Production and Characterization of Aluminosilicate Refractory Brick Using Unwana Beach Silica Sand, Ekebedi and Unwana Clays

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ABSTRACT

Production and characterization of aluminosilicate refractory brick using Unwana beach silica sand, Ekebedi and Unwana clays has been carried out with a view to determining the suitability of locally available raw materials for refractory bricks production. The sand was used to compose six (with 65%, 60%, 55%, 50%, 45%, and 40% of Unwana beach silica sand) refractory brick body together with Ekebedi and Unwana clays. The properties of the produced bricks investigated after firing shows that samples A - F had total shrinkage ranging from 3.2% to 8.3% with % porosity of 16.58 to 18.66 respectively. The bulk density result revealed that Samples A- F had 1.63g/cm³ to 1.53g/cm³ with compressive strength ranging from 7.90Mpa to 21.49Mpa respectively. The estimated refractoriness result indicated that the samples A-F had values ranging from 1608°C to 1630°C. The percentage shrinkage, percentage porosity and compressive strength increased progressively with decrease in Unwana beach silica sand contents, while bulk density decreased with decrease in Unwana beach silica sand content. The refractoriness of the bricks increased by 40°C or 50°C with decrease in Unwana beach silica sand content and increase in Ekebedi and Unwana clays content. However, all the samples were good refractory bricks; Sample F was the best in terms of refractoriness and compressive strength. But Sample A was the best in terms of lower shrinkage and porosity with higher bulk density and moderate refractoriness of 1608°C. Therefore, Unwana beach sand can be used for (aluminosilicate) refractory brick production.
1. introduction

Refractory bricks fall under the different types of material derived from ceramics technology. This aspect of ceramics technology is the technology that deals with utilization of sand and/or clay or clay based materials by mixing with water, shaping and drying them, made durable by firing for various uses in the world. Many authors have defined refractory, no doubt but one of them is that “Refactories are high temperature and chemical resistant ceramic materials used in the construction (insulation) of kilns, ovens and annealing chambers against heat and chemical attacks”[1]. Also, aluminosilicate refractories are known to be refractories based upon \( \text{Al}_2\text{O}_3 \) (alumina) and \( \text{SiO}_2 \) (silica). The bricks produced from aluminosilicate refractories are silica bricks, siliceous bricks, fire bricks, high alumina bricks and aluminous fire brick. Silica bricks are refractories formed of at least 90% silica cemented with slurred lime used to line furnace roofs.

In high temperature industries, refractories play a vital role in different areas of the industrial machines. Some of the areas that desperately use refractories are the machine tool industry which uses refractory as abrasives, electrical and electronic industries as insulators, nuclear power industries as moderators in nuclear fuel, aerospace industries, ceramic industries, glass industries and iron and steel industries which consume more than 70% refractory for kiln, furnace and others [1].

Refractory is referred to as resisting control or resistant to treatment or heat [2]. The ceramic materials that can withstand high temperature without appreciable deformation under service conditions are called refractory materials [3]. The ceramic materials such as magnesia with melting point of 2800\(^\circ\)C and alumina (with melting point at 2040\(^\circ\)C) are excellent materials for refractory purposes. Such materials are used for firing at high temperature furnace, boilers, crucibles, converts etc. Some common refractory materials are silica, magnesia, dolomite, silicon, carbide, zirconia and graphite [3].

Refractories are non metallic materials that have unusual high melting temperatures and maintain their structural properties at very high temperatures. Principally, they are composed of oxides of silicon and aluminum. They are employed in great quantities in the metallurgical, glassmaking and ceramics industries, where they are formed into variety of shapes to line the interiors of furnaces, kilns and other devices that process materials at very high temperatures [4].

Refractory materials must be chemically and physically stable at high temperature depending on the environment they operate in. They need to be resistant to thermal shock, be chemically inert and/or have specific ranges of thermal conductivity and of low co-efficient of thermal expansion. Furthermore, the oxides of aluminum (alumina), silicon and magnesium are the most important materials used in the manufacture of refractories. Another oxide usually found in the refractories is the oxide of calcium (lime). Fire clay is also widely used in the manufacture of refractories [1]. Any material can be described as a ‘refractory’, if it can withstand the action of abrasive or corrosive solids, liquids or gases at high temperatures. The various combinations of operating conditions, in which refractories are used, make it necessary to manufacture a range of refractory materials with different properties. Refractory materials are made in varying combinations and shapes depending on their applications [5]. General requirements of refractory materials are: Withstand high temperatures, withstand sudden changes of temperatures, withstand action of molten (eg.
metals slag, glass, hot gases, etc.), withstand load at service conditions, withstand load and abrasive forces, have low coefficient of thermal expansion, conserve heat and Should not contaminate the material with which it come in contact [5].

Refractories are the primary materials used by the metallurgical industry in the construction of all, in the internal linings of melting, smelting in vessels for holding and transporting metal and slag, also for heating before further processing and in the flues or stacks through which hot gases are conducted. The metallurgical processes require very high operating temperatures and then the refractory materials become more essential because it can withstand very high temperature without rapid physical and chemical deterioration [6,7].

Refractories can be classified on the basis of chemical composition, end use and methods of manufacture as shown in Table 1.

<table>
<thead>
<tr>
<th>Classification method</th>
<th>Examples</th>
<th>Chemical composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACID, which readily combines with bases</td>
<td>Silica, Semisilica, Alumino-silicate</td>
<td></td>
</tr>
<tr>
<td>BASIC, which consists mainly of metallic oxides that resist the action of bases</td>
<td>Magnesite, Chrom-magnesite, Magnesite-chromite, Dolomite</td>
<td></td>
</tr>
<tr>
<td>NEUTRAL, which does not combine with acids nor bases</td>
<td>Fireclay bricks, Chrome, Pure Alumina</td>
<td></td>
</tr>
<tr>
<td>Special</td>
<td>Carbon, Silicon Carbide, Zirconia</td>
<td></td>
</tr>
<tr>
<td>End use</td>
<td>Blast furnace casting pit</td>
<td></td>
</tr>
<tr>
<td>Method of manufacture</td>
<td>Dry press process, fused cast, hand moulded, formed normal, fired or chemically bonded, unformed (monolithics, plastics, ramming mass, gunning castable, spraying)</td>
<td></td>
</tr>
</tbody>
</table>

2. MATERIALS AND METHODS

2.1 Collection of Raw Materials

The silica sand was obtained from Unwana Beach, Afikpo North of Ebonyi State, Nigeria. The remaining two clays (Unwana and Ekebedi clays) were collected from Unwana community of Afikpo North, Ebonyi State and Ekebede in Ikwuano-Oboro of Abia State respectively.

3. CHEMICAL ANALYSIS

3.1 Determination of Chemical Composition of Unwana Beach Silica Sand, Unwana and Ekebedi Clays

A chemical analysis of Unwana beach silica sand was carried out to determine the % oxide composition of the sample. The oxides include: SiO$_2$, CaO, MgO, Na$_2$O, K$_2$O, Fe$_2$O$_3$, ZnO, MnO, Al$_2$O$_3$, and loss on ignition. The oxides were determined using Buck Scientific model
210 VGP Atomic Absorption Spectrophotometer (AAS) [9]. This process was repeated for Unwana and Ekebedi clays.

### 3.2 Batch Materials Weighing

The raw materials used for the production were weighed using a weighing balance. 45% fine particles size sand, 10% medium particular size sand and 45% coarse particle size sand were mixed. The percentage of the beach silica sand, Ekebedi and Unwana clays were recorded on the Table 2 containing the total batch weighing and the mixing percentages.

#### Table 2. Body composition of the bricks

<table>
<thead>
<tr>
<th>Name of raw materials</th>
<th>Composition (%)</th>
<th>Weight (g)</th>
<th>% of water (ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sample A</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unwana Beach Silica Sand</td>
<td>65</td>
<td>10,920</td>
<td></td>
</tr>
<tr>
<td>Ekebedi Clay (grog )</td>
<td>15</td>
<td>2,520</td>
<td></td>
</tr>
<tr>
<td>Unwana Clay (binder)</td>
<td>20</td>
<td>3,360</td>
<td>2,520</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>16,800</td>
<td></td>
</tr>
<tr>
<td><strong>Sample B</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unwana Beach Silica Sand</td>
<td>60</td>
<td>10,080</td>
<td></td>
</tr>
<tr>
<td>Ekebedi Clay (grog )</td>
<td>17.5</td>
<td>2,940</td>
<td></td>
</tr>
<tr>
<td>Unwana Clay (binder)</td>
<td>22.5</td>
<td>3,780</td>
<td>2,520</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>16,800</td>
<td></td>
</tr>
<tr>
<td><strong>Sample C</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unwana Beach Silica Sand</td>
<td>55</td>
<td>9,240</td>
<td></td>
</tr>
<tr>
<td>Ekebedi Clay (grog )</td>
<td>20</td>
<td>3,360</td>
<td></td>
</tr>
<tr>
<td>Unwana Clay (binder)</td>
<td>25</td>
<td>4,200</td>
<td>2,520</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>16,800</td>
<td></td>
</tr>
<tr>
<td><strong>Sample D</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unwana Beach Silica Sand</td>
<td>50</td>
<td>8,400</td>
<td></td>
</tr>
<tr>
<td>Ekebedi Clay (grog )</td>
<td>22.5</td>
<td>3,780</td>
<td></td>
</tr>
<tr>
<td>Unwana Clay (binder)</td>
<td>27.5</td>
<td>4,620</td>
<td>2,520</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>16,800</td>
<td></td>
</tr>
<tr>
<td><strong>Sample E</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unwana Beach Silica Sand</td>
<td>45</td>
<td>7,560</td>
<td></td>
</tr>
<tr>
<td>Ekebedi Clay (grog )</td>
<td>25</td>
<td>4,200</td>
<td></td>
</tr>
<tr>
<td>Unwana Clay (binder)</td>
<td>30</td>
<td>5,040</td>
<td>2,520</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>16,800</td>
<td></td>
</tr>
<tr>
<td><strong>Sample F</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unwana Beach Silica Sand</td>
<td>40</td>
<td>6,720</td>
<td></td>
</tr>
<tr>
<td>Ekebedi Clay (grog )</td>
<td>27.5</td>
<td>4,620</td>
<td></td>
</tr>
<tr>
<td>Unwana Clay (binder)</td>
<td>32.5</td>
<td>5,460</td>
<td>2,520</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>16,800</td>
<td></td>
</tr>
<tr>
<td><strong>Grand Total</strong></td>
<td></td>
<td>100,800</td>
<td>15,120</td>
</tr>
</tbody>
</table>

### 3.3 Production Process

The sample “A” percentage composition of the batch was weighed and mixed thoroughly before addition of water. Proper mixing of the batch was observed and allowed to aged for 30 minutes. This process was repeated with the other five (5) samples. The moulds were lubricated with heavy oil inside to serve as separator. Properly mixed body with water was
put into the moulds and hydraulic press was used to press the material for strength and uniformity in texture. Each product was released (demould) from the mould after two (2) minutes of proper compression. The bricks were left to dry in open air for two (2) weeks on the floor and loaded into a dryer (oven) at a temperature of ±110°C for a period of one week for proper drying before firing. Properly dried bricks were carefully loaded inside electric kiln and fired to 1650°C.

4. DETERMINATION OF REFRACTORY BRICKS PROPERTIES

4.1 Determination of Linear Shrinkage

Three samples from each batch were given mark (line) with sharp object measured 10cm length at the center point of the top side of the bricks at wet stage. The changes in length of the marks (line) were determined after drying and firing to give linear drying shrinkage, firing shrinkage and total shrinkage of the brick produced.

\[
\% \text{ Drying shrinkage} = \frac{\text{Wet length (cm)} - \text{Dry length (cm)}}{\text{Wet length (cm)}} \times 100
\]

\[
\% \text{ Firing shrinkage} = \frac{\text{Dry length (cm)} - \text{Fired length (cm)}}{\text{Dry length (cm)}} \times 100
\]

\[
\% \text{ Total shrinkage} = \frac{\text{Wet length (cm)} - \text{fired length (cm)}}{\text{Wet length (cm)}} \times 100
\]

4.2 Determination of Bulk Density

The length, breadth and height of the bricks (Samples A - F) were measured and recorded in cm. Chemical balance was used to weigh each sample to the nearest gram. The results obtained were used to first calculate the bricks’ bulk volume and consequently the bulk density and the result is expressed in g/cm³.

4.3 Determination of Porosity

Three samples from each batch were weighed accurately and immersed in boiling water in a large pot and boiled for 4 hours. During the boiling process, the water level was maintained by adding more water if the water level was not covering the bricks again. The bricks were left to cool for 12 hours. They were then removed from the water and their bodies cleaned with a dry towel and were immediately weighed. The differences in weight between the boiled and unboiled bricks were recorded as water porosity. This was expressed as percentage of the original weight.

\[
\% \text{ Porosity} = \frac{\text{Wet weight} - \text{fired weight}}{\text{Fired weight}} \times 100
\]

4.4 Determination of Compressive Strength

Three samples from each batch were crushed using compressive strength tester (Buehler hydraulic press). The load (force) applied before the bricks fractured were recorded. Samples were mounted in turn on a compressive strength tester and load was applied axially at a uniform rate by operating the pump handle in an up and down movement till it
failed. Compressive strength in Mpa was taken as the maximum pressure shown by the
gauge dial which was read off from the machine tester (Buehler hydraulic press).

4.4 Determination of Refractoriness

The refractoriness of the bricks produced were estimated using Shuen’s formula.

\[ K = \frac{360 + Al_2O_3 - RO}{0.228} \]

Where

- \( K \) = Refractoriness (°C)
- \( Al_2O_3 \) = Alumina Content in the clay
- \( RO \) = Sum of all the oxides beside SiO\(_2\) in the clay (or materials)

360 and 0.228 are constants [9].

5. RESULTS AND DISCUSSION

The Unwana beach sand was used to compose six refractory bricks body (with 65%, 60%,
55%, 50%, 45%, and 40%) together with Ekebedi and Unwana clays (Table. 2). The
properties of the produced bricks (Fig.1) investigated after firing showed that samples A – F
had % drying shrinkage, firing shrinkage and total shrinkage (Fig.2) of: A - 3.0%, B – 3.5%,
C – 4.0%, D – 4.1%, E – 4.5% and F – 5.0% (Fig.3), 0.2%, 0.4%, 0.8%, 1.8%, 2.67% and
3.5% (Fig.4) and 3.2%, 3.9%, 4.8%, 5.8%, 7% and 8.3% (Fig.5) respectively with the
following % porosity of 16.58%, 16.74%, 17.11%, 17.39%, 17.69% and 18.66% (Fig.6). This
is mainly attributed to the expansion that accompanied the low quartz to high quartz and the
high quartz to high cristobalite polymorphic transformations that occur on firing at 573°C and
1400°C. The more the brick shrinks, the less porous it is, hence the denser it becomes [9].
The shrinkage values of the produced bricks were found within the acceptable value (2-10%)
recommended [10] and their porosity values also found within the acceptable range of 10-
30% as suggested for refractory bricks [10]. The bulk density result revealed that Sample A-
F had: A - 1.6g/cm\(^3\), B – 1.63g/cm\(^3\), C – 1.62g/cm\(^3\), D – 1.62g/cm\(^3\), E - 1.61g/cm\(^3\) and F -
1.53g/cm\(^3\) (Fig.7) with compressive strength of 7.90Mpa, 9.87Mpa, 10.97Mpa, 15.35Mpa,
18.35Mpa and 21.49Mpa (Fig.8) respectively. The high compressive strength value may be
attributed to the formation of the glassy phase necessary to provide bond strength. The
presence of lime content, sodium and potassium oxides (CaO, Na\(_2\)O and K\(_2\)O) in the
chemical composition (Table. 3) of the materials are sufficient to enhance this process. Also,
the nearer to the sintering point a material is fired, the more pronounced becomes the
sintering at the temperature [9], thereby reducing porosity and increasing density and total
shrinkage. The values of bulk density recorded fell bellow recommended values (1.7 –
2.1g/cm\(^3\)) and (2.3 g/cm\(^3\)) [10,11]. Also, compressive strength of samples were higher than
the recommended minimum of 5Mpa for refractories [10]. The estimated refractoriness
result indicated as follows: A - 1608°C, B – 1612°C, C - 1617°C, D - 1621°C, E - 1625°C and
F - 1630°C (Fig.9). This may be due to appreciation of the alumina content (Al\(_2\)O\(_3\)) and high
melting temperature of silica content (SiO\(_2\)) precent in the body composition (Tables. 2 and
3). All the samples fell within the pyrometric segar cone of 26(1580°C), 27(1610°C) and
28(1630°C) which are still within the recommended ranges of pyrometric cone equivalent
(PCE) 16 – 32 for refractory bricks.
The drying shrinkage, firing shrinkage as well as total shrinkage increased progressively with decrease in silica sand (Fig.2 - 5). Shrinkage, Porosity, Compressive strength and % of Unwana Beach sand are interrelated quantities. The decrease in Unwana Beach sand content increased the shrinkage, made it more porous and increased the compressive strength of the brick (Fig.3 - 5 and 8). This may be due to the increase in the binding clay (Ekebedi and Unwana clays content) (Table. 2). But the bulk density increased with increase in Unwana Beach sand content (Fig.7). This may be as a result of high % of Unwana Beach sand in the body composition (Tables. 2).
The refractoriness of the bricks increase by 4°C or 5°C with decrease in Unwana beach silica sand content and increase in Ekebedi and Unwana clay (Fig.8). This may be due to higher alumina content of Ekebedi and Unwana clays than that of Unwana beach silica sand (Tables 2 and 3). It is a well known fact that the temperature rises as alumina content increases. Samples A – F had the refractoriness of 1608°C, 1612°C, 1617°C, 1621°C, 1625°C and 1630°C respectively. All of these fell within the pyrometric segar cone of 26(1580°C), 27(1610°C) and 28(1630°C). Therefore, all the samples were good refractory bricks. Sample F was the best in terms of refractoriness and compressive strength. But sample A is the best in term of lower shrinkage and porosity with higher bulk density and moderate refractoriness of 1608°C.
Fig. 4. Graph of % firing shrinkage of the refractory bricks

Fig. 5. Graph of % total shrinkage of the refractive bricks
Fig. 6. Graph of % porosity of the refractory bricks

Fig. 7. Graph of bulk density of the refractory bricks
Fig. 8. Graph of compressive strength of the refractory bricks

Fig. 9. Graph of refractoriness of the refractory bricks
Table 3. Result of chemical analysis of the unwana beach silica sand, unwana and ekebedi clays

<table>
<thead>
<tr>
<th>Parameters</th>
<th>% Oxides compositions</th>
<th>Unwana beach silica sand</th>
<th>Unwana clay</th>
<th>Ekebedi clay</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO</td>
<td>0.37</td>
<td>0.14</td>
<td>0.28</td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>0.06</td>
<td>0.04</td>
<td>0.08</td>
<td></td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.20</td>
<td>0.54</td>
<td>0.03</td>
<td></td>
</tr>
<tr>
<td>K₂O</td>
<td>0.08</td>
<td>0.54</td>
<td>0.033</td>
<td></td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>0.02</td>
<td>7.00</td>
<td>1.41</td>
<td></td>
</tr>
<tr>
<td>SiO₂</td>
<td>95.86</td>
<td>52.24</td>
<td>58.53</td>
<td></td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>0.4</td>
<td>27.20</td>
<td>28.03</td>
<td></td>
</tr>
<tr>
<td>ZnO</td>
<td>0.02</td>
<td>-</td>
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<td></td>
</tr>
<tr>
<td>MnO</td>
<td>0.01</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>TiO₂</td>
<td>-</td>
<td>1.52</td>
<td>1.12</td>
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</tr>
<tr>
<td>L.O.I</td>
<td>2.79</td>
<td>10.50</td>
<td>8.0</td>
<td></td>
</tr>
<tr>
<td>Other oxides/impurities</td>
<td>0.19</td>
<td>0.28</td>
<td>2.19</td>
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</tr>
</tbody>
</table>

7. CONCLUSION

In this research work, chemical analysis of Unwana beach sand, Unwana and Ekebedi clays were investigated. These materials were used to produce refractory bricks of samples A – F. The characteristics and necessary properties required of a refractory brick were tested. The percentage shrinkage and porosity of the bricks were found to be within the acceptable value recommended. The bulk density of the products fell below recommended value while their compressive strengths were higher than the recommended value. According to the refractoriness values, all the bricks fell within the recommended ranges of pyrometric cone equivalent (PCE) for refractory bricks. Moreover, with the investigation stated above, sample F was picked as the best with respect to its highest refractoriness and compressive strength which indicates good resistance to abrasion and more resistant to impact. However, sample A can be referred to as the best when making reference to lower shrinkage and porosity with higher bulk density and moderate refractoriness. This is because lower porosity values give better resistance to slag attack and spalling resistance. An increase in bulk density of a given refractory increases its volume stability, heat capacity and resistance to slag penetration. The Unwana beach sand is therefore suitable for manufacturing of (aluminosilicate) refractory brick.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES