

**MECHANICAL PROPERTIES OF CERAMIC FLOOR
TILES MADE FROM SELECTED MINERALS IN
UGANDA**

**BY
OCHEN WILLIAM
2009/HD/160/MScP**

**A DISSERTATION SUBMITTED TO GRADUATE SCHOOL IN PARTIAL
FULFILMENT OF THE REQUIREMENTS OF THE AWARD OF
THE DEGREE OF MASTER OF SCIENCE IN PHYSICS OF
KYAMBOGO UNIVERSITY**

JUNE 2012

DECLARATION

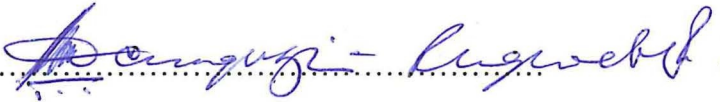
I **OCHEN WILLIAM** declare that the work presented in this study is original and has never been presented by any person or submitted to any University for an academic award.

Signed.....

Date.....17/12/2012


APPROVAL

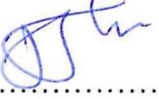
This is to certify that the study by OCHEN WILLIAM was carried out under our supervision and its approved for submission to the Board of Graduate school and Senate of Kyambogo University.

Signed: 

SUPERVISOR ONE: Dr MUCUNGUZI Eric (Assoc. Professor)

Physics Department, Kyambogo University

Date: 

Signed: 

SUPERVISOR TWO: Dr. OLUPOT Peter Wilberforce

Department of Mechanical Engineering, Makerere University

Date: 

DEDICATION

This study is dedicated to the Almighty God who gave me wisdom and ability.

ACKNOWLEDGEMENT

I wish to express special thanks to my father Mr. Apunyo Juventine for the love and financial input he offered during my elementary, secondary and tertiary education. My mother madam Asio Florence for the care and advice she gave me during my days in school.

My heart felt gratitude goes to my supervisors, Assoc. Prof. Eric Mucunguzi of Kyambogo University Faculty of Science and Dr Peter Wilberforce Olupot of Makerere University faculty of Technology for the guidance throughout this research. The time spent reading and thinking through, their constant encouragement are attributes I will forever appreciate.

I extend my gratitude to Assoc. Prof. Obwoya Sam Kinyera of Kyambogo University and Mr Ben Enjiku (H.O.D) Physics KyU for the valuable time spent reading through the dissertation and giving advice where necessary. Your advice was taken seriously that is why this work was a success.

My sincere thanks goes to the Department of Ceramics, Uganda Industrial Research Institute (UIRI) for their cooperation especially Robert Twesigye, for firing the samples. Not forgetting Dicky and Paul for the technical advice and brotherly help they offered. It is also my pleasure to thank the management of Uganda National Bureau of Standards (UNBS) for giving me permission to use their Laboratories, most especially Eng. Okumu John for his guidance during testing and formulation of samples. I would also like to thank the Management of Makerere University most especially the Head of Physics Department Dr. Florence D'ujang and the Laboratory attendant Mr Okiror Micheal for

giving me permission to use their moulds and sieves. Not forgetting Mr Kasala Moses of Department of Geology and Petroleum for carrying out the chemical analysis of the minerals. Mr Muluga Paul of Ministry of Works Laboratory Kireka for his advice during the strength tests on the samples.

Special thanks to my fiancée Cathy and my son Einstein , no words can describe my love for you both. My appreciation also goes to classmates Mr. Abiriga Faustin, Mr. Okullo Michael, Mr. Byaruhanga Bonaventure, Ms Talibawo Joan, Ms Nasejje Stella and Ms Mbambu Joyce. Grandparents Mzee Ocen Willy, Mrs Sanyu Ocen and Akello Mary (R.I.P). My siblings Emma, Eve, Lydia, Suzan, Winnie and Pius have been a great inspiration. I hope this achievement on my part serves as an inspiration to them too. Employer Mr. Mike Kironde, friends Frony Odeke and Eric Ssebunya have been a great support to me. Finally with pleasure and gratitude, I extend my sincere thanks to all those that helped me in all the activities leading to this dissertation, both in their individual and institutional capacities due to limited space it was not possible to mention them all.

TABLE OF CONTENTS

Declaration.....	i
Approval.....	ii
Dedication.....	iii
Acknowledgment.....	iiv
Table of Content.....	vi
List of Figures.....	x
List of Tables	xi
List of abbreviations	xiii
Abstract.....	xiv
 CHAPTER ONE: INTRODUCTION	
1.1 Background of the Study	1
1.2 Statement of the Problem.....	5
1.3 Purpose of the Study	6
1.4 Objective of the Study	6
1.5 Significance of the Study	6
1.6 Scope of the Study	7
 CHAPTERTWO: REVIEW OF RELATED LITERATURE	
2.1 Introduction.....	8
2.1.1 Clay.....	8
2.1.2 Kaolinitic Minerals	10

2.1.3 Chemical Composition of Raw Materials.....	14
2.1.4 Mineral Composition in Ceramic Tiles.....	15
2.1.5 Bonding.....	16
2.1.6 Particle Size, Shape and Distribution.....	17
2.1.7 Forming.....	20
2.1.8 Firing.....	21
2.1.9 Effect of Firing on Porosity	24
2.1.10 Effect of Firing on Strength	25
2.2.1 Porosity	27
2.2.2 Water Absorption.....	30
2.3.1 Modulus of Rupture and Water Absorption Rating.....	39
2.3.2 Tiles.....	41
CHAPTER THREE: METHODOLOGY	
3.1 Research Design.....	43
3.2 Preparation of Raw Materials	45
3.2.1 Ball Clay	45
3.2.2 Kaolin.....	45
3.2.3 Feldspar.....	46
3.2.4 Sand.....	46
3.3 Composition Ratios of Raw Materials in the Samples	46
3.4 Chemical Analysis of Minerals.....	47

3.5 Mixing of Raw Materials	48
3.6 Production of Sample Tiles.....	49
3.6.1 Forming.....	50
3.6.2 Firing.....	52
3.7 Experimental Measurements.....	53
3.7.1 Linear Shrinkage.....	53
3.7.2 Measurement of Modulus Of Rupture (MOR)	54
3.7.3 Measurement of Water Absorption.....	55
CHAPTER FOUR: RESULTS AND DISCUSSIONS	
4.1 Introduction.....	56
4.2 Results of Chemical Composition of Minerals.....	56
4.3 Discussion of Chemical Composition of Minerals	57
4.4 Results of Shrinkage	58
4.5 Discussion of Shrinkage	59
4.6 Results on Appearance of Fired Samples	61
4.6.1 Effects of Firing on Shape	62
4.6.2 Effect of Firing on Surface Finish	63
4.6.3 Effect of Firing on Color.....	63
4.7 Statistical Analysis.....	64
4.8 Results of Modulus of Rupture (MOR)	66
4.9 Discussion of Modulus Of Rupture (MOR).....	68

4.10 Comparison of MOR with South African National Standards	73
4.11 Statistical Analysis of Strength.....	75
4.12 Results of Water Absorption.....	75
4.13 Discussion on Water Absorption	77
4.14 Comparison of Water Absorption with SANS	78
4.15 Statistical Analysis of Water Absorption.....	79
CHAPTER FIVE: CONCLUSIONS AND RECOMMENDATIONS	
5.1 Conclusion	81
5.2 Recommendations.....	83
REFERENCES.....	85
APPENDIX A	<u>90</u>
APPENDIX B	95

LIST OF FIGURES

PAGE

Figure 2.1: Effect of particle size variation on densification.....	19
Figure 2.2: Bending strength of floor tiles as a function of temperature.....	26
Figure 2.3: Schematic Diagram of the Three Point Loading test	34
Figure 3.1: Experimental layout.....	44
Figure 3.2: XRF machine.....	47
Figure 3.3: Picture of the Automatic Mixer.....	49
Figure 3.4: Picture of a Green Body (tile) after Ejection from the Mould.....	50
Figure 3.5: Picture of the Forming Process using a Compact Compression Machine.....	51
Figure 3.6: Picture of a Body on a Jigger Table ready for Leveling.....	51
Figure 3.7: Picture of dry Samples in an Automatic Electronic Kiln ready for Firing.....	52
Figure 3.8: Picture Showing Strength Test using a Three Point Loading Machine.....	55
Figure 4.1: Linear Shrinkage (%) of the Studied Tiles.....	57
Figure 4.2 Samples fired at different temperatures.....	60
Figure 4.3: Warped Samples (right) and Unwrapped Samples (left).....	61
Figure 4.4: MOR of the Sample Tiles at different Temperature.....	66
Figure 4.5: Water Absorption of the Sample Tiles.....	75

LIST OF TABLES

Table 2.1: Chemical composition of minerals from different deposits in Uganda.....	14
Table 2.2: Relationship between Particle sizes and Strength of ceramic ware.....	18
Table 2.3: Physical requirements for dust-pressed (group B) tiles (source SASN, 2008).....	40
Table 2.4: Physical requirements for extruded (group A) tiles.....	40
Table 3.1: Composition of Formulated Tile Samples by Weight	47
Table 4.1: Chemical composition of raw materials.....	56
Table 4.2: ANOVA Table	65
Table 4.3: ANOVA Table for analysis of shrinkage.....	66
Table 4.4: Comparison of strength of formulated samples with SANS.....	74
Table 4.5: ANOVA for strength.....	75
Table 4.6: Comparison of water absorption with SANS.....	79
Table 4.7: ANOVA of water absorption.....	80

LIST OF ABBREVIATIONS

UNBS	Uganda National Bureau of Standards
UIRI	Uganda Industrial Research Institute
H.O.D	Head of Department
KyU	Kyambogo University
R.I.P	Rest In Peace
B.C	Before Christ
www	World Wide Web
http	hyper text transfer protocol
UCL	Uganda Clays Limited
SANS	South African National Standards
ISO	International Organization for Standardization
BGS	British Geological Survey
MOR	Modulus of Rupture
W.A	Water Absorption
wt	weight
SEM	Scanning Electron Microscope
XRF	X-Ray Florescence
XRD	X-Ray Diffraction
INAA	Instrumental Neutron Activation Analysis

MPa	Mega Pascals
kN	Kilo Newton
ANOVA	Analysis Of VArience
SS _E	Sum Square of Error
SS _{Temp}	Sum Square of Temperature
SS _T	Sum Square of Total observations
L.S-b	Linear Shrinkage of breadth
L.S-L	Linear Shrinkage of Length
L.S-t	Linear Shrinkage of thickness
Av. L.S	Average Linear Shrinkage
P.r	Proving ring reading

ABSTRACT

This study was designed to produce porcelain floor tiles using raw materials found in Uganda and to determine mechanical properties of the tiles produced. The properties were then compared with South African National Standards. Porcelains are made of clays, kaolin, feldspar and sand. In this study, the raw materials were mixed in proportions of 40-60% clays, 30-40% feldspar and 10-30% sand. The tests carried out include shrinkage, strength, water absorption and chemical analysis.

Samples were pressed at 30MPa with 12% moisture content by weight. Firing was at peak temperatures of 1050, 1100, 1150, 1200 and 1250⁰C. Firing process involved heating dry samples from room temperature to 105⁰C at 100⁰C/hr, after 2 hrs of holding the rate was then raised to 360⁰C/hr and soaked for 1 hr at peak temperatures. Samples were left to cool naturally in the kiln for 16 hrs.

Chemical analysis of the minerals by XRF method revealed that kaolin had an alumina content of 24.9 (wt %) whereas sand had silica at 95.0 (wt %). Iron oxide which influences the color of samples was high in ball clay at 3.2 (wt %).

Maximum strength of 34MPa was exhibited by composition 2 at 1250⁰C with linear shrinkage of 9.1% and water absorption of 0.0 (wt %). South African National Standards classifies tiles whose strength is above 30MPa as B₁. All samples fired in the temperature range of 1100 to 1250⁰C exhibited properties in the range recommended by SANS both in strength and water absorption for flooring as B₁ to B₄ tiles.

Minerals from the studied deposits yielded high grade ceramic floor tiles recommended by SANS for flooring. No cracks were exhibited by samples fired at 1100 to 1250⁰C

CHAPTER ONE: INTRODUCTION

1.1 Background of the Study

Ceramic products are made by action of heat on molded earthy minerals. Keramos is a Greek word from which the term ceramics was derived. Keramos literally means “burned stuff” when referring to products obtained through action of fire upon materials (Olupot, 2010). Ceramic materials are compounds of alumina and silica bonded by strong ionic and covalent bonds hence making them strong and useful in nature (Grimshaw, 1971). The metallic and non metallic elements in ceramics are compounds of aluminum and oxygen, calcium and oxygen, silicon and nitrogen. The type of atoms and bonding primarily affects the chemical, mechanical, electrical, magnetic and optical properties of ceramics

The use of ceramics ranks as one of man’s earliest scientific achievements though the exact date can never be known with certainty. Around 2600 B.C, the Egyptians built the step pyramids with colorful glazed ceramic tiles (<http://www.enotes.com>). Besides tiles ceramics are used for making sanitary and kitchen ware. In the refractory industry, ceramics are used to make fire bricks and insulators. This is because of their durability at high temperature (Raymond, 1994). Materials made from ceramics have high electrical resistance that is why they are employed as resistors (Grimshaw, 1971). As a result of technological advancement, modern applications of ceramics include magnetic memory systems (ferrite ceramics) and optical communication devices among others.

A tile is any hard-piece of material with a flat surface. Tiles can be made of wood, stones, ceramics and plastic. Floor tiles are commonly made of ceramics and are categorized as

porcelain stoneware, porcelain earthenware and quarry tiles. Porcelain stoneware tiles are dense, impermeable and of high strength unlike earthenware and quarry tiles. The sintering temperature of earthenware tiles is about 900°C. However, porcelain stoneware tiles are sintered at peak temperatures of 1100°C to 1300°C for a soaking time of 40 minutes depending on the fluxing content (Tucci and Esposito. et. al. 2007). Stoneware porcelain tiles are strong, hard and wear-resistant this is because of the phase transitions of the ingredients during firing. Ceramic stoneware tiles have good appearance, they are easy to clean. They are also resistant to corrosion and have a high strength.

According to Sanchez and Garcia (2010), both porcelain stoneware and porcelain earthenware contain similar ingredients notably ball clay, kaolin, feldspar and sand. Quarry tiles are basically made from only ball clay according to Uganda Clays Limited (1994). Grimshaw (1971), suggested that stoneware tiles can further be categorized as fine and coarse. Fine stoneware tiles are made of kaolin, ball clay, feldspar and flint or fine sand whereas coarse tiles can be made from fireclays with sufficient amount of fluxes such as feldspar. Hutchings and Xu. et. al. (2006), reported that the use of sand in the tiles should be minimized. This is because sand raises the porosity of the fired specimen hence causing a decrease in strength.

The raw materials used in porcelains have different functionalities. Ball clay provides plasticity to the body that makes it easy to be shaped into other forms. Kaolin contains high alumina which improves on the strength (Kaahwa, 1999). Feldspar is a mineral which forms molten glass at a moderate temperature. It is used to reduce the kiln temperature during firing. Finally sand has a high melting point. It is used to maintain shape of the formed article during firing (Olupot, 2010). The clay content in porcelains

stoneware is a combination of ball clay and kaolin. The three ingredients (clay, feldspar and sand) constitute the term triaxial porcelains commonly used in ceramics.

Clay is one of the major raw materials used for producing ceramic stoneware tiles. The term clay refers to a naturally occurring mineral composed of fine particles which gains plasticity at an appropriate water content and will harden when dried or fired. Clay is mainly found in two major forms, kaolin also known as china clay and ball clay. According to Olupot (2006), Kaolin deposits are found in Mutundwe in Kampala and Mutaka in Bushenyi. Other places with known deposits include Namasera, Buwambo, Kilembe and Kisai with reasonable quantities of clay minerals. Ball clay deposit is at Ntaawo found in Mukono district. Feldspar deposits are found in Mutaka and Lunya in Mukono whereas sand deposits can be found in lido beach in Wakiso district. All these deposits have widely been studied and found to have good quality minerals compared to those marketed in Europe (Kirabira, 2005).

According to the South African National Standards (SANS, 2008), the critical factors in evaluating ceramic floor tiles are water absorption, strength, hardness, shrinkage and the coefficient of linear thermal expansion. Cristina and Federica. et. al. (2001), investigated mechanical properties of ceramic stoneware tiles, the parameters evaluated include shrinkage, strength, water absorption and hardness.

The strength of tiles is determined by analyzing modulus of rupture (MOR) which ranges between 15MPa to 30MPa for pressed tiles (South African National Standards, 2008). Pressed tiles are considered stronger due to high particle densification compared to extruded tiles (Cristina and Federica. et. al. 2001). Currently in Uganda, quarry floor tiles

are manufactured by extrusion and their strength is 15.4MPa(Uganda Clays Limited, 1994). Fired quarry tiles are brown in color due to the impurities such as iron, manganese and titania present in ball clay (Olupot, 2010). Surface scratch resistance determines the hardness of the tile. (South African National Standards, 2008), reported that unglazed tiles experience a volume abrasion of 200to 550 cubic millimeters for pressed tiles. Harder tiles exhibit low volume abrasion compared to weaker ones.

According to South African National Standards, water absorption and strength are parameters which define the class to which the tile product belongs. This is because the two parameters are important in the study of densification. Water absorption for unglazed ceramic tiles ranges from 0.0 to 20%. The lower ranges are specified for surfaces which are vulnerable to water such as bathrooms unlike high ranges which are suitable for high traffic areas such as shopping mall. Quarry tiles from Uganda Clays Limited are fired at temperatures between 800°C to 950°C, they have a water absorption of between 10to 12%.

In triaxial porcelains shrinkage is caused by contraction of voids created by escaped moisture. High shrinkage is associated with high densification hence increased strength and low water absorption. Low shrinkage signifies increased number of pores in the body hence high water and low strength. Work by Cristina and Federica et al (2001) on mechanical properties of porcelain stoneware tiles recorded linear shrinkage of 5-8%. This figure is in range with 5-7.5% which is recommended for tiles by South African National Standards.

Forming methods applied in tile manufacturing includes dry-pressing, semi-dry pressing and extrusion (Ryan, 1978). Currently in Uganda quarry floor tiles from Uganda clays limited are produced by extrusion method. The most appropriate method of manufacturing high strength ceramic floor tiles is dry pressing. This is because no moisture is involved therefore high densification is achieved since no voids are left by moisture after firing. Semi-dry pressing involves adding moisture to the powder to improve on plasticity for easily forming. According to Rayan (1978), a moisture content of about 4-10% is recommended. High moisture content of more than 20% by weight causes cracking after drying or firing. Sanchez and Garcia. et. al (2010), reported that a forming pressure of about 30-45MPa can be applied to the dump powder so as to achieve a strong fired body.

1.2 Statement of the Problem

In Uganda there is currently no readily available information on the mechanical properties of ceramic porcelain stoneware floor tiles made from selected local minerals. Uganda Clays Limited is the leading national producer of floor tiles, they manufacture only quarry. Most of the porcelain stoneware tiles and other ceramic construction materials such as drainage pipes, water sinks are imported into Uganda from Europe, America and Asia especially India. The raw materials needed for production of porcelain stoneware tiles can be obtained locally and they include kaolin, ball clay, feldspar and sand.

High quality ceramic mineral deposits such as those found in Mutaka, Mutundwe, Ntaawo, Kisai, Buwambo and Namusera so far have not been exploited to make high

grade ceramic floor tiles in Uganda to meet the increasing demand. This study intends to investigate the suitability of these deposits in attaining porcelain stoneware tiles evaluated in accordance with SANS.

1.3 Purpose of the Study

Uganda Clays Limited manufactures quarry tiles which are of low strength compared to porcelains. This study was designed to develop a prototype ceramic floor tile (porcelain) from selected raw materials in Uganda. The mechanical properties of the formulated samples were evaluated by comparing data with South African National Standards.

1.4 Objective of the Study

The objectives of this study were:

- (a) To establish alumina and silica content in the raw materials and procedures for making ceramic floor tiles from selected minerals in Uganda.
- (b) To analyze properties such as shrinkage, strength and water absorption of the formulated tiles.
- (c) To classify the formulated floor tiles basing on strength and water absorption by comparing data with the South African National Standards.

1.5 Significance of the Study

The study obtained the right proportions of different raw minerals from Ugandan deposits. The production parameter for floor tiles of specified properties, evaluated in comparison with the South African National Standards was attained.

The results of the study could be availed to the government or private sectors so that a facility could be set up to manufacture porcelain stoneware tiles for local consumption and export. This can create employment and also earn revenue to the government through tax.

1.6 Scope of the Study

The study was limited to use kaolin and feldspar from Mutaka in Bushenyi, ball clay from Ntaawo in Mukono and sand from Lido in Wakiso district. This is because these deposits have been widely studied. The particle sizes used were 25 μ m, 45 μ m, 53 μ m and 80 μ m for sand, ball clay, feldspar and kaolin respectively. Four compositions were formulated containing 20% ball clay, 20-40% kaolin, 30-40% feldspar and 10-30% sand by weight. The type of tiles manufactured was fine ceramic porcelain stoneware floor tiles. The forming method used was semi-dry pressing with a water content of 12% by weight. The samples were pressed at a pressure of 30Mpa and fired at peak temperatures of 1050, 1100, 1150, 1200 and 1250⁰C. The study was confined to measure water absorption, shrinkage and strength.

CHAPTER TWO: REVIEW OF RELATED LITERATURE

2.1 Introduction

The review of literature was focused on three major areas of the study.

(a) Literature on raw minerals including chemical compositions, morphology, manufacturing procedures and their effects on mechanical properties is discussed in this chapter.

(b) The critical parameters considered when evaluating floor tiles namely water absorption, porosity, shrinkage and strength.

(c) Classification of tiles based on data on water absorption and modulus of rupture (MOR) based on South African National Standards has been discussed.

2.1.1 Clay

The major raw material for the production of ceramic tiles is clay. Clay is a mineral consisting of fine grained particles which gains plasticity at appropriate water content (Grimshaw, 1971). Clay can be molded into various shapes but when heated it shrinks and on cooling becomes very hard.

The color of moist ball clay ranges from grey to brown and white for kaolin. The presence of impurities such as iron, magnesia, titania, potassium oxide and calcium oxide among others affects the color of both fired and unfired clay bodies. British Geological Survey (2011), reported that the presence of iron in ball clay constitutes the color of fired samples. Fired clay samples turn red, orange,

brown, white or grey (<http://www.pottery.com>). According to Weems (1988), the iron oxide present in clay will produce certain definite colors dependent on the amount of the oxide present. When iron combines with silica and alumina the color becomes grayish or even black.

The types of clay recognized contain mainly silicate and alumina. These are crystalline and amorphous in nature. A crystalline substance must have a characteristic form or shape. When clay occurs in crystalline state it is fairly pure. The size of the grain in clay enables the material to be classified into coarse-crystalline, in which the grains are readily visible to the unaided eye. Fine or micro-crystalline in which the grains are much finer such that they require a strong microscope to identify their structure. Though many ceramic materials especially plastic clay appears to be amorphous under a microscope, the use of x-rays and an electronic microscope shows that they are crystalline in nature, this therefore means clay can exist in both situations. An amorphous solid has neither a recognizable shape nor geometrical internal structure (Grimshaw, 1971).

The most commonly recognized groups of clay are kaolin and ball clay. Ceramics are generally formulated by using clay of different types and adding silica and feldspar to aid their plasticity and firing consistency. Kaolin mineral is based on the combination of two layers, first is the silica layer composed of silicon and oxygen atoms. Second is the gibbsite composed of aluminum atom and the hydroxyl group. The chemical composition of kaolin is $\text{Al}_2\text{O}_3\text{Si}_2\text{O}_5(\text{OH})_5$ implying sheets of $n(\text{Si}_2\text{O}_5)^{2-}$ linked to $(\text{Al}_2(\text{OH})_4)^{2+}$ units. These sheets are linked by hydrogen bonds which are weaker compared to ionic and covalent bonds (Ryan,

1978). All kaolin minerals are crystalline and they have the same basic structure, the difference between them being the way these pairs of layers are stacked on top of one another to form a crystal.

2.1.2 Kaolinitic Minerals

These clays contain kaolinite as the main clay mineral but with various proportions of other types of non clay materials such as feldspar, mica and talc. These clays can be classified as china clays, ball clays, fire clays and brick clays.

According to Ryan (1978), china clay which is also referred to as kaolin is mainly residual clay, generally composed of clay mineral kaolinite contaminated with mica, feldspar and silica. Residual clay is formed and deposited at its place of origin. Kaolin is coarse and more pure than ball clay, it has low plasticity and contains higher concentration of alumina than silica. Because of its low impurity content, china clay is useful in the production of white decorative ware and in refractory. In the manufacture of floor tiles, kaolin is used as the main source of alumina because the higher the alumina contents in the mixture the greater the strength of the ware formed (Kaahwa, 1999).

Olupot (2006), reported that heating kaolin to a temperature of 1100°C results in the development of mullite, whose chemical formula is $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$. The strength of ceramic ware depends on the interlocking of the fine mullite needles implying that the higher the mullite content the higher the strength. Factors such as firing temperature, composition of alumina and silica in the raw material affect the formation of mullite hence the strength of the ceramic body (Senapati and

Cathy,1998).Kirabira (2005) and Obwoya (2004), reported that alumina combines with silica under the influence of heat to form mullite according to equation 2.1



Yaseen (2000) and Senapati (1998), emphasized that mullite is formed from both clay and feldspar. The one formed from clay is called primary mullite, it occurs at low temperature, however it acts as a seed in the formation of mullite from feldspar. At higher temperature alkalis diffuses out of feldspar, forming

Secondary mullite, these are much thinner in size and they yield stronger ceramic bodies because they interlock perfectly.

Kaolin is highly refractory clay and has a melting point of above 1800^oC when pure. It is not advisable to use kaolin alone when making floor tiles because it has poor plasticity that makes it difficult to be shaped into other objects. If used alone, it is difficult to mature into a hard and dense body during firing. Practically kaolin has to be added to other minerals such as ball clay and feldspar to improve on plasticity and lower the kiln temperature necessary to produce hard and dense ceramic floor tiles (Kirabira, 2005).

Ball clays are sedimentary clays and essentially composed of kaolinite mineral. Sedimentary clays are transported and re deposited far away from its place of origin. They are characterized by their fine grain size and often high content of organic material. Ball clay is named so because of the practice in England of forming dump clay that could be rolled into wagons for transportation. Ball clay is much finer than china clay therefore giving it great plasticity (Rayan, 1978). They

also contain a great quantity and variety of impurities such as iron, Titania, magnesia, calcium and potassium oxides which are finer than the clay particles making it difficult to remove, these impurities reduce their refractoriness and strength.

If whiteness of the final ware is needed then not more than 15% ball clay can be added to required composition. This is because ball clay impairs whiteness and also reduces translucency of vitrified ware (Olupot, 2010). Both kaolin and ball clay contain quartz as a major impurity which can be removed by sieving. Ball clay is usually dark grey and turns red or brown when fired due to the burning off of impurities. The clay particles tighten into a dense structure at a temperature of about 1300°C (Rhodes, 1973). Although ball clay has low alumina content and high silica content, it can be used as a binder in the production of ceramic ware. In the manufacture of ceramic tiles the amount of ball clay has to be controlled because large amounts of water must be added to develop high plasticity which results into high shrinkage, cracking and warping during drying (Olupot, 2006).

Fireclays contain essentially kaolinite and are residual in some cases but the bulks are sedimentary and may have been transported over long distances before being deposited. They are of fine particle size and therefore plastic just like ball clay. According to Rhodes (1973), fireclays are useful for a great variety of products, principally in the manufacture of fire bricks and other refractory for kilns, furnaces, boilers and melting pots.

Brick clays comprise of kaolinite and they are inevitably high in iron content. They also contain gross amounts of other impurities notably calcium compounds. Because of their high impurity content they are fired at relatively low temperature since they do not need any fluxing.

Feldspar is a mineral that forms molten glass at a moderate temperature. Naturally feldspar is usually a mixture of aluminum silicates of sodium or potassium. According to Olupot (2010), feldspars are used in ceramics as fine powder of $\leq 45 \mu\text{m}$, they act as a flux by forming a glassy phase in bodies. This improves on vitrification and translucency. Both sodium and potassium feldspar melt at a temperature of 1100°C to 1200°C . The glassy phase formed as a result of melting of feldspar fill the pores created hence increase in strength and decrease of water absorption of the body (Abadir and Sellam.et.al. 2003).

Sand is purely silica (SiO_2) in nature. In the manufacture of ceramic tiles, sand is used to avoid warping. It also maintains the shape of the fired body by reducing shrinkage. This is because sand has a high melting point therefore at high temperature it cements the gaps created hence maintaining shape (Grimshaw, 1971). According to Kingery (1976), sand must be used as fine powder. This is because the expansion of crystals in a matrix due to heating, leads to stress that may give rise to cracking. The stress in the grains can be reduced if the grain size is reduced, therefore properties of stoneware are improved if fine grains are used rather than coarse material (Stathis and Ekonomaku. et.al.2004).

2.1.3 Chemical Composition of Raw Materials

The chemical composition of the starting minerals greatly influences the processing procedures and the mechanical properties of the fired samples. The minerals used in porcelain stoneware include clay, feldspar and sand. These minerals contain alumina, silica, calcium oxide, potassium oxide, titania and iron to mention but a few. Olupot (2010), analyzed the chemical composition of these minerals from some deposits in Uganda as indicated in Table 2.1.

Table 2.1: Chemical compositions from different deposits in Uganda (wt %).

Compound	Mutaka Feldspar	Mutaka Kaolin	Mukono ball clay	Lido sand
SiO ₂	62.9	48.8	67.2	100.0
Al ₂ O ₃	18.3	36	18.2	0.127
CaO	<0.09	<0.09	0.306	<0.09
Fe ₂ O ₃	0.065	0.238	2.83	0.201
K ₂ O	11.2	1.14	0.975	<0.06
MgO	<0.02	0.038	0.363	<0.02
MnO	<0.003	0.0277	0.0262	0.0092
Na ₂ O	0.409	0.0481	0.185	<0.05
P ₂ O ₅	0.0874	0.0094	0.049	0.0171
TiO ₂	0.0036	0.0041	1.38	0.1720
LOI	3.1	12.6	8.1	0.4
Total	<100.98	<99.0	<99.61	<100.75

Kirabira (2005), argues that high content of alumina in the clay sample yields a much stronger ceramic ware. But high amounts of impurities such as oxides of

iron, manganese, potassium and calcium could lower its strength. Mutaka deposits have the required composition of alumina in kaolin and feldspar necessary to manufacture porcelain ware (Olupot, 2006).

Nyakairu and Koeberl (2001) and Bergmann and Zimmer (2006), reported that oxides of elements of aluminum and silicon in raw materials is determined by X-ray Fluorescence (XRF). However these elements can also be determined by Instrumental Neutron Activation Analysis (INAA). Using INAA samples of about 150g are measured and irradiated for 8 hours at a neutron flux of $2.10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$.

2.1.4 Mineral Composition in Ceramic Tiles

Sanchez and Garcia. et. al. (2010), reported that the composition by weight of the raw materials in porcelain stoneware tiles should be between 40%-60% clay, feldspar is 40%-50% and quartz (sand) is 10%.

The materials used have different functions. Ball clay helps in forming by providing high plasticity, kaolin provides drying strength after firing, feldspar acts as fluxing body by developing a glassy phase at low temperature. Sodium feldspar being mainly used in the sintering processes. Quartz promotes dimensional stability due to its high melting point (Olupot, 2010). According to Hutchings et. al. (2006) quartz (sand) increases the porosity of the fired specimen because it reduces the sinterability of the body therefore its content in the ware should be minimized. The raw materials should be crushed and powdered to particles sizes of less than $160 \mu\text{m}$ for clay and less than $40 \mu\text{m}$ for feldspar and quartz.

2.1.5 Bonding

Grimshaw (1971), reported that the basic principles of crystal structures and bonding give a better understanding of the different strength and characteristics between metallic and non metallic materials. Ceramics can be defined as inorganic, nonmetallic minerals. They are compounds formed between aluminum and oxygen, calcium and oxygen and silicon and nitrogen (Olupot, 2010). The type of bonding between atoms and the way atoms are packed together in ceramics primarily affects the chemical, mechanical, electrical, magnetic and optical properties of ceramics.

The main types of bonds present in a crystal lattice are covalent and ionic bonds in which electrons are transferred or shared between adjacent bonds. Other types include metallic, hydrogen and van der Waals which are weaker bonds. The types of bonds majorly present in ceramic materials are covalent and ionic bonds (Kingery, 1976). According to Grimshaw (1971), the ionic character is given by difference in electro-negativity between cations and anions while covalent nature by sharing of valence electrons. Materials with covalent and ionic bonds are stronger, rigid at room temperature, hard and rupture after a small displacement. Although these materials are hard they are brittle this is because of the covalent bonds present (Raymond, 1994).

When a crystal containing ionic or covalent bond is subjected to compression, the ionic components or atoms are being forced closed hence strengthening the bonds. If a similar material is subjected to tensional or shear force, the atoms are pulled

from their equilibrium position hence weakening the bond resulting into cracks or breakage. Ceramic materials are made up of mainly covalent and ionic bonds making them strong in compression but weak in tension or shear hence brittleness.

2.1.6 Particle Size, Shape and Distribution

The morphology of the raw material which refers to the size, size distribution, shape and other parameters that may help to define how the powder will flow, pack and react to form the final body. The analysis of particle sizes is a very important factor since varying their sizes directly affects the way the grains are agglomerated. The strength of a ceramic material is affected by size, shape, distribution and density or closeness of the particles. This is because these properties directly affect the texture, porosity, plasticity, specific gravity and bulk density of the ceramic material both raw and fired (Grimshaw, 1971). The texture of the ceramic material is said to be coarse when the particles are large or loosely spaced and fine when the particles are small and closely spaced. Besides affecting the porosity and plasticity, texture also has influence on the shrinkage of the material after firing or drying. Open-textured materials whose particles are not closely packed and have many large voids are found to shrink and crack when fired or dried compared to close-texture materials with small voids and more closely packed (Kingery, 1976).

Rahbar and Adubar.et.al. (2010), reports that when larger particle sizes are sintered, large pore sizes are produced compared to smaller particle sizes. Due to

larger pores the strength of the ceramic body formed is lower. Hutchings et. al. (2006), reported that particle size greater than 100 μ m creates large pores in tiles. This is because the particles do not totally deform during the pressing stage and the voids they form remain in the tile after firing. Generally finer particles create stronger fired ceramic tiles than coarse ones. This is because they form smaller pores within the tile. From Table 2.2 it is clear that finer particles have high modulus of rupture than coarse particles.

Table 2.2: Relationship between Particle sizes and Strength of Ceramic ware.

Particle sizes / μ m	MOR/MPa
45-63	68.7
63-90	49.8
90-125	35.1
125-250	16.6

Source: (Rahbar et. al. 2010)

Finer particles are more plastic because they absorb more water than coarse particles. Firing samples made of finer particles creates many microspores due to escaped moisture. This therefore increases linear shrinkage, warping and cracking of samples (Grimshaw, 1971).

As reported by Grimshaw (1971), ceramic samples made of equal-sized particles form a system with 38% void spaces. When three particles sizes were packed

together the void spaces of the system reduced to 5.4%. Mixing three particle sizes is important in that much smaller particles tend to occupy most of the voids as seen in Figure 2.1.

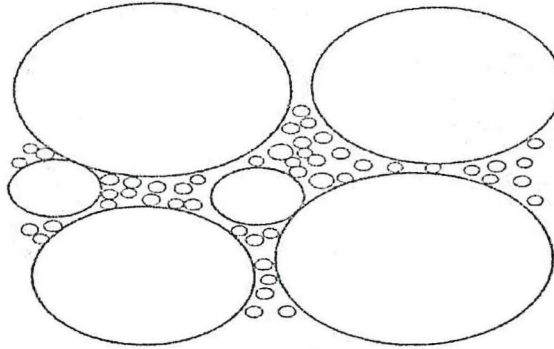


Figure 2.1: Effect of particle size variation on densification. (Source: Kingery, 1976).

This therefore creates a stronger closed packed arrangement of particles hence improving on the strength of the ceramic tile formed.

Maximum strength can be achieved if grading is done such that the composite material is to consist of 65.6% of larger particles, 24.9% of medium particles and 9.5% of finer particles (Grimshaw, 1971). This is because maximum densification is achieved, porosity is reduced by smaller particles filling the voids hence increase in strength. The use of carefully graded materials greatly lessens the possibility of shrinking, cracking and warping when the material is subjected to sudden changes in temperature because the number of pores is reduced.

2.1.7 Forming

According to Kaahwa (1999), forming involves feeding the powder or plastic material into a mould and compacting or pressing it to an appropriate pressure. Pressing is a method used to produce floor tiles and other ceramic wares (SANS 2008). Pressing can be categorized into dry-pressing where no moisture content is applied to the powder and semi dry-pressing where a moisture content of approximately 4-10% is used hence forming a damp powder. Semi dry-pressing is used in the manufacture of bricks and floor tiles and can also be applied in industrial porcelain products (Rayan, 1978). Forming pressure of about 30-55MPa compacts particles more tightly hence improving its densification and strength (Sanchez and Garcia. et. al 2010).

Hot-pressing is a more advanced and complex procedure, it combines the simultaneous application of pressure and temperature to enhance densification. This process involves filling the mould with powder, then applying pressure while the entire system is held at an elevated temperature (<http://www.azom.com>). This process produces stronger tiles since particles are highly compacted and pore sizes are smaller because no or very little moisture is applied during the pressing process. The use of high forming pressure improves on densification and greatly improves strength and lowers water absorption of the tiles (<http://www.onlinelibrary.willy.com>).

