# INVESTIGATION OF THE COMPRESSIVE STRENGTH OF NTAWO BALL CLAY FIRED TO DIFFERENT TEMPERATURES FOR DIFFERENT HOLDING TIMES

BY

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## DECLARATION

I Isabirye Jeromy Moses do hereby declare that this research report contains my original work and has not been submitted to any academic institution for an academic award.

**Signed:**.................................................................

**Date:** 08/12/2017

# APPROVAL

This research by Isabirye Jeromy Moses was carried out under our supervision and is hereby approved for submission to the Graduate Board and Senate of Kyambogo University with our due approvals.

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# DEDICATION

I dedicate this work to my father Mr. Kabi George for his support that has led to my academic success up to this level.

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## ABSTRACT

This study focused on ball clay from Ntawo deposit from Mukono District, in central Uganda with respect to its compressive strength. The chemical compounds by percentage weight were determined using RIX 3000 Spectrometer machine. The compressive force was determined by 500SN-1299417 machine. One hundred and fifty samples of Ntawo ball clay fired from 500°C to 1000°C at intervals of 100°C were used to find out how holding time affected compressive strength for each set firing temperature. The same number of samples were used to investigate how the compressive strength of fired Ntawo ball clay to find out how they were affected by firing temperature for each set holding time between 20 minutes and 60 minutes at intervals of 10 minutes.

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 fluxes. The compressive strength decreased from 4.2% to 0.4% withholding time for firing between 500°C and 800°C. The compressive strength variation was negligible, less than 1% for temperatures beyond 900°C to 1000°C. The highest compressive strength was 25.45 MPa, obtained by firing to a temperature of 1000°C. In all cases compressive strength increased by $298 \pm 7$ % for all firing temperatures at each of holding times of 20, 30, 40, 50 and 60 minutes respectively. The compressive strength of ball clay beyond 800°C does not need longer holding time.

It was further established that the compressive strength of fired clay depended on holding time when fired to various firing temperatures and the compressive strength of fired clay depended on firing temperature when the firing is held for holding time were tested at $\propto = 0.01$ and $\propto = 0.05$ levels of significance. The compressive strength for ball clay fired above 1000°C and holding time exceeding 60 minutes is worth investigation in order to find out whether any change in the mineral compounds of ball clay affect the above results.

# CHAPTER ONE: INTRODUCTION

## 1.1Background of the Study

The word "clay" is generally understood to refer to soil composed of small mineral particles that exhibit plastic behavior when containing a moderate amount of water. The plasticity behavior allows clay to be easily molded. Minerals in this context refer to finely ground chemical compounds. Clay has those minerals that exhibit plasticity. There are several groups of chemical compounds that make up clay minerals. The finest sizes of the grain, typically smaller than 0.075 $\mu m$, are referred to as clay sizes only for purposes of definition (Cater etal, 2007).

The formation of clay from rock is a continuous process, taking place daily everywhere in the world. Clay deposits typically form over long periods of time as a result of physical and chemical weathering of rocks, usually from silicate- bearing rocks, by low concentrations of carbonic acid and other naturally occurring acids. These acidic solvents, attack and weather rock after leaching through upper weathered layers. The decomposition of igneous rocks such as granite, which were themselves formed by solidification of molten materials from the interior of the earth get decomposed to form clay. Granite is composed of approximately equal proportions of mica, quartz and feldspar. The feldspar is the least stable of the minerals when exposed to the action of water and air. The clay after formation could also be transported by water and re-deposited at some distance from its place of origin. The clay then becomes sedimentary or secondary clay.

Sedimentary clays are of fine particle size and remain in suspension whilst they are being transported. The large particle clamp are likely to be lost en route and only the very fine particles would be carried to the final deposit. Sedimentary clays are thus usually of finer particle size than residual clays. During their transportation by water erosion, sedimentary clays become contaminated with other material and the variety of non-clay material found in them is greater than in residual clays. These non clay materials are usually fine and are much more difficult to separate from the clay mineral than non clay materials found in residual clays. Deposits of ball clay are most likely to be found in the plains and valleys or along rivers. In Uganda the most pronounced areas where ball clay is found are areas of Kajjansi in Wakiso District, Kitiko, Masooli, Ntawo deposits near Kampala, Kamonkoli in Budaka (Nasenyi, Bugungu near Jinja in Buikwe District. Other areas which are not most pronounced include Butende in Iganga District, Buteraniro in Mbarara District, Kasukengo in Masaka District, Malawa in Tororo District, Butema in Hoima District, Agu (wetland) in Ngora District and Kangole in Moroto District. The deposits are scattered with clays deposited under lacustrine and alluvial environmental conditions. All the mentioned deposits belong to the sedimentary group (Kaliisa, 1983).

Clay is widely used for various purposes. In most countries in the world, ball clays are used in producing every day articles of high strength. The articles of high strength include: wall tiles, roof tiles, floor tiles, wash basins, toilet bowls, plates, cups and saucers, linoleum, acoustic ceiling tiles, insulated electrical cables, pale coloured bricks, drainage pipes, wind screen wipers, spark plugs, engine mountings, hoses and fertilizers. In Uganda, ball clay is used for making pottery, both utilitarian and decorative, and construction products such as bricks, wall and floor tiles of low strength. It is used in many industrial processes such as paper making, cement production and chemical filtering. Clay in wet form is also used, where natural seals are needed such as in the cores of dams or as a barrier in landfills against toxic seepage (lining the land fill preferably in combination with geotextiles) since it does not permit passage of fluid.

Fired clay exhibits high melting point, high mechanical strength, porosity and water absorption when fired at high temperatures. Fired clay is resistant to chemical reactions, in particular situation in which it is used. The fired clays have dense structure with low permeability at high temperature. It displays a high strength, lower porosity and water absorption. Other properties for fired ball clay include: clay and its mixture, molding of the clay and shrinkage test. The clay and its mixture are very important because the quality of clay and water to be added for proper plasticity and consistency for moulding clay articles can be found. Water is one of the essential requirements in production of clay articles. Pure water is perfect for clay article production since it does not change the properties of prepared materials.

Different clays are blended to obtain clay products of properties ideal for each ceramic application. The technical expertise is to: identify the properties of each ball clay seam as they change across the deposit, understand how the clay will behave when formed and fired as part of a ceramic body, blend together different seams to produce a large number of consistent products suitable for each individual customer's particular manufacturing process.

In Uganda, most people utilize the enormous deposit of clay. It is mostly used by local people for pottery purposes. Nyakairu etal., (2001) examined the mineralogical, geo chemical and sediment logical characteristics of clay deposits from central Uganda and their applications. The deposits contained silica and alumina minerals together with iron and fluxes. Harris, (1946) and Kagobya, (1950) studied the clay deposit at Mukono –Ntawo. It was reported that Ntawo clay exhibited marked shrinkage and cracked on firing and the quality of clay product was inferior when it was evaluated for pottery production. McGill, (1965) studied the nature and distribution of clays from several occurrences around Kampala and determined their plasticity with a view to establish a fine ceramics industry. Nyakairu and Kaawa, (1998) made a study of some clay samples from various deposits in Uganda and indicated that they are medium – quality Kaolinic- illitic clays. There is ample demand for quality clay products of high strength and thus, the present study evaluates strength of clay products based on firing temperatures and holding times. This will help to give a better understanding of the ball clay material, as well as of their compressive strength when firing temperature and holding time are being varied.

## 1.2 Statement of the Problem

Both high and low compressive strength clay products can be manufactured using ball clay as a raw material. Fewer studies have been done on the mechanical properties of fired ball clay as compared to studies which have been done on the mineral composition of ball clay from Ntawo deposit. There is scanty information documented on mechanical properties of fired clay and on use of local clays in the manufacture of fired ball clay products of high strength in Uganda.

## 1.3 Purpose of the Study

The purpose of the study was to establish how the compressive strength of Ntawo ball clay is affected by firing it to different temperatures and by varying the holding time of firing.

## 1.4 Objectives of the Study

In order to achieve the above aim, the following objectives were set to:

(i) Find out the major chemical compounds which constitute the ball clay from Ntawo deposit.

(ii) Measure compressive strength of fired ball clay sample for different holding time fired to different temperatures.

(iii)Determine the compressive strength of fired ball clay samples fired to different firing temperature held for different holding time.

(iv)Establish the relationship between compressive strength of fired Ntawo ball clay and holding time.

(v) Establish the relationship between compressive strength of fired Ntawo ball clay and firing temperature.

## 1.5 Research Hypotheses:

To establish the relationships between compressive strength and holding time, and also firing temperature, the following null hypotheses were stated to be tested at $\alpha = 0.01$ and $\alpha = 0.05$.

(i) $H_{01}$-There is no relationship between compressive strength and holding time when ball clay samples are fired to the set firing temperatures.

(ii) $H_{02}$-There is no relationship between compressive strength and firing temperature when ball clay samples are fired to the set holding times.

## 1.6 Scope of the Study

This study was focused on ball clay from Ntawo deposit Mukono District in central Uganda. The deposit is located in Ntawo a distance of twenty five (25) kilometers from Kampala along Jinja Road. The ball clay in Ntawo is known to be composed of kaolinite sedimentary clay that consists of kaolinite, mica, quartz and carbonaceous material such as lignite. The ball clay is fine grained and of intermediate plasticity which aid in its rheological stability during shaping process.

The degree of firing was a function of holding time of heating and firing temperature at which clay was heated. The firing temperature and holding time were among the factors which affect physical and mechanical properties of fired clay products. Other factors that include the rate of heating and firing schedule were kept constant. The samples were of the same size and shape of the particles. The variables which were considered in the study included: the holding time, firing temperature and compressive strength of the clay samples manually molded. Compressive strength was regarded as the dependent variable, while holding time and firing temperature of ball clay samples were considered as the independent variables.

Six firing temperatures were selected in the range of 500°C to 1000°C at intervals of 100°C for five holding times within the range of twenty (20) minutes to sixty ( 60) minutes. This was to make sure all organic material in the samples got completely burnt. The holding time was controlled at most for one hour.

### 1.7 Significance of the Study

The Ntawo ball clay contained silica (67.2%), alumina (18.2%), $Fe_2O_3$ (2.83%) and fluxing components CaO (0.31%), $K_2O$ (0.98%), MgO (0.36%) and $Na_2O$(0.19%). The weight percentage of the mineral compounds of Ntawo ball clay for the clay samples were within the range of values investigated by Duggal, (2000) and Nyakairu etal. (2001). The strength of fired clay samples increased with increase in firing temperature between 500°C and 1000°C for fixed holding times. The percentage increase in compressive strength was 305.3%, 303.6%, 294.9%, 293.1% and 290.8% for holding times 20, 30, 40, 50 and 60 minutes respectively. The null hypothesis $H_{02}$ set was that there is no relationship between compressive strength and firing temperature at fixed holding time. The relationship between compressive strength and firing temperature of samples fired at fixed holding time was significant. The highest compressive strength was 25.45 MPa at firing temperature 1000°C for holding times between 40 minutes and 60 minutes.

The compressive strength significantly increased with holding time from 500°C to 800°C. The percentage increase in compressive strength was 4.2%, 2.5%, 1.6%, 1.8% at 500°C, 600°C, 700°C and 800°C respectively. At firing temperatures higher than 800°C , compressive strength of the fired clay samples was not depending on holding time. The relationship between compressive strength and holding time of the samples was not significant. The percentage increase in compressive strength was 0.4% at 900°C and 1000°C.

# CHAPTER TWO: REVIEW OF RELATED LITERATURE

## 2.1 Introduction

The review of related literature for this study was to provide the theoretical back ground of the specific objectives in line with knowledge of other researchers about the study. It was to explain the theories about nature of clay used for production of fired clay products, mineral composition of clay, Processes of manufacture of fired clay and factors that influence compressive strength of fired clay. Also the chapter focused on type of kiln best for firing clay products to attain high strength and effect of kiln temperature on the strength of clay product.

## 2.2 Nature of clay used for manufacturing of Fired clay Products

Clay is one of the most abundant natural mineral on earth. It is the basic material in the manufacture of clay products. The quality of fired clay product depends mainly on mineral compounds, degree of firing and difference in manufacturing method. The plasticity of clay is a result of the enormous amount of surface area inherent in particle size and shape. Clay when mixed with water, easily molded or formed into the desired shapes, gain sufficient tensile strength to maintain the shapes after moulds are removed. The particles of clay are highly fused at high temperatures. So it is important to know the types and mineralogical composition of clay which determines the quality of the end product (Worrall, 1986).

It is from the silicate of feldspar in the decomposition or kaolinisation of feldspar in the presence of air and water over long periods of time that the kaolinitic clays have been formed. All the potash and part of the silica in felspar have been dissolved away, the residue combining with water to give the clay mineral called kaolinite. The clay once formed from the parent rock may be deposited at its place of origin or may be transported by water and re-deposited at some distance from its place of origin. In the first case clay is known as residual or primary clay and in the second case as sedimentary or secondary clay.

According to McGill (1965), the clays in central Uganda, can generally be classified as sedimentary and transported alluvial clays. Clays derived from gneissic and granitoid rocks of the basement are leached and enriched in quartz. These clays are thought to have formed by leaching of the decomposed bed rock and are normally separated from the bedrock by a layer of large quartzite pebbles (Harris, 1946). The clays occur as surficial layers with a general thickness varying between 2 and 5 meters (Kallisa, 1983). The main features of clays are their low wet-to-dry shrinkage, refractory nature and an extremely high plasticity, which is attributed to high kaolinite content. Due to their high plasticity, these clays can be classified into surface clays, shale, fire clay and ball clays. The types of clay are found in different deposits with varying characteristics. The surface clay is found close to the earth's surface. It is the most accessible and simply mined. So it is the least expensive clay soil with high oxide content, ranging from 10 to 25 percent. The shale is a metamorphic form of clay hardened and layered under natural geologic conditions. It is very dense and harder to remove from the ground than other clays and as a result is more costly to be mined. Like surface clay, shale contains a relatively high percentage of oxide fluxes. Fire clay is found at greater depths than either surface clay or shale. It generally has fewer impurities, more uniform chemical and physical properties and only 2 to 10 percent oxides. The lower percentage of oxide fluxes gives fire clay a much higher softening point than surface clay and shale, and the ability to withstand very high temperatures. This refractory quality makes fire clay best suited to produce brick for furnaces, fire places, flue liners, ovens and chimney stacks.

Ball clays are highly plastic fine particle clays grouped under sedimentary clays. They are transported over a relatively short distance from the parent rock from which the clay is formed. They are characterized by their fine grain size and often high content of organic material. Ball clays vary widely in plasticity, particle size, raw colour and drying properties. It is difficult to compare them by quantitative tests. This is because pure samples are difficult to mix, form and crack during drying. Ball clays are easily slaked in water when dry. The fine particle sizes are impermeable to the passage of water. Ball clay is not a clay mineral in itself, but contains other minerals primarily kaolinite. Mica and quartz are also normally present in substantial amounts (Campbell and James, 2003).

### 2.3 Mineralogical composition of clays

Mineralogical content of clay is one of the most important factors which determine the quality of burnt clay products. Clays are mainly composed of silica and alumina with varying amounts of metallic oxides and other impurities. Metallic oxides act as fluxes to promote fusion of clay particles at low temperatures, influence the range of temperatures in which the material vitrifies and give burnt clay product the necessary strength. The varying amounts of iron, calcium and magnesium oxides also influence the colour of fired clay (Worrall, 1986).

According to Nyakairu et al., (1998), the chemical composition is mostly used to identify the raw material characteristics and the source of raw clay. The determination of the mineralogical composition of clay soil allows the identification of possible deficiencies that may occur during production of clay products, like the presence of organic matter, lime nodules, harmful soluble salts and other impurities that might influence the durability of the clay products.

The mineralogical composition of ball clay may vary from one pit to another when we go deeper during excavation. Therefore, it requires continuous testing of the raw materials or clay mineralogical compositions in the laboratory during production. Generally, the mineralogical composition of clay material was done by Duggal, (2000) and came up with results shown in Table 2.1.

**Table 2.1:** Mineral Composition of clay (**Source: Duggal, 2000**)

| Minerals | Chemical formula | Weight % |
|---|---|---|
| Silica | $SiO_2$ | 50 – 70 |
| Alumina | $Al_2O_3$ | 18 – 30 |
| Lime | $CaO$ | 10 |
| Magnesia | $MgO$ | < 1 |
| Ferric Oxide | $Fe_2O_3$ | < 7 |
| Alkalis | $NaOH$ | < 10 |
| Carbon dioxide | $CO_2$ | Very small percentage. |
| Sulphur trioxide | $SO_3$ | |
| Water | $H_2O$ | |

The range of results in Table 2.1 was based on clays from different deposits that can be used as a guide to estimate mineral composition.

