X-ray diffractograms of the Kankara clay and the bagasse ash were taken using Cu Kα radiation at scan speed of 3^o/ min [7]. The sample was rotated at precisely one-half of the angular speed of the receiving slit, so that a constant angle between the incident and reflected beams is maintained. The receiving slit is mounted in front of the counter on the counter tube arm, and behind it is usually fixed a scatter slit to ensure that the counter receives radiation only from the portion of the specimen illuminated by the primary beam. The intensity diffracted at the various angles was recorded automatically on a chart and the appropriate (Θ) and (d) values were then obtained.

A chemical analysis of the Kankara clay was determined in the Scientific section of the National Metallurgical Development Center, Jos, Nigeria by

using Mini Pal compact energy dispersive X-ray spectrometer (XRF) [8]. The grog preparation started by beneficiating the Kankara clay in which the lumps were first grinded and slacked by soaking in water for two days. The resultant slurry was screened through the Tyler sieve of 200 mesh (75µm) size [9-10]. The sieved clay suspension was de-watered in biscuit fired clay (decanting) pots and left for two days, while the coarse material (impurities) was discarded. The clay slurry-pulp was worked into a plastic body of a stiff consistency. The clay was packed in a rectangular wooden mould. The mould was properly packed and left to dry for three days. After drying in the oven (110°C) the bricks (bricks or solid particles) formed were fired in the furnace at 1500°C. The produced grog was crushed, ground and sieved into 75µm[12]. Before the

production of the crucibles a preliminary test was conducted to determine the combination of bagasse ash, grog and Kankara clay. The test confirmed that the combination of 20wt% bagasse ash, 33.3wt%Kankara clay and 47.7wt%grog give the best properties. Slip casting was used in the production of the crucible. Slip is formed by suspension of clay, grog and bagasse ash in water. The suspension was poured into a porous mold (made of plaster of paris), water from the slip is absorbed into the mold, leaving behind a solid layer on the mold wall, the thickness of which depend on the time. This process continued until the entire mold cavity becomes solid (solid casting), by inverting the mold and pouring out the excess slip; this is term drain casting. As the cast piece dries and shrinks, it was pull away (or release) from the mould walls; at this time the mold was dissembled and the cast piece removed(see Figure 1). 200 and 500grams crucibles were produced with this method. The crucibles were dried in open air for three days, followed by drying in oven for 12 hours at 110^{O}C to expel any moisture left in the samples and to avoid crack during firing. Firing was done in muffle electric heating furnace at heating rate of 7^{O}C/minute[13-14]. The firing procedure used involved heating and soaking the samples at various temperatures: 250^{O}C for 6 hours, 650 ^{O}C hours for 4 hours, 950 ^{O}C for 3 hours, 1100 ^{O}C hours for 8 hours and 2000 ^{O}C hours for 8hours. After firing the samples were allowed to cool in the furnace at a cooling rate of 1 ^{O}C /minute [13-16].

Figure1: the Crucible when been casted in a mould using slip casting method

Glazing of the crucibles was done using the weight percentage of materials shown in Table 1. From the literature [10-11], glaze for application on porous or green wares should possess a high fluidity and a specific gravity of 1.40 which corresponds to about 50% water and 50% of the mass [10, 14]. The procedures that were followed for preparation of the glaze slip are; 0.2% by weight (0.24g) of sodium carbonate was added as a deflocculant, 50% of the water required for the glazing slip was also added [6].

Table 1: Raw material composition in glaze

Raw Material	Glaze wt%
Kaolin	40
Feldspar	30
Quart	20
Silicon carbide	10

The mixture was ball milled for 18 hours bearing in mind that homogenous mixture is required that will remain in suspension for a reasonable length of time and a glaze slip that also has a high fluidity. After ball milling, the resultant slip was poured out and mixed with the remaining water to give the required specific gravity of about 1.47. Immersion method was employed to glaze the body of the crucible. The glazed crucible was place in the kiln and heated in a reduced atmosphere for 2 hours to a temperature of 1260°C and allowed to cool in the kiln for 24 hours before the crucible was removed [10]. The porosity and bulk density of the glazed crucibles was determined by standard method [4-6] by keeping the crucible in the oven at 110^{O}C for 3 hours to obtained constant weight D the crucibles was then suspend in distilled water and boiled on a hot plate for 30 minutes, after boiling, while still in hot water, the water was now displaced with cold water and the weight W was measured on a spring balance hinged on the a tripod stand. The test sample were removed from the water and extra water wiped off from the surface by lightly blotting the sample with wet towel and the weight S in air was measured, the apparent porosity (P_a) of the sample was determined from the relationship [4].

