

Influence of firing temperature on physical properties of Same clay and Pugu kaolin for ceramic tiles application

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Abstract: This study focuses on the effect of firing temperature on the physical properties of Same clay and Pugu kaolin for potential application in ceramic tiles. X-Ray diffractometer (XRD) is used to analyze the phases formed after firing. XRD result shows that quartz, sillimanite and cristobalite appears at temperature beyond 1200°C while mullite phase develops in Pugu kaolin. Pugu kaolin shows low porosity (1.3%) at 1400°C compared to that of Same clay (12.7%). Water absorption at 1200°C is 7.8 % for Pugu kaolin and sharply reduced to 0.47% at a temperature between 1300°C to 1400°C suggesting complete sintering to have taken place at this temperature.

Keywords: Clay, Ceramic Tile, Phase Development, Linear Shrinkage, Water Absorption

1. Introduction

Clay is the main raw material in the traditional ceramic industry and an important ingredient in advanced ceramic products. For example, clay is used as a raw material in many industrial fields such as ceramics, paper and paint. Their applications are closely dependent on their structure, composition, and physical properties [1-4].

Ceramic tiles are composed essentially of clays and other inorganic raw materials such as quartz, feldspar, and carbonates, the clays being the most important raw materials. In fabrication of ceramic products, raw materials selection plays vital role in the final [1]. The physico-chemical properties of the resulting ceramics is influenced by the properties of the components present in the clay and the temperature they have been subjected to [5]. During firing, a series of transformation occurs which determines their final ceramic properties [6, 7]. Sintering of ceramic mass is fundamental in adjusting desired properties and occurs in general during liquid-phase formation. Thus the presence of components such as low-melting clays is very important. Fluxes (alkaline oxides, mainly K₂O and Na₂O), in reaction with silica and alumina, promote liquid phase formations that facilitate densification [8].

For the purpose of determining the quality of the clays

used in the formulation of ceramic tiles, it is important to monitor and understand the phase transformations which take place during the firing process [9].

Tanzania is importing industrial scale ceramic tiles and most of other related ceramic products from outside the country, as they are not adequately produced locally. There are, however, abundant ceramic raw materials locally available that can be exploited and utilized for such ceramic application. Clay deposits have been reported in places like Singino-Lindi, Pugu-Dar Es Salaam, Malangali-Iringa, and Matamba-Mbeya, Same-Kilimamjaro, Arusha and Mwanza have been reported [10-12]. Basically the studies have been on geological occurrence and suggestions on their possible use. Pugu deposit found to be with white kaolinitic sandstone [10]. Neither deposit has been studied for the use in the ceramic tile industry. Thus, the knowledge of these clays has both academic value and immediate commercial interest in the ceramic tile industry.

This work is therefore to study the influence of firing temperature on physical properties of Same clay and Pugu kaolin for ceramic tile application. It focuses on the phase changes and microstructural development in relation to the physical properties due to change in firing temperature.

2. Experimental Procedure

2.1. Material

Clay samples are collected from Same, Kilimanjaro region in the northern zone of Tanzania and Pugu kaolin hills approximately 25 km south-south-west of Dar es Salaam and 20 km inland from the Indian Ocean.

2.2. Specimen Preparation

Clay samples are planetary ball milled to form powder. The powder is pressed to form pellets which are dried at 105°C until constant weight was achieved. The samples are then heated to 600, 800, 1000, 1200, 1300 and 1400°C, respectively at a rate of 5°C/min with soaking time of 1h at each maximum temperature.

2.3. Specimen Characterization

The XRD patterns are obtained with Rigaku, D.Max 2200 diffractometer operating at tube voltage and current 40kV and 40mA, respectively using monochromatic Cu-K α radiation. Diffraction patterns were recorded by scanning from 10° to 80° (2 θ) at a rate of 2°/min. The morphology was observed by

scanning electron microscopy (SEM), in JEOL JSM-6380.

The chemical composition of the samples is determined by inductively coupled plasma optical emission spectroscopy (ICP-OES) Optima 5300 DV. Linear shrinkage is calculated by measuring dimensions of the prepared specimen before and after firing as per BS EN ISO 1045-3:1997. Water absorption and apparent porosity are measured using Auto Pore IV 9500.

3. Results and Discussion

3.1. Chemical Composition, Microstructures and Phase Analysis

Table 1 shows the chemical compositions in terms of oxide contents, as well the loss on ignition of Pugu kaolin and Same clay. Both clays contain mainly SiO₂ and Al₂O₃, which corresponds to 85.6% for Same clay and 83.6% for Pugu kaolin. Same clay shows the higher amount of Fe₂O₃ (2.62%) compared to Pugu kaolin which contains 2.22% of Fe₂O₃ content. Iron oxide gives reddish color to the product after sintering and the reddish color becomes stronger as amount of Fe₂O₃ increases [13, 14].

Table 1. Chemical composition of raw materials (wt. %)

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O	LOI*	Total
Pugu Kaolin	48.9	34.7	2.22	0.6	0.01	0.11	0.07	0.34	13.05	100
Same Clay	62.1	23.5	2.62	1.55	0.02	0.09	0.02	0.08	10.03	100

*LOI=loss on ignition

The loss on ignition is observed to be relatively high for Pugu kaolin (13.05%) compared to (10.03%) that of clay from Same. The high loss on ignition (13.05%) associated with low SiO₂ and high Al₂O₃ content is due to the significant content of alumina in Pugu kaolin. The content of alkaline oxides (K₂O and Na₂O) is relatively low for Same clay indicating low percentage of flux minerals and that will result in increase in sintering temperatures [2, 15]. The low earth-alkaline oxides (CaO and MgO) content for Pugu kaolin and Same clay indicates that the studied clays are poor in carbonates [15].

The X-ray diffraction patterns of raw clay and after firing are presented in Figure 1. The result shows phase

compositions of both raw clays are kaolinite but during firing phase changes are observed. Same clay forms quartz, cristobalite and sillimanite above 1200°C. In Pugu kaolin, quartz starts to be formed above 1200°C. Quartz and mullite phase dominates at a temperature above 1400°C.

Past studies [3, 16, 17], shows that formation of mullite facilitated the densification. The present study gives results which agree with the phenomena presented, as Pugu kaolin densified much than Same clay (Table 2).

The examination of microstructure by Scanning Electron Microscope (SEM) presented in Figure 3 evidences the densification improvement when sintering is done.

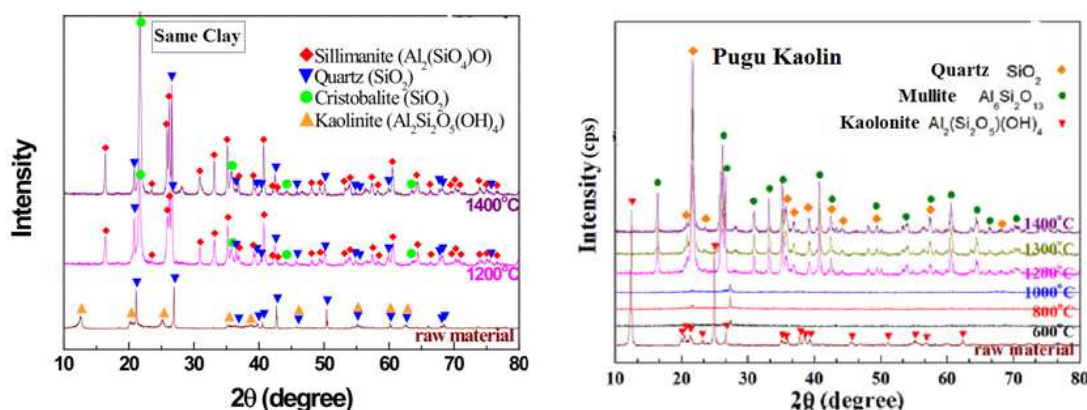


Figure 1. X-ray diffraction pattern of the clay sintered at various temperatures.

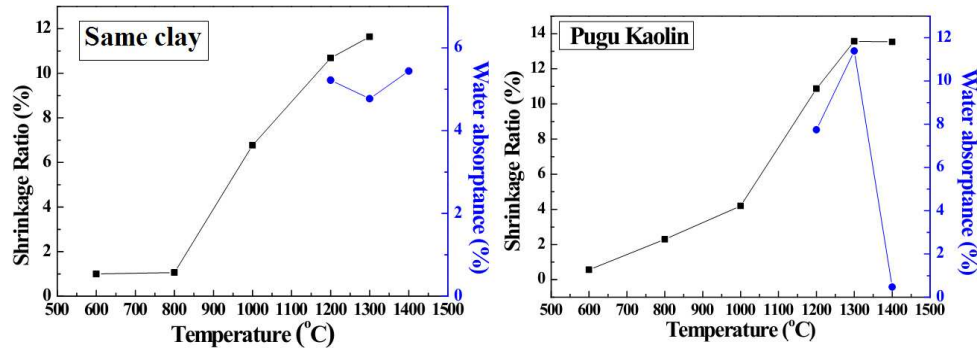


Figure 2. Water absorption and linear shrinkage of clay samples depending on sintering temperature

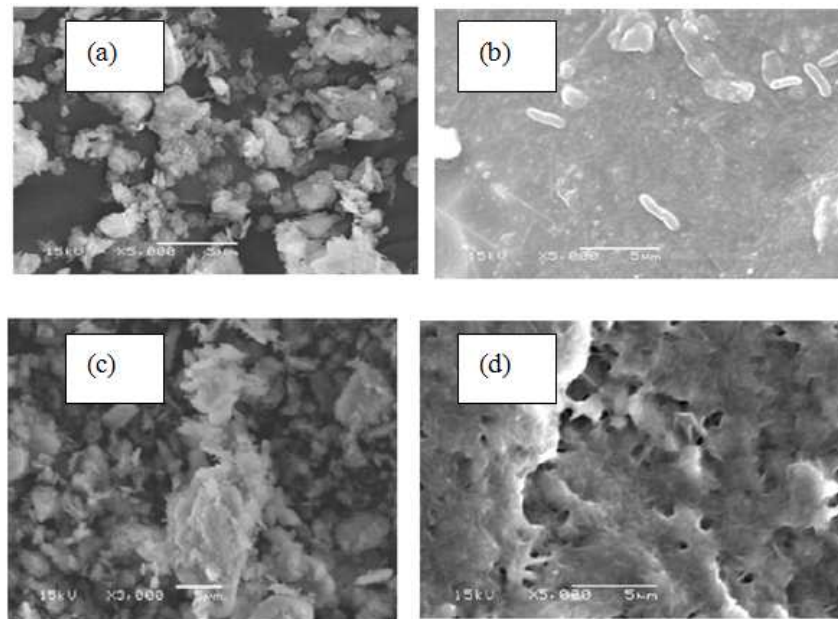


Figure 3. SEM micrographs of raw (a) Same clay, (c) Pugu kaolin and sintered at 1400°C (b) Same clay, (d) Pugu kaolin respectively

Table 2. Bulk properties of Pugu kaolin and Same clay depending on firing temperature

Heat-treatment temperature (°C)	Pugu kaolin			Same clay		
	Apparent density (g/cm ³)	Bulk density (g/cm ³)	Apparent porosity (%)	Apparent density (g/cm ³)	Bulk density (g/cm ³)	Apparent porosity (%)
1200	2.742	2.261	17.5	2.673	2.345	12.3
1300	2.725	2.079	23.7	2.724	2.41	11.5
1400	2.676	2.642	1.3	2.679	2.338	12.7

3.2. Evaluation of Physical Properties

Densification behavior, in terms of linear shrinkage and water absorption as a function of firing temperature is well presented in Figure 2. Results shows linear shrinkage increases as the firing temperature is increased. Water absorption values at 1200°C are 5.2% for Same clay and 7.8 % for Pugu kaolin. The values are decreased to 0.47% and increased to 5.44% at 1400°C for Pugu kaolin and Same clay respectively.

The decrease in water absorption for Pugu kaolin is due to densification that has taken place. This is further evidenced by the sharp decrease in apparent porosity at firing temperature between 1300°C and 1400°C (Table 2). The

increase in water absorption in Same clay at temperature between 1300°C and 1400°C as shown in Figure. 2 is due to increased apparent porosity which may be due to the formation of cristobalite which hinders easy densification of the component. These results are supported by El-Kheshen et al. [18] who investigated on sinterability, microstructure and properties of glass/ceramic composites and Zawrah et al. [17] who studied the effect of cristobalite formation on sinterability, microstructure and properties of glass/ceramic composites. On both studies, cristobalite phase precipitation in the composite lead to a more difficult densification.

4. Conclusion

Pugu kaolin is rich in kaolinite. During firing, quartz and

mullite are formed. At room temperature, Same clay is kaolinite. As it is fired it phase transformation is observed. It forms quartz, sillimanite and cristobalite above 1200°C. As the temperature is increased cristobalite peaks increases. The development of mullite phase in Pugu kaolin causes linear shrinkage increase with temperature while porosity and water absorption decreases. Water absorption values at firing temperature of 1400°C are 0.47% and 5.44% for Pugu kaolin and Same clay respectively. The sharp decrease in water absorption at a firing temperature between 1300°C and 1400°C in Pugu kaolin suggests the complete sintering to have been achieved at this temperature.

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