Other forming methods can include jiggering, extrusion and throwing. Jiggering is a similar process used for flat ware or tile production where a flat disc of the body is first formed by spreading the body on a flat revolving plaster head with a flat tool brought down from above. The flat body is then thrown onto the plaster mould which

forms a flat shape (Rayan, 1978). The mould is rotated in a jigger head and a profile tool is brought down on to the body to shape the back of the ware formed. These methods yield weaker tiles because no compaction is done so the particles are loosely attached. Extrusion is also used as one of the methods of forming, here the plastic body is forced through a mould of the required cross-section and the extruded column is cut into suitable length. This method is mainly used in making bricks, pipes and rods including quarry floor tiles by UCL. Throwing is the old method of shaping a plastic body by hand while it is rotating on a potter's wheel, considerable skill and experience is needed. This method is mainly used for pottery. After forming the body, firing is done to improve its appearance and strength.

Carneane et. al. (2001), explains that in the process of ceramic tile manufacture, ceramic powder is wetted and plunged into a ball mill. To obtain a mixture which is then compacted at a pressure of 30MPa using an automated hydraulic press. The compacted samples of dimension 58x126x10 mm, are then dried at a temperature of 110°C for 2 hours and subsequently fired at 950°C-1300°C in a kiln for a soaking time of 55 minutes to form a strong ceramic ware. According to Sanchez and Garcia (2010) industrial processing of tiles involves three stages (i) wet milling (ii) pressing at 30MPa to 45Mpa with moisture content of 5%-7% (iii) firing for 40-60minutes at 1180°C to 1220°C to obtain maximum densification.

2.1.8 Firing

Ceramic ware can under go at least one firing which converts the shaped ware irreversibly into a strong material. Unglazed ware is fired only once and glazed ware

is fired twice (Singer, 1963). A glaze is a glassy finish which seals the body and at the same time provides a smooth and brilliant finish. Firing can be done in different ways, first unglazed ware is heated to a low temperature and the glaze applied. The two are then heated to a high temperature for both to mature. The raw ware is glazed then both bodies are fired at a high temperature to mature.

During firing, the removal of water takes place in different stages. When the body dries the mechanical water is removed. At a temperature of 100°C, any moisture (hygroscopic water) left in the ware after drying is driven off. At about 400-500°C the hydroxyl group (chemically held water) in the clay mineral is driven off (Rayan, 1978). The organic matter mainly impurities present in the body may be removed at a temperature of between 300°C to 700°C. The ease of removal of this organic matter depends on the type present. Besides, firing temperature of ceramic ware is affected by various factors such as mineral composition of the raw materials in the body and particle preparation which involves size and distribution (Grimshaw, 1971).

A ceramic body should be heated to a desired temperature at a recommended rate so as to produce tiles of required strength and quality. If the ceramic material is overheated the effect may be dangerous instead of useful. Overheating may create excessive amount of fussed mater which may endanger the stability of the mass by creating cracks. This decreases the strength of the ceramic tile formed. Under heating also causes weakness in the tile, due to poor maturity of the raw materials. Excessive or prolonged heating also causes shrinkage of clay ware and swelling or bloating (Grimshaw, 1971).

In triaxial porcelains firing yields a possibility of mullite formation. According to Olupot (2006) mullite content in porcelains increases the strength of the body according to the mullite hypothesis. Kirabira (2005) and Obwoya (2004), reported that when a ceramic body is fired beyond 1100°C , alumina combines with silica to form mullite.

Consequently the formation of mullite leads to a reduction in porosity hence resulting to an increase in strength. A reduction in porosity can also be caused by grain growth which results from increase in firing temperature. Firing at a temperature of 1000 to 1200°C melts feldspar hence forming a glassy phase. The formation of glassy matrix leads to a decrease in porosity. This is because molten glass fills the voids created in the ceramic body. This leads to formation of a dense structure attributed to vitrification due to fluxing action (Obwoya, 2004).

Cristina et al. (2001), explains that during tile manufacture, samples of dimension $110\text{mm}\times 55\text{mm}\times 10\text{mm}$ were formulated at a pressure of 33MPa. Firing was carried out at peak temperatures of 1160, 1180, 1200 and 1220°C with a heating rate of $25^{\circ}\text{C min}^{-1}$ to form a dense body. According to Agenor and Danchamir. et. al. (2009), sample tiles of dimension $80\times 20\times 7\text{mm}$ were pressed at a pressure of 45Mpa with a moisture content of 5.5% by weight. They were then dried in an oven at 110°C and fired in an electric kiln at a heating rate of 70°C/min from 25°C to 500°C and 25°C/min from 500°C to 1230°C , with a hold at peak temperature for 6min. Tucci and Esposito. et. al. (2007), reported that samples with 6% (wt) water content and sieved powder were pressed at 52Mpa. The sintering behavior was evaluated by firing in a furnace at peak temperature of 1180, 1200, 1220, 1240, 1260 and 1280°C for a

soaking time of 40 minutes. The heating rate from room temperature to 600°C was 60°C/min and from 600°C to different firing temperature was 20°C/min. After heating the samples were left inside the furnace until naturally cooling to room temperature.

During cooling physical changes take place such as cracking, shrinkage and warping. Olupot (2010) and Senapati and Cathyl (1998), reported that, as a body starts cooling, pyroplastic deformation and relaxation within the glass phase prevents the development of residual stress until the glass transition temperature is reached. As a body cools below the glass transition temperature, residual stress develops because of thermal expansion mismatch between mullite and sand. This causes cracks, the cracking is dictated by sand particle size and the cooling rate.

2.1.9 Effect of Firing on Porosity

The porosity of unfired materials is related solely to the size and shape of the particles used and the manner in which particles blend together. On firing, some particles fuse hence interlocking the pores. At a higher temperature finer particles fuse more rapidly than coarse particles. The liquid formed fills the voids in the mass thus reducing porosity (Kingery, 1976). However, the porosity of a body may increase or decrease when the temperature is raised depending largely on the composition and minerals which are present. In the presence of a reasonable amount of carbonates or finely powdered coke in the clay sample its porosity increases on heating. Clay samples containing mica or feldspar reduce their porosity because these minerals turn glassy on firing hence filling the voids created (Grimshaw, 1971). Kaolinitic clays which are used for making floor tiles especially china clays which have finer particles when

mixed with reasonable amount of mica or feldspar would reduce their porosity on heating hence increase in strength.

