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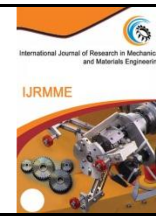

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GROUNDNUT SHELL ASH AS ALTERNATIVE RAW MATERIAL FOR WHITEWARE BODY FORMULATIONS

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ABSTRACT

Groundnut shell ash (GSA) is a product of controlled burning of groundnut shells. The ash contains high percentage of silica with smaller amount of aluminium, iron, alkali and alkaline earth oxides. In this study an attempt has been made to use this waste ash as partial replacement for quartz in whiteware body formulations. Chemical analyses were carried out on the groundnut shell ash and other raw materials utilized for the body formulations. Six different samples were formulated by progressively replacing quartz with groundnut shell ash in steps of 5wt. % (from 0wt. % to 25wt. %). The samples were shaped, dried and fired between 1150^0C-1300^0C at the interval of 50^0C. Percentage linear shrinkage, flexural strength and water absorption tests were used to characterized the samples. The results obtained show that the linear shrinkage and flexural strength of the samples increased progressively with increase in GSA contents. The flexural strength of the samples increased with increasing substitution of quartz by GSA content up to 15% before decrease in strength is observed for most of the samples. 15wt. % groundnut shell ash containing sample fired at 1250^0C exhibited the maximum flexural strength value of 94.7MPa, which represents an increase in strength of 22.0% in comparison with the standard whiteware composition without GSA fired at the same temperature. The results of water absorption test show that the incorporation of GSA into the samples resulted in verification at low temperature in most of the samples. The microstructural examinations obtained from Scanning Electron Microscope (SEM) reveal that GSA containing samples have well-interlocked structure of mullite needles in the microstructure which enhanced their flexural strength.

INTRODUCTION

In recent times, researchers in material engineering field have devoted special attention to the waste minimization practices, pollution prevention, and sustainable development. The development of environment-friendly production processes has been a major focus. Despite this, large volumes of agricultural and industrial solid wastes are generated worldwide every day and the volume will still continue to increase [1]. One of the agricultural wastes usually generated in huge volume is groundnut shell. Groundnut is produced in large quantity in Nigeria and many other parts of the world. To date, limited use of the groundnut shells after extraction of the seeds has been reported in literatures [2-3]. Conversion of these agricultural wastes to products suitable for use as alternative raw material in ceramic will help to extend the frontiers of knowledge of whiteware development and applications, and contribute in agricultural waste management. Groundnut shell ash (GSA) is a product of controlled burning of groundnut shells. GSA contains appreciable amount of active silica (SiO_2) and other refractory oxides such as Alumina (Al_2O_3) and hematite (Fe_2O_3). It also contains small amount of alkalis and other trace elements. The chemical composition of GSA varies, depending on the variety, geographic location and climate.

About 80 million tonnes of groundnut shell ash is produced in Nigeria annually, which is mostly thrown away as waste [3].

The GSA waste is one of the major concerns of the agricultural industry which the common method of disposal in Nigeria is by burning in open air which causes pollution and destroys the soil nutrients. The management of this abundant waste in an environmentally safe way is challenge that must be met. To recycle the waste material in ceramic industry is an advantage of environmental protection and also saving the raw materials. Ceramic bodies, such as whiteware, are heterogeneous materials, consisting mainly of natural raw materials with wide range of composition [4]. For this reason, such bodies could tolerate different types of alternative raw materials, even in high percentages. Utilization of these agro-wastes for formulation of whiteware body will bring about reduction in the use of natural raw materials, reduces production cost, energy consumption as well as serving as a means of safe disposal of these agro-wastes [5].

Whitewares are usually manufactured by using different raw materials such as sodium or potassium feldspar, ceramics frits together with clay, kaolin and silica sand. The clay, gives plasticity to the ceramic mixture; flint or quartz (SiO_2), maintains the shape of the formed article during firing; and feldspar, serves as flux [6]. The products are usually shaped and sintered at high temperature. Quartz is one of the most important raw materials for whiteware production because of its ability to introduce crystalline phase in the body. However, several authors have reported that quartz grains embedded in the whiteware glassy matrix have a deleterious effect on the mechanical strength mainly because of its transformation during cooling which usually causes cracks in the body and results in the development of stresses which initiate fracture [7-9]. Kingery [10] reported that the thermo-mechanical properties of whiteware bodies change greatly during the reconstructive and the displacive transformation of free silica due to change in volume. Few authors have made attempts to replace quartz with different materials such as rice husk ash and fly ash in whiteware bodies and improvement in mechanical properties of the final products were reported [11-12].

In spite of the fact that groundnut shells are produced in large quantity in Nigeria and other parts of the world, insufficient attention has been devoted to characterizing this waste material as alternative raw material for whiteware bodies. In this present investigation, quartz was progressively replaced with GSA and its influences on the properties of whiteware compositions were extensively studied.

EXPERIMENTAL PROCEDURE

Materials

The raw materials selected for this research work were potash feldspar, calcined quartz, Kaolinitic clay, and groundnut shell ash. All the raw materials selected are abundant in Nigeria and they are currently being utilized for manufacturing different ceramic products.

Preparation of groundnut shell ash

Groundnut shells were gathered in large quantities. The groundnut shells were dried in the sun for three days so as to remove the water content from it and then to aid the burning process. A metallic drum with perforated holes on its body was used to burn the groundnut shells. The dried groundnut shells were placed in the metallic drum and burnt in open air in order to enhance the combustion. The ash obtained in the drum was allowed to cool in the drum before removal. The ash obtained was then conditioned in a furnace at the temperature of 650^0C for 180 minutes so as to reduce the carbonaceous and volatile constituents of the ash in accordance with [13]. After conditioning, sieve shaker was used to carry out particle size analysis of the groundnut shell ash. Chemical analysis was carried out on the GSA using X-ray fluorescence spectroscopy. The result of the chemical analysis is as presented in table 1

Mixtures preparation

A standard whiteware body composition (SP_0) was selected as the starting composition and calcined quartz was gradually replaced with GSA in the body compositions as shown in Table 2. The processed raw materials were weighed according to the composition in table 2 using accurate electronic weighing balance. The weighed raw materials were charged into pot mill containing porcelain grinding media (pebbles) and 35% water of the total charge was added together with sodium tripoliphosphate as deflocculant. The pot mill was allowed to run for two hour before its content was discharged into a dry pan. The pan and its content were dried in electric oven at the temperature of 105^0C for two hours. The dried material was then crushed and 7% weight of water was added before passing through a 100 mesh (150µm) to obtain suitable powders for pressing. The powder was used to form square samples of size 100mm x 100mm x 10mm by using uniaxial hydraulic pressing machine at the pressure of 50bars. The pieces were then dried at 110 ºC for 24 h. The samples were fired in an electric laboratory kiln between 1150^0C and 1300^0C at the interval of 50^0C, and with soaking time at the maximum temperature of 1 h. The following technological properties were determined in the fired pieces in accordance with standardized procedures: linear shrinkage, water absorption, and flexural strength. The microstructural features were also studied by SEM on fracture-etched surfaces. The fracture-etched specimens, etched in 10% HF for 3 min were studied for the existence of various phases in the fired specimens.

RESULTS AND DISCUSSION

Characterization of raw materials

The results of chemical analyses of the raw materials are as presented in table 1. From the results presented in table 1, it is observed that the GSA contains a large amount of silicon oxide (SiO_2) which plays a major role in the formation of mullite phase, and to a lesser extent aluminum oxide (Al_2O_3), iron oxide (Fe_2O_3), calcium oxide (CaO), and potassium oxide (K_2O). The results of analyses carried out on other raw materials show that they have constituents suitable for producing whiteware body as reported by Norton [14].

