

Influence of Rate of Temperature Rise during Firing Process on Compressive Strength of Mukono Ntawo Ball Clay

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Abstract

This study was focused on the effect of varying the rate of temperature rise during firing process on the compressive strength of ball clay slab from Ntawo clay deposit from Mukono District, in central Uganda. The rectangular clay slabs of dimensions of 16cm by 4cm by 1cm were produced of clay particles of 45 μ m size. They were dried under open shade and fired at varied rate of temperature rise of 2^oC min⁻¹, 3^oC min⁻¹, 4^oC min⁻¹, 5^oC min⁻¹ and 6^oC min⁻¹ to firing temperatures of 500^oC, 600^oC, 700^oC, 800^oC, 900^oC and 1000^oC and held at the temperatures for one hour. The fired slabs were then cooled to room temperature in the electric furnace. The chemical composition by percentage weight of the unfired clay sample was determined by x-ray diffraction using RIX 3000 spectrometer. The compressive force of the samples were measured using 500SN-1299417 compressive machine. It was observed that Ntawo ball clay contained 67.20% of silica, 18.20% of alumina, 2.83% of iron oxide, 1.38% of titanium, 0.06% of phosphorous penta-oxide, 0.31% of calcium oxide, 0.98% of potassium oxide and 0.19% of sodium oxide. The compressive strength of the samples decreased with increase in the rate of temperature rise. The null hypothesis that the compressive strength of the fired samples did not depend on the rate of temperature rise during the firing process was rejected at $\alpha=0.05$ and $\alpha=0.01$ levels of significance. The decrease of compressive strength with increase of the rate of temperature rise between 2^oC min⁻¹ to 3^oC min⁻¹ was negligible for all the firing temperatures. The percentage decrease of compressive strength with the rate of temperature rise was appreciable for firing temperatures of 500^oC and 1000^oC of 11%.

Keywords: Rate of temperature rise; Compressive strength; Firing temperature and Fired clay.

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1. Introduction

Clay is composed of fine-grained natural rock particles that contain one or more clay minerals with traces of metal oxides and organic matter[29]. Clay has minerals that make it exhibit the property of plasticity when wet , making it easy to be molded into any shape. The chemical compounds that make up clay minerals have grain sizes typically smaller than 2 μ m and consequently referred to as clay sizes only for the purpose of definition [30]. The clay minerals originate typically from a long periods of gradual chemical weathering of rocks like the silicate-bearing rocks which are eroded by low concentration of carbonic acid and other dilute acidic solvents[5] . These acidic solvents attack and weather rock layer by layer as the acid reacts with dissolvable rock substances. The decomposition of rock materials can also be initiated by physical processes such as the breaking up of the rock due to thermal stresses created in the rocks. The rocks explode and break into smaller particles producing clay particles. The loose particles on the rocks can also be worn away by water and strong winds that blow over the rocks. The primary clays are formed as residual deposits at the site of formation. Secondary clays are clays that have been transported from their original location by water and wind erosion and deposited in a new place as sedimentary deposit [9]. Clay deposits are typically associated with very low energy depositional environments such as large lakes and marine basins [9]. This explains why clay deposits are associated with valleys and low lying areas.

The plastic behavior of clays and their hardened forms after firing has made them particularly useful since the beginning of civilization, for making cooking pots, bricks, porcelain tiles and also drainage pipes .The thermal properties of clays makes clay suitable for making refractory materials need for lining the surfaces of kilns, ovens and furnaces in the manufacture of metallic products. The clay used for refractory material production are often called fire clays. They are used to manufacture heat resistant tiles and bricks..Ball clays from China clay, predominantly containing kaolin because of its property is used to make ceramics filter and is also used in drug manufacturing. .Expanded clay blocks are used for insulation in many applications. .In general clays are widely used in the manufacture of many traditional ceramics. products and each ceramic product requires clays that has particular and appropriate characteristics. The mineralogical composition of clay bodies and the heating processes determine the final property of the ceramic product. The mineralogical compounds undergo several changes during drying and firing stages. Many researches have investigated the properties of fired clay products [31, 32].The purpose of this study was to further look at local clay resource where so far few studies have been done to understand their properties. In this particular study the influence of rate of temperature rise on compressive strength of fired clay for selected firing temperatures has been undertaken.