Porosity of clay made materials increases as they are heated until a temperature of about 750°C-850°C. This is because clay expands slightly and loss of water and impurities occur at the same time (Grimshaw, 1971). When the temperature is increased above 900°C, partial fusion occurs and molten matter begins to fill up the pores causing a reduction in porosity.

According to Martin and Ma-Rinco.et. al. (2007), open porosity consists of fine and interconnected pores with irregular shape. On the other hand, closed porosity is due to large, isolated and spherical pores. The size of the closed pores increases with firing temperature, from an average diameter of $\approx 5\mu\text{m}$ at 1200°C to 100 μm at 1300°C for porcelain stoneware. It is established that porcelain stoneware is fired at optimum vitrification temperature range of 1260-1280°C. Firing above vitrification temperature for tiles made of kaolinitic clay, feldspar and quartz results into a fall in physical properties due to forced expulsion of entrapped gases resulting into blister and bloating.

2.1.10 Effect of Firing on Strength

When clay samples are heated there is direct removal of water, this improves on the densification of the particles hence increase in the hardness and strength of the product. At a certain temperature of above 900°C finer particles diffuse and fill the voids in the sample this strengthens the bonding of these particles within the samples hence resulting in improvement in strength.

Martin and Ma-Rinco et. al. (2007), reported that stoneware tiles containing 50% clay, 40% feldspar and 10% quartz have an average bending strength of 34 ± 2 MPa for samples fired at 1260-1280°C range. SANS (2008), categories this as B₁ tiles which happens to be the strongest.

An experiment to establish the effect of firing temperature on the strength of floor tiles using atomized ceramic powder showed that the bending strength varies with firing temperature according to Fig 2.4. The atomized ceramic powder used contained clay, quartz, carbonates, feldspar and mica (Obwoya, 2004).

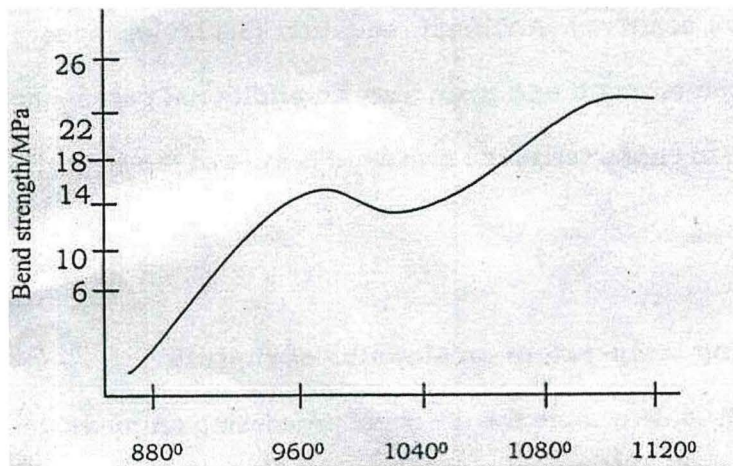


Figure 2.2: Bending strength of floor tiles as a function of temperature.

According to Obwoya (2004), the major microstructural flaw, which controls MOR, is micro-cracks which is caused by sand in the body. The formation of cracks in ceramic samples is dictated by temperature. At extreme temperatures sand grains expand and this leads to spontaneous cracking during cooling (Stathis and Ekonomaku. et.al 2004).

Over firing creates dark spots on the samples especially with clay samples containing carbonaceous elements as impurities. The dark spots may lead to decrease in strength of the body due to pores created by the burnt substances.

Bloating occurs when the firing cycle does not match the clay or minerals being fired. The symptoms are voids created within the body. Generally when a body is being fired and the gases formed within can not escape, with no place to go they expand and generate a void called a bloat. Lack of enough oxygen due to over packing of samples in the kiln can also cause bloats, their strength is reduced. Bloating can be prevented by slow firing rate because this gives time for carbon and sulphur to combine with oxygen and work its way out of the clay body. Provision of an oxygen rich environment in the kiln by use of ventilation can help minimize bloating (<http://www.lagunaclay.com>).

2.2.1 Porosity

Ceramic products with the exception of glass are to some extent porous, some fraction of the total volume is composed of air in form of pores or voids. According to Grimshaw, (1971) porosity of a material is the total proportion of air spaces contained between solid particles of which the body is composed. Pores can be classified as: closed or sealed pores, channel pores connecting different pores, micro pores which are so small and cannot be filled with water and other liquids during soaking. These types of pores can be categorized into two main sets: true and apparent porosity. True porosity is the ratio of the volume of all voids both open and closed to the total volume of the article. Apparent porosity is the ratio of the volume of water or liquid capable of being absorbed to the exterior volume of the article. Mostly, the term porosity is

applied to apparent porosity as estimated from the amount of water or suitable liquid absorbed by a given weight or volume of sample. The apparent porosity, p , expressed as a percentage is calculated using equation 2.2

$$p = \frac{m_2 - m_1}{v} \times 100\% \quad (2.2)$$

The exterior volume, v , expressed in cubic centimeters is calculated from equation 2.3.

$$v = m_2 - m_3 \quad (2.3)$$

Where m_1 the mass of the dry tile, m_2 is the mass of wet tile after impregnation with water and m_3 is the mass of the suspended tile impregnated by immersion.

The volume of open pores, v_o , and the volume of the impervious portion, v_i , expressed in cubic centimeters, are given by the equations 2.4 and 2.5.

$$v_o = m_2 - m_1 \quad (2.4)$$

$$v_i = m_1 - m_3 \quad (2.5)$$

The apparent relative density, T , of the impervious ceramic ware is calculated using equation 2.6.

$$T = \frac{m_1}{m_1 - m_3} \quad (2.6)$$

The bulk density, B , expressed in grams per cubic centimeters of a specimen is the quotient of its dry mass divided by the exterior volume, including pores. The bulk density is calculated from equation 2.7 (ISO 10545-3, 1995)

$$B = \frac{m_1}{v} \quad (2.7)$$

Apparent and bulk densities signify densification of particles in the samples. High densification means low porosity and high strength.

According to Grimshaw, (1971), porosity of ceramic materials can be influenced by shape and size of the particles, treatment to which the material is subjected during manufacture such as moisture content, firing temperature and forming pressure. Porosity of ceramic materials has great