INVESTIGATION OF THE COMPRESSIVE STRENGTH OF NTAWO BALL CLAY FIRED TO DIFFERENT TEMPERATURES AT DIFFERENT RATES OF TEMPERATURE RISE

BY

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DECLARATION

I, Byekwaso Ronald do hereby declare that this dissertation contains my original work and it has not been submitted to any academic institution for any academic award.

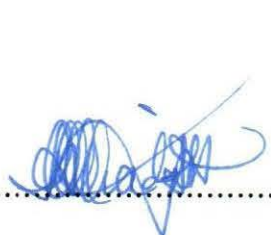Signed: ....

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APPROVAL

This dissertation by Byekwaso Ronald was developed and carried out under our supervision and is hereby cleared for submission to the Graduate Board and Senate of Kyambogo University with our due approval.

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DEDICATION

I dedicate this work to all my family members.

ACKNOWLEDGEMENT

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ABSTRACT

This study was to investigate the compressive strength of fired Ntawo Ball clay found in Mukono District in Central Uganda. The clay sample was first analyzed using X-ray Diffraction Spectrometer Model RIX3000 to determine the relative abundance of the chemical components and then compressive strengths were determined.

Ball clay of particle size 45.0 μm was used to produce 150 rectangular clay slabs fired in an electric furnace, model P5910. They were then subjected to compressive strength test by first determining the breaking force for each sample using a compressive test machine, compact 500Sn-12994917 on samples fired at different rates of temperature rise, ranging between $2°Cmin^{-1}$ and $6°Cmin^{-1}$ in intervals of $1°Cmin^{-1}$ for set firing temperatures. The compressive strength tests were then carried out on another set of samples fired at set rates of temperature rise to different firing temperatures ranging between $500°C$ and $1000°C$ in intervals of $100°C$. The compressive strength of fired Ntawo ball clay was found to decrease with increase in rates of temperature rise between $2°Cmin^{-1}$ and $6°Cmin^{-1}$ for the six firing temperatures. For temperatures of $500°C$ to $1000°C$ in intervals of $100°C$, the percentage decrease in compressive strength were 11%, 3%, 3%, 8%, 2.9%, and 11% respectively. At a level of significance $\alpha = 0.05$ and $\alpha = 0.01$, it was established that compressive strength was related to the rate of temperature rise. The study further revealed that compressive strength increased with increase in firing temperature. The percentage increase was 279%, 273%, 247%, 257% and 279% for the rates of temperature rise between $2°Cmin^{-1}$ and $6°Cmin^{-1}$ respectively, overall average percentage increase being 267%. At a level of significance $\alpha = 0.05$ and $\alpha = 0.01$, compressive strength depended on firing temperature. Ntawo clay was found to be composed of mainly silica (67.20%), alumina (18.20%), ferrous oxides (2.83%) and other fluxing oxides such as CaO (0.31%), K_2O (0.98%), and Na_2O (0.19%), among other compounds. The compressive strength of fired samples decreased with increase in rates of temperature rise during firing between $500°C$ and $1000°C$. Other investigations should be made to find out the effect of firing beyond $1000°C$ and firing at a rate above $6°Cmin^{-1}$ on the compressive strength, and the effect of changing the mineralogical composition of clay on the compressive strength.

CHAPTER ONE: INTRODUCTION

1.1 Background of the Study

Clay is a fine-grained natural rock or soil material that combines one or more clay minerals with traces of metal oxides and organic matter (Guggenheim & Martin, 2016). Clay has the property of plasticity when wet, making it to be easily molded. About the mineralogical composition, clay is composed of minerals that exhibit plasticity. There are several groups of chemical compounds that make up clay minerals. The finest sizes of the grain size typically smaller than 2 μm, are referred to as clay sizes only for purposes of definition (Guggenheim & Martin, 2016).

Clay minerals typically form over long periods of time as a result of the gradual chemical weathering of rocks, usually silicate-bearing, by low concentrations of carbonic acid and other diluted solvents. These acidic solvents attack and weather rock after leaching through upper weathered layers. The decomposition of igneous rocks such as granite, 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. Feldspar is the least stable of minerals when exposed to the action of water and air. In addition to the weathering process, some clay minerals are formed through hydrothermal activity.

There are two types of clay deposits: primary and secondary. Primary clays form as residual deposits in soil and remain at the site of formation. Secondary clays are clays that have been transported from their original location by water erosion and deposited in a new sedimentary deposit. Clay deposits are typically associated with very low energy depositional environments such as large lakes and marine basins (Keaves, 2015).

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 (Meland & Norrman, 2010). 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 (Tigue, 2001).

Deposits of ball clay are normally found in plains and valleys or along rivers. In Uganda, ball clay is normally found in Kajjansi, Wakiso District, Ntawo in Mukono District, and Kamonkoli in Budaka District. Other mostly pronounced areas 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 (McGrill, 1965).

Clay has been in use since the beginnings of civilization, for making cooking pots, bricks, porcelain, and also drainage pipes. Both brick clays and other clays are used for other purposes, such as the manufacture of clay pipes, and for floor and wall tiles. Fireclays are used for more refractory purposes such as heat-resistant tiles or bricks (Scarre, 2005). Ball clays from China clay, predominantly kaolinite, is used in ceramics, as a filer and in drug manufacture. Expanded clays are used as a lightweight aggregate in the manufacture of expanded clay blocks used for insulation. However, the major use of clay, after brick manufacture, is in the manufacture of cement (Benny & Theng, 2006). Highly absorbent, bentonite is much used in foundry work for facing the moulds and preparing the moulding sands for casting metals. The less absorbent bentonites are used chiefly in the oil industry as filtering and deodorizing agents in the refining of petroleum and mixed with other

materials as drilling muds to protect the cutting bit while drilling. Other uses include making of fillers, sizings, dressings, construction, purifying water and wine; purifying sewage, paper, ceramics, plastics, and rubber industries. On the other hand, clay is also used as medicine since prehistoric times. The Armenian bole, which used to soothe an upset stomach, is a good example. Kaolin clay and attapulgite have been used as anti-diarrheal medicines (Grim & Ralph, 2016).

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, namely: wall tiles, roof tiles ,floor tiles, wash basins, toilet bowls, plates, cups and saucers ,linoleum, acoustic ceiling tiles, insulated electrical cables, pale colored 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 also used in many industrial processes such as paper making, cement production and chemical filtering. Fired clay exhibits high melting point, high mechanical strength, porosity and water absorption when fired at high temperatures. It is resistant to chemical reactions, in particular situation in which it is used. Fired clays have dense structure with low permeability at high temperatures. It displays a high strength, lower porosity and water absorption. Other properties for fired ball clay include: clay and its mixture, molding of clay and shrinkage test. Clay and its mixture is very important because the quality of clay and water to be added for proper plasticity and consistency for moulding clay articles can be found. Safe drinking water is an essential requirement in production of clay articles due to its purity that does not change the properties of prepared materials. The source rock gives rise to clay minerals with very fine particle size - giving high green strength - and a pale ivory to buff colour.

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 (Wilson, 2008).

Various researchers have investigated the properties of fired clay made from other clay deposits. Nyakairu, et al., (1998) 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.

1.2 Statement of the Problem

. The Ntawo ball clay with a very high percentage of minerals responsible for mechanical strength of fired ball clay has not been explored. Fewer studies have been done on mechanical properties of ball clay from Ntawo compared to studies done on chemical composition. There is scanty information documented on the mechanical properties of ball clay from Ntawo deposit.

1.3 Purpose of the Study

This study investigated the compressive strength of Ntawo ball clay fired to different temperatures at varying rates of temperature rise.

1.4 Objectives of the Study

In order to achieve the purpose of the study, the following objectives were set;

(i) To find out the major chemical composition of Ntawo ball clay.

(ii) To determine the compressive strength of Ntawo ball clay for different rates of temperature rise fired to different temperatures.

(iii) To determine the compressive strength of Ntawo ball clay fired to different temperatures at different rates of temperature rise.

(iv) To establish the relationship between compressive strength of Ntawo ball clay and the rate of temperature rise during firing, when fired to given temperatures.

(v) To establish the relationship between the compressive strength of Ntawo ball clay and firing temperature, at set rate of temperature rise.

1.5 Research hypotheses

To determine the extent to which compressive strength was related with rate of temperature rise and firing temperature the following null hypotheses were set to be tested at $\alpha = 0.01$ and $\alpha = 0.05$.

(i) HO_1- The compressive strength of Ntawo ball clay does not vary with the rate of temperature rise when fired to set temperatures.

(ii) $H0_2$- The compressive strength of Ntawo ball clay does not vary with the

temperature at which it is fired at set rate of temperature rise.

1.6 Scope of the Study

The study was limited to the use of ball clay from Ntawo ball clay deposits in Mukono District in Uganda. It was limited to only two variables; firing temperature and rate of temperature rise. The focus was on six different firing temperatures of 500°C, 600°C, 700°C, 800°C, 900°C and 1000°C; and five different rates of temperature rise of; $2°Cmin^{-1}$, $3°Cmin^{-1}$, $4°Cmin^{-1}$, $5°Cmin^{-1}$ and $6°Cmin^{-1}$. The holding time was maintained at 60 min for all the samples. The sieve of mesh size of 45 μm was used to sieve clay particle size for the samples. Compressive strength was the only dependent variable. A total of 150 samples were considered for the tests carried out under the different conditions.

Other variables considered in this study were; sample shrinkage, porosity, water absorption, and density of the sample. The study was designed to generate results to test two null hypotheses namely: **H0₁:** The compressive strength of Ntawo ball clay did not vary with the rate of temperature rise when samples were fired to set temperatures, and **H0₂:** The compressive strength of Ntawo ball clay did not vary with the temperature at which it was fired at set rate of temperature rise.

1.7 Significance of the Study

The ball clay from Ntawo contained silica (67.20%), alumina (18.20%), ferrous oxides (2.83%) and other fluxing oxides such as CaO (0.31%), K_2O (0.98%), MgO (0.36%) and Na_2O (0.19%), by percentage weight. The values were in range with those as investigated by Nyakairu (2001). The compressive strength of the fired clay samples decreased with increase in the rate of temperature rise between $2°Cmin^{-1}$ and $6°Cmin^{-1}$ for each of the fixed firing temperatures between 500°C and 1000°C. The percentage decrease in compressive strength was 11%, 3%, 3%, 8%, 2.9%, and 11% for the rates of temperature rise between $2°Cmin^{-1}$ and $6°Cmin^{-1}$ respectively. Therefore, the null hypothesis $H0_1$ set was significant. The relationship between compressive strength and firing temperature of the samples at fixed rates of temperature rise was also significant. The highest value of compressive strength was 28.11MPa at firing temperature of 1000°C for the rate of temperature rise of $2°Cmin^{-1}$. The compressive strength of the fired samples increased significantly with increase in firing temperature from 500°C and 1000°C in intervals of 100°C for each rate of temperature rise. The percentage increase in compressive strength was 279%, 273%, 247%, 257% and 279% for the set rates of temperature rise of $2°Cmin^{-1}$, $3°Cmin^{-1}$, $4°Cmin^{-1}$, $5°Cmin^{-1}$, and $6°Cmin^{-1}$, respectively. Therefore, the null hypothesis $H0_2$ was rejected at $\alpha = 0.05$ and $\alpha = 0.01$ level of significance.

CHAPTER TWO: REVIEW OF RELATED LITERATURE

2.1 Introduction

The literature mainly focused on mineralogical composition of clay, classification of clay, effect of firing clay bodies, and compressive strength of fired clay materials. Many studies have found out that compressive strength of fired clay depends on firing temperature, rate of temperature rise, among other factors. The literature review explores more on issues related to different aspects of the study objectives such as: Clay and clay types, clay mineralogical composition, forming methods of clay, firing of the clay material, rate of temperature rise, Compressive strength of fired clay body, water absorption and porosity of fired clay.

2.2 Mineralogical Composition of Clay

Clay is composed of compounds referred to as clay minerals. They are chemical compounds of different elements that include oxides (Wilson, 2008). The types of mineral content in clay determine the quality of fired clay products in terms of its strength. Clays are mostly composed of Silica and alumina with varying amounts of metallic oxides and other minor ingredients. Some of the metallic oxides in clay bodies such as calcium, magnesium, sodium, and potassium oxides act as fluxes to promote fusion of clay particles at lower temperatures (Keaves, 2015). They influence the temperatures at which materials that contain fluxes vitrify, and give heated clay products necessary strength for structural purposes. Varying amount of iron, calcium and magnesium oxides also influence the colour of fired clay products. Clays form flat hexagonal sheets similar to micas. Clay minerals are common weathering products (including weathering of feldspar) and low-temperature hydrothermal alteration products. They are very common in soils, in fine-grained sedimentary rocks such as shale, mudstone, and siltstone, and in fine-grained metamorphic slate and phyllite.

Clay is a fine-grained natural rock or soil material that combines one or more clay minerals with traces of metal oxides and organic matter (Guggenheim & Martin, 2010). Geologic clay deposits are mostly composed of phyllosilicate minerals containing variable amounts of water trapped in the mineral structure (Keiliors & Mones, 2016). In clays, tetrahedral sheets are always bonded to octahedral sheets formed from small cations such as; aluminium or magnesium, and coordinated by six oxygen atoms. The unshared vertex from tetrahedral sheet also forms part of one side of the octahedral sheet but an additional oxygen atom is located above the gap in tetrahedral sheet at the center of six tetrahedra. This oxygen atom is bonded to a hydrogen atom forming an OH group in a clay structure. Clays can be categorized depending on the layers tetrahedral and octahedral sheets are packaged into. If there is only one tetrahedral and one octahedral group in each layer, the clay is known as 1:1 clay. The alternative known as 2:1 clay has two tetrahedral sheets, with an unshared vertex of each sheet pointing towards each other and forming each side of an octahedral sheet (Kenoya & Jonathan, 2005).

Like all phyllosilicates, clay minerals are characterised by two-dimensional sheets of corner sharing SiO_4 tetrahedra and/or AlO_4 octahedra. The sheet units have chemical composition $(Al_2Si)_3O_4$. Each silica tetrahedron shares three of its vertex oxygen atoms with other tetrahedra forming a hexagonal array in two-dimensions. The fourth vertex is not shared with another tetrahedron, and all tetrahedra "point" in the same direction; i.e. all unshared vertices are on same side of the sheet (Wall & Mike, 2013).

The mineralogical composition of clay may vary within a quarry as well as with depth of exploration. Therefore, it requires continuous testing of the raw clay materials for mineralogical composition. Clay minerals are usually (but not necessarily) ultrafine-grained (normally considered to be less than $2\,\mu m$ in size on standard particle size classifications) and so may require special analytical techniques for their identification and

study. These include x-ray diffraction, electron diffraction methods, various spectroscopic methods such as Mössbauer spectroscopy, infrared spectroscopy, Raman spectroscopy, and SEM-EDS or automated mineralogy processes. These methods can be augmented by polarized light microscopy, a traditional technique establishing fundamental occurrences or petrologic relationships (Agle, Brown, & Dwayne, 2013). Table 2.1 shows the proportions of different minerals in typical clay.

Table 2. 1: Common Mineral Ingredients/Oxides of Clay **[Source: (Agle, Brown, & Dwayne, 2013)]**

Ingredients/oxides	Proportions
Silica (S_iO_2)	50 - 60%
Alumina (Al_2O_3)	20 – 30%
Lime (CaO)	10%
Magnesia (MgO)	<1%
Ferric Oxide (Fe_iO_3)	<7%
Alkalis (NaOH, KoH, …)	<10%
Carbondioxide (CO_2)	Very small
Sulpher trioxide (SO_3)	Very small
Water (H_2O)	Very small

According to New Delhi (2000), the functions of ingredients or oxides in clay are; Silica (S_iO_2), which enables clay material to retain its shape and impart durability, and prevents shrinkage and warping. However, excess of silica makes clay brittle and weak on burning. If alumina (Al_2O_3), which absorbs water and renders clay plastic, is present in excess, it produces cracks in clay material on drying. Lime (CaO) that normally constitutes less than 10% of clay reduces shrinkage on drying and helps to bind the clay. In carbonated form, lime lowers the fusion point but excess of lime causes clay material to melt and lose its shape. Magnesium oxide (MgO) affects colours and makes the material yellow on burning. It causes the clay to soften at a slower rate. When excess oxygen is available, iron oxide (Fe_2O_3) gives columns on burning, and when oxygen available is insufficient, it gives dark brown or even black colour. However, excess of ferric oxide makes the

material dark blue. Iron oxide also improves impermeability, durability, and also gives strength and hardness to clay products.

