

DEVELOPMENT AND TESTING OF COMPOSITE REFRACTORY BRICKS
(A case study of selected Kenyan clays)

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**A thesis submitted to Graduate School in partial fulfilment of the requirements of
Master of Science Degree in Engineering Systems and Management of Egerton
University**

EGERTON UNIVERSITY

NOVEMBER 2016

DECLARATION AND RECOMMENDATION

Declaration

I, Kimutai Charles Keter declare that this thesis is my original work and has not been wholly or in part presented for the award of a degree in any other university known to me.

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Recommendation

This thesis is the candidate's original work and has been prepared with our guidance and assistance and is submitted for examination with our approval as university supervisors.

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DEDICATION

I dedicate this work to my wife Josephine Keter, children; Kiprotich Mutai, Kibet Keter, Kipkirui Mutai, Chepkemoi Keter and Kipkorir Keter for their patience and moral support during the entire period of my studies. To my Parents William Kipketer Marintany and Alice Bugaga who denied themselves the comfort of life for the sake of my education.

ACKNOWLEDGEMENT

I would like to express my sincere thanks to all lecturers of the Faculty of Engineering and Technology, Egerton University for sharing their wisdom and extensive understanding as well as training me during my study. Distinguished appreciation and sincere gratitude goes to my supervisors Prof. Nyaanga, D.M. and Dr. Owino, G.O. of Egerton University for the invaluable support and guidance in the realization of this thesis.

More appreciation goes to the Director Kenya Industrial Research and Development Institute (KIRDI), for according me ample time and space to carry out the experiments. Special thanks go to the technical staff of (KIRDI) ceramic division, who assisted me while carrying out the experiments and collecting data. Thanks to the Ministry of Mines and Geology laboratory division for granting me the opportunity to carry out chemical composition analysis of clay samples at their facility.

Above all, I acknowledge the Almighty God for according me strength, hope and care. It is through his unseen interventions that this work has been a success.

ABSTRACT

Refractory bricks (refractories) are used in the construction of furnaces and kiln internal linings that hold, melt and transfer raw materials being processed. Kenya imports refractories mainly for its cement, metal smelting and sugar processing industries. Kaolin, bauxite, salama, soapstone and ball clays exhibited Loss of Ignition of 7-15%, silica of 46-55% and alumina of 25-34%. Soapstone and salama clays exhibited high values of potassium (5.6%) and iron oxide (16%), while salama clay had low alumina of 20% disqualified as refractory clay material. Alumina, silica and iron oxides in kaolin, bauxite and ball clays made them suitable as composite refractory clays. The properties of the developed composite bricks were determined at different mix ratios and particle sizes and their results compared with American Society of Testing Materials (ASTM) standards. Kaolin and bauxite clays were mixed at different ratios with 10% binder (ball clay). Developed bricks were moulded to volumes of (70x70x70) mm, subjected to a pressure of 4.1N/m² and dried at 110°C. The bricks were fired in the furnace at 200°C for 6 hours, 650°C for 3 hours, 950°C for 4 hours and 1250°C for 8 hours and left to cool to room temperature. They were then subjected to physical and thermal tests and data obtained analysed using Statistical Analysis Software at 5% level of significance. Cold crushing strength, thermal shock resistance and bulk density were directly proportional to the increase in kaolin ratio. Decrease in linear shrinkage and apparent porosity was directly proportional to bauxite ratio. As kaolin to bauxite ratio increased from 2:7:1 to 7:2:1, apparent porosity decreased from 38% to 29% and bulk density increased from 1.45g/cm³ to 1.61g/cm³, cold crushing strength increased from 2.2 to 3.3KN/m², linear shrinkage decreased from 8.89% to 3.69%, and thermal shock resistance increased from 14 to 27 cycles. Bulk density, cold crushing strength, linear shrinkage and thermal shock resistance decreased with increase in particle sizes but apparent porosity increased with increase in particle size. As particle sizes increased from 150µm to 600µm, apparent porosity increased from 29% to 36%, bulk density decreased from 2.23g/cm³ to 1.166g/cm³, cold crushing strength decreased from 3.3 to 2.64MN/m², linear shrinkage from 3.69% to 2.0%, and thermal shock resistance from 27 to 19 cycles. Particle sizes of 150µm at a mix ratio of 7:2:1 produced apparent porosity of 29%, bulk density of 2.23g/cm³, cold crushing strength of 3.3MN/m², linear shrinkage of 3.69% and thermal shock resistance of 27 cycles which was suitable for commercial production. This study adds knowledge to existing literature on refractories local clays.

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LIST OF ACRONYMS AND ABBREVIATION

Al ₂ O ₃	Alumina oxide
CaO	Calcium oxide
Fe ₂ O ₃	Iron oxide
K ₂ O	Potassium oxide
MgO	Magnesium oxide
Na ₂ O	Sodium oxide
SiO	Silica oxide
D	Dry Weight
R	Percentage of clay sample retained
S	Weigh at room temperature
W	Weight in air
SAS	Statistical analysis for sciences
ANOVA	Analysis of Variance
ASTM	American Society for Testing Materials
KIPPRA	Kenya Institute of public policy and Research Analysis.
KIRDI	Kenya Industrial Research and Development Institute
LSD	Least square deviation
α	Alpha
μ	Micro
CCS	Cold crushing strengeth
AP	Aparent porosity
BD	Bulk density
LSF	Linear fired shrinkkkage
TSR	Thermal shock resistance
LOI	Lost on ignition
CYC	Cycles
PCE	Pyrometric Cone Equivalent

DEFINITION OF TERMS

Refractory bricks	Materials having high melting points with properties that make them suitable to act as heat resisting barriers between high and low temperature zones.
Vitrification	The temperature levels between which the clay begins to fuse and when it achieves its final fusion or hardness
Plasticity	That property which enables a material to be changed in shape without rupturing by the application of external force and retain that shape when the force is removed or reduced below a certain value
Thermal shock resistance	This test method indicates the ability of a refractory product to withstand the stress generated by sudden changes in temperature
Apparent porosity	A ratio of volume of vacant space/pores to the total volume of materials expressed in percentage
Bulk Density	The ratio of mass in a solid state to bulk volume of refractory brick
Crushing Strength	The capacity of refractory brick to provide resistance to a compressive load at room temperature
Linear fired shrinkage	The change in linear dimensions that has occurred in test specimens after soaking heat for a period of 24 h and then cooled to room temperature

CHAPTER ONE

INTRODUCTION

1.1 Background information

Refractory bricks (refractories) are the main components in metallurgical and cement processing industries in the construction of furnaces and kiln internal linings (Krishna *et al.*, 2013). Kiln internal linings are used as vessels for holding, melting and transferring raw materials under process. The major consumers of refractory products are cement, sugar, incineration, metal processing industries, chemical, glass, boilers and petrochemical industries.

East African Portland Cement alone consumes approximately 1200 tons of refractory bricks annually for production of clinker (Ministry of Industrialization, 2013). This demand of refractory bricks is bound to increase tremendously owing to the increased growth of Kenya's manufacturing sector (World Bank, 2013).

Kenya imported 100 % refractory bricks in 2012 spending approximately 3 billion Kenya shillings annually mostly from India and China for its cement, metal smelting and sugar processing industries (KIPPRA, 2013). But according to Kenya Central Bureau of Statistics (2014), Kenya has a potential of cutting down imports of refractory bricks by 20% and increase exports by 15 % in a span of 10 years, despite the abundant deposits of clay raw materials for local production. The principal raw materials used in the production of refractory bricks are $Al_2O_3-SiO_2$ clay. Range of raw materials is utilized to achieve the desirable chemical composition, physical and thermal properties of refractory bricks (Harbison, 2005). kaolin, bauxite and ball clays are listed among the primary raw materials for refractory brick production

According to Kenya Government report (Ministry of Environment and Natural Resources, 2006), Kenya is endowed with vast deposits of kaolin, bauxite and ball clays at Nakuru, Kericho and Nyeri that can be exploited for production of refractory bricks. Unfortunately, there has not been economical utilization of these clays locally. However, production of refractory bricks in Kenya will add value to local clays and help in achieving the Vision 2030 (Ministry of Industrialization, 2013).

In the recent past, there have been researches towards developing refractory products for furnace linings in steel industries in Nigeria and Uganda utilising their locally available clay deposits. John (2003) among other researchers has indicated the suitability of Nigerian clay

deposits as refractories for furnace linings in steel industries, while Agha (1998) showed that local clay has better thermal and physical properties than imported ones.

1.2. Statement of the problem

Even though refractory bricks are the main source of energy conservation in furnace in Kenya industries, its production has not been exploited locally. Reliance on imports is hampered by price escalation. Refractory bricks produced under different mix ratios have been reported to have different physical and thermal characteristics. Previous researches on this subject focused on the chemical, physical and thermal properties derived from one type of refractory clay material, but little has been reported on the characteristics of composite refractory bricks produced at varying ratios and particle sizes of kaolin and bauxite using ball clay as binder. The optimal mix ratios of such composite refractory bricks are unknown and have not been documented in Kenya.

1.3 Objectives

The broad objective of this study was to develop and test properties of composite refractory bricks using selected Kenyan clays.

The specific objectives are:

- i. To determine the chemical composition of selected Kenyan clays.
- ii. To develop composite refractory bricks and determine the effects of kaolin and bauxite clay mix ratios on their physical and thermal properties.
- iii. To determine how particle sizes affect physical and thermal properties of the developed composite refractory bricks.

1.4 Research questions

The research questions are:

- i. What is the chemical composition of kaolin, bauxite and ball clays?
- ii. What are the effects of kaolin and bauxite clay mix ratios on physical and thermal properties of developed composite refractory bricks?
- iii. How do particle sizes affect physical and thermal properties of composite refractory bricks?

1.5 Justification

Kenya has substantial deposits of kaolin, bauxite and ball clays that can be economically utilized for the production of refractory bricks. An in-depth understanding of the chemical composition, physical, thermal properties and particle size distribution are important if optimal material properties of refractory bricks are to be achieved. These properties influence the formulation and processing conditions of the final product.

Characterizations of refractory raw materials will boost utilization of clays for commercial application to improve local production, development of refractory technology and increased revenue. Production of refractory bricks in Kenya presents a lucrative opportunity to create employment through value addition to natural resources. It is feasible to supplement bricks importation by providing information on chemical composition, physical and thermal properties suitable for refractory bricks from Kenyan kaolin, bauxite and ball clays.

1.6 Scope and limitations

This study utilized kaolin and bauxite mixed with ball clay in production of composite refractory bricks. The collected kaolin and bauxite clays were dried, milled and sieved before firing in muffle furnace at 1250°C. they were then mixed at ratios of (kaolin to bauxite) 2:7:1, 3:6:1, 4:4:1, 6:3:1 and 7:2:1 and particle sizes of 150µm, 380µm and 600µm with 10% ball clay as binder; moisture content of the mixture was maintained at 8%. Bricks were compressed using hydraulic press at 41KN with dwell time being maintained at 5 minutes using cubical moulds of 70x70x70 mm. The study was limited to apparent porosity, bulk density, cold crushing strength, linear shrinkage and thermal shock resistance.

CHAPTER TWO

LITERATURE REVIEW

2.1 Refractory materials

Refractory is a material which retains its shape and chemical properties when subjected to high temperatures. It is used in applications which require extreme heat resistance. Most raw materials used in refractory production do not occur naturally, but are processed in such a way as to lower the fluxes and unwanted oxides. Generally, refractory materials consists of oxides such as silica oxide (SiO_2) alumina oxide (Al_2O_3), magnesia oxide (MgO), calcium oxide (CaO) and zirconia oxide (ZrO_2). Different types of refractories are used in various combinations and shapes depending on their applications such as in boilers, furnaces, kiln and ovens.

Characteristics of refractory materials include the ability to withstand high temperatures, resist sudden changes of temperatures, withstand abrasive forces, conserve heat and have low coefficient of thermal expansion. Clays are typically anhydrous complex compounds of alumina oxide (Al_2O_3) and silica oxide (SiO_2) that exist in various proportions and contain varied amounts of impurities of iron, organic matters and residual minerals (Sanni, 2005). These are used to regulate rate of heating, peak temperatures and soaking (resident) time in the muffle furnaces that affect chemical reactions and the development in the refractory clay body. The firing temperature depends upon the service for which the bricks are intended.

In firing refractory bricks, a heating regime of four temperatures must be adhered to (Al-Amaireh, 2009). The regime includes heating at a temperature of 200°C where free water is evaporated; at 450°C combined water is driven off; iron, sulfur compounds, gasses formed are eliminated and organic matter are oxidized at $600^\circ\text{C} - 950^\circ\text{C}$ and gasses formed are eliminated. Mineral transformations and changes in volume are effected between $870^\circ\text{C} - 1250^\circ\text{C}$, and finally the particles of clay are bonded together into mechanically strong refractory brick. Refractory clay materials are those with high refractoriness and possess the capability of maintaining both physical and chemical identity at high temperatures, thus are resistant to fusion and softening at elevated working temperatures (Sanni, 2005). The clay materials used for furnace and kiln linings in metallurgical and cement industries are classified as refractory clays. However, the degree of refractoriness and plasticity of any clay material is often influenced by the amount of the impurities contained in them. Moreover, the ability of selected refractory clay

to withstand high temperature and resist physical and chemical corrosion determines the quality and the suitability of such material for use as furnace lining.

Since metallurgical processes require very high operating temperatures, refractories used, must withstand very high temperatures without rapid chemical, physical and thermal deterioration. Other important characteristics are bulk density, apparent porosity, apparent specific gravity, crushing strength, refractoriness, linear shrinkage, thermal resistant and thermal expansion.

In Kenya, clay deposits are found in various locations. For instance, ball clay is found in Nyeri County, kaolin in Naivasha and soapstone in Kisii (Tabaka area). Bauxite materials currently used in cement industries as additives (0.3%) in the clinkering process are mined from Tanzania and Kenya in Kericho County. The development of our locally available raw materials for the production of refractories was justified by the need to meet the technological requirements of the country, and to conserve the much-needed foreign exchange (Hassan, 2000)

2.1.1 Application of refractory bricks

Refractory bricks are the primary materials used by industries dealing with iron and steel, glass, pottery, cement kilns, chemical plants, sugar refineries, incinerators and power stations as internal linings of furnaces for melting and heating. Refractories are normally used for: lining of plants for thermal processes (melting, firing, and heat treatment furnaces and transport vessels), heat insulation, heat recovery (regenerators and recuperators) and construction of design components (functional products).

Refractory clays are generally characterized by low thermal expansion coefficient, low thermal conductivity, low specific gravity, low specific heat, low strength at high temperature and less slag penetration. Since it is hard to build closed-cell structures into high-void-volume ceramics, these materials are all “open” (Carniglia and Barna, 2002). Refractory bricks are principally used in furnace construction to confine hot atmospheres, and to thermally insulate structural members from excessive temperatures (Callister, 2003).

2.1.2 Refractory clays

Clay refractoriness refers to its ability to resist melting at high temperature, prevent heat flow across its cross-sectional boundary layer as much as possible and maintain volume stability

at high temperature, withstand unsteady thermal and physical shock, resist abrasion and corrosion, exhibit higher hot strength and be resistant to hot fluids. In addition, refractory materials must be dense and porous. Hence, insulating refractory clays have high porosity, low thermal conductivity and high thermal insulating properties. They are capable of minimizing heat loss in furnaces as much as possible and maximizing heat conservation to a great extent.

Refractory clay material obtained from a single site might not possess all the required properties that would make it a perfect refractory material. Therefore, it becomes imperative to select clays based on the physical, chemical and thermal analysis of samples. The selected refractory clay is improved with others from different sites adequately and blended to required physical, thermal and chemical properties (Nuhu and Abdullahi, 2008).

Clays are classified according to their relative plasticity or malleability, their strength when moist (green strength), their strength after drying (dry strength), their air shrinkage properties and their vitrification range. Clay's vitrification range describes the temperature levels between which the clay begins to fuse and when it achieves its final fusion or hardness. Clays are often mixed or blended to achieve the desired properties dictated by their end use. Plasticity is that property which enables a material to be changed in shape without rupturing by the application of external force and retain that shape when the force is removed or reduced below a certain value.

Abolarin *et al.*, (2004) investigated the properties of Nigerian local refractory materials from Kuru, Ikaleri, Arkin-ladi and Bauchi locations and concluded that refractory clays from Ikaleri and Barkin-ladi are suitable for furnace lining construction due to higher thermal shock resistance, crushing strength, bulk density and refractoriness values which had similar result with this research work. Omowumi (2001) also found a close relationship of properties between Nigerian bricks and imported ones. Research work by Irabor (2002) revealed that kaoline clays from Kankara in Nigeria, Jos and Oshiele are suitable for paper, paint and pharmaceutical manufacture.