Technical Properties

The linear shrinkage indicates the degree of densification during firing, and is very important for the dimensional control of the clay ceramic products. The technical properties of the whiteware samples fired at different temperature range (1150^0C, 1200^0C, 1250^0C and 1300^0C) are shown in figure 1-3. The firing shrinkage of the samples (Fig. 1) is observed to increase progressively with increase in the percentage of GSA. The increase in firing shrinkage of 39% is observed for 25wt% GSA containing sample (SP_{25}) in comparison with standard whiteware body composition (SP_0) at maturing temperature of 1150^0C. In the case of samples fired at 1300^0C, increase in fired shrinkage of 8% is observed for 25wt% GSA containing sample (SP_{25}) in comparison with the standard whiteware body composition (SP_0). Fired shrinkage value for all the samples ranges between 7.4% and 12.4%. This increase in fired shrinkage of the samples can be attributed to floppy nature of groundnut shell ash incorporated into the whiteware body compositions which tend to undergo much shrinkage during firing of the samples.

Figure 2 shows the flexural strength test result for the samples. The results show that the flexural strength of the samples increased with increasing substitution of quartz by GSA content up to 15% before decrease in strength is observed for most of the samples. 15wt% groundnut shell ash containing sample (SP_{15}) fired at 1250^0C exhibited the maximum flexural strength value of 94.7MPa, this represents an increase in strength of 22.0% in comparison with the standard whiteware composition (SP_0) without GSA fired at the same temperature. Also, decrease in flexural strength value of 21.3% is observed on complete replacement of quartz with GSA in sample SP_{25} fired at 1250^0C in comparison with 15wt% GSA containing sample fired at the same temperature. It is also observed from figure 2 that there was slight decrease in flexural strength (3.27%) for 15wt% GSA containing sample (SP_{15}) fired at 1300^0C in comparison with same sample fired at 1250^0C. The decrease in flexural strength for 20wt% and 25wt% GSA containing samples can be attributed to increase in glassy phase of the microstructure due to higher percentage of GSA in the composition. Islam, *et al* [15] has reported that the best mechanical and dielectric properties can be achieved in whiteware body by high mullite and quartz content with low amount of the glassy phase and in absence of micro cracks.

The water absorption results for the samples are presented in figure 3. It can be observed from figure 3 that 20wt% and 25wt% GSA containing samples (SP_{20} and SP_{25}) fired at 1150^0C attained 0.2% water absorption value when compared with standard whiteware composition (SP_0) fired at the same temperature with water absorption value of 3.7%. The standard whiteware composition (SP_0) fired at 1300^0C attained early verification with water absorption value of 0%, on complete replacement of quartz with GSA in the body composition, the water absorption value increased to 0.8%. The results of water absorption rate indicated that replacement of GSA with quartz in the body composition resulted in the formation of additional liquid phase and the enhancement of the densification process. These results obtained are in agreement with previous investigation reported elsewhere [11].

Microstructural characterization of the samples

Microstructural changes in the whiteware bodies were examined with scanning electron microscope (SEM). Scanning electron micrographs of fracture — etched surfaces of the specimens are presented in figure 4(a-d). The samples exhibit different shapes of the mullite needles upon firing depending on the amount of glass forming constituent in the composition. Detailed SEM in figure 4(a) indicates the presence of pores in the standard whiteware body without GSA fired at 1150^0C. The high water absorption rate of the sample and low strength could be attributed to presence of the pores. Figure 4(b) shows the detail SEM image for 15wt. % GSA containing samples fired at 1150^0C. The image shows that the sample contains different shapes of mullite needles with very few pores which are responsible for the improvement in flexural strength of the sample.

Figure 4(c) and 4(d) show SEM images of standard whiteware body composition and 15wt. % GSA containing sample fired at 1300^0C. Figure 4(c) indicates presence of pores with few mullite needles while figure 4(d) extensively shows interlocked secondary mullite needles embedded in the glassy matrix which is responsible for improvement in flexural strength. An optimum temperature and composition at which considerable amounts of quartz and mullite crystals existed in the glass phase maximized the physical and mechanical properties of the whiteware bodies.