$$P_a = \frac{W-D}{W-S} x100(\%)$$

Pa=apparent porosity

The Bulk density (B_d) was also calculated from the relationship as [8]

$$Bd = \frac{D}{W-S} \ (g\backslash cm^3)$$

Bd=bulk density, D = Dried weight, W = Soaked weight, S = Suspended weight

The sample was tested for crushing strength using hydraulic strength testing machine. The crushing strength was then calculated using the relationship [2]:

$$\text{Cold Crushing strength} = \frac{load(KN)}{Area(m^2)}$$

A thermal shock resistance test sample was put in the furnace that was maintained at a temperature of 1600^{O}C and soaked at this temperature for 30mintues after which the sample was brought out to cool for 10 minutes. The sample was then tested for failure using a standard rig, if failure did not occur the sample was then put back inside the furnace and heated for a period of ten minutes, this cycle of heating, cooling and testing was repeated until failure occurred. The number of complete cycles to produce failure in each sample was noted [4].

Experimental evaluation of the crucible was conducted by melting process; firstly a 50-gram button of commercial purity aluminium ingot was placed in the crucible. After charging the Crucible in the furnace, argon was injected to maintain a control atmosphere. The charge was heated at approximately 10^{o}C/per minute to 700 $\pm 50^{o}$C and held for 5 minutes at that temperature. During the melting of the aluminium, sample of aluminium was taken out at temperatures of 550, 600, 650, 700 and 750^{o}C for chemical analysis for the contamination of Al_2O_3 and SiO_2. Secondly, the crucible was used to pour actual castings of Al-12%Si alloy. The crucible was then charged with 200 grams of commercial Al-12%Si alloy and placed in a melting furnace. The charged crucible was then heated at approximately 10^{o}C per minute and argon was injected through the system. The sample was subjected to metallographic examination [7].

3. Results and Discussion

The X-ray diffraction patterns of the clay, scanned from 0-90°(2θ) showed several peaks due to the different minerals (Figure 2). The major diffraction peaks were 10.29, 29.03, 31.08, 32.08 and 49.76° and their inter-planar distance are: 9.98, 3.57, 3.34, 3.23 and 2.12Å, and their relative intensity of X-ray scattering were 1.43, 9.31, 100.00, 0.65 and 4.19, phases at these peaks were: Magnesium Aluminum Silicate(Mg O Al_2 O_3 Si O_2), Sillimanite(Al_2 (Si O_4) O), Quartz low(Si O_2), Mayenite, syn((Ca O)$_{12}$ (Al_2 O_3)$_7$), Silicon Oxide(Si O_2), while each of these phases has a score of 30, 20, 86, 24 and 25 respectively(see Figure 2).