2.1.3 Properties of bricks

An understanding of properties of refractories is fundamental to the development, improvement, quality control and selection of linings for high temperature applications. Various applications of refractory materials in different types of industries require diversified properties to meet the physico-chemical and thermal requirements at different phases of use. In some

industrial units more than one phase are present. For instance in steel-making vessels, slag/metal/gases are simultaneously present in the vessel at high temperatures. In the heat treating furnaces, solid/reducing or oxidizing gases are simultaneously present (Chukwudu, 2008).

Chemical composition, bulk density, apparent porosity, apparent specific gravity and strength at ambient temperatures are used as control parameters in the manufacturing and quality control process of refractory bricks. The elemental oxide and phase composition of the clays and bricks can be determined by the X-ray diffraction analysis. X-ray diffraction method works with the principle that every crystal has characteristic diffraction angle of incident rays that give the method its versatility. These parameters determine applicability of the final end product. The chemical composition serves as a basis for the classification of refractory bricks, while density, porosity and strength are used in quality evaluation of the brick. Bulk density and apparent porosity are determined by the boiling water method. This method assumes that when the brick is boiled in water for a period of not less than two hours, all the pores will absorb water. The soaked weight in water, suspended weight in water and dry weight are related and can be used to determine apparent porosity and bulk density. Bulk density, apparent porosity and strength are affected by the type and quality of raw materials, the size and fit of the particles, moisture content at the time of pressing, pressure in the mould, temperature, duration of firing and the rate of cooling. The strength of a product at room temperature is an important indicator of its ability to withstand abrasion and impact in low temperature applications and to withstand handling and shipping (Harbison, 2005).

2.2 Chemical properties of refractory materials

Alumina (Al_2O_3) occurs in clays as allumino-silicates and would very rarely be found in clays in the Free State. Silica (SiO_2) is used to reduce plasticity, unfired strength and drying contraction. Iron Oxide (Fe_2O_3) and titania (TiO_2) are oxides that primarily determine the fired colours (1250°C) of the refractory clay. Lime (CaO) and Magnesia (MgO) in clays act as mild fluxes and combine with the oxides of alumina and silica on firing to form eutectics which reduce the vitrification temperature and refractoriness of the clay (Ryan and Ranford, 2012). Omotoyinbo and Oluwole (2008) reported that clay with aluminum oxide (Al_2O_3) and silica (SiO_2) as major constituents are suitable as alumino-silicate refractory materials. Harbison

(2005) reported that there are generally five American society of testing materials (ASTM) standard classes of refractory bricks, namely; super-duty, high-duty, medium-duty, low-duty and semi-silica with composite as shown in Table 2.1.

Table 2. 1: ASTM classes of refractory bricks

Brick	%SiO ₂	% Al ₂ O ₃	% of other Constituents	PCE
Super duty	49-53	40-44	5-7	3175-3200
High duty	50-60	35-40	5-9	3075-3175
Medium duty	60-70	26-36	5-9	2975-3075
Low duty	60-70	23-33	6-10	2770-2900

Source: Harbison (2005).

Chemical properties of refractory materials include uniformity of composition, reactions between base materials and operating environment and issues such as volatilization of the constituents or binding agents, corrosion, chemical attack or diffusion and reactions with the product. According to Abdullahi and Samaila (2007), the chemical composition of clay samples from Gur in Nigeria tested had alumina (Al₂O₃) content of 21.25% while that of Yamarkumi was 19.68%. Both were found to be applicable as high melting clay but not as refractory clay. This was because the values of their alumina content lay within the recommended range for high melting clay. The silica (SiO₂) content of Gur clay (59.20%) met the standard for refractory clay (46-62%) while that for Yamarkumi clay (41.80%) was short of the standard (Nnuka and Agbo, 2000). Tables 2.2 show the chemical composition and Loss On Ignition (LOI) of clays from different Nigeria clays.

Omotoyinbo and Oluwole (2008) reported that chemical analysis refractory bricks contain aluminum oxide (Al₂O₃) of 26.29 – 30.68% and silica (SiO₂) of 45.22 – 48.66% as major constituents making them suitable as alumino-silicate refractory materials.

Table 2. 2: : Chemical composition of selected clays in Nigeria

Clay samples	Al ₂ O ₃	SiO ₂	CaO	MgO	Fe ₂ O ₃	Na ₂ O	TiO ₂	K ₂ O	LOI
A	28.76	47.06	2.828	1.37	1.568	0.1	1.128	0.0925	13.974
B	20.5	50.5	1.7	0.98	12.3	-	-	-	12.5
Refractory clays	25 - 39	46 - 62	0.2 – 1.0	0.2 – 1.0	0.4 - 2.7	-	-	-	8 – 18

Source: Nnuka and Agbo(2000)

Table 2.3 by East African Portland Cement gives the chemical composition for clays used for cement production process. The clays, except kaolin are either rich in alumina or silica.

Table 2. 3: Chemical composition

Oxides (%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O
Salama	49.09	22.42	14.15	2.11	1.70	0.32	0.31
Pozzolana	60.04	13.59	7.81	1.76	2.15	3.72	4.45
Buaxite	24.71	40.64	9.66	1.27	0.61	0.02	0.05
Kaolin	40-50	18-38	1	0.10	0.20	0.5	0.01
Vermiculite	46.00	16.00	13.00	5.00	35.00	6.00	

Source: East African Portland Cement Company (2013).

2.3 Effects of particle size on refractory materials

The primary function of precision particle analysis is to obtain quantitative data about the size and distribution of particles in a material. The size of a spherical particle is uniquely defined by its diameter. Particle analysis assists in ensuring that the samples have the required particle size needed for the production of refractory bricks or moulding sand mixtures (Omotoyinbo and Oluwole, 2008). Properties of any refractory depend on its mineral composition, particle size distribution of the minerals and how minerals react to high temperatures and furnace environments. Particles varying in size from 6mm to less than 74µm in diameter are recommended for unfired refractories. Upon firing, the fine particles form a ceramic bond between the larger particles. The fired refractory consists of bonded crystalline mineral particles and glass (K₂O) depending largely on the composition of the oxides (Callister, 2003).

2.3.1. Particle size analysis

Sieving is the classification of particles in terms of their ability or inability to pass through an aperture of controlled size. Sieving accomplishes the separation of particles mechanically on the basis of size and their acceptance or rejection by a screening surface. Particles are introduced onto a stack of particles (screening surfaces) with finer apertures below. This could be done using a dry sample (dry sieving) or wet sample as specified by ASTM C 92-(2014) standard.

The three common methods employed for particle analysis are:

- i) A cumulative–log plot: a semi-log plot of either the cumulative mass % passing (undersize) or the cumulative mass % retained (oversize) against log particle size which gives a curve.
- ii) The Gates-Gaudin-Schuhmann method: a log-log plot giving a straight line.
- iii) The Rosin–Rammler-Benneth method: a log-log straight-line plot.

Particle analysis would assist to narrow down to a specific particle size that gives results similar to the standard particle materials. The percentage retains and pass is calculated using the equations 2.1 and 2.2

$$\% \text{ of clay sample retained} = \frac{\text{Mass of clay retain on particle}}{\text{Mass of each sample of clay}} \times 100\% \text{ --- 2.1}$$

$$\% \text{ of clay sample passing through particle} = 100\% - \% \text{ of clay sample retained --- 2.2}$$

2.3.2. Bricks Production

There are three main refractory brick manufacturing processes: soft mud, stiff mud and dry press method based on amount of water added. Harbison (2005) reported that water content of 10 to 15 % is added into the clay to produce plasticity in the case of stiff mud process; for stiff mud process, clays are mixed with water above 30% while the dry press method requires less than 10% water. Production of refractory bricks requires utilization of chemically controlled raw materials and processing temperatures in order to achieve the desired physical, mechanical and thermal properties. Refractories are composed of thermally stable inorganic mineral aggregates, a binder phase and additives that enhance certain mechanical properties. In the production of

refractory bricks, particles of ground clay must include a range of graded sizes, each in appropriate proportion. The clays are typically ground in a dry pan, which is a rotating pan-shaped grinding mill having slotted openings in the bottom. The batches are screened to the desired sizes and thoroughly mixed with a binder and a small but closely controlled amount of water (< 10%). Normally, refractory bricks are processed by dry method where the raw materials are mixed directly with water to produce a homogenous plastic body. In some cases, the homogenous mixture is allowed to age. Aging refers to change in plasticity within the body leading to change in particle–particle interactions which helps to distribute moisture evenly through the body. The moistened batch is then fed to a mechanically or hydraulically operated press in which the brick is formed under pressure (Matsui *et al.* 2001).

Bricks formed by any of the processes described above are dried in tunnel or humidity driers and fired. The rate of heating, peak temperatures, soaking (resident) time and the atmosphere in the kiln affect chemical reactions and the micro structural development in the ceramic body and consequently are important in the processing. Free and combined water are driven off, while iron and sulfur compounds and organic matter are oxidized. Besides, mineral transformations and changes in volume are effected while the particles of clay are ceramically-bonded together into a mechanically-strong brick.

2. 4 Physical and thermal properties of refractory bricks

When selecting a refractory material for a particular application, its physical properties such as bulk density, apparent porosity and strength at room temperature should be considered. Bulk density, strength and apparent porosity are influenced by the quality of the materials, the size of particles, moisture content, pressure, temperature, duration of firing, kiln condition and cooling rate (Idenyi and Nwajagu, 2003).

2.4.1 Bulk density

Bulk density is mass per unit volume of the refractory without taking into account the volume occupied by pores. It depends upon the specific gravity and porosity. The density of refractories is an indirect measure of their capacity to store heat and increases with decrease in particle size (Idenyi and Nwajagu, 2003). According to Aigbodion *et al.* (2010), bulk density ranges from 1.90 to 2.30g/cm³ for refractory clays. Abdullahi and Samaila, (2007) and Omowumi, (2001) reported an average bulk density of the clay to range between 2.06-2.11 g/cm³

which makes it suitable for siliceous fireclays. This property is important in the transportation or handling of refractory materials. Factors known to affect this property include particle size, treatment during manufacturing and the nature of the materials in the clay sample (Omotoyinbo and Oluwole, 2008).

2.4.2 Apparent porosity

Apparent Porosity is a measure of the volume of the open pores into which a liquid can penetrate in the total volume. It is an important property especially where the refractory is in contact with molten charge and slags. A low porosity is desirable since it would prevent easy penetration of the molten materials and have influences on refractory behaviour. A large number of small pores are generally preferable to an equivalent number of large pores. In many applications low porosity is recommended since insulating refractories are very porous. Aigbodion *et al.* (2010) reported the porosity for tin tailings(industrial waste) to increase with particle size ranging from 32.66% to 39% for particle sizes between 0.090mm and 0.39mm inclusive as shown in Figure 2.1. Those values of apparent porosity were higher than the recommended value of 22 - 25% for medium heat duty and 23 - 26% for high heat duty clay Harbison(2005).

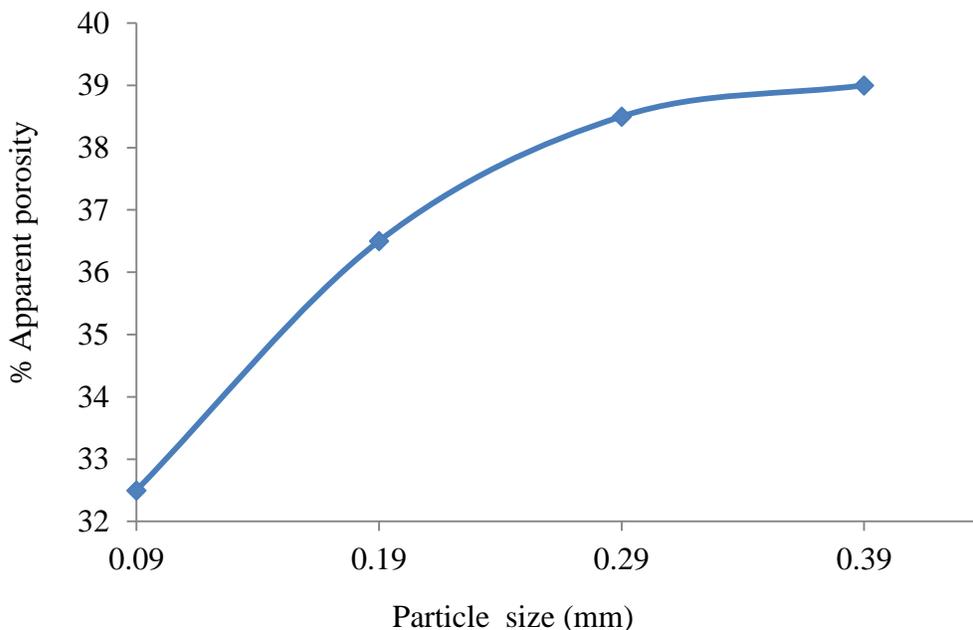


Figure 2.1: Variation of porosity with particle size.

(Adopted from Aigbodion *et al.* 2010)

2.4.3 Cold crushing strength

Cold crushing strength (CCS) indicates the ability of a refractory to withstand handling, impact or abrasion at low temperatures. However, it does not give strength of refractory at service temperatures. Factors such as composition, ramming pressure, firing temperature, particle size and the amount of water content determine the ccs of the clay materials (Omotoyinbo and Oluwole, 2008). The ccs of refractories is determined by placing a sample on a flat surface followed by application of a uniform load to it through a bearing block in a standard mechanical or hydraulic compression testing machine. The load at which it initiates cracks on the specimen represents the cold crushing strength Figure 2.2.

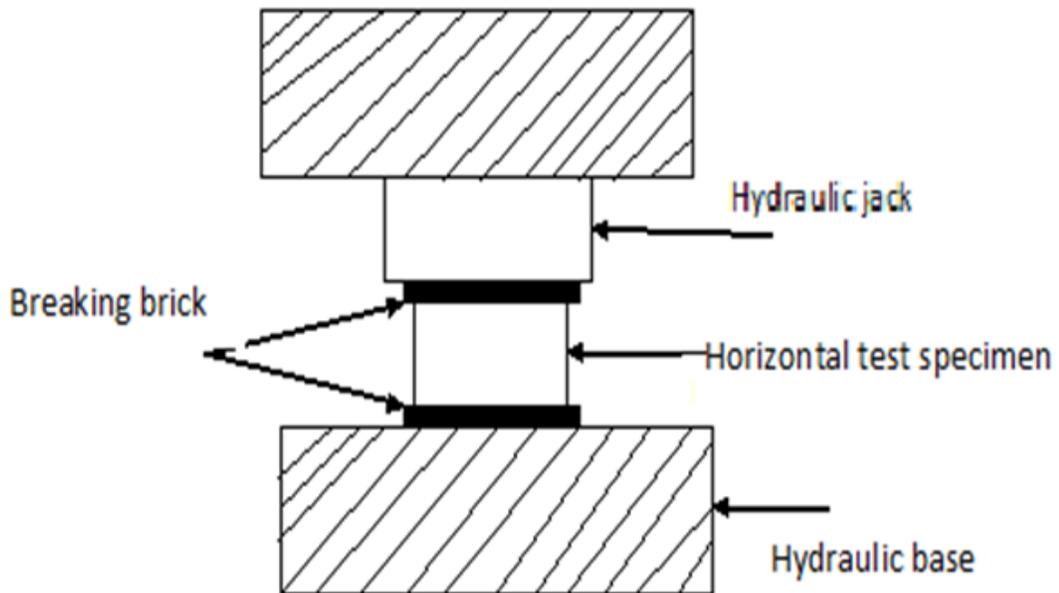


Figure2.2: cross section of cold crushing

According to Aigbodion *et al.* (2010), cold crushing strength increases with decreasing particle size. Figure 2.3 demonstrates how variation in particle size influences cold crushing strength at 0.09mm, 0.19mm, 0.29mm and 0.35mm were 55.9MN/m², 42.17 MN/m², 32.36 MN/m² and 29.42 MN/m² respectively.