Each chemical compound in the clay has property for the clay product. Silica enables the clay product to retain its shape. It prevents shrinkage and warping of the clay product. Excess of silica makes the clay body brittle and weak on burning. Alumina determines the plasticity in the clay which is an important component for high strength clay products. Alumina absorbs water and renders the clay plastic. If alumina is present in excess of the specified quality, it produces cracks in the clay body on drying. The high percentage of hydrated alumina silicates in the clay is needed during clay product forming and high firing shrinkage. However, to compensate for the high firing shrinkage due to high alumina content, free silica in the form of sand or silt is added to clay. Alkalis for example soluble salts of potassium when present in quantities greater than 1% produces higher fired body densities.

Iron oxide representing 3% to 8% within a clay soil will produce a reddish clay product when fired to temperature between 900°C and 1000°C. Iron Oxide improves impermeability and durability of the fired clay. It also gives strength and hardness (Duggal, 2000). Higher alkali oxides of greater than 3% ($MgO$,$K_2O$,$Na_2O$ e.t.c) contents results in low temperature vitrification and increases the firing shrinkage. Lime causes silica in clay to melt on burning and thus helps to bind it. Excess of lime causes the fired clay body to melt and clay body loses its shape. Magnesia affects colour and makes the fired clay or pottery product yellow.

Harris (1946) investigated fired clay and came up with the following findings on the composition of clay and its technical importance. The chemical composition of clays was silica, alumina, iron oxide, magnesia, lime and alkalis. Variation in chemical compounds than the required amount of any of these constituents may make a substantial difference in clay product quality especially in short cycle firing systems. Nyakairu et al., (2001), carried out a study on chemical composition of kaolin and clay from central Uganda. The findings are as shown in Table 2.2.

**Table 2.2:** Chemical composition of kaolin and clay from central Uganda ( **Source: Nyakairu et al., (2001)**

| Compound | Chemical formula | Weight percentage Clay | Weight percentage Kaolin |
|---|---|---|---|
| Silica | $SiO_2$ | 67.18 | 46.40 |
| Alumina | $Al_2O_3$ | 18.20 | 38.70 |
| Calcium Oxide | CaO | 0.306 | 0.090 |
| Iron Oxide | $Fe_2O_3$ | 2.832 | 0.791 |
| Potassium Oxide | $K_2O$ | 0.975 | 0.214 |
| Magnesium oxide | MgO | 0.363 | 0.020 |
| Sodium Oxide | NaO | 0.185 | 0.040 |
| Titanium dioxide | $TiO_2$ | 1.380 | 0.039 |
| Phosphorus oxide | $P_2O_5$ | 0.049 | 0.043 |
| Loss on ignitition | LOI | 8.430 | 13.80 |

The researcher found out that the most components were $Al_2O_3$ and $SiO_2$, with other mineral oxides of $Fe_2O_3$, $TiO_2$, MgO, $K_2O$, MnO, CaO and $P_2O_5$ which have a decisive influence on the strength of the clay product.

According to Punmia etal. (2003), the normal chemical properties of clay show that the weight percentages are : silica ( not less than 60%), alumina (not less than 15%), Ferric oxide (not less than 3%), lime (not less than 3%), Magnesia (not less than3%), alkalis (not less than 4%), total water solutes (not less than 1%) and loss of Ignition (not less than 7%).

### 2.4 Processes of manufacture of fired clay

According to Clews (1977), the common manufacturing process has six stages: mining and storage of raw materials, preparing raw materials, forming the clay products, drying, firing and cooling. The clay is mined from a quarry and transported to the work place or processing plant. At work place the following processes are performed: drying with air, blending, mixing and crushing of the raw materials. Crushing of the raw materials is done to reduce the over sized particles of the raw materials. The crushed particles are then sieved to come up with particle size required of the raw material. The fineness to the clay influences not only the external appearance of the finished clay product but also physical characteristic such as compressive strength, porosity and water absorption.

According to Clews (1934) tempering is the first step in the forming processes that produces homogeneous plastic clay mass. Usually, this is achieved by adding water to the clay until it becomes plastic which makes it easy to be shaped to the desired sizes. There are three principal processes for forming clay products, namely: stiff mud press, soft mud press and dry press. The soft mud press is suitable for clays having high plastic index. The amount of water used for mixing the clay is in the range of 20 to 30 percent. For the stiff mud press, the amount of water used for mixing clay is in the range of 10 to 15 percent. During the process of mixing, the tempered clay goes through a de- airing chamber that preserves a vacuum. De-airing removes air bubbles, giving the clay increased workability and plasticity, resulting in greater strength. The dry press process is suited for clays of very low plasticity. The clay is mixed with a minimum amount of water up to 10 percent, and then pressed into moulds under pressures. The green clay body product contains large percentage of moisture, depending on the forming method. Before the firing process begins, most of this moisture is evaporated in dryer chambers or they can be dried in open shed where there is sufficient place for drying (Cater etal. 2007).

According to American Standard for Testing and Material (ASTM C 43- 98a), firing is the process of heating material to elevated temperatures. The temperatures are usually in excess of 930°C. The extent of firing is a function of both time and temperature. The firing develops the inter- particulate bond, strengths, pore structure and colour of the clay product. The firing temperature has to be sufficient to produce a fired clay product which is durable. The bond may result from fusion or melting of one or more constituents of the composition or surface of the particles. Other thermal mechanisms such as inter particle reaction may be responsible for the bond. The higher the heat treatment, the greater the extent of bonding and consequently the greater the developed strength and the lower the resulting porosity. The bond developed has to be sufficient to provide the specified strength, porosity and durability for any particular clay product (Ahamadi etal. 2008).

According to Sameer and Maithel, (2003) firing temperature management for clay products in the kiln, affect the quality of the clay product. The clay product can either be under or over burnt. Both over and under burning of clay products are as a result of poor firing temperature management. Controlling the degree of firing in recent method of production is done by using a thermocouple in the kiln. But in the traditional methods of clay product manufacturing the degree of firing is decided by the technicians who follow the firing process through the inspection hole on the kiln.

Kang and Joong (2005) investigated the firing of clay under the following processes: dehydration, oxidation and vitrification. Dehydration is the stage of firing, were water is removed from the clay mass. Oxidation is burning of carbonaceous material in the clay mass.

During vitrification stage, the mixed and shaped clay material is irreversibly changed into a hard, glassy and durable material called fired clay product. A clay particle does not develop its full strength until it has vitrified. The vitrification range starts at about 900°C and extends up to the highest temperature clay can be held without melting. Depending on the kind of clay minerals and the impurities present, the upper temperature limit of the vitrification ranges may vary from 1000°C to 1500°C.

Throughout the process of firing of clay, a series of physical changes takes place. The important physical changes during clay body firing are shown in Table 2.4 (Goffer, 2007).

**Table 2.3:** Physical changes caused by wet clays Heated at different Temperature Ranges ( **Source :Goffer, 2007)**

| **Heating temperature / °C** | **Physical changes** |
|---|---|
| Room temperature – 100 | Loss hygroscopic water. |
| 100 - 500 | Loss of water of plasticity. Burn out of carbonaceous water. |
| 500 - 600 | Dehydration (loss of chemically combined water and modification of clay structure). |
| 600 - 900 | Break down of clay structure and incipient vitrification. |
| 900 - 1500 | Sintering and vitrification leading to development of fired strength. |
| Above 1500 | Melting. |

At a temperature of 100°C, moisture in the clay mass is driven off. The moisture in the clay mass is driven off until clay products have attained 150°C. This part of the firing is called the water smoking period. Clays contain carbonaceous matter in some degree. When heated, the organic material usually dissociates at temperature from 350°C upwards, into volatile hydrocarbons and a more carbonaceous residue. Whether the volatile matter burns or not is dependent not only on the presence of sufficient oxygen for combustion but also on whether the temperature is sufficiently high for the gases to ignite. Carbon monoxide for example requires a temperature of 450°C before it will burn (William Ryan, 1978).

At about 450°C to 500°C the clay mineral starts to decompose. Hydroxyl groups present in the clay structure are driven off as water in this reaction, which is known as dehydroxylation.

$$\underset{\text{Kaolinite}}{Al_2O_3.2SiO_2.2H_2O} \rightarrow \underset{\text{metakaolin}}{Al_2O_3.2SiO_2} + \underset{\text{steam}}{2H_2O}$$

This reaction results in a weakening of the body, since the metakaolin formed has no binding power like that of the clay. Once the clay structure has been broken down due to this reaction its ability to become plastic when mixed with water is lost.

After clay body has been heated to about 600°C , decomposition takes place and its plasticity cannot hold longer. A clay test piece fired in this way will not break down and recombine with water when immersed and its resistance to such attack increases as the firing temperature is increased. Traces of combined water may continue to be given off until 850°C to 900°C is reached but for the most part the water is driven off at 650°C (Clews and Green, 1934).

The huge volumes of water that have to be driven off from a kiln chamber of clay articles should be appreciated. The rate of rise of temperature during the water smoking periods must be relatively slow. If it were not so, the rapid evolution of steam might disrupt the clay products and also might exclude the air or oxygen required to effect the oxidizing reactions that are normally needed by clay products in the firing range 400°C to 800°C.

There being no oxidizable material in clay, the oxidizing reactions are referable entirely to the organic or carbonaceous matter present, the sulphur compounds, such as the sulphides of iron and the ferrous compounds, which are oxidized to the ferric condition. The oxidizing period in the firing of clay articles, although not very definite, may be considered as extending to 950°C. It thus overlaps at the lower end of the range with the period when the clay is decomposing and the combined water is being evolved. At the upper end it may extend into the vitrification period. It is important that the kiln atmosphere should be strongly oxidizing to enable oxidation of carbon to carbon dioxide to take place as early as possible. Where iron is present in the body, ineffective removal of carbon can be serious, leading to faults known as "black cores" or "red hearts". It is usually desirable that iron compounds should be oxidized to the ferric state, the colour of which is bleached by certain oxides present in the body such as calcium oxide and alumina.

The oxidation of the more carbonaceous solid residue and its elimination as gaseous carbon monoxide or dioxide is also governed by several conditions: Firstly, sufficient oxygen should be present in the atmosphere immediately surrounding the clay. This may easily not be the case if the clay is in the process of decomposing or even if adjacent clay goods are decomposing and by the evolution of water, reducing the proportion of oxygen adjacent to the ware in question. Secondly, the readiness with which the available oxygen can penetrate through the system of pores to the carbonaceous matter beneath the surface is important. This depends on the permeability of the ware. During the stage of firing from 350°C to 950°C, the permeability of clay goods may be very low. Consequently long periods may be necessary for the complete removal of carbonaceous matter from clay goods, especially for clay articles of several millimeters thick. Thirdly, the effect of temperature, acts in two opposite ways. The higher the temperature, the more readily any particular form of carbon will react with oxygen. If, however, the oxygen has poor access to the carbon and the products of combustion cannot readily escape from the site within the clay mass where they are formed, then oxidation cannot proceed and the effect of rising temperature will be to convert the solid residue into a form of carbon more closely resembling graphite which is not so readily oxidizable. Under some conditions it may happen that the carbonaceous residue is still incompletely oxidized in the centre of a clay mass by the time the surface has become vitrified. This accounts for black core in fired clay products like clay bricks, tiles and other clay ware. Under normal conditions it is usually inadvisable to attempt to burn out carbon at temperature above 950°C (Clews and Green, 1934).

The oxidation of sulphur compounds during firing of clay goods is perhaps best exemplified by considering the behavior of iron pyrites, $FeS_2$. If the kiln atmosphere is neutral or only slightly oxidizing, iron pyrites dissociates at about 450°C into iron sulphide and sulphur which is distilled off probably subsequently burnt to sulphur dioxide and sulphur trioxide. In the presence of sufficient air the iron sulphide, FeS, may be converted at slightly higher temperatures into ferric oxide and oxides of sulphur or into ferrous and ferric sulphate.

Kiln atmosphere, is important in affecting the reactions which occur during firing. Normally an oxidizing atmosphere is required so that organic matter can be burnt out and iron kept in the ferric state. There are, however, some instances where a reducing atmosphere is needed. A reducing atmosphere is usually obtained by allowing insufficient air into the kiln to enable all the carbon from the fuel to be converted to carbon dioxide. Some carbon is converted only to carbon monoxide CO, a reducing gas. By extracting oxygen from other materials, carbon monoxide can become oxidized to carbon dioxide, whilst reducing the material providing the oxygen. Other reducing gases like hydrogen can be introduced into kilns for special purposes. Instances where a reducing atmosphere is required at least for part of fire are: in the glost, firing of hard porcelain where the small amount of impurity iron present is purposely converted to the ferrous state, giving the fired porcelain a blue tint which is preferred to the yellow colour obtained from ferric iron. This is achieved by using a reducing atmosphere in the kiln from about 1000°C to the top temperature of about 1400°C. Heavy clay products like engineering bricks have their firing completed in a reducing atmosphere. This is normally done by converting the iron to the ferrous form and producing the blue colour. Certain colours depend on obtaining a low oxidation state of the metal involved and require reducing conditions for example cupric oxide, which gives green colours under oxidizing conditions (Garve, 1949).

Under the conditions usually existing in kilns the iron sulphates dissociate into ferric oxide again around 700°C. The presence of carbon will assist the decomposition of sulphates. The chemical reactions possible are numerous, most of them are reversible and the course followed is largely dependent on the temperature and composition of the atmosphere surrounding the clay. For temperatures above 700°C and low proportions of sulphur trioxide in the kiln atmosphere leads to the formation of ferric oxide. Lower temperatures around 500°C to 650°C and high proportions of sulphur dioxide and trioxide in the kiln atmosphere favour the formation of ferric sulphate. However, all the reactions that involve combination with a constituent of the gases surrounding the clay are hindered if the permeability of the clay mass is low (Clews and Green, 1934).

Vitrification of ceramic products as glass formation may start at any temperature above 900°C and extends up to the highest temperature. The clay body can withstand without serious distortion, depending on the composition of the body. It is the period in which increased compressive strength develops. At the lower end of vitrification period, increased strength may be considered to be caused by sintering, a process in which closely adjacent particles of solid become united through the constituent ions of each escaping into the other and eventually re-ordering themselves in crystalline form and making a solid link between the two. At the same time, strength is being increased as new crystalline compounds are produced from the residue left of the clay at 550°C. Alumina crystallizes about 950°C and mullite at a somewhat higher temperature. Particles of fluxing material react with particles of other body constituents with which they are in contact to form liquid and the proportion of liquid increases as the temperature increases. The body contracts due to the formation of liquid (firing shrinkage) and the porosity is reduced. If vitrification is allowed to go too far, so much liquid may be formed that the body may lose shape. New materials may crystallize from the liquid as the temperature is increased (Garve, 1949).

On cooling the liquid solidifies and cements together the unmelted particle and crystals formed during heating, give strength to the fired body. Reactions can occur on heating without formation of a liquid. Such solid phase reactions are the first step towards densification even in the cases where solid phase reaction is followed by the formation of a liquid. In other cases densification may take place, resulting in a dense, hard product without the formation of a liquid at all. Such a case is that of pure alumina ceramics, which are fired at about 1800°C or more than 200°C below the melting temperature. This results into mutual diffusion of atoms between touching particles which is known as sintering. As a result of sintering, particles become fewer and larger pores are eliminated. Strength in the fired clay body under these cases is not due to glass formation, but due to inter locking of crystals.

The process of densification depends on contact between particles, the more particles – particle contacts in unfired material, the more readily the reactions leading to densification will take place. Thus clay has fine particle sizes, which implies that the greater its surface area per unit weight, the more readily it will fire. Atoms at the surface of the material have a higher energy than those in the bulk, since they are combined to other atoms only on one side and so are in a state of unbalance. The greater the surface area of the powder, the greater the ratio of surface atoms to atoms in the bulk, hence greater the surface energy of the powder. The surface energy is reduced due to decrease in the surface area after firing the clay body. It is this reduction in energy which is the "driving force" behind sintering (William Ryan, 1978).

Clay, unlike metal, softens slowly and melts or vitrifies gradually when subjected to rising temperatures. Incipient fusion occurs when the clay particles become sufficiently soft to stick together in a mass when cooled. Table 2.4 shows the changes and events that occur in the kiln, when a clay product is fired at different firing temperatures.