Determination of mineralogical composition of clay allows for identification of unwanted materials in clay that can lead to poor quality clay products. The durability of clay bricks, for example, might be compromised by the presence of organic matter, lime modules, soluble salts and other impurities. Mineralogical analysis can provide information about firing temperature and degree of vitrification which is relevant for manufacturing clay products such as bricks. Nyakairu et al. (2001) carried out mineral and chemical compound analysis of Kaolins and clays used to formulate industrial bricks in central Uganda, and found out that the most predominant compounds were alumina (Al_2O_3) and silica (S_iO_2) with other minor minerals of Fe_2O_3, TiO_2, MgO, K_2O, MnO, CaO and P_2O_5 which have a decisive influence on the strength of final products. Mureramanzi (2010) carried out a research on village brick making in Africa and found out that the chief chemical composition of clay is silica, alumina, iron oxide, magnesia, lime and alkalis. Variation in the required amount of any of these compounds may make a substantial difference in the quality of final product especially in short cycle firing systems.

Clay is extensively found on earth where water has interacted with other minerals and organic matter (Rilem, 1984). Clay minerals have also been detected at several locations in the solar system such as on Mars including Echus Chasma and Mawrth Vallis and the Memnonia quadrangle and the Elysium quadrangle. Spectrography has confirmed presence of clays on asteroids including the dwarf planet Ceres and Tempel as well as Jupiter's moon Europa (Nortion, 1970).

Bonding between the tetrahedral and octahedral sheets requires that the tetrahedral sheet becomes corrugated or twisted; causing ditrigonal distortion to the hexagonal array, and the octahedral sheet is flattened. This minimizes the overall bond-valence distortions of

the crystallite (Kerry, 2014). Depending on the composition of tetrahedral and octahedral sheets, the layer will have no charge, or will have a net negative charge. If layers are charged this charge is balanced by interlayer cations such as Na^+ or K^+. In each case the interlayer can also contain water. The crystal structure is formed from a stack of layers interspaced with the interlayers (Bowman & Hutka, 2002).

Kaolinite is a clay mineral with a soft consistency and earthy texture. It is easily broken and can be molded or shaped especially when wet. Kaolinite is a lackluster and uninteresting mineral on its own, but it occasionally forms interesting pseudomorphs especially after feldspars. It is also a common accessory to other minerals, including gem crystals in decomposing feldspar pegmatites.

The term kaolinite describes the name of a group of closely-related clay minerals, and an individual member mineral of a group. All members of the kaolinite group have same (or similar) chemical formula, and they are dickite, kaolinite, nacrite, halloysite, and odinite. kaolinite also has a very similar chemical formula to Serpentine, and is sometimes considered a member of the serpentine group. Kaolinite is the most common clay mineral, and entire clay deposits can be composed of this mineral. There are many commercial kaolinite mines where this mineral is mined in large volumes for its various industrial uses. Kaolinite is named after the Kao-ling, a mountain in Jiangxi Province in China where this mineral was well-known from early times (Ogunye & Boussabaine, 2002).

Clay mineralogy of the Amboseli Basin; Water supplied to the Amboseli Basin from Mount Kilimanjaro, the principal recharge area, has a relatively high content of Mg^{2+} and on evaporation it becomes supersaturated with respect to dolomite, sepiolite, and kerolite (Stoessell and Hay 1978). Stoessell also reported the occurrence of kerolite in the Sinya Beds, which he be at least partly an alteration product of sepiolite. Clays' highest ill alumina content are composed of hydrated aluminum oxides, and paradoxically not

inferred to of clay minerals, in the way they have been defined tentatively by China International Practice Exhibition of Architecture (CIPEA) as "hydrated silicates with layer or chain lattices (McGeary, 1961). The processes of origin of high-alumina minerals, based on first-hand observations and studies have been well documented in a number of papers (Highley & Bloodworth, 2006).

2.3 Classification of Clay Types

According to various literature, clay is classified into surface clays, shale and fire clays. Their unique properties are as listed in Table 2.2.

Table 2. 2: Types of Clay and their Properties (**Ultrone et al., 2004**)

Clay soil types	Properties
Surface clay	- Found close to the earth surface. It is the most accessible and simply mined. - Has high oxide content ranging from 10 to 25% - This types of clay are usually preferred by brick factories
Shale	- It is a metamorphic form of clay hardened and layered under natural geologic conditions - It is very dense and hard to remove from the ground than other clays and as a result is more costly. - It also contains a relatively high percentage of oxide fluxes such as CaO, Na_2O, K_2O.
Fire clay	- Found at greater depths than either surface clay or shale - It generally has fewer impurities, more uniform chemicals and physical properties and only 2-10% oxide. - The lower percentage of oxide fluxes gives fire clay 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 finances, fire places, fire liners, ovens and chimney stacks.

Most potters use surface clays containing comparatively higher oxide content. Using surface clays reduces raw material exploration cost but blending of these surface clays with other clays have lower oxide content of these raw materials within the desired limit and it adjusts their physical properties. Plasticity of clay is one of the physical characteristics which determine its workability (Kang & Joong, 2005). In clay, the three principle identified clay minerals can be characterized in terms of activity and

plasticity,these are; Montinorillonite (or smectite). Is found to have more absorption and interparticle attraction compared with others. This makes the mineral ideal for use as a drilling mud in soil exploration and in drilling oil wells. It is also commonly injected into the ground and basement walls as a water barrier to stop basement leaks. Illite clay mineral is intermediate in terms of activity. The plastic Index (PI) of pure illite ranges from about 30-50. Kaolinite is a clay mineral with the least activity. This mineral is commonly used in the ceramic industry and for brick manufacture. . The plastic index of a pure kaolinite ranges from about 15-20 (Kang & Joong, 2005).

2.4 Effect of Firing of Clay Bodies

Firing is the heating of material to elevated temperatures above 600°C. The effect of firing of clay depends on the temperature range to which it is subjected. The final results of the firing process showed that clay is converted to a new hard and rigid ceramic material that is stable (Goffer, 2007).

Firing temperatures have varying effects on different clay types and are often quite critical. During firing, clay material undergoes a physical change. Clay particles and impurities are fused together to produce a hard durable and weather resistant product by vitrification. This is usually accompanied by further shrinkage and colour change. The varying temperatures have different effects on the clay type but are generally in the range of 900°C-1200°C. Fired bricks are permanently hardened if firing is done appropriately catering for the characteristics of the materials used to make the clay products. According to Goffer (2007), the effects caused by heat on clay are as summarised in Table 2.4.

Table 2. 3: The Physical and Chemical Changes Caused by Heat on Wet Clay (Sourc: Goffer, 2007)

TEMPERATURE °C	CHANGE
Room temperature – 100	Drying or loss of water of formation.
100-500	Loss of water of plasticity.
500-600	Dehydration (loss of chemically combined water and modification of clay structure).
600 -900	Break down of clay structure and incipient vitrification.
900 -about 1700	Vitrification and formation of new structures.
Above 1700	Melting.

The most common stages of clay firing are; dehydration stage in which the firing leads to removal of water ; oxidation stage at which burning of carbonaceous material takes place and vitrification stage where the mixed and shaped clay material is irreversibly changed to a hard, glassy and durable clay material called fired clay brick. A clay body does not develop to its full strength until it has vitrified. The vitrification process starts at about 900°C and extends up to the highest temperature the clay can, without melting. Depending on the kind of clay minerals and impurities present, upper temperature limit of vitrification ranges may vary from 1000°C to 1500°C. During vitrification period, porosity of the mass decreases and its strength and hardness increases (Mazen, 2009). Cooling: This is the final stage of firing in which fired clay will be cooled.

According to the American Standard for Testing and Material (ASTM) Committee on standards (1999), the temperatures are usually in excess of 930°C. The extent of firing is a function of both time and temperature. To get good products, clay materials should not be subjected to sudden temperature rise. The firing of clay is therefore done in three stages to avoid sudden temperature rise; pre-heating to ensure total dryness of the clay material, and utilizes combustion gases in the Kiln to raise the clay temperature, firing where fuel, usually natural gas, LPG, oil or coal is used to raise and maintain temperature to the required level over a few hours, cold air is drawn into the kiln to cool clay materials slowly for sorting and packing (Kang & Joong, 2005). Firing can be done in one of the

two types of oven or kiln. A periodic kiln consists of a single, refractory- lined, sealed chamber with burner ports and electric heating elements. It can only fire one batch of ware at a time, but it is more flexible since firing cycle can be adjusted for each product. A tunnel kiln is a refractory chamber which maintains a certain temperature none continuously, with the ware being pushed from one zone to another. The ware enters a pre-firing zone, then through a firing zone at the center of a kiln before leaving through the cooling zone. This type of kiln is more economical and energy efficient than a periodic kiln.

The extent of firing should be sufficient to produce the level of these properties required by specifications for a particular product. Firing irreversibly transforms clay particles into solid bodies, accompanied by shrinkage and colour change. According to ASTMC 43-980, fired bricks are developed between particulate constituents of fired clay solely as a result of the firing process. The bond may result from fusion or melting of one or more constituents of the composition of surface or particles between 900°C to 1700°C. 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 development should be sufficient to provide the specified strength, porosity and durability for any particular product. The degree of firing depends on the duration of firing and amount of temperature used for firing. Burning is accomplished by controlled firing in Kiln to achieve fusion of clay particles and hardening of fired clay material. Since so many of the properties of fired clay depend on the method and control of firing, the development over the years of more sophisticated Kilns have been instrumental in improving the quality and durability of fired clay materials.

In addition the firing of clay brings about some colour changes at different temperatures as shown in Table 2.4 for the kiln firing chart.

Table 2. 4: Kiln Firing Chart(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		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.
	Dull red	
700		
	Dark red	
600		
	Dull red glow	
500		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. Between 480 - 700°C, chemical water (referred to as 'water smoke') is driven off.
	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.

Sintering is the process of heating and compacting a powdered material at a temperature below its melting point in order to weld the particles together into a single rigid shape. This is the stage where clay particles begin to cement themselves together to create a hard

material called bisque. It occurs between 800-900°C (www.potterymaking.org). Sintering is aimed at controlling the grain size in ceramics to make dense ceramics. Changes that occur during the firing process are related to changes in grain size and shape. Firing is aimed at maximizing the properties such as compressive strength, water absorption and porosity which has to be eliminated as much as possible (Lin, 2006). Sintering is aimed at producing sintered parts with reproducible and if possible, designed microstructure through control of sintering variables. Microstructure control means the control of grain size, sintered density, and size distribution of other phases including pores. In most cases, the final goal of microstructure control is to prepare a fully dense body with a fine grain structure (Kang, 2005). In the clay brick manufacturing process, stable initial raw materials 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 material due to the decrease or increase in the volume of the system.

Rate of temperature rise of clay materials is how fast or slow a material can be fired to reach the required temperature. It is a measure of how fast the required temperature can be reached. In general, the firing process of clay bricks at different heating rates generates a range of gas emissions into the atmosphere. In one investigation, four different heating rates were used: 0.7 °C min^{-1}, 2 °C min^{-1}, 5 °C min^{-1} and 10 °C min^{-1}. The samples were fired in solid form from room temperature to 1050 °C (Dondi M et al., 1999).

During the firing process, carbon monoxide, carbon dioxide, nitrogen oxides, hydrogen cyanide and chlorine emissions were measured at different heating rates. All bricks were also tested for their physical and mechanical properties including dry density, compressive strength, tensile strength, water absorption and initial rate of absorption. Results show that gas emissions were reduced significantly with higher heating rates (10 °C min) followed by 5 °C min^{-1} and 2 °C min^{-1}. For both types of brick samples. Higher heating rates also

decrease the compressive strength and tensile strength value but demonstrate an insignificant effect on the water absorption properties respectively. There are variables in clay that influence the moulding properties: rate of firing, type of clay mineral, soluble salts and adsorbed ions, particle shape and organic matter. For brick clays, the amount and kind of accessory minerals be indicated as an additional factor. Norton suggested that grain size, could be readily controlled by settling or centrifuging from a dilute suspension. The use of settling troughs and centrifuges makes possible a reasonable degree of mineral separation and gives a satisfactory rate of firing control (Cater, et al., 2007).

Rate of firing analysis is a reliable, reproducible technique that eliminates factors that may affect field texture such as organic matter content, clay mineralogy, cation composition and the presence of cementing agents (Bowman & Hutka, 2002). According to Nishikawa (1984), like for ceramic materials, rate of firing distribution has an influence on packing of clay. This affects flow, porosity and hence strength in clay bricks. Most inorganic building materials such as brick, stone and concrete are porous and permeable to water vapour and liquid water. It is well known that many of the durability problems in building construction are related to water transport in porous building materials.

2.5 Compressive Strength of Fired Clay Materials

Compressive strength is the capacity of a material or structure to withstand loads tending to reduce its size. It is the maximum compressive stress that, under a gradually applied load, a given solid material can sustain without fracture.

Compressive strength of clay material is remarkably improved by firing at high temperatures (Ankara, 1986). With increase in firing temperature, compressive strength increases. Sharp increase in compressive strength at 1000°C and above may be attributed to enhanced vitrification in the clay material. Increase in compressive strength is due to decrease in porosity and increase in bulk density with increasing temperature (Ultrone, et

al., 2005). The density of bricks increases as temperature decreases along with increasing compressive strength. Compressive strength is an important property of clay bricks which is used as an indicator of masonry strength and as a result, bricks strength has become an important requirement in brick work design. A considerable amount of past research and studies as Masonry indicated that stronger bricks contribute to greater backward strength. Compressive strength is the resistance to compression of a material and is measured in the laboratory by applying perpendicular load to the largest face (Dondi, et al., 1999). A wide range of fired clay such as bricks is available in this country. Bricks vary in compressive strength due to the differing qualities of raw material and the method of firing.

Modern methods of manufacture produce bricks with consistent qualities. For bricks made from natural occurring materials, the compressive strength of individual bricks in a given batch inevitably varies. Hence compressive strength of clay bricks is not always indicative of their durability. Clay products for load- bearing designs can be provided to suitable close tolerances and strength.

Table 2. 5: Approximate Compressive Strength of Bricks and Porosity[Source; Compell and James (2003)]

Type	Compressive strength, N/mm^2	Water Absorption % by mass, max
CLAY BRICKS		
Clay bricks – range	4-180	7.5
Load bearing common bricks	7-60	6.3
Engineering Class A	70.0	4.5
Engineering Class B	50.0	7.0

Mureramanzi (2010) confirmed that the heating of the brick increases its compressive strength. The compressive strength of fired clay mainly depends on the percentage of clay

particles contained in their raw materials. This contributes to the quality of burnt clay such as bricks. Although the criteria for establishing the quality of material are variable and diverse, strength of a material is the main controlling factor. Compressive strength is found to be a durability indicator of materials Ogunye, (2002). Bricks that pass the durability test must also have an ample strength, low permeability characteristics and high dry density. Other factors that affect compressive strength of fired clay include; porosity, water absorption, particle size of clay, holding time among other factors.