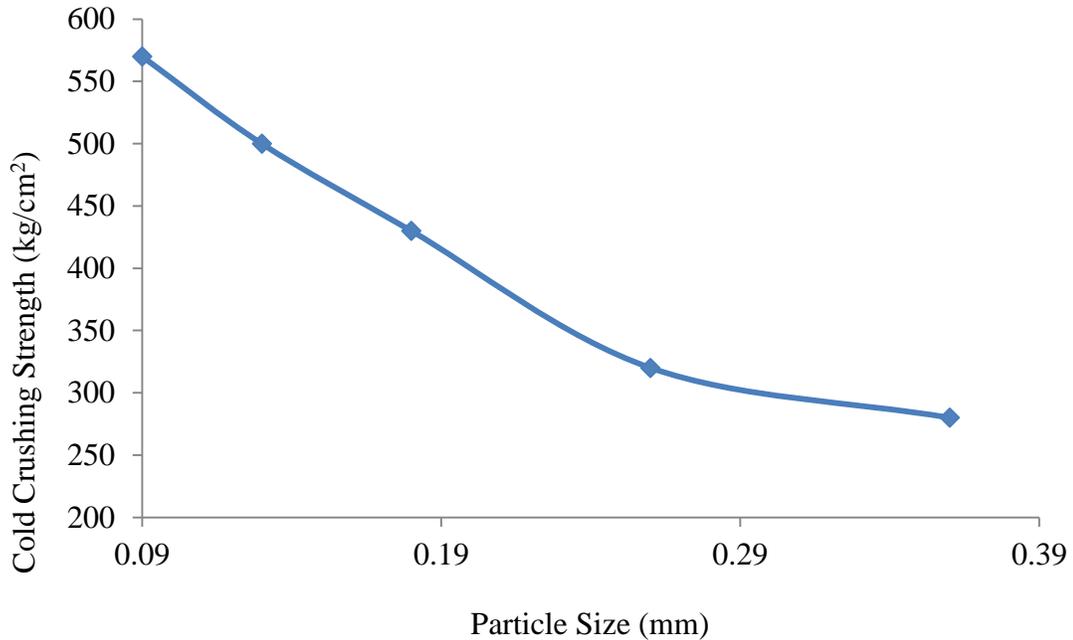


Figure 2.3: Effect of particle size on cold crushing strength.

(Adopted from Aigbodion *et al.*, 2010).

Omotoyinbo and Oluwole (2008) concluded that the presence of high silica content and alkali metal in clay are responsible for high compressive strengths. The findings by Abolarin *et al.*, (2008) on the cold crushing strength of 32 MN/m² were comparable to standard values of high duty (28 – 35MN/m²) silica bricks. This indicated that the bricks were properly fired and can easily be transported without damages. It also shows the ability of the clay to withstand abrasion and loading.

2.5 Thermal properties of refractory bricks

The thermal properties of refractory bricks include refractoriness, linear fire shrinkage, and coefficient of thermal expansion and loss of ignition which are discussed in the subsequent sub topics.

2.5.1 Linear firing shrinkage

Refractories are produced in the form of standard bricks. Total shrinkage of refractory brick indicates the size of the mould and generally, high shrinkage indicates a lower melting point. Linear firing shrinkage indicates how fusibility of the mixture increases with decrease in

particle size as shown in Figure 2.5. The linear shrinkage value of 3% and 3.6% obtained in the Tin tailings is below the recommended maximum value of 7% taken as adequate for refractory production (Aigbodion *et al.* 2010).

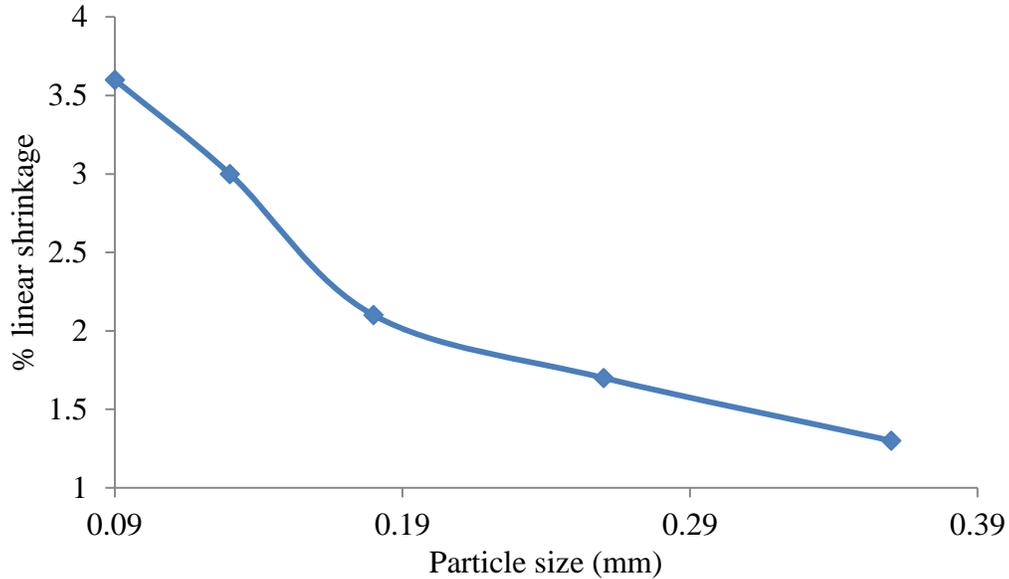


Figure 2.4: Effect of particle size on linear shrinkage.

(Adopted from Aigbodion *et al.*, 2010).

According to Omowumi (2001), the linear shrinkage of 3.5% was lower than the recommended range of 4-10% for fireclay but was more desirable since higher shrinkage values results in warping and cracking of the brick which may cause loss of heat in the furnace. The linear firing shrinkage of the blended sample after drying and firing is generally low and ranges from 0.8% to 3%. This is due to variation in chemical composition, particle size and apparent porosity. Chukwudu (2008) reported that the linear drying shrinkage of 9.9% and linear firing shrinkage of 13.9% compared to the recommended linear shrinkage of 7 % to 10% for refractory clays.

2.5.2 Loss on ignition

Loss On Ignition (LOI) is the combustion of volatile matter present in the clay. Addullahi and Samaila (2007) reported losses on ignition for samples to be between 11.5% to 14.05%, which was lower than the 18% specified upper limit for refractory clays. LOI is calculated using equation 2.3.

$$LOI = \frac{W_2 - W_3}{W_2 - W_1} \times 100\% \text{ ----- 2.3}$$

Where: W_1 = Dry crucible

W_2 = Dry sample plus crucible

W_3 = Fired sample plus crucible.

2.5.3 Thermal shock resistance

Thermal stability is the ability of a refractory to withstand sudden temperature changes without fracture. The number of thermal cycles (heating and abrupt quenching in water or air) a refractory brick can undergo characterizes its thermal shock resistance.

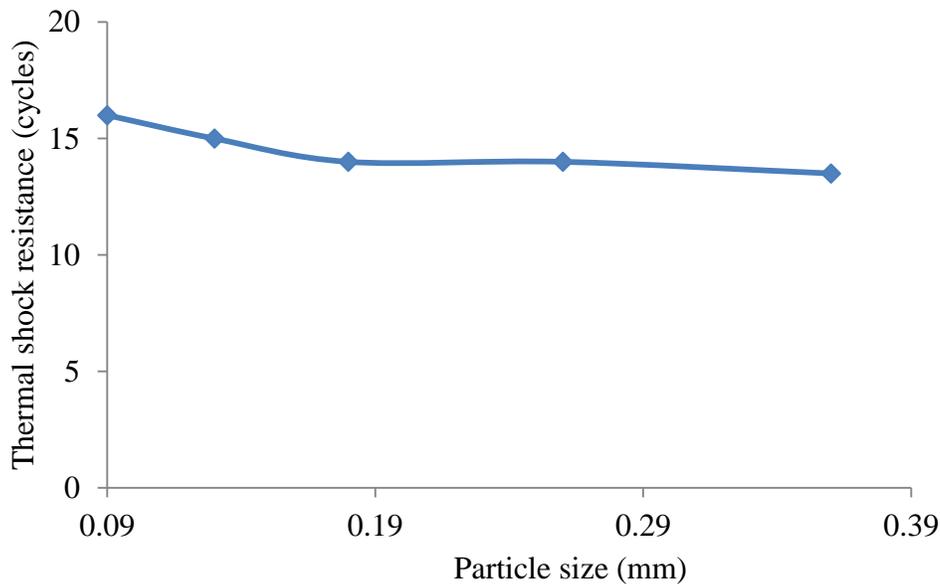


Figure 2.5: Effect of particle size on thermal shock resistance.

(Adopted from Aigbodion *et al.*, 2010).

Thermal shock resistance of the Tin tailings increases as the particle size decreases which indicates poor characteristics. For bricks, it increases from 14 to 16 cycles at 0.09mm particle size as shown in Figure 2.6. However, for blended clay, it falls within the accepted range of 15+ cycles (Aigbodion *et al.*, 2010). Thermal shock resistance of a material is influenced by the particle size, coefficient of linear expansion and thermal conductivity. Refractory materials with low thermal coefficient of expansion and coarse textures have increased resistance to sudden changes in temperature (Omotoyinbo and Oluwole 2008) Chukwudi (2008), reported the number of cycles to failure to be 32 cycles for Nsu clay deposit in Imo state of Nigeria. This value

narrowly falls outside the 20-30 numbers of cycles recommended by Chester (1973). According to Addullahi and Samaila (2007), the thermal shock is acceptable if it falls between 25-30 cycles for Gur and Yamarkumi clays in Nigeria.

From the other researchers work referred in the literature review on the performance of chemical composition, physical and thermal properties approach are applied in characterization of kenyan clays and formulation thereof in chapter three accompanied by statistical analysis software at 5% level of significance.

2.54 Refractoriness (fusion point)

Refractories melt progressively over a wide range of temperatures depending on their chemical compositions. Refractoriness or fusion point is ideally assessed by the cone fusion method. Alumina content of clay determines its refractoriness and presence of alkali metals in the clay usually lowers its fusion temperature (Chesti, 1986). The equivalent standard cone which melts to the same extent as the test cone is known as the Pyrometric Cone Equivalent (PCE). According to ASTM C24- 09 (2013), standard PCE is measured by making a cone of the material and firing it until it bends to a 3 O'clock position giving a PCE value of 28 and 27.

The test pieces are generally made to form triangular pyramids having a height 4 times the base. Thus, the predetermined pyrometric cone equivalents of standard test pieces are placed along with cone made of the samples to be tested in the furnace and the PCE's of the samples are determined by comparison. Softening point is noticed when the tip of the cone starts bending with the rise of the temperature. It has been observed that, the higher the alumina content in the fireclay, the higher the fusion point. Tin tailing from Jos, Nigeria has a refractoriness of 1600⁰C which corresponds with the standard value for medium heat duty and high heat duty of the Indian fireclay refractory (Aigbodion *et al.*, 2010). Nnuka and Agbo (2000) noted that Otukpo clay had refractoriness of 1710⁰C, which was similar to imported refractories into Nigeria. Ire clay golden was below 1500⁰C while Ire clay purple was above 1500⁰C (Abolarin *et al.* 2004).

Chukwudu (2008), reporting on refractory properties of Nsu clay deposit in Imo state of Nigeria showed that refractoriness of the sample occurred at a temperature of 1683⁰C, attributed this to appreciation of the alumina content (36.2wt %) in the fired sample. Representative PCE values for selected refractories include cones 33-34 for super duty fireclay, cones 29-31 and 36-

37 for medium duty fire clay and 60% alumina product, respectively. The cone values reported for refractories are based on a defined standard time-temperature relationship, such that different heating rates will result in different PCE values. PCE can be useful for quality control purposes to detect variations in composition of raw material formulation. Refractoriness and plasticity are the two main properties required in fireclay for its suitability in the manufacture of refractory bricks. Depending upon their capacity to withstand high temperatures before melting, the fireclays are graded into low duty that withstands temperatures between 1515-1615°C (PCE 19 to 28) while intermediate duty withstands upto 1650°C (PCE 30), high duty upto 1700°C (PCE 32) and super duty upto 1775°C (PCE 35).

CHAPTER THREE

3 MATERIALS AND METHODS

3.1 Characterization of clays

Description

The raw materials used in this study were kaolin, bauxite and ball clays due to their availability and good properties such as high alumina and silica content. Kaolin, bauxite, Kisii soapstone and Salama clay were collected from Naivasha (Eburru Complex), Kericho (Kipchimchim), Kisii (Tabaka) and Machakos, while the ball clay was sourced from Nyeri (Mukurweini).

Chemical properties were analysed at the Ministry of Environment and Minerals Resources, Department of Mines and Geology. These clays were dried, crushed in to powder form and pelleted. The selected clay samples of kaolin, Kisii soapstone, bauxite, salama and ball clay were subjected to chemical composition analyses using Panalytical B.V 4KW Energy dispersive X-ray spectrometer. The samples for analysis were dried at a temperature of 250⁰ C (Refer to Plate A.1 in the appendix) to remove moisture and crushed into powder form. Twelve grams of each clay sample (refer to Plate A.2) was then grounded in an agate mortar, 1.5g of a binder (used in laboratory) was added, before mixing and pressing in a hydraulic press into a pellet (Plate A.3). The pellet was loaded into the sample chamber (Plate A.4) of the spectrometer. A voltage of 30KV and a current of 1mA were applied to produce X-rays to excite the sample for a preset time of 10mins. The spectrum from the samples was then analyzed to determine the concentration of Al₂O₃, SiO₂, CaO, MgO, Fe₂O₃, Na₂O, K₂O and LOI.

One hundred grams of each clay sample was dried at 110°C and cooled in the desiccators (Plate A.5) to room temperature. A clean and dry porcelain crucible (Plate A.6) was then weighed (W_1) and the dry sample plus crucible were weighed (W_2). The crucible with the sample was placed in a muffle furnace and heated to a temperature of 1000°C for 14 hours. Eventually, the crucible and its contents were cooled in a desiccators to room temperature and then weighed (W_3). The data obtained was analysed using equation 2.3.

3.2 Physical and thermal properties of composite refractory bricks

3.2.1 Production of composite bricks

Mould used to developed refractory bricks could only hold a maximum of 1000g of clay. Sample was made of individual material or mixture of kaolin and bauxite plus 10% binder, therefore the remain space in the mould of 900gms was proportioned to kaolin and bauxite as indicated in Table 3.1.

The mix ratios of kaolin (Ka), bauxite (Ba) and ball (Bc) clays were prepared as shown in Table 3.1. The physical and thermal properties of the developed bricks that were investigated included porosity, bulk density, cold crushing strength, thermal shock resistance and linear shrinkage.

Table 3 1: : Mix ratios for different clay materials

Treatment	(Ka :Ba) Ratios	% binder (Bc)
1	0:9:1	10
2	2:7:1	10
3	3:6:1	10
4	4:4:1	10
5	6:3:1	10
6	7:2:1	10
7	9:0:1	10

Where; Ka is kaolin, Ba is Bauxite, Bc is Ball clay.

(a) Production flow chart

The research followed the procedure outlined in Figure 3.1 to produce the composite refractory bricks; it shows various stages of the experimental set up employed in the study. The excavations were done in the respective quarries, preliminary crushing and chemical analysis were carried out at the Ministry of Environment and Minerals Resources, Department of Mines and Geology. Samples were mixed in different ratios before being compressed, bonded and fired. Preliminary drying and testing for quality control were analysed at Kenya Industrial Research and Development Institute (KIRDI).

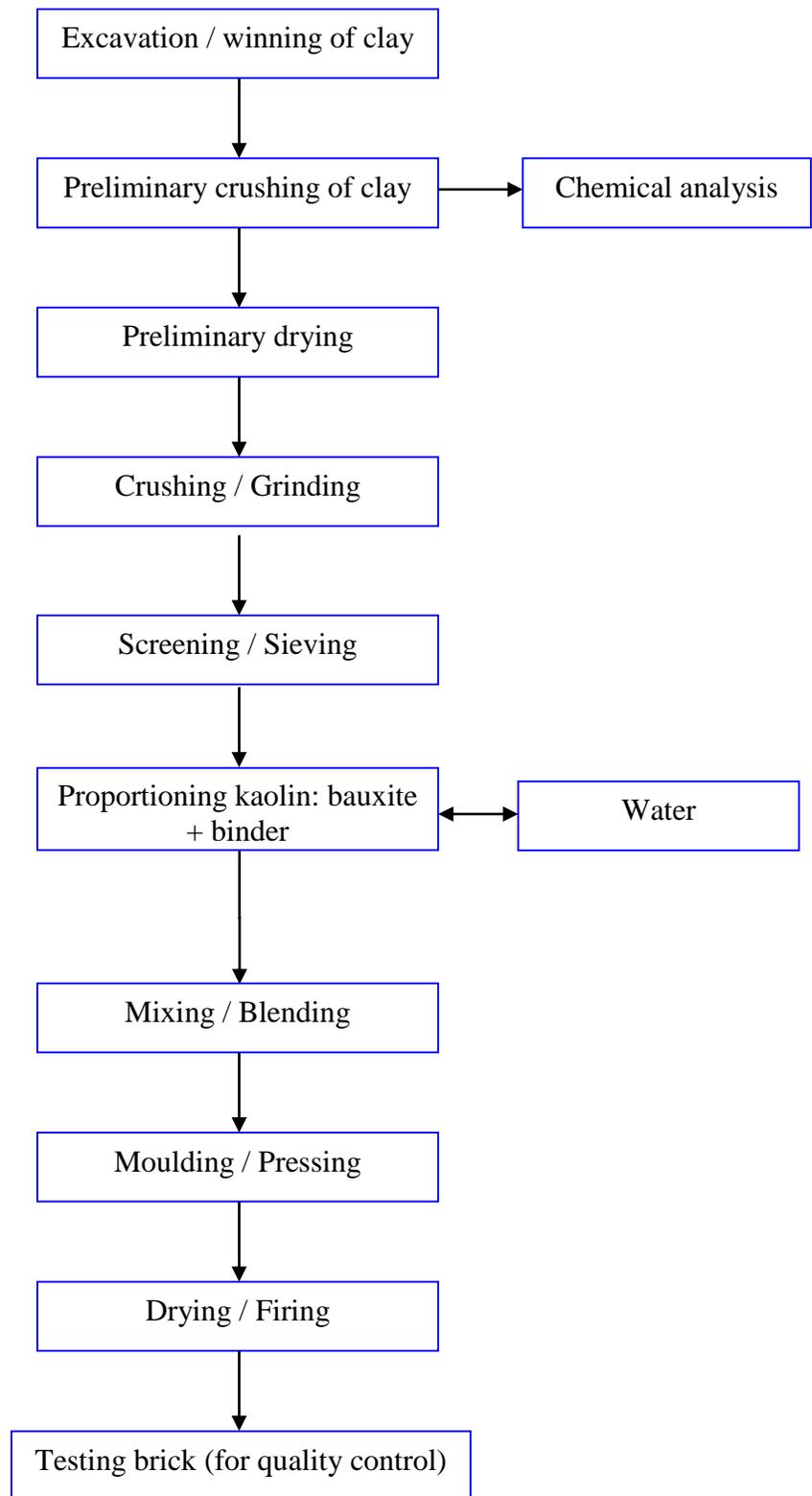


Figure 3.1: Production Flow chart and testing.