**Table 2. 4:** The effect of Kiln Temperature on Clay Product (**Source :www.potterymaking.org**)

| TEMPERATURE /°C | COLOR | EVENT |
|---|---|---|
| 1400 | Brilliant white | End of porcelain |
| 1300 | White | End of stoneware range |
| | Yellow-white | |
| 1200 | | End of earthenware (red clay) range. |
| | Yellow | Between 1100°C-1200°C , mullite and cristobalite (two types of silica) form when clay starts converting to glass. Clay and ceramic particles start to melt together and form crystals. These changes make the material shrink as it becomes denser. Soaking (holding the end temperature) increases the amount of fused matter and the amount of chemical action between the fluxes and the more refractory materials. |
| 1100 | Yellow – orange | |
| | Orange | |
| 1000 | | |
| | Red – orange | |
| 900 | | |
| | Cherry red | Between 800°C-900°C sintering begins. This is the stage where clay particles begin to cement themselves together to create a hard material called bisque. |
| 800 | | |

| | | |
|---|---|---|
| | Dull red | Between 300-800°C ,the temperature must be raised steadily and ample air must be present to permit the complete burning of carbonaceous materials (impurities in the clay along with paper, wax etc).After 800°C ,the clay surface will start to seal off, trapping unburned carbonaceous materials and sulfides, which could cause bloating and black coring.<br>Quartz inversion occurs at 573°C. When clay is refired for a glaze firing, quartz crystals change from an alpha crystal structure to a beta crystal structure. The inversion is reversed on cooling. This conversion creates stresses in the clay so temperature increase and decrease must be slow to avoid cracking the work.<br>Between 480-700°C, chemical water (referred to as 'water smoke') is driven off. |
| 700 | | |
| | Dark red | |
| 600 | | |
| | Dull red glow | |
| 500 | | |
| | Black | |
| 400 | Black | |
| 300 | Black | Upon cooling, cristobalite, a crystalline form of silica found in all clay bodies, shrinks suddenly at 220°C. Fast cooling at this temperature will cause ware to crack. |
| 200 | Black | |
| 100 | Black | Water boils and converts to steam. Trapped water will cause clay to explode so all water should be evaporated below 100°C. Begin a firing by keeping the kiln below 100°C until all water has evaporated. |

## 2.5 Factors that influence compressive strength of fired clay

Compressive strength is one of the physical properties of clay that is a good predictor of fired clay's ability to resist cracking of the surface. Compressive strength is highly affected by firing temperature, method of production, physical, chemical and mineralogical properties of raw clay. The strength of clay products depend strongly on the production technology. The splitting process, firing temperature, the restraint effect of the platens, the height and shape of the specimen can have an effect on the strength as well.

There are two possible ways of producing clay product. The clay product has to be formed either by extrusion or by moulding. The former method is applied to clays with high level of plasticity. When the plasticity of the clay is lower, the latter procedure is used characteristically. In this case the clay cake is filled into moulds and then pressed.

Pate and Noble (1958) carried out an investigation on the relationship between the elastic modulus and compressive strength of clay products. The relationship between the elastic modulus and strength of clay products was linear in case of low strength. In case of higher strength the Young's modulus increased exponentially with temperature. The variations between compressive strength of the fired clay products with firing temperature and Young's modulus were determined. It was found that proportionality exists between the two mechanical features.

In the study of factors that influence the strength of fired clay, Livingston et al., (1998) suggested that the durability and compressive strength of fired clay are related to their microstructure and mineralogy. In unfired clay, the strength and water permeability are related to the size and shape of particles present and the forming process, but upon heating, the nature of the mineral comprising the mass has a very important influence on the compressive strength because of the chemical reactions and partial fusing which occur then.

The porosity in clay unit depends on the type of clay used in manufacturing and temperature of firing. According to Khalaf et al., (2002), the porosity of the clay influences its compressive strength, water absorption and permeability. During the sintering process in the clay product manufacturing process, stable initial raw material transform into complex compounds at high temperatures. New compounds are also formed due to chemical reactions that take place. These compounds have impacts on the stability of the clay product due to the decrease or increase in the volume of the system.

The study carried out by Tile et al., (1975), showed that vitrification of ceramic material is an important factor that influences the quality and physical properties of the end product such as compressive strength and permeability. The microstructure changes with the sintering temperature. At 800°C and 900°C, the clay has not yet experienced full solid state sintering process since the individual clay particles have not fused together. Tile et al., (1975) concluded that the clay structure formed at lower temperatures of 840°C to 960°C remained essentially the same until temperatures of over 1080°C are reached.

According to Johari et al., (2010), the porosity of clay showed an increment of 1.4 percent and 0.1 percent from 800°C to 900°C and 900°C to 1000°C respectively. The increase in porosity was a result of diffusion at relatively low temperature without significant shrinkage. The shrinkage value for temperature 800°C, 900°C and 1000°C was 0.31 percent, 0.50 percent and 1.04 percent respectively. In his study, clay products that were sintered until 1000°C were considered as having a porous structure since their water absorption rates were higher than 25 percent. Between1000°C and 1100°C, the solid state sintering becomes very significant since the clay body had been fully sintered. Very few pores can be seen in the microstructure. Clay product porosity values reduced significantly from 39.33 percent to 27.06 percent and it was 31 percent reduction. The importance of the solid state sintering process was to develop atomic bonding between particles by a diffusion mechanism. This diffusion followed by grain growth which created a dense structure with significant shrinkage value increase of 74 percent causing the reduction in pore for clay product sintered from temperature 1000°C to 1100°C. A progressive gain in compressive strength was observed on clay sintered at 1100°C where the strength increased from 25.4N/mm$^2$ to 71.8 N/mm$^2$.

Starting from 1100°C, the liquid phase sintering becomes a very important sintering mechanism. German, (1985) emphasized that the liquid phase sintering was existent if there is a liquid phase that coexists with particulate solids during the sintering process. During this process, the reduction of pores becomes more significant as compacted structure starts to increase its performance such as compressive strength and water permeability. The fired clay sintered at 1100°C begins to diffuse and shrink as the liquid phase starts to form and fill up the pores creating smaller pores. The clay product shrinks by 37 percent when sintered from 1100°C to 1200°C causing the porosity to reduce 47.5 percent. The effect of firing also caused the water absorption value to reduce 42 percent lower than the value for clay product sintered at 1000°C and hence compressive strength of the fired clay increases. In their study, the internal surface of pores in clay product sintered at 1200°C and 1250°C had a 'glazed' view. The sintering process reached the optimum temperature at 1200°C, whereby its microstructure contained minimum pores with porosity value 14.2 percent and produced the highest strength 89.5 MPa. However at 1250°C, the microstructure showed larger pore sizes and lower porosity value which is 5.87 percent with brittle fracture behavior. The clay product becomes more brittle due to a larger portion of glassy phase in the microstructure. Therefore, the compressive strength of the clay product became lower. The firing has a positive influence on the microstructure of the clay product promoting a dense structure with low permeability (Green, 1969).

Plasticity of the clay bodies is of great importance in the shaping of clay materials. The plasticity of a normal clay product is -26.75 percent. When the rock residue content in the mixture varied from 10 percent to 50 percent, the plasticity value changed from -25.08 percent to 16.26 percent respectively. Plasticity is an important parameter for the production of clay material. Because, clay industry mainly uses extrusion shaping and therefore the insufficient plasticity creates failures and heterogeneities in the clay body, this causes variation in mechanical properties. The plasticity has an important technological application, since it indicates the minimum percentage of moisture content necessary to reach a plastic condition. With high plasticity, there will be more difficulty in drying the samples and causing the appearance of dimension problem or even cracks. It was observed that the residue addition caused no difficulties during the mixing and extrusion process. The percentage of plasticity of clay body samples decreases with an increase in the rock residue mixture in the clay samples.

The density affects a number of properties of clay materials but probably the most important effect is on compressive strength. The drying density of clay products is a function of rock residue addition. The density of a normal reference clay body is 1.781g/cm$^3$ and the values of density of clay bodies vary from 1.888 to 2.349g/cm$^3$. The drying density of clay products increases with the increase in the rock residue mix in the samples. Therefore the density of the clay body is directly proportional to the quantity of rock residue added in the mixture.

# CHAPTER THREE: METHODOLOGY OF THE STUDY

## 3.1 Introduction

In this chapter, sample preparation of clay, laboratory tests conducted in determination of chemical compounds of Ntawo ball clay and breaking force of fired ball clay have been discussed. The research design employed in the study was descriptive and relational. Clay samples were dried and fired at the same rate of heating to different firing temperatures for different holding times. The study focused on compressive strength of samples of Ntawo clay, how to relate it to holding time for a specific firing temperature and to relate firing temperature at given holding times of 20, 30, 40, 50, 60 minutes for firing temperatures of 500°C, 600°C, 700°C, 800°C, 900°C and 1000°C. Measurement of cross sectional area of each sample was done. The compressive strength of each sample was determined

## 3.2 Research Design

The design was aimed at preparing and testing the compressive strength of Ntawo fired ball clay product by varying firing temperature and holding time while firing it. An electric kiln model carbolite 1400 for firing the samples, Compressive machine 500 SN- 12994917 for determining the breaking force and an X-Ray Diffractometer RIX 3000 machine for identifying chemical compounds in ball clay, were used during the investigation. Two hundred samples based on the particle size of 0 - 45 $\mu m$ sieve were made to study the material performance during formation and firing. The dimensions length, width and height of the fired samples were measured which later used to determine their cross sectional area. The average cross sectional area was calculated which was used to determine compressive strength of the samples. The design was found appropriate for the study since the study focused on investigation of compressive strength of Ntawo ball clay fired to different temperatures for different holding time. The design was used to measure compressive strength of fired ball clay sample for different holding time fired to specified temperatures and compressive strength of fired ball clay sample fired to different firing temperature held for specified holding times. Also the design was used to establish the relationship between compressive strength and holding time under the null hypothesis $H_{01}$ and relationship between compressive strength and firing temperature under the null hypothesis $H_{02}$.

### 3.3 Sample Preparation

Forty kilograms of clay soil was dug from five different pits within the same quarry of the Ntawo deposit. This was to increase on the chances of minerals of ball clay picked and also to increase the sample space within the same quarry of the Ntawo deposit. The clay soil was packed in two polythene bags and then transported to Uganda Industrial Research Institute's Laboratory shade. The clay soil was removed from the polythene bags and left outside in an open area for two weeks to allow organic matter in it to decompose and add plasticity in the clay. After the clay was removed from the open area, to the bin containing water before the unwanted particles in the clay such as big stones, metals and other vegetative materials were removed. In the bin, clay was thoroughly mixed with water using a hoe. The added water to ball clay was used to bring the slurry to a suitable consistency. The slurry formed was poured in a stack of sieves on a mechanical vibrator as shown in Figure 3.1 to get clay of particle size 0 - 45 $\mu m$. The paste was left for a week to gain wet strength, before moulding the clay samples.

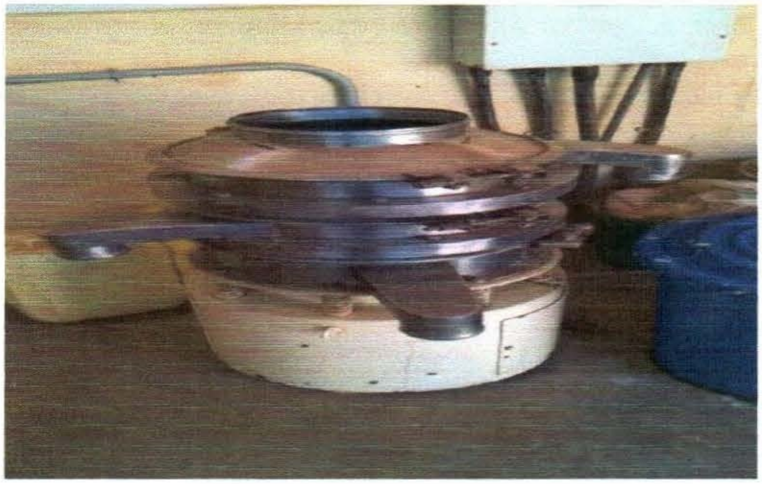

**Figure 3. 1:** A stack of sieves on a mechanical vibrator

Preparing a clot of clay was done, by dusting the clot on five of the six surfaces to prevent it from sticking to sides of the mould. Starting with the cut off piece from the previous clay mass, a new lump of clay was put on top. Using the human palm, the new ball of clay was rolled forward till the previous cut off came over the top.

It was pressed down making a wedge shape clot of clay. The clot was lifted up on edge to sand the narrow edges. After sanding, the second narrow edge, the clot was rolled forward to apply sand to the end. The clot was tempered with a wide top and narrow bottom end ready to throw into the mould cavity shown in Figure 3.2.

**Figure 3.2:** Rectangular moulds used to fabricate clay samples

A rectangular metallic mould was obtained by assembling rectangular metal blocks, hollow rigid metal frame and a metal plate from mild steel. The rectangular mould was of dimension 4 cm x 16 cm x 1 cm. This type of clay mould was selected so that the clay samples obtained from these moulds would fit in the small space of the electric Kiln used for firing of the clay articles.

The hand moulding method with a rectangular metallic mould was used. The clay was shaped by hand into a lump. The use of a mould was to give shape to the clay which resulted in more accurate and better clay samples. The metallic moulds were soaked in oil before use. This was to lubricate the moulds so that it prevents the plastic clay from sticking on the metal mould. The plastic clay was thrown into the wetted bottomless mould of metal as it rested upon a wooden pallet.

**Figure 3.3:** Clay in the rectangular moulds

The clay mass was manually thrown in the mould and then pressed using hands to reduce the voids and cracks 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. Fast drying on extremely hot days may lead to cracks in the green clay article which is undesirable, therefore to minimize crack and quick moisture loss, clay samples were not left open to extremely heavy sunshine. The clay samples were laid out in stacks with a horizontal- vertical alignment as to minimize usage of space in the laboratory. The clay samples were left to dry for another week and after they were turned over for another week to facilitate uniform drying and prevent them from warping. After two weeks, the samples were sufficiently hard to allow them to be stacked on end in a hurrying bone pattern with a finger's width between them to allow further drying. This is called a hack or a hack stead. This was done in an open room, to protect the clay samples from the rain or harsh sun. After the four weeks, the clay samples were dry and ready to be fired. The time of drying is dependent on the moisture content of the clay samples and the humidity of the production area.

The dried green clay samples were heated to a temperature of 100°C in an Electric Kiln (pottery crafts kiln), Model carbolite 1400 for 12 hours. This was to ensure total dryness of ball clay samples to prevent the swelling or bloating of the samples when fired at high temperatures, caused by the expansion of entrapped water. The clay samples were allowed to cool and then each clay sample was marked before firing to high temperatures was done in an electric kiln.

**Figure 3.4:** An electric kiln used for firing the clay samples

Firing for this investigation was done using an electric kiln model carbolite 1400 shown in Figure 3.5. The firing temperatures used for this study were 500°C, 600°C, 700°C, 800°C, 900°C, and 1000°C . The clay samples were fired separately for holding times 20, 30, 40, 50 and 60 minutes with these six different firing temperatures. One hundred fifty samples were put in sets of five clay samples. The different sets of five clay samples were fired for holding times of 20,30, 40, 50, and 60 minutes separately at fixed temperatures of 500°C, 600°C, 700°C, 800°C, 900°C, and 1000°C. This was to test the effect of duration of firing temperature on the compressive strength of the fired clay samples and effect of firing temperature on compressive strength of clay at fixed holding times 20, 30, 40, 50 and 60 minutes. Here hold time means duration of firing maintained after the corresponding firing temperature was attained. The rate of firing was very important as clay sample was fired to gain high strength. This was to ensure that heat penetrate deep into the clay body. Rapid firing causes the bloating of clay due to the formation of an impermeable vitrified outer skin preventing the loss of gases such as water vapour and carbon dioxide from the interior of the clay. Therefore, the electric kiln temperature was gradually increased from 100°C to the set firing temperature of each treatment.

Having firing completed, hot clay samples were allowed to cool down, gradually decreasing temperature of the electric kiln. The electric kiln was turned off and clay samples were allowed to stay for two hours in the kiln and then removed from the kiln.

After the firing process, the fired clay samples' physical properties were observed. The dimensions of each fired clay sample were measured using the vernier caliper. The dimensions were used to determine the cross sectional area. The overall average cross sectional area for all the fired clay samples was determined and found to be $1.1 \times 10^{-3} m^2$. Later, the average cross sectional area was used to determine the average compressive strength of the fired samples in each batch of five samples. Then the fired clay samples were subjected to breaking force tests.

### 3.4 Determination of chemical composition of Ntawo Clay

The clay soil sample was dried at 100°C and then finely milled to obtain fine particle size to determine the chemical compounds in Ntawo ball clay. Ten grams of clay samples were put in small 30 mm diameter plastic containers. The clay samples were taken to Mintek Mineral Research Institute, South Africa through the Uganda Industrial Research Institute.

At Mintek Mineral Research Institute, identification of chemical compounds was carried out by the X-ray diffraction technique using a Bruker D8 Advance diffractometer with a Lynx Eye detector. The X-ray diffraction patterns were scanned in steps of 0.034° in a range of diffraction angles from 10° to 59° of $2\theta°$ for ball clay using copper ($K\alpha$) as X-ray source with a wavelength 1.541Å. An X-ray spectrometer machine, Rigaku RIX 3000 was used to determine the chemical compounds of ball clay. The clay samples were irradiated and the radiations generated by the clay elements were characterized by specific wavelength and intensity. This was related to their concentration and hence easy identification in the X-ray spectrum.

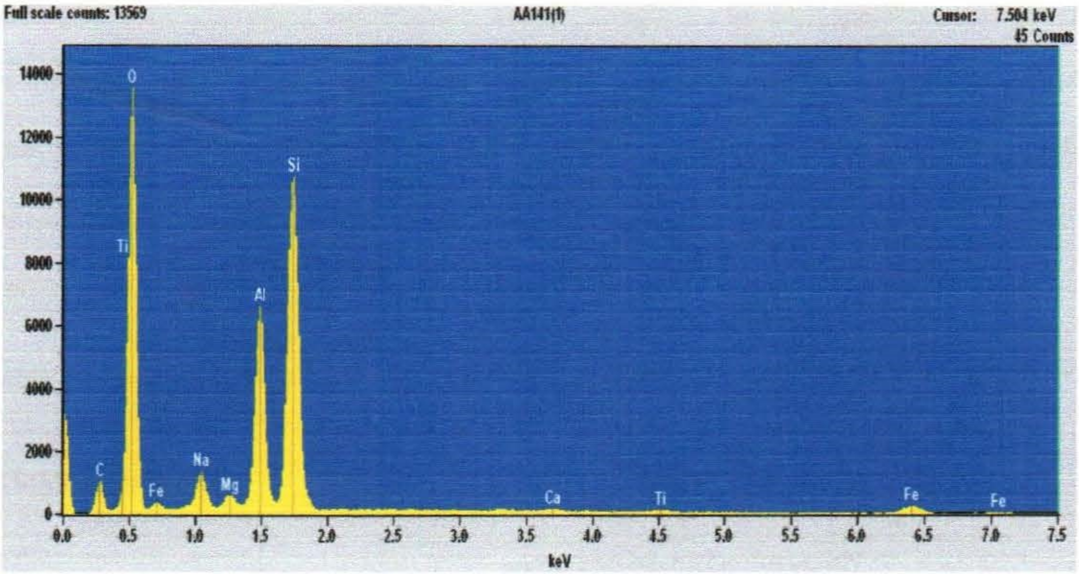

**Figure 3.5:** X- Ray Diffractogram of Ntawo Ball Clay

The chemical elements which were identified included: carbon, oxygen, sodium, magnesium, aluminum, silicon, calcium, titanium and iron as shown on Figure 3.6, the X-ray diffractogram of the clay material taken from Ntawo deposit used in the study. The relative amounts by weight percentage were estimated using the Autoquan program.

### 3.5 Determination of Breaking Force of fired Ntawo Clay Samples

The measurement of breaking force of the clay samples was done using a compression 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 bearing surfaces of all the platens of the machine were wiped clean. This was to ensure a uniform bearing for each clay sample, placed between the platens of the machine. Each fired clay sample was placed between the two parallel plates of the compression test machine. The test procedure started in load control until the specimen was loaded with a force of 25 kN. The vertical linear variable displacement transducer (LVDT) was adjusted at a uniform rate of 14Nmm$^{-2}$ per minute, until the fired clay sample experienced explosive collapse or failure. The load at failure was the maximum load at which the clay samples failed to produce any further increase in the indicator reading on the compressing test machine. The breaking force for each specimen was therefore read from the test machine. The results of breaking force of samples fired for varying holding times to different firing temperatures is in appendix C. The values of breaking force of fired Ntawo ball clay samples for the five holding times are showed in table 3.1. The table gives average values of breaking force in kilonewtons (kN).

**Table 3.1** Values of breaking force of clay samples fired to different firing temperatures held at different holding times.

| **Holding time / min** | **Firing temperature/°C** | | | | | |
|---|---|---|---|---|---|---|
| | 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 |

The average breaking force values in Table 3.1 were used to determine compressive strength of the fired clay samples. The compressive strength of the fired clay samples in a batch was calculated by dividing average breaking force (kN) on the sample by the average cross sectional area ($m^2$).

# CHAPTER FOUR: RESULTS OF THE STUDY

## 4.1 Introduction

This study focused on the chemical compounds of Ntawo ball clay, how compressive strength of fired Ntawo ball clay was affected by varying holding time at six different firing temperatures and how compressive strength was affected by varying firing temperature for five holding times. The study was to generate results to respond to the null hypothesis, $H_{O1}$, that at given firing temperatures there was no difference in compressive strength of the fired samples. This was to find if the null hypothesis hold for all the six selected firing temperatures. The second null hypothesis, $H_{O2}$, was that there was no difference in compressive strength of the fired samples for different firing temperatures at fixed holding times. This was to find if indeed the null hypothesis hold for all the five selected holding times. Two levels of significance $\alpha =$ 0.05 and $\alpha = 0.01$ respectively were used to test the null hypothesis.

The degrees of freedom (4, 20) and (4, 25) in the study were used to test the null hypotheses $H_{01}$and $H_{02}$ respectively. Graphs and tables to interpret the findings of the study were considered.

The chemical compounds in the clay sample were first determined using X-ray diffraction method.

## 4.2 Chemical Composition of Ntawo Ball Clay

From the X-ray diffraction, relative amounts by percentage weight of chemical compounds were determined using the Autoquan program. They are as shown in Table 4.1.

**Table 4.1:** Chemical Composition of Ntawo ball clay

| Chemical compounds | Chemical formula | 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 | NaO | 0.19 |
| Titanium dioxide | $TiO_2$ | 1.38 |
| Phosphorous oxide | $P_2O_5$ | 0.10 |
| Loss on ignition | LOI | 8.14 |

As shown in Table 4.1 , the Ntawo ball clay has more silica (67.2%), less alumina (18.2%) content and $Fe_2O_3$(2.83%). The Ntawo clay has relatively small oxides so that fusion of the clay particles may take place at higher temperature. These oxides include: CaO (0.31%), NaO (0.19%), $K_2O$ (0.98%) and MgO(0.36%). They act as fluxes and facilitate fusion of clay particles and influence the colour of the fired clay product. The iron content in the ball clay from Ntawo influences the brown reddish colour of clay products when fired at higher temperature. The weight percentages of mineral compounds obtained are within the range compared to those obtained by S.K.Duggal (2000) and Nyakairu etal (2001). The results by S.K.Duggal were : silica (50-70%), alumina (18-30%), $Fe_2O_3$(< 7%), CaO (10%), MgO(< 1%),and NaOH(< 10%) . And results by Nyakairu etal were: silica (67.18%), alumina (18.23%), $Fe_2O_3$(2.832%), CaO (0.306%), MgO(0.363%), and NaO(0.185%).

## 4.3 The Compressive Strength of Fired Ntawo ball clay for different Holding Times

This study was to find out the variation of compressive strength for temperatures set from 500°C to 1000°C at intervals of 100°C for holding times 20 minutes to 60 minutes. The relationship between compressive strength and holding time was set using the null hypothesis $H_{01}$. For each firing temperature, two levels of significance $\alpha = 0.01$ and 0.05 were used. Values of F for both theoretical and calculated to test the null hypothesis were determined.

The theoretical values of $F_{0.01}$ and $F_{0.05}$ were 4.31 and 2.866 respectively at degrees of freedom of 4 and 20 of the data for each firing temperature.

The null hypothesis, $H_{O1}$ was that there was no difference between the compressive strength of the fired samples for different holding times, when the ball clay was fired to different temperatures.

The null hypothesis was first tested when the clay was fired to 500°C. The result for samples fired to 500°C was obtained and are as shown in Table 4.2 .

**Table 4. 2:** Values of compressive strength for different holding times of samples fired to 500°C .

| **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** |

The Table 4.2 includes the average variance within the mean and average variance with sample data. These values including the degrees of freedom of 4 and 20 for the data were used to calculate the F-value for the data shown in table.

A graph showing the relationship between compressive strength and holding times was as shown in Figure 4.1.

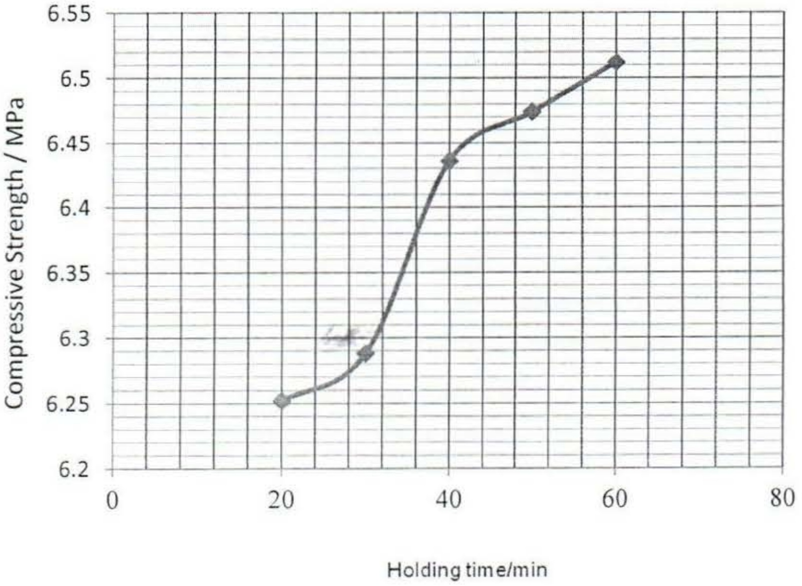

**Figure 4.1:** A graph of compressive strength against holding time for samples fired to 500°C

There was gradual increase in compressive strength between holding time 20 and 30 minutes, followed by sharp increase in strength between 30 and 40 minutes, then gradual increase in strength. From the results shown in Figure 4.1 compressive strength increased with increase in holding time. The overall percentage increase in compressive strength obtained between holding time 20 and 60 minutes was 4.2 percent for samples fired at 500°C.

The F- value calculated (6.9168) was greater than the two theoretical F- values 4.431 and 2.866 respectively at the two levels of significance $\alpha = 0.01$ and $\alpha = 0.05$. Hence the null hypothesis $H_{01}$ was rejected. This showed that there was significant relationship between average compressive strength and holding times for samples fired at 500°C

Another set of results was used to test the null hypothesis $H_{01}$ for the samples fired at 600°C. The result for samples fired to 600°C obtained was as shown in Table 4.3.

**Table 4.3:** Values of compressive strength for different holding times of samples fired to 600°C .

| 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** |

The Table 4.3 includes the average variance within the mean and average variance with sample data. These values including the degrees of freedom of 4 and 20 for the data were used to calculate the F-value for the data shown in the table.

A graph showing the relationship between compressive strength and holding times for samples fired to 600°C is shown in Figure 4.2.

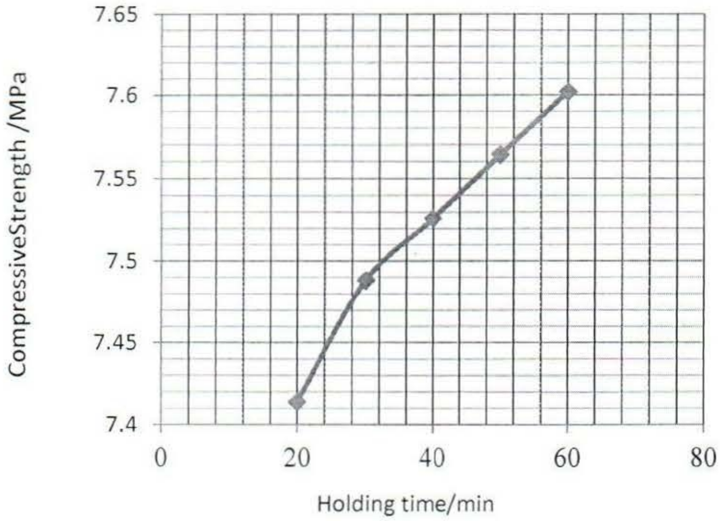

**Figure 4.2**: A graph of compressive strength against holding time for samples fired to 600°C

The curve is steeper between holding time 20 and 30 minutes. This indicated that average compressive strength increases with holding time. It was then followed by a gradual increase in strength between holding time 30 and 60 minutes. The overall percentage increase in

Compressive strength between holding time 20 and 60 minutes was 2.5 percent for samples fired at 600°C.

The value obtained for F (14.0918) exceeds the theoretical $F_{0.05} = 2.866$ and $F_{0.01} = 4.431$ values with 4 and 20 degrees of freedom. The null hypothesis $H_{O1}$ was rejected at the 0.01 and 0.05 levels of significance. Therefore, there was a difference between average compressive strength and holding times for clay samples fired to 600°C.

Another set of results was used to test the null hypothesis $H_{O1}$ for the samples fired at 700°C. The result for samples fired to 700°C obtained was as shown in Table 4.4.

**Table 4.4:** Values of compressive strength for different holding times of samples fired to 700°C

| Holding time /min | Average compressive strength $\overline{x}$ /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** |

This table 4.4 includes the average variance within the mean and average variance with sample data. These values including the degrees of freedom of 4 and 20 for the data were used to calculate the F-value for the data shown in table 4.4.

A graph showing the relationship between compressive strength and holding times for samples fired to 700°C is shown in Figure 4.3.

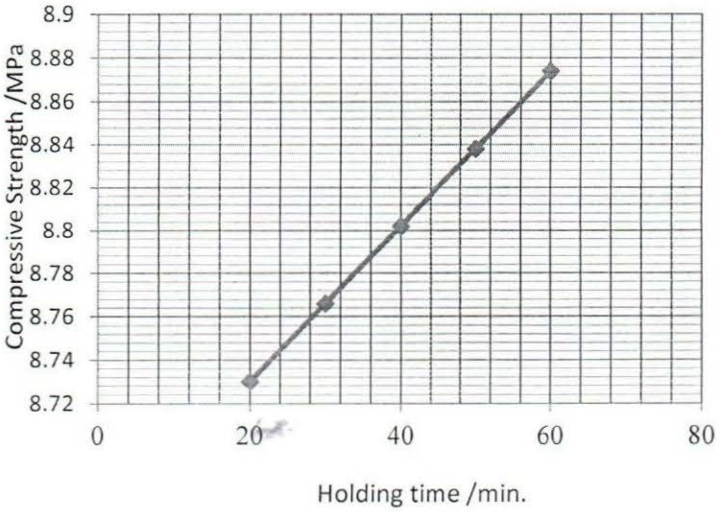

**Figure 4.3:** A graph of compressive strength against holding time for samples fired to 700°C

The relationship between average compressive strength and holding times is as shown in Figure 4.3. The graph was a straight line and less steep. 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 20 and 60 minutes was 1.6 percent for samples fired at 700°C. Therefore, the results obtained for the five holding times at firing temperature of 700°C are significant.

The F- value calculated (29.3478) was greater than the two theoretical F- values 4.431 and 2.866 respectively at two levels of significance $\alpha = 0.01$and $\alpha = 0.05$. Thus the null hypothesis $H_{01}$ was rejected. This indicated that there was significant relationship between average compressive strength and holding times for samples fired at 700°C.

Another set of results was used to test the null hypothesis $H_{01}$for the samples fired at 800°C. The result for samples fired to 800°C obtained was as shown in Table 4.5.

**Table 4. 5:** Values of compressive strength for different holding times of samples fired to 800°C .

| Holding time /min | Average compressive strength $\overline{x}$ /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** |

The Table 4.5 includes the average variance within the mean and average variance with sample data. These values including the degrees of freedom of 4 and 20 for the data were used to calculate the F-value for the data shown in Table 4.5 above. A graph showing the relationship between average compressive strength and holding times at 95% confidence level was as shown in Figure 4.4.

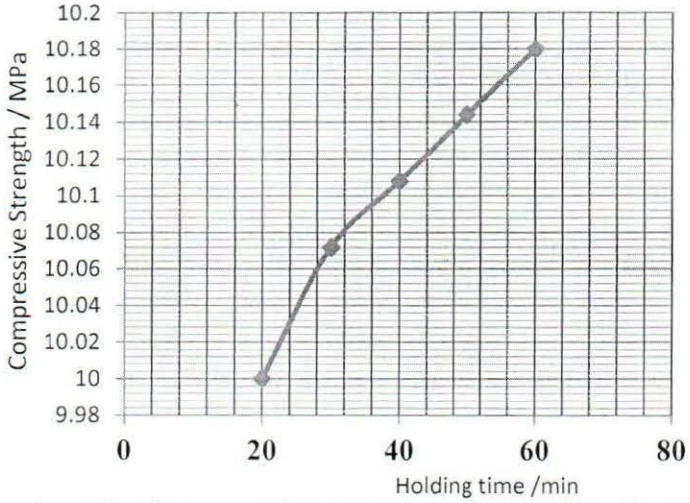

**Figure 4. 4:** A graph of compressive strength against holding time for samples fired to 800◦C

The graph in Figure 4.4 shows a steep increase in average compressive strength 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°C.

The F- value calculated (3.6999) exceeded the theoretical F-value at the level of significance $\alpha = 0.05$ and was less at $\alpha = 0.01$. Therefore, the null hypothesis $H_{O1}$ was rejected at 95% confidence level and accepted at 99% confidence level with 4 and 20 degrees of freedom. It was noted that for 99% confidence level, there was a difference in compressive strength of fired ball clay for the five holding times, while at 95% confidence level; the difference in compressive strength and the five holding times was significant.