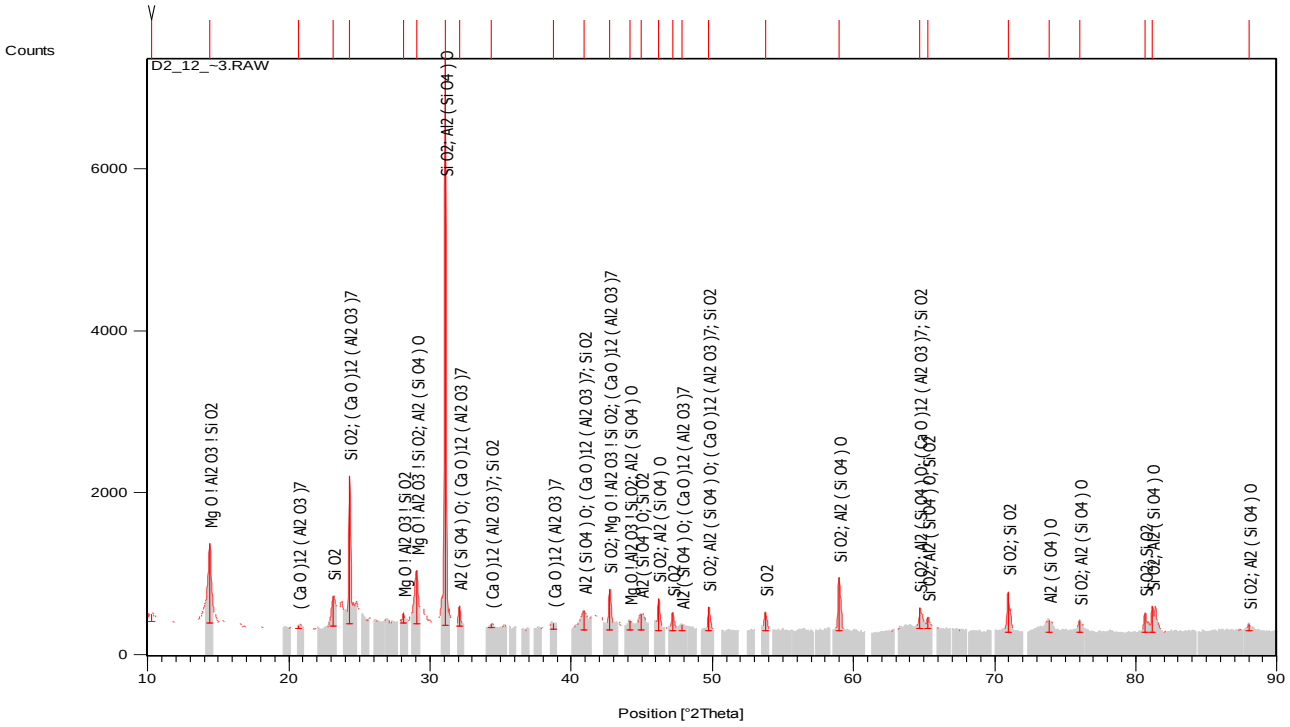

Figure 2: XRD pattern of Kankara Clay

The XRF chemical composition of the clay is presented in Table 2. The XRF analysis confirmed that SiO_2 and Al_2O_3 were found to be the major constituents of the clay. Silicon dioxide and alumina were known to be among the hardest substances. Some other oxides viz. Fe_2O_3 MgO, K_2O, Na_2O was also found to be present in traces. The presence of hard elements like SiO_2, Al_2O_3 suggested that, the Kankara clay belong to Alumino-Silicate clay [4]. This result of XRF is in agreement with the resulted of XRD analysis.

Table 2: Chemical Analysis of the Kankara Clay

Compound	Al_2O_3	SiO_2	Fe_2O_3	CaO	MgO	Ka_2O	I.O.I
% composition	36.40	46.48	1.09	0.73	0.87	0.10	14.31

The XRD pattern (see Figure 3) of the bagasse ash revealed that, the major diffraction peaks are 47.1, 22.1 and 24.78° and their inter-planar distance are: 1.92, 4.01 and 3.50Å, and their relative intensity of X-ray

scattering are 21.08, 100.00 and 23.45 and phases at these peaks are: Carbon(graphite), Silica and Carbon(graphite) while each of these phases have a score of 31, 28 and 27 respectively. From this analysis it was confirmed that the bagasse ash has carbon in graphite form necessary for the production of graphite crucible [10, 15].