Porosity represents the absorption of water by immersion under vacuum (RILEM, CPC, 1984). The water absorption of a brick is defined as the weight of water taken up by a brick under a given laboratory test conditions or specification and is expressed as a percentage of the dry weight of the unit. Water absorption is a measure of available pore space and is expressed as a percentage of dry brick weight. It is affected by properties of clay, method of manufacturing and degree of firing. Water absorption capacity of clay brick affects the surface finishing of a brick- laid wall, Cultrone et. al., (2004). The brick contains pores that will allow passage of water. Due to capillary action at the pores of a brick, the pores will absorb water content from mortar that lay on bricks. The absorption of water will affect properties of mortar and thus affect the bonding of mortar between bricks. The bond between brick and mortar is largely influenced by the demand of a brick to absorb water by suction and the ability of mortar to retain water necessary for the hydration of cement. The initial rate of absorption by clay bricks should fall between a range of 0.25 and 2.05 $kg/min/m^2$ in order to form strong bond between mortar and bricks. If the initial rate of clay bricks is less than 0.25 $kg/min/m^2$, bricks do not absorb much water from the mortar and water may tend to float on the mortar. If the initial rate of absorption value is too high, too much moisture is drawn from the mortar (Hall & Hoff, 2002). If too much moisture is drawn from the mortar, the mortar may dry and harden faster than the bonds made with bricks. The bonds between mortar and bricks may not be

strong as the mortar hardens. The water absorption and porosity are an effective index for the quality and densification of clay brick.

Porosity and water absorption of clay also affect the compressive strength. The term porosity relates to the total volume of voids in a material. It is defined as the ratio of the volume of pore space to a bulk volume of a material (Hall and Hoffw, 2002). "Traditional building materials have porosities ranging between 0.4 and 20 for fired clay bricks, marble and granites respectively (Hall and Hoffw, 2002). Materials made by generating gas bubbles within the paste or during firing usually have high porosity. An example of this is Aerated Auto claved concrete block. These materials have micro structures different from those of sintered or cementitious materials. A process such as sintering act to close or isolate some of the pore space and as a result open porosity reduces because parts of the pore space become disconnected. During sintering, open and closed pores are usually formed. The minimum density corresponds to the maximum volume of closed pores in the sample. Densification is a pore- filling process that occurs during the liquid- phase flow and by pore shrinkage. If cavities or porosity are more in the matrix, specimens will exhibit less density and absorb more water.

The smaller the size of the particles, the more they fuse together to form a hard structure, which increases compressive strength of the fired clay body. The Porosity in clay material depends on the type of clay used in manufacturing and temperature of firing. According to Khalaf et al., (2002), the porosity of the fired clay influences its compressive strength, water absorption and permeability. The amount of porosity will depend on the rate of firing, size distribution of the particles and shaping mentioned.

CHAPTER THREE: METHODOLOGY OF THE STUDY

3.1 Introduction

The methodology of this study focused on determining the compressive strength of Ntawo ball Clay samples in terms of finding the relationship between the rate of temperature rise while firing Ntawo ball clay samples, and compressive strength of the fired samples and the firing temperature with compressive strength. The compressive strength of fired samples was determined using the compact 500Sn12994917 and the heating of samples was done using the craft furnace model P5910. The research design, sample preparation, determination of chemical ingredients of Ntawo Ball Clay, firing of clay samples, determination of average breaking force of samples, and determination of compressive strength of the fired samples fired to different temperatures for different rates of temperature rise have herein been discussed.

3.2 Research Design

The research was based on descriptive and relative designs. The descriptive design focused on the compressive strength against rate of temperature rise and firing temperature. The relational design was to test the two hypotheses by using the F-values for the data for the values obtained for the different conditions. The calculated F-value was then compared with the theoretical F-values of rate of temperature rise and varying of firing temperature against compressive strength to confirm or reject the null hypotheses set. The relational design focused on comparing the relationship between the dependent variables i.e. compressive strength of the samples and the independent variables of rate of temperature rise and firing temperature. The characteristic ingredients of Ntawo ball clay were investigated. Ntawo ball clay of $45.0\,\mu m$ particle size was used to find the characteristic values of compressive strength of fired samples at specific rates of temperature rise for specific firing temperatures. This was to test the null hypothesis if

there was no difference between the compressive strength of the samples for different rates of temperature rise for all ranges of heating temperatures used. The characteristic compressive strength of the samples was also determined for each rate of temperature rise for different heating temperatures. The null hypothesis that there was no relation between compressive strength and heating temperature for a range of rates of temperature rise was also tested.

3.3 Sampling and Sample Preparation

This study was based on studying Ntawo ball clay. The ball clay was collected from Ntawo clay deposits in Mukono District, central Uganda, a well known area for clay brick making by local people. The making of Clay Bricks in Mukono started in the early 1930s up to-date. The recent study found out that Ntawo clay deposits contained high levels of ball clay.

The clay for this study was obtained from eight (8) pits. The clay was extracted from a depth of about 2m for each pit where ball clay was found. This is usual depth at which clay is extracted. Clay amounting to 40 kg was obtained from each pit. The clay from each pit was collected separately in different containers labeled A, B, C, D, E, F, G, and H. These were then transported to the laboratory where the clay in each container was separately sorted to remove stones, sand and vegetation. Equal proportions of the clay from the different containers after sorting was soaked in water and mixed thoroughly to form the clay slurry.

Sieving of this slurry was then done by pouring it into a sieve of mesh size 45.0 μm. This was achieved by assembling sieves which were connected to mechanical vibrator machine as shown in Figures 3.1;

Figure 3.1: A Stack of Sieves on a Mechanical Vibrator.

The slurry of particle size $0 - 45.0\mu m$ from the sieves was then kept for one week in the laboratory to allow some water to evaporate to form a clay paste, i.e. a range of particle sizes of the clay was used. This was then compacted into rectangular standard molds of dimensions 160 mm x 40 mm x 10 mm and left there for four days during which the excess water evaporated from the clay. The slabs were then removed from the molds using hands. A total of 160 rectangular clay slabs were produced at this stage.

Figure 3. 2: Clay in the Rectangular Molds.
The slabs were left to dry under room temperature and open air controlled room for four

weeks to ensure total drying of the slabs.

3.4 Determination of the Composition of Ntawo Ball Clay

A total of 10 dried clay slabs were then ground into a fine powder and this was used for

the chemical composition analysis of the ball clay samples. The fine clay powder sample

was then taken to Mintek Mineral Research Institute in South Africa through Uganda

Industrial research Institute for analysis of the chemical composition. Ten grams of the

clay powder was accommodated in small 30 mm diameter plastic tablets and was put in

the spectrometer for analysis. The chemical compounds in the samples were determined

by the use of X-ray diffraction (XRD) using Rigaku RIX 3000 X-ray spectrometer. The

instrument used was connected to a computer to control the set-up and for data storage.

The equipment uses the following detector crystals: LiF220, Pe, Ge and TLAP.

The sample was irradiated with x-rays. The radiation that was generated by the clay

elements were characterized by specific wave lengths and intensity which is related with

its concentration, allowing therefore their identification in the x-ray spectrum. The

elements and compounds found in this study were: Silicon Oxide (SiO_2), Aluminum Oxide

(Al$_2$O$_3$), Iron Oxide (Fe$_2$O$_3$), potassium oxide (K$_2$O), Titanium Dioxide (TiO$_2$), Sodium Oxide (Na$_2$O), Calcium Oxide (CaO), Magnesium Oxide (MgO), Phospherous Penta Oxide (P$_2$O$_5$) and Manganese Oxide (MnO).

The X-Ray spectrograph in Figure 3.4 was used to analyze the chemical elements and hence compounds in the clay sample.
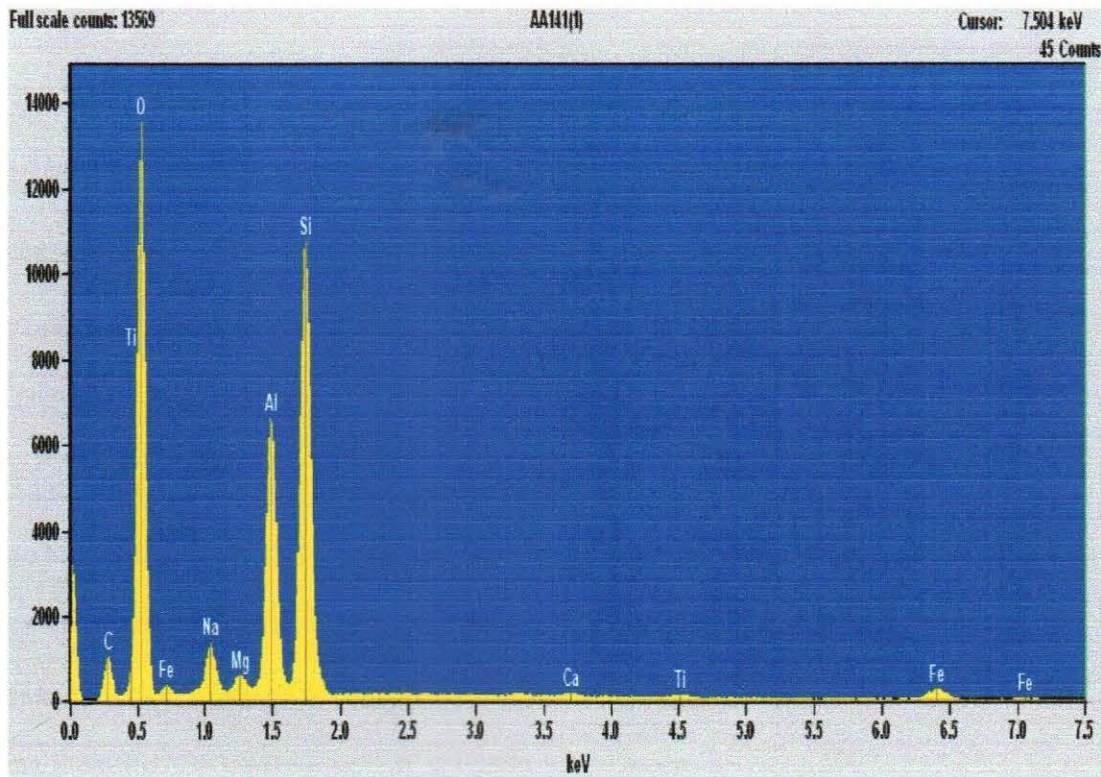
Figure 3. 3: XRD diffractogram of Natwo Ball Clay

The XRD spectrograph shown in figure 3.4 showed that Ntawo ball clay contained carbon, titanium, iron, sodium, magnesium, aluminium, oxygen, silicon, and calcium.

By analysis of the XRD spectrogram above, the relative abundances by percentage weight of the various elements was determined. Table 3.1 shows the chemical compounds and their corresponding percentage weights.

Table 3.1: Chemical Compounds in Ntawo ball clay:

COMPOUND	CHEMICAL SYMBOL	WEIGH (%)
Silica	SiO_2	67.20
Alumina	Al_2O_3	18.20
Calcium Oxide	CaO	0.31
Iron Oxide	Fe_2O_3	2.83
Potassium Oxide	K_2O	0.98
Magnesium Oxide	MgO	0.36
Sodium Oxide	Na_2O	0.19
Titanium dioxide	TiO_2	1.38
Phosphorous Penta Oxide	P_2O_5	0.06

From Table 3.1, the most predominant compound in Ntawo ball clay were; silica (67.2%), alumina (18.2%), calcium oxide (0.31%), and iron oxide, (2.8%), potassium oxide (0.98%),magnesium oxide (0.36%), sodium oxide, titanium dioxide (1.38%) and phosphorous pentaoxide (0.06%) which are generally responsible for the mechanical strength of clay materials, and plasticity of the clay.

3.5 Firing of Samples for Compressive Strength Test

One hundred fifty (150) dried clay samples were subjected to pre-heating at 110°C using an electric kiln for three hours. This was done to remove moisture left over from the drying process. This was done to ensure total dryness of the clay samples. Five samples were then fired for each of the 6 6° different temperatures between 500°C and 1000°C, at intervals of 100°C for the same holding time of 60 minutes using the craft furnace model P5910. Five samples were also fired at each of the five different rates of temperature rise of $2°Cmin^{-1}$, $3°Cmin^{-1}$, $4°Cmin^{-1}$, $5°Cmin^{-1}$ and $Cmin^{-1}$ from room temperature to each of the six different temperatures of 500°C to 1000°C.

The craft furnace (oven) used for firing the samples is shown in figure 3.6.

Figure 3. 4: Picture of an Electric kiln used for firing the Clay Samples

After firing, the hot clay samples were allowed to cool down gradually after switching off the electric kiln. The samples were then left to stay for two hours in the Kiln and then later they were removed from the Kiln after cooling.

The samples fired at different temperatures turned reddish brown because of the oxidation of Ferrous Silicate. At these temperatures, dehydration takes place and carbonaceous organic matter started to burn off (Technical memorandum No. 6, 1984). The maximum temperature was 1000°C. For each temperature such as 500°C - 1000°C to be reached, the temperature of Kiln or furnace was gradually increased from room temperature. This prevents bloating of the clay due to formation of an impermeable vitrified outer skin which prevents loss of gases such as water vapor and carbon dioxide from the inside part of the Clay material (Ankara, 1986).

After firing the samples to the six different temperatures and rates of temperature rise, the fired samples were studied for their physical properties such as shrinkage, This was done

by measuring the dimensions using a vernier caliper and the cross-sectional area of the broadest face of each sample was determined. An average area of $1100mm^2$ was obtained. This was then used to determine the compressive strength of the samples in each batch. The compressive strength of different samples fired at different temperatures and different rates was determined.

3.7 Measurement of Compressive Strength of the Fired Ball Clay Samples

The compressive strength is the capacity of material or structure to withstand loads tending to reduce the size or distort it (Urbanek & Lee, 2010). The ultimate compressive strength of a material is that value of uniaxial compressive stress reached when material fails completely. The compressive strength is usually obtained experimentally by means of compressive test.

Figure 3. 5: Picture of Compressive Strength Machine

The compressive strength of the fired clay samples was calculated by determining the breaking force of the samples using the compressive test machine; compact 500 Sn-12994917. This was done first, for samples fired to different rates of temperature rise at fixed temperatures and secondly, for different firing temperatures for each of the five fixed rates of temperature rise. The breaking force for the samples was determined using compact 500 Sn-12994917 shown in figure 3.7. This was done by placing the different samples in turn in the machine and determining the compressive strength. The compressive strength was determined by dividing the applied breaking force by average cross-sectional area. The values of the average breaking force are shown the Tables 3.2 and 3.3.

Table 3. 2 : Average breaking force of samples at different firig temperatures

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

Table 3. 3 : Average breaking force of samples at different rates of temperature rise

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

The samples A, B, C, D and E were used for each of the five rates at each particular temperature and for each of the six firing temperatures. The breaking force for each

sample was determined from which the values of compressive strength were calculated and recorded in MPa to two decimal places.

Tables 3.2 and 3.3 show a summary of the average values of breaking force for the samples calculated from the five individual breaking forces at the corresponding temperatures and rates of temperature rise for all the samples. The values of the breaking force of the samples fired at the different rates of temperature rise at fixed temperatures between 500°C and 1000°C are shown in Table 3.2.

Table 3.3 also shows the values of the breaking force for the samples fired to different temperatures between 500°C and 1000°C at fixed rates of temperature rise.

For each of the six firing temperatures considered in this study, five samples A, B, C, D and E were used and the values of the breaking force were obtained corresponding to each of the rates of temperature rise. Therefore, in this experiment, a total of 150 fired samples were used. Thus, for each of the values of the average at every temperature, five samples were used. This means that for the 30 average values obtained, a total of (30 x 5) samples were used. The values of breaking force were then used for determining the compressive strength of the samples for the different conditions considered.

$$Compressive\ strength = \frac{Applied\ force}{Average\ cross-sectional\ area}$$

The machine was fitted with rectangular metallic plates of area corresponding to the area of the largest face of the fired slabs (1100 mm²) and a perpendicular force was applied to the largest face of the fired slabs in turn and this was measured for each of the samples.

The compressive strength of each sample in a batch fired at different rates and temperatures was calculated by dividing maximum compressive load on the sample by the initial cross-sectional area of the sample.

$$Compressive\ strength\ (N/mm^2) = \frac{F}{A}$$

Where F = load applied (kN)

A = Area (mm^2). The area of the base used was 1100 mm².

CHAPTER FOUR: RESULTS OF THE STUDY

4.1 Introduction

This study was designed to determine the characteristics of Ntawo ball clay in terms of the chemical composition, and in terms of compressive strength of the fired Ntawo ball clay heated at five different rates of temperature rise, and in terms of compressive strength of samples fired to six different temperatures. In the first experiment, compressive strength of the samples was determined against rate of temperature rise. This was determined for each of the six firing temperatures. It was done to test the null hypothesis that there was no difference between compressive strength of the samples and rate of temperature rise. Secondly, it was to test the null hypothesis that there was no difference between compressive strength and firing temperature.

4.2 Chemical Compounds in Ntawo Ball Clay

This was the first result of the study of the components of Ntawo clay. They were given in terms of percentage weight of each compound as determined by the XRD model RIX3000 Spectrometer. The details of the results were as shown in Table 4.1.

Table 4. 1: Chemical composition of Ntawo ball clay

COMPOUND	CHEMICAL FOMULA	WEIGHT PERCENTAGE
Silica	SiO_2	67.20
Alumina	Al_2O_3	18.20
Calcium Oxide	CaO	0.31
Iron Oxide	Fe_2O_3	2.83
Potassium Oxide	K_2O	0.98
Magnesium Oxide	MgO	0.36
Sodium Oxide	Na_2O	0.19
Titanium dioxide	TiO_2	1.38
Phosphorous Penta Oxide	P_2O_5	0.06

These results are close in value with the findings by El-Din et al., (2010) who carried out similar chemical analysis on clay and obtained the following results in percentage weight;

SiO_2 (65.20), Al_2O_3 (15.26), CaO (1.01), Fe_2O_3 (7.36), MgO (0.83), Na_2O (0.62), TiO_2.(1.92) and P_2O_5 (0.15).

4.3 Compressive Strength of Ntawo Ball Clay Fired at different Rates of Temperature rise

The compressive strength of fired Ntawo ball clay was determined against rate of temperature rise for each of the six firing temperatures. This was done to test the Null hypothesis $H0_1$ that there was no difference between the compressive strength of the fired samples and the rate of temperature rise during firing for each of the temperatures from 500°C to 1000°C at intervals of 100°C. The compressive strength at each rate of temperature rise, grand mean compressive strength, the variance within the mean, and variance within the samples were calculated in order to find the F-values at 95% and 99% confidence levels or levels of significance $\alpha = 0.05$ and $\alpha = 0.01$ respectively for each firing temperature. The theoretical F-values at 95% and 99% confidence levels were 2.8 and 4.43 respectively at the degrees of freedom of 4 and 20.

The results of the compressive strength for the firing temperature of 500°C were obtained as shown in Table 4.2.

Table 4. 2: Values of compressive strength at different rates of temperature rise of samples fired to 500°C

Rate of temperature rise/°C min^{-1}	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
2	7.41	0.051984	0.006500
3	7.38	0.039204	0.009275
4	7.37	0.035344	0.012925
5	7.13	0.002704	0.022700
6	6.62	0.315844	0.009200
Grand Mean Compressive strength	7.182	0.089016	0.012210
Overall Variance within the mean			0.44508
Calculated F-value			36.45209

The graph of compressive strength was plotted against rate of temperature rise as shown in Figure 4.1 to find the relationship for the samples when fired to 500°C.
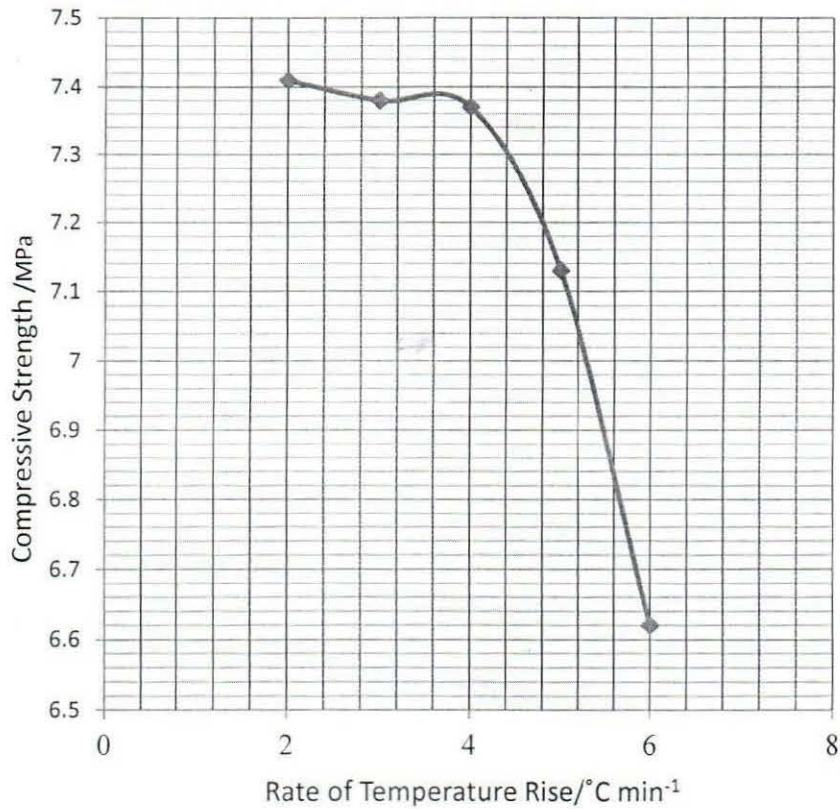
Figure 4. 1: A graph of compressive strength against rate of temperature rise for samples fired to 500°C

The graph in figure 4.1 has shown that the compressive strength of fired Ntawo ball clay decreased with increase in the rate of temperature rise from 7.41 MPa to 6.62 MPa between the rates 2°C min^{-1} and 4°C min^{-1}. There was a small change in the compressive strength from 7.41 MPa to 7.37 MPa indicating a percentage decrease in compressive strength of 2.9% for the rate of temperature rise from 2°C min^{-1} to 4°C min^{-1}. However, a sharp decrease in the compressive strength was realized between 4°C min^{-1} and 6°C min^{-1}. At temperature of 500°C, the lowest value of of compressive strength of 6.62 MPa was

attained at a rate of 6°C min^{-1}. The percentage decrease in compressive strength of 10% was noted in the compressive strength.

Since the calculated F-value (36.45209) was greater than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis $H0_1$ that there was no difference between compressive strength and rate of temperature rise was rejected.

This means that there was significant relationship between the compressive strength and rate of temperature rise for the samples fired to 500°C.

The results of the compressive strength for the firing temperature of 600°C were obtained as shown in Table 4.3.

Table 4. 3: Values of compressive strength at different rates of temperature rise of samples fired to 600°C

Rate of temperature rise/°C min^{-1}	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
2	7.55	0.007056	0.017580
3	7.56	0.008836	0.010850
4	7.53	0.004096	0.010850
5	7.38	0.007396	0.009725
6	7.31	0.024336	0.006500
Grand Mean Compressive strength	7.466	0.010344	0.001110
Overll Variance within the mean			0.05172
Calculated F-value			4.65904

The graph of compressive strength was plotted against rate of temperature rise as in Figure 4.2 to find the relationship for the samples when fired to 600°C.
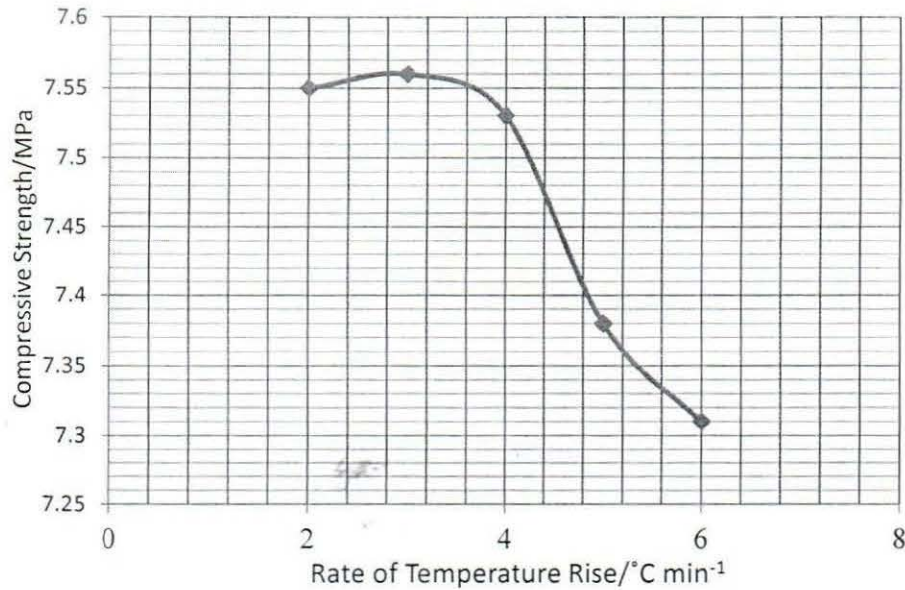
Figure 4. 2: A graph of compressive strength against rate of temperature rise for samples fired to 600°C

The graph that there was no difference between compressive strength and rate of temperature rise in figure 4.2 has shown that the compressive strength of Ntawo ball clay fired to a temperature of 600°C at the five rates of temperature rise decreased from 7.55 MPa to 7.31 MPa. Between $2°Cmin^{-1}$ and $3°Cmin^{-1}$, there was a small change in the compressive strength from 7.55 MPa to 7.56 MPa indicating a percentage change of 0.1% in compressive strength. Between $4°Cmin^{-1}$ $6°Cmin^{-1}$, there was a pronounced decrease in compressive strength from 7.53 MPa to 7. 31 MPa showing a percentage decrease of 3% in the compressive strength. The overall percentage decrease in compressive strength for the samples fired to 600°C was 3.4%.

Since the calculated F-value (4.65904) was greater than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis $H0_1$ that there was no difference between compressive strength and rate of temperature rise was rejected. This means that there was significant relationship between the compressive strength and rate of

temperature rise for the samples fired to 600°C. The results of compressive strength for the

firing temperature of 700°C were obtained as shown in Table 4.4.

Table 4. 4: Values of compressive strength at different rates of temperature rise of samples fired to 700°C

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

The graph of compressive strength was plotted against rate of temperature rise as in Figure

4.3 to find the relationship for the samples when fired to 700°C.
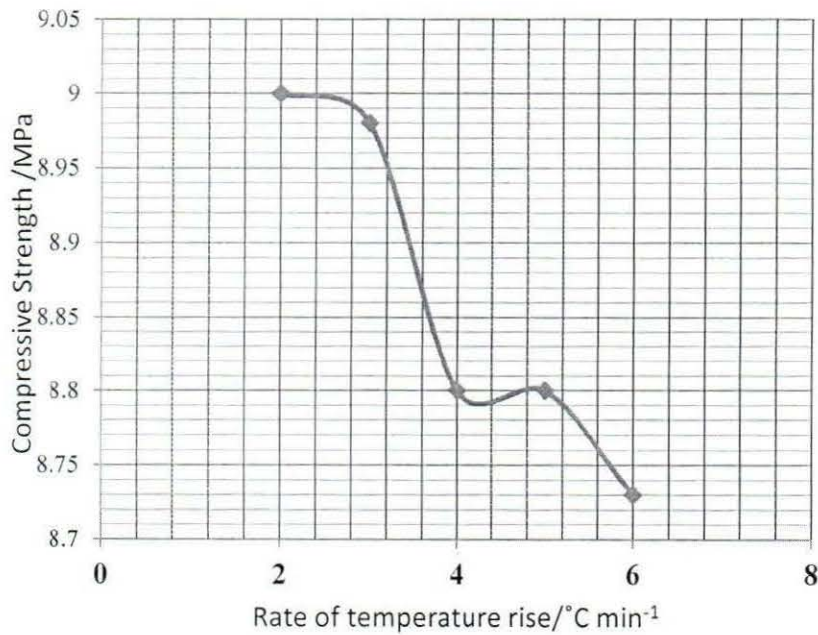
Figure 4. 3: A graph of compressive strength against rate of temperature rise for samples fired to 700°C

The graph in figure 4.3 has shown that between $2°Cmin^{-1}$ and $3°Cmin^{-1}$, there was small decrease in compressive strength of samples from 9.00 MPa to 8.98 MPa indicating a percentage decrease of 0.2% in the compressive strength. This was followed by a sharp decrease in compressive strength between $3°Cmin^{-1}$ and $4°Cmin^{-1}$ from 8.98 MPa 8.80 MPa representing a percentage decrease of 2% in compressive strength. Between $4°Cmin^{-1}$ and $5°Cmin^{-1}$ there was no change in compressive strength followed by a sharp decrease between $5°Cmin^{-1}$ and $6°Cmin^{-1}$ from 8.80 MPa to 8.73 MPa.

Since the calculated F-value (0.778552) was unexpectedly less than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis $H0_1$ that there was no difference between compressive strength and rate of temperature rise was accepted. This means that there was no significant relationship between the compressive strength and rate of temperature rise for the samples fired to 700°C. The percentage decrease in compressive strength was 3%.

The results of compressive strength for the firing temperature of 800°C were obtained as shown in Table 4.5.

Table 4. 5: Values of compressive strength at different rates of temperature rise of samples fired to 800°C

Rate of temperature rise/°C min⁻¹	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
2	11.05	0.173056	0.006500
3	10.95	0.099856	0.006500
4	10.66	0.000676	0.009425
5	10.29	0.118336	0.009725
6	10.22	0.171396	0.006500
Grand Mean Compressive strength	10.634	0.112664	0.007730
Overall Variance within the mean			0.563320
Calculated F-value			72.8745

The graph of compressive strength was plotted against rate of temperature rise as in Figure 4.4 to find the relationship for the samples when fired to 800°C.
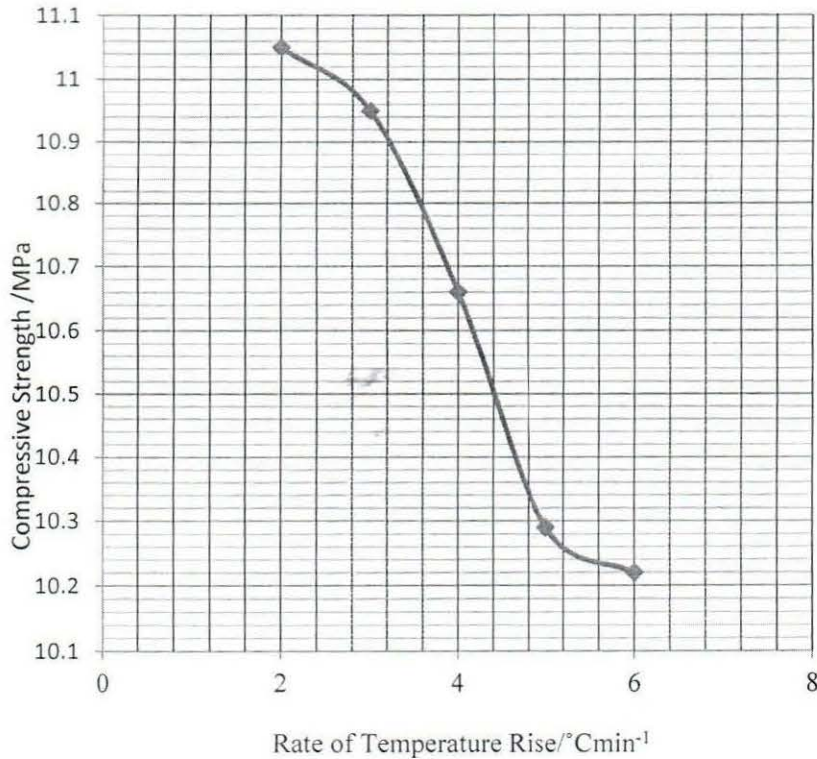
Figure 4.4: A graph of compressive strength against rate of temperature rise for samples fired to 800°C

The graph in figure 4.4 has shown that the highest value of compressive strength of 28.11 MPa was attained at a rate of 2°Cmin^{-1} for the samples fired at 800°C. A sharp decrease in compressive strength occurred between the rates of temperature rise of 2°Cmin^{-1} and 5°Cmin^{-1} from 11.05 MPa to 10.29 MPa ,indicating a percentage decrease of 7% in a compressive strength. This was followed by a gradual decrease between 5°Cmin^{-1} and 6°Cmin^{-1} from 10.29 MPa to 10.22 MPa,indicating a percentage decrease of 0.6% in the compressive strength. It should be noted that all values of compressive strength at the five rates at this temperature are higher than the corresponding values of compressive strength at other lower temperatures. The overall percentage decrease in the compressive strength for the samples fired to 800°C was 8%.

Since the calculated F-value (72.8745) was greater than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis $H0_1$ that there was no difference between compressive strength and rate of temperature rise was rejected. This means that there was significant relationship between the compressive strength and rate of temperature rise for the samples fired to 800°C.

The results of compressive strength for the firing temperature of 900°C were obtained as shown in Table 4.6.

Table 4. 6: Values of compressive strength at different rates of temperature rise of samples Fired to 900°C

Rate of temperature rise/°C min⁻¹	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
2	19.05	0.043264	0.022700
3	19.13	0.082944	0.006500
4	18.87	0.000784	0.038900
5	18.65	0.036864	0.009050
6	18.51	0.110224	0.024875
Grand Mean Compressive strength	18.842	0.054816	0.020405
Overall Variance within the mean			0.27408
Calculated F-value			13.432

The graph of compressive strength was plotted against rate of temperature rise as in figure 4.5 to find the relationship for the samples when fired to 900°C.
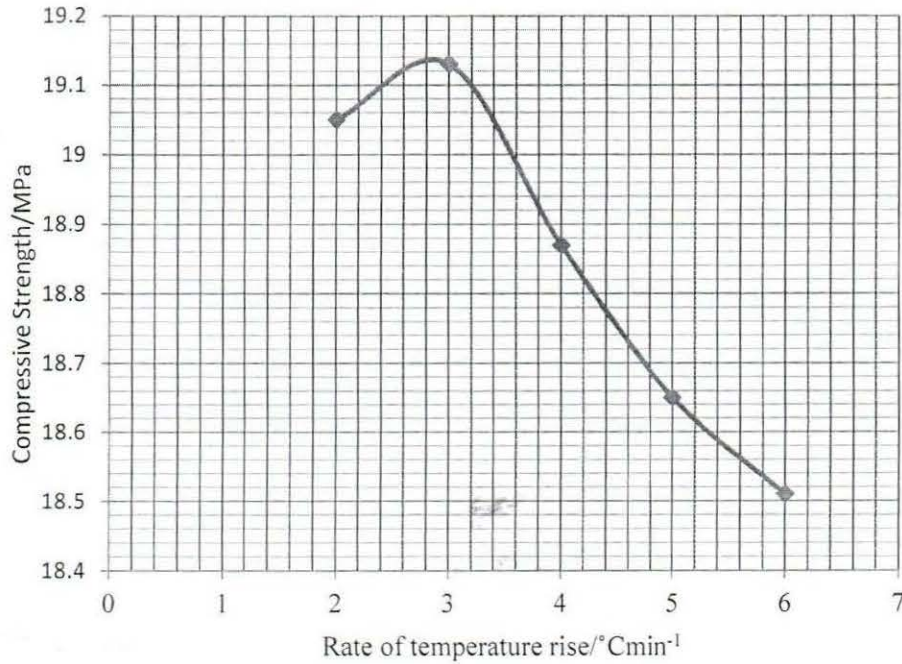
Figure 4. 5: A graph of compressive strength against rate of temperature rise for samples fired to 900°C

The graph in Figure 4.5 has shown that, the compressive strength values decreased with increase in rates of temperature rise for the samples fired to 900°C. However, there was a slight increase in compressive strength between 2°Cmin^{-1} and 3°Cmin^{-1} from19.05MPa to19.13MPa, indicating a percentage change of 0.4%. This was followed by a sharp decrease in the compressive strength from 3°Cmin^{-1} to 6°Cmin^{-1} from 19.13 MPa to 18.5MPa indicating a percentage decrease of 3.3% in the compressive strength. The highest value of compressive strength of 19.13 MPa was attained at a rate of 3°Cmin^{-1}. The overall percentage decrease in compressive strength for samples fired to 900°C was 2.9%.

Since the calculated F-value (13.432) was greater than the two theoretical F-values at 95% and 99% confidence there levels, then the null hypothesis $H0_1$ that there was no difference between compressive strength and rate of temperature rise was rejected. This means that

there was significant relationship between the compressive strength and rate of temperature rise for the samples fired to 900°C.

The results of compressive strength for the firing temperature of 1000°C were obtained as shown in Table 4.7.

Table 4. 7: Values of compressive strength at different rates of temperature rise of samples fired to 1000°C

Rate of temperature rise/°C min^{-1}	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
2	28.11	2.9929	1.014025
3	27.53	1.3225	0.770500
4	25.56	0.6724	0.882700
5	25.45	0.8649	0.165625
6	25.25	1.2769	0.393675
Grand Mean Compressive strength	26.38	1.4259	0.645305
Overall Variance within the mean			7.1296
Calculated F-value			11.04842

The graph of compressive strength was plotted against rate of temperature rise as in Figure 4.6 to find the relationship for the samples when fired to 1000°C
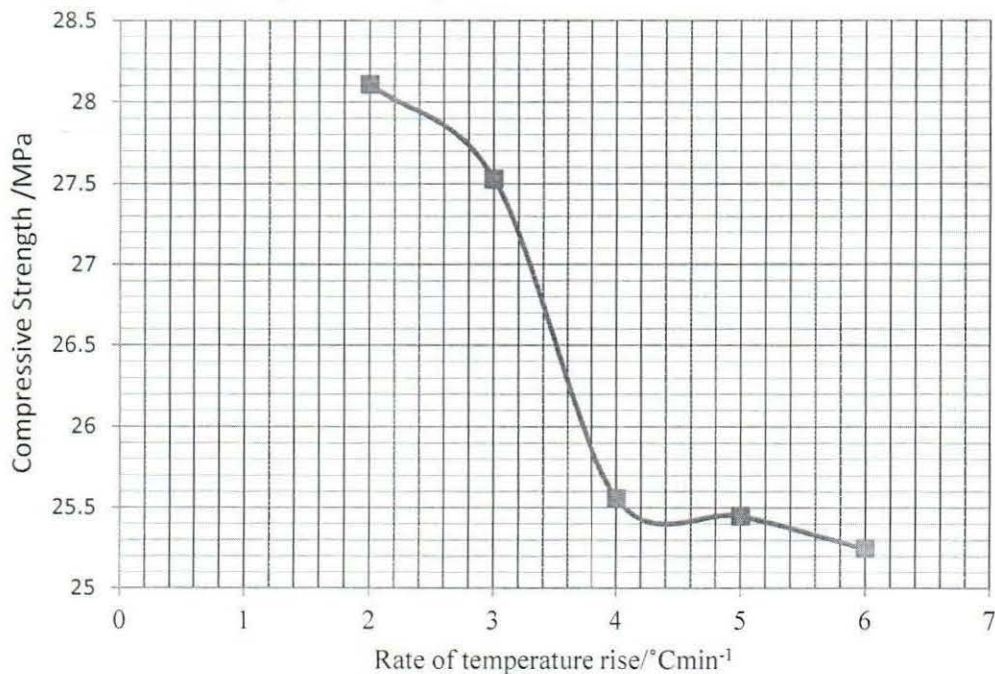
Figure 4. 6: A Graph of compressive strength against rate of temperature rise for samples fired to 1000°C

The graph in Ffigure 4.6 has shown that the highest value of compressive strength of 28.11MPa was attained at a rate of $2°Cmin^{-1}$ and the lowest value of 26.38 MPa was attained at $6°Cmin^{-1}$. There was a sharp decrease in the compressive strength between $2°Cmin^{-1}$ and $4°Cmin^{-1}$ from28.11 MPa to 25.56 MPa, which represents a percentage decrease of 9.9% This was followed by a small change in compressive strength between $4°Cmin^{-1}$ and $6°Cmin^{-1}$ from 25.56 MPa to 25.25 MPa, a percentage decrease of 1.2%. The overall percentage decrease in compressive strength of the samples fired to 1000°C was 11%.

Since the calculated F-value (11.04842) was greater than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis $H0_1$ that there was no difference between compressive strength and rate of temperature rise was rejected. This means that there was significant relationship between the compressive strength and rate of temperature rise for the samples fired to 1000°C.

In general, compressive strength of the fired samples decreased with increase in the rate of temperature rise for all the fixed temperatures between 500°C and 1000°C.

4.4 Compressive strength of Ntawo ball clay fired to different temperatures

The compressive strength of fired Ntawo ball clay was determined for each of the five rates of temperature rise. This was done to test the null hypothesis that there was no difference between the compressive strength of fired samples and firing temperature for each rates of temperature rise from $2°Cmin^{-1}$ to $6°Cmin^{-1}$.

Compressive strength at each firing temperature, grand mean compressive strength, the variance within the mean and variance within samples were calculated in order to find the F-values at 95% and 99% confidence levels or level of significance $\alpha = 0.05$ and $\alpha = 0.01$ respectively for each rate of temperature rise. The theoretical F-values at 95% and 99% confidence levels were 2.76 and 4.18 respectively at degrees of freedom of 4 and 25.

The results of compressive strength for rate of temperature rise at $2°Cmin^{-1}$ were obtained as shown in Table 4.8.

Table 4. 8: Values of average compressive strength at different firing temperatures for s fired at $2°Cmin^{-1}$

Firing temperature/°C	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
500	7.41	39.501225	0.00650
600	7.55	37.761025	0.01760
700	9.00	22.043025	0.00813
800	11.05	6.996025	0.00650
900	19.05	28.676025	0.02270
1000	28.11	207.792225	1.03403
Grand Mean	13.695	57.12825	0.18258
Overall Variance within the mean			342.76955
Calculated F-value			1877.417774

The graph of compressive strength was plotted against temperature as shown in Figure 4.7 to find the relationship for samples when fired at $2°Cmin^{-1}$.
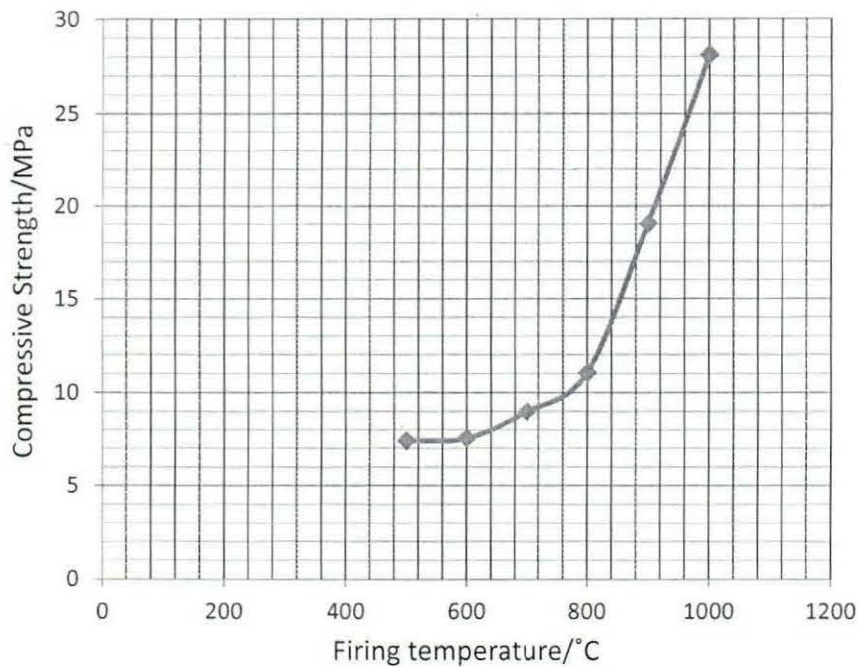
Figure 4. 7: A graph of compressive strength against firing temperature for samples fired at $2°Cmin^{-1}$

The graph in figure 4.6 has shown the compressive strength of the samples increased with increase in firing temperature initially exponentially from 500°C to 800 °C and proportionally from 800°C to 1000°C. The increase in compressive strength was from 7.41MPa to 11.05 MPa indicating a percentage increase of 49% between 500°C and 800. From 800°C to 1000°C there was a sharp increase in compressive strength from 11.05 MPa to 28.11 MPa, a percentage increase of 15.4%. This may be attributed to the sintering of the clay particles in this temperature range. The highest value of compressive strength at this rate of temperature rise was 28.11 MPa when the samples were fired to a temperature of 1000°C, and the lowest was 7.41 MPa when the samples were fired to 500°C. The overall percentage increase between 500°C and 1000°C in the compressive strength at 2°Cmin⁻¹ was 279%.

Since the calculated F-value (1877.417774) was greater than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis was rejected. This means that there was a significant relationship between compressive strength and firing temperature for the samples fired at 2°Cmin⁻¹ rate of firing temperature rise.

The results of compressive strength for rate of temperature rise at 3°Cmin⁻¹ were obtained as shown in Table 4.9.

Table 4. 9: Values of average compressive strength at different firing temperatures for Ntawo clay fired at $3°Cmin^{-1}$

Firing temperature/°C	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
500	7.38	38.539264	0.009725
600	7.56	36.336784	0.010850
700	8.98	21.233664	0.009725
800	10.95	6.959044	0.006500
900	19.13	30.713764	0.006500
1000	27.53	194.379364	0.210500
Grand Mean	13.588	54.693650	0.042300
Overall Variance within the mean			328.161884
Calculated F-value			7757.964

The graph of compressive strength was plotted against temperature as in figure 4.7 to find

the relationship for samples when fired at $3°Cmin^{-1}$.
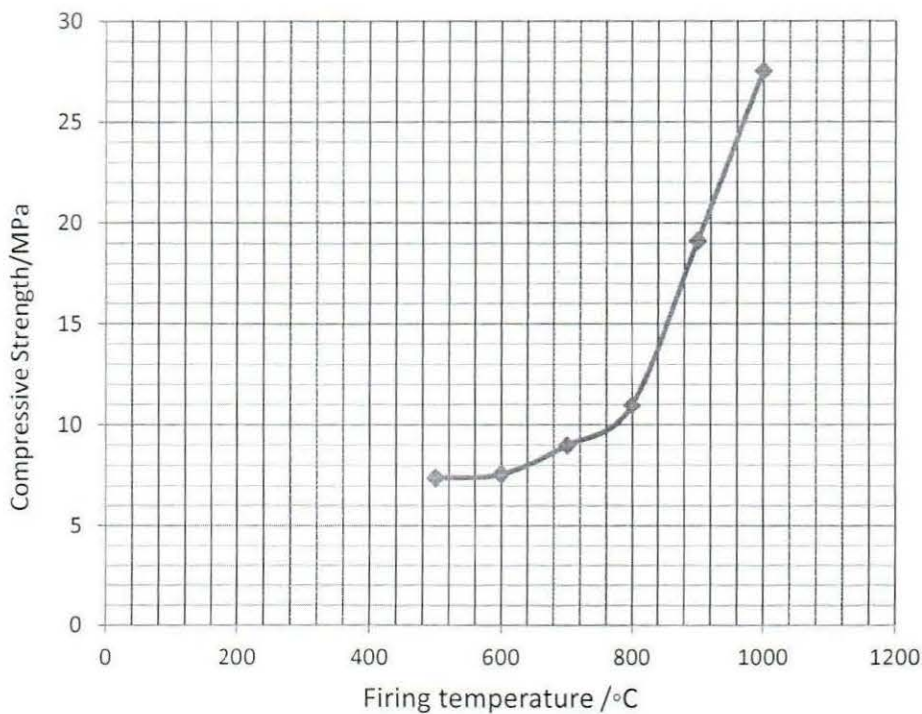
Figure 4. 8: A graph of compressive strength against firing temperature for samples fired at $3°Cmin^{-1}$

The graph in Figure 4.8 has shown that the compressive strength of the samples increased with increase in firing temperature initially exponentially from 7.38 MPa to 10.95MPa between 500°C and 800°C indicating a percentage increase of 48%, and then exponentially from 10.95 MPa to 27.52 MPa between 800°C and 1000°C indicating a percentage increase of 151%. However, at this rate, the maximum value of the compressive strength of 27.53MPa was achieved when a sample was fired to 1000°C, and it is lower than that attained at a rate of 2°Cmin^{-1} for the samples fired to the same temperature. The overall percentage increase in the compressive strength was 273%.

Since the calculated F-value (7757.964) was greater than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis that there was no difference between compressive strength of the sample and firing temperature was rejected. This means that there was a significant relationship between compressive strength and firing temperature for the samples fired at 3°Cmin^{-1} rate of firing temperature rise.

The results of compressive strength for rate of temperature rise at 4°Cmin^{-1} were obtained as shown in Table 4.10.

Table 4.10: Values of average compressive strength at different firing temperatures for samples fired at 4°Cmin^{-1}

Firing temperature/°C	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
500	7.37	33.200644	0.012925
600	7.53	31.382404	0.010850
700	8.80	18.766224	0.009725
800	10.66	6.110784	0.009425
900	18.87	32.924644	0.038900
1000	25.56	154.455184	0.882700
Grand Mean	13.132	46.139981	0.160754
Overall Variance within the mean			276.839884
Calculated F-value			1722.132

The graph of compressive strength was plotted against temperature as in figure 4.9 to find the relationship for samples when fired at $4°Cmin^{-1}$.
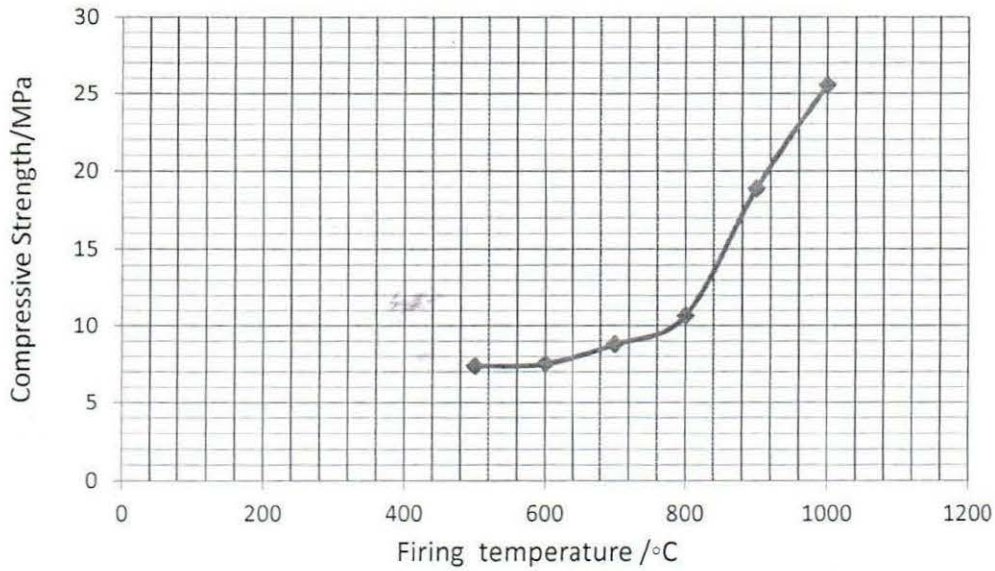
Figure 4. 9: A graph of compressive strength against firing temperature for Ntawo ball clay fired at $4°Cmin^{-1}$

The graph in Figure 4.9 has shown that the compressive strength increased as firing temperature was increased. A gradual exponential increase in compressive strength occured between 500°C and 800°C from 7.37 MPa to 10.66 MPa giving a percentage increase of 44.6% followed by a sharp proportional increase between 800°C and 1000°C from 10.66 MPs to 25.56 MPa giving a percentage increase of 140%. The sharp increase may be attributed to the sintering process of the clay particles in this temperature range. The overall percentage increase in the compressive strength of the samples fired at $4°Cmin^{-1}$ was 247%.

Since the calculated F-value (1722.132) was greater than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis that there was no difference between compressive strength of the sample and firing temperature was rejected. This

means that there was a significant relationship between compressive strength and firing temperature for the samples fired at $4°Cmin^{-1}$ rate of firing temperature rise.

The results of compressive strength for rate of temperature rise at $5°Cmin^{-1}$ were obtained as shown in Table 4.11.

Table 4. 10: Values of average compressive strength at different firing temperatures for samples fired at $5°Cmin^{-1}$

Firing temperature/°C	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
500	7.13	33.8724	0.022700
600	7.38	31.0249	0.009725
700	8.80	17.2225	0.009725
800	10.29	7.0756	0.009725
900	18.65	32.4900	0.009050
1000	25.45	156.2500	0.165625
Grand Mean	12.950	46.3227	0.037758
Overall Variance within the mean			277.9354
Calculated F-value			7360.9087

The graph of compressive strength was plotted against temperature as in Figure 4.10 to find the relationship for samples when fired at $5°Cmin^{-1}$.
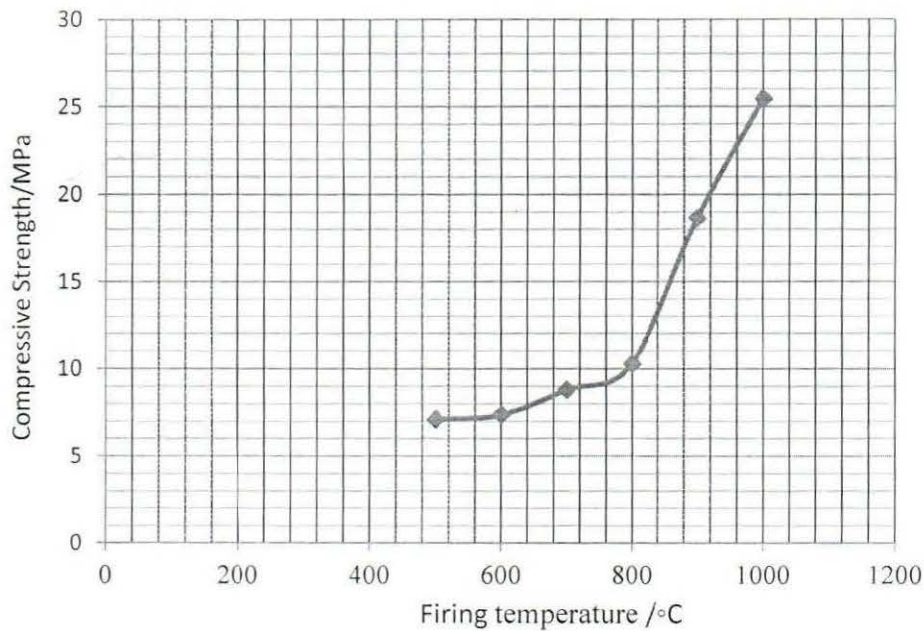
Figure 4. 10: A graph of compressive strength against firing temperature for samples fired at $5°Cmin^{-1}$

The graph in figure 4.10 has shown that the compressive strength of the samples initially increased exponentially with increase in firing temperature. The variation follows the same trend as that when the rate was $4°Cmin^{-1}$. The gradual exponential increase in compressive strength between 500°C and 800°C from 7.13 MPa to 10.29 MPa is attributed to the loss of water of plasticity, dehydration, modification of clay structure and vitrification. The percentage increase was 44%. There was a sharp proportional increase in the compressive strength between 800°C and 1000°C from 10.29 MPa to 25.45MPa indicating a percentage increase of 147% which was due to the vitrification and sintering taking place in this temperature range. The overall percentage increase in compressive strength was 257%.

Since the calculated F-value (7360.9087) was greater than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis that there was no difference between compressive strength of the sample and firing temperature was rejected. This means that there was a significant relationship between compressive strength and firing temperature for the samples fired at $5°Cmin^{-1}$ rate of firing temperature rise.

The results of compressive strength for rate of temperature rise at $6°Cmin^{-1}$ were obtained as shown in Table 4.11.

Table 4.12: Values of average compressive strength at different firing temperatures for samples fired at $6°Cmin^{-1}$.

Firing temperature/°C	Average Compressive strength/MPa	Variance, δ_m^2 within the mean	Variance, δ_x^2 within the sample
500	6.65	37.859409	0.009200
600	7.31	29.844369	0.006500
700	8.73	16.345849	0.000000
800	10.22	6.517809	0.006500
900	18.51	32.913169	0.023075
1000	25.25	155.675529	0.039368
Grand Mean	12.773	46.526020	0.073158
Overall Variance within the mean			279.156134
Calculated F-value			3815.7821

The graph of compressive strength was plotted against temperature as in figure 4.11 to find the relationship for samples when fired at $6°Cmin^{-1}$.
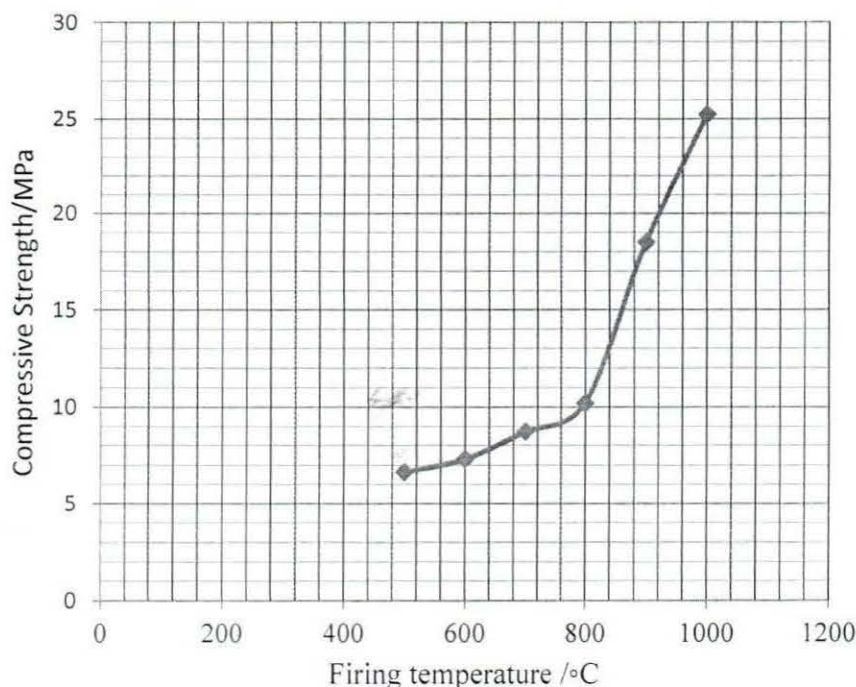
Figure 4.11: A graph of compressive strength against firing temperature for samples fired at $6°Cmin^{-1}$

The graph in Figure 4.11 has shown that the compressive strength of the sample increased exponentially from 500°C to 800°C from 6.65MPa to 10.22 MPa during the firing process, giving a percentage increase of 54% in the compressive strength. A sharp proportional increase in the compressive strength occurred between 800°C and 1000°C from 10.22 MPa to 25.25 MPa leading to a percentage increase of 147%. In this temperature range, there is cementing together of the clay particles giving rise to a hard rigid structure and this accounted for a high values of compressive strength within this temperature range. At this rate of temperature rise of $6°Cmin^{-1}$, the overall percentage increase in compressive strength was 279%.

Since the calculated F-value (3815.7821) was greater than the two theoretical F-values at 95% and 99% confidence levels, then the null hypothesis that there was no difference

between compressive strength of the sample and firing temperature was rejected. This means that there was a significant relationship between compressive strength and firing temperature for the samples fired at $6°Cmin^{-1}$ rate of firing temperature rise.

In general, increase in firing temperature of the ball clay samples during firing increases the compressive strength of the fired samples. For the samples fired to temperatures between 500°C to 1000°C in intervals of 100°C for rates of temperature rise, the average percentage increase in the compressive strength for all the five rates of temperature rise was 267%. The increase in compressive strength with firing temperature did not depend on the rate of rise of temperature.

CHAPTER 5: DISCUSSION, CONCLUSIONS, AND RECOMMENDATIONS

5.1 Discussion of Results.

During firing of clay material, a series of transformation occurs which determine the final properties of the product. The main factors often advanced that affect the property of fired clay products are the type of Clay materials, fabrication method, drying procedure, firing temperature and firing profile. The properties of durability and strength of clay material are decreased in terms of the microstructure and mineralogy of the clay products. In fired clay materials, the nature of the minerals in the clay mass has as much important influence as the heating treatment on the mechanical strength of the clay product. In this study, there were three different findings that were established with respect to the compressive strength of Ntawo clay.

The chemical composition analysis that was carried out indicated that the most predominant compounds of the Ntawo ball clay by percentage weight were; Silca (67.2%), Alumina (18.20%), Iron Oxide (2.83%), which constitutes about 90% of the compounds. These compounds are generally responsible for the mechanical strength of the clay products. The fluxing oxides such as K_2O (0.98%), CaO (0.31%) and Na_2O (0.19%) together contribute less than 2% of the minerals.

The relationship between rate of temperature rise and compressive strength of the fired Ntawo Ball Clay was found. Results obtained for the ball clay samples indicated that at any given firing temperature between 500°Cand 1000°C, the rate of temperature rise had a very significant influence on the compressive strength of the fired samples except when fired to700°C. At very low rates of temperature rise, the compressive strength of the fired samples was greater than at very high rates of temperature rise. Generally, as the rate of temperature rise increased, compressive strength of the samples reduced. This is due to the fact that at low rates of temperature rise, the rate of heat and air to penetrate the inner

layers of the clay material to burn off the organic compounds particularly carbon to produce carbondioxide and carbonmonoxide is low. In addition, the heat penetrates to remove the remaining moisture inside the clay body and this leads to the closing of the voids leading to close packing of the clay particles which results in high compressive strength of the sample. However, at very high rates of temperature rise such as $5°Cmin^{-1}$ or $6°Cmin^{-1}$, during the firing process, an impermeable layer develops on the surface of the clay body which prevents thermal conduction of heat into the inner layers of the clay material that would lead to removal of moisture and combustion of carbon compounds inside the material. The result is that the voids inside the material persist and this prevents the close packing and fusion of clay particles. The heat takes very long time to reach inner parts of the clay body. This accounted for the decrease in the compressive strength of the samples fired at very high rates of temperature rise. However, it was noted that even at the same rates, the compressive strength increased as the firing temperature was increased, for example for the temperature of $500°C$, the compressive strength of $2°Cmin^{-1}$ was 7.41 MPa and 28.11 MPa at $1000°C$. The results obtained for the relationship between compressive strength and rate of temperature rise showed that the average percentage decrease in compressive strength was 6.7%.

Firing temperature has a considerable effect on the compressive strength of the clay material. During firing, a series of transformations take place in the clay body which leads to changes in the compressive strength of the material. Between the temperatures of $500°C$ and $600°C$, there was loss of water of plasticity and dehydration leading to modification of the clay structure. At higher firing temperatures, above $800°C$, sintering took place in the material leading to compacting, cementing and fusion of clay particles together to form a single rigid mass. This led to complete elimination of voids from the mass. Therefore, the clay body becomes hard and requires a large force to break it leading to an increase in compressive strength of the fired clay.

The results obtained for the variation of firing temperature with compressive strength indicated that generally compressive strength increased with increase in firing temperature. Sharp increase in compressive strength of 147.8% was noted between the temperatures of 800°C and 1000°C for all rates of temperature rise. This was the temperature range with in which sintering took place. The average percentage increase in the compressive strength for all the rates of temperature rise considered in this study was 267%. For the firing temperatures considered in this study, the highest value of compressive strength of 28.11MPa was obtained at a firing temperature of 1000°C at a rate of temperature rise of 2°Cmin^{-1}, and the lowest compressive strength of 6.62 Mpa was obtained at a firing temperature of 500°C at a rate of temperature rise of 6°Cmin^{-1}

5.2 Conclusion

Compressive strength decreases with increase in rate of temperature rise for all temperatures except when fired to 700°C. The percentage decrease between 2.8% and 11% was noted giving an average percentage decrease of about 6.7% in the compressive strength of the samples. Therefore, there is a strong relationship between compressive strength and rate of temperature rise during firing of the samples.

The study also showed that compressive strength of the fired samples increases with increase in firing temperature for all the rates of temperature rise. The average percentage increase in compressive strength was about 267% showing that there is a strong relationship between compressive strength and firing temperature of the samples.

5.3 Recommendations

Basing on the results of this research, it is recommended that clay material should be fired to high temperatures above 800°C at very low rates of temperature rise below $2°Cmin^{-1}$ to ensure high compressive strength of the fired sample.

The research should continue with other types of soils to see if other soils can give the same results or not. This is because different soil types may have different particle sizes unlike clay which has uniform particle size and yet this also affects the mechanical properties of soil. This is because these also affect the compressive strength of the fired clay material and hence affects the quality of the products.

Other mechanical and physical properties such as color, porosity, density, weight loss, firing shrinkage, and bending strength should also be investigated for Ntawo Ball Clay. Compressive strength of Ball Clay Materials should be investigated for firing temperatures much higher than 1000°C. This is because above this temperature, very many transformations take place which may also affect the mechanical properties of the sample.

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APPENDIX A

SYMBOLS AND FORMULAE USED TO COMPUTE THE VARIOUS

QUANTITIES

1. Compressive strength $= \dfrac{Breaking\ Force}{Cross\ Sectional\ Area} = \dfrac{F}{A}$

2. Degree of Freedom

   For Numerator $= K - 1$

   For denominator $= K(n-1)$

   where K = Number of sets of samples

   n = Number of samples in each set

3. F-value $= \dfrac{Variance\ with\ in\ Mean}{Variance\ with\ in\ Sample}$

4. Grand total for the average $\bar{x}_G = \dfrac{\sum \bar{x}}{n}$

   where $\sum \bar{x}$ = sum of the average of compressive strength
   n = Number of samples

5. Variance $\sigma^2 = \bar{x} - \bar{x}_G$
   where $\bar{x}$ = mean or average of values

6. $\bar{x} = \dfrac{V Total\ number\ of\ sample\ values}{Number\ of\ samples}$

7. Variance with in samples $= \dfrac{1}{N} \sum \left[\dfrac{1}{n-1} \sum (\bar{x} - \bar{x}_G) \right]$

8. Average Variance $= \dfrac{1}{n-1} \sum (\bar{x} - \bar{x}_G)^2$

APPENDIX B

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 δ^2, then a central B% confidence interval for μ, 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.

    (a) Variation of compressive strength with rates of temperature rise of Ntawo Ball clay.

Firing temperature of 1000°C for $2°Cmin^{-1}$

$\bar{X} = 26.380$MPa. $\delta = 0.645305$ and n = 5.

    (i)   At $\alpha = 0.01$, $t_{0.01/2} = t_{0.005} = 4.43$ at the degree of freedom = n -1= 5-1 = 4.

$$t_{\alpha/2} \times \frac{\delta}{\sqrt{n}} = \frac{4.43 \times 0.645305}{\sqrt{5}} = 1.2785.$$

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

$$26.380 - 1.2785 < \mu < 26.380 + 1.2785$$

$$25.1015 < \mu < 27.6585$$

    (ii)   At $\alpha = 0.05$, $t_{0.05/2} = t_{0.025} = 2.87$ at the degree of freedom = n -1= 5-1 = 4.

$$t_{\alpha/2} \times \frac{\delta}{\sqrt{n}} = \frac{2.87 \times 0.645305}{\sqrt{5}} = 0.82825$$

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

$$26.380 - 0.82825 < \mu < 26.380 + 0.82825$$

$$25.55 < \mu < 27.20825$$

Firing temperature of 900°C at $2°Cmin^{-1}$:

$\bar{X} = 18.842$MPa. $\delta = 0.020405$ and n = 5.

    (i)   At $\alpha = 0.01$, $t_{0.01/2} = t_{0.005} = 4.43$ at the degree of freedom = n -1= 5-1 = 4.

$$t_{\alpha/2} \times \frac{\delta}{\sqrt{n}} = \frac{4.43 \times 0.020405}{\sqrt{5}} = 0.04043$$

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

$$18.842 - 0.04043 < \mu < 18.842 + 0.04043$$

$$18.80157 < \mu < 18.88243$$

(ii) At $\alpha = 0.05$, $t_{0.05/2} = t_{0.025} = 2.87$ at the degree of freedom = n -1= 5-1 = 4.

$$t_{\alpha/2} \times \frac{\delta}{\sqrt{n}} = \frac{2.87 \times 0.020405}{\sqrt{5}} = 0.02619$$

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

$$18.842 - 0.02619 < \mu < 18.842 + 0.02619$$

$$18.81581 < \mu < 18.86819$$

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

At firing temperature of 1000°C for rate of temperature rise of $2°Cmin^{-1}$:

$\bar{\bar{X}} = 13.695$. $\delta = 0.182575$ and n = 5.

(iii) At $\alpha = 0.01$, $t_{0.05/2} = t_{0.005} = 4.18$ at the degree of freedom = n -1= 6-1 = 5.
$$t_{\alpha/2} \times \frac{\delta}{\sqrt{n}} = \frac{4.18 \times 0.182575}{\sqrt{5}} = 0.341297$$

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

$$13.695 - 0.341297 < \mu < 13.695 + 0.341297$$
$$13.353703 < \mu < 14.036297$$

(iv) At $\alpha = 0.05$, $t_{0.05/2} = t_{0.025} = 2.76$ at the degree of freedom = 4.

$$t_{\alpha/2} \times \frac{\delta}{\sqrt{n}} = \frac{2.76 \times 0.182575}{\sqrt{5}} = 0.22535$$

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

$$13.46965 - 0.22535 < \mu < 13.46965 + 0.22535$$

$$13.46965 < \mu < 13.92035$$

APPENDIX C

VALUES OF BREAKING FORCE OF SAMPLES FOR DIFFERENT RATES OF

TEMPERATURE RISE

Values of breaking force of samples fired to 500°C

Rate of temperature rise/°C min^{-1}	Breaking Force/kN					
	A	B	C	D	E	Average
2	8.2	8.2	8.2	8.0	8.2	8.16
3	8.2	8.2	8.0	8.0	8.2	8.12
4	8.2	8.0	8.0	8.0	8.2	8.12
5	8.0	7.8	7.8	7.6	7.8	7.84
6	7.4	7.2	7.2	7.2	7.4	7.28

Values of breaking force of samples fired to 600°C

Rate of temperature rise/°C min^{-1}	Breaking Force/kN					
	A	B	C	D	E	Average
2	8.4	8.2	8.4	8.4	8.1	8.30
3	8.4	8.4	8.2	8.4	8.2	8.32
4	8.4	8.4	8.2	8.2	8.2	8.23
5	8.0	8.2	8.2	8.0	8.2	8.12
6	8.0	8.0	8.2	8.0	8.0	8.04

Values of breaking force of samples fired to 700°C

Rate of temperature rise/°C min^{-1}	Breaking Force/kN					
	A	B	C	D	E	Average
2	10.0	9.8	10.0	10.0	9.8	9.92
3	10.0	9.8	10.0	9.8	9.8	9.88
4	9.6	9.6	9.8	9.8	9.6	8.23
5	7.8	9.8	9.6	9.6	9.8	8.12
6	9.6	9.6	9.6	9.6	9.6	8.04

Values of breaking force of samples fired to 800°C

Rate of temperature rise/°C min⁻¹	Breaking Force/kN					
	A	B	C	D	E	Average
2	12.2	12.2	12.0	12.2	12.2	12.16
3	12.0	12.2	12.2	12.0	12.0	12.08
4	11.8	11.6	11.6	11.8	11.6	11.68
5	11.4	11.4	11.2	11.2	11.4	11.32
6	11.4	11.2	11.2	11.2	11.2	11.24

Values of breaking force of samples fired to 900°C

Rate of temperature rise/°C min⁻¹	Breaking Force/kN					
	A	B	C	D	E	Average
2	21.2	21.0	20.8	20.8	21.0	20.96
3	21.2	21.0	21.0	21.0	21.0	21.04
4	20.8	21.0	20.8	20.8	20.4	20.76
5	20.6	20.1	20.4	20.6	20.4	20.48
6	20.2	20.1	20.2	20.4	20.4	20.32

Values of breaking force of samples fired to 1000°C

Rate of temperature rise/°C min⁻¹	Breaking Force/kN					
	A	B	C	D	E	Average
2	30.6	32.2	32.0	29.8	30.0	30.9
3	29.8	30.0	29.8	29.8	32.0	30.3
4	27.8	29.8	27.0	27.0	27.6	28.0
5	23.0	28.8	27.8	27.8	27.8	26.9
6	27.8	28.8	28.0	27.4	27.6	27.9

APPENDIX D

VALUES OF COMPRESSIVE STRENGTH OF SAMPLES FIRED FOR DIFFERENT RATES OF TEMPERATURE RISE TO DIFFERENT HEATING TEMPERATURES

Values of compressive strength of samples fired to 500°C

Rate/°C min⁻¹	Compressive strength/MPa					
	A	B	C	D	E	Average
2	7.45	7.45	7.45	7.27	7.45	7.41
3	7.45	7.45	7.27	7.27	7.45	7.38
4	7.45	7.22	7.27	7.45	7.45	7.37
5	7.27	7.09	7.27	6.91	7.09	7.13
6	6.73	6.55	6.55	6.55	6.73	6.62

Values of compressive strength of samples fired to 600°C

Rate/°C min⁻¹	Compressive strength/MPa					
	A	B	C	D	E	Average
2	7.64	7.45	7.64	7.64	7.36	7.55
3	7.64	7.64	7.45	7.64	7.45	7.56
4	7.64	7.64	7.45	7.45	7.45	7.53
5	7.27	7.45	7.45	7.27	7.45	7.38
6	7.27	7.27	7.45	7.27	7.27	7.31

Values of compressive strength of samples fired to 700°C

Rate/°C min⁻¹	Compressive strength/MPa					
	A	B	C	D	E	Average
2	9.09	8.91	9.09	9.01	8.91	9.00
3	9.09	8.91	9.09	8.91	8.91	8.98
4	8.73	8.73	8.91	8.91	8.73	8.80
5	7.09	8.91	8.73	8.73	8.73	8.80
6	8.73	8.73	8.73	8.73	8.73	8.73

Values of compressive strength of samples fired to 800°C

Rate/°C min⁻¹	Compressive strength/MPa					
	A	B	C	D	E	Average
2	11.09	11.09	10.91	11.09	11.09	11.05
3	10.91	11.09	10.91	10.91	10.91	10.95
4	10.72	10.55	10.73	10.73	10.55	10.66
5	10.36	10.36	10.18	10.18	10.36	10.29
6	10.36	10.18	10.18	10.18	10.18	10.22

Values of compressive strength of samples fired to 900°C

Rate/°C min^{-1}	Compressive strength/MPa					
	A	B	C	D	E	Average
2	19.27	19.09	18.91	18.91	19.09	19.05
3	19.27	19.09	19.09	19.09	19.09	19.13
4	18.91	19.09	18.91	18.91	18.55	18.87
5	18.72	18.72	18.55	18.73	18.55	18.65
6	18.36	18.72	18.36	18.55	18.55	18.51

Values of compressive strength of samples fired to 1000°C

Rate/°C min^{-1}	Compressive strength/MPa					
	A	B	C	D	E	Average
2	27.82	29.27	29.09	27.09	27.27	28.11
3	27.09	27.27	27.09	27.09	29.09	27.53
4	25.27	27.09	25.27	24.55	25.6	25.56
5	20.91	26.18	25.27	25.27	25.27	25.45
6	25.27	26.18	24.45	24.91	25.09	25.25

APPENDIX E

VALUES OF COMPRESSIVE STRENGTH OF SAMPLES FIRED TO

DIFFERENT FIRING TEMPERATURES

Values of compressive strength of samples fired at $2°C$ min^{-1}

Firing Temperature/°C	Compressive strength/MPa					
	A	B	C	D	E	Average
500	7.45	7.45	7.45	7.27	7.45	7.41
600	7.64	7.45	7.64	7.64	7.36	7.55
700	9.09	8.91	9.09	9.01	8.91	9.00
800	11.09	11.09	10.91	11.09	11.09	11.05
900	19.27	19.09	18.91	18.91	19.09	19.05
1000	27.82	29.29	29.09	27.09	27.27	28.11

Values of compressive strength of samples fired at $3°C$ min^{-1}

Firing Temperature/°C	Compressive strength/MPa					
	A	B	C	D	E	Average
500	7.45	7.45	7.27	7.45	7.45	7.38
600	7.64	7.45	7.45	7.64	7.65	7.56
700	9.09	8.91	9.09	8.91	8.71	8.98
800	10.91	11.09	10.91	10.91	10.91	10.95
900	19.27	19.09	19.09	19.09	19.09	19.13
1000	27.09	27.27	27.09	27.09	27.09	27.53

Values of compressive strength of samples fired at $4°C$ min^{-1}

Firing Temperature/°C	Compressive strength/MPa					
	A	B	C	D	E	Average
500	7.45	7.22	7.27	7.45	7.45	7.37
600	7.64	7.64	7.45	7.45	7.45	7.53
700	8.75	8.73	8.91	8.91	8.73	8.80
800	10.72	10.55	10.73	10.73	10.55	10.66
900	18.91	19.09	18.91	18.91	18.87	18.87
1000	25.27	27.09	25.27	24.55	25.60	25.56

Values of compressive strength of samples fired at 5°C min⁻¹

Firing Temperature/°C	Compressive strength/MPa					
	A	B	C	D	E	Average
500	7.27	7.09	727	6.91	7.09	7.13
600	7.27	7.45	7.45	7.27	7.45	7.38
700	8.91	8.91	8.73	8.73	8.73	8.80
800	10.36	10.36	10.18	10.18	10.36	10.29
900	18.72	18.72	18.55	18.75	18.55	18.65
1000	25.25	26.18	25.27	25.27	25.27	25.45

Values of compressive strength of samples fired at 6°C min⁻¹

Firing Temperature/°C	Compressive strength/MPa					
	A	B	C	D	E	Average
500	6.72	6.55	6.55	6.55	6.73	6.62
600	7.27	7.21	7.45	7.27	7.27	7.31
700	8.73	8.73	8.74	8.73	8.73	8.73
800	10.36	10.18	10.18	10.18	10.18	10.22
900	18.36	18.72	18.36	18.55	18.55	18.51
1000	25.27	26.18	24.45	25.09	25,09	25.25

APPENDIX F

VALUES OF COMPRESSIVE STRENGTH AND VARIANCES FOR THE

SAMPLES FOR DIFFERENT RATES OF TEMPERATURE RISE

Compressive strength and variances for samples fired to 500°C

Rate of temperature rise/°Cmin⁻¹	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
2	7.45	7.45	7.45	7.27	7.45	7.41	
	0.0016	0.0016	0.0016	0.0196	0.0016		0.006500
3	7.45	7.45	7.27	7.27	7.45	7.38	
	0.0049	0.0049	0.0121	0.0121	0.0049		0.009725
4	7.45	7.22	7.27	7.45	7.45	7.37	
	0.0064	0.0225	0.010	0.0064	0.0064		0.01295
5	7.27	7.09	7.27	6.91	7.09	7.13	
	0.0196	0.0016	0.0196	0.0484	0.0016		0.02270
6	6.72	6.55	6.55	6.55	6.73	6.62	
	0.0100	0.0049	0.0049	0.0049	0.00121		0.00920
Grand Total							0.06105
Average Variance with in samples							0.01221

Compressive strength and variances for samples fired to 600°C

Rate of temperature rise/°Cmin⁻¹	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
2	7.64	7.45	7.64	7.64	7.36	7.55	
	0.0081	0.0100	0.0081	0.0081	0.0361		0.01758
3	7.64	7.64	7.45	7.64	7.45	7.56	
	0.0064	0.0064	0.0121	0.0064	0.0121		0.01085
4	7.64	7.64	7.45	7.45	7.45	7.53	
	0.0121	0.0121	0.0064	0.0064	0.0064		0.01085
5	7.27	7.45	7.45	7.27	7.45	7.38	
	0.0121	0.0049	0.0049	0.0121	0.0049		0.009725
6	7.27	7.27	7.45	7.27	7.27	7.31	
	0.0016	0.0016	0.0196	0.0016	0.0016		0.006500
Grand Total							0.055505
Average Variance with in samples							0.011101

Compressive strength and variances for samples fired to 700˚C

Rate of temperature rise/°Cmin⁻¹	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
2	9.09	8.91	9.09	9.01	8.91	9.00	
	0.0081	0.0081	0.0081	0.0001	0.0081		0.008125
3	9.09	8.91	9.09	8.91	8.91	8.98	
	0.0121	0.0049	0.0121	0.0049	0.0049		0.009725
4	8.73	8.73	8.91	8.91	8.73	8.80	
	0.0049	0.0049	0.0121	0.0121	0.0049		0.009725
5	8.91	8.91	8.73	8.73	8.73	8.80	
	0.0121	0.0121	0.0049	0.0049	0.0049		0.009725
6	8.73	8.73	8.73	8.73	8.73	8.73	
	0.000	0.000	0.000	0.000	0.000		0.00000
Grand Total							0.03730
Average Variance with in samples							0.00746

Compressive strength and variances for samples fired to 800˚C

Rate of temperature rise/°Cmin⁻¹	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
2	11.09	11.09	10.91	11.09	11.09	11.05	
	0.0016	0.0016	0.0196	0.0016	0.0016		0.00650
3	10.91	11.09	10.91	10.91	10.91	10.95	
	0.0016	0.0196	0.0016	0.0016	0.0016		0.00650
4	10.72	10.55	10.73	10.73	10.55	10.66	
	0.0049	0.0100	0.0064	0.0064	0.0100		0.009425
5	10.36	10.36	10.18	10.18	10.36	10.29	
	0.0049	0.0049	0.0121	0.0121	0.0049		0.009725
6	10.36	10.18	10.18	10.18	10.18	10.22	
	0.0196	0.0016	0.0016	0.0016	0.0016		0.006500
Grand Total							0.038650
Average Variance with in samples							0.007730

Compressive strength and variances for samples fired to 900°C

Rate of temperature rise/°Cmin^{-1}	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
2	19.27	19.09	18.91	18.91	19.09	19.05	
	0.0484	0.0016	0.0196	0.0196	0.0016		0.0227
3	19.27	19.09	19.09	19.09	19.09	19.13	
	0.0196	0.0016	0.0016	0.0016	0.0016		0.0065
4	18.91	19.09	18.91	18.91	18.55	18.87	
	0.0016	0.0484	0.0016	0.0016	0.1024		0.0389
5	18.72	18.72	18.55	18.73	18.55	18.65	
	0.0049	0.0049	0.0100	0.0064	0.0100		0.00905
6	18.36	18.72	18.36	18.55	18.55	18.51	
	0.0121	0.0625	0.0121	0.0064	0.0064		0.024875
Grand Total							0.102025
Average Variance with in samples							0.020405

Compressive strength and variances for samples fired to 1000°C

Rate of temperature rise/°Cmin^{-1}	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
2	27.82	29.27	29.09	27.09	27.27	28.11	
	0.0841	1.3456	0.9604	0.9604	0.7056		1.014025
3	27.09	27.27	27.09	27.09	29.09	27.53	
	0.1936	0.0676	0.1936	0.1936	2.4336		0.7705
4	25.27	27.09	25.27	24.55	25.60	25.56	
	0.0841	2.3409	0.0841	1.0201	0.0016		0.8827
5	25,27	26.18	25.27	25.27	25.27	25.45	
	0.0324	0.5329	0.0324	0.0324	0.0324		0.165625
6	25.27	26.18	24.45	25.27	25.09	25.25	
	0.0081	1.000	0.5329	0.0081	0.0256		0.393675
Grand Total							3.226525
Average Variance with in samples							0.645305

APPENDIX G

VALUES OF COMPRESSIVE STRENGTH AND VARIANCES FOR THE SAMPLES FOR DIFFERENT FIRING TEMPERATURES

Compressive strength and variances of samples for the rate of temperatures rise of $2°Cmin^{-1}$

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
500	7.45	7.45	7.45	7.27	7.45	7.41	
	0.0016	0.0016	0.0016	0.0196	0.0016		0.00650
600	7.64	7.45	7.64	7.64	7.36	7.55	
	0.0081	0.0100	0.0081	0.0081	0.0361		0.01760
700	9.09	8.91	9.09	9.01	8.91	9.00	
	0.0081	0.0081	0.0081	0.0001	0.0081		0.008125
800	11.09	11.09	10.91	11.09	11.09	11.05	
	0.0016	0.0016	0.0196	0.0016	0.0016		0.00650
900	19.27	19.09	18.91	18.91	19.09	19.05	
	0.0484	0.0016	0.0196	0.0196	0.0016		0.0227
1000	27.82	29.27	29.09	27.09	27.27	28.11	
	0.0841	1.3456	0.9604	1.0404	0.7056		1.034025
Grand Total							1.09545
Average Variance with in samples							0.182575

Computation of compressive strength and variances of samples for different firing temperatures at rate of $3°Cmin^{-1}$

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
500	7.45	7.45	7.27	7.27	7.45	7.38	
	0.0049	0.0049	0.0121	0.0121	0.0049		0.009725
600	7.64	7.64	7.45	7.64	7.45	7.56	
	0.0064	0.0064	0.0121	0.0064	0.0121		0.01085
700	9.09	8.91	9.09	8.91	8.91	8.98	
	0.0121	0.0049	0.0121	0.0049	0.0049		0.009725
800	10.91	11.09	10.91	10.91	10.91	10.95	
	0.0016	0.0196	0.0016	0.0016	0.0016		0.00650
900	19.22	19.09	19.09	19.09	19.09	19.13	
	0.0196	0.0016	0.0016	0.0016	0.0016		0.006500
1000	27.09	27.27	27.09	27.09	27.09	27.53	
	0.1936	0.0676	0.1936	0.1936	0.1936		0.21050
Grand Total							0.25300
Average Variance with in samples							0.04230

Compressive strength and variances of samples for the rate of temperatures rise of 4°Cmin^{-1}

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
500	7.45	7.22	7.27	7.45	7.45	7.37	
	0.0064	0.0225	0.0100	0.0065	0.0065		0.012925
600	7.64	7.64	7.45	7.45	7.45	7.53	
	0.0121	0.0121	0.0064	0.0064	0.0064		0.01085
700	8.73	8.73	8.91	8.91	8.73	8.80	
	0.0049	0.0049	0.0121	0.0121	0.0049		0.009725
800	10.72	10.55	10.73	10.73	10.55	10.66	
	0.0049	0.0100	0.0064	0.0064	0.0100		0.009425
900	18.91	19.09	18.91	18.91	18.55	18.87	
	0.0016	0.0484	0.0016	0.0016	0.1024		0.03890
1000	25.27	27.09	25.27	24.55	25.60	25.56	
	0.841	2.3409	0.0841	1.0201	0.0016		0.8827
Grand Total							0.964525
Average Variance with in samples							0.160754

Compressive strength and variances of samples for the rate of temperatures rise of 5°Cmin^{-1}

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
500	7.27	7.09	7.27	6.91	7.09	7.13	
	0.0196	0.0016	0.0196	0.0484	0.0016		0.02270
600	7.27	7.45	7.45	7.27	7.45	7.38	
	0.0121	0.0049	0.0049	0.0121	0.0049		0.009725
700	8.91	8.91	8.73	8.73	8.73	8.80	
	0.0121	0.0121	0.0049	0.0049	0.0049		0.009725
800	10.36	10.36	10.18	10.18	10.36	10.29	
	0.0049	0.0049	0.0121	0.0121	0.0049		0.009725
900	18.72	18.72	18.55	18.73	18.55	18.65	
	0.0049	0.0049	0.010	0.0064	0.010		0.00905
1000	25.27	26.18	25.27	25.27	25.27	25.45	
	0.0324	0.5329	0.0324	0.0324	0.0324		0.165625
Grand Total							0.22655
Average Variance with in samples							0.0377583

Compressive strength and variances of samples for the rate of temperatures rise of $6°Cmin^{-1}$

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2
	A	B	C	D	E		
500	6.72	6.55	6.55	6.55	6.73	6.62	
	0.0100	0.0049	0.0049	0.0049	0.00121		0.00920
600	7.27	7.27	7.45	7.27	7.27	7.31	
	0.0016	0.0016	0.0196	0.0016	0.0016		0.006500
700	8.73	8.73	8.73	8.73	8.73	8.73	
	0.000	0.000	0.000	0.000	0.000		0.000
800	10.36	10.18	10.18	10.18	10.18	10.22	
	0.0196	0.0016	0.0016	0.0016	0.0016		0.006500
900	18.36	18.72	18.36	18.55	18.55	18.51	
	0.0225	0.0441	0.0225	0.0016	0.0016		0.023075
1000	25.27	26.18	24.45	25.27	25.09	25.25	
	0.0081	1.000	0.5329	0.0081	0.0256		0.393675
Grand Total							0.43895
Average Variance with in samples							0.0731583

APPENDIX H
COMPUTATION OF VARIANCE AND F-VALUES FOR VARYING RATES OF TEMPERATURE RISE

Values of samples heated to 500°C

Rate of temperature rise /°Cmin^{-1}	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
2	7.45	7.45	7.45	7.27	7.45	7.41	0.051984
3	7.45	7.45	7.27	7.27	7.45	7.38	0.039204
4	7.45	7.22	7.27	7.45	7.45	7.37	0.035344
5	7.27	7.09	7.27	6.91	7.09	7.13	0.002704
6	6.72	6.55	6.55	6.55	6.73	6.62	0.315844
Grand average						7.182	
Overall variance with in mean							0.445080
Variance with in sample							0.012221
Calculated F - value							36.45209

Values of samples heated to 600°C

Rate /°Cmin^{-1}	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
2	7.64	7.45	7.64	7.64	7.36	7.55	0.007056
3	7.64	7.64	7.45	7.64	7.45	7.56	0.008836
4	7.64	7.64	7.45	7.45	7.45	7.53	0.004096
5	7.27	7.45	7.45	7.27	7.45	7.38	0.007396
6	7.27	7.27	7.45	7.27	7.27	7.31	0.024336
Grand average						7.466	
Overall variance with in mean							0.051720
Variance with in sample							0.011101
Calculated F - value							4.659040

Values of samples heated to 700°C

Rate of temperature rise/°Cmin^{-1}	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
2	9.09	8.91	9.09	9.01	8.91	9.00	0.019044
3	9.09	8.91	9.09	8.91	8.91	8.98	0.013924
4	8.73	8.73	8.91	8.91	8.73	8.80	0.003844
5	8.91	8.91	8.73	8.73	8.73	8.80	0.003844
6	8.73	8.73	8.73	8.73	8.73	8.73	0.017424
Grand average						8.862	
Overall variance with in mean							0.058080
Variance with in sample							0.007460
Calculated F - value							0.778552

Values of samples heated to 800°C

temperature Rate of rise/°Cmin^{-1}	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
2	11.09	11.09	10.91	11.09	11.09	11.05	0.173056
3	10.91	11.09	10.91	10.91	10.91	10.95	0.099856
4	10.72	10.55	10.73	10.73	10.55	10.66	0.000676
5	10.36	10.36	10.18	10.18	10.36	10.29	0.118336
6	10.36	10.18	10.18	10.18	10.18	10.22	0.171396
Grand average						10.634	
Overall variance with in mean							0.563320
Variance with in sample							0.007730
Calculated F - value							72.87450

Values of samples heated to 900°C

Rate of temperature rise/°Cmin^{-1}	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
2	19.27	19.09	18.91	18.91	19.09	19.05	0.043264
3	19.27	19.09	19.09	19.09	19.09	19.13	0.082944
4	18.91	19.09	18.91	18.91	18.55	18.87	0.000784
5	18.72	18.72	18.55	18.73	18.55	18.65	0.036864
6	18.36	18.72	18.36	18.55	18.55	18.51	0.110224
Grand average						18.842	
Overall variance with in mean							0.274080
Variance with in sample							0.020405
Calculated F - value							13.432

Values of samples heated to 1000°C

Rate of temperature rise/°Cmin^{-1}	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
2	27.82	29.27	29.09	27.09	27.27	28.11	2.9929
3	27.09	27.27	27.09	27.09	29.09	27.53	1.3225
4	25.27	27.09	25.27	24.55	25.60	25.56	0.6724
5	25.27	26.18	25.27	25.27	25.27	25.45	0.8649
6	25.27	26.18	24.45	25.27	25.09	25.25	1.2769
Grand average						26.380	
Overall variance with in mean							7.1296
Variance with in sample							0.645305
Calculated F - value							11.04842

APPENDIX I

COMPUTATION OF VARIANCE AND F-VALUES FOR VARYING FIRING

TEMPERATURE

Values for samples heated at a rate of temperature rise of $2°Cmin^{-1}$

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
500	7.45	7.45	7.45	7.27	7.45	7.41	39.501225
600	7.64	7.45	7.64	7.64	7.36	7.55	37.761025
700	9.09	8.91	9.09	9.01	8.91	9.00	22.043025
800	11.09	11.09	10.91	11.09	11.09	11.05	6.996025
900	19.27	19.09	18.91	18.91	19.09	19.05	28.676025
1000	27.82	29.29	29.09	27.09	27.27	28.11	207.792225
Grand average						13.695	
Overall variance with in mean							342.7655
Variance with in sample							0.182575
Calculated F - value							1877.41774

Values for samples heated at a rate of temperature rise of $3°Cmin^{-1}$

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
500	7.45	7.45	7.27	7.27	7.45	7.38	38.539264
600	7.64	7.64	7.45	7.64	7.65	7.56	36.336784
700	9.09	8.91	9.09	8.91	8.71	8.98	21.233664
800	10.91	11.09	10.91	10.91	10.91	10.95	6.959044
900	19.27	19.09	19.09	19.09	19.09	19.13	30.713764
1000	27.09	27.27	27.09	27.09	27.09	27.53	194.379364
Grand average						13.588	
Overall variance with in mean							328.161884
Variance with in sample							0.04230
Calculated F - value							7757.964

Values for samples heated at a rate of temperature rise of 4°Cmin^{-1}

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
500	7.45	7.22	7.27	7.45	7.45	7.37	33.200644
600	7.64	7,64	7.45	7.45	7.45	7.53	31.382404
700	8.73	8.73	8.91	8.91	8.73	8.80	18.766224
800	10.72	10.55	10.73	10.73	10.55	10.66	6.1107840
900	18.91	19.09	18.91	18.91	18.55	18.87	32.924644
1000	25.27	27.09	25.27	24.55	25.60	25.56	154.45518
Grand average						13.132	
Overall variance with in mean							276.83988
Variance with in sample							0.1607542
Calculated F - value							1722.132

Values for samples heated at a rate of temperature rise of 5°Cmin^{-1}

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
500	7.27	7.09	7.27	6.91	7.09	7.13	33.8724
600	7.27	7.45	7.45	7.27	7.45	7.38	31.0249
700	8.91	8.91	8.73	8.73	8.73	8.80	17.2225
800	10.36	10.36	10.18	10.18	10.36	10.29	7.0756
900	18.72	18.72	18.55	18.75	18.55	18.65	32.4900
1000	25.27	26.18	25.27	25.27	25.27	25.45	156.2500
Grand average						12.950	
Overall variance with in mean							277.9354
Variance with in sample							0.0377583
Calculated F - value							7360.9087

Values for samples heated at a rate of temperature rise of 6°Cmin^{-1}

Firing temperature /°C	Compressive strength /MPa					Average compressive strength /MPa	Variance δ^2 with in the mean
	A	B	C	D	E		
500	6.72	6.55	6.55	6.55	6.73	6.62	37.859409
600	7.27	7.27	7.45	7.27	7.27	7.31	29.844369
700	8.73	8.73	8.73	8.73	8.73	8.73	16.345849
800	10.36	10.18	10.18	10.18	10.18	10.22	6.517809
900	18.36	18.72	18.36	18.55	18.55	18.51	32.913169
1000	25.27	26.18	24.45	25.27	25.09	25.25	155.675529
Grand average						12.773	
Overall variance with in mean							279.156134
Variance with in sample							0.0731583
Calculated F - value							3815.7821