Physical and Mechanical Properties of Porous Kaolin Based Ceramics at Different Sintering Temperatures

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(Received 25 November 2015; Revised 17 April 2016; Accepted 6 June 2016)

Abstract: In this work, kaolin based ceramics using styrofoam, sawdust, and powdery high density polyethylene as pore formers were experimentally investigated. Prior to batch formulations, the kaolinite used was wet beneficiated. This was followed by mixing starting materials with pore formers, producing green bodies which were then uni-axially compacted into standard sample dimensions and fired at various sintering temperatures of 850°C, 1000°C and 1150°C for 2 hours in a furnace. The physical and mechanical properties of the sintered samples were investigated and the generated data analyzed. It was observed that the apparent porosity and water absorption of the samples decreased with increased sintering temperature, while the bulk density, apparent density and cold crushing strength of the samples increased with increased sintering temperature. It was concluded that the samples which were sintered at 850°C with 5% wt pore former of powdery high density polyethylene gave the optimum properties in terms of the porosity and mechanical strength of the samples.

Keywords: Sintering temperature, Kaolin based ceramics, Pore formers

1. Introduction
Porous ceramics have attained increasing importance in industry recently due to their numerous applications and utilisation involving different materials like metals, polymers, composites, semiconductors and biomaterials (Muhammad, 2008). Porosity can affect performance properties, strength (both flexural and compressive), and density of materials. There has been a long tradition in producing porous materials mainly for structural applications which include concrete, cements, bricks and refractories (Nettleship, 1996). In all applications of porous material, transport through the pore phase is very important and can be achieved if the materials contain more than 10% connected porosity and pore volume. This type of porous ceramic finds key applications as supports for heterogeneous catalysts, membranes for bioreactors, environmental filters for hot flue gases and diesel engine emissions.

The development of porous ceramic materials has presented a new challenge to several industries, because porous ceramics are specifically more durable in extreme environments and their surface characteristics permit them to fulfil very specific requirements. With the incessant demand for porous ceramics in industrial applications, a number of technologies have been developed lately for fabricating these materials and their pore characteristics, and for identifying pore-related properties. A tailor-made porous ceramic can be made through a critical understanding and interpretation of the relationship between various pore-related properties and optimising them for specific uses. Nowadays, different porous ceramics with more delicate and uniform pore structures, having wide pore size range (few micrometers to a few nanometers) can be prepared for diverse purposes via varied physical and chemical processing (Bilung, 2012). Figure 1 is a pictorial view of well distributed pores in a ceramic body.

Porous ceramics with well-defined macroscopic shapes and also high mechanical stability can be fabricated using novel processing routes, while retaining the inherent porosity of the porous powder from which they are produced (Sheng, 2001; Dibandjo et al, 2008). Sintering is a thermal process that transforms a compact powder into a bulk material, and is used in mass-producing complex-shaped components. Sintering is one of the most important technological processes in the powder metallurgy and ceramic industries. A rational theory of sintering should predict the routes for production of the required structure of a sintered body in order to provide the desired physio-chemical and physio-mechanical properties (Olevsky, 1998).
Nigeria and Ghana are endowed with large deposits of ceramic raw materials such as pegmatite; alumina; montmorillonite and illite; mica; clay; feldspar; quartz; and bauxite. Clay mineral deposits have been found in most regions of Nigeria and Ghana and are mainly high grade clays containing kaolinite \( \text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \). With the growing demand for porous ceramics in industrial applications, several technologies have been developed for fabricating these materials while also attempting to control their pore characteristics and properties (Sheppard, 1992; Montonnaro et al., 1996).