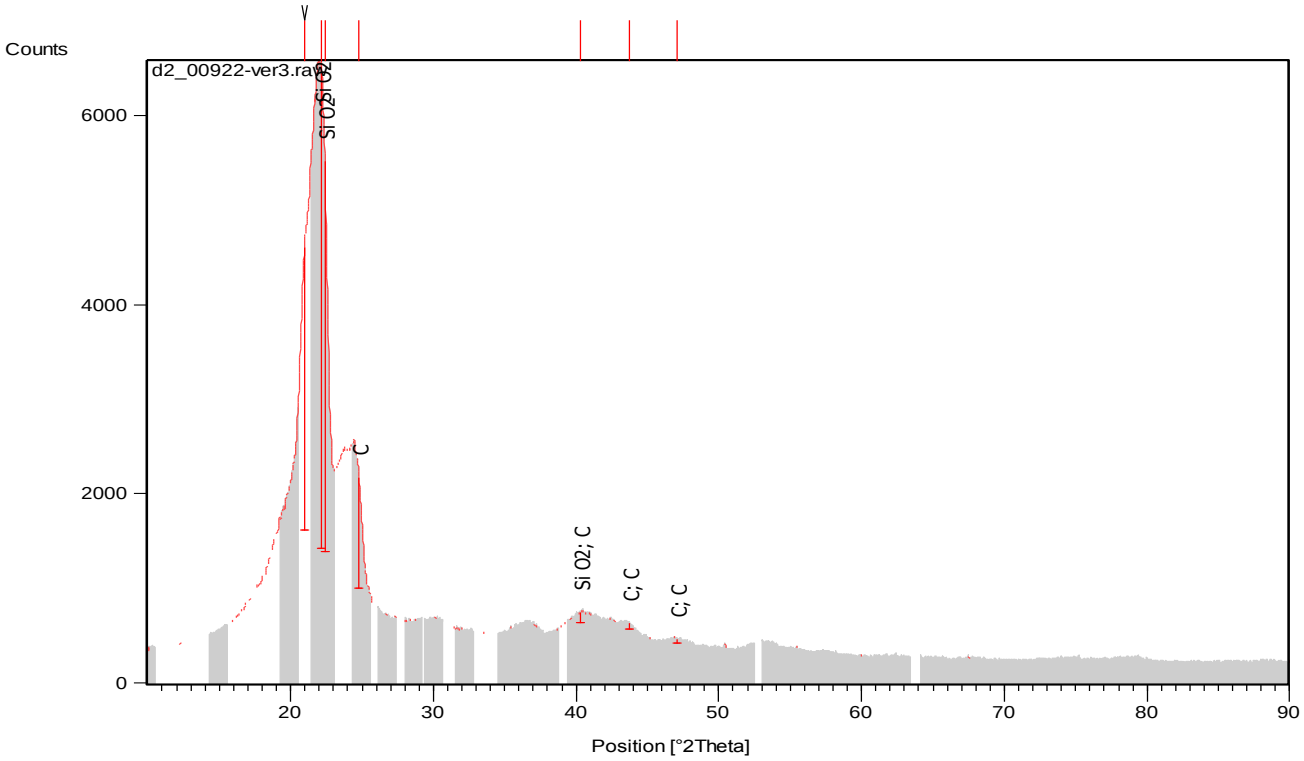

Figure 3: XRD pattern of Bagasse ash

After casting the crucibles they were removed from the mould accordingly following the standard procedure [11] (see Figure 4). The green products were black in colour and this was evidence of the colour of bagasse ash. The macroscopic observation of the crucible after the casting;, it was observed that well forming occurred during the casting process; this confirmed to proper bonding of the clay-bagasse ash particles. After preheating using oven, the water was absorbed and the colours of the crucibles were ash-black in colour. After firing of the glazed crucible at temperature of 1260°C, the crucible was found to be under-glazed which shows that the temperature of firing was not enough to glazed the crucible and later the crucible was glazed at 1650°C to obtained proper bonding(see Figure 5) [14].

The apparent porosity of the crucible was 11.05%. The value was within the recommended standard of 11-14% for clay-graphite bonded crucibles. This means that with this value of porosity the crucible will have good thermal conductivity to transfer heat to the charge materials [10-11]. Bulk density of the crucible was 1.90g/cm^3 the values was within the standard of 1.89 to 1.96 g/cm^3 [1-2] The cold crushing strength obtained was 265kg/cm^3 the value is within the ranges of 250-300 kg/cm^2 recommended. This is attributed to good bonding and vitrification during firing [1, 4].

Figure 4: Crucible after been removed from the mould

Figure 5: Glazed Crucible after been fired in the furnace

The thermal shock resistance of crucible is +30cycles. The thermal shock resistance was very good. The thermal shock resistance falls within the accepted range of 15+ cycles [4-6]. This means that the produced crucible can withstand sudden change in temperature. The amount of incorporated crucible material into the prepared aluminium was determined by chemical analysis using XRF (for SiO_2 and Al_2O_3). The result of the contamination analysis made by XRF on the prepared aluminum samples can be summarized in Figure 6. From the result it may be observed that aluminium melted in developed bagasse ash- clay crucible had a higher stability against contamination. However, this stability decreased when the amount of alumina and silica incorporated in the aluminium increased with melting temperature. As Menezes et al [3] noted that alumina addition in aluminium increases the stability against devitrification up to a certain percentage, allowing the preparation of big pieces.

