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The Effect of Holding Time on the Compressive Strength of Mukono Ntawo Ball Clay

Isabirye Jeromy Moses[1], Enjiku Ben D.D[2]
[1]Department of Physics, Kyambogo University, P.O.Box 1 Kyambogo – Uganda
[2]Department of Natural Sciences, Kampala University, P. O. Box 25454 Kampala

Abstract:-This study was to find the effect of holding time on the compressive strength of fired slabs produced from ball clay obtained from Ntawoclay deposit in Mukono District, in central Uganda .The chemical composition by percentage weight of the clay was analyzed based on x-ray diffraction pattern obtained using RIX 3000 Spectrometer machine. The clay samples wereproduced from clay slurry of particle size of 45µm. The dried samples were fired in electric furnace to temperatures of 500^0C, 600^0C, 700^0C, 800^0C, 900^0C and 1000^0C.The temperatures were held for holding times of 20minutes, 30mintes,40minutes 50minutes and 60minutes..and allowed to cool to room temperature in the furnace. The compressive force required to break the fired slabs was measured using 500SN-1299417 machine. The breaking forces were converted into the compressive strengthof the slabs to find out how holding time affected compressive strength of the slabs..A null hypotheses, H_{01}- that compressive strength of fired clay does not depend on holding time when fired to various firing temperatures at $\propto = 0.01$ and $\propto = 0.05$ levels of significance. The Ntawo ball clay was found to contain 67.2% of silica, 18.2% of alumina, 2.83 % of Fe_2O_3 and 1.84% of sodium, calcium and magnesium fluxes. The compressive strength of the slabs was found to decrease from 4.2% to 0.4% with holding time for firing temperatures between 500℃ and 800℃. The compressive strength variation was negligible, less than 1% for temperatures beyond 900℃ to 1000℃. The compressive strength of ball clay beyond 800℃ does not need longer holding time because according to this study will not affect the compressive strength. The compressive strength for ball clay fired beyond 1000℃ and 60 minutes is worth investigation to find whether any change in the mineral compounds of ball clay can affect the above results.

Keywords:-Holding Time; Firing Temperature; Fired Clay Product; Compressive Strength.

I. INTRODUCTION

The properties of clay bodies is known to be affected by its thermal treatment when fired, and by its chemical and mineralogical composition. The chemical composition is oftendetermined by source of the clay. or source of the clay. Deposits of ball clay are located in the plains and valleys or along rivers banks. The ball clays are scattered in areas characterized by deposits of lacustrine and alluvial soil types..Most ball clay deposits belong to the sedimentary group type [11, 14, 15, 20, 28,29].. Compressive strength is one of the major physical properties of fired ball clay products, which makes clay products to resist shrinkage and cracking of the face. Compressive strength is known to be highly affected by firing profiles, method of manufacture, porosity, physical, chemical and mineralogical properties of the ball clay used [8, 12, 17, 31]. Firing of clay products for example make its shrinkage increase with higher temperature firing[8].To compensate for high firing shrinkage due to high alumina content; silica in form of sand is added to clay in production of ceramic products. The chemical composition of common clay often contain not less than 60% of silica by weight not less than 15% of alumina and not less than 3% of ferric oxide..The processes of production of fired clay productsinclude: mining, of the clay preparation of the clay, forming, drying and firing [4,5].The extent of firing is a function of both time and temperature. The firing facilitates chemical reactions that develops theinter-particulate bond, the strength, pore structure and colour of the clay product [2, 10, 16, 22].The chemical reaction of oxidation, long periods f heating may be necessary for complete removal of carbonaceous matter from clay samples of several millimeters thick [6, 9].Varying holding time has been fund to affect slightly the quality of some fired clay products. In Uganda, ball clay is used for making pottery and bricks for construction, but are found to be of low strength. Fired ball clay exhibits high melting point, compressive strength, porosity and water absorption when fired at high temperatures. The properties for fired ball clay that has been done include: shrinkage of clay and its mixture.Some studies have been conducted involving ball clay used for pottery products in Uganda [11, 14, 15, 20, 27, 28, 29]. The results have been found to be useful to produce high and low strength ceramic products using ball clay as a raw material. In Uganda, high compressive strength fired ball clay products are imported from a broad, yet ball clay is in plenty, but not tried for production of high quality products of high compressive strength. The information on properties of fired local ball clay used in the manufacture of fired ball clay products of high strength would enhance quality of fired ceramic materials locally produced. The purpose of the study was to investigate the effect of holding time on compressive strength of Ntawo fired ball clay to add to the needed information on local n ball clay.

II. MATERIALS AND METHODS

The ball clay used in this study was dug from five different pits within the same clay siteof Ntawoclaydeposit in MukonoDistrict in central Uganda.Ten grams of clay sample from each of the five pits was mixed and finely milled into a powder.The powder was then compressed into 30mmdiameter tablets and the tablets were sent to South Africa Mintek Mineral Research Institute for identification of the clay components using x-ray diffraction meter RIX3000.

The X-ray diffraction patterns were obtained by scanning the samples in steps of 0.034° in a range of diffraction angles from 10° to 59° of 2θ° for ball clay using copper ($K\alpha$) as X-ray source with a wave length 1.541Å. The results wereas shown in figure 1 below.

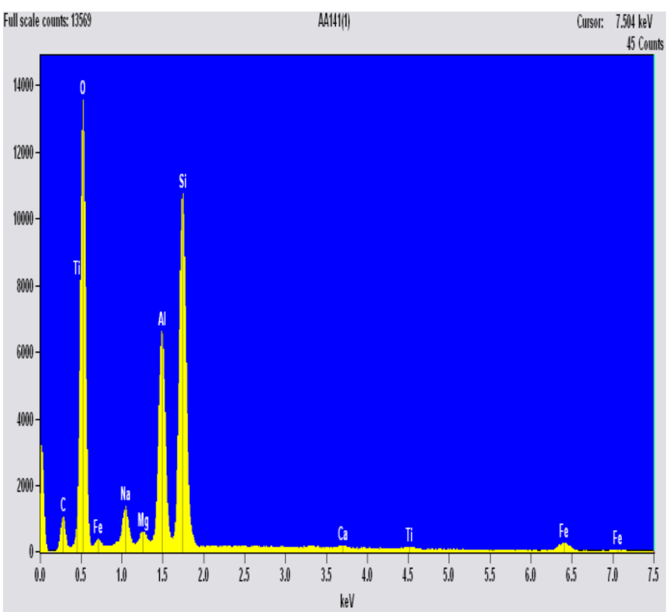

Figure 1: X- Ray Diffractogram of Ntawo Ball Clay

The chemical elements identified from the diffraction pattern were used to determine the components of the Ntawo ball clay.

The rest of the clay samples from the five pits were mixed in a bucket of water. The clay slurry formed was poured into a stack of sieves fitted on a vibrator to allow clay of particle size 45µm to pass through the sieves. . The sieved slurry was left in the open air for a week for the water content to evaporate and form a paste that was strong enoughto be molded in a metal mold of dimensions of 16cm by 4cm by 1cm. The paste was then wet pressed into a mold by hand to reduce the voids and cracks that could develop in the clay sample produced. The wet clay samples produced were put under open shade for slow drying to avoid cracking and reduce its moisture content for a week While drying for the three weeks, the samples were regularly turned over to facilitate uniform drying and prevent them from warping. The dried green clay samples were heated slowly to a temperature of 90^{0}C in an electric furnace for 12 hours. This was to ensure total dryness of ball clay samples and to prevent the swelling or bloating of the sample when fired at high temperatures that would be caused by the expansion of entrapped water.

A set of five samples were fired to a final temperature of 500^{0}C and each set was held at the firing temperature for.varying holding time 20minutes, 30minutes, 40minutes, 50minutes, and 60minutes. This procedure was repeated for final firing temperatures of 600℃, 700℃, 800℃, 900℃ and 1000℃ .The fired samples were allowed to cool in the electric furnace to room temperature and removed from the furnace.

The dimensions of the fired samples were measured and used to determine the cross sectional area of each of the samples. Later the cross sectional areas were used to determine the compressive strength of the fired clay samples The the fired clay samples were subjected to breaking force tests using a compressive strength tester 500SN- 1299417 machine equipped with a compression load cell with a maximum capacity of 25 kN, connected to an appropriate control system.

The values of breaking force of fired Ntawo ball clay samples obtained were as shown in table 1.0 below.The table gives average values of breaking force in kilonewtons.

Holding time / min	Firing temperature/℃					
	500	600	700	800	900	1000
	Average breaking force/kN					
20	6.83	8.16	9.60	11.00	20.48	27.88
30	6.92	8.20	9.64	11.08	20.52	27.92
40	7.08	8.25	9.68	11.12	20.56	27.96
50	7.12	8.30	9.72	11.16	20.56	28.00
60	7.16	8.32	9.76	11.20	20.56	28.00

Table 1: Breaking Force of Clay Samples Fired To Different Firing Temperatures Held At Different Holding Times.

The average breaking force values in table 1 were used to determine the average compressive strength of the fired clay samples.

The statistical parameters, variance, variance within sample and variance within mean were calculated for the compressive strength at each holding time. The relationship between holding time and compressive strength was determined by use F- value and t- distribution statistics. The two were used to test the null hypothesis Ho1 that there was no relationship between the compressive strength and holding time for the different firing temperatures.

III. RESULTS AND DISCUSSIONS

A. Chemical Components of Ntawo Ball Clay

The x-ray diffraction analysis gave the following chemical composition of Ntawo ball clay by percentage weight of various clay compounds

Component	Chemical equation	Percentage weight
Silica	SiO_2	67.20%
Alumina	Al_2O_3	18.20%
Calcium oxide	CaO	0.31%
Iron oxide	Fe_2O_3	2.83%
Potassium oxide	K_2O	0.98%
Magnesium Oxide	MgO	0.36%
Sodium oxide	Na_2O	0.19%
Phosphorous Pentaoxide	P_2O_5	0.06%
Titanium dioxide	TiO_2	1.38%

Table 2: X-ray Diffraction Results of Ntawo Clay Sample Components

TheNtawoball clay has a high silica content and a low alumina content. The values of the components are in close agreement to those found by Nyakiuri(1998)

B. Compressive strength of Ntawo ball Clay

This study was also to find the compressive strength of fired Ntawo ball clay sample for varying holding times of firing of 20minutes, 30minutes,40minutes, 50minutes and 60minutes.This was was for various final firing temperatures of 500^{0}C, 600^{0}C 700^{0}C 800^{0}C 900^{0}C and 1000^{0}C.

The results to show the relationship between compressive strength and holding time when the sample was fire to 500^{0}C. was as shown in table 3 below

Holding time /min	Average compressive strength $\overline{x}$ /MPa	Variance within sample	Variance within mean
20	6.252	0.00972	0.0197
30	6.288	0.00972	0.0109
40	6.436	0.01083	0.0019
50	6.474	0.01083	0.0067
60	6.512	0.00722	0.0143
Grand mean	**6.3924**	**0.009664**	**0.0134**
Over all variance within mean			0.0668
Calculated F- value			**6.9168**

Table3: Compressive Strength for Different Holding Times of Samples Fired to 500°C .

The table 3 includes the average variance within mean and average variance within sample (data). The F-values=6.92 above for the degrees of freedom of 4 and 20 of the data used wasgreater than the expected F-value of 4.43 at the level of significance α=0.01 and F-value of 2.87 at the level of significance α=0.05. The null hypothesis HO1 that the holding time does not affect the compressive strength of the sample fired to 500^{0}C is rejected at the two levels of significance. .The compressive strength is thus significantly affected by the holding time at the two levels of significance.

A graph to show the variation of compressive with holding times was also plotted as shown in figure 2 below.