Numerous pore-forming agents have been investigated such as:

- Starch (Sheffler, 2005; Zivcova et al., 2012; Khatab et al., 2012; Garrido et al., 2011; Chen et al., 2011; Zivcova et al., 2009; Zivcova et al., 2010)
- Graphite (Gregorova et al., 2006; Sarikaya and Dogan, 2013; Sanson et al, 2008; Ding et al., 2007)
- Lycopodium (Boaro et al., 2003; Zivcova et al., 2007; Zivcova et al., 2008)
- Sucrose (Sarikaya and Dogan, 2013; Ray et al., 2010; Wang et al., 2005)
- Polymethyl methacrylate (Zeng et al., 2007; Yao et al., 2005; Kumar et al., 2005).

Even though starch is the most frequently used pore forming agent, possibly due to its biological origin and availability, the difficulties in maintaining the pore structure formed by the starch burn out, and the narrow size range of commercially available starch types (typically between 5 and 50 μm) limits its application when large pores are desirable (Gregorova and Pabst, 2007). Svinka et al. (2009) studied the production of porous alumina ceramics by the slurry casting method and investigated pore formation by elimination of hydrogen as a result of a chemical reaction of aluminium powder with water. The purpose of their study was to determine various ways of producing high porosity alumina ceramics having high mechanical strength and other properties significant for refractory ceramics.

The commercial ceramic supports are traditionally manufactured from compounds such as alumina \( (\text{Al}_2\text{O}_3) \), cordierite \( (2\text{MgO}.2\text{Al}_2\text{O}_3.5\text{SiO}_2) \), and mullite \( (3\text{Al}_2\text{O}_3.2\text{SiO}_2) \) (Zhong, et al., 2002; Saffaj, et al., 2004; Mohammadi and Pak, 2003), all of which have a relatively elevated cost. More recently, different processing routes for kaolin based ceramic and membrane supports have been proposed (Bouzerara et al, 2006) in order to decrease cost of manufacturing supports and to evaluate the feasibility of using local natural resources (Bouzerara et al., 2006; Zhou et al., 2008).

Therefore, this work investigates the physio-mechanical properties of low-cost kaolin based ceramics using styrofoam, sawdust, and high density polyethylene as pore formers at varying temperatures for its possible application as catalyst supports amongst others. It is important to highlight the use of powdery high density polyethylene (PHDPE) as a pore former, because to the best of our knowledge PHDPE has not been explored as a pore former in castable ceramics.

2. Experimental Procedure

The clay materials that were used in this study were obtained from Kankara and Kibi Kaolin deposits in Nigeria and Ghana respectively. The sawdust used was obtained from a local sawmill in Ghana. The styrofoam and powdery high density polyethylene powders were processed at the Materials Laboratory of the University of Ghana.

2.1 Raw material beneficiation and powder preparation

The mined kaolinite containing impurities was removed by washing, sieving and drying of the slurry in a Plaster of Paris (POP) mould. The washing was done by adding water to a constantly stirred kaolin blunger. The slurry was then passed through a sieve cloth to eliminate coarse grained impurities. The slurry filtrate was poured into a plaster of Paris (P.O.P) mould. The water was then separated from slurry by capillary action. Next, the solid was further dried in an electric oven for 7 hours. The dried samples were fed into a Thomas Hammer Mill (Arthur H. Thomas Co, USA), to break the soft lumps into powder.

Pellets were made by dry pressing in a hydraulic press (Carver Press USA) at a load of 3.5 MPa, with a 25% pump speed and dwell time of 90 seconds. A high carbon, high chrome steel die (12.5 mm diameter) was used. The pellet thickness was 6 mm. Acetone was used for cleaning the die to prevent contamination and sticking and 5% stearic acid solution was used for lubrication.
2.2 Characterisation Techniques for Kaolin
The essential properties of the raw material such as particle size ranges were experimentally determined. A laser method, low-angle laser light scattering (LALLS), was used for the particle size analysis with levels of sensitivity in the 0nm to 1000nm micron range using a nanoparticle analyser (Horiba Scientific, SZ-100). 10mg of the kaolin samples (from Kankara and Kibi) plus freshly deionized water (10mg of kaolinite + 10 ml of water) were subjected to continuous ultrasound treatment in an ultrasonic bath for 15 mins to ensure dilution and homogenous dispersion. The pore formers comprising saw dust, styrofoam and high density polyethylene were prepared using an analytical mill to reduce the sizes.

