# Purification of Meetiyagoda Kaolin for boron free glaze manufacturing

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## Introduction

A tile is a manufactured piece of hard-wearing material such as ceramic, stone, metal, or even glass for covering of roofs, floors, walls, or other objects. Tiles are often made from ceramic with a hard glaze finish. But sometimes with other materials such as glass, marble, granite and slate are also used to produce tiles. Among them, floor tiles are commonly made of ceramic, porcelain and stones due to their attractiveness, durability, and easiness to clean. The main components of a tile are tile body, glaze layer, and printed layer. The raw materials used to form a tile consist of clay minerals, quartz, feldspar which is used to lower the firing temperature and chemical additives required for the shaping process. Tile decoration is mainly depending on the glaze layer. Sodium feldspar, potassium feldspar, quartz, wollastanite and kaolinite are abundantly used materials in glaze production. Even through Sri Lanka contains most of the above raw materials; at present all required glaze materials as imported to the country due to the cost, lack of technology and some quality problems of raw materials. So this research aims to manufacture a low cost glaze medium mainly using local mineral materials and few imported materials.

## Methodology

The current study was carried out at Uva Wellassa University, Badulla, Sri Lanka and Lanka Tiles PLC, Jaltara, Sri Lanka. First, the raw materials were purified to make frit and glaze. The acid leach process was carried out for purification of kaolin and the magnetic process was carried out feldspar, wollastanite and quartz. The clay sample selected for investigation was kaolinite clay obtained from Meetiyagoda area. Then the clay sample was ground using a laboratory ball mill to –149 μm (100 meshes). The ground clay sample was placed on the sieve, and then mechanically shacken for 5 min. The oversize was further grounded followed by sieving with the same sieve. The procedures were repeated till the entire clay sample passed through the sieve. After that ground clay sample passing 100 mesh was subjected to calcination. The sample was heated at 600℃ for 1 h (using a muffle furnace with a maximum temperature of 1200 $^{o}$C ) to activate the clay before acid treatment (Al-Zaharani and Abdulmajid, 2009). Calcineated clay sample passing 100 mesh were leached using 3M hydrochloric acid for different periods of time (10-150 min) and at different leaching temperatures (25$^{o}$C to boiling temperature) using a constant temperature shaking water bath at a fixed shaking rate of 160 cycles/min and using boiling under reflux (Hulbert and Huff, 1970). At the end of leaching, the resulted slurry was filtered to separate undissolved materials and, washed in distilled water. The filtrate and washings were continued

until ions were removed in the sample. The resulting sample was dried for 24 h in Laboratory oven. Likewise the feldspar and wollastanite were purified by magnets. The purity and quality of leached kaolinite were tested using common base (CS 100) in tile industry. After that the frit was made using 80g of sodium tetraborate pentahydrate ($Na_2B_4O_7.10H_2O$), 100g quartz, 160g of kaolin, 40g of wollastonite, 20g of zirconium silicate and 5g sodium chloride. The raw material was mixed in a pot mill. Water was added slowly step by step until the mixture forms in to a crumb. The crumb was heated at 100 $C^0$until it was completely dry. Then the dried product was transferred to porcelain cups and it was placed in a laboratory muffle kiln. The kiln was heated at a rate of 4$^0$C per minute up to a 1050 $C^0$ and kept at same temperature for 90 minutes (Simon et al, 2007). The furnace was switched off and the crucible was allowed to cool down to ambient temperature in the furnace. The product was removed, wrapped in a plastic film hammered to break in to small pieces. Then the base formula was generated by using it. Glass formation and formulation mechanisms (fluxes, vitrifying agent, opecifier), thermal expansion of the materials and melting point (softening temperature) were considered for base making. The materials in the base thermal expansion were adjusted according to frit thermal expansion. Frit (30g), kaolin (8g), sodium feldspar (30g), wollastanite (32g), zirconium silicate (10g), aluminium oxide (2.5g), zinc oxide(1.5g), C.M.C (0.2g) and S.T.P.P (0.2g) were added to the base formula and it was grind with 100 ml water in pot mill for about 20 minutes. Resulted base medium was sieved using a 100 µm sieve and it was sprayed by spray gun on to engobe green tile. Base density and base viscosity was measured before spraying. Based tile was fired in the kiln. Finally fired tile surface properties, thermal shock, abrasion, strain, cracking and acid resistance were checked to ensure the quality.

## Results and Discussion

Leach kaolin base is whiter than the reference base which is used to compare. But there were pin holes on glaze surface which may have caused by organic materials in kaolin. Pin holes are considered as a defect in the tile body. The challenge of reducing pin holes can be achieved by changing firing zone, but it cannot be adjust for one tile. Therefore, pin holes were reduced by making rougher the tile surface by adding alumina in to the base. After adding alumina, the result shows that it can use for glaze making.

Prepared frit is opaque and I has matt surface when it was fired with the 90% of frit and 10% kaolin. This frit generates a slightly rough surface compared to the other commercially available verities as this cooled under slow cooling environment and there, it produces a crystalline surface instead of a vitreous surface. Even though fast cooling is essential in making frit, it is impossible to achieve that with a laboratory muffle kiln. Therefore this frit was used in production of base by adjusting other raw materials.

Prepared base is white in colour and has a glossy surface and also fulfil the all quality tests.

- **Tile code – Sample**
- **Ceramic Body – White**
- **Tile Size – 300 X 300 mm**

Table 1: Technical characteristics of new tile

| | CHARACTERISTICS | RESULT |
|---|---|---|
| 1 | STAIN RESISTANCE | PASS |

| | | |
|---|---|---|
| 2 | ACID RESISTANCE | PASS |
| 3 | ALKALI RESISTANCE | PASS |
| 4 | PEI CLASS | 3 |
| 5 | SLIP RESISTANCE (PENDULUM TEST) | WET DRY |
| | | FAIL PASS |
| 6 | THERMAL SHOCK RESISTANCE | PASS |
| 7 | CRAZING ABILITY | PASS |
| 8 | CLEANING ABILITY | VERY EASY EASY ~~DIFFICULT TO CLEAN~~ |
| 9 | CUTTING ABILITY | YES |

Table 2: Dimensional characteristics of new tile

| Product tile Warpage (mm) | | | | | | Centre curvature (mm) | | |
|---|---|---|---|---|---|---|---|---|
| Tile no | 1 | 2 | 3 | 4 | Range | 1 | 2 | Range |
| 01 | -0.01 | 0.22 | -0.11 | 0.30 | 0.44 | -0.11 | -0.04 | -0.03 |
| 02 | 0.12 | 0.27 | 0.25 | 0.04 | 0.21 | -0.05 | -0.14 | -0.09 |
| 03 | 0.05 | 0.28 | -0.15 | 0.11 | 0.43 | 0.29 | 0.36 | 0.07 |

## Conclusion

Main objective of this research is to prepare boron free glaze from purification of Meetiyagoda kaolin and other local minerals. Purification was successfully achieved by hydrochloric acid leaching of kaolin and removal of iron using a magnet in feldspar, quartz and wollastanite. Using standard methods these materials were formed in to a suitable base material to be used in glaze, which is satisfy all quality test.

Pin holes were appeared due to presence of organic materials, by adding aluminium oxide in to the base those can be reduced.

Cost of leaching Meetiyagoda kaolin is high; therefore overall cost of the base was also high. Because of that the imported kaolin (*BO kaolin*) was used in the process and the resulted also shows the same quality.

## References

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