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DEVELOPMENT OF POROUS CERAMICS FROM LOCALLY SOURCED KANKARA CLAY

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In this study, porous ceramic was produced from Kankara clay with cassava starch as a pore former. Physical properties of the clay were determined using particles size distribution and FESEM. Chemical, phase analysis and thermal properties of the clay were determined using XRF, XRD and TGA/DTA respectively. To determine the porous ceramic properties, the clay was mixed with cassava starch 5-30wt%, compacted at a pressure of 60MPa and fired at 1200°C. Maturation properties of the fired ceramics were determined using bulk density and apparent porosity. In addition, the mineralogy and morphology of the fired porous ceramics were determined using XRD and FESEM respectively. From the results of the XRF, XRD and TGA/DTA, the clay is kaolin which transformed to mullite after firing at 1200°C. The bulk density and the apparent porosity obtained were in the range 1.44-2.12g/cm^3 and 12-47% respectively. The FESEM morphology of the porous ceramics shows that the porosity increases with percentage starch content. This shows that porous ceramics can be produced from local Kankara clay which can be used in filtration and separation applications.

Keywords: Kankara clay; mulllite, bulk density; apparent porosity

1.0 INTRODUCTION

Traditional clays have been used to produce ceramic materials such as white wares, sanitary wares, kitchen wares and tiles. Recently, clays are being used to produce porous ceramics for more advanced applications such as filtration and purification. Different materials such as steel, wood, wire screens, fibre glass, bonded aggregates, pressed ceramic strainers and ceramic forms have been used to developed porous materials. However, high chemical, mechanical and thermal stability of porous ceramics coupled with the ability to tailor the pore sizes for specific operation has increased the demand for porous ceramics in industrial applications (Kwame Efavi *et al.*, 2012).Porous ceramics have wide applications in several industries. For example, in foundry operations, porous ceramic filters have been used to separate molten metal from suspended particles captured in the melt. In tanning, electroplating, juice and mining industries, porous ceramics have been used to treat the wastewater generated from these industries. Many researches have been conducted on the use of traditional clay as porous ceramic materials. For example, (Kwame *et al.*, 2005) conducted a research on the development of porous ceramic from kaolin deposits in Ghana, an apparent porosity as high as 38% was achieved.(Vasanth *et al.*, 2011), reported the fabrication of porous ceramic from kaolin and quartz, porosity of 30% and shrinkage of 12% have been reported. In another research,(Vasanth *et al.*, 2012) reported a porosity of 30% from a porous ceramic prepared from kaolin..(Emani *et al.*, 2014a), reported an apparent porosity in the range of 30.1-37.4% when preparing a porous ceramic from kaolin clay. In addition, (Emani *et al.*, 2014b), reported the fabrication of porous ceramics from kaolin clay and a porosity of 37% has been reported.

Furthermore, clays from different countries have been used to fabricate porous ceramic materials.(Harabi *et al.*, 2012) developed a porous ceramics from Algerian clay; a porosity of 48% and average pore size of

7µm have been achieved. In addition, (Majouli *et al.*, 2012) produced porous ceramics from Moroccan clay an average pore size and porosity of 6.6 µm and 42% were obtained respectively.(Hedfi *et al.*, 2014) produced porous ceramics from Tunisian clay and reported an average porosity and bulk density of 30% and 1.85g/cm^3 respectively.

Nigeria has the largest deposits of clay in the African continent but such deposits are highly underutilized (Ekosse, 2010). Clay deposits have been found in all states of Nigeria, which contain kaolinite $Al_2Si_2O_5(OH)_4$ with the growing demand of ceramics and expensive nature of engineering, inexpensive clay minerals are used as a starting materials for the development of porous ceramics.

Several researchers have reported the use of kaolin clay for the development of porous ceramics. However, there is no research on the development of porous ceramics from Nigerian clay. The objective of this work is to characterize Kankara clay and produce a porous ceramic from the same clay.