X-ray diffraction (XRD) was performed on powders of kaolin clay to determine their phase compositions. This was done on an empyrean diffractometer (Bruker AXS, D8 Advance) with theta/theta geometry, operating a Cu Kα radiation tube (λ = 1.5418 Å) at 40 kV and 30 mA. The XRD patterns of all the randomly oriented powder specimens were recorded in the 10.0°-90° 2θ range with a step size of 0.017° and a counting time of 14 s per step. The surface morphology of the kaolin was investigated using an ultra-high vacuum and high resolution FEI, XL-30 scanning electron microscopy. Samples were metalized with gold/platinum coating prior to the analysis. Images were acquired at magnifications of 8000 and 15000 X.

2.2 Sintering of compacted pellets
The compacted pellets were sintered in a muffle furnace at 850°C, 1000°C and 1150°C. At each sintering temperature, the holding times for different batches were 2 hours. The samples were heated at 5 °C/min till final sintering temperature. The furnace was switched off for cooling and the samples were taken after the furnace temperature reached below 100 °C.

2.3 Physical Properties’ Test
Physical properties tested for were apparent porosity; bulk density; apparent density; percentage water absorption; and total shrinkage. The test specimens were dried at 100°C for 24 hours to ensure total water loss, and later fired up to 850°C, 1000°C and 1150°C in an electric furnace. Their fired weights were measured and recorded. They were then allowed to cool in a beaker of water. Bubbles were observed as the pores in the specimens were filled with water. Their soaked weights were measured and recorded. They were then suspended in a beaker one after the other using a sling. Each specimen was lightly wiped with a moistened smooth cotton cloth to remove all excess water from the surface, and then the saturated weight, W, was recorded.

The apparent porosity (the amount of void or pores within a volume of porous solid) of the kaolin based ceramic bodies was calculated using Eq. (1):

\[
\text{Apparent Porosity} = \frac{(W-D)}{(W-S)} \times 100
\]  

Their respective bulk densities, apparent densities and percentage water absorption were calculated using the formulae proposed by Chesti (1986):

\[
\text{Bulk Density} = \frac{D}{(W-S)} \times 100 \text{ (g/cm}^3\text{)}
\]

\[
\text{Apparent Density} = \frac{D}{(D-S)} \times 100 \text{ (g/cm}^3\text{)}
\]

\[
\text{Water Absorption} = \frac{W}{(W-S)} \times 100
\]

Where: D = Weight of fired specimen, S = Weight of fired specimen suspended in water, and W = Weight of soaked specimen suspended in air.

Also the total shrinkage was calculated for each test specimen using the following formula taken from Norsker (1987):

\[
\%\text{Total Shrinkage} = \frac{\text{OL-FL}}{\text{OL}} \times 100
\]

Where: OL means original length; DL stands for dry length and FL is fired length.

2.4 Mechanical Property Test
The mechanical property tested for was cold crushing strength. It was used to determine the compression strength at failure for each sample which is an indication of its probable performance under load. The standard ceramic samples were dried in an oven at a temperature of 110 °C and allowed to cool. The compressive strength testing was carried out on a hydraulic testing machine (Carver Press, USA). The compressive strengths of the porous clay ceramics were obtained by compressive loading of samples prepared according to ASTM C133-97 (2003); and the cold crushing strengths, of standard and conditioned samples, were calculated from the equation:

\[
\text{CS} = \frac{P}{BW}
\]

Where: P is load at failure; and B and W are the respective width and breadth of the specimens.

Table 1 shows the batch formulations of clay (Kankara and Kibi) and pore formers (sawdust, styrofoam and powdery high density polyethylene in four different ratios (clay-to-pore formers) by weight.

3. Results and Discussion
3.1 SEM Images of Kaolin
The SEM images of the beneficiated Kankara and Kibi kaolin respectively scanned at 8000x and 15000x shown in Figures 4 and 5, reveal the platelet structure of kaolinite clay reported in the literature (Abo-El-Enein, 2013; Bergaya et al., 2013) which normally portrays booklets morphology. The average particle size was
estimated as 2.0 μm for both the raw and beneficiated kaolin.

Figure 6 shows the XRD pattern of the beneficiated Kankara and Kibi kaolin. It could be seen from the XRD pattern that the peaks at Bragg’s angles of 12.35, 19.89, 20.38, 24.88, 34.94, 35.95, 36.06, 38.35, 45.24, 54.88 and 62.37° responsible for the kaolinite mineral were more prominent after beneficiation. The XRD pattern was dominated by the kaolinite peaks after beneficiation of the kaolin.

Table 1. Composition of test Samples by weight (Total weight = 100g)

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Kaolin (g)</th>
<th>Plasticizer (Kibi Kaolin) (g)</th>
<th>Saw dust (g)</th>
<th>Styrofoam</th>
<th>High Density Polyethylene (HDPE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0% CS</td>
<td>80</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>5%SD</td>
<td>75</td>
<td>20</td>
<td>5</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>10%SD</td>
<td>70</td>
<td>20</td>
<td>10</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>15%SD</td>
<td>65</td>
<td>20</td>
<td>15</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>20%SD</td>
<td>60</td>
<td>20</td>
<td>20</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>5%SYF</td>
<td>75</td>
<td>20</td>
<td>-</td>
<td>5</td>
<td>-</td>
</tr>
<tr>
<td>10%SYF</td>
<td>70</td>
<td>20</td>
<td>-</td>
<td>10</td>
<td>-</td>
</tr>
<tr>
<td>15%SYF</td>
<td>65</td>
<td>20</td>
<td>-</td>
<td>15</td>
<td>-</td>
</tr>
<tr>
<td>20%SYF</td>
<td>60</td>
<td>20</td>
<td>-</td>
<td>20</td>
<td>-</td>
</tr>
<tr>
<td>5%HDPE</td>
<td>75</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>5</td>
</tr>
<tr>
<td>10%HDPE</td>
<td>70</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>10</td>
</tr>
<tr>
<td>15%HDPE</td>
<td>65</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>15</td>
</tr>
<tr>
<td>20%HDPE</td>
<td>60</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>20</td>
</tr>
</tbody>
</table>

Figure 4. SEM images of Beneficiated Kankara Kaolin: A) 8000X; B) 15000x

Figure 5. SEM images of Beneficiated Kibi Kaolin: A) 8000X; B) 15000x
3.2 Particle size distribution

The ranges of particle size of the samples after particle size analysis are shown in Figure 7. There is a wide range of particle sizes (0nm-8000nm) in the samples which is necessary for close packing configuration (Kingery, 1976; Richerson, 2006).

![Figure 6: XRD pattern of kaolinite (Kankara and Kibi).](image)

![Figure 7: Particle size distribution of Kankara and Kibi Kaolin](image)

3.3 Effects of Sintering Temperature on the Porous Kaolin based Ceramics

3.3.1 Shrinkage

The shrinkage plots for the control and porous samples with different fractions of pore formers (SD, STY, and HDPE) are shown in Figure 8. The plot shows that shrinkage increases with increasing sintering temperature. The onset of shrinkage marks the start of the densification process. The control sample showed reduced shrinkage because of the absence of pores in the sample. Thus, the addition of pore formers creates voids in the green (unfired) compact of kaolin-based ceramic which requires higher sintering temperatures for its removal and shrinkage tendency. Thus, at a particular temperature, samples containing pore formers will be more porous than samples without pores. The control samples and samples with saw dust, styrofoam and HDPE at various composition of pore formers had shrinkage values ranging from 7.50-14.25%, 10.00-16.75%, and 12.50-20.00%; 7.50-12.50%, 9.50-10.00% and 11.75-12.50%; 7.50-13.75%, 10.00-17.00% and 12.50-19.75% for sintering temperatures of 850°C, 1000°C and 1150°C, respectively. These values are all within tolerable limits for kaolin-based porous ceramics.

![Figure 8. Variation in shrinkage values of fired samples (fired at 850°C, 1000°C and 1150°C) due to change in sintering time and percentage of pore formers.](image)

3.3.2 Apparent porosity and water absorption

The porosity and water absorption rate, which are the void contents and the weight of the moisture in the pores as a fraction of the weight of the sintered specimen, is an effective index of the quality of porous ceramics. From Figures 9 and 10, the effects of sintering temperature on the apparent porosity and water absorption of the sintered clay-based ceramic are clearly shown. All the plots show a similar trend, that is an increasing porosity and water absorption trend with pore former content. The control samples showed significant reduction in apparent porosity, this is due to the absence of pore formers. However, the absolute value of porosity and water absorption decreases with an increase in sintering temperature and time. This implies that at higher sintering temperature and/or longer holding time, the
pore mobility increases. This causes reduction in porosity and water absorption. These results are in agreement with Surabhi (2012).

From Figure 9, it is observed that the apparent porosity of the sintered sample decreases with increases in sintering temperature. This is due to the fact that voids exist between particles of the newly formed green (unfired) ceramic, much of these inter-particle voids are eliminated during firing/sintering to produce sintered ceramic. However, it is often the case that this pore elimination process is incomplete and some residual porosity will remain. A lot of factors determine the amount of these pores that will be eliminated during sintering, these include, the temperature at which the ceramic is sintered; the higher the sintering temperature, the higher the amount of the pores that will be filled/eliminated during the sintering operation. Moreover, the composition of the ceramic raw materials from which the ceramic is fabricated together with the sintering temperature equally affects the elimination of the pore during sintering.

Control samples and samples with saw dust, styrofoam and HDPE at various composition of pore formers had apparent porosity values ranging from 30.87-51.97%, 30.22-51.16%, and 28.63-47.00%; 30.87-48.82%, 30.22-44.40% and 28.63-39.19%; 30.87-57.06%, 30.22-54.36% and 28.63-51.66% for sintering temperatures of 850°C, 1000°C and 1150°C respectively. However, samples with saw dust, styrofoam and HDPE as pore formers had water absorption values ranging from 19.87-54.34%, 18.75-52.58%, and 17.07-42.10%; 19.87-61.96%, 18.75-51.56% and 17.07-42.10%; 19.87-69.70%, 18.75-61.54% and 17.07-53.84% for sintering temperatures of 850°C, 1000°C and 1150°C, respectively. These values are all within tolerable limits for kaolin-based porous ceramics.

The results (see Figure 10) show that as the percentage by mass of pore formers increased, the water absorption of the porous ceramic samples increased. A near-linear dependence of porosity and water absorption on the pore former contents (from 5 to 20 wt%) in the samples was observed. Furthermore, the amount of water absorbed by the porous ceramics decreased with an increase in the heating temperature. The decline in the rate of water absorption with increasing heating temperature suggests that local liquid-phase sintering occurred, which contributed to a decrease in pore volume and the water absorption rate. Water absorption values of the samples show that the sintering behaviour of ceramics is affected by the formation of a transitory liquid phase, which improves the densification of the sintered samples. The bonding capacity of the mixture is related to the amount of the pore formers added to the mixture.

Control samples and samples with saw dust, styrofoam and HDPE at various compositions of pore formers had apparent density values ranging from 1.90-2.34 g/cm³, 1.93-2.31 g/cm³ and 1.97-2.34 g/cm³; 1.44-2.24 g/cm³, 1.47-2.31 g/cm³ and 1.49-2.34 g/cm³; 1.85-2.24 g/cm³, 1.89-2.31 g/cm³ and 1.92-2.34 g/cm³ for sintering temperatures of 850°C, 1000°C and 1150°C, respectively. While bulk density values ranged from 0.86-1.55 g/cm³, 0.89-1.61 g/cm³ and 0.98-2.75 g/cm³; 0.66-1.55 g/cm³, 1.47-2.31 g/cm³ and 0.79-2.75 g/cm³; 0.81-1.61 g/cm³ and 0.88-2.75 g/cm³ for sintering temperatures of 850°C, 1000°C and 1150°C, respectively. These values are all within tolerable limits for kaolin-based porous ceramics.

### 3.3.3 Apparent and Bulk density

Figures 11 and 12 show the variation of apparent and bulk densities in the sintered control and porous kaolin-based ceramics as a function of pore former content for different sintering temperatures. All the plots show similar trends, that is a decreasing bulk density trend with pore former content. However, the absolute value of apparent and bulk densities increase with an increase in sintering temperature. This implies that at higher sintering temperature, the pore mobility increases. This causes a reduction in porosity and an increase in bulk density.

Control samples and samples with saw dust, styrofoam and HDPE at various compositions of pore formers had apparent density values ranging from 1.90-2.34 g/cm³, 1.93-2.31 g/cm³ and 1.97-2.34 g/cm³; 1.44-2.24 g/cm³, 1.47-2.31 g/cm³ and 1.49-2.34 g/cm³; 1.85-2.24 g/cm³, 1.89-2.31 g/cm³ and 1.92-2.34 g/cm³ for sintering temperatures of 850°C, 1000°C and 1150°C, respectively. While bulk density values ranged from 0.86-1.55 g/cm³, 0.89-1.61 g/cm³ and 0.98-2.75 g/cm³; 0.66-1.55 g/cm³, 1.47-2.31 g/cm³ and 0.79-2.75 g/cm³; 0.81-1.61 g/cm³ and 0.88-2.75 g/cm³ for sintering temperatures of 850°C, 1000°C and 1150°C, respectively. These values are all within tolerable limits for kaolin-based porous ceramics.
Contrary to the relationship which existed between the sintering temperature and the shrinkage, water absorption and apparent porosity as discussed above, it is observed that the bulk density of the clay-based ceramic samples increased with increases in the sintering temperature. This is expected because as the inter-particle voids/pores are progressively filled up with increasing sintering temperatures, the volume of the ceramic samples can be said to reduce with increased sintering temperature. This behaviour is also due to the reduced porosity of the sample as explained above which leads to an increase in the amount of matter in the sample per unit volume (Aramide, 2012).

It is critical to note that, for this study, a sintering temperature of 850°C and a 5% weight pore former addition by mass can be suggested as the optimum conditions for the specific batches. This is in terms of the porosities and water absorption of the ceramic bodies vis-à-vis their mechanical strength. However, properties of ceramic bodies for the other conditions investigated are tolerable and of huge significance considering potential high temperature applications of these substrates.

### 4. Conclusions

This study examined the physical and mechanical properties of low-cost kaolin-based ceramics in the preparation of porous substrates. Based on the physical and mechanical properties of the sintered samples at different temperatures, it can be concluded that:

i. Samples with high density polyethylene as a new pore former considered in this study resulted in highly porous bodies, confirming that the choice of pore formers is critical to achieving porous ceramic bodies. Formulations containing 80% kaolin can be used for the production of ceramics with porosities as high as 59.84% if the right pore formers are used.

ii. Apparent porosity, and water absorption of the samples decrease with increased sintering temperature.

iii. Bulk and apparent densities and cold crushing strength of the samples increased with increased sintering temperature.
iv. Samples with 5% wt pore former (HDPE), sintered at 850°C gave the optimum properties in terms of the porosity, water absorption and mechanical strength of the samples.

v. The physical and mechanical properties of all samples fired at different sintering temperatures (water absorption, bulk density, apparent density and linear shrinkage) were all within tolerable limits for kaolin-based ceramics.

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