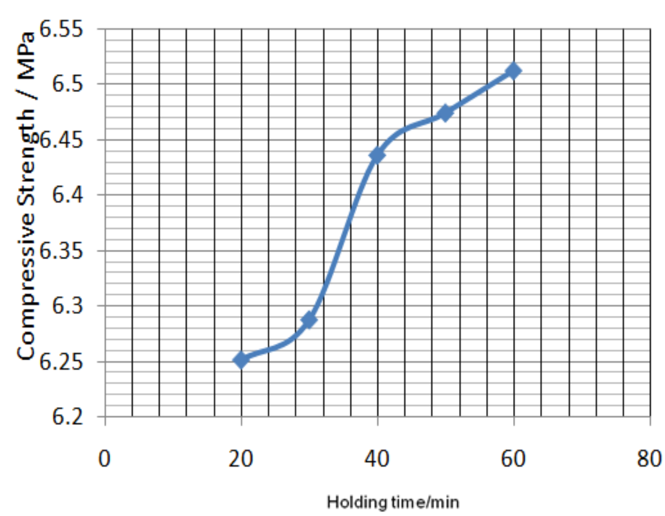

Figure 2: Variation of Compressive Strength with Holding Time for clay samples fired at 500°C

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There was gradual increase in compressive strength with holding time 20 and 30 minutes, followed by sharp increase in strength between 30minutes and 40 minutes, then gradual increase in the compressive strength to 60 minutes. From the results shown on the graph compressive strength increased for holding time. The overall percentage increase in compressive strength obtained between holding time 20 and 60 minutes was by 4.2 percent for samples fired at 500℃.

The results to show the relationship between compressive strength and holding time when the sample was fire to 600^0C. was obtained as shown in table 4 below

Holding time /min	Average compressive strength $\bar{x}$ /MPa	Variance within sample	Variance within mean
20	7.414	0.00648	0.0110
30	7.488	0.00722	0.0009
40	7.526	0.01083	0.0001
50	7.564	0.01083	0.0020
60	7.602	0.00181	0.0069
Grand mean	**7.519**	**0.00743**	**0.0209**
Over all variance within mean			0.1047
Calculated F- value			**14.0918**

Table4: Compressive Strength for Different Holding Times of Samples fired to 600℃ .

The calculated F-value of 14.09 above is greater than the expected F-values of 4.43 at the level of significance α=0.01 and F-value of 2.87 at the level of significance α=0.05. The null hypothesis HO1 that the holding time does not affect the compressive strength of the sample fired to 600^0C is rejected at the two levels of significance. .The compressive strength is thus significantly affected by the holding time at the two levels of significance.

A graph of the compressive strength was drawn against the holding to ime as shown in figure 3 below..

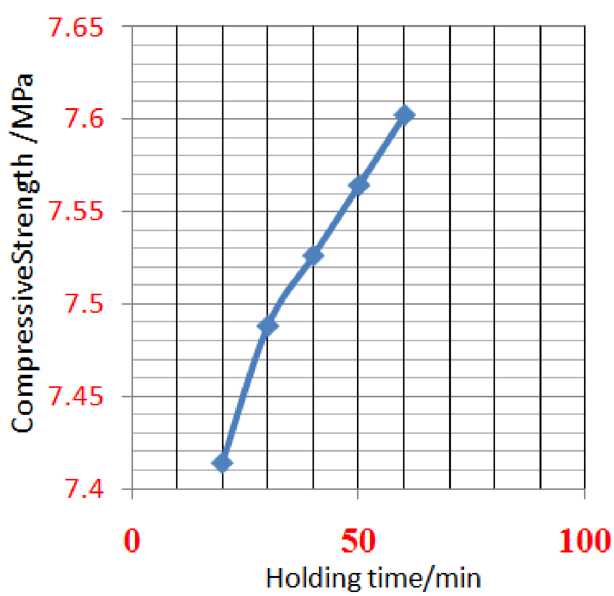

Figure 3: Variation of Compressive Strength with Holding Time for clay samples fired to 600℃

The curve was steep between holding time 20 and 30 minutes and less steep but uniform up to 60minutesThis indicated that average compressive strength increased with holding time. The overall percentage increase in compressive strength between holding time 20minutes and 60 minutes was 2.5 percent for samples fired at 600℃.. This is less than for the sample heated to 500^0C

Another set of results was obtained for samples heated to firing temperature of 700^0C.The result for samples fired to 700℃ obtained was as shown in table 5 below.