(b) Moulding and firing composite bricks

Kaolin, bauxite, Salama clay, Kisii soapstone and ball clays were dried and thoroughly mixed to obtain a homogeneous sample (Plate A.7) and 8% of water was added to improve plasticity. The samples were stored in a stop pad container for 24hrs to allow moisture distribution and weathering, which improves plasticity in the clays. The samples were put in a cubical mould box (Plate A.8) of volume 70x70x70mm and pressed using a hydraulic jack to a pressure of 4.1N/m². The bricks were then withdrawn from the mould (plate A.9) and weighed before leaving them in open air for 24hrs to dry naturally. The bricks were then placed in an oven (Plate A.10) for 24 hrs at a temperature of 110°C to expel any moisture as recommended by AddulRasheed (2009), and Aigbodion (2007).

The dried brick was fired in an automatic digital electric furnace (Plate A.11) at a heating rate of 7°C/min up to 200°C for 6 hours, 650°C for 3 hours, 950°C for 4 hours and 1250°C for 8 hours, as indicated in Figure 3.2 as recommended by Al-Amaireh (2009). After firing, the bricks were cooled in the furnace at a rate of 1°C/min as recommended by Aigbodion *et al.* (2007) and Chesti (1986). Results for five replications for each ratio and sample are presented in section 4.2.

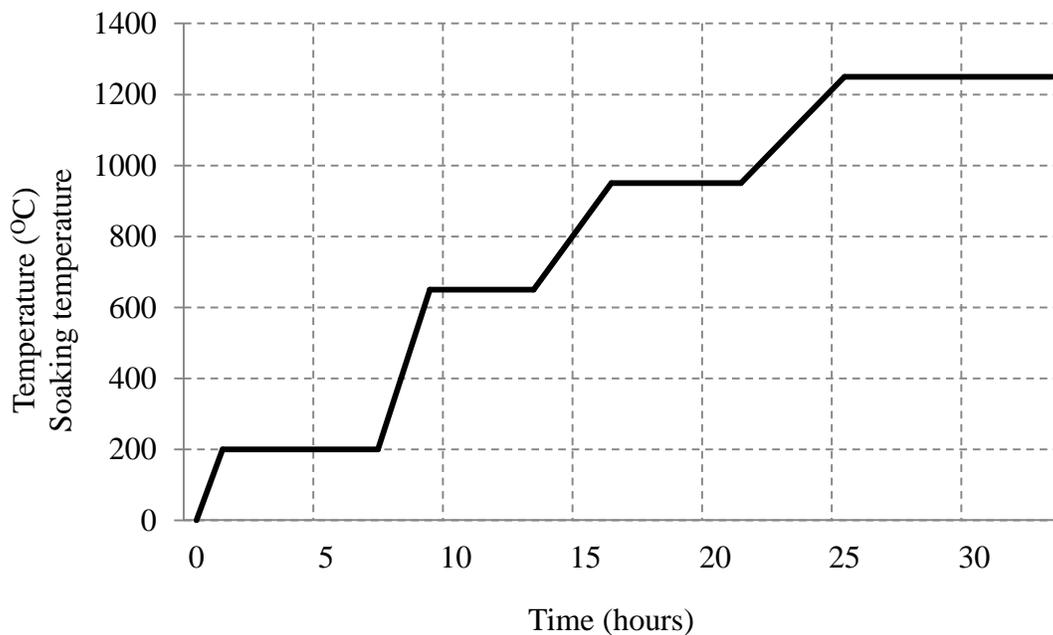


Figure 3.2: Heating Regime.

(Adopted from Al-Amaireh, 2009).

3.2.2 Properties of composite brick

(a) Apparent porosity

Apparent porosity (AP) of the developed bricks from each mix ratio was determined in accordance with ASTM C20-00 (2015). The cuboid samples of measuring 70mm were initially dried in oven at 110°C for 24 hours ASTM C20-00, 2015 (Plate A. 10) to obtain a constant weight which was recorded as D using digital weighing scale. The dry sample was suspended in distilled water (plate A.12) and boiled for two hours. It was left suspended in water to cool at room temperature and its weight recorded as S. Afterwards, it was removed from distilled water and the surface wiped off. The sample was then weighed and recorded as W. Eventually, apparent porosity was calculated using equation 3.1

$$AP = \frac{S - D}{S - W} \times 100 \% \text{ -----3.1}$$

where; AP is Apparent Porosity, D is dry weight, S is Suspended weight in distilled water and W is Weight in air.

Results for five replications (appendix c) for each ratio are presented and discussed in section 4.2.

(b) Bulk density

The procedures used on apparent porosity test were applied to determine bulk density (ASTM C20) since it is a function of the method of manufacture. Bulk densities of the bricks were determined using equation 3.2.

$$BD = \frac{D}{S - W} \times 100 \% \text{ -----3.2}$$

where: BD is Bulk density, D is dry weight, S is Suspended weight in distilled water and W is Weight in air.

Results for two replications (appendix c) for each ratio are presented in section 4.2

(c) Cold crushing strength

Cold crushing strength (CCS) is the ability of the refractory clay materials to withstand abrasion and loading without damaging or crumbling into powdered form after it has been fired

to a temperature of 1250°C. The cold crushing strength of a refractory sample was determined in accordance with ASTM C133-97 (2015) standard. Standard bricks measuring 70mm cube were tested for strength under universal strength testing machine, take readings of load/area at failure, CCS was obtained using Eq. 3.3 as used by Hassan, (2000).

$$CCS = \frac{\text{Maximum load (MN)}}{\text{Cross sectional area (m}^2\text{)}} \text{-----}3.3$$

Results for five replications for each ratio are presented and discussed in section 4.2

(d) Linear shrinkage

The samples of the refractory materials were moulded into cubes with sides of 70 mm and hydraulically compacted at 4.1N/m². A green brick length (undried brick) was measured on each sample and recorded as L₁. The samples were dried in an oven at 110⁰C for 24hrs, then placed inside a furnace preset at 1250⁰C before cooling to room temperature at a rate of 1⁰C/min. Linear shrinkage of the samples was then determined in accordance with ASTM C 356 (2014) standard using equation 3.4.

$$\% LS = \frac{L_1 - L_2}{L_1} \times 100 \% \text{-----}3.4$$

where: LS is linear shrinkage, L₁ is green brick length and L₂ is fired length.

Results for five replications for each ratio are presented and discussed in section 4.2

(e) Thermal shock resistance

Thermal shock resistance was determined by heating the sample in a furnace to a preset temperature of 1100°C for 30 minutes. Afterwards, the sample was removed from the furnace and cooled for 10 minutes in accordance with ASTM C1171 (2015), Standard. The sample was recharged for another 10 minutes at 1100⁰C and then cooled again for 10 minutes. This cycle of heating and cooling was repeated until the brick fractures. The number of complete cycles before occurrence of failure on each sample was taken as the measure of the thermal shock resistance according to Aigbodion (2007). Results for five replications (appendix c) for each ratio are presented and discussed in section 4.2

3.4 Effect of particle sizes on physical and thermal properties of composite refractory bricks

The clay materials at a mix ratio of 7:2 (based on results obtained in section 3.2) were crushed (Plate A.1) and thereafter passed through a set of three sieves of aperture of 600 μ m, 380 μ m and 150 μ m corresponding to American Society of Testing Materials (ASTM) standards sieves numbers 30, 50 and 100, respectively. These sieved clay materials were then dried in the sun for one week before packing them in labeled plastic bags to avoid absorbing moisture from the atmosphere. Apparent porosity, bulk density, cold crushing strength, linear shrinkages and thermal shock resistances of the samples were tested using the procedures employed in section 3.2. Results for five replications (Appendix D) for each ratio are presented and discussed in section 4.3

3.5 Experimental design and data analysis

Statistical Analysis Software (SAS) was used to analyse physical and thermal properties of composite refractory bricks. The properties were subjected to Two-way Analysis of variance (ANOVA) and which was used to examine whether there was significant difference in their means. Least Significant Differences (LSD) was determined at 5% level of significance. The input of data and output of SAS version 8.1 are presented in Appendices B and C.

The output of SAS on the performance characteristics mentioned above are tabulated in mean tables and discussions thereof in chapter four accompanied by observations made on the results.

CHAPTER FOUR
RESULTS AND DISCUSSIONS

In this study, physical and thermal properties of kaolin, bauxite and ball clays were characterized and performance of the developed composite refractory bricks evaluated on mix ratios and particle size. The physical and thermal properties investigated were apparent porosity, bulk density, cold crushing strength, linear shrinkage and thermal shock resistance.

4.1 Chemical composition of kaolin, bauxite and ball clays

This section includes the details of the chemical analysis performed. The chemical composition of selected Kenyan clays is given in Table 4.1 and Appendix B-1(a) and (b)

Table 4. 1: Chemical composition (wt %) of clays and ASTM values

Clay	Al ₂ O ₃	SiO ₂	CaO	MgO	TiO	Fe ₂ O ₃	Na ₂ O	K ₂ O	LOI%
Kaolin	30 ^b	55 ^a	0.1 ^d	0.2 ^b	1.1 ^c	0.7 ^d	0.2 ^b	0.1 ^c	7 ^e
Soapstone	34 ^a	46 ^c	0.2 ^c	0.1 ^c	1.1 ^c	0.3 ^d	1.3 ^a	5.6 ^a	8 ^d
Salama	20 ^d	50 ^b	0.3 ^b	0.1 ^c	1.4 ^b	16.0 ^a	0.4 ^b	1.2 ^a	14 ^b
Bauxite	25 ^c	46 ^c	1.3 ^a	0.0 ^c	1.4 ^b	9.4 ^b	0.1 ^b	0.1 ^c	12 ^c
Ball clay	31 ^b	47 ^c	0.1 ^d	0.3 ^a	2.5 ^a	3.3 ^c	0.4 ^b	1.0 ^a	15 ^a
ASTM Values	25 - 39	46 - 62	0.2 – 1.0		0.4 - 2.7		0.3 – 3.0		8 - 18
Average ASTM	32	54	0.6	0.6	1.5		1.6	1.6	13
LSD	0.93	0.93	0.07	0.07	0.09	0.43	0.08	0.07	0.93

NB: The values followed by the same letters (superscript, a, b, c, d or e) in the same column are not significantly different at $\alpha= 0.05$ and their Least Significant Difference (LSD).

The aluminum oxide in kaolin (30%) and ball (31%) clays are not significantly different at $\alpha= 0.05$ and are within acceptable limits as per ASTM values of 25-39% (Nnuka and Agbo,2000) . In kaolin, bauxite, soapstone and ball clays, aluminum oxide (Al₂O₃) ranged between 25% and

34% and silica (SiO_2) between 46% and 55% which lies within the ASTM values of 25 - 39% and 46 - 62, respectively, agrees with Omotoyinbo and Oluwole (2008) who reported Al_2O_3 ranging from 26.29 to 30.68% and SiO_2 from 45.22 to 48.66%. These are major constituents which make them suitable as refractory materials. The mean values of aluminum oxide (Al_2O_3) for Salama (20%) clay agree with those reported by Abdullahi and Samaila (2007) of 21.25% and 19.68%. Both values were found to be applicable as high melting clay but not as refractory clay. The alumina content in clay is a strong indicator of its refractoriness and the higher the amount of alumina in clay, the better is the refractoriness of that clay. Silica oxide content in bauxite (46%), soapstone (46%) and ball clay (47%) are not significantly different at 5% level of significance which agrees with the findings by Yami and Umaru (2007) of 41.8 - 54.75%. These results fall within the ASTM range for refractory clay of 46 - 62% by Nnuka and Agbo (2000). However, soapstone contained high values (5.6%) of potassium oxide (K_2O) which exceeded the refractory standard ASTM of 0.3 to 3%. Since, high potassium in clays influences formation of glass during sintering process at 1250°C , soapstone could not qualify as a refractory material at elevated temperatures.

Salama clay had 16% of iron oxide which was higher than 11.39% recommended by Mazen (2009) and Ndaliman (2007). Onyeji (2010) stated that clays with appreciable amounts of iron cannot be used as sources of aluminium as the presence of iron tends to influence early melting points of refractory materials during the firing process. The high iron oxide content in salama clay also tends to affect its firing strength. Therefore, salama clay failed to meet minimum requirement as refractory clay material. The quantities for CaO and MgO impurities in the entire samples fell within the recommended ASTM standard of 0.2 - 1.0%.

Based on the results obtained on chemical composition analysis, Kaolin, bauxite and ball clay (binder) met the minimum requirements as refractory clay materials. Therefore the three clays were chosen for refractory brick production and their physical and thermal properties on composite bricks manufacture.

The loss on ignition (LOI) of refractory clays was determined as the percentage of moisture loss to ignition on firing the clay samples. The results indicated that ball and kaolin clays had the highest and lowest percentage of LOI the values being 15% and 7%, respectively. These values represented the amount of moisture the clay materials could hold (or the percentage weight

reduction of samples) which is a reflection of their grain structure and fines. This suggests that ball clay is of finer grains and more compact compared to the rest. The LOI values for all the five Kenyan clays agree with values recommended by ASTM values Nnuka and Agbo(2000) . Addullahi and Samaila (2007) reported values of 11.5% – 14.05% which is similar to those of Kenyan salama, bauxite, ball clays.

4.2 Physical and thermal properties of refractory bricks

4.2.1 Physical properties

(a) Colour appearance and observation

Figure 4.1 presents unfired (physical appearance of green-composite brick) bricks made of individual clay materials. After firing, observations were made to determine clays that met minimum requirements for purposes of narrowing down to an appropriate composite refractory material. The physical colour of unfired individual kaolin, bauxite, soapstone, salama and ball clays were ivory white, brown, ivory white, sienna brown and dim grey, respectively.



Figure 4.1: Various colours of unfired/ green bricks.

(Where: BC is Ball clay, NC is Kaolin, BX is Bauxite, SC is Salama clay and KSI is Kisii soft stone).

After the sintering process at 1250⁰C, the colour of bricks turned white, reddish, snow white, wine red and tan for kaolin, salama, soapstone, bauxite and ball clays as shown in Figure 4.2. The red colour indicates the presence of Fe₂O₃ content.



Figure 4.2: Fired bricks of individual clays.

The physical appearance of these bricks after firing revealed that at sintering temperature of 1250°C soapstone (snow-white) started to form glass due to high levels of potassium (5.6%). Salama and ball clays experienced several cracks at lower temperatures due to the presence of iron oxide and reaction of water of crystallization. In addition, bauxite bricks chipped off more than those made from kaolin which demonstrated low plasticity and incomplete sintering process. Hence, observations made on the fired bricks made of individual clay materials led to the conclusion that mixed ratios of kaolin, bauxite and ball clays would be suitable for manufacturing composite refractory bricks. Results obtained agrees with Omotoyinbo and Oluwole (2008) who reported that aluminum oxide (Al_2O_3) of 26.29 – 30.68% and silica (SiO_2) 45.22 – 48.66% are suitable as refractory materials.

To ascertain the best binder ratio, mix ratios of kaolin and bauxite were subjected to varying ratios of ball clay at 5%, 10%, 15% and 20%, respectively as shown in Figure 4.3 and judged on consistency, holding, colour and zero cracks. Ball clay of 10% binder was found ideal for the mix ratios.



Figure 4.3: Binder ratios.

