Production of Vitrified Porcelain Tiles Using Local Raw Materials from South Western Nigeria

Idowu Oluwatuse Idowu

Glass /Ceramic Department
Federal Polytechnic, Ado – Ekiti, Nigeria.

Abstract
Ceramic tiles, described as a flat piece baked clay used for covering walls are very expensive in Nigeria. About 80% of this product are imported to Nigeria from other countries since the few tiles producing industries in the country do not have the capacity to satisfy the huge demand for the product. As a matter of fact, the highly demanded porcelain tiles are not even produced in Southwestern part of Nigeria. To produced prototype porcelain tiles from this part of the country. Field trips were made to different locations in Southwestern Nigeria to collect samples of Kaolin, (from Ifon), Feldspar, (from Ijero) and Silica (from Igbokoda) A 21 member Triaxial blend test was performed on the selected material. As a result of the test, blend 12 with composition of 20% Kaolin, 60% Silica and 20% was chosen for the study. However the test with the above composition was not successful, thus the need for re-adjustment of the composition and better method of material processing. Re – adjusted composition of 50% feldspar, 17% Kaolin, 19% Ball clay 13% Limestone which was fire to a temperature 1280°C was eventually seen to be successful when compared with both the Nigerian (NIS) and International Standard (ISO).the adoption of this findings by indigenous producers will definitely enhance the local production of porcelain tiles in Nigeria which is currently put at only 20%

Keywords: porcelain, kaolin, ball clay, triaxial blend, feldspar, silica

INTRODUCTION
The government of the Federal Republic of Nigeria is considering diversifying from sole dependence on petroleum products to harnessing the opportunities that abound in solid minerals sub-sector of the economy in order to boost the economy. Ceramic raw materials are among the probable options among the available minerals in Nigeria. The products take a prime position next to food in the materials that are required in large quantity. Exportation of finished ceramic wares made of these solid minerals would go a long way at complementing revenue generation from oil. Of all the ceramic articles, the tiles take the lead among the materials that are demanded in large quantities all over the world. Alan (2005) described tile as a flat piece of baked clay used for covering of walls.

Today, most modern houses are embellished with tiles of various sizes and colours, but about 80% of tiles are imported from abroad since the few tiles producing industries in the country can no longer satisfy the huge demand for the products (Ali, 2004). Alkheia (2010) lamented that Nigeria continue to rely heavily on imported building materials and components. It reported that at least 70% of the fabric and other materials for finishing of a building materials including ceramic tiles increase yearly on the average of 10%-15%. Heavy reliance on imported tiles coupled with the depreciation of the Naira (Nigerian currency)has an immediate impact on both the supply and pricing of these building materials. This is further compounded by the cumbersome clearing processes causing delay and congestion in the Nigerian ports; therefore, users bear the consequence by paying a significant premium on tiles which is 3 to 4 times of the shelf price in other economies. According to Oaikhinan (2014) the total value of ceramics imported into Nigeria is over $600million. The report went further to state Nigeria occupies the 9th position among world’s ceramic consumer and remain the only country among the emerging economics without export of ceramics products.

There are abundant natural resources needed for ceramic tiles production in Nigeria (Ajayi, 2006). Also Kashim (2004), observed that Africa is richly blessed with natural resources which could make her develop her capacity for high-tech and value added manufacturing activities to fully participate in world trade. Mbahi (1999) remarked that it is rewarding to utilize these local materials and human resources to develop the society. Unfortunately, the reverse is the case. Both human and natural resources lay waste in Nigeria while the citizens engage in capital flight activities with wanton abandonments.

Therefore this research work is aimed at producing vitrified porcelain tiles using raw materials from Southwestern Nigeria.
LIMITATION OF THE STUDY
This study was limited to the use of Alumino – Silicate materials from Southwestern Nigeria.