2. Materials and Methods

The ball clay for this study was obtained from eight pits within the same clay deposit at Ntawo deposit Mukono in central Uganda .The clay from the different pits were mixed together thoroughly and dried. The dried clay was then ground into powder form. A portion of the powder was compressed into 30mm tablets and these were sent to the Mintek Mineral Research Institute in South Africa for chemical composition analysis using an X-ray diffract meter RIX 3000 machine.

The rest of the powdered clay was soaked and thoroughly mixed in a bucket of water to get a slurry of clay. The slurry was poured into a stack of sieves connected to a mechanical vibrator to sieve through clay of particles of size of 45µm.

The sieved slurry was left to dry for a week to form a paste which was pressed into rectangle metal mold of dimensions of 16cm by 4cm by 1cm. The molded samples were dried under open shade to avoid fast drying that could lead to cracks forming on the samples. While drying the samples were regularly turned over to prevent warping of the samples.

A total of one hundred and fifty dried samples were subjected to preheating at a temperature of 1100C in an electric furnace to remove the moisture left from the open air drying process. This was to prevent swelling and bloating of the samples during the high temperature firing of the samples. Five of the dried clay samples were loaded into the electric furnace for each of the rate of temperature rise and fired at the rate of temperature rise of 2⁰C min⁻¹, 3⁰C min⁻¹, 4⁰C min⁻¹, 5⁰C min⁻¹ and 6⁰C min⁻¹. The firing at each of the rate of temperature rise was done reaching the temperature of 500⁰C and held at the final temperature for one hour. The above firing process was done to reach the firing temperatures of 600⁰C, 700⁰C, 800⁰C, 900⁰C and 1000⁰C but each time allowing the holding time to be for one hour

3. Results and Discussion.

3.1 Chemical Composition of the clay

The result of the X-ray diffraction are shown in the figure 1 below. The x-ray diffraction spectrogram was used to work out the percentage of chemical compounds in Ntawo ball clay

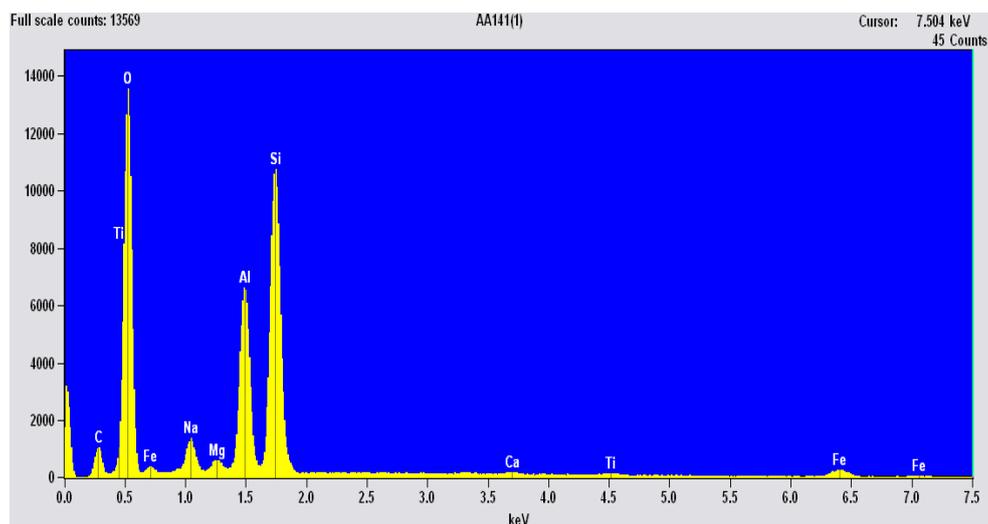


Figure 1: X-ray Diffract Pattern of Ntawo ball clays

The percentage composition by weight of the different chemical components in Ntawo ball clay from the diffraction spectrogram are as shown in table 1 below

Table 1: Chemical Composition of Ntawo Ball Clay

| Compound | Chemical Symbol | Percentage weight |
|------------------------|--------------------------------|-------------------|
| Silica | SiO ₂ | 67.20 |
| Alumina | Al ₂ O ₃ | 18.20 |
| Calcium Oxide | CaO | 0.31 |
| Iron Oxide | Fe ₂ O ₃ | 2.83 |
| Potassium Oxide | K ₂ O | 0.98 |
| Magnesium Oxide | MgO | 0.36 |
| Sodium Oxide | Na ₂ O | 0.19 |
| Titanium dioxide | TiO ₂ | 1.38 |
| Phosphorus Penta oxide | P ₂ O ₅ | 0.06 |

The percentage of the different mineral compounds found in the samples were within the range of those obtained by earlier by Nyakairu [2001] where silica was 67.18% alumina was 18.23% iron oxide was 2.83% calcium oxide was 0.306% magnesium oxide was 0.363% and sodium oxide was 0.185%.