Refractory bricks of different mixed ratios were prepared using the procedures mentioned on the individual materials stipulated in section 3.3. Different bricks from the five mix ratios were replicated five (5) times and were as shown Figure 4.4. and fired to a maximum temperature of 1250°C.



Figure 4.4: Fired bricks samples at different mix ratios.

The results of apparent porosity, bulk density, cold crushing strength, linear shrinkage and thermal shock resistance for each mix ratios are shown in Table 4.2 and Figures 4.5 through 4.9.

Table 4. 2: Physical and thermal properties of kaolin and bauxite clays.

Ka:Ba:Bc Ratios	AP(%)	BD (g/cm ³)	CCS (MN/m ²)	LFS (%)	TSR (CYC)
0:9:1	43 ^a	1.56 ^c	17 ^f	9.7 ^a	8 ^f
2:7:1	38 ^b	1.56 ^c	22 ^e	8.89 ^b	14 ^e
3:6:1	36 ^c	1.54 ^c	24 ^d	7.23 ^c	15 ^e
4:4:1	35 ^c	1.60 ^c	27 ^c	6.43 ^d	20 ^c
6:3:1	33 ^d	2.03 ^a	30 ^b	4.44 ^e	22 ^b
7:2:1	29 ^e	2.23 ^b	33 ^a	3.69 ^e	27 ^a
9:0:1	27 ^f	1.60 ^c	27 ^c	2.81 ^f	18 ^d
(ASTM)	20-30	1.71-2.10	15	4-10	20-30
LSD	1.716	0.102	1.0634	0.2353	1.2484

Means followed by the same letter superscript (a, b, c, d, e or f) are not significantly different at $\alpha=0.05$ and Least Significant Difference (LSD).

where Ka = kaolin, Ba = bauxite, Bc = ball, AP = Apparent porosity, BD = Bulk Density, CCS = Cold crushing strength LFS = Linear Fired Shrinkage, TSR = Thermal Shock Resistance and CYC = Cycle

(b) Apparent porosity

Apparent porosity is a key property on brick insulation and heat conservation in a furnace or kiln. Low apparent porosity indicates better insulation and heat retention of refractory bricks. From the results presented in Table 4.2 and Figure 4.5, it is evident that apparent porosity depends on the characteristics of the original material. The apparent porosity of the pure kaolin and bauxite clay alone were 27% and 43%, respectively.

The apparent porosity for bauxite was higher than for kaolin which could be attributed to the more air pockets contained in it. The lower porosity of the refractory brick, the better the insulation of the brick for refractoriness. Results show that if kaolin and bauxite are mixed in ratios of 6:3:1 and 4:4:1, they would exhibit similar but inferior apparent porosity of 36-38% at LSD of 1.7.

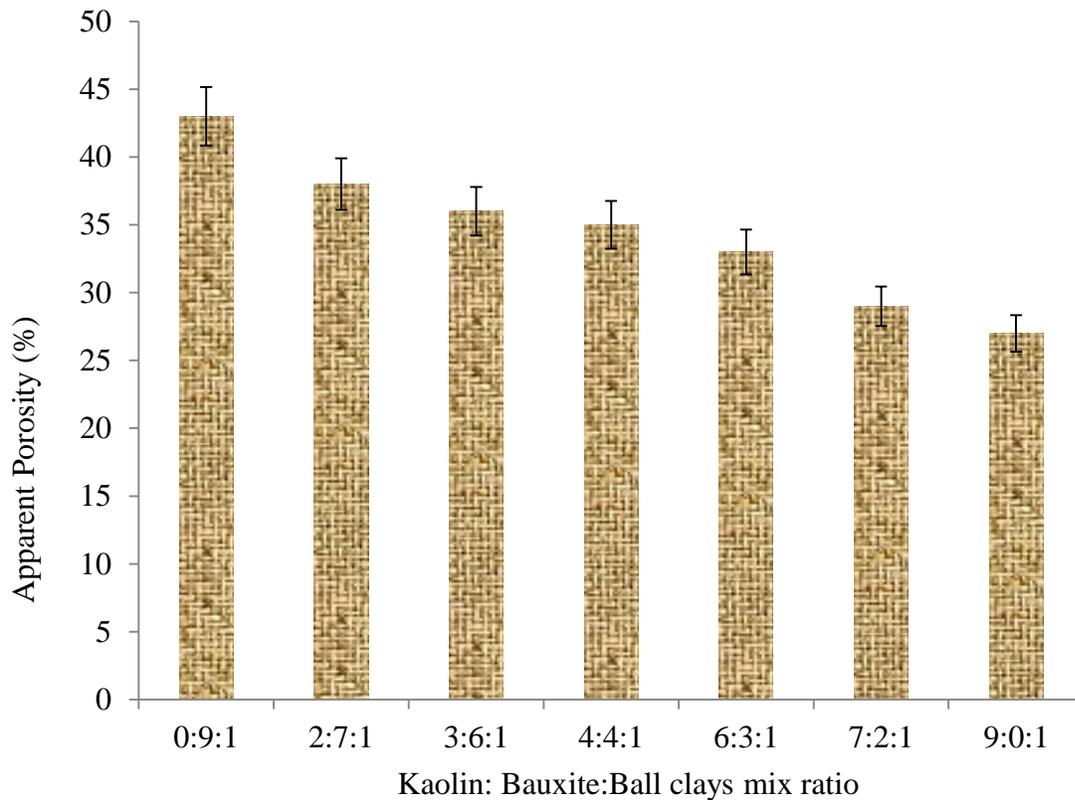


Figure 4.5: Effect of mix ratios on apparent porosity.

The apparent porosity values of 27% and 29% for kaolin and 7:2:1 (kaolin and bauxite) mix ratio, respectively falls within the values specified by ASTM C20-00 (2015) of 20 – 30%. Mix ratios 2:7:1, 3:6:1 and 0:9:1 had the highest porosity of 38%, 36% and 43%, respectively which were much higher than the standard. Error bars on mix ratios 2:7:1, 3:6:1, 4:4:1 and 6:3:1 overlap which shows that they are not significantly different at $\alpha = 0.05$ and LSD of 1.7. This implies that these ratios are not appropriate for refractory bricks since the apparent porosity is higher than the ASTM values. This finding implies that when kaolin was mixed with bauxite at a ratio of 7:2, apparent porosity improved from 43% to 29% which qualifies it as refractory brick material. It was also realized that as the quantity of kaolin was increased in the mix, the apparent porosity decreased from 38% to 29%, this is due to the voids and fineness in kaolin clay.

(c) Bulk density of composite bricks

High bulk density is commonly desired for clay refractories because high fired-density bricks usually confer high physical strength at high service temperatures.

Effects of mix ratios on bulk density are presented in Table 4.2 and Figure 4.6. The bulk densities for kaolin and bauxite clays were 1.60g/cm^3 and 1.56g/cm^3 , respectively. It is evident from these results that bulk density is dependent upon the characteristics of the original material and increased proportionally with increase in kaolin. As the quantity of kaolin in the ratio was increased from 20% to 70%, bulk density increased from 1.54g/cm^3 to 2.23g/cm^3 . This could be attributed to the porosity of bauxite clay which is higher than that of kaolin. This suggests that kaolin has higher specific gravity compared to bauxite which depends on the type of raw materials as was observed by Mazen (2009).

The bulk densities obtained with ratios of 2:7:1, 6:3:1, 4:4:1, kaolin and bauxite were not different at $\alpha= 0.05$ and *LSD* of 0.102 but are lower than the ASTM values of $1.71 - 2.10\text{g/cm}^3$. Mix ratios of 6:3:1 and 7:2:1 had recommendable values since they met the minimum requirements for refractory bricks.

The value of bulk density equal to 2.03g/cm^3 lies within the findings of Yami & Umaru (2007) and Abolarin *et al.*, (2004), who reported values of $2.02\text{-}2.11\text{ g/cm}^3$ and $1.94 - 2.04\text{ g/cm}^3$, respectively.

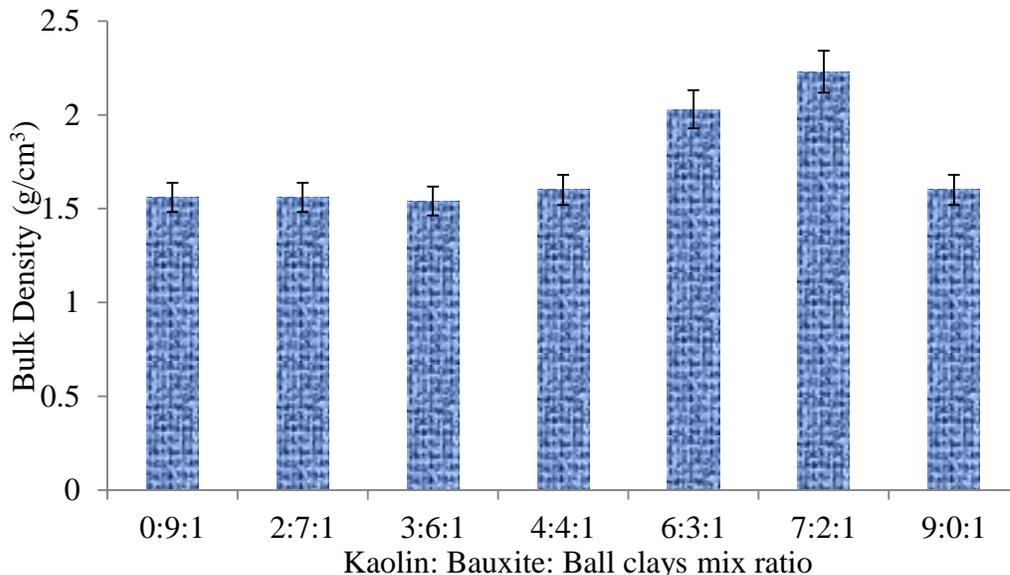


Figure 4.6: Effect of mix ratios on bulk density.

(d) Cold crushing strength

Cold crushing strength indicates the ability to withstand handling, transportation and installation of a refractory brick at low temperatures. Table 4.2 presents mean values of cold crushing strength for various ratios of kaolin to bauxite bounded by ball clay while Figure 4.7 is used to compare cold crushing strengths using error bars at 5% level of significance with LSD of 1.1. The values obtained account for good bonding and vitrification during firing.

Bricks from Kaolin and bauxite presented cold strengths of 27MN/m² and 17MN/m², respectively. This shows that pure kaolin bricks has a higher cold crushing strength than pure bauxite bricks. Cold crushing strength for 2:7:1 ratio was 22MN/m² which increased to 33MN/m² as kaolin portion increased to 7:2:1. As Kaolin increases, the cold crushing strength increased too. This shows that cold crushing strength of the produced refractory brick depends on the original material since kenyan Kaolin has better chemical properties i.e silica content of 55% compared to 46% for bauxite, which is responsible for the higher strengths.

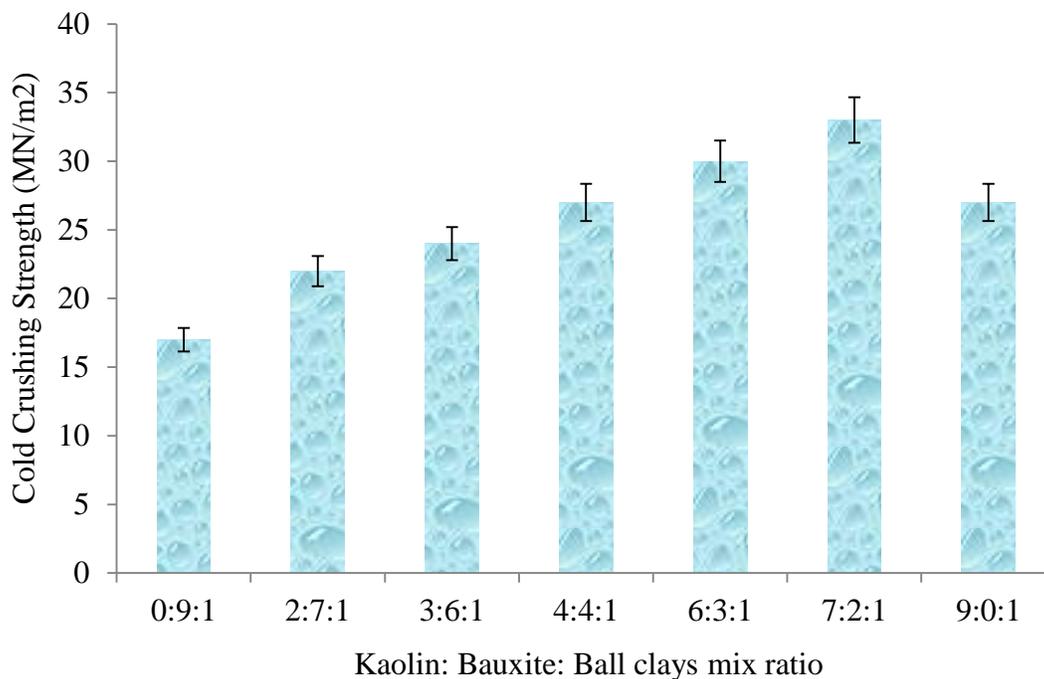


Figure 4.7: Effect of mix ratios on cold crushing strength.

The ratios 9:0:1 and 4:4:1 depict similar cold crushing strength property of 27MN/m² it is evident from their error bars which are overlapping, while other ratios portray different values at $\alpha= 0.05$ and LSD of 1.1.

The values of cold crushing strength obtained in this study range between 17MN/m² and 33 MN/m² all of which are higher than the ASTM C133-97 (2015) value of 15MN/m². This is due to high silica content of 46% to 55% as supported by Omotoyinbo and Oluwole (2008), who reported that the presence of high silica content and alkali in clay are responsible for high compressive strengths. This shows that the bricks had been properly fired which enables them to withstand loading and can easily be transported without damage as noted by Abolarin *et al.* (2008).

4.2.2 Thermal properties

(a) Linear shrinkage

Linear shrinkage is an important parameter in the standardization of refractory bricks. It is an indicator of the efficiency of a firing regime and indicates the size of a mould. The results on this parameter are presented in Table 4.2 and Figure 4.8.

It is observed that linear shrinkage is inversely proportional to increase in kaolin. At a mix ratio of 0:9:1, linear shrinkage was 12.62 % which reduced to 2.81% with increase in kaolin. This could be attributed to the particles sizes distribution in kaolin. Kaolin and bauxite mix ratios of 2:7:1, 3:6:1, 4:4:1, 6:3:1, 7:2:1 and 9:0:1 produced linear shrinkage values ranging from 8.89 to 2.81% which falls within the recommended limit of 4-10% ASTM C20-00(2015), Standard . It was also observed that all ratios produced different linear shrinkage values at $\alpha= 0.05$ and LSD of 0.24 and the un-overlapping error bars in Figure 4.8.

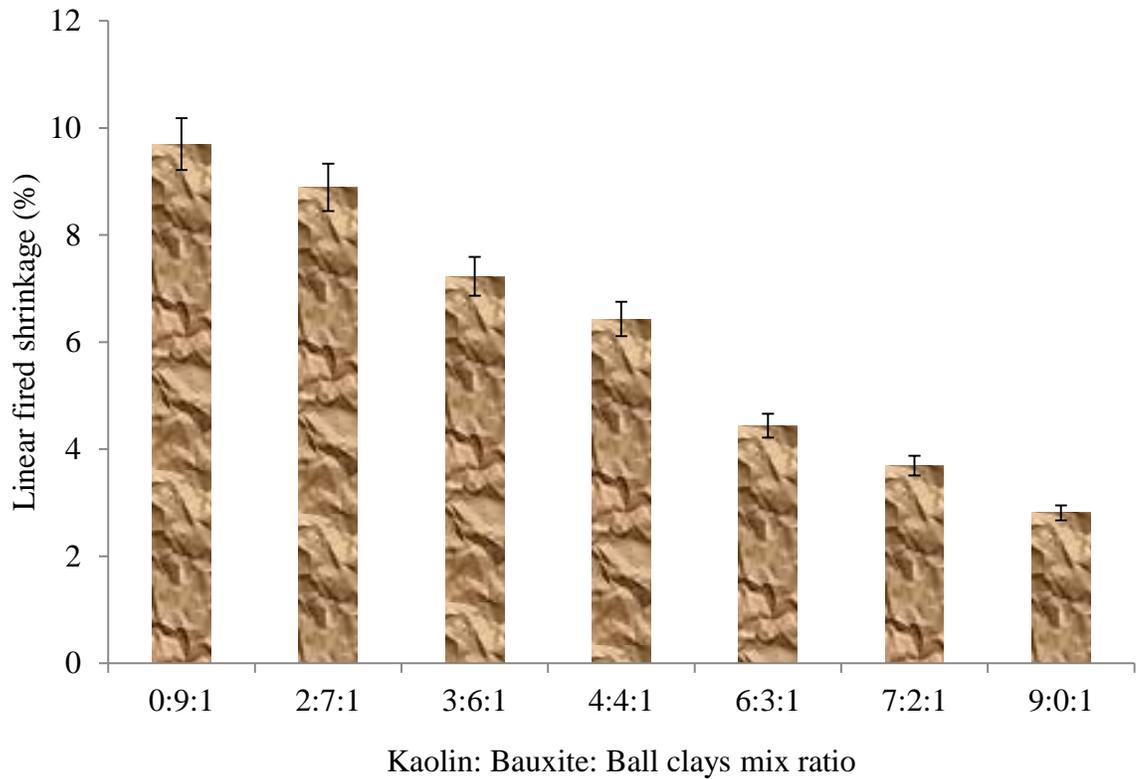


Figure 4.8: Effect of mix ratios on linear shrinkage.