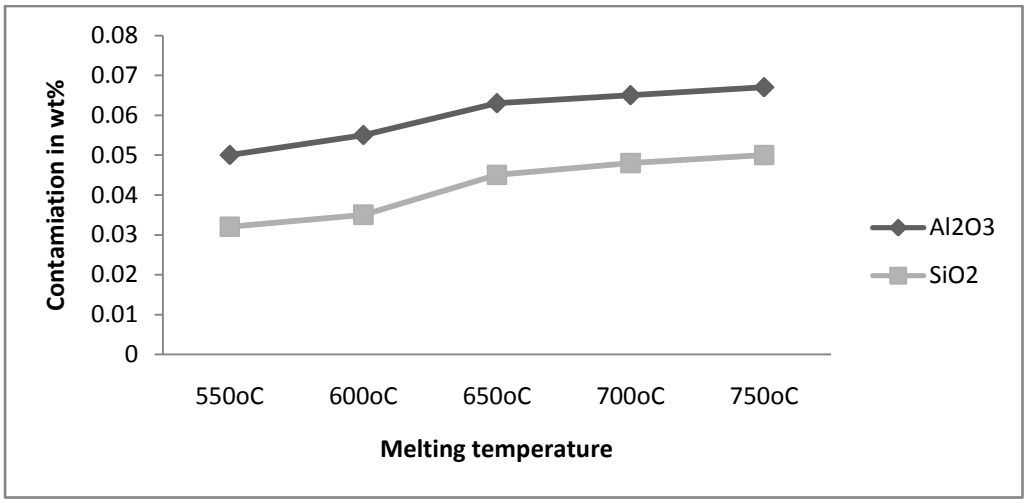

Figure 6: Variation of Contamination with melting temperatures

The optical microstructure of the cast Al-12wt%Si alloy is showed in Figure 7.

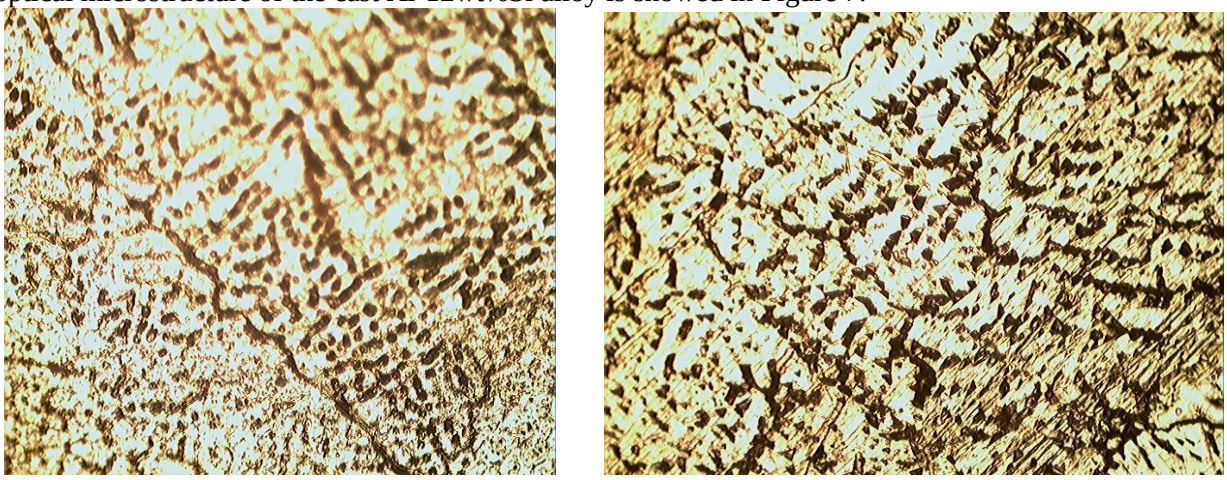

Figure 7: Optical microstructure of the casted aluminium alloy.

The microstructure of the aluminum alloy was analyzed using optical microscopy (see Figure 7). The structure reveals the eutectic phase containing Al_3Si, in α-aluminum matrix. These structures are in agreement with phase reported by other researchers [7]. In the examined microstructure, no effects of unfavorable phenomena were observed, which are frequently formed in the structures of cast aluminium alloy, such as sedimentation, as well as the formation of particle agglomerates or gas blisters. This shows that the developed crucible can be used in the casting of non-ferrous alloy with good structures.

Conclusions

1. From the results obtained from this investigation it can be concluded that suitable size of bagasse ash crucible can be produced with 20% bagasse, 47.7% grog and 33.3% of clay, using the slip casting method.
2. The crucibles produced using plastic clay as binder give very less open pores and hence low permeability, high compacted and hence good heat conductivity, good resistance to molten metal and slag, and high strength.

3. Based on the results obtained after glazing, locally available raw materials which includes; feldspar, quartz, clay and silicon carbide are suitable for making the glaze for bagasse ash crucible.
4. The results of the density, porosity, crushing strength and thermal shock resistance obtained are within the recommended standard.
5. The crucible has a high stability to the contamination of cast aluminium
6. The crucible can be employed in the casting of non-ferrous alloy of good microstructures

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