2.0 MATERIALS AND METHODS

The clay used was obtained from Kankara local government in Katsina state. Chemical composition, thermal properties and mineralogical composition of the clay was determined using XRF(Philip PW2400),Particle size distribution (PSD) on the clay sieved through 50µm was conducted using Malvern particle size analyzer. Thermal analysis (TGA/DTA) of the clay was performed using PerkinElmer. Phases present in the clay and the fired clay were determined using XRD machines Siemens D500. The clay was mixed with starch in the range 5-30%wt and pressed at 60MPa using Instron machine. The pressed clay was fired at different temperatures (1000-1200°C). Mineralogical properties of the fired clays were determined using XRD.

The porous fired specimens were initially weighed in air before being weighed suspended in deionised water inside a desiccator. The desiccator was connected to a vacuum pump and pumped to ensure complete penetration of water into the pores of the fired specimens for 24 hours. After 24 hours, the specimens were gently rubbed with a cloth to remove the surface water not absorbed by the porous specimens and re-weighed; the mass increase after the immersion was recorded. Finally, the specimens were immersed in deionized water and the mass inside water was recorded(ASTM C373-88, (1999) and ASTM B962-14, (2014))

The morphology of the porous ceramics wasrecorded using Field Emission Scanning Electron Microscopy (FESEM).

3.0 RESULTS AND DISCUSSION

3.1 Chemical composition and particle size distribution

The chemical composition of the clay sieved through 50µm (Table 1) shows that Al_2O_3 and SiO_2 are the major compounds found in the clay. Other compounds present in the clay are potassium oxide (K_2O), iron oxide (Fe_2O_3), calicium oxide (CaO), titanium oxide (TiO_2), magnesium oxide (MgO), manganese oxide (MnO) and potasium oxide (P_2O_5).

Table 1 Chemical composition of Kankara clay used in this research (wt%)

Compound	%wt
SiO_2	48.86
Al_2O_3	37.83
K_2O	1.15
Fe_2O_3	0.27
CaO	0.05
TiO_2	0.04
MgO	0.04
MnO	0.01
P_2O_5	0.01
LOI	11.81

Particle size distribution of the clay is depicted in Figure 1. As can be seen from figure, the clay has a unimodal population distribution with a maximum peak at 19.31µm, which presents the modal particle size of the clay. The mean ze of the clay was found to be approximately 16.19μm.

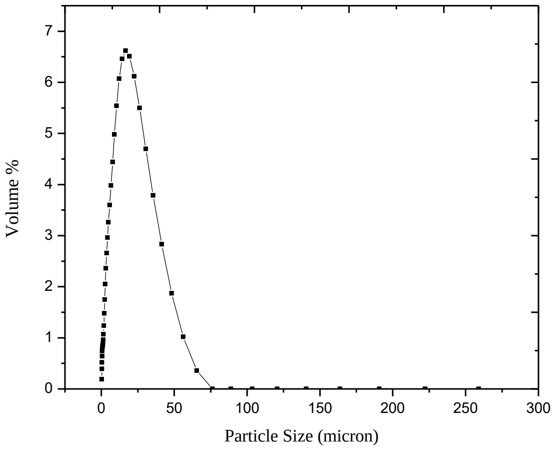

Figure 1 Particle size distribution of Kankara clay sieved to 50μm

3.2 Thermal analysis of clay, glycerol and starch mixture

The thermal analysis by differential thermal and thermogravimetric analyses of the clay mixture with glycerol (binder) and starch is shown in Figure 2. The DTA analysis of the clay shows various endothermic and exothermic peaks during heating to 1200°C. The first peak at a temperature within 70-100°C is due to the removal of physical combined water present in the clay. The peak at the temperature range 200-250°C is due burn out of starch. The peak at 480°C is due to the conversion of kaolin to metakaolin (equation 2.5). The peak at 1000°C is due to the formation of amorphous silica and conversions of metakaolin to spinel, while at 1100°C, spinel converted to mullite. The formation of mullite is in agreement with Lecomte-Nana *et al.*, (2011). The importance of this analysis is that it shows the various reactions/degradation of the mixture (clay, starch and glycerol) at various temperatures so that a heating cycle can be design to avoid cracking during firing.

The TGA analysis (Figure 2) shows various decreases in mass due to the glycerol and starch burn out, transformation of kaolin into metakaolin. The decrease in mass of kaolin, starch and glycerol mixed in the clay from a temperature of 70°C to 900°C is about 12.9%.

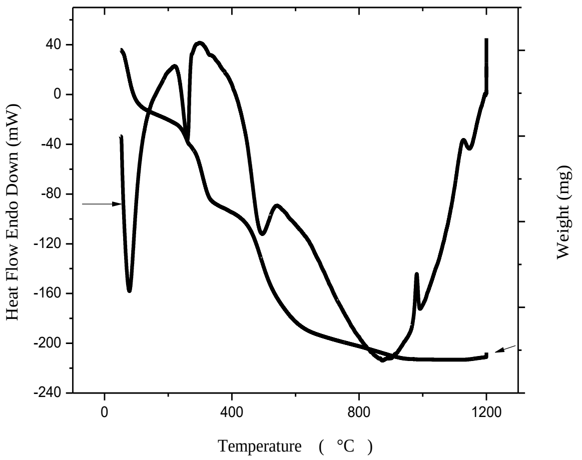

Figure 2 TGA/DTA analyses of Kankara clay

3.3 Phase analysis of raw clay and fired clay

XRD phase analysis of the raw clay is shown in Figure 3. The clay contains sharp peaks of kaolinite phase, with some peaks of illite and quartz. The relative low intensity peak of the quartz in the clay indicates insignificance presence of free silica. These observations were reported by Ptacek, (2010); Panda *et al.*, (2010); Gougazeh and Buhl, (2014).

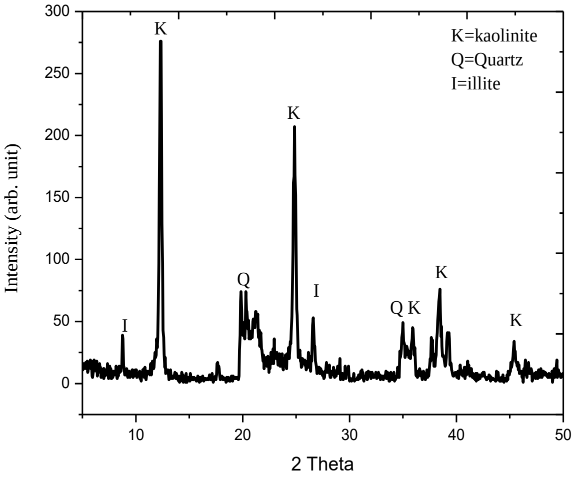

Figure 3 XRD spectrum of Kankara clay

The XRD patterns of the specimens at temperatures of 1000-1200°C were presented in Figure 4. At 100°C, the XRD pattern shows the existence of quartz phase. After firing to temperature 1100°C, the intensity of the quartz peak diminishes.

Further heating to 1200°C, mullite phase appears. Heating from 1000-1100°C, only spinel appears in the XRD results, the spinel formed later converted to mullite. However, faster heating rate promotes the formation of mullite direct from spinel. Heating rate of 3-20°C/min is enough to cause the formation of mullite direct from spinel without any cristobalite intermediate phase. This observation has been reported by Castelein *et al.,* (2001).