Lower linear shrinkage values are more desirable and values below 4% would be acceptable as Abolarin *et al.* (2004) pointed out. The values of linear shrinkage using 7:2:1 and 9:0:1 ratios were 3.69 and 2.81, respectively, which compares reasonably well with values obtained by Aigbodion *et al.*, (2010) who reported 3 % to 3.6%. Hence, these two mix ratios are appropriate for manufacturing of composite refractory brick. This means that the brick produced would be less susceptible to volume change.

(b) Thermal shock resistance

Thermal shock resistance is the ability of a refractory brick to withstand sudden temperature changes without fracture. This is determined by the number of thermal cycles (heating and cooling) the refractory bricks withstood. The results of the means on this parameter are indicated in Table 4.2 and Figure 4.8.

The thermal shock resistance increased with increase in the amount of kaolin. Bricks without kaolin (0:9:1) had thermal shock resistance of 8 cycles and as kaolin content increased to 7:2:1, it increased to a maximum of 27 cycles. Kaolin and bauxite at ratios 4:4:1, 6:3:1 and 7:2:1

presented recommendable results of 20, 22, and 27 cycles, respectively. Thermal shock resistance for ratios 2:7:1 and 3:6:1 were not significantly different at $\alpha=0.05$ and LSD OF 1.25.

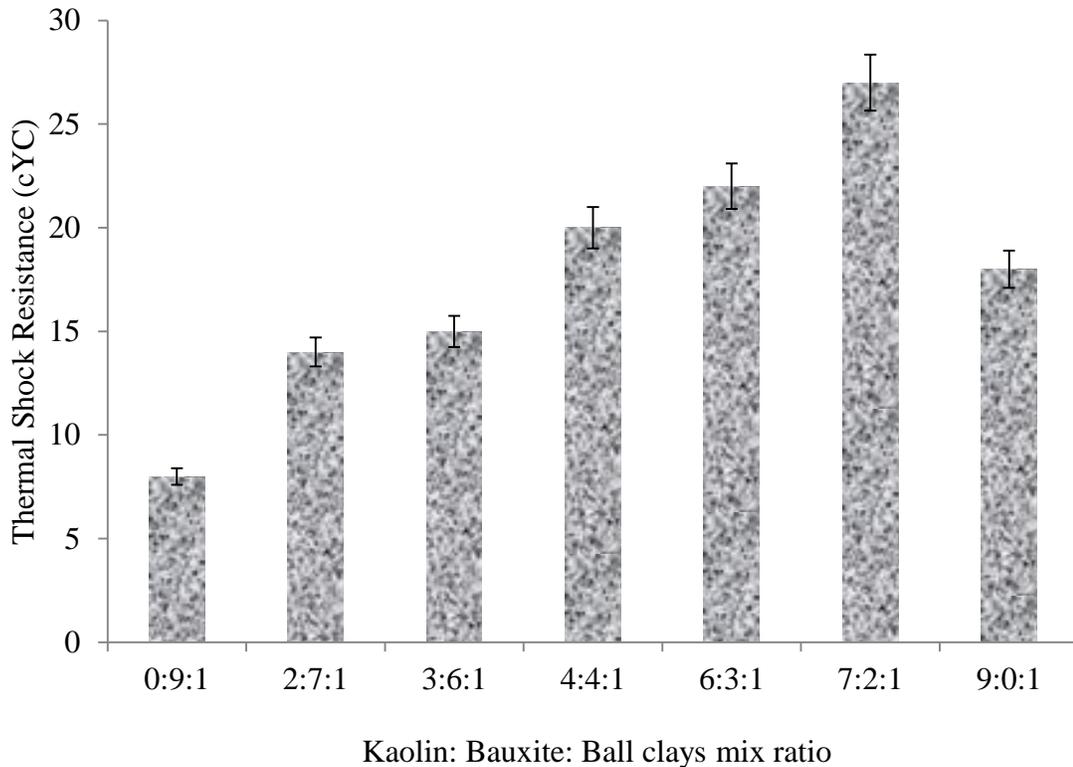


Figure 4.9: Effect of mix ratios on thermal shock resistance.

The thermal shock resistances for mix ratios of 4:4:1, 6:3:1, 7:2:1 and 9:0:1 exhibited similar results reported by Mazen (2009) and Ndaliman (2007) of 17 to 28 cycles and ASTM values of 20-30% reinforcing importance of kaolin in refractory brick making. The numbers of cycles are used to measure the longevity of refractory bricks which in turn would result to cost savings due to minimal replacement. The results obtained of 27 cycles for 7:2:1 mix ratio is recommendable for refractory brick production.

4.3 Effect of particle size on physical and thermal properties of developed bricks

Analysed data for particle size distribution on physical and thermal properties of composite refractory bricks with particle sizes of 150 μm , 380 μm and 600 μm for kaolin, bauxite and ball clay-binder mixed at a ratio of 7:2:1 evaluated against ASTM values at 5% level of significance are presented in Table 4.3. The output on mix ratio 7:2:1 the performance composite characteristics of composite refractory bricks mentioned in section 4.2 qualified for particle size

analysis. Particle size adapted are borrowed from previous researchers. Trends in the mean values for apparent porosity, bulk density, cold crushing strength, linear shrinkage and thermal shock resistance are indicated in Figures 4.10 through 4.14.

Table 4. 3: Physical and thermal properties for 7:2:1 mix ratio at compaction of 4.1N/m².

Particle size (μm)	AP (%)	BD (g/cm^3)	CCS (MN/m^2)	LFS (%)	TSR (cycles)
150	29 ^c	2.23 ^a	33 ^a	3.69 ^a	27 ^a
380	33 ^b	2.04 ^b	27 ^b	2.4 ^b	24 ^b
600	36 ^a	1.6 ^c	26.4 ^b	2.0 ^c	19 ^c
ASTM	20-30	1.71 -2.10	15	4 - 10	20-30
LSD	0.7956	0.0734	1.0744	0.1282	1.2579.

NB: The apparent porosity values followed by the same letter superscript (a,b or c) are not significantly different at $\alpha= 0.05$ and Least Significant Difference.

4.3.1 Physical properties for varying particle size

(a) Apparent porosity

Table 4.3 presents mean values of apparent porosity with variations in particle size for 7:2:1 mix ratio based on ASTM C20-00 (2015) values at 5% level of significance and least significance difference of 0.7956.

The mean values of apparent porosity increased with increase in particle sizes of the composite brick. For particle size of 150 μm , the value of apparent porosity was 29% and this value increased linearly as particle size increased to 36% at 600 μm as shown in Figure 4.10. All the apparent porosities are significantly different at $\alpha= 0.05$ and LSD of 0.7956 though values for particle sizes 380 μm and 600 μm fell outside the ASTM C20-00 (2015) range of 20 – 30%. This could be attributed to presence of voids in the raw materials.

Figure 4.10 shows the trend of apparent porosity with variations in particle size for 7:2:1 mix ratio based on ASTM values and specified level of significance and least significance difference.

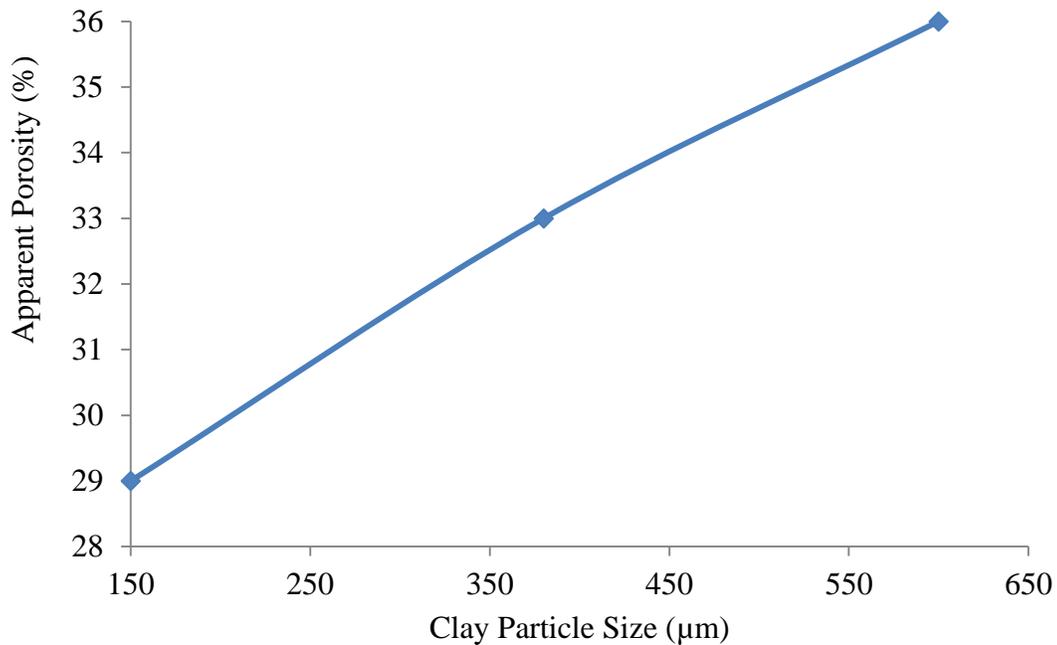


Figure 4.10: Effect of particle size on apparent porosity.

The result for particle size of 150µm met the ASTM C20-00 (2015) requirement of 20-30% porosity, but is higher than the findings of Aigbodion *et al.* (2010) who reported apparent porosity of values of 22-25% for medium heat with particle sizes of 150µm of ratio 7:2:1.

(b) Bulk density

The mean values of bulk density for 150µm, 380µm and 600µm were 2.23g/cm³, 2.04g/cm³ and 1.6kg/cm³, respectively Table 4.3. There was a reduction in the bulk density with increase in the particle size which might have been due to reduced pressure per unit area since constant pressure of 4.1N/m² was maintained during manufacturing process. The means of bulk density for various particle sizes were significantly different at $\alpha=0.05$ and LSD of 0.0734 suggesting that particle sizes has influence on the bulk density.

The bulk density trend presented in Figure 4.11 shows that as particle size increased from 150µm to 600µm, bulk density decreased from 2.23g/cm³ to 1.6g/cm³, respectively, which could be attributed to bigger particle size result in bigger void between the particles hence a lower density.

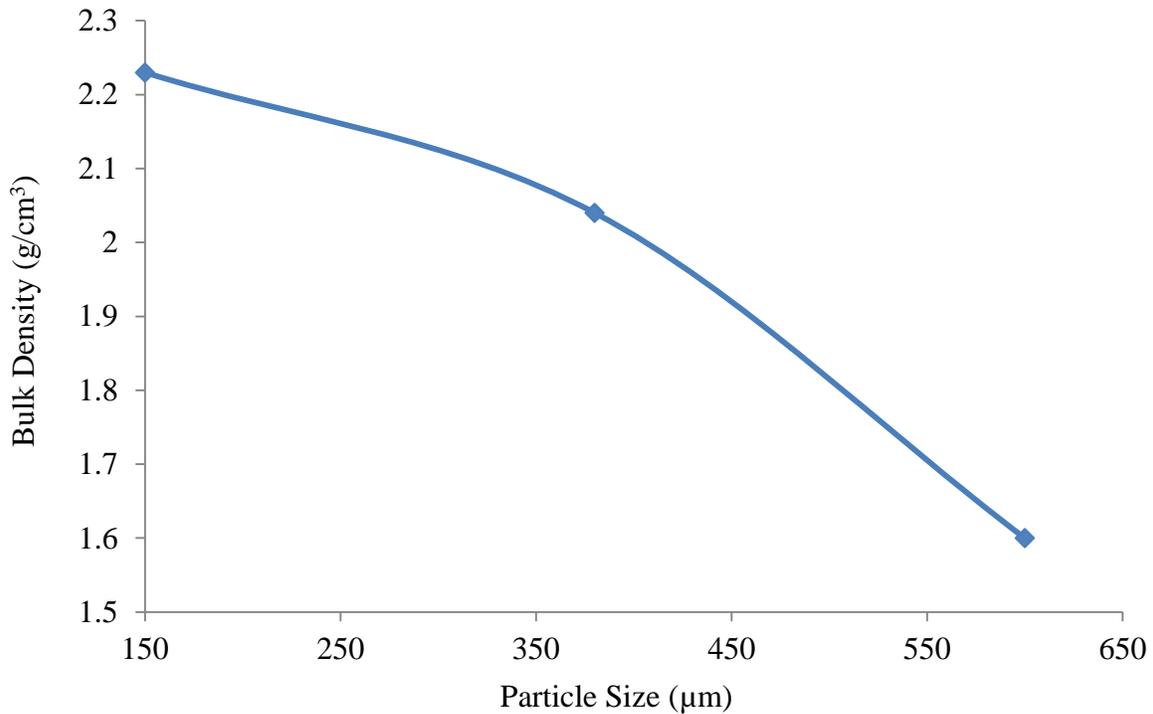


Figure 4.11: Effect of particle size on bulk density.

The result of bulk density for particle size 380µm (2.04 g/cm³) agrees with ASTM C20-00 (2015) values of 1.17 – 2.1g/cm³, but the results for the two particle sizes disagree with the findings of Mazen (2009) who reported average bulk densities of 1.96 and 1.98 g/cm³. The bulk density results obtained for particle sizes 150µm (2.23g/cm³) and 380µm (2.04g/cm³) are in close agreement with the findings of Hassan (2000) who results ranged between 1.90 and 2.30g/cm³ for the same particle sizes.

(c) Cold crushing strength

The cold crushing strength of a refractory brick is an indication of its suitability for use of refractories in construction. It was employed in this study to measure the ability of the composite refractory bricks for the strength of the grains and also of the bonding system.

Table 4.3 and Figure 4.12 shows particle size and the cold crushing strength of 150µm, 380µm and 600µm were 33, 27 and 26.4 MN/m², respectively. These values exceed 15 MN/m², the minimum specified value by ASTM C133-97(2015). It is evident from these results that, as particle size increased, cold crushing strength decreased which indicates that cold crushing

strength is inversely proportional to the increase in particle size as reported by as Aigbodion *et al.*, (2010).

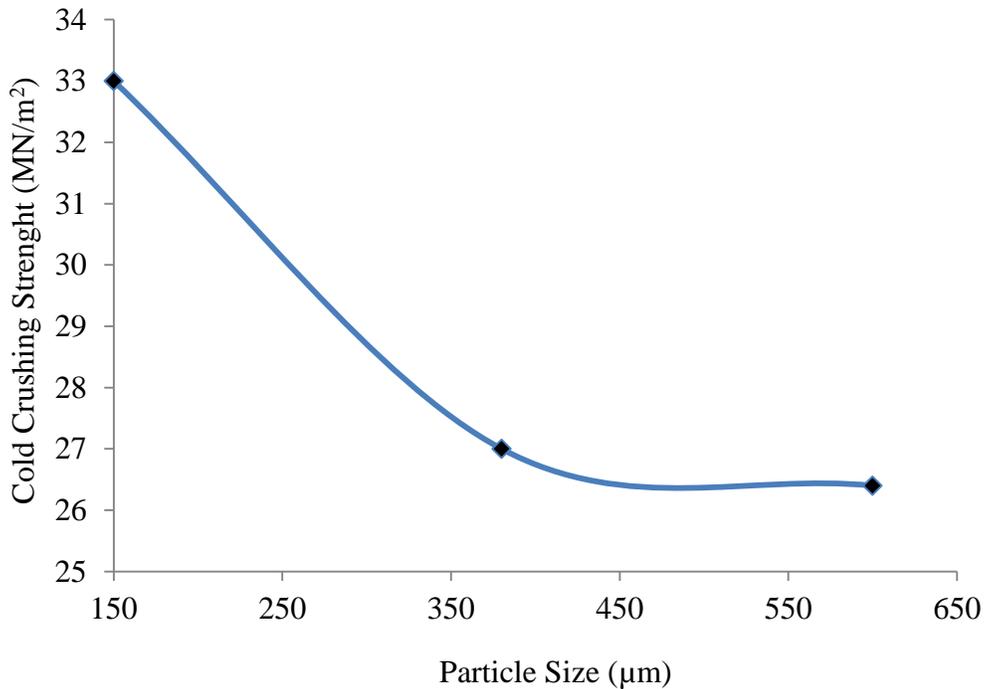


Figure 4.12: Effect of particle size on cold crushing strength.

The cold crushing strength values for particle sizes 380µm and 600µm were not significantly different at $\alpha= 0.05$ and least significant difference of 1.1. Results obtained compares with Aigbodion *et al.*, (2010) who reported, 32.36 MN/m² and 29.42 MN/m² at 290µm and 350µm respectively. These clays require adequate firing to ensure suitability for transportation of slags and fluxes.

4.3.2 Thermal properties for varying particle size

(a) Linear shrinkage

Linear shrinkage refers to change in linear dimensions occurring on the refractory brick after soaking in heat for a period of 24 hours (refer to Figure 3.2) and then cooling to room temperature. This is used to predict the limit of permissible shrinkage in service, i.e the degree of linear shrinkage to be tolerated by a refractory brick material when subjected to soaking heat.

The effect of different particle size are indicated in Table 4.3 and the trend of linear shrinkage with variation in particle size plotted in Figure 4.13.. It is evident from these results that linear shrinkage reduced with increase in particle size of a refractory material.

The mean value of linear shrinkage for 150 μm was 3.69% which reduced gradually to 2.0% at particle size of 600 μm at $\alpha= 0.05$ and LSD of 0.13. This reduction in linear shrinkage could be attributed to particle size and chemical composition of the composite clay, as some compounds are known to expand readily than others at the same temperature.

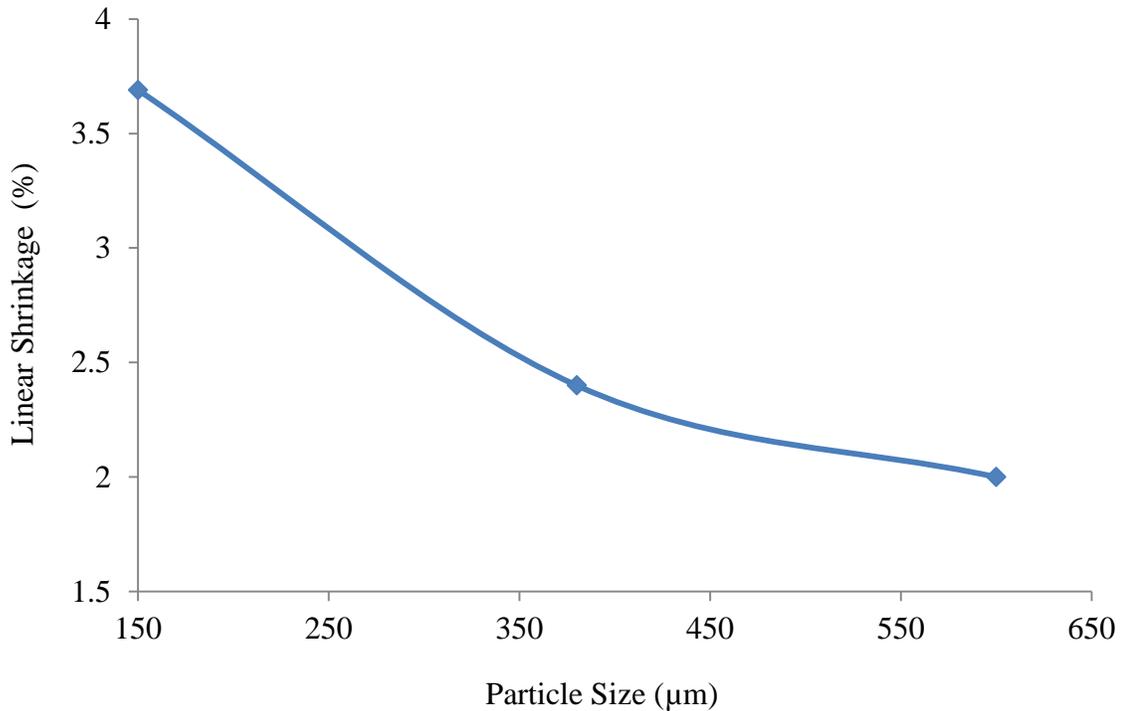


Figure 4.13: Effect of particle size on linear shrinkage.

Linear shrinkage values obtained from this study were slightly lower than the minimum ASTM value of 4% and Omowumi (2001) who reported a range of 4-10% for fireclays. The lower the values of linear shrinkage the better the performance as pointed by Abolarin *et al.* (2004). in their study implying that the brick would be less susceptible to volume change.

(b) Thermal shock resistance

Thermal shock resistance indicates the ability of a refractory brick to withstand the stress generated by sudden changes in temperature (heating and cooling cycles). The mean values of thermal shock resistance of the developed refractory bricks decreased with increase in particle size of the composite brick. In addition, all the three values of thermal shock resistance differed at $\alpha= 0.05$ and LSD of 1.3. At particle size of 150 μm the thermal shock was 27 cycles, but

reduced to 24 and 19 cycles when the particles size were increased to 380 μm and 600 μm , respectively.

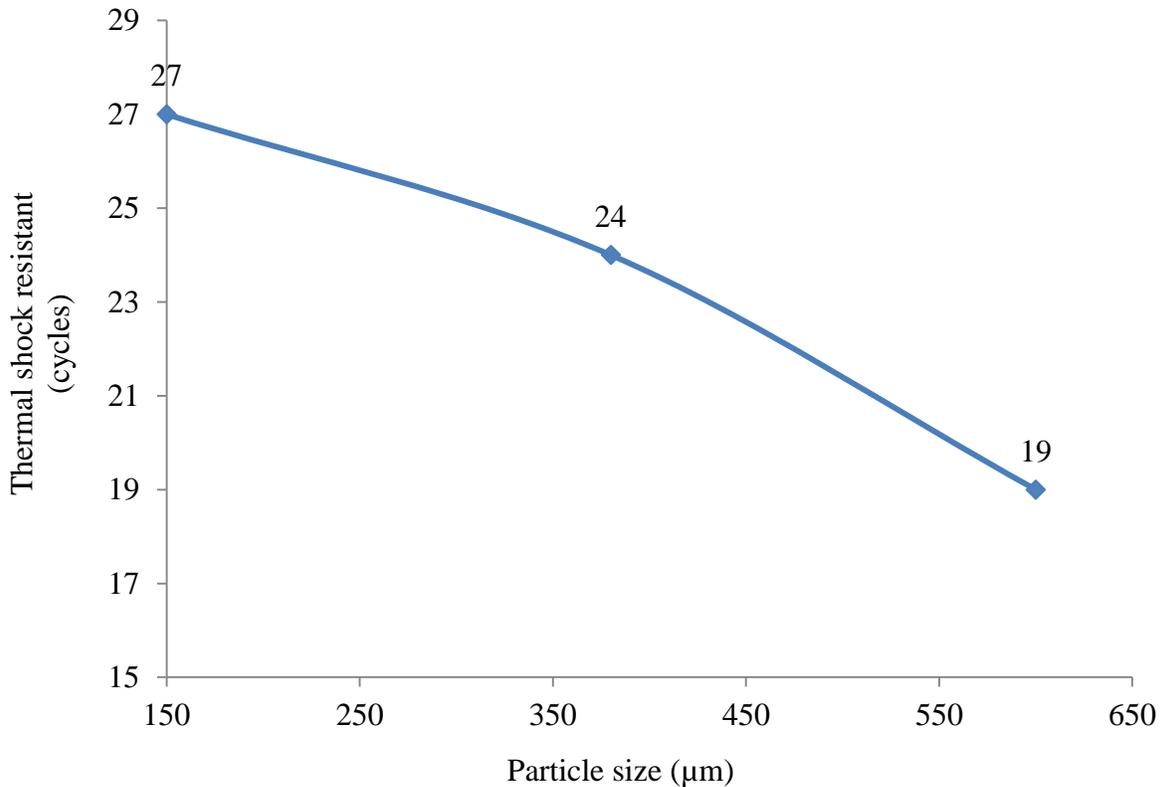


Figure 4.14: Effect of particle size on thermal shock resistance.

The thermal shock resistance values for particle sizes 150 μm and 380 μm were 27 and 24 cycles which fell within ASTM C1171 range of 20 – 30 cycles. The thermal shock resistance values of 19- 27 cycles were close to those of Mazen (2009) and Ndaliman (2007) who reported 17 – 28 cycles on average. This makes these bricks excellent for long time refractory use. The particle size of 150 μm gave a value of 27 cycles, found more appropriate for refractory brick making.

4.4 Optimum Kenyan refractory Brick and Properties.

The refractory brick made from mix ratio 7:2:1 gave the best values of apparent porosity (29) bulk density (2.23g/cm³), cold crushing strength (33), linear firing shrinkage (3.69) and thermal shock resistance (27) Table 4.2. This implies that mix ratio 7:2:1 can be adapted for brick manufacturing. Particle size of 150 μm produced best results compare to 380 and 600 μm Table 4.3, therefore recommended for brick manufacturing.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Alumina oxide, silica and iron oxide contents in kaolin, bauxite and ball clays made them suitable for use as composite refractory clays. This study has characterised kaolin, bauxite, salama, soapstone and ball clays and established their suitability as refractory materials. All the selected clays met the minimum ASTM requirements of Loss of Ignition (7-15%), silica (46-55%) and alumina (25-34%), except salama clay which exhibited lower alumina of 20%. Soapstone and salama clays exhibited high values of potassium (5.6%) and iron oxide (16%), respectively which are inferior, hence, disqualified as source of refractory material.

The refractory brick made from mix ratio 7:2:1 gave the best values of apparent porosity (29) bulk density (2.23g/cm^3), cold crushing strength (33), linear firing shrinkage (3.69) and thermal shock resistance (27). Apparent porosity and Linear shrinkage decreased with increase in kaoli. Bulk density, Cold crushing and Thermal shock resistance was directly proportional to increase in kaolin. All the mix ratios of kaolin and bauxite clays produced linear shrinkage values within the ASTM C133 range of 4-10% though 7:2:1 and 9:0:1 yielded the lower values implying it is better. Mix ratios of 4:4:1, 6:3:1 and 7:2:1 yielded thermal shock resistances above the minimum ASTM value of 15 cycles.

Particle sizes of $150\mu\text{m}$ at a mix ratio of 7:2:1 produced composite refractory bricks with the best performance suitable for commercial production. Apparent porosity increased with increase in particle size. Linear shrinkage, Thermal shock resistance, Bulk density, Cold crushing strength increased with decrease in clay particle size.

5.2 Recommendations

Besides encouraging investors to utilize local clays to manufacture refractory bricks and in so doing create employment, reduce importation and improve the balance of trade, further research can be conducted in the following areas:

- i. Thermal (Pyrometric Cone Equivalent) and physical properties of the composite refractory brick at a temperature of 1600°C .
- ii. Thermal conductivity tests for insulating refractories where the thermal gradients from the hot face to the cold face dictate the use of a refractory material for the specific uses.
- iii. Physical and thermal properties of composite brick from other local clay materials.

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APPENDICES

APPENDIX A: Plates for Research activities and experimental



Plate A.1: Materials drying at 250⁰C



Plate A.2: Raw material clay (12g) and binder (1.5g)

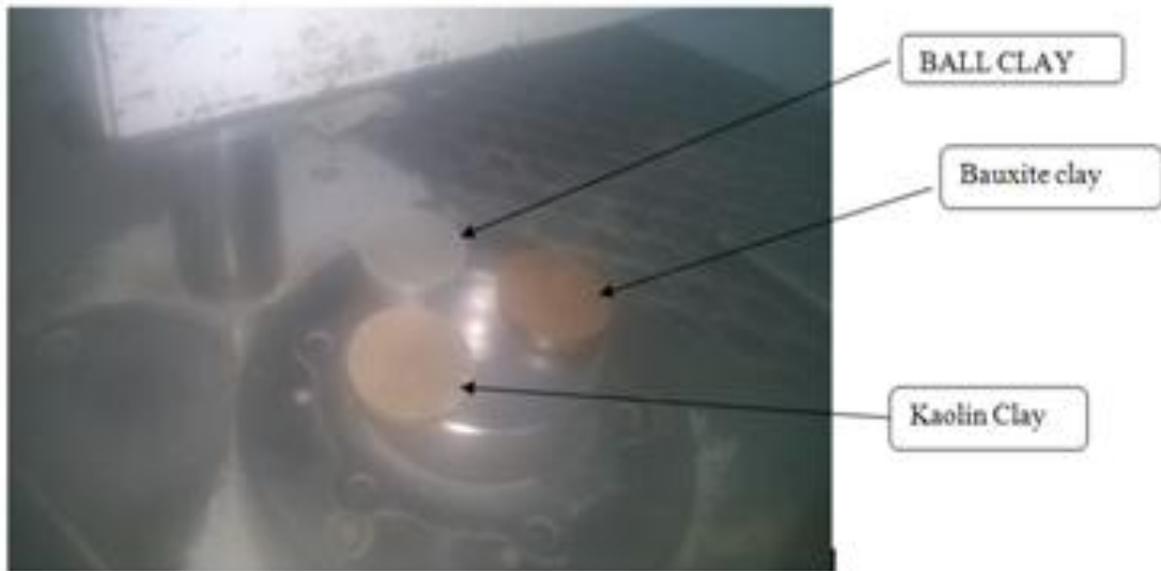


Plate A.3: Pellets

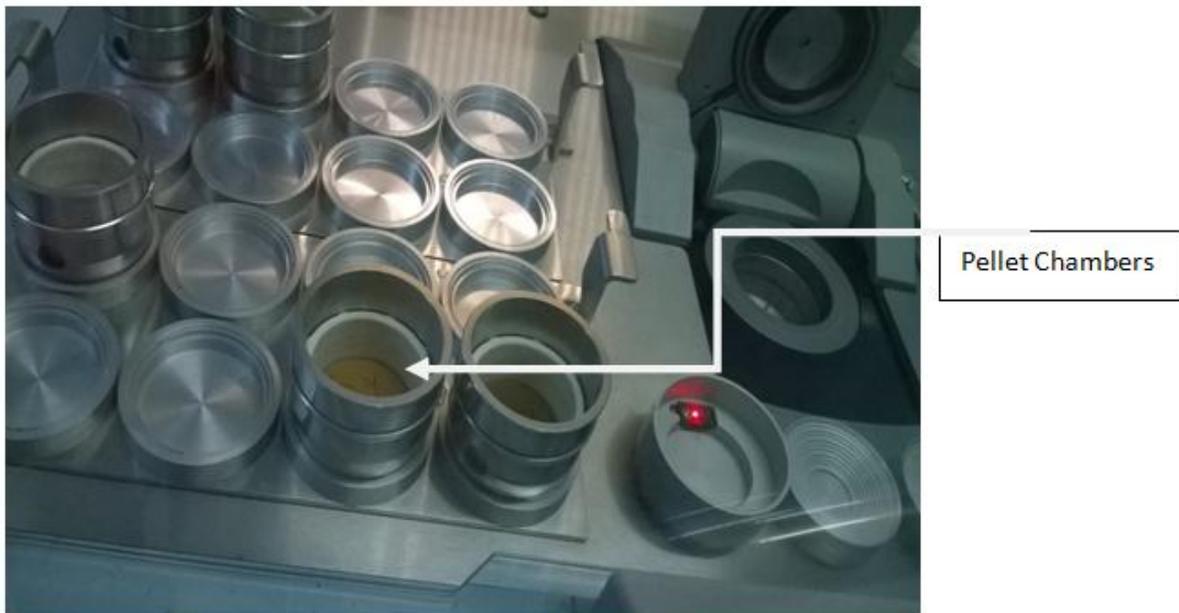


Plate A.4: X-ray pellet chambers



Plate A.5: Samples in desiccators



Plate A.6: Porcelain crucible with Samples



Plate A.7: Mix sample at varying ratios



Plate A.8: Cubical mould box of side 70 mm.