Another set of results was used to test the null hypothesis $H_{01}$for the samples fired at 900°C. The result for samples fired to 900°C obtained was as shown in Table 4.6.

**Table 4.6:** Values of compressive strength for different holding times of samples fired to 900°C

| **Holding time /min** | **Average compressive strength $\overline{x}$ /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** |

The Table 4.6 above includes the average variance with in the mean and average variance with sample data. These values including the degrees of freedom of 4 and 20 for the data were used to calculate the F-value for the data shown in Table 4.6.

A graph showing the relationship between compressive strength and holding time is as shown in Figure 4.5 for clay samples fired to 900°C .

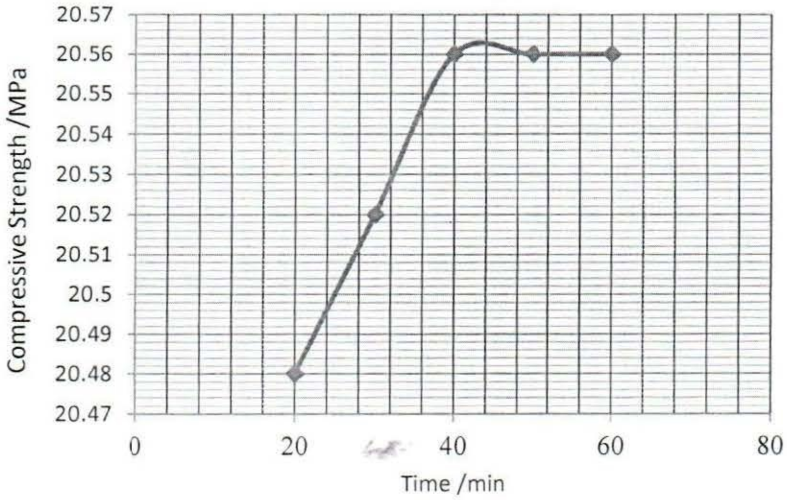

**Figure 4.5**: A graph of compressive strength against hold time for samples fired to 900°C

The graph is a straight line and has the highest slope between holding time 20 minutes and 40 minutes, but remained constant between holding time 50 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°C.

The F- value calculated (0.5333) was less than the theoretical F- values 4.431 and 2.866 respectively at the two levels of significance $\alpha = 0.01$ and $\alpha = 0.05$. Thus, the null hypothesis, $H_{01}$ was accepted at both 95% and 99% confidence levels. For both confidence levels, there was a difference in compressive strength and holding time for samples fired at 900°C. Therefore, the results obtained for the five holding times at firing temperature of 900°C showed that there was no effect of holding time on compressive strength of the fired ball clay.

Another set of results was used to test the null hypothesis $H_{01}$ for the samples fired at 1000°C.

The average compressive strength was determined for different holding times when the samples were heated to a temperature of 1000°C .The result obtained for samples fired to 1000°C was as shown in Table 4.7 .

**Table 4. 7:** Values of compressive strength for different holding times of samples fired to 1000°C.

| 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** |

The Table 4.7 includes the average variance within the mean and average variance with sample data. These values including the degrees of freedom of 4 and 20 for the data were used to calculate the F-value for the data shown in Table 4.7.

A graph of compressive strength against holding time for samples fired to 1000°C is as shown in Figure 4.6.

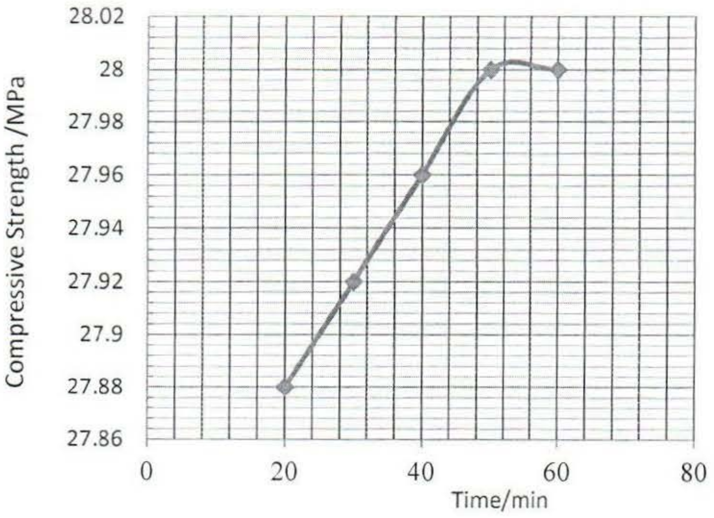

**Figure 4.6:** A graph of compressive strength against holding time for samples fired to 1000°C

The graph is a straight line, steep between 20 and 50 minutes. The overall percentage increase in compressive strength obtained between holding time 20 and 60 minutes was 0.4 percent for samples fired at 1000°C.

The calculated F- value (1.70) was less than the theoretical F- values at the levels of significance $\alpha = 0.05$ and $\alpha = 0.01$. The null hypothesis, $H_{01}$ was accepted at both levels of significance. At both levels of significance, there was a difference in average compressive strength of fired ball clay for the five holding times. Therefore, the results obtained for the five holding times at firing temperature of 1000°C show that there was no effect of holding time on the strength of the fired ball clay.

### 4.4 Compressive Strength of Ntawo ball clay for different Firing Temperatures

This study was to find out the variation of compressive strength for holding times set from 20 minutes to 60 minutes at intervals of 10 minutes for firing temperatures 500°C to 1000°C. The relationship between compressive strength and firing temperature was set using the null hypothesis $H_{02}$. For each holding time set, two levels of significance $\alpha = 0.01$ and $\alpha = 0.05$ were used to test null hypothesis $H_{02}$. The theoretical values of $F_{0.01}$ and $F_{0.05}$ were 2.76 and 4.18 respectively at degrees of freedom 4 and 25 of the data for each holding time set.

The null hypothesis, $H_{O2}$ was that there was no difference between compressive strength for the different firing temperatures of samples and the different holding time, when ball clay was fired.

The hypothesis $H_{02}$ was first tested when the clay was fired at holding time 20 minutes. The result obtained for samples fired at holding time 20 minutes was as shown in Table 4.8.

**Table 4. 8**: Values of compressive strength for different firing temperatures at the holding time of 20 minutes

| **Firing temperature /°C** | **Average compressive strength $\overline{x}$ /MPa** | **Variance within sample** | **Variance within mean** |
|---|---|---|---|
| 500 | 6.252 | 0.00972 | 41.92174 |
| 600 | 7.414 | 0.00648 | 28.22478 |
| 700 | 8.730 | 0.00000 | 15.97361 |
| 800 | 10.000 | 0.00000 | 7.43489 |
| 900 | 18.622 | 0.00972 | 34.75456 |
| 1000 | 25.342 | 0.00972 | 154.14579 |
| **Grand mean** | **12.727** | **0.00594** | **47.90923** |
| **Over all variance within mean** | | | 287.45537 |
| **Calculated F- value** | | | **48393.15993** |

The Table 4.8 includes the average variance within the mean and average variance within sample data. These values including the degrees of freedom of 4 and 25 for the data were used to calculate the F- value for the data shown in Table 4.8.

A graph showing the relationship between average compressive strength and different firing temperatures at holding time of 20 minutes is shown in Figure 4.7.

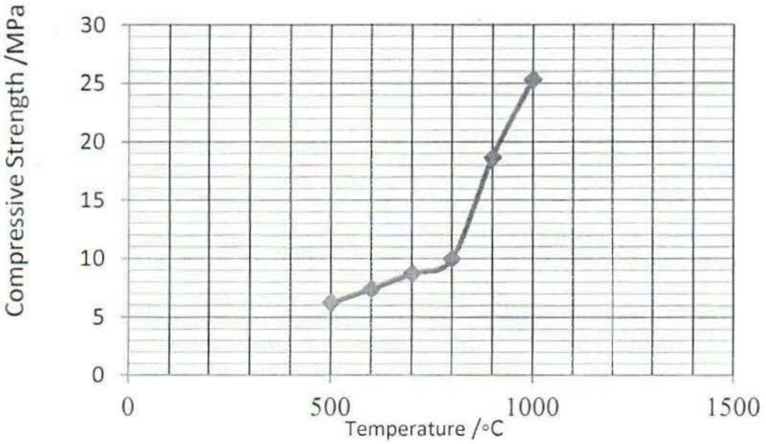

**Figure 4.7:** A graph of compressive strength against firing temperature for samples fired at holding time of 20 minutes

The average compressive strength gradually increased between firing temperature 500°C and 800°C, followed by a steep rise between 800°C and 900°C and slightly reduces between 900°C and 1000°C.The overall percentage increase in compressive strength obtained between firing temperature 500°C and 1000°C was 305.3 percent for samples fired at holding time of 20 minutes.

The calculated F- value (48393.16) exceeds the two theoretical F- values 2.76 and 4.18 at two levels of significance $\alpha = 0.01$and $\alpha = 0.05$ respectively. The null hypothesis, $H_{02}$ was rejected. Therefore, there was significant relationship between average compressive strength and firing temperatures of samples fired for the holding time of 20 minutes.

Another set of results was used to test the null hypothesis $H_{02}$ for the samples fired for holding time of 30 minutes.

The result obtained for samples fired at holding time 30 minutes for six different firing temperatures was as shown in Table 4.9.

**Table 4.9:** Values of compressive strength for different firing temperatures at the holding time of 30 minutes

| **Firing temperature /°C** | **Average compressive strength $\overline{x}$ /MPa** | **Variance within sample** | **Variance within mean** |
|---|---|---|---|
| 500 | 6.288 | 0.00972 | 42.081169 |
| 600 | 7.488 | 0.00722 | 27.952369 |
| 700 | 8.766 | 0.00648 | 16.072081 |
| 800 | 10.072 | 0.00972 | 7.306209 |
| 900 | 18.658 | 0.00972 | 34.60969 |
| 1000 | 25.378 | 0.00972 | 158.83561 |
| **Grand mean** | **12.775** | **0.008763** | **47.80952** |
| **Over all variance within mean** | | | 286.85713 |
| **Calculated F- value** | | | **32735.03663** |

The Table 4.9 includes the average values of variance within mean and variance within sample (data).These values including the degrees of freedom of 4 and 25 for the data were used to calculate the F- value for the data shown in Table 4.9.

A graph showing the relationship between average compressive strength and firing temperatures at holding time of 30 minutes is as shown in Figure 4.8.

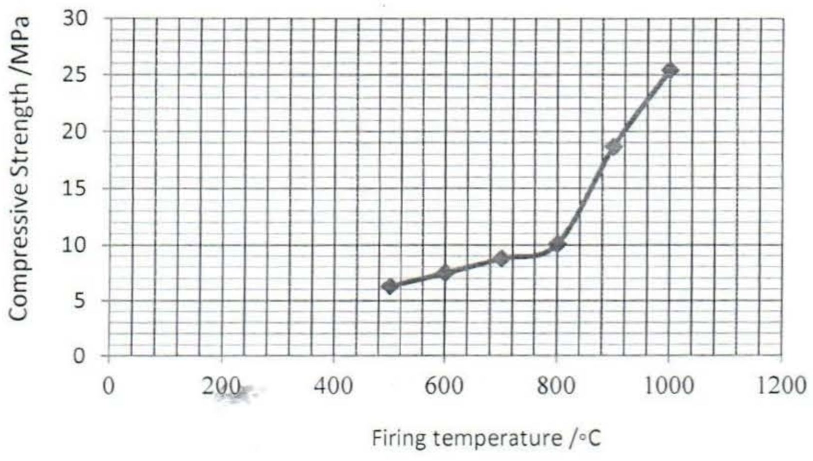

**Figure 4.7:** A graph of compressive strength against firing temperature for samples fired at holding time of 30 minutes

The average compressive strength gradually increased between firing temperature 500°C and 800°C, followed by a steep increase between firing temperature 800°C and 1000°C . The overall percentage increase in compressive strength obtained between firing temperature 500°C and 1000°C was 303.6 percent for samples fired at the holding time of 30 minutes.

The calculated F- value (32735.04) was greater than the two theoretical F- values 2.76 and 4.18 at two levels of significance $\alpha = 0.01$ and $\alpha = 0.05$ respectively. At the two levels of significance the null hypothesis, $H_{02}$ was rejected. This shows that the relationship between compressive strength and firing temperatures was significant for samples fired at the holding time of 30 minutes.

Another set of results was used to test the null hypothesis $H_{02}$ for the samples fired for holding time of 40 minutes. The result for samples fired for six different firing temperatures at holding time 40 minutes was as shown in Table 4.10.

Two levels of significance $\alpha = 0.01$ and $\alpha = 0.05$ were used for each test. The theoretical values of $F_{0.01}$ and $F_{0.05}$ with degrees of freedom 4 and 25 were 2.76 and 4.18 respectively.

**Table 4.10:** Values of compressive strength for different firing temperatures at the holding time of 40 minutes

| **Firing temperature /°C** | **Average compressive strength $\bar{x}$ /MPa** | **Variance within sample** | **Variance within mean** |
|---|---|---|---|
| 500 | 6.436 | 0.010830 | 40.88324 |
| 600 | 7.526 | 0.010830 | 28.13242 |
| 700 | 8.802 | 0.007128 | 16.22478 |
| 800 | 10.108 | 0.009720 | 7.40928 |
| 900 | 18.694 | 0.006480 | 34.38650 |
| 1000 | 25.414 | 0.006480 | 158.35706 |
| **Grand mean** | **12.830** | **0.008578** | **47.56554** |
| **Over all variance within mean** | | | 285.39327 |
| **Calculated F- value** | | | **33270.37445** |

The Table 4.10 includes the average values of variance within mean and variance within sample (data).These values including the degrees of freedom of 4 and 25 for the data were used to calculate the F- value for the data shown in Table 4.10.

A graph showing the relationship between average compressive strength and firing temperatures at holding time of 40 minutes is shown in Figure 4.9.

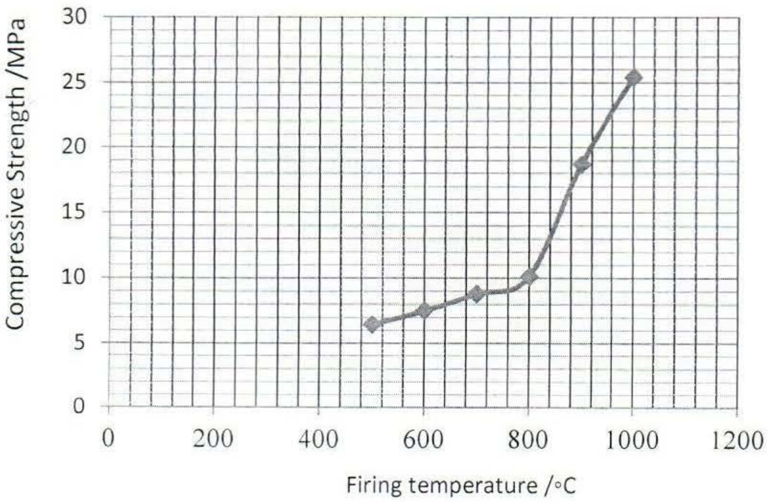

**Figure 4.8:** A graph of compressive strength against firing temperature for samples fired at holding time of 40 minutes

The compressive strength gradually increased between firing temperature of 500°C and 800°C, and then followed by a steep rise between firing temperature of 800°C and 1000°C. The overall percentage increase in compressive strength obtained between 500°C and 1000°C was 294.9 percent for samples fired at the holding time of 40 minutes.

The calculated F-value (33270.37) was greater than the two theoretical F- values 2.76 and 4.18 respectively at the two levels of significance $\alpha = 0.01$ and $\alpha = 0.05$ respectively. Therefore the null hypothesis $H_{O2}$ was rejected and the relationship between average compressive strength for the different firing temperatures was significant at holding time of 40 minutes

Therefore, there was significant relationship between average compressive strength and firing temperatures of samples fired for the holding time of 40 minutes.

Another set of results was used to test the null hypothesis $H_{O2}$ for the samples fired for holding time of 50 minutes.

The result for samples fired for holding time 50 minutes are as shown in Table 4.11.

**Table 4.11:** Values of compressive strength for different firing temperatures at the holding time of 50 minutes.

| **Firing temperature /°C** | **Average compressive strength $\overline{x}$ /MPa** | **Variance within sample** | **Variance within mean** |
|---|---|---|---|
| 500 | 6.474 | 0.01083 | 40.79377 |
| 600 | 7.564 | 0.01083 | 28.05821 |
| 700 | 8.838 | 0.00972 | 16.18453 |
| 800 | 10.144 | 0.00648 | 7.38209 |
| 900 | 18.694 | 0.00648 | 34.02389 |
| 1000 | 25.450 | 0.00000 | 158.48292 |
| **Grand mean** | **12.861** | **0.00739** | **47.487570** |
| **Over all variance within mean** | | | 284.92541 |
| **Calculated F- value** | | | **38555.53532** |

The Table 4.11 includes the average values of variance within mean and variance within sample (data).These values including the degrees of freedom of 4 and 25 for the data were used to calculate the F- value for the data shown in Table 4.11.

A graph showing the relationship between average compressive strength and firing temperature at holding time of 50 minutes is shown in Figure 4.10.

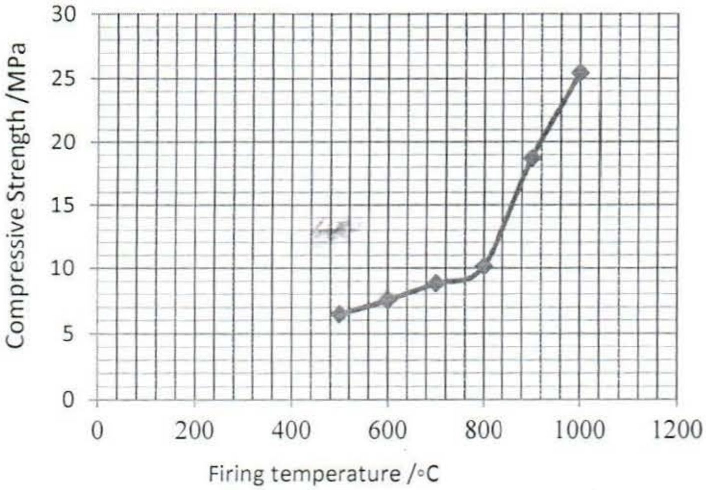

**Figure 4.9:** A graph of compressive strength against firing temperature for samples fired at holding time of 50 minutes

Average strength increased gradually from 500°C to 800°C, and then followed by a steep rise upto 1000°C. The overall percentage increase in compressive strength obtained between 500°C and 1000°C was 293.1 percent for samples fired at holding time of 50 minutes.

The F- value calculated (38555.5) was greater than the two theoretical F- values 2.76 and 4.18 at the two levels of significance $\alpha = 0.01$ and $\alpha = 0.05$ respectively. The null hypothesis $H_{O2}$ was rejected and the relationship between compressive strength for the different firing temperatures was significant at holding time of 50 minutes. The relationship between compressive strength and firing temperatures was not consistent at the holding time of 50minutes. Therefore, there was significant relationship between average compressive strength and firing temperatures of samples fired for the holding time of 50 minutes.

Another set of results was used to test the null hypothesis $H_{02}$ for the samples fired for holding time of 60 minutes. The result obtained for samples fired at holding time 60 minutes was as shown in Table 4.12.

**Table 4.12:** Values of compressive strength for different firing temperatures at the holding time of 60 minutes

| Firing temperature /°C | Average compressive strength $\bar{x}$ /MPa | Variance within sample | Variance within mean |
|---|---|---|---|
| 500 | 6.512 | 0.007220 | 40.61895 |
| 600 | 7.602 | 0.001805 | 27.91326 |
| 700 | 8.874 | 0.006480 | 16.09053 |
| 800 | 10.180 | 0.000000 | 7.31865 |
| 900 | 18.694 | 0.006480 | 33.74100 |
| 1000 | 25.450 | 0.000000 | 157.89169 |
| **Grand mean** | **12.8853** | **0.003664** | **47.25901** |
| **Over all variance within mean** | | | 283.55407 |
| **Calculated F- value** | | | **77385.701** |

The Table 4.12 above includes the average values of variance within mean and variance within sample (data).These values including the degrees of freedom of 4 and 25 for the data were used to calculate the F- value for the data shown in Table 4.12.

A graph showing a relationship between average compressive strength and different firing temperature is as shown in Figure 4.11.

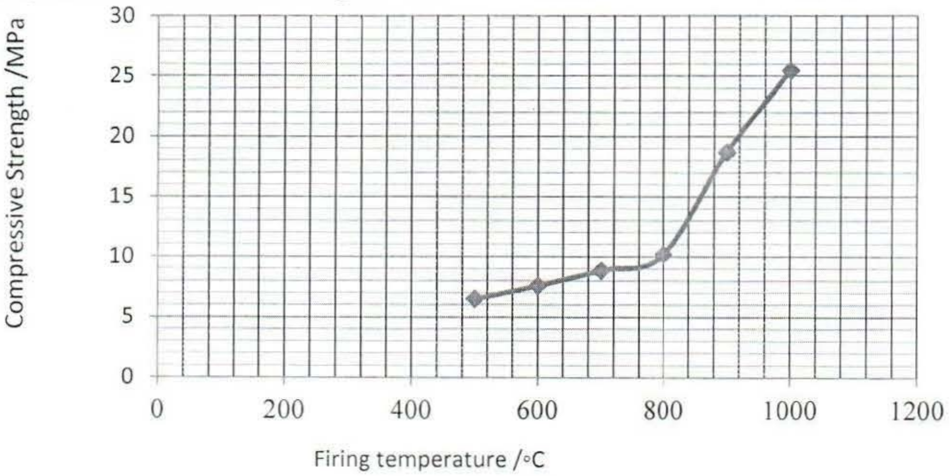

**Figure 4.10:** A graph of compressive strength against firing temperature for samples fired at holding time of 60 minutes

The compressive strength gradually increased between firing temperature of 500°C and 800°C, and then followed by a steep rise from firing temperature of 800°C to 1000°C. The overall percentage increase in compressive strength obtained between 500°C and 1000°C was 290.8 percent for samples fired at the holding time of 60 minutes.

The F- value calculated (77385.701) was greater than the two theoretical F-values 2.76 and 4.18 at $\alpha = 0.01$ and $\alpha = 0.05$ levels of significance. The null hypothesis was rejected at the $\alpha = 0.01$ and $\alpha = 0.05$ levels of significance. This indicated that there was a relationship between compressive strength and firing temperature of the sample fired at holding time 60 minutes. Therefore, there was significant relationship between compressive strength and firing temperature for samples fired at holding time of 60 minutes.

# CHAPTER 5: DISCUSSION, CONCLUSION AND RECOMENDATIONS

## 5.1 Discussion of results

In this study there were three investigations, namely chemical composition of Ntawo ball clay, establishment of relationship between compressive strength and holding time for specific temperatures and relationship between compressive strength and firing temperature for specific holding time.

The Ntawo ball clay exhibited high silica (67.2%) good for plasticity and alumina (18.2%) which is suitable for high strength. Also, contain $Fe_2O_3$(2.83%), MgO (0.36%), $K_2O$(0.98%), and CaO(0.31%). The metallic oxides CaO and MgO act as a flux and facilitate fusion of ball clay particles. The metal oxides influence the colour of the fired clay.

The results indicated that optimum firing temperature was found to be 1000°C . The percentage increase in compressive strength significantly increased with holding time between 500°C and 800°C. The percentage increase in strength was 4.2%, 2.5%, 1.6%, 1.8% at 500°C, 600°C, 700°C and 800°C respectively. Beyond 800°C, the percentage increase in compressive strength was 0.4% for 900°C and 1000°C. The traces of moisture in the clay test sample may continue to be given off until 800°C. Beyond 800°C, particles begin to fuse themselves together to create a hard material. The holding time beyond 800°C can't affect any change in the compressive strength of clay samples fired. Prolonged holding time had no significant effect on the compressive strength of fired clay investigated for temperatures above 800°C. Thus un necessary longer holding times should be avoided to save time and energy.

Also, in all cases compressive strength was increasing for all varying firing temperatures at fixed holding times. The percentage increase in compressive strength was 305.3%,303.6%,294.9%, 293.1% and 290.8% for holding times of 20, 30, 40, 50 and 60 minutes respectively. The overall average percentage increase in compressive strength was

298± 7 %. The relationship between compressive strength and firing temperature was significant. Therefore for all firing temperatures, the hypothesis $H_{02}$ was significant, showing that there was a relationship between compressive strength and firing temperature at specific holding times. The highest compressive strength was 25.45 MPa at firing temperature 1000°C for holding times 50 and 60 minutes. From the Table 4.12, compressive strength increased from 6.25 MPa at a holding time of 20 minutes to 25.45 MPa at the holding time of 60 minutes. This was attributed to reactions which occurred as firing temperature increased. Clay minerals decompose, hydroxyl groups present in the clay structure were driven off and oxidation took place and particles fused together yielding an increase in compressive strength.

## 5.2 Conclusions

Ntawo ball clay contained chemical compounds needed for fired clay products. It exhibited the high weight percentage of silica and alumina, iron and other metal oxides which act as fluxes.

According to this study, ball clay from Ntawo has a high compressive strength of

25.45 MPa. The optimum firing temperature of the local ball clay samples produced was 800°C and 1000°C. As the firing temperature increased within the optimum firing temperature range of the ball clay samples, average compressive strength of the fired ball clay samples increased. This confirms that firing temperature significantly affect compressive strength of fired ball clay products.

The results obtained for the relationship between compressive strength and firing temperature indicated an average percentage increase in compressive strength. The percentage increase between 290.8 and 305.3% was noted giving an average percentage increase of about 297.5±7% in compressive strength of all fired samples. Therefore there was a relationship between compressive strength and firing temperatures of the fired clay samples.

For specific firing temperatures, as the duration of firing increased from 20 minutes to 60 minutes, fired clay samples showed small increase in compressive strength. At firing temperatures higher than 800°C, compressive strength of the fired clay samples was not depending on holding time. This was the temperature at which the clay body reached sintering. 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 ball clay 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°C. Therefore unnecessary longer holding times should be avoided to save time and energy.

At temperatures of 800°C or above, the quality and durability of fired clay samples was generally superior. It displayed a high compressive strength value. The findings also indicated that the physical and mechanical properties of ball clay products can be controlled to a significant extent by varying the firing temperature. The best firing temperature for fired clay samples with good performance of mechanical properties was found to be 1000°C.

### 5.3 Recommendations

Basing on the results obtained; interest in compressive strength of ball clay beyond 800°C no need for longer duration of firing. Other investigations be conducted to find out what will happen to the trend of compressive strength for ball clay fired beyond 1000°C and 60 minutes. An investigation be carried out to find whether any change in the mineral compounds of ball clay affect the above results.

Research should continue with the availabilities and potential of ball clay sources from other clay deposits to see if they offer the needed compressive strength characteristics. Production of over and under fired clay products can be minimized by testing the optimum firing temperature of the raw materials to achieve target average compressive strength of the clay products and testing the optimum hold time of clay soil to achieve target physical properties of clay products. Using a temperature sensor device help to know the amount of firing temperature in the kiln, which minimises under firing, over firing and even melting of the clay products.

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## APPENDIX A

## FORMULAE USED IN ANALYSIS OF RESULTS

Computation of mean compressive strength test result for each sample and average Variances.

Compressive strength (MPa) = $\frac{Average\ breaking\ force\ (kN)}{Averagecross-sectional\ area\ (m^2)}$

Average cross – sectional area for the clay sample used in the study = 1100mm$^2$ or 1.1 x 10$^{-3}$m$^2$.

Grand average $\overline{X_G}$ $= \frac{\sum \bar{X}}{n}$ , where n- number of samples and $\bar{X}$ - average compressive strength.

Variance,$\delta^2 = (\bar{X} - \bar{X}_G)^2$.

Variance within mean = n $\left[\frac{1}{n-1} \sum (\bar{X} - \bar{X}_G)^2\right]$

Variance within samples = $\frac{1}{N} \sum \left[\frac{1}{n-1} \sum (X - \bar{X})^2\right]$ , where N – the number of average variances and $\bar{X}$ - average compressive strength.

Average variance = $\frac{\sum (X - \bar{X})^2}{n-1}$ and Variance, $\delta^2 = (X - \bar{X})^2$ , where $\bar{X}$ - average compressive strength.

Calculated F – value = $\frac{Variance\ with\ in\ mean}{Variance\ with\ in\ sample}$ .

**Levels of significance:**

$\alpha = 0.05$ and $\alpha = 0.01$ .

**Confidence levels:**

95% and 99% confidence levels

**Degrees of freedom:**

Degrees of freedom for numerator = k – 1 and

Degrees of freedom for denominator = k (n – 1).

Where, k – number of samples and n – number of variables such as firing temperatures, holding time considered in a table of results for treatments.

# APPENDIX B

## FORMULAE FOR CALCULATING STATISTICAL VALUES

**Formulae for interval estimation (t- distribution statistics).**

If $\bar{X}$ is the mean of a random sample of size n from a normal population with known variance $\delta^2$, then a central B% confidence interval for $\mu$, the population mean, is given by :

$\bar{X} \pm Z\left(\frac{\delta}{\sqrt{n}}\right)$ , where Z is the $\frac{100-B}{2}$ % point of a N(0, 1) distribution.

OR $Z = t_{\alpha/2}$ and $\alpha = \left(\frac{100-B}{2}\right)$ % .

**Determination of ranges in which the mean lies.**

Variation of compressive strength and holding time of firing of Ntawo ball clay.

Firing temperature of 900°C:

$\bar{X} = 18.6724$. $\delta = 0.0882$ and n = 5.

(i) At $\alpha = 0.01$, $t_{0.01/2} = t_{0.005} = 4.604$ at the degree of freedom = n -1= 5-1 = 4.

$$t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}} = \frac{4.604x\ 0.0882}{\sqrt{5}} = 0.18160.$$

$$\bar{X} - t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}} < \mu < \bar{X} + t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}}$$

$$18.6724 - 0.1816 < \mu < 18.6724 + 0.1816$$

$$18.4908 < \mu < 18.854$$

(ii) At $\alpha = 0.05$, $t_{0.05/2} = t_{0.025} = 2.776$ at the degree of freedom = n -1= 5-1 = 4.

$$t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}} = \frac{2.776x\ 0.0882}{\sqrt{5}} = 0.10950$$

$$\bar{X} - t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}} < \mu < \bar{X} + t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}}$$

$$18.6724 - 0.10950 < \mu < 18.6724 + 0.10950$$

$18.5629 < \mu < 18.7819$

Firing temperature of 1000°C:

$\bar{X} = 25.407$. $\delta = 0.072$ and n = 5.

(i) At $\alpha = 0.01$, $t_{0.01/2} = t_{0.005} = 4.604$ at the degree of freedom = n -1= 5-1 = 4.

$$t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}} = \frac{4.604x\ 0.072}{\sqrt{5}} = 0.14825$$

$$\bar{X} - t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}} < \mu < \bar{X} + t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}}$$

$$25.407 - 0.14825 < \mu < 25.407 + 0.14825$$

$$25.25875 < \mu < 25.55525$$

(ii) At $\alpha = 0.05$, $t_{0.05/2} = t_{0.025} = 2.776$ at the degree of freedom = n -1= 5-1 = 4.

$$t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}} = \frac{2.776x\ 0.072}{\sqrt{5}} = 0.089385$$

$$\bar{X} - t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}} < \mu < \bar{X} + t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}}$$

$$25.407 - 0.089385 < \mu < 25.407 + 0.089385$$

$$25.31761 < \mu < 25.49639$$

**The relationship between compressive strength and firing temperature of Ntawo ball clay.**

Holding time 50 minutes.

$\bar{X} = 12.861$. $\delta = 0.08597$ and n = 6.

(iii) At $\alpha = 0.01$, $t_{0.01/2} = t_{0.005} = 4.032$ at the degree of freedom = n -1= 6-1 = 5.

$$t_{\alpha/2} \text{ x } \frac{\delta}{\sqrt{n}} = \frac{4.032x\ 0.08597}{\sqrt{6}} = 0.14151.$$

$\bar{X}$ - $t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$ $< \mu < \bar{X} + t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$

$12.861 - 0.14151 < \mu < 12.861 + 0.14151$

$12.71949 < \mu < 13.00251$

(iv) At $\alpha = 0.05$, $t_{0.05/2}$ = $t_{0.025}$ = 2.571 at the degree of freedom = n -1= 6-1 = 5.

$t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$ = $\frac{2.571x\ 0.08597}{\sqrt{6}}$ = 0.09023

$\bar{X}$ - $t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$ $< \mu < \bar{X} + t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$

$12.861 - 0.09023 < \mu < 12.861 + 0.09023$

$12.77077 < \mu < 12.95123$

Holding time of 60 minutes:

$\bar{X} = 12.8853$. $\delta = 0.06053$ and n = 6.

(iii) At $\alpha = 0.01$, $t_{0.01/2}$ = $t_{0.005}$ = 4.032 at the degree of freedom = n -1= 6-1 = 5.

$t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$ = $\frac{4.032x\ 0.06053}{\sqrt{6}}$ = 0.09964

$\bar{X}$ - $t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$ $< \mu < \bar{X} + t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$

$12.8853 - 0.09964 < \mu < 12.8853 + 0.09964$

$12.78566 < \mu < 12.98494$

(iv) At $\alpha = 0.05$, $t_{0.05/2}$ = $t_{0.025}$ = 2.571 at the degree of freedom = n -1= 6-1 = 5.

$t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$ = $\frac{2.571x\ 0.06053}{\sqrt{6}}$ = 0.06353

$\bar{X}$ - $t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$ $< \mu < \bar{X} + t_{\alpha/2}$ x $\frac{\delta}{\sqrt{n}}$

$12.8853 - 0.06353 < \mu < 12.8853 + 0.06353$

$12.82177 < \mu < 12.94883$

# APPENDIX C

## VALUES OF BREAKING FORCE OF SAMPLES FIRED FOR VARYING HOLDING TIMES

Breaking force of samples fired to 500°C

| Holding time /min | Breaking force /kN | | | | | Mean breaking force, F/ kN | Standard deviation. |
|---|---|---|---|---|---|---|---|
| | F1 | F2 | F3 | F4 | F5 | | |
| 20 | 6.8 | 7.0 | 6.8 | 7.0 | 6.8 | 6.83 | 0.833 |
| 30 | 6.8 | 7.0 | 7.0 | 6.8 | 7.0 | 6.92 | 0.098 |
| 40 | 7.0 | 7.2 | 7.2 | 7.0 | 7.0 | 7.08 | 0.098 |
| 50 | 7.0 | 7.2 | 7.2 | 7.2 | 7.0 | 7.12 | 0.098 |
| 60 | 7.2 | 7.2 | 7.2 | 7.2 | 7.0 | 7.16 | 0.080 |

**Breaking force of samples fired to 600°C**

| Holding time/min | Breaking force / kN | | | | | Mean breaking force, F /kN | Standard deviation. |
|---|---|---|---|---|---|---|---|
| | F1 | F2 | F3 | F4 | F5 | | |
| 20 | 8.2 | 8.2 | 8.2 | 8.0 | 8.2 | 8.16 | 0.080 |
| 30 | 8.2 | 8.2 | 8.4 | 8.2 | 8.2 | 8.20 | 0.815 |
| 40 | 8.4 | 8.2 | 8.2 | 8.2 | 8.4 | 8.25 | 0.711 |
| 50 | 8.4 | 8.4 | 8.2 | 8.2 | 8.4 | 8.30 | 0.585 |
| 60 | 8.4 | 8.4 | 8.4 | 8.2 | 8.4 | 8.32 | 0.821 |

Breaking force of samples fired to 700°C

| Holding time / min | Breaking force / kN | | | | | Mean breaking force, F / KN | Standard deviation. |
|---|---|---|---|---|---|---|---|
| | F1 | F2 | F3 | F4 | F5 | | |
| 20 | 9.6 | 9.6 | 9.6 | 9.6 | 9.6 | 9.60 | 0.000 |
| 30 | 9.6 | 9.6 | 9.6 | 9.8 | 9.6 | 9.64 | 0.080 |
| 40 | 9.6 | 9.8 | 9.6 | 9.8 | 9.6 | 9.68 | 0.098 |
| 50 | 9.8 | 9.6 | 9.8 | 9.6 | 9.8 | 9.72 | 0.098 |
| 60 | 9.8 | 9.8 | 9.8 | 9.6 | 9.8 | 9.76 | 0.080 |

Breaking force of samples fired to 800°C

| Holding time / min | Breaking force / kN | | | | | Mean breaking force, F / kN | Standard deviation. |
|---|---|---|---|---|---|---|---|
| | F1 | F2 | F3 | F4 | F5 | | |
| 20 | 11.0 | 11.0 | 11.0 | 11.0 | 11.0 | 11.00 | 0.000 |
| 30 | 11.0 | 11.0 | 11.0 | 11.2 | 11.2 | 11.08 | 0.098 |
| 40 | 11.2 | 11.2 | 11.2 | 11.0 | 11.0 | 11.12 | 0.098 |
| 50 | 11.2 | 11.0 | 11.2 | 11.2 | 11.2 | 11.16 | 0.080 |
| 60 | 11.2 | 11.2 | 11.2 | 11.2 | 11.2 | 11.20 | 0.000 |

Breaking force of samples fired to 900°C

| Holding time /min | Breaking force / kN | | | | | Mean breaking force, F / kN | Standard deviation. |
|---|---|---|---|---|---|---|---|
| | F1 | F2 | F3 | F4 | F5 | | |
| 20 | 20.4 | 20.6 | 20.4 | 20.6 | 20.4 | 20.48 | 0.098 |
| 30 | 20.4 | 20.6 | 20.4 | 20.6 | 20.6 | 20.52 | 0.098 |
| 40 | 20.6 | 20.6 | 20.6 | 20.6 | 20.4 | 20.56 | 0.080 |
| 50 | 20.6 | 20.6 | 20.6 | 20.4 | 20.6 | 20.56 | 0.080 |
| 60 | 20.6 | 20.4 | 20.6 | 20.6 | 20.6 | 20.56 | 0.080 |

**Breaking force of samples fired to 1000°C**

| **Holding time /min** | **Breaking force /kN** | | | | | **Mean breaking force, F /kN** | **Standard deviation.** |
|---|---|---|---|---|---|---|---|
| | **F1** | **F2** | **F3** | **F4** | **F5** | | |
| 20 | 27.8 | 27.8 | 28.0 | 27.8 | 28.0 | 27.88 | 0.098 |
| 30 | 27.8 | 27.8 | 28.0 | 28.0 | 28.0 | 27.92 | 0.098 |
| 40 | 28.0 | 27.8 | 28.0 | 28.0 | 28.0 | 27.96 | 0.080 |
| 50 | 28.0 | 28.0 | 28.0 | 28.0 | 28.0 | 28.0 | 0.000 |
| 60 | 28.0 | 28.0 | 28.0 | 28.0 | 28.0 | 28.0 | 0.000 |

## APPENDIX D

## VALUES OF COMPRESSIVE STRENGTH AND VARIANCES OF SAMPLES FIRED FOR VARYING HOLDING TIMES

**Compressive Strength and Variances of samples fired to 500°C**

| Holding time/min | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$/ MPa | Average Variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 20 | 6.18 | 6.36 | 6.18 | 6.36 | 6.18 | 6.252 | |
| | 0.005184 | 0.011664 | 0.005184 | 0.011664 | 0.005184 | | 0.00972 |
| 30 | 6.18 | 6.36 | 6.36 | 6.18 | 6.36 | 6.288 | |
| | 0.011664 | 0.005184 | 0.005184 | 0.011664 | 0.005184 | | 0.00972 |
| 40 | 6.36 | 6.55 | 6.55 | 6.36 | 6.36 | 6.436 | |
| | 0.005776 | 0.012996 | 0.012996 | 0.005776 | 0.005776 | | 0.01083 |
| 50 | 6.36 | 6.55 | 6.55 | 6.55 | 6.36 | 6.474 | |
| | 0.012996 | 0.005776 | 0.005776 | 0.005776 | 0.012996 | | 0.01083 |
| 60 | 6.55 | 6.55 | 6.55 | 6.55 | 6.36 | 6.512 | |
| | 0.001444 | 0.001444 | 0.001444 | 0.001444 | 0.023104 | | 0.00722 |
| Grand total | | | | | | | 0.04832 |
| Average variance within samples | | | | | | | **0.009664** |

**Compressive Strength and Variances of samples fired at 600°C**

| Holding time/min | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$/ MPa | Average Variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 20 | 7.45 | 7.45 | 7.45 | 7.27 | 7.45 | 7.414 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.020736 | 0.001296 | | 0.00648 |
| 30 | 7.45 | 7.45 | 7.64 | 7.45 | 7.45 | 7.488 | |
| | 0.001444 | 0.001444 | 0.023104 | 0.001444 | 0.001444 | | 0.00722 |
| 40 | 7.64 | 7.45 | 7.45 | 7.45 | 7.64 | 7.526 | |
| | 0.012996 | 0.005776 | 0.005776 | 0.005776 | 0.012996 | | 0.01083 |
| 50 | 7.64 | 7.64 | 7.45 | 7.45 | 7.64 | 7.564 | |
| | 0.005776 | 0.005776 | 0.012996 | 0.012996 | 0.005776 | | 0.01083 |
| 60 | 7.64 | 7.64 | 7.64 | 7.45 | 7.64 | 7.602 | |
| | 0.001444 | 0.001444 | 0.001444 | 0.001444 | 0.023104 | | 0.00181 |
| Grand total | | | | | | | 0.03717 |
| Average variance within samples | | | | | | | **0.00743** |

**Compressive Strength and Variances of samples fired at 700°C**

| Holding time/min | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$/ MPa | Average Variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 20 | 8.73 | 8.73 | 8.73 | 8.73 | 8.73 | 8.730 | |
| | 0.000000 | 0.000000 | 0.000000 | 0.000000 | 0.000000 | | 0.000000 |
| 30 | 8.73 | 8.73 | 8.73 | 8.91 | 8.73 | 8.766 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.02073 | 0.001296 | | 0.006480 |
| 40 | 8.73 | 8.91 | 8.73 | 8.91 | 8.73 | 8.802 | |
| | 0.005184 | 0.011664 | 0.005184 | 0.011664 | 0.005184 | | 0.007128 |
| 50 | 8.91 | 8.73 | 8.91 | 8.73 | 8.91 | 8.838 | |
| | 0.005184 | 0.011664 | 0.005184 | 0.011664 | 0.005184 | | 0.00972 |
| 60 | 8.91 | 8.91 | 8.91 | 8.73 | 8.91 | 8.874 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.020736 | 0.001296 | | 0.00648 |
| Grand total | | | | | | | 0.029808 |
| Average variance within samples | | | | | | | **0.005962** |

**Compressive Strength and Variances of samples fired at 800°C**

| Holding time/min | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$/ MPa | Average Variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 20 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.000 | |
| | 0.000000 | 0.000000 | 0.000000 | 0.000000 | 0.000000 | | 0.000000 |
| 30 | 10.00 | 10.00 | 10.00 | 10.18 | 10.18 | 10.072 | |
| | 0.005184 | 0.005184 | 0.005184 | 0.011664 | 0.011664 | | 0.009720 |
| 40 | 10.18 | 10.18 | 10.18 | 10.00 | 10.00 | 10.108 | |
| | 0.005184 | 0.005184 | 0.005184 | 0.011664 | 0.011664 | | 0.009720 |
| 50 | 10.18 | 10.00 | 10.18 | 10.18 | 10.18 | 10.144 | |
| | 0.001296 | 0.020736 | 0.001296 | 0.001296 | 0.001296 | | 0.006480 |
| 60 | 10.18 | 10.18 | 10.18 | 10.18 | 10.18 | 10.180 | |
| | 0.000000 | 0.000000 | 0.000000 | 0.000000 | 0.000000 | | 0.000000 |
| Grand total | | | | | | | 0.025920 |
| Average variance within samples | | | | | | | **0.005184** |

**Compressive Strength and Variances of samples fired at 900°C**

| Holding time/min | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$/ MPa | Average Variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 20 | 18.55 | 18.73 | 18.55 | 18.73 | 18.55 | 18.622 | |
| | 0.005184 | 0.011664 | 0.005184 | 0.011664 | 0.005184 | | 0.009720 |
| 30 | 18.55 | 18.73 | 18.55 | 18.73 | 18.73 | 18.658 | |
| | 0.011664 | 0.005184 | 0.011664 | 0.005184 | 0.005184 | | 0.009720 |
| 40 | 18.73 | 18.73 | 18.73 | 18.73 | 18.55 | 18.694 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.001296 | 0.020736 | | 0.006480 |
| 50 | 18.73 | 18.73 | 18.73 | 18.55 | 18.73 | 18.694 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.020736 | 0,001296 | | 0.006480 |
| 60 | 18.73 | 18.55 | 18.73 | 18.73 | 18.73 | 18.694 | |
| | 0.001296 | 0.020736 | 0.001296 | 0.001296 | 0.001296 | | 0.006480 |
| Grand total | | | | | | | 0.038880 |
| Average variance within samples | | | | | | | **0.007776** |

**Compressive Strength and Variances of samples fired at 1000°C**

| Holding time/min | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$/ MPa | Average Variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 20 | 25.27 | 25.27 | 25.45 | 25.27 | 25.45 | 25.342 | |
| | 0.005184 | 0.005184 | 0.011664 | 0.005184 | 0.011664 | | 0.009720 |
| 30 | 25.27 | 25.27 | 25.45 | 25.45 | 25.45 | 25.378 | |
| | 0.011664 | 0.011664 | 0.005184 | 0.005184 | 0.005184 | | 0.009720 |
| 40 | 25.45 | 25.27 | 25.45 | 25.45 | 25.45 | 25.414 | |
| | 0.001296 | 0.020736 | 0.001296 | 0.001296 | 0.001296 | | 0.006480 |
| 50 | 25.45 | 25.45 | 25.45 | 25.45 | 25.45 | 25.450 | |
| | 0.000000 | 0.000000 | 0.000000 | 0.000000 | 0.000000 | | 0.000000 |
| 60 | 25.45 | 25.45 | 25.45 | 25.45 | 25.45 | 25.450 | |
| | 0.000000 | 0.000000 | 0.000000 | 0.000000 | 0.000000 | | 0.000000 |
| Grand total | | | | | | | 0.025920 |
| Average variance within samples | | | | | | | **0.005184** |

**Compressive Strength and variances of samples fired at 500°C**

| **Holding time/min** | **Compressive strength / MPa** | | | | | **Average compressive strength /MPa** | **Variance, $\delta^2$** |
|---|---|---|---|---|---|---|---|
| | **A** | **B** | **C** | **D** | **E** | | |
| 20 | 6.18 | 6.36 | 6.18 | 6.36 | 6.18 | 6.252 | 0.0197 |
| 30 | 6.18 | 6.36 | 6.36 | 6.18 | 6.36 | 6.288 | 0.0109 |
| 40 | 6.36 | 6.55 | 6.55 | 6.36 | 6.36 | 6.436 | 0.0019 |
| 50 | 6.36 | 6.55 | 6.55 | 6.55 | 6.36 | 6.474 | 0.0067 |
| 60 | 6.55 | 6.55 | 6.55 | 6.55 | 6.36 | 6.512 | 0.0143 |
| Grand average | | | | | | **6.392** | **0.0134** |
| Variance within mean | | | | | | | **0.0134** |
| Variance within sample | | | | | | | **0.0097** |
| **Calculated F - value** | | | | | | | **6.9168** |

**Compressive Strength and variances of samples fired at 600°C**

| **Holding time/min** | **Compressive strength / MPa** | | | | | **Average compressive strength /MPa** | **Variance, $\delta^2$** |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 20 | 7.45 | 7.45 | 7.45 | 7.27 | 7.45 | 7.414 | 0.0110 |
| 30 | 7.45 | 7.45 | 7.64 | 7.45 | 7.45 | 7.488 | 0.0009 |
| 40 | 7.64 | 7.45 | 7.45 | 7.45 | 7.64 | 7.526 | 0.0001 |
| 50 | 7.64 | 7.64 | 7.45 | 7.45 | 7.64 | 7.564 | 0.0020 |
| 60 | 7.64 | 7.64 | 7.64 | 7.45 | 7.64 | 7.602 | 0.0069 |
| Grand average | | | | | | 7.519 | 0.0209 |
| Variance within mean | | | | | | | 0.1047 |
| Variance within sample | | | | | | | 0.0074 |
| Calculated F - value | | | | | | | **14.0918** |

**Compressive Strength and variances of samples fired at 700°C**

| **Holding time/min** | **Compressive strength / MPa** | | | | | **Average compressive strength /MPa** | **Variance $\delta^2$** |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 20 | 8.73 | 8.73 | 8.73 | 8.73 | 8.73 | 8.730 | 0.0117 |
| 30 | 8.73 | 8.73 | 8.73 | 8.91 | 8.73 | 8.766 | 0.0207 |
| 40 | 8.73 | 8.91 | 8.73 | 8.91 | 8.73 | 8.802 | 0.0324 |
| 50 | 8.91 | 8.73 | 8.91 | 8.73 | 8.91 | 8.838 | 0.0467 |
| 60 | 8.91 | 8.91 | 8.91 | 8.73 | 8.91 | 8.874 | 0.0635 |
| Grand average | | | | | | 8.622 | 0.0350 |
| Variance within mean | | | | | | | 0.1750 |
| Variance within sample | | | | | | | 0.0060 |
| Calculated F - value | | | | | | | **29.3478** |

**Compressive Strength and variances of samples fired at 800°C**

| **Holding time/min** | **Compressive strength / MPa** | | | | | **Average compressive strength /MPa** | **Variance $\delta^2$** |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 20 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.000 | 0.0102 |
| 30 | 10,00 | 10.00 | 10.00 | 10.18 | 10.18 | 10.072 | 0.0008 |
| 40 | 10.18 | 10.18 | 10.18 | 10.00 | 10.00 | 10.108 | 0.0001 |
| 50 | 10.18 | 10.00 | 10.18 | 10.18 | 10.18 | 10.144 | 0.0019 |
| 60 | 10.18 | 10.18 | 10.18 | 10.18 | 10.18 | 10.180 | 0.0063 |
| Grand average | | | | | | 10.101 | 0.0038 |
| Variance within mean | | | | | | | 0.0192 |
| Variance within sample | | | | | | | 0.0052 |
| Calculated F - value | | | | | | | **3.6999** |

Compressive Strength and variances of samples fired at 800°C

| **Holding time/min** | **Compressive strength / MPa** | | | | | **Average compressive strength /MPa** | **Variance $\delta^2$** |
|---|---|---|---|---|---|---|---|
| | **A** | **B** | **C** | **D** | **E** | | |
| 20 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.000 | 0.0102 |
| 30 | 10.00 | 10.00 | 10.00 | 10.18 | 10.18 | 10.072 | 0.0008 |
| 40 | 10.18 | 10.18 | 10.18 | 10.00 | 10.00 | 10.108 | 0.0001 |
| 50 | 10.18 | 10.00 | 10.18 | 10.18 | 10.18 | 10.144 | 0.0019 |
| 60 | 10.18 | 10.18 | 10.18 | 10.18 | 10.18 | 10.180 | 0.0063 |
| Grand average | | | | | | 10.101 | 0.0038 |
| Variance within mean | | | | | | | 0.0192 |
| Variance within sample | | | | | | | 0.0052 |
| Calculated F - value | | | | | | | **3.6999** |

Compressive Strength and variances of samples fired at 900°C

| **Holding time/min** | **Compressive strength / MPa** | | | | | **Average compressive strength /MPa** | **Variance $\delta^2$** |
|---|---|---|---|---|---|---|---|
| | **A** | **B** | **C** | **D** | **E** | | |
| 20 | 18.55 | 18.73 | 18.55 | 12.73 | 18.55 | 18.622 | 0.0025 |
| 30 | 18.55 | 18.73 | 18.55 | 18.73 | 18.73 | 18.658 | 0.0002 |
| 40 | 18.73 | 18.73 | 18.73 | 18.73 | 18.55 | 18.694 | 0.0005 |
| 50 | 18.73 | 18.73 | 18.73 | 18.55 | 18.73 | 18.694 | 0.0005 |
| 60 | 18.73 | 18.55 | 18.73 | 18.73 | 18.73 | 18.694 | 0.0005 |
| Grand average | | | | | | 18.672 | 0.0008 |
| Variance within mean | | | | | | | 0.0041 |
| Variance within sample | | | | | | | 0.0078 |
| Calculated F - value | | | | | | | **0.5333** |

**Compressive Strength and variance of samples fired at 1000°C**

| **Holding time/min** | **Compressive strength / MPa** | | | | | **Average compressive strength /MPa** | **Variance $\delta^2$** |
|---|---|---|---|---|---|---|---|
| | **A** | **B** | **C** | **D** | **E** | | |
| 20 | 25.27 | 25.27 | 25.45 | 25.27 | 25.45 | 25.342 | 0.0042 |
| 30 | 25.27 | 25.27 | 25.45 | 25.45 | 25.45 | 25.378 | 0.0008 |
| 40 | 25.45 | 25.27 | 25.45 | 25.45 | 25.45 | 25.414 | 0.0001 |
| 50 | 25.45 | 25.45 | 25.45 | 25.45 | 25.45 | 25.450 | 0.0019 |
| 60 | 25.45 | 25.45 | 25.45 | 25.45 | 25.45 | 25.450 | 0.0019 |
| Grand average | | | | | | 25.407 | 0.0018 |
| Variance within mean | | | | | | | 0.0088 |
| Variance within sample | | | | | | | 0.0052 |
| Calculated F - value | | | | | | | **1.7000** |

## APPENDIX E

## VALUES OF COMPRESSIVE STRENGTH AND VARIANCES OF SAMPLES FIRED TO DIFFERENT FIRING TEMPERATURES

### Compressive Strength and variances of samples fired for Holding Time 20 minutes

| Firing temperature /°C | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$ /MPa | Average variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 500 | 6.18 | 6.36 | 6,18 | 6.36 | 6.18 | 6.252 | |
| | 0.005184 | 0.011664 | 0.005184 | 0.011664 | 0.005184 | | 0.00972 |
| 600 | 7.45 | 7.45 | 7.45 | 7.27 | 7.45 | 7.414 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.020736 | 0.001296 | | 0.00648 |
| 700 | 8.73 | 8.73 | 8.73 | 8.73 | 8.73 | 8.730 | |
| | 0.00000 | 0.00000 | 0.00000 | 0.00000 | 0.00000 | | 0.00000 |
| 800 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.000 | |
| | 0.00000 | 0.00000 | 0.00000 | 0.00000 | 0.00000 | | 0.00000 |
| 900 | 18.55 | 18.73 | 18.55 | 18.73 | 18.55 | 18.622 | |
| | 0.005184 | 0.011664 | 0.005184 | 0.011664 | 0.005184 | | 0.00972 |
| 1000 | 25.27 | 25.27 | 25.45 | 25.27 | 25.45 | 25.342 | |
| | 0.005184 | 0.005184 | 0.011664 | 0.005184 | 0.011664 | | 0.00972 |
| Grand total | | | | | | | 0.03564 |
| Average variance within samples | | | | | | | **0.00594** |

### Compressive Strength and variances of samples fired for Holding Time 30 minutes

| Firing temperature /°C | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$ /MPa | Average variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 500 | 6.18 | 6.36 | 6.36 | 6.18 | 6.36 | 6.288 | |
| | 0.011664 | 0.005184 | 0.005184 | 0.011664 | 0.005184 | | 0.00972 |
| 600 | 7.45 | 7.45 | 7.64 | 7.45 | 7.45 | 7.488 | |
| | 0.001444 | 0.001444 | 0.023104 | 0.001444 | 0.001444 | | 0.00722 |
| 700 | 8.73 | 8.73 | 8.73 | 8.91 | 8.73 | 8.766 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.02073 | 0.001296 | | 0.00648 |
| 800 | 10.00 | 10.00 | 10.00 | 10.18 | 10.18 | 10.072 | |
| | 0.005184 | 0.005184 | 0.005184 | 0.011664 | 0.011664 | | 0.00972 |
| 900 | 18.55 | 18.73 | 18.55 | 18.73 | 18.73 | 18.658 | |
| | 0.011664 | 0.005184 | 0.011664 | 0.005184 | 0.005184 | | 0.00972 |
| 1000 | 25.27 | 25.27 | 25.45 | 25.45 | 25.45 | 25.378 | |
| | 0.011664 | 0.011664 | 0.005184 | 0.005184 | 0.005184 | | 0.00972 |
| Grand total | | | | | | | 0.05258 |
| Average variance within samples | | | | | | | **0.008763** |

**Compressive Strength and variances of samples fired for Holding Time 40 minutes**

| Firing temperature /°C | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$ /MPa | Average variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 500 | 6.36 | 6.55 | 6.55 | 6.36 | 6.36 | 6.436 | |
| | 0.005776 | 0.012996 | 0.012996 | 0.005776 | 0.005776 | | 0.010830 |
| 600 | 7.64 | 7.45 | 7.45 | 7.45 | 7.64 | 7.526 | |
| | 0.012996 | 0.005776 | 0.005776 | 0.005776 | 0.012996 | | 0.010830 |
| 700 | 8.73 | 8.91 | 8.73 | 8.91 | 8.73 | 8.802 | |
| | 0.005184 | 0.011664 | 0.005184 | 0.011664 | 0.005184 | | 0.007128 |
| 800 | 10.18 | 10.18 | 10.18 | 10.00 | 10.00 | 10.108 | |
| | 0.005184 | 0.005184 | 0.005184 | 0.011664 | 0.011664 | | 0.009720 |
| 900 | 18.73 | 18.73 | 18.73 | 18.73 | 18.55 | 18.694 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.001296 | 0.020736 | | 0.006480 |
| 1000 | 25.45 | 25.27 | 25.45 | 25.45 | 25.45 | 25.414 | |
| | 0.001296 | 0.020736 | 0.001296 | 0.001296 | 0.001296 | | 0.006480 |
| Grand total | | | | | | | 0.051468 |
| Average variance within samples | | | | | | | **0.008578** |

**Compressive Strength and variances of samples fired for Holding Time 50 minutes**

| Firing temperature /°C | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$ /MPa | Average variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 500 | 6.36 | 6.55 | 6.55 | 6.55 | 6.36 | 6.474 | |
| | 0.012996 | 0.005776 | 0.005776 | 0.005776 | 0.012996 | | 0.01083 |
| 600 | 7.64 | 7.64 | 7.45 | 7.45 | 7.64 | 7.564 | |
| | 0.005776 | 0.005776 | 0.012996 | 0.012996 | 0.005776 | | 0.01083 |
| 700 | 8.91 | 8.73 | 8.91 | 8.73 | 8.91 | 8.838 | |
| | 0.005184 | 0.011664 | 0.005184 | 0.011664 | 0.005184 | | 0.00972 |
| 800 | 10.18 | 10.00 | 10.18 | 10.18 | 10.18 | 10.144 | |
| | 0.001296 | 0.020736 | 0.001296 | 0.001296 | 0.001296 | | 0.00648 |
| 900 | 18.73 | 18.73 | 18.73 | 18.55 | 18.73 | 18.694 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.020736 | 0.001296 | | 0.00648 |
| 1000 | 25.45 | 25.45 | 25.45 | 25.45 | 25.45 | 25.450 | |
| | 0.000000 | 0.000000 | 0.000000 | 0.000000 | 0.000000 | | 0.00000 |
| Grand total | | | | | | | 0.04434 |
| Average variance within samples | | | | | | | **0.00739** |

**Compressive Strength and variances of samples fired for Holding Time 60 minutes**

| Firing temperature /°C | Compressive strength / MPa | | | | | Average compressive strength $\bar{X}$ /MPa | Average variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 500 | 6.55 | 6.55 | 6.55 | 6.55 | 6.36 | 6.512 | |
| | 0.001444 | 0.001444 | 0.001444 | 0.001444 | 0.023104 | | 0.007220 |
| 600 | 7.64 | 7.64 | 7.64 | 7.45 | 7.64 | 7.602 | |
| | 0.001444 | 0.001444 | 0.001444 | 0.001444 | 0.023104 | | 0.001805 |
| 700 | 8.91 | 8.91 | 8.91 | 8.73 | 8.91 | 8.874 | |
| | 0.001296 | 0.001296 | 0.001296 | 0.020736 | 0.001296 | | 0.006480 |
| 800 | 10.18 | 10.18 | 10.18 | 10.18 | 10.18 | 10.180 | |
| | 0.000000 | 0.000000 | 0.000000 | 0.000000 | 0.000000 | | 0.000000 |
| 900 | 18.73 | 18.55 | 18.73 | 18.73 | 18.73 | 18.694 | |
| | 0.001296 | 0.020736 | 0.001296 | 0.001296 | 0.001296 | | 0.006480 |
| 1000 | 25.45 | 25.45 | 25.45 | 25.45 | 25.45 | 25.450 | |
| | 0.000000 | 0.000000 | 0.000000 | 0.000000 | 0.000000 | | 0.000000 |
| Grand total | | | | | | | 0.021985 |
| Average variance within samples | | | | | | | **0.003664** |

**Compressive Strength and variances of samples fired for Holding Time 20 minutes**

| **Firing temperature /°C** | **Compressive strength /MPa** | | | | | **Average compressive strength $\bar{X}$ /MPa** | **Average variance $\delta^2$** |
|---|---|---|---|---|---|---|---|
| | **A** | **B** | **C** | **D** | **E** | | |
| 500 | 6.18 | 6.36 | 6.18 | 6.36 | 6.18 | 6.252 | 41.92174 |
| 600 | 7.45 | 7.45 | 7.45 | 7.27 | 7.45 | 7.414 | 28.22478 |
| 700 | 8.73 | 8.73 | 8.73 | 8.73 | 8.73 | 8.730 | 15.97361 |
| 800 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.000 | 7.43489 |
| 900 | 18.55 | 18.73 | 18.55 | 18.73 | 18.55 | 18.622 | 34.75456 |
| 1000 | 25.27 | 25.27 | 25.45 | 25.27 | 25.45 | 25.342 | 154.14579 |
| Grand average | | | | | | 12.727 | 47.90923 |
| Variance with in mean | | | | | | | 287.45537 |
| Variance with in sample | | | | | | | 0.00594 |
| Calculated F- value | | | | | | | **48393.15993** |

**Compressive Strength and variances of samples fired for Holding Time 30 minutes**

| Firing temperature /°C | Compressive strength /MPa | | | | | Average compressive strength $\overline{X}$ /MPa | Average variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 500 | 6.18 | 6.36 | 6.36 | 6.18 | 6.36 | 6.288 | 42.081169 |
| 600 | 7.45 | 7.45 | 7.64 | 7.45 | 7.45 | 7.488 | 27.952369 |
| 700 | 8.73 | 8.73 | 8.73 | 8.91 | 8.73 | 8.766 | 16.072081 |
| 800 | 10.00 | 10.00 | 10.00 | 10.18 | 10.18 | 10.072 | 7.306209 |
| 900 | 18.55 | 18.73 | 18.55 | 18.73 | 18.73 | 18.658 | 34.60969 |
| 1000 | 25.27 | 25.27 | 25.45 | 25.45 | 25.45 | 25.378 | 158.83561 |
| Grand average | | | | | | 12.775 | 47.80952 |
| Variance within mean | | | | | | | 286.85713 |
| Variance within sample | | | | | | | 0.008763 |
| Calculated F- value | | | | | | | **32735.03663** |

**Compressive Strength and variances of samples fired for Holding Time 40 minutes**

| Firing temperature /°C | Compressive strength /MPa | | | | | Average compressive strength $\overline{X}$ /MPa | Average variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 500 | 6.36 | 6.55 | 6.55 | 6.36 | 6.36 | 6.436 | 40.88324 |
| 600 | 7.64 | 7.45 | 7.45 | 7.45 | 7.64 | 7.526 | 28.13242 |
| 700 | 8.73 | 8.91 | 8.73 | 8.91 | 8.73 | 8.802 | 16.22478 |
| 800 | 10.18 | 10.18 | 10.18 | 10.00 | 10.00 | 10.108 | 7.40928 |
| 900 | 18.73 | 18.73 | 18.73 | 18.73 | 18.55 | 18.694 | 34.38650 |
| 1000 | 25.45 | 25.27 | 25.45 | 25.45 | 25.45 | 25.414 | 158.35706 |
| Grand average | | | | | | 12.830 | 47.56554 |
| Variance within mean | | | | | | | 285.39327 |
| Variance within sample | | | | | | | 0.008578 |
| Calculated F- value | | | | | | | **33270.37445** |

**Compressive Strength and variances of samples fired for Holding Time 50 minutes**

| Firing temperature /°C | Compressive strength /MPa | | | | | Average compressive strength $\overline{X}$ /MPa | Average variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 500 | 6.36 | 6.55 | 6.55 | 6.55 | 6.36 | 6.474 | 40.79377 |
| 600 | 7.64 | 7.64 | 7.45 | 7.45 | 7.64 | 7.564 | 28.05821 |
| 700 | 8.91 | 8.73 | 8.91 | 8.73 | 8.91 | 8.838 | 16.18453 |
| 800 | 10.18 | 10.00 | 10.18 | 10.18 | 10.81 | 10.144 | 7.38209 |
| 900 | 18.73 | 18.73 | 18.73 | 18.55 | 18.73 | 18.694 | 34.02389 |
| 1000 | 25.45 | 25.45 | 25.45 | 25.45 | 25.45 | 25.450 | 158.48292 |
| Grand average | | | | | | 12.861 | 47.48757 |
| Variance within mean | | | | | | | 284.92541 |
| Variance within sample | | | | | | | 0.00739 |
| Calculated F- value | | | | | | | **38555.53532** |

**Compressive Strength and variances of samples fired for Holding Time 60 minutes**

| Firing temperature /°C | Compressive strength /MPa | | | | | Average compressive strength $\overline{X}$ /MPa | Average variance $\delta^2$ |
|---|---|---|---|---|---|---|---|
| | A | B | C | D | E | | |
| 500 | 6.55 | 6.55 | 6.55 | 6.55 | 6.36 | 6.512 | 40.61895 |
| 600 | 7.64 | 7.64 | 7.64 | 7.45 | 7.64 | 7.602 | 27.91326 |
| 700 | 8.91 | 8.91 | 8.91 | 8.73 | 8.91 | 8.874 | 16.09053 |
| 800 | 10.81 | 10.18 | 10.18 | 10.18 | 10.18 | 10.180 | 7.31865 |
| 900 | 18.73 | 18.55 | 18.73 | 18.73 | 18.73 | 18.694 | 33.74100 |
| 1000 | 25.45 | 25.45 | 25.45 | 25.45 | 25.45 | 25.450 | 157.89169 |
| Grand average | | | | | | 12.885 | 47.25901 |
| Variance within mean | | | | | | | 283.55407 |
| Variance within sample | | | | | | | 0.0036642 |
| Calculated F- value | | | | | | | **77385.701** |