3.2 Compressive Strength

The fired samples were subjected to breaking force in order to determine the compressive strength of the samples.

The values of the average breaking force of the fired samples obtained were as shown below in table 2 for the different firing temperatures for each of the rise of temperature to the firing temperature

Table 2: Values of average breaking force of clay samples fired at different rates of temperature rise at six different set firing temperatures.

| Rate of temperature rise/°Cmin ⁻¹ | Average breaking force/ kN | | | | | |
|--|----------------------------|-------|-------|-------|-------|--------|
| | 500°C | 600°C | 700°C | 800°C | 900°C | 1000°C |
| 2 | 8.16 | 8.30 | 9.92 | 12.16 | 20.96 | 30.90 |
| 3 | 8.12 | 8.32 | 9.88 | 12.08 | 21.04 | 30.30 |
| 4 | 8.12 | 8.23 | 8.23 | 11.68 | 20.76 | 28.00 |
| 5 | 7.84 | 8.12 | 8.12 | 11.32 | 20.48 | 26.90 |
| 6 | 7.28 | 8.04 | 8.04 | 11.24 | 20.32 | 27.90 |

The breaking forces were used to calculate the compressive strength of the samples. The first results were for samples fired to 500⁰C and the values obtained for firing temperature of 500⁰C was as shown in table 3 below

Table 1: Compressive Strength at different Rates of Temperature Rise of samples fired to 500⁰C

| Rate of temperature rise/ ⁰ C min ⁻¹ | Average Compressive strength/MPa | Variance, δ_m^2 within the mean | Variance, δ_x^2 within the sample |
|--|----------------------------------|--|--|
| 2 | 7.41 | 0.051984 | 0.006500 |
| 3 | 7.38 | 0.039204 | 0.009275 |
| 4 | 7.37 | 0.035344 | 0.012925 |
| 5 | 7.13 | 0.002704 | 0.022700 |
| 6 | 6.62 | 0.315844 | 0.009200 |
| Grand Mean Compressive strength | 7.182 | 0.089016 | 0.01221 |
| Overall Variance within the mean | | | 0.44508 |
| Calculated F-value | | | 36.45209 |

The F-value for the data was calculated to test the null hypothesis HO₁ that the compressive strength at the firing temperature of 500⁰C did not depend on the rate of rise of temperature at two levels of significance $\alpha=0.05$ and $\alpha=0.01$ respectively. For degrees of freedom of 4 and 20 of the data used. The calculated F-value of 36.45 was greater than the expected values of 2.4 and 4.43 at the set levels of significance of $\alpha=0.05$ and $\alpha=0.01$ respectively. The study therefore rejects the null hypothesis and confirms that there was a relationship between compressive strength and the rate of rise of temperature when the samples were fired to 500⁰C. The graph of compressive strength was then plotted against the rate of rise of temperature to obtain the graph in figure 2 below to find any relationship between the compressive strength and rate of temperature rise.

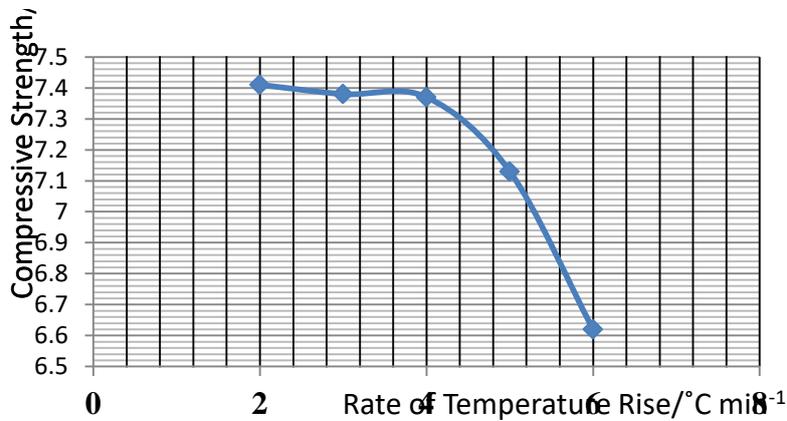


Figure 2: Effect of the Rate of Temperature Rise on the Compressive Strength of Samples fired to 500⁰C

The graph indicates that the variation of compressive strength was negligible for values of rate of temperature rise between 2^oC min⁻¹ and 4^oC min⁻¹ but decreases appreciably by 10% between the rate of temperature rise of 4^oC min⁻¹ and 6^oC min⁻¹.

The second set of results was for samples fired to 600^oC. The results of the compressive strength for the firing temperature of 600^oC were obtained as shown in Table 4.3 below.

Table 2: Values of Compressive Strength at different Rates of Temperature Rise of Samples Fired to 600^oC

| Rate of temperature rise/ ^o C min ⁻¹ | Average Compressive Strength/MPa | Variance, δ_m^2 within the mean | Variance, δ_x^2 within the sample |
|--|----------------------------------|--|--|
| 2 | 7.55 | 0.007056 | 0.01758 |
| 3 | 7.56 | 0.008836 | 0.01085 |
| 4 | 7.53 | 0.004096 | 0.01085 |
| 5 | 7.38 | 0.007396 | 0.009725 |
| 6 | 7.31 | 0.024336 | 0.006500 |
| Grand Mean Compressive strength | 7.466 | 0.010344 | 0.0011101 |
| Overall Variance within the mean | | | 0.05172 |
| Calculated F-value | | | 4.65904 |

For degrees of freedom of 4 and 20 of the data above, the F-value of 4.66 was obtained and this was greater than the expected F-values of 2.4 and 4.43 at the set levels of significance of $\alpha=0.05$ and $\alpha=0.01$ respectively. The hypothesis that the compressive strength did not depend on the rate of temperature rise at the two levels were rejected. The compressive strength for samples fired to 600^oC were affected by the rate of temperature rise.

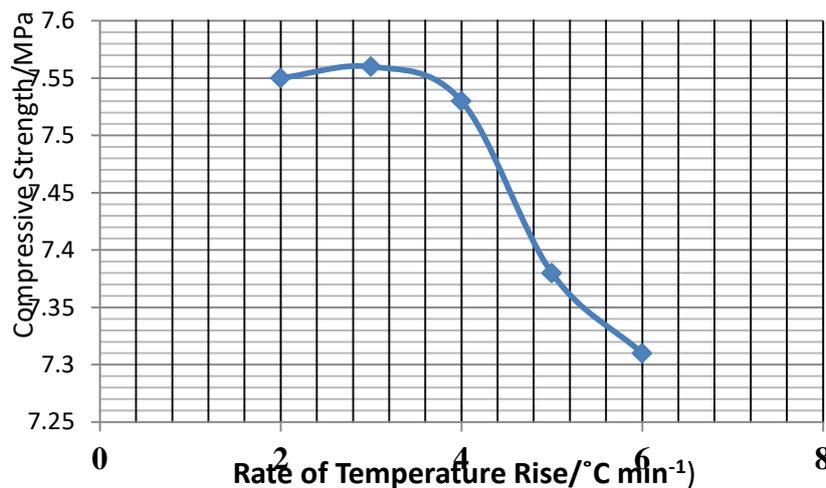


Figure 2: Effect of the Rate of Temperature Rise on the Compressive Strength of Samples fired to 600^oC

The graph of the compressive strength against the rate of rise of temperature was plotted as shown figure 3 below to find the relationship between the compressive strength and the rate of temperature rise for the samples fired to 600°C.

The graph indeed showed a negligible change of compressive strength with the rate of temperature rise for values between 2°C min⁻¹ and 3°C min⁻¹. The compressive strength this time fell by a small amount by 3% between 3°C min⁻¹ and 6°C min⁻¹. The decrease in compressive strength with rate of rise of temperature this time for values of temperature rise greater than 3°C min⁻¹ was small.

The result for the variation of compressive strength of samples fired to 700°C with rate of temperature rise obtained was as shown table 5 below

Table 3: Values of Compressive Strength at different Rates of Temperature Rise of Samples Fired to 700°C

| Rate of temperature rise/°C min ⁻¹ | Average Compressive strength/MPa | Variance, δ_m^2 within the mean | Variance, δ_x^2 within the sample |
|---|----------------------------------|--|--|
| 2 | 9.00 | 0.019044 | 0.008125 |
| 3 | 8.98 | 0.013924 | 0.007725 |
| 4 | 8.80 | 0.003844 | 0.009725 |
| 5 | 8.80 | 0.003844 | 0.009725 |
| 6 | 8.73 | 0.017424 | 0.000000 |
| Grand Mean Compressive strength | 8.862 | 0.011616 | 0.074600 |
| Overall Variance within the mean | | | 0.058080 |
| Calculated F-value | | | 0.778552 |

The calculated F-value 0.78 for firing temperature of 700°C this time was much smaller than the expected value of 2.4 and 4.43 at the set levels of significance, of $\alpha=0.05$ and $\alpha=0.01$ respectively. This implies that the rate of rise of temperature does not affect the compressive strength of the samples fired to 700°C. The graph of compressive strength was plotted against rate of temperature rise as in figure 4 to find the relationship for the samples when fired to 700°C. The graph in figure 4 has shown that between the rate on temperature rise of 2°Cmin⁻¹ and 3°Cmin⁻¹, there was negligible decrease in compressive strength of samples and even the decrease of compressive strength between the rate of temperature rise of 3°C min-1 and 6°C min-1 was by 2.7% which is very small and comparable to that of samples fired to 60°C.. The results of compressive strength of samples fired to the temperature of 800°C were obtained as shown in Table 4.5 below.

The calculated F-value 72.87 for firing temperature of 800°C was much greater than the expected F-values of 2.4 and 4.43 at the set levels of significance, of $\alpha=0.05$ and $\alpha=0.01$ respectively. This implies that the rate of rise of temperature this time affected the compressive strength of the samples fired to 800°C. The graph of compressive strength was plotted against rate of temperature rise as in figure 5 below to find the relationship between the compressive strength and the rate of temperature rise for the samples when fired to 800°C.

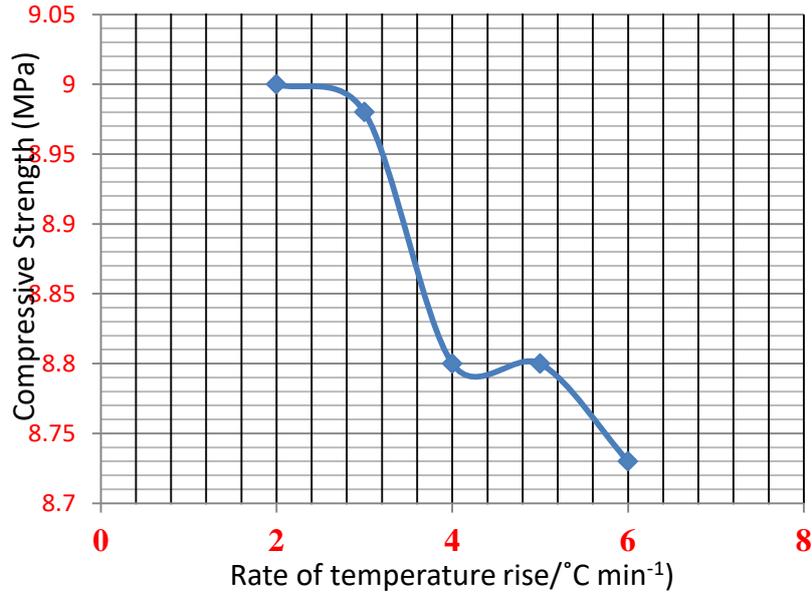


Figure 4: Effect of the Rate of Temperature Rise on the Compressive Strength of Samples fired to 700°C

Table 4: Values of compressive strength at different Rates of Temperature Rise of Samples Fired to 800°C

| Rate of temperature rise/°C min ⁻¹ | Average Compressive strength/MPa | Variance, δ_m^2 within the mean | Variance, δ_x^2 within the sample |
|---|----------------------------------|--|--|
| 2 | 11.05 | 0.173056 | 0.006500 |
| 3 | 10.95 | 0.099856 | 0.006500 |
| 4 | 10.66 | 0.000676 | 0.009425 |
| 5 | 10.29 | 0.118336 | 0.009725 |
| 6 | 10.22 | 0.171396 | 0.006500 |
| Grand Mean Compressive strength | 10.634 | 0.112664 | 0.0077300 |
| Overall Variance within the mean | | | 0.563320 |
| Calculated F-value | | | 72.8745 |

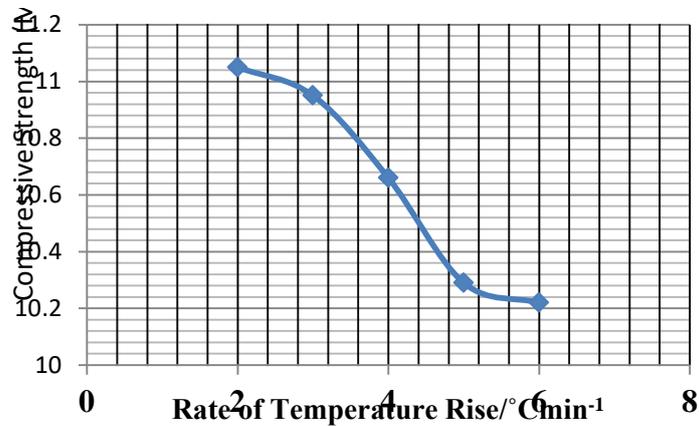


Figure 3: Effect of the Rate of Temperature Rise on the Compressive Strength of Samples fired to 800°C

The change compressive strength of samples fired to 800°C was sharp right the rate of temperature rise of 2°C min⁻¹ to 6°C min⁻¹ by 7.5%

The results of compressive strength of samples fired to a temperature of 900°C were obtained as shown in Table 7 below.

Table 5: Compressive Strength at Different Rates of Temperature Rise of Samples Fired to 900°C

| Rate of temperature rise/°C min ⁻¹ | Average Compressive strength/MPa | Variance, δ_m^2 within the mean | Variance, δ_x^2 within the sample |
|---|----------------------------------|--|--|
| 2 | 19.05 | 0.043264 | 0.02270 |
| 3 | 19.13 | 0.082944 | 0.00650 |
| 4 | 18.87 | 0.000784 | 0.03890 |
| 5 | 18.65 | 0.036864 | 0.00905 |
| 6 | 18.51 | 0.110224 | 0.024875 |
| Grand Mean Compressive strength | 18.842 | 0.054816 | 0.020405 |
| Overall Variance within the mean | | | 0.27408 |
| Calculated F-value | | | 13.432 |

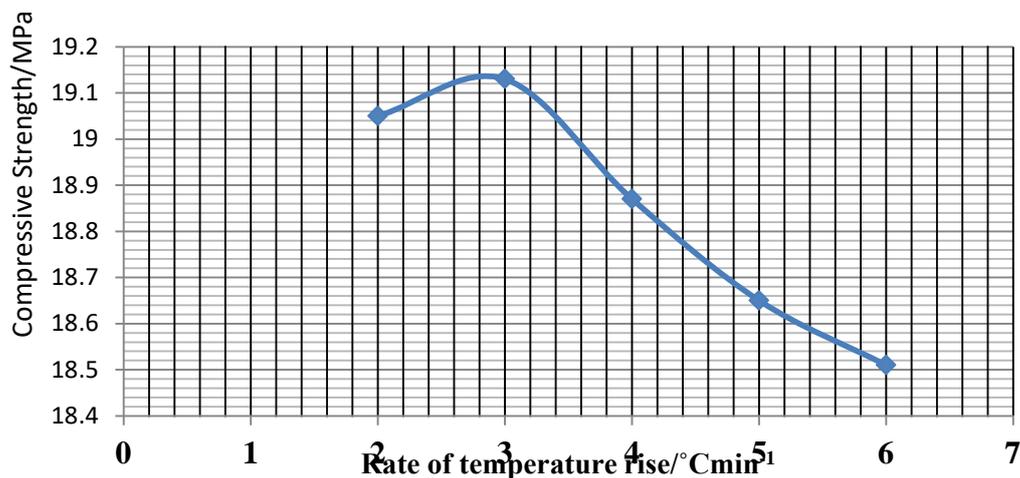


Figure 4: Effect of the Rate of Temperature Rise on the Compressive Strength of Samples fired to 900°C

The calculated F-value is 13.43 for samples fired to 900°C which is much greater than the expected F-values of 2.4 and 4.43 at the set levels of significance, of $\alpha=0.05$ and $\alpha=0.01$ respectively. This implies that the rate of rise of temperature affected the compressive strength of the samples fired to 900°C. The graph of compressive strength was plotted against rate of temperature rise as in figure 6 below to find the relationship between the

compressive strength and the rate of temperature rise for the samples when fired to 900°C. The graph in figure 6 has shown that, the compressive strength values decreased with increase in rates of temperature rise for the samples fired to 900°C by small percent of 3%. The last set of results was for samples fired to 1000°C and the results are as shown table 8 below.

Table 6: Values of Compressive Strength at different Rates of Temperature Rise of Samples Fired to 1000°C

| Rate of temperature rise/°C min ⁻¹ | Average Compressive strength/MPa | Variance, δ_m^2 within the mean | Variance, δ_x^2 within the sample |
|---|----------------------------------|--|--|
| 2 | 28.11 | 2.9929 | 1.014025 |
| 3 | 27.53 | 1.3225 | 0.770500 |
| 4 | 25.56 | 0.6724 | 0.882700 |
| 5 | 25.45 | 0.8649 | 0.165625 |
| 6 | 25.25 | 1.2769 | 0.393675 |
| Grand Mean Compressive strength | 26.38 | 1.42592 | 0.645305 |
| Overall Variance within the mean | | | 7.1296 |
| Calculated F-value | | | 11.04842 |

The calculated F-value is 11.05 for samples fired to 1000°C which is greater than the expected F-values of 2.4 and 4.43 at the set levels of significance, of $\alpha=0.05$ and $\alpha=0.01$ respectively. This implies that the rate of rise of temperature rise affected the compressive strength of the samples fired to 1000°C. The graph of compressive strength was plotted against rate of temperature rise as in figure 7 to find the relationship between the compressive strength and the rate I of rise of temperature for the samples fired to 1000°C.

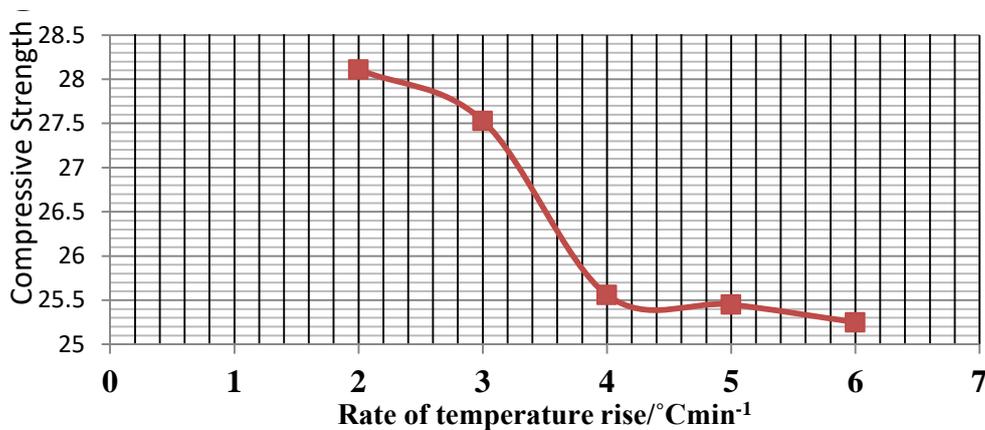


Figure 5: Effect of the Rate of Temperature Rise on the Compressive Strength of Samples fired to 1000°C

The graph shows that the compressive strength decreased from the rate of temperature rise of 2°C min⁻¹ to 4°C

min⁻¹ by 9.1% and the change was negligible beyond 4⁰C min⁻¹ remained unaltered at about 25.5 MPa.

In general, there was decrease in compressive strength of the fired samples with increase in the rate of temperature rise for all the fixed temperatures between 500°C and 1000°C except for the firing temperature of 700⁰C. There was no difference between compressive strengths.

4. Conclusion

The research carried out in this study has established that fast heating rates or high rates of temperature rise decreased the compressive strength and slow heating rates or low rates of temperature rise increased the compressive strength. The results obtained for the relationship between compressive strength and rate of temperature rise indicated an average percentage decrease in compressive strength. The highest compressive strength of 28.11MPa was obtained at a rate of temperature rise of 2°Cmin⁻¹ for the samples fired to 1000°Cmin⁻¹. The overall average percentage decrease in compressive strength was 6.7%.

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