Holding time /min	Average compressive strength $\bar{x}$ /MPa	Variance within sample	Variance within mean
20	8.730	0.000000	0.0117
30	8.766	0.006480	0.0207
40	8.802	0.007128	0.0324
50	8.838	0.009720	0.0467
60	8.874	0.006480	0.0635
Grand mean	**8.622**	**0.005962**	**0.0350**
Over all variance within mean			0.1750
Calculated F- value			29.3478

Table5: Compressive Strength for Different Holding Times of Samples Fired to 700℃

The calculated F-value of 29.35 in the table above is much greater than the expected F-value of 4.43 at the level of significance α=0.01 and F-value of 2.87 at the level of significance α=0.05. The null hypothesis HO1 that the holding time does not affect the compressive strength of the sample fired to 700^0C is rejected at the two levels of significance. .The compressive strength is thus significantly affected by the holding time at the two levels of significance when the sample is heated to 700^0C.

A graph of the compressive strength was drawn against the holding time as shown in figure 4 below

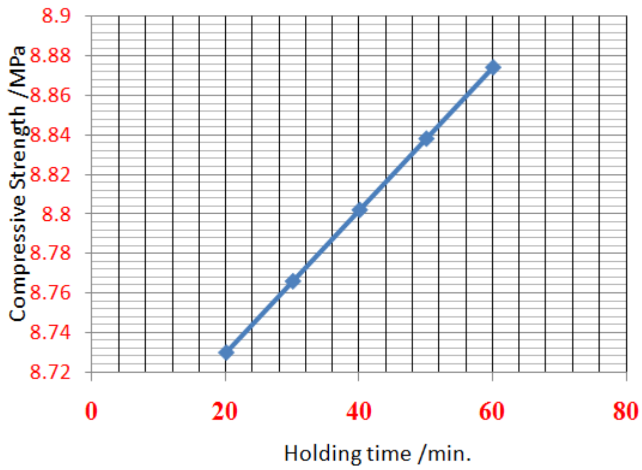

Figure 4:Variation of Compressive Strength with Holding Time for clay samples fired at 700◦C

The graph obtained is a straight line that indicates that the compressive strength is directly proportional to holding time for samples fired to 700^0C.. This shows that an increase in average compressive strength is directly proportional to an increase in holding time. The overall percentage increase in compressive strength obtained between holding time 20minutes and 60 minutes was 1.6 percent for samples fired at 700℃.The percentage increase is less than that for 600^0C..

Another set of results was obtained for samples fired to a temperature of 8000C and the results are as shown table 6 below

Holding time /min	Average compressive strength $\bar{x}$ /MPa	Variance within sample	Variance within mean
20	10.000	0.000000	0.0102
30	10.072	0.009720	0.0008
40	10.108	0.009720	0.0001
50	10.144	0.006480	0.0019
60	10.180	0.000000	0.0063
Grand mean	**10.101**	**0.005184**	**0.0038**
Over all variance within mean			0.0192
Calculated F- value			**3.6999**

Table6: Compressive Strength for different Holding Times of samples fired to 800°C .

The F-value for the set of results above of 3.70 is below the expected F-value of 4.43 at the level of significance α=0.01 but above the expected F-valueof 2.87 at the level of significance α=0.05. This means at the level of significance α=0.01 there is no relationship between the compressive strength and holding time . The relationship is only at the level of significance of α=0.05..