Plate A.9: Green Samples



Plate A.10: Oven Dry samples at 1100C



Plate A.11: Samples in automatic digital electric furnace



Plate A.12: Apparent porosity and bulk density testing equipment

APPENDIX B: Chemical analysis and statistical analysis systems

Table B.1 (a): Chemical analysis

CLAY SAMPLE	REPLICATION	AL ₂ O ₃	SiO ₂	CaO	MgO	TiO	Fe ₂ O ₃	Na ₂ O	K ₂ O	LOI
kaolin	1	30	56	0.14	0.2	1.1	0.6	0.1	0.1	7
kaolin	2	29	55	0.09	0.1	1.2	0.7	0.2	0.1	7
kaolin	3	30	55	0.08	0.3	1.1	0.8	0.2	0.05	8
kaolin	4	31	54	0.07	0.2	1.1	0.7	0.2	0.05	6
kaolin	5	30	55	0.12	0.2	1	0.7	0.3	0.2	7
Soapstone	1	34	46	0.2	0.1	1.1	0.4	1.2	5.6	8
Soapstone	2	33	46	0.2	0.05	1.2	0.3	1.4	5.7	9
Soapstone	3	34	45	0.1	0.05	1.1	0.3	1.3	5.5	8
Soapstone	4	35	47	0.3	0.2	1.1	0.2	1.3	5.6	8
Soapstone	5	34	46	0.2	0.1	1	0.3	1.3	5.6	7
Salama clay	1	20	49	0.4	0.1	1.4	16	0.3	1.2	14
Salama clay	2	19	50	0.3	0.1	1.4	16	0.4	1.2	14
Salama clay	3	21	50	0.3	0.1	1.5	17	0.5	1.3	15
Salama clay	4	20	51	0.2	0.1	1.4	15	0.4	1.2	14
Salama clay	5	20	50	0.3	0.1	1.3	16	0.4	1.1	13
bauxite	1	25	46	1.3	0.004	1.4	9.4	0.1	0.1	12
bauxite	2	25	46	1.2	0.005	1.3	9.5	0.1	0.1	12
bauxite	3	25	47	1.4	0.006	1.5	9.4	0.1	0.1	11
bauxite	4	26	45	1.3	0.005	1.4	9.3	0.1	0.1	13
bauxite	5	24	46	1.3	0.005	1.4	9.4	0.1	0.1	12
ball clay	1	32	47	0.1	0.2	2.4	3.4	0.4	1	15
ball clay	2	30	48	0.1	0.3	2.5	3.3	0.3	0.95	16
ball clay	3	31	46	0.1	0.3	2.6	3.2	0.3	0.9	14
ball clay	4	31	47	0.1	0.3	2.7	3.2	0.4	0.95	15
ball clay	5	31	47	0.1	0.4	2.5	3.2	0.4	1	15

Table B.1 (b): Loss On Ignition

Clay	Crucible (gm)	Raw sample(gm)	Dry sample(gm)	LOI %
Kaolin	7.7502	9.2213	9.1150	7.2
Soapstone	8.4775	9.4446	9.3628	8.4
Salama	8.8566	9.6627	9.5504	13.6
Bauxite	8.3722	9.4556	9.3257	12.0
Ball clay	8.4411	9.4447	9.2964	14.0

Table B-1(c): ANOVA t Test (LSD) output for chemical analysis

a) t Tests (LSD) for AL₂O₃

Alpha 0.05
 Error Degrees of Freedom 20
 Error Mean Square 0.5
 Critical Value of t 2.08596
 Least Significant Difference 0.9329

t Grouping	Mean	N	CLAYSAMPLE
A	34.0	5	soapstone
B	31.0	5	BALL
B	30.0	5	KAOLIN
C	25.0	5	BAUXITE
D	20.0	5	Salama

NB: AL₂O₃ Means with the same letter are not significantly different

b) t Tests (LSD) for SiO₂

Alpha 0.05
 Error Degrees of Freedom 20
 Error Mean Square 0.5
 Critical Value of t 2.08596
 Least Significant Difference 0.9329

t Grouping	Mean	N	CLAYSAMPLE
A	55.0	5	KAOLIN
B	50.0	5	Salama
C	47.0	5	BALL
C	46.0	5	BAUXITE
C	46.0	5	soapstone

NB: SiO₂ Means with the same letter are not significantly different

c) t Tests (LSD) for CaO

Alpha 0.05
 Error Degrees of Freedom 20
 Error Mean Square 0.00317
 Critical Value of t 2.08596

Least Significant Difference 0.0743

t Grouping	Mean	N	CLAYSAMPLE
A	1.3	5	BAUXITE
B	0.3	5	Salama
C	0.2	5	Kisii
D	0.1	5	KAOLIN
D			
D	0.1	5	BALL

NB: CaO Means with the same letter are not significantly different

d) t Tests (LSD) for MgO

Alpha 0.05
 Error Degrees of Freedom 20
 Error Mean Square 0.002456
 Critical Value of t 2.08596
 Least Significant Difference 0.0654

t Grouping	Mean	N	CLAYSAMPLE
A	0.3	5	BALL
B	0.2	5	KAOLIN
C	0.12	5	Kisii
C			
C	0.10	5	Salama
C			
C	0.05	5	BAUXITE

NB: MgO Means with the same letter are not significantly different.

e) t Tests (LSD) for TiO

Alpha 0.05
 Error Degrees of Freedom 20
 Error Mean Square 0.005
 Critical Value of t 2.08596
 Least Significant Difference 0.0933

t Grouping	Mean	N	CLAYSAMPLE
A	2.5	5	BALL
B	1.4	5	BAUXITE
B	1.4	5	Salama
C	1.1	5	Kisii
C	1.1	5	KAOLIN

NB: *TiO* Means with the same letter are not significantly different.

f) t Tests (LSD) for Fe₂O₃

Alpha 0.05
 Error Degrees of Freedom 20
 Error Mean Square 0.104
 Critical Value of t 2.08596
 Least Significant Difference 0.4255

t Grouping	Mean	N	CLAYSAMPLE
A	16.0	5	Salama
B	9.4	5	BAUXITE
C	3.3	5	BALL
D	0.7	5	KAOLIN
D	0.3	5	Kisii

NB: *Fe₂O₃* Means with the same letter are not significantly different.

g) t Tests (LSD) for Na₂O

Alpha 0.05
 Error Degrees of Freedom 20
 Error Mean Square 0.004
 Critical Value of t 2.08596
 Least Significant Difference 0.0834

t Grouping	Mean	N	CLAYSAMPLE
A	1.3	5	Kisii
B	0.4	5	BALL
B			
B	0.4	5	Salama
B			
B	0.2	5	KAOLIN
B			
B	0.1	5	BAUXITE

NB: Na_2O Means with the same letter are not significantly different.

h) t Tests (LSD) for K_2O

Alpha 0.05
 Error Degrees of Freedom 20
 Error Mean Square 0.00275
 Critical Value of t 2.08596
 Least Significant Difference 0.0692

t Grouping	Mean	N	CLAYSAMPLE
A	5.6	5	Kisii
B	1.2	5	Salama
B	1.0	5	BALL
C	0.1	5	BAUXITE
C			
C	0.1	5	KAOLIN

NB: K_2O Means with the same letter are not significantly different.

i) t Tests (LSD) for LOI

Alpha 0.05
 Error Degrees of Freedom 20
 Error Mean Square 0.5
 Critical Value of t 2.08596
 Least Significant Difference 0.9329

t Grouping	Mean	N	CLAYSAMPLE
A	15.0	5	BALL
B	14.0	5	Salama
C	12.0	5	BAUXITE
D	8.0	5	Kisii
E	7.0	5	KAOLIN

NB: LOI Means with the same letter are not significantly different

APPENDIX C: Physical and thermal properties and statistical analysis systems

Table C-1 (a): Effects of mix ratios on physical and thermal properties of refractory bricks.

REPLICATION	MIX RATIOS	Apparent Porosity	Bulk Density	Cold strength	Linear Shrinkage	Thermal Sshock
1	9:0	27	1.6	26	2.83	17
2	9:0	28	1.6	26	2.8	18
3	9:0	26	1.6	27	2.79	18
4	9:0	28	1.62	28	2.8	19
5	9:0	26	1.63	28	2.83	18
1	0:9	40	1.7	18	9.25	6
2	0:9	44	1.6	16	9.95	9
3	0:9	38	1.6	17	9.75	9
4	0:9	36	1.4	15	9.55	8
5	0:9	42	1.5	14	9.85	8
1	2:7	38.5	1.5	22.5	9	15
2	2:7	38	1.6	22	9	13
3	2:7	37.5	1.7	22	9	14
4	2:7	38	1.4	22	8.5	14
5	2:7	38	1.6	21.5	9	14
1	1:2	37	1.6	24	7.5	14
2	1:2	36	1.6	25	7	16
3	1:2	35	1.5	24	7	15
4	1:2	36	1.5	23	7.5	15
5	1:2	36	1.5	24	7	15
1	1:1	34.5	1.6	27	6.5	22
2	1:1	35.5	1.7	27	6.3	20
3	1:1	35	1.6	26.8	6.4	18
4	1:1	35	1.6	26.8	6.3	21
5	1:1	35	1.6	27.4	6.5	19
1	2:1	32.75	1.9	29	4.5	22
2	2:1	33	2.1	30	4.5	23
3	2:1	33.25	1.9	30	4.3	22
4	2:1	33	2.1	31	4.4	22
5	2:1	33	2.1	30	4.5	21
1	7:2	29	2.23	33.25	3.7	28
2	7:2	28	2.22	33	3.7	27
3	7:2	30	2.25	32.75	3.85	27
4	7:2	29	2.23	33	3.6	27
5	7:2	29	2.25	33	3.6	26

Table C-1 (b): ANOVA output for Physical and Thermal properties for developed composite refractory bricks on mix ratios

a. Dependent Variable: Apparent Porosity

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	6	660.0	110.00	62.70	<.0001
Error	28	49.125	1.75		
Corrected Total	34	709.125			

R-Square	Coeff Var	Root MSE	Apparent Porosity Mean
0.930724	3.895770	1.324562	34.00000

Source	DF	Anova SS	Mean Square	F Value	Pr > F
MIXRATIOS	6	660.0	110.0	62.70	<.0001

b. Dependent Variable: Bulk Density

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	6	2.30	0.38	61.90	<.0001
Error	28	0.17	0.006		
Corrected Total	34	2.47			

R-Square	Coeff Var	Root MSE	BD Mean
0.929899	4.536914	0.078722	1.735143

Source	DF	Anova SS	Mean Square	F Value	Pr > F
MIXRATIOS	6	2.30	0.38	61.90	<.0001

c. Dependent Variable: Cold Crushing Strength

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	6	928.57	154.76	229.70	<.0001
Error	28	18.86	0.67		
Corrected Total	34	947.4364286			

R-Square Coeff Var Root MSE CCS Mean
0.980088 3.209921 0.820823 25.57143

d. Dependent Variable: Linear Fired Shrinkage

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	6	206.24	34.37	1042.31	<.0001
Error	28	0.923	0.032		
Corrected Total	34	207.166			

R-Square Coeff Var Root MSE LFS Mean
0.995543 2.948736 0.181600 6.158571

e. Dependent Variable: Thermal shock resistance

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	6	1127.142	187.85	202.31	<.0001
Error	28	26.00	0.93		
Corrected Total	34	1153.14			

R-Square Coeff Var Root MSE TSR Mean
0.977453 5.439814 0.963624 17.71429

Source	DF	Anova SS	Mean Square	F Value	Pr > F
MIXRATIOS	6	1127.14	187.85	202.31	<.0001

Table C-1 (c): t Test (LSD) for physical and thermal properties for develop composite refractory bricks on mix ratios

a. t Tests (LSD) for Apparent Porosity

Alpha 0.05
 Error Degrees of Freedom 28
 Error Mean Square 1.754464
 Critical Value of t 2.04841
 Least Significant Difference 1.716

t Grouping	Mean	N	MIXRATIOS
A	43.0	5	0:9
B	38.0	5	2:7
C	36.0	5	1:2
C	35.0	5	1:1
D	33.0	5	2:1
E	29.0	5	7:2
F	27.0	5	9:0

NB: Apparent Porosity Means with the same letter are not significantly different

b. t Tests (LSD) for Bulk Density

Alpha 0.05
 Error Degrees of Freedom 28
 Error Mean Square 0.006197
 Critical Value of t 2.04841
 Least Significant Difference 0.102

t Grouping	Mean	N	MIXRATIOS
A	2.23	5	7:2
B	2.02	5	2:1
C	1.62	5	1:1
C	1.61	5	9:0
C	1.56	5	0:9
C	1.56	5	2:7
C	1.54	5	1:2

NB: Means with the same letter are not significantly different

c. t Tests (LSD) for Cold Crushing Strength

Alpha 0.05
 Error Degrees of Freedom 28
 Error Mean Square 0.67375
 Critical Value of t 2.04841
 Least Significant Difference 1.0634

Grouping	Mean	N	MIXRATIOS
A	33.0	5	7:2
B	30.0	5	2: 1
C	27.0	5	9:0
C	27.0	5	1:1
D	24.0	5	1:2
E	22.0	5	2:7
F	17.0	5	0:9

NB: Means with the same letter are not significantly different

d. t Tests (LSD) for Linear Fired Shrinkage

Alpha 0.05
 Error Degrees of Freedom 28

Error Mean Square 0.032979
 Critical Value of t 2.04841
 Least Significant Difference 0.2353

t Grouping	Mean	N	MIXRATIOS
A	9.67	5	0:9
B	8.90	5	2:7
C	7.20	5	1:2
D	6.40	5	1:1
E	4.44	5	2:1
E	3.69	5	7:2
F	2.81	5	9:0

NB: Linear Fired Shrinkage Means with the same letter are not significantly different

e. t Tests (LSD) for Thermal Shock Resistance

Alpha 0.05
 Error Degrees of Freedom 28
 Error Mean Square 0.928571
 Critical Value of t 2.04841
 Least Significant Difference 1.2484

t Grouping	Mean	N	MIXRATIOS
A	27.0	5	7:2
B	22.0	5	2:1
C	20.0	5	1:1
D	18.0	5	9:0
E	15.0	5	1:2
E	14.0	5	2:7
F	8.0	5	0:9

NB: Means with the same letter are not significantly different

APPENDIX D: Physical and thermal properties for particle sizes

Table D-1(a): Physical and Thermal Properties of the developed composite refractory brick

REPLICATION	Particle size(μm)	Apparent porosity(%)	Bulk density	Cold crushing	Linear shrinkage	thermal shock
1	150	28.5	2.24	32.75	3.68	26
2	150	29.5	2.23	33	3.71	27
3	150	29	2.24	33	3.73	27
4	150	29	2.23	33.15	3.68	28
5	150	29	2.23	33.1	3.65	27
1	380	33	2	28	2.3	22
2	380	33	2.1	27	2.5	24
3	380	34	2	27	2.4	24
4	380	32.5	2.1	27	2.4	25
5	380	32.5	1.98	26	2.4	25
1	600	37	1.6	26	2	19
2	600	36	1.7	25	1.8	20
3	600	36	1.6	28	2.2	19
4	600	36	1.6	27	2	18
5	600	35	1.5	26	2	19

Table D-1 (b): ANOVA output for physical and thermal properties of the developed composite refractory bricks

a) Dependent Variable: apparent porosity

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	2	123.33	61.6	185.00	<.0001
Error	12	4.0	0.33		
corrected Total	14	127.33			

R-Square Coeff Var Root MSE apparent porosity Mean
 0.968586 1.767399 0.577350 32.66667

b) Dependent Variable: Bulk density

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	2	1.052	0.52	185.45	<.0001
Error	12	0.034	0.003		
Corrected Total	14	1.086			

R-Square Coeff Var Root MSE Bulk density Mean
 0.968659 2.721995 0.053260 1.956667

c) Dependent Variable: cold crushing strength

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	2	133.20	66.60	109.55	<.0001
Error	12	7.295	0.607		
Corrected Total	14	140.495			

R-Square Coeff Var Root MSE cold crushing Mean
 0.948076 2.707257 0.779690 28.80000

d) Dependent Variable: linear shrinkage

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	2	7.80	3.90	450.89	<.0001
Error	12	0.1038	0.0086		
Corrected Total	14	7.904			

b) t Tests (LSD) for Bulk density

Alpha 0.05
 Error Degrees of Freedom 12
 Error Mean Square 0.002837
 Critical Value of t 2.17881
 Least Significant Difference 0.0734

t Grouping	Mean	N	sieve size(μm)
A	2.234	5	150
B	2.036	5	380
C	1.6	5	600

NB: Bulk density Means with the same letter are not significantly different

c) t Tests (LSD) for Cold Crushing Strength

Alpha 0.05
 Error Degrees of Freedom 12
 Error Mean Square 0.607917
 Critical Value of t 2.17881
 Least Significant Difference 1.0744

t Grouping	Mean	N	sieve size(μm)
A	33.0	5	150
B	27.0	5	380
B	26.4	5	600

NB: Cold Crushing Strength Means with the same letter are not significantly different

d) t Tests (LSD) for linear shrinkage

Alpha 0.05
 Error Degrees of Freedom 12
 Error Mean Square 0.00865
 Critical Value of t 2.17881
 Least Significant Difference 0.1282

t Grouping	Mean	N	sieve size(μm)
A	3.69	5	150
B	2.4	5	380
C	2.0	5	600

NB: linear shrinkage Means with the same letter are not significantly different

e) t Tests (LSD) for thermal shock resistance

Alpha 0.05
Error Degrees of Freedom 12
Error Mean Square 0.833333
Critical Value of t 2.17881
Least Significant Difference 1.2579

t Grouping	Mean	N	sieve size(μm)
A	27.0	5	150
B	24.0	5	380
C	19.0	5	600

NB: Thermal shock resistance Means with the same letter are not significantly different