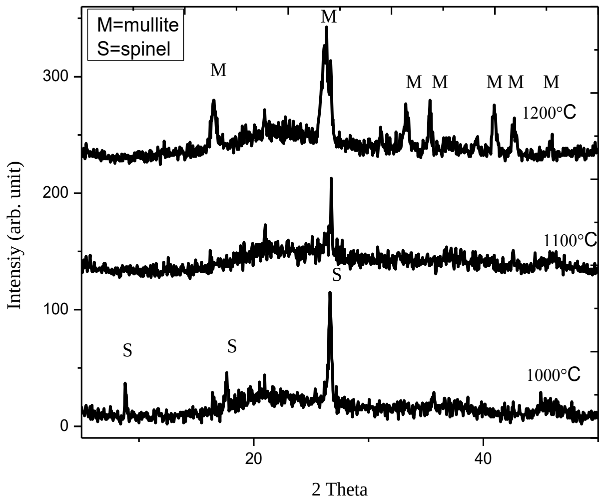

Figure 4 XRD analysis of fired specimens from 1000-1200°C

3.4 Apparent porosity and bulk density

The variation of apparent porosity and bulk density with varying percentage of starch at firing temperature of 1200°C is presented in Figure 5 (optimum firing temperature of the clay). The plot shows an increase in porosity as the percentage of starch increases. Also, the plot shows decrease in bulk density with increase in starch content. This behaviour of increase in apparent porosity and decrease in bulk density can be attributed to the starch content, which completely burns out at temperature of 460-520°C as shown in the TGA/DTA diagram (Figure 2), which resulted in the formation of porosity and voids. The higher the starch content, the more the population of voids/porosity is expected. The decrease in the bulk density is the consequent of an increase in inter-particle void volume and internal void volume created as the result of the starch burnout. The inter-particle and internal void volume created increases with increasing percentage of starch in the specimens. Hence, this results in a decrease in the bulk density. There is an inverse relationship between bulk density and apparent porosity; the higher the apparent porosity the lower the bulk density and vice versa.

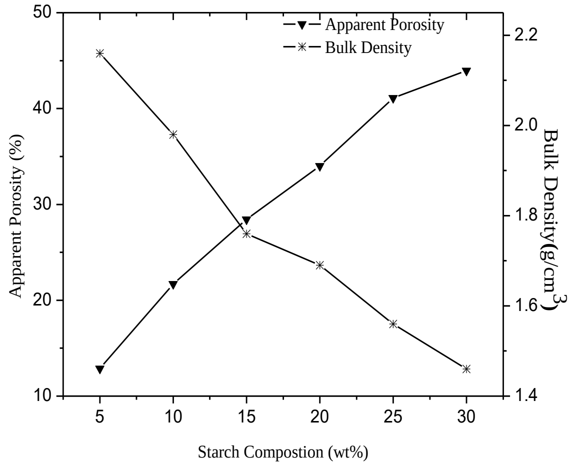

Figure 5 Variation of apparent porosity and bulk density at 1200°C

3.5 Morphology of porous fired clay

The morphology of specimens fired at 1200°C is shown in Figure 6. Figure 6a, which contains 5wt% starch, shows pockets of relatively rounded pores which are not uniformly distributed through specimen. Figures 6b and 6c show the morphology of specimens containing 10 and 15wt% starch respectively, which show the porosity formed are relatively of rounded shape (slit pores) with relative uniform distribution throughout the morphology. Figures 6d-f of specimens containing 20, 25 and 30wt% respectively, show the presence of interconnected pores in their morphology, this is as a result of the starch content, which make the slit pores to coalesce due to the burn out of the starch at firing temperature of 1200°C. The microstructures (Figures 6d-f) seem to open up due to increasing amount of starch burn-out as its content increases. Increase in starch to ceramic products increases their porosity and interconnection of pores after firing (Yang and Tsai, 2008; Almeida *et al.*, 2009; Sahnoun and Baklouti, 2013).

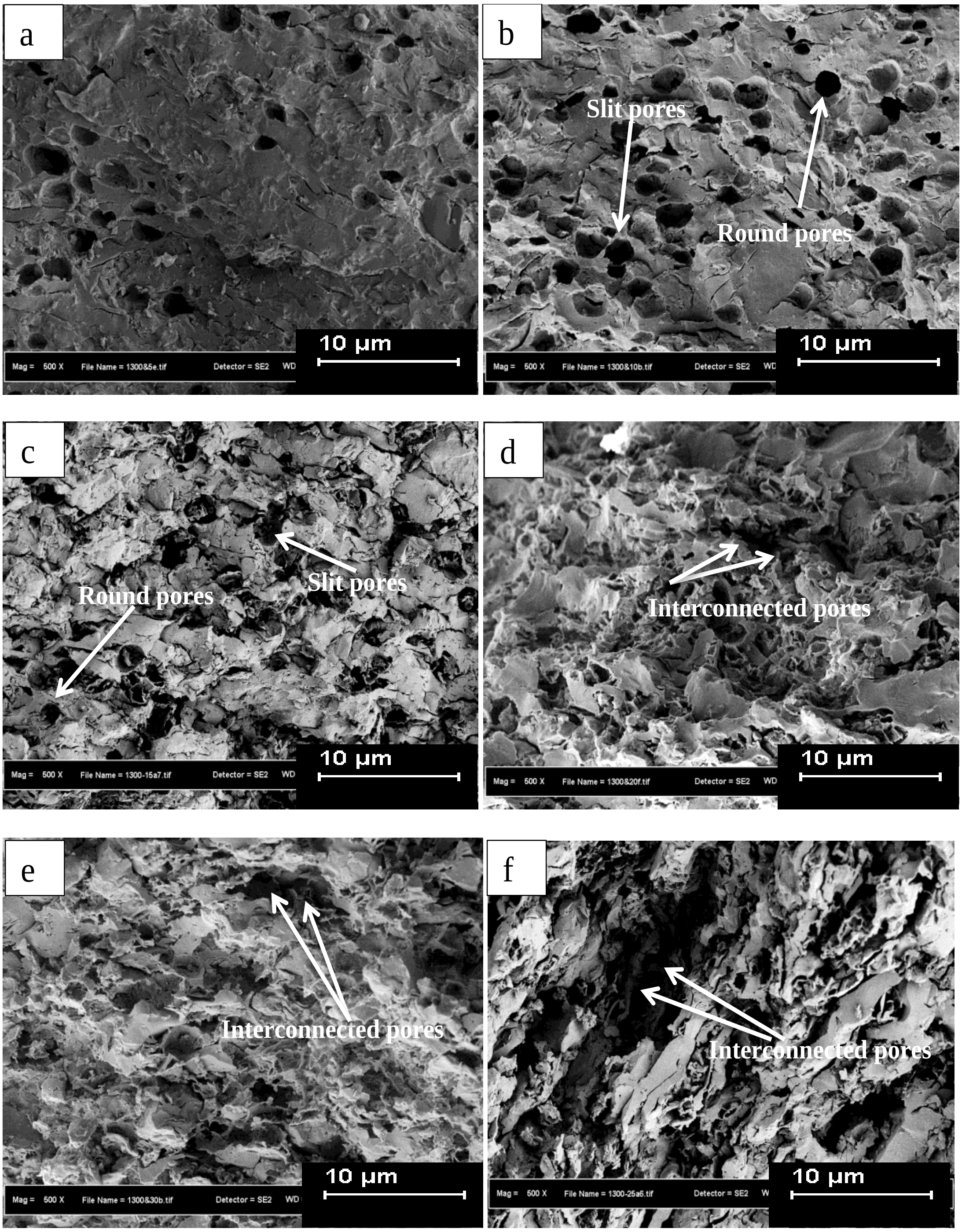

Figure 6 Fractured surface morphology of fired specimens (a) 5wt% (b) 10wt% (c) 15wt% (d) 20wt% (e) 25wt% and (f) 30wt% at 1200°C

4.0 CONCLUSION

In this research, porous ceramics were produced from Kankara clay by mixing with cassava starch and fired at 1200°C. From the TGA/DTA results and XRD it shows that 1200°C is enough for the densification of the clay. Bulk density of the porous ceramics shows decrease with increase stach content. However the apparent porosity shows increase with increase starch content. The FESEM morphology shows that as percentage starch increases from 5-30wt% amount of porosity increases by formation of interconnected pores. The produced porous ceramics will have application in filtration and separation processes.

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