The graph of the values of the compressive strength and holding time was plotted as shown in fu figure 5 below

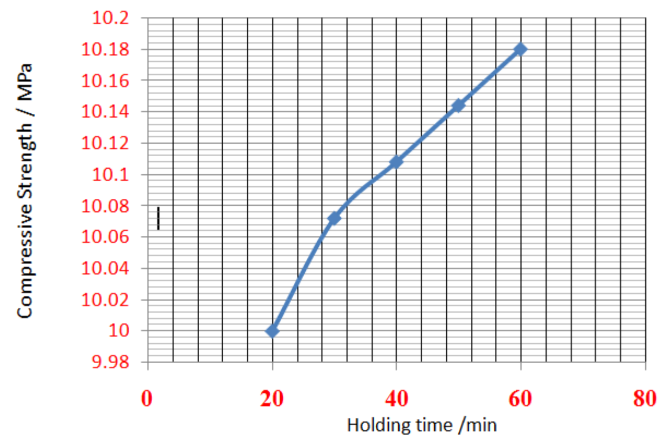

Figure 5:Compressive Strength against Holding Time for clay samples fired at 800◦C

The graph indicated that there was steep increase in average compressive strength with holding time between holding time 20 and 30 minutes, followed by gradual increase in strength between 30 and 60 minutes. The overall percentage increase in compressive strength obtained between holding time 20 and 60 minutes was 1.8 percent for samples fired at 800℃.

The results for samples fired to 900^0C was as shown in table 7 below.

Holding time /min	Average compressive strength $\bar{x}$ /MPa	Variance within sample	Variance within mean
20	18.622	0.009720	0.0025
30	18.658	0.009720	0.0002
40	18.694	0.006480	0.0005
50	18.694	0.006480	0.0005
60	18.694	0.006480	0.0005
Grand mean	**18.6724**	**0.007776**	**0.0008**
Over all variance within mean			0.0041
Calculated F- value			**0.5333**

Table7: Compressive Strength for different Holding Times of Samples fired to 900℃

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The calculated F-value 0.53 in the table above is much smaller than the expected F-values of 4.43 at the level of significance α=0.01 and F-value of 2.87 at the level of significance α=0.05. The null hypothesis HO1 that the holding time does not affect the compressive strength of the sample fired to 700^{0}C cannot be rejected at the two levels of significance. .The compressive strength is thus not affected by the holding time at the two levels of significance when the sample is heated to 900^{0}C.

A graph showing the relationship between compressive strength and holding time is as shown in figure 6 below for clay samples fired at 900℃ .

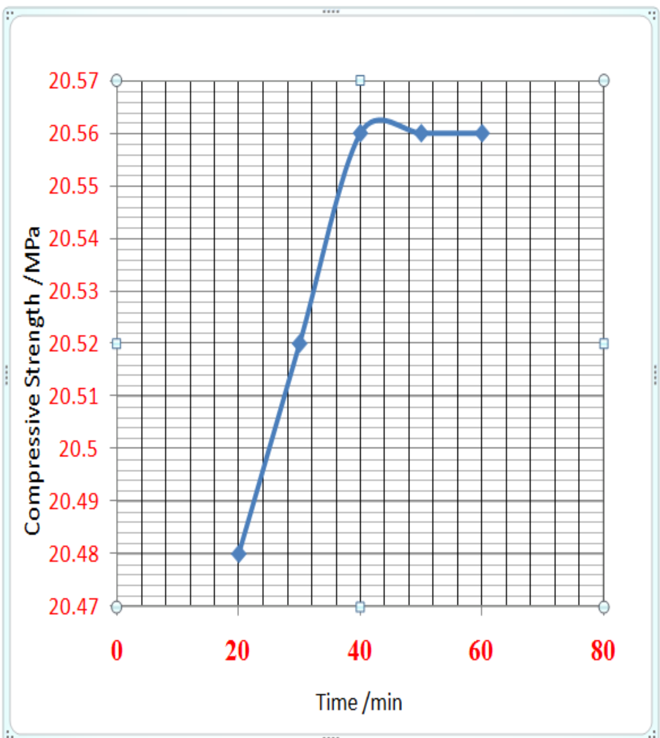

Figure 6: Variation of Compressive Strength with Hold time for Clay fired at 900℃

The graph was a straight line and steep between holding time 20 minutes and 40 minutes, but remained constant between holding time 40 and 60 minutes.