Table 1. Chemical analysis of Raw Materials

Constituents	Potash feldspar	Kaolinitic clay	Quartz	Groundnut Shell Ash
SiO_2	65.66	47.21	96.10	62.72
Al_2O_3	15.48	35.47	1.07	7.64
Fe_2O_3	0.74	0.60	0.10	5.27

MgO	0.28	0.05	0.03	2.13
K_2O	9.04	0.73	0.12	8.47
Na_2O	3.12	0.08	0.51	1.02
CaO	0.87	0.12	0.11	6.74
LOI	3.26	12.83	0.64	3.78

*LOI represents loss on Ignition

Table 2. Mixture Composition (Wt %)

Mixtures	Raw Materials			
	Kaolinitic Clay	Potash Feldspar	Quartz	GSA
SP_0	50	25	25	0
SP_5	50	25	20	5
SP_{10}	50	25	15	10
SP_{15}	50	25	10	15
SP_{20}	50	25	5	20
SP_{25}	50	25	0	25

Figure 1. Result of Fired Shrinkage Test for Samples Fired at Different Temperature

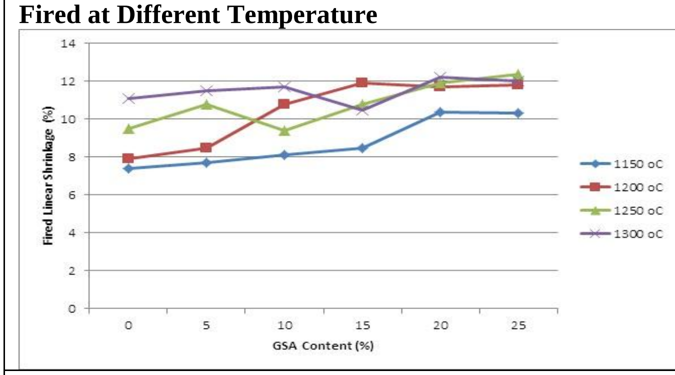


Figure 2. Result of Flexural Strength Test for Samples Fired at Different Temperature

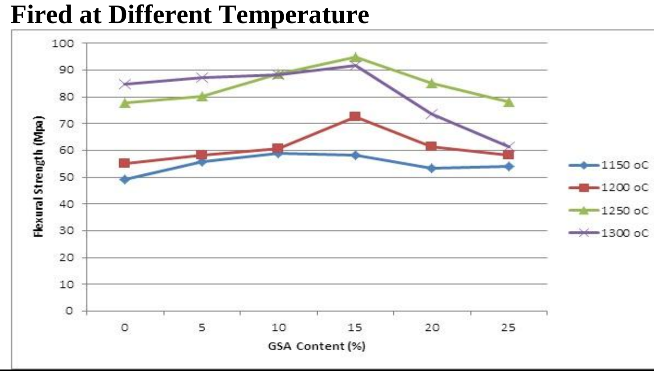


Figure 3. Result of Water Absorption Test for Samples Fired at Different Temperature

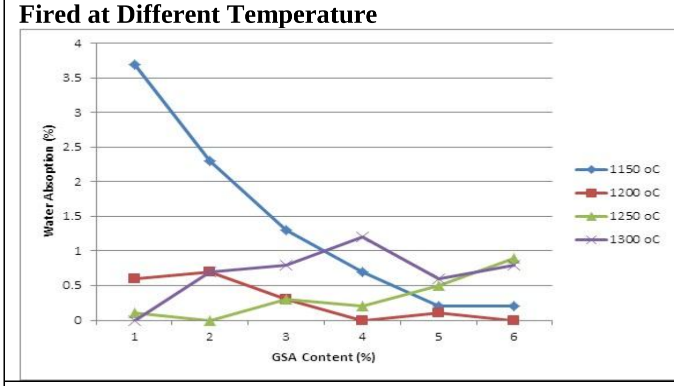


Figure 4(a). SEM Image of Standard whiteware Body Fired at 1150^0C (SP_0)

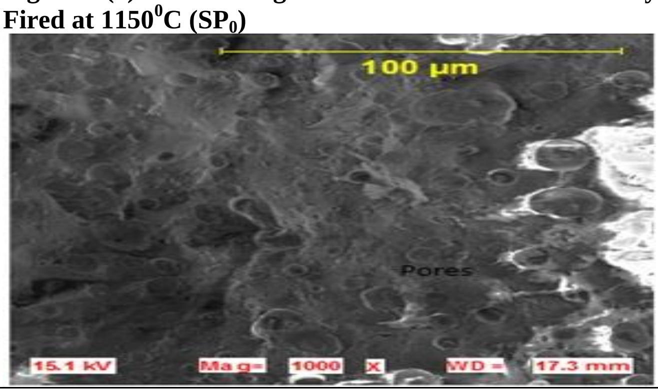


Figure 4(b). SEM image of 15wt. % GSA Containing Whiteware Body Fired at1150^0C (SP_{15})

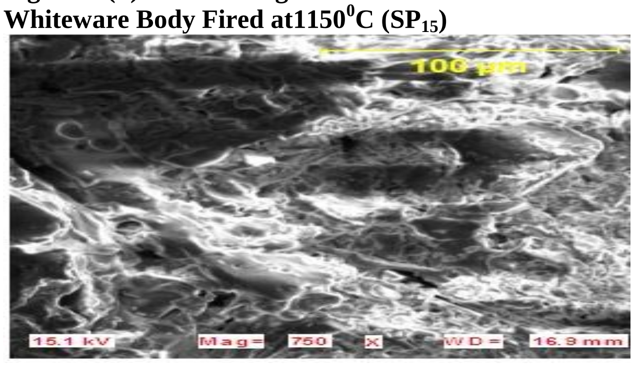


Figure 4(c). SEM Image of Standard whiteware Body Fired at 1300^0C (SP_0)

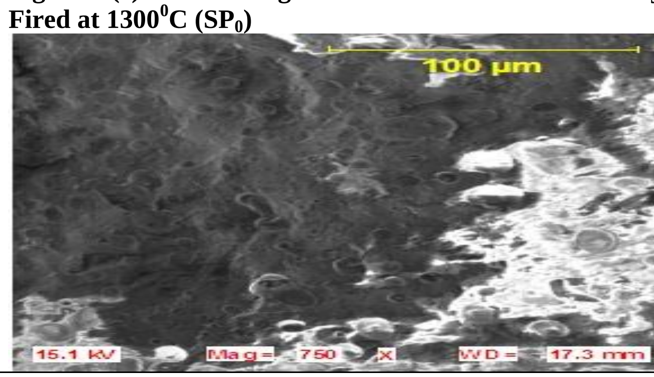
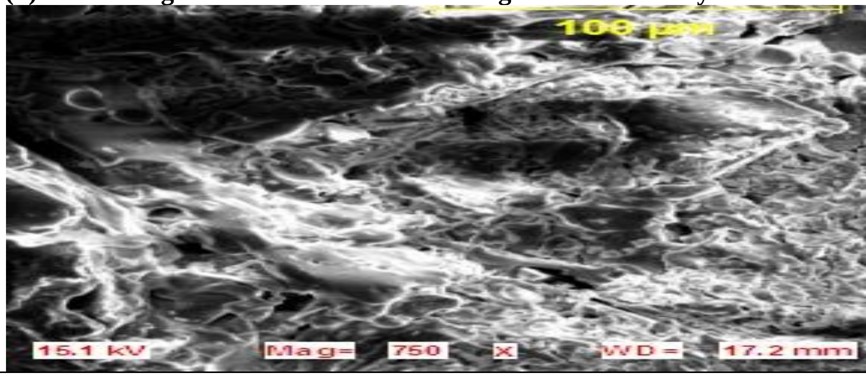


Figure 4(d). SEM Image of 15wt. % GSA Containing Whiteware Body Fired at 1300^{0}C (SP$_{15}$)



CONCLUSION

In this research work, the partial replacement of quartz by GSA in standard whiteware body composition was investigated. The results obtained show that the linear shrinkage and flexural strength of the samples increased with addition of GSA in the body formulation with 15wt. % GSA containing samples fired at different temperatures exhibiting the maximum flexural strength values. The results of water absorption rate also indicated that GSA containing samples attained early verification at low temperature which implies that the addition of GSA result in low water absorption rate at low temperature. The SEM images of the samples also revealed that the addition of 15wt. % GSA increased the formation of mullit phase which enhanced the physical properties of the whiteware bodies. These results affirmed that whiteware body with improve physical properties can be produced at low temperature by incorporating 15wt. % GSA into the body formulation.

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