



**Full Length Research Article**

**THE INFLUENCE OF GRAIN SIZE AND CALCINATION CONDITIONS ON THE COMPRESSIVE STRENGTH OF THE MAHINA LIME- RICH BRICK CLAYS**

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**ABSTRACT**

Lime-rich clays from Mahina, Northwest part of Tanzania were used in this work. Chemical analysis showed that Mahina clays contain considerable amounts of CaO (16.25%) and CaCO<sub>3</sub> (25.41%). Mineralogical characterization of raw materials was done by XRD and DTA both of which revealed the presence of kaolinite, montmorillonite, quartz, calcite, feldspar and minor accessories. Bricks constructed from different grain size (63, 125, 300, 450 and 600 μm) were each calcined at 950 °C for 30 and 60 minutes. New phases identified were Gehlenite, wollastonite, anorthite and calcium aluminate. Coarser (600 μm) and finer (63 μm) particles gave lower values of compressive values, while the 450 μm samples showed maximum compressive strength. Bricks of 450 μm size were heated for 60, 30, 90, and 120 minutes at temperatures between 650 and 950 °C. The compressive strength increased with the calcination temperature and with a soak time of 120 minutes. The maximum strength (325 kg/cm<sup>2</sup>) was obtained from materials calcined for 90 minutes at 900 °C. Calcination duration had an influence on compressive strength. For instance samples calcined at 950 °C had strengths of 131 and 278 kg/cm<sup>2</sup> for calcination period of 5 and 90 minutes, respectively. However, there was reduction of compressive strength at higher temperatures i.e. >900. The results showed that when grain size and calcination conditions are properly chosen for lime rich clays, carbonate affects the quality of the products such as bricks positively.

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**INTRODUCTION**

The use of clay bricks in construction can be traced back more than five thousand years, and until present an increased production of burnt clay bricks is very important in many parts of the world. Almost any clay mineral composition of the clay is satisfactory for the manufacture of 'common brick' unless it contains a large percent of coarse stony material which cannot be eliminated in the preparation process or ground to adequate fineness. Furthermore, a high concentration of non-clay material in a silt-size range may cause difficulties by reducing the green and fired strength of the ware. Presence of harmful constituents found in clays which are sometimes known as impurities is another setback. However, the effect of impurities in a clay depends on several factors, among them being their nature and behaviour when the clay is used; their proportion in which they occur; the size and shape of the grains of clay, and of the impurities and the conditions under

which interaction takes place which include the temperature reached, the duration of heating and the effect of any substances which may be present. Clays composed of mixtures of clay minerals with appreciable amounts (25 to 50%) of unsorted fine-grained non-clay minerals are most satisfactory. Large amounts of montmorillonite clays, iron, alkalies and alkaline earth, either in the clay mineral or as other constituents, are detrimental because they cause too much shrinkage and reduce the vitrification range. Much kaolinite normally increases the temperature required for firing. Among all the detrimental components mentioned, calcium compounds in the form of lime are one of the most harmful constituent for clays which are to be used for the manufacture of bricks. Presence of calcium carbonate in higher amount has adverse properties on the fired bodies due to the distortion of the bricks on firing and also to the presence of CaO. Calcium carbonate when heated to about 800 °C is converted into lime which if it remains uncombined on cooling may absorb moisture from the air and swell so much so as to crack or burst the body. This is a serious defect in some clays used for brick-making where it is termed as lime-blowing (Elert *et al.*, 2003).

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Peter and Iberg (1978) have studied the influence of clay composition on the quality of fired bricks, and have reported positive effects of moderate amounts of carbonate which act as a flux. It has been found that carbonates lead to reduction in shrinkage and anisotropic behaviour and to an increase in compressive strength (Peters and Iberg, 1978; Cultrone *et al.*, 1998; Tite and Maniatis, 1975). Common bricks correspond to compressive strength of about 100 kgcm<sup>-2</sup> (Koeppel, 1976). According to Peters and Iberg (1978), the compressive strength of brick made from carbonate – poor raw materials could be increased from 380 to 550 kgcm<sup>-2</sup> by the addition of 5–10% CaCO<sub>3</sub>. With larger CaCO<sub>3</sub> additions no further increase of compressive strength resulted. Thus this shows that when the raw material is dressed properly, finely dispersed carbonate affects the quality of clay products positively. In Tanzania, there are several locations of suitable clay deposits which are used locally for the manufacture of bricks. However, their usage as sources of raw material has all along been too insignificant. Hitherto no detailed scientific study has been carried out on the deposits. In the present study investigation on the compressive strength of lime-rich clay from Mahina clay deposits in Kwimba district as influenced by the grain size and calcination conditions was carried out with the aim of assessing its suitability for the manufacture of bricks.

## MATERIALS AND METHODS

Lime-rich clay used in this work was collected from Mahina deposits in Kwimba district, Northwest part of Tanzania. Clay was milled and the particle size distributions was found to be about 20 wt% for clay particle whose size is <63µm (<63µm); 49 wt% of the particle >63µm but <500µm and 31 wt% of 500<X<600 µm. Small bricks from grain sizes (63, 125, 300, 450 and 600 µm) were fabricated manually using a clay to water ratio of 1:0.4 and were fashioned in steel moulds of 15 cm X 8 cm X 6 cm and allowed to dry at room temperature for 7 days and then subsequently oven dried at 110 °C for another 24 hours. Once dry they were calcined separately each at a rate of about 10 °C min<sup>-1</sup> until it reached 950 °C and was maintained for 30 and 60 minutes before cooled down. Then they were tested for compressive strength. Furthermore, the 450 µm grain size bricks were heated for 5, 30, 60, 90 and 120 minutes at temperatures between 650 and 950 °C, and then they were also tested for compressive strength.

### Mineralogical analysis

The mineralogical composition of the raw material as well as mineralogical changes taking place upon firing (calcined) were performed on Siemens D-5005 X-ray Diffractometer using Cu K 1 radiation (λ = 1.54056 Å) with 40KV, 40 mA. The mineralogical compositions of the raw and calcined materials were supplemented by the Differential Thermal Analysis (DTA) which was performed by the Stanton-Redcroft model with an attached digital voltmeter connected to a PC. The Thermocouple utilised was Pt/Pt: 13% Rh. Samples were heated at 10 °C/min to about 1200 °C with alumina as a reference material.

### Chemical analysis

Chemical analysis was determined by Siemen SRS 3000 X-ray fluorescence (Rhodium anode, 8 analyzer crystals with beryllium windows 125 µm) spectrometry.

## Compressive Strength tests

Compressive Strength tests were determined by using the tensile machine type UPD 40, Max load 400 KN, energy 400 v,50 Hz, 6A.

Compressive Strength (C<sub>s</sub>) was determined by the formula:

$$C_s = L/A$$

Where L = load in Newton, and A = area in mm<sup>2</sup>.

## RESULTS AND DISCUSSION

### The chemical composition

The chemical composition of clay is given in Table 1. The chemical composition indicated that Mahina clay contains considerable amounts of Fe<sub>2</sub>O<sub>3</sub>, CaO, MgO, SO<sub>3</sub>, CaCO<sub>3</sub>, and low Al<sub>2</sub>O<sub>3</sub>.

### Mineralogical characterization

The X-ray diffraction pattern of the raw lime-rich clay is given in Figure 1a. The pattern of clay fraction indicated the presence of kaolinite, illite and montmorillonite while non-clay minerals found were quartz, calcite and feldspar and weak signals of some minor accessories. DTA results of the raw material are given in Figure 2a, they closely agree with X-ray diffraction results. The endothermic peaks in the range 100–200 °C, 600 and 750–800 °C are associated to a mixture of clay minerals montmorillonite, illite and kaolinite (Mackenzie, 1957; Peters and Iberg, 1978). However, kaolinite appears to be present in minor and trace quantities here than is seen in X-ray. Another endothermic peak has been observed to occur between 880 and 910 °C, this is associated with the decomposition of CaCO<sub>3</sub>. Both XRD and DTA results are in good agreement with the chemical analysis given in Table 1.

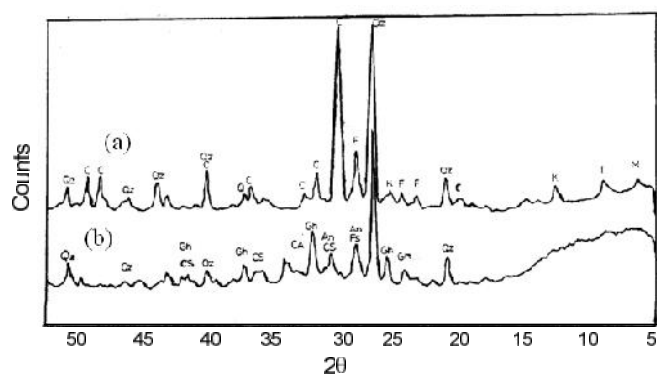


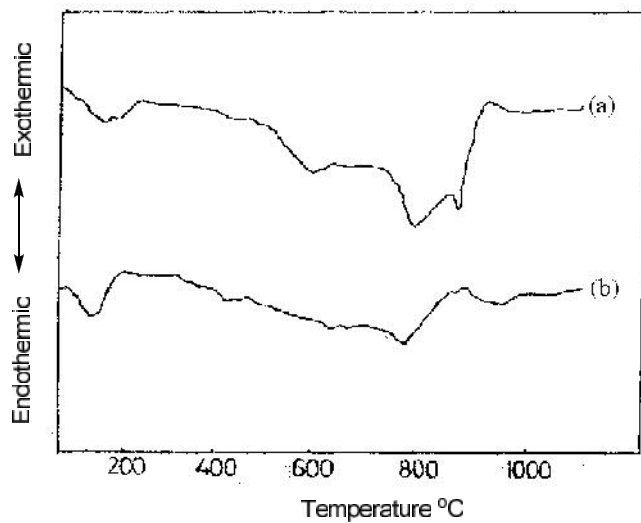
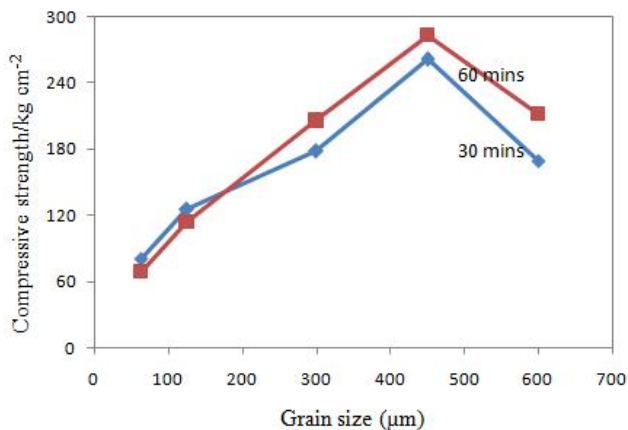
Fig. 1. X-Ray diffraction pattern of (a) Raw lime-rich brick clay: M = Montmorillonite, I = Illite, K = Kaolinite, Qz = Quartz, C = Calcite, F = Feldspar; (b) Calcined at 950 °C for 30 min: CS = Wollastonite, CA = Calcium Aluminate, Gh = Gehlenite, An = Anorthite

### Influence of grain size of raw materials on compressive strength

The grain size of the raw material is one of the most influential factors in determining the compressive strength of the product. Figure 3 gives results of the grain size (63, 125, 300, 450 and 600 µm) of lime rich bricks calcined at 950 °C for both 30 and 60 minutes. Results obtained have shown that grain size has an

**Table 1. Chemical composition of lime-rich clay (in mass %)**

Component	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	CaCO <sub>3</sub>	LOI
% mass	40.02	13.76	4.23	16.25	2.81	3.01	0.74	0.61	25.41	17.49

**Fig. 2. DTA Thermograms of (a) Raw lime-rich clay (b) Calcined at 950 °C for 30 min****Fig. 3. Effect of grain size (63, 125, 300, 450 and 600 μm) of raw material on compressive strength**

influence on the properties of the product. As indicated in Figure 3, courser (600 μm) and finer (63 μm) particles had lower values of compressive strength when fired for both 30 and 60 minutes, while the 450 μm samples showed maximum compressive strength. The lower values of compressive strength for courser grains in lime rich clay is due to activation of particles in the raw samples which leads to the development of relatively large CaO grains which are unevenly distributed throughout the fired sample such that some places are rich in lime and others have very little lime. This results into a product of a weak structure due to inhomogeneous distribution of lime and are responsible for voids and for initiating cracking into the body as they act as stress concentration points within the product (Davidge, 1979; Martin-Marquez, 2008). Furthermore, some of them might not react with the aluminosilicates and other components in the fired body. If at all the brick is exposed to moisture/water or humidity, hydration takes place and the hydration reactions of the unreacted cores are accompanied by volume increase (lime-blowing) which is followed by cracks which develop inside the body.

Funk and Dinger (1994) pointed out that grain size and particle size distribution variation affect homogeneity because reactions begin where species make contact which in turn enhances the probability of the desired reactions to take place. When homogeneity is poor, large masses of single components can sinter before having the opportunity to react with other components. Although homogeneity is achieved when particles are finely ground yet the compressive strength was low an observation which is not expected for fine grains. This could be viewed as being due to interlocking of grains (particles) inside the body resulting in increase of glassy material. Consistent with this study Hemaly *et al.* (1984) found that bricks of grain size 149 μm gave compressive strengths of 83 and 66 kgcm<sup>-2</sup> for calcination times 30 and 60 minutes, respectively, while 1410 μm sample gave 281 kgcm<sup>-2</sup> under same conditions.

The sample which showed maximum compressive strength (450 μm) was a sample having a range of grain sizes in proper proportions so that tight packing of the granular materials could be achieved. Thus the preparation route might consist of grinding the raw material to a proper fineness which in this study was 450 μm. The same Figure 3 depicts that particle size distribution in the range 63 μm to 200 μm fired for 30 minutes gave higher compressive strengths than in the particles calcined for 60 minutes. But as the size of particles got larger it reached a point where the compressive strength for the particles calcined for 60 minutes became higher than the 30 minutes calcined particle compressive strength, this took place at >200 μm. This is because prolonged calcinations on larger particles enabled CaCO<sub>3</sub> dissociation to be completed. In addition the reaction between lime and clay was enhanced. For fine grains, calcinations at high temperatures ( 950 °C) form glassy materials with hardly any hydraulic properties. The glassy product formed show less tendency to react with lime to produce a binding agent. The compressive strength therefore decreases.

### Influence of calcination conditions

#### Mineralogical phase changes

Mineralogical phase changes observed with X-ray diffraction for calcined clay at 950 °C are given in Figure 1b. It should be noted that throughout the process of raising the temperature to 950 °C, the fired bricks were subjected to a series of changes in their physical properties, while complicated chemical reactions occurred both within the separate mineral constituents and between the different minerals. Peters and Iberg (1978) observed that no major mineralogical changes occur up to 500 °C. Kaolinite is destroyed between 500 and 550 °C, and the breakdown of dolomite starts and is completed at 650 °C. Normally the breakdown of fine-grained calcite begins at 600 °C, increases at 650 °C and is completed at 700 °C. Their amounts decrease rapidly between 800 °C and disappear completely at 950 °C. Along with the decomposition of calcite, the crystallization of CaO can be observed when it reaches a maximum at 725 °C; above 800 °C the CaO content

decreases and CaO commonly disappears between 900 °C and 950 °C. In the present work (Figure 1b), it was observed that calcite peaks disappeared and no calcium oxide peaks were detected an observation which agrees with Peters and Iberg's (1978) work. New phases formed by the interaction between lime and other constituents during calcination were also seen. These were identified as Gehlenite – calcium aluminium silicate ( $C_2AS - Ca_2Al(AlSi)O_7$ ), wollastonite – calcium silicate ( $CS - CaSiO_3$ ), anorthite – calcium aluminium silicate ( $CAS_2 - CaAl_2Si_2O_8$ ) and calcium aluminate. It is pointed out that normally appreciable amounts of gehlenite are formed rapidly above 860 °C, but above 920 °C the formation slows down and reaches a maximum at 1050 °C (Peters and Iberg, 1978). In Ca-rich samples, the gehlenite content decreases during soaking as a result of the reactions



And



Whereby quartz is used and anorthite and wollastonite are formed. The crystallization of wollastonite, diopside ( $CaMgSi_2O_6$ ) and anorthite also start at 860 °C but at a slower rate than that of gehlenite (Peters and Iberg, 1978). The same observations have been reported by Cultrone *et al.*, (2001). The DTA thermogram results given in Figure 2b were in good agreement with the X-ray patterns. The endothermic peaks were found at 140 °C; 740 – 800 °C; and 850 – 900 °C. They are associated with loss of absorbed water; decomposition of  $CaCO_3$  and calcium aluminosilicates, respectively.

### Compressive strength

A grain size of 450 µm was used in the subsequent work. Results of the compressive strength of bricks of particle size of 450 µm fired at 650, 700, 750, 800, 900 and 950 °C for 60, 30, 90, and 120 minutes are given in Figure 4.

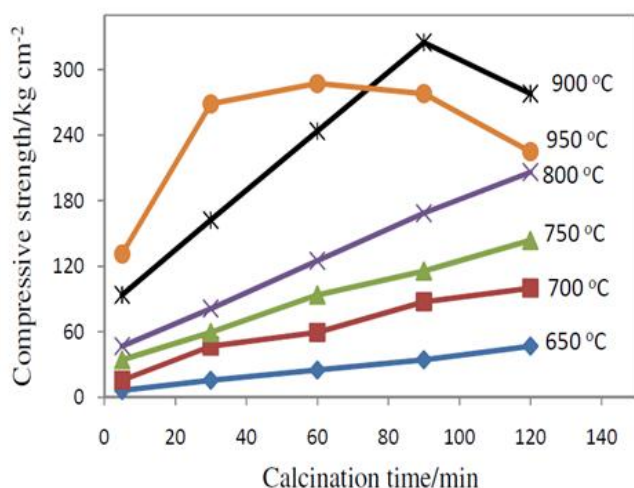


Fig. 4. Effect of calcination conditions of 450 µm grain size on compressive strength

The results of the effect of the activation on the compressive strength have shown that compressive strength increased with the firing temperature and with a soak time of 120 minutes. However, there was reduction of compressive strength at

higher temperatures i.e. >900 °C. This reduction might be due to the formation of glassy material which could not react with lime to produce calcium silicates as binding agent. The effect of the firing temperature on compressive strength was clearly seen when the sample was subjected at 650 and 900 °C for 90 minutes. The compressive strength values were 35 and 325  $kgcm^{-2}$  respectively. The observed increase in compressive strength of the bricks with the firing temperature could be viewed as being due to the decrease of porosity of the materials, which is also related to the increase of bulk density (Akwilapo, 1999; Elwan and El-Didamony, 1999). Furthermore, results showed that calcination period had influence on compressive strength. For instance bricks fired at 950 °C had compressive strengths of 131 and 278  $kgcm^{-2}$  for calcination period of 5 and 90 minutes, respectively. This is because prolonged calcination period (soaking time) allows for  $CaCO_3$  complete dissociation and the reaction between lime and other clay constituents, and mechanical/physical properties such as compressive strength are enhanced (Elwan and El-Didamony, 1999; Elert, 2003). The same observation was seen in other firing temperatures. However, the maximum compressive strength (325  $kgcm^{-2}$ ) obtained in this study was from materials calcined for 90 minutes at 900 °C.

### Conclusion

Findings from the present study indicated that:

- Upon firing, significant mineralogical changes do occur. Carbonate rich clay undergo transformation at about 800 °C and new calcium silicates and aluminium-silicates are formed. Results which are in line with other researchers (Peters and Iberg, 1978, Cultrone *et al.*, 2001)
- When lime rich clays are used for the manufacture of bricks, the grain size, content of carbonate and calcination conditions should be controlled in order to avoid lime blowing. Proper choice of good range of grain size (450 µm) and calcination conditions such as firing temperatures (900 °C) and calcination periods (90 minutes) will increase the comprehensive strength of lime rich bricks to a significant value. Appropriate grain size of raw materials will be finely dispersed in the matrix/body so that they will all react with other constituents in the body and this will produce bricks of high quality.

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