The overall percentage increase in compressive strength obtained between holding time 20 and 60 minutes was 0.4 percent for samples fired at 900℃. This a negligible increase which statistically is not significant.

The last set of results was for samples fired at 1000^{0}C as shown in table 8 below.

Holding time /min	Average compressive strength $\bar{x}$ /MPa	Variance within sample	Variance within mean
20	25.342	0.009720	0.0042
30	25.378	0.009720	0.0008
40	25.414	0.006480	0.0001
50	25.450	0.000000	0.0019
60	25.450	0.000000	0.0019
Grand mean	25.407	0.005184	0.0018
Over all variance within mean			0.0088
Calculated F- value			1.7000

Table8: Values of Compressive Strength for Different Holding Times of Samples fired to 1000℃.

The calculated F-value of 1.7 in the table 7 was smaller than the expected _F-values of 4.43 at the level of significance α=0.01 and F-value of 2.87 at the level of significance α=0.05. The null hypothesis HO1 that the holding time does not affect the compressive strength of the sample fired to 1000^{0}C cannot be rejected at the two levels of significance. .The compressive strength is thus not affected by the holding time at the two levels of significance when the sample is heated to1000^{0}C.

A graph of compressive strength against holding time for samples fired at 1000℃ is as shown in figure 7 below.

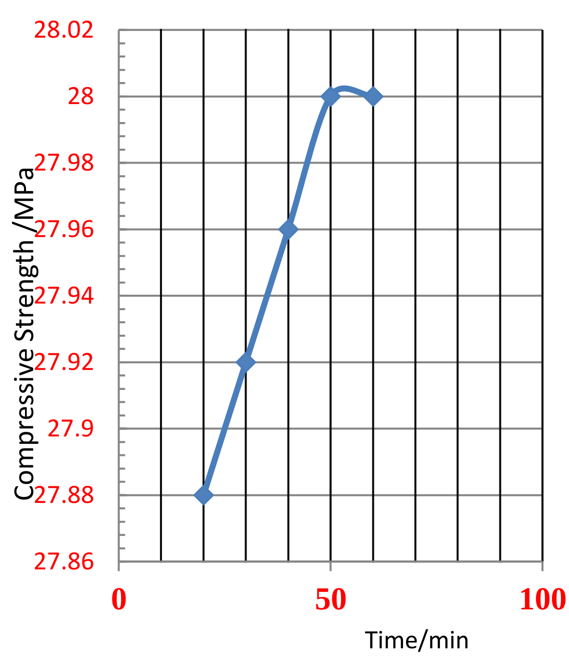

Figure 7:Variation of Compressive Strength with Holding Time for clay fired at 1000℃

The graph was a straight line, less steep between 20 and 50 minutes and then remained constant. The overall percentage increase in compressive strength obtained between holding time 20 and 60 minutes was 0.4 percent for samples fired at 1000℃.This value is of no consequence

Therefore, the results obtained for the five holding times at firing temperature of 1000℃ show that there was no effect of holding time on the strength of the fired ball clay.

IV. CONCLUSIONS

For specific firing temperatures, as the duration of holding time increased from 20 minutes to 60 minutes, fired clay samples showed small increase in compressive strength. At firing temperatures higher than 800℃, compressive strength of the fired clay samples was not depending on holding time. The percentage increase in strength between 4.2 and 0.4% was noted giving a percentage rise of about 1.82% in strength of the samples fired at fixed temperatures, but varying the holding time. Prolonged holding time had no significant effect on the compressive strength of fired clay samples investigated beyond 800℃. Therefore it is unnecessary to keep longer holding times for higher firing temperature. It would simply be wasting time and energy.

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