MATERIALS AND METHOD
Preparation of Clay
Kaolin (primary clay) collected from Isan – Ekiti, Ifon and Onibode were levigated. Levigation which is the method of removing impurities from clay by water floating, was carried out in the ceramic workshop of Federal Polytechnic, Ado – Ekiti. According to Cardew (1979) levigation of clay helps in increasing the refractoriness, since with the method, the impurities and free silica are removed. The clays from the above sources were soaked separately for four days before levigation.

After levigation, the clays were dried, pulverized and kept in powder form.

Clay Test
To formulate suitable recipes that will manifest the characteristics of standard porcelain tiles, the pulverized clay samples were tested for shrinkage and porosity.

a. Shrinkage test on clay samples
The collected clay samples were mixed into plastic state and made into slabs of 14 x 14 x 1cm with a line of 10cm long drawn on each slab. The line was measured and the measurements were recorded. (see fig.1)

Later, the slabs were fired to 1300\(^\circ\)c and measured the second time to determine the total shrinkage of the slabs.

b. Porosity test on clay samples
Porosity test was carried out on clay slabs used for shrinkage test. The test was carried out to determine the rate of water absorption. Each of the slabs was weighed and the weight recorded. They were immersed in water to boil for two hours. The slabs were left in the water for 24 hours after which they were brought out and the surface water wiped off and weighed. The % porosity, was calculated thus:

\[
\text{Porosity} = \frac{\text{wet weight} - \text{dry weight}}{\text{dry weight}} \times 100
\]

Based on the result shown in table (2), Ifon Kaolin readily become the most dependable of the three Kaolin samples that were used for the test since it exhibited less shrinkage and less porosity than the others.

FELDSPAR SAMPLES
Feldspars are used as fluxes in ceramic bodies and glazes. They are some of the very few sources of water – insoluble fluxes in the ceramic industry. When the body containing them was fired, the feldspar melts and forms molten glass, which causes the particles of the clay to cling together, when this glass solidifies, it gives strength and hardness (which are distinct qualities of porcelain) to the body. Norton (1956) recommends potash feldspar for glaze formulation.

The Ijero – Ekiti feldspar which is potash in nature (Garkida, 2003) is used for this study. Ijero feldspar was mined and pulverized, though with grits. Hence, the grits were sifted out. The resulting product was washed, dried, milled, sieved and stored for use.

SILICA SAMPLE
The quartz sand obtained, gotten from Igbokoda in Ilaje – Ese – Odo Local Government Area of Ondo State, was thoroughly washed, wet – shifted to remove the more fine particles, which usually contain other mineral impurities, different from silica. The product was dried, milled, sifted and stored for use.

According to singer and singer (1963) finely ground silica accounts for about 20 - 50% of normal ceramic bodies, therefore, any form of silica (for example, ground calcined flint, or ground quartz sand, sandstone etc) may be used in the composition of a ceramic body. Hence, the silica sand, sourced from Igbokoda, was used in this study.

BODY COMPOSITION
After preliminary investigation were conducted on different material samples, Ifon Kaolin, Ijero feldspar and Igbokoda silica sand were adopted for further experiments.

Triaxial body blends
A 21 member traixial blend was performed on the selected material (i.e silica, feldspar and Kaolin).

Gleen (1971) suggests that the method to arrived at a suitable porcelain material body composition was to start the experiment with material singularly for instance, feldspar independently first. This should be followed with decrease in ratio of material in certain proportion using test blend method until the desired result was obtained and established. In line with above statement, triaxial blend was composed.

The ratio in grams as indicated on the fig.(2) was used to compose the blends.

For convenience and easy identification, each of the boxes in fig. (2) was numbered as shown in fig. (3) Numbers were inscribed on each tablet produced from the blends. By these, one can easily identify a particular blend by the number.

The tablets were dried and fired to 1250\(^\circ\)c. The product after firing were then subjected to physical
examination to detect brittleness, density/porosity and hardness. The result of the porosity test are produced in the table 2.

In trying to determine the useful blends, apart from the porosity test, sharp metal objects as nails were used to scratch the fired tablets to determine hardness and brittleness.

After scrutinizing the blends, blend 12 was selected for further experiments because of its hardness, porosity and level of vitrification.

In order to produce vitrified porcelain tiles with the above selected materials, there was the need to construct frame mould.

CONSTRUCTION OF MOULD
Pressing method of producing tiles was considered more appropriate than casting and extrusion methods. All components made by the above methods apart from pressing have low strength (Jayatilaka, 1979).

The mould for pressing (Ramming) was designed and constructed, however, there are some necessary factors to be considered while constructing mould for production of tiles, some of the factors include:

a. The need for a very strong iron that could bear the ramming pressure.
b. The need for a very tight fitting of the top mould for it to be able to hold the body-mixture intact during the process of pressing.
c. The need for a very effective and equal distribution of pressure on the material during ramming.
d. The need for an effective and equal method of removing the produced tiles from the mould after forming in order to retain the formed shape.
e. The need for a strong welding of the parts that makes up the mould in order to stand the pressure exerted by ramming.

MOULD FOR THE PRODUCTION OF TILES
The component parts for frame mould for the production of tiles are listed as follows:

a. The mould’s top: The mould’s top was made of very thick iron – plate with a dimension of 14.5cm x 14.5cm x 5mm. A 4cm x 4cm plate was attached to the mould’s top.
b. The mould’s frame: The mould’s frame formed the rectangular shape of the tiles. It was constructed with angle iron with 5mm thickness. It has an inner dimension of 14.5cm x 14.5cm.
c. The mould’s base: The mould’s base was made from a very strong 5mm thick iron – plate with a dimension of 14.5cm x 14.5cm x 5mm.
d. The removal boards: Several removal boards made of wooden material were constructed. The newly produced tiles were released on each. They have a dimension of 20cm x 20cm x 9mm.
e. Leveling wood: 10 x 3 x 0.8cm wood used for leveling materials in the mould.

PRODUCTION METHODOLOGY
As a result of the test conducted on the blends. Blend 12 with composition of 20% kaolin, 60% silica and 20% feldspar was chosen for the study.

Measurement of the materials which was done at a dry state to ensure thorough mixing. Edem (2004) stressed how important it is for materials needed for the production of tiles to be properly prepared in order to facilitate proper fusion after thorough mixture. Starch was added to the selected blend, mixed with 10% water (in volume) to serve as temporary binder as recommended by Basty(1989). It pointed out some organic addictives for dry pressing, stating further that temporary binders which includes plant rubber, starch, dextrin and others could be added for green strength. Ramming with hammar was adopted as the method of production.

First Experiment
The mould’s frame which was attached to the mould’s base was placed on a very strong wood. Nylon was spread inside the mould frame to act as separator. The material was fed into the mould to the level of 1cm. The leveling wood was used to level up the materials in the mould with a little bit of pressing.

Hammar was gently used to ram the material inside the mould, after which another Nylon was spread on the gently rammed material.

The mould’s top was placed on top of the Nylon inside the frame. Hammar was then rammed to pressed the material until the rammed material could not go down further.

Ejection of the Materials
To remove the rammed tile from the mould, the following process were carried out.

(a) The mould’s top was first removed
(b) The Nylon on top of the tile was removed.
(c) The removal board was placed on top of the frame.
(d) The arrangement was then turn upside down.
(e) The mould’s base was opened towards the left side.
(f) The tile automatically dropped on the removal board.

Result and Observation
a. It was discovered that the percentage of water used for mixing the starch was too
much as the pressed tile was too soft and got distorted.

b. It was also noticed that the pressed tile cracked after two days as a result of the high water content in the material.

Second Experiment
Another attempt to produce good tile was made. Unlike before, the percentage of water was reduced from 10% to 5% (in volume) the material after the water mixture still looked dried.

The earlier process of production was repeated.

Result and Observation
After the tile was removed. It was discovered that the percentage of water added was normal as the pressed tile was considered to be of good quality. Thus 5% of water was used to produce tiles from the blend that was selected earlier.

Drying
Drying of the produced tiles was done indoor. The tiles were left on the removal boards for about two weeks to ensure that the tiles are properly dried.

Firing
The dried tiles were taking to West African Ceramic Industry, Ajaokuta to be fired to 1300°C, the expected temperature of a typical porcelain product.

The result of fired tiles did not meet the standard required of a typical porcelain tile based on both National and International standards. For example the shrinkage test conducted revealed that of 6.6% as against the National and International standard of 0.5% expected. Not that alone, the water of absorption average of 20% was recorded instead of the standard 0.5%. And most importantly, the tile produced did not fuse as expected.

Mcmeekin (1973) states, “porcelain is extremely hard, and non – porous, fired between 1250°C and very high temperature in the 1400°C area and beyond for some industrial porcelain”. In this case, they must be completely fused and vitified, air free and impervious to the entry of gases and liquid of any kind”

The researcher had to think of alternative that will solve the earlier stated problem. The best alternative was to change the medium of pressing out the tiles, instead of using the fabricated manual mould, Hydraulic laboratory press was used at West African Ceramic Limited, Ajaokuta. The press was built with quality material and accurately furnished, valid instrument for producing laboratory tile samples. The mould and punch are fitted in such a way that the tile thickness, dimension can evenly be obtained.

The press has maximum continuous operating pressures of 250 bars, a control board with general switch having two hand control lever for pressing and it also has a push button for activating the extractor/expeller. The press machine can be used with overall dimension of pressed tile at 100 – 100 mm.

Need to process the Material in better form
There was need to process the materials using the Standard Equipment in West African Ceramic Ltd., for optimum result. And in addition ball clay was added to the tested compositions as well as Sodium Silicate to replace starch earlier used.

Process of Preparation
The materials were weighed on the electronic balance and then charged in a pot mill containing Alumina Grinding Media (AGM). The pot mill is allowed to run for twenty minutes with the addition of 35% water of the total charge.

After milling, the slurry or slip is discharged into a dry pan and allowed in the pan for drying. Afterward, the dried material was ground into fine granules using mesh #60 to sieve the granules. An addition of 6% water of the total weight of the granules was added to humidify it for easy pressing (body compaction) into tile slab. The pressed tile slabs were made to go through the dry process in the dryer to expel the moisture content. The size measurement of the tile slab was obtained using Vienier Caliper before the tile slab was sent to the kiln (furnace) for firing using a special refractory metal plate.

After the firing which was done in a continuous kiln, the weight of the tile slab was obtained again. The modulus of rupture (M.O.R) was determined; the size measurement was obtained to determine the shrinkage percentage between the dried and fired tiles slab.

Formular for calculating the modulus of rupture is:

$$\frac{3 \times P \times L}{2 \times bx + 2}$$

P = Pressure of force at which the tile slab is broken
L = Length between the two support on which the tile slab rest while breaking it.

b = The size measurement of the tile slab
+2 = The thickness of the tile slab.

When the modules of Rupture of the tile slab had been determined, using the above formular, the water of absorbency rate was determined with the aid of water absorption tester machine. This machine is operated in two stages, firstly, the tile slabs were arranged inside the machine pressurized vacuum chamber with a transparent lid to make it air tight. It
was allowed to run for 15 minutes, after which water was introduced into the pressurized vacuum chamber and allowed to run for another 15 minutes.

At the expiration of the second 15 minutes, the slabs were removed and the water allowed to drip. The tile slabs were then mopped with clean towel to drain the water and their weight obtained to determine the water absorbency rate of the tile slab using this formula:

\[ W_{1} = \frac{W_{0} - W_{2}}{W_{0}} \times 100 \]

The composition of the six samples fired and tested were stated in tables: 4 and The result is shown in table 5.

The overall results suggest a porcelain tiles but needed to be further adjusted to meet at least the National Standard and the water of absorption in the samples were too high compare with the National Standard.

In view of the above, feldspar content in the composition was increased as recommended by Mcmeekin (1973) stating that, “for porcelain to be made, the materials should be of high quality refractoriness”, this, according to the researcher, “will give good fusion and vitrification of the desired products. The body composition in the formulation should be held thus: Limestone and most importantly Feldspar be relatively high”. In line with the above, the percentage of feldspar was increased drastically and limestone from Ewekoro was used in place of silica. The Re-adjusted composition is given in table 4.

DISCUSSION

Modulus of Rupture test was carried out on the adjusted sample (table 6), Ahmed (1986) described M.O.R. as the fracture strength of the material under bending load. In other words, the test was aimed at determining the strength of the glazed tiles after firing. At the end of the test, M.O.R. of 48.35 kg/cm² was recorded on the adjusted samples. This surpasses both the International Standard (I.S.O.) and Nigerian Standard (NIS) of 35 kg/cm² and 44 kg/cm² respectively. The result can be said to be successful since it indicated that both the International standard and Nigerian standards were surpassed.

Water of absorbency rate of 0.43% was also recorded on the tested sample (Table 6) Although, this result did not meet the International Standard (I.S.O.) of 0.5%, but it surpasses the Nigerian Standard which was put at 1% maximum. A shrinkage test of 6.63% was recorded as against 0.6% and 0.1% of both the International and Nigerian Standards respectively (Table 6). The high shrinkage rate may be due to the addition of Feldspar. But the problem of shrinkage can be tackled by increasing the size of the mould for tile production as recommended by Chris (2000). Apart from the shrinkage, the result indicated that the materials are of good quality for fusion, hardness, tensile strength, flux, and refractoriness.

CONCLUSION

A closer study of the prototype tiles produced at the end of the research revealed that acceptable vitrified porcelain tiles could be achieved with raw material from Southwestern Nigeria. Although the manually constructed mould was not good enough for the production of porcelain tile, it could however, be useful for earthenware production which does not require a high level of fusion expected of a porcelain product. The produced tiles surpasses both the International and Nigeria Standard in terms of modules of Rapture test, it also surpasses the Nigerian Standard in terms of water of absorbency test. But it couldn’t meet both the International and National Standard in terms of shrinkage. Shrinkage, however, is not a very serious issue since it could be corrected by adjusting the size of the mould.

With the application of the knowledge derived from this study, the culture of mass importation of porcelain tiles into Nigeria might be reduced and this will give way for local production of this facing product thereby boosting Nigerian economy.

CONTRIBUTIONS TO THE BODY OF KNOWLEDGE

This research is expected to contribute to the body of existing knowledge in the following ways.

1. It will generate synergies of collaboration between the academic community, local industries producing ceramic tiles and other stakeholders in order to fashion out how to improve the local product and make them acceptable to consumers.
2. Contribute to the evolution of suitable body composition for the production of vitrified in Southwestern Nigeria.
3. Provide a referral base for further studies on the production of ceramic tiles in order to push further the frontiers of knowledge.

REFERENCES


**APPENDIX**

![Fig. Slab for Shrinkage and water of absorption tests.](image)

**Table 1: shrinkage tests on clay samples**

<table>
<thead>
<tr>
<th>Kaolin (primary clay)</th>
<th>Temp. of firing</th>
<th>Measurement Net</th>
<th>dry</th>
<th>fired</th>
</tr>
</thead>
<tbody>
<tr>
<td>Onibode</td>
<td>1300°C</td>
<td>10cm 9.6cm 9.2cm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Issan</td>
<td>1300°C</td>
<td>10cm 9.6cm 9.3cm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ifon</td>
<td>1300°C</td>
<td>10cm 9.7cm 9.4cm</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Source: Compiled by the Researcher

**Table 2: Porosity tests on clay samples**

<table>
<thead>
<tr>
<th>Kaolin (primary lay)</th>
<th>porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Onibode</td>
<td>16</td>
</tr>
<tr>
<td>Issan</td>
<td>15.5</td>
</tr>
<tr>
<td>Ifon</td>
<td>15.3</td>
</tr>
</tbody>
</table>

Source: Compiled by the Researcher
Kaolin

Silica                        feldspar

Fig: 2 Triaxial blend of three selected material

Table 3: The result of porosity test on blend.
Identification Number               Percentage Porosity at 1250⁰C

1  18:65
2  16:78
3  15:13
4  16:13
5  14:55
6  16:83
7  15:03
8  14:77
9  15:68
10 16:66
11 15:31
12 11:74
13 14:12
14 12:12
15 16:33
16 14:03
17 16:48
18 20:92
19 13:37
20 20:92
21 22:87

Source: Result of laboratory test by Researcher

Table 4: The summary of % material composition of the tested samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Feldspar</th>
<th>Silica</th>
<th>Kaolin</th>
<th>Ball clay</th>
<th>Limestone</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>36</td>
<td>24</td>
<td>24</td>
<td>16</td>
<td>2</td>
</tr>
<tr>
<td>B</td>
<td>40</td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>15</td>
</tr>
<tr>
<td>C</td>
<td>40</td>
<td>25</td>
<td>25</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>D</td>
<td>40</td>
<td>20</td>
<td>25</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>E</td>
<td>35</td>
<td>25</td>
<td>25</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>F</td>
<td>30</td>
<td>25</td>
<td>25</td>
<td>20</td>
<td>19</td>
</tr>
<tr>
<td>Re-adjusted composition</td>
<td>51</td>
<td>-</td>
<td>17</td>
<td>19</td>
<td>13</td>
</tr>
</tbody>
</table>

Source: Compiled by the researcher

Table 5: Results of the Tests conducted on clay samples

<table>
<thead>
<tr>
<th>Type of test</th>
<th>Sample A</th>
<th>Sample B</th>
<th>Sample C</th>
<th>Sample D</th>
<th>Sample E</th>
<th>Sample F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulus of Rupture</td>
<td>42.81 kg/cm²</td>
<td>52.68 kg/cm²</td>
<td>53.74 kg/cm²</td>
<td>42.16 kg/cm²</td>
<td>46.19 kg/cm²</td>
<td>45.25 kg/cm²</td>
</tr>
<tr>
<td>Water absorbency</td>
<td>7.57%</td>
<td>8.76%</td>
<td>9.58%</td>
<td>7.33%</td>
<td>9.87%</td>
<td>7.77%</td>
</tr>
<tr>
<td>Shrinkage</td>
<td>3.16%</td>
<td>4.28%</td>
<td>4.85%</td>
<td>4.39%</td>
<td>3.89%</td>
<td>3.11%</td>
</tr>
</tbody>
</table>

Source: Compiled by the researcher

Table 6: Result of the Test conducted on Re-adjusted Body composition VIS – A –VIS the National and International Standard

<table>
<thead>
<tr>
<th></th>
<th>Re-adjusted Composition</th>
<th>International standard (ISO)</th>
<th>Nigerian standard (NIS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulus of Rupture</td>
<td>48.35 kg/cm²</td>
<td>35 kg/cm²</td>
<td>44 kg/cm²</td>
</tr>
<tr>
<td>Water absorbency</td>
<td>0.43%</td>
<td>0.5%</td>
<td>1% maximum</td>
</tr>
<tr>
<td>Shrinkage</td>
<td>6.63%</td>
<td>0.6%</td>
<td>0.1%</td>
</tr>
</tbody>
</table>

Source: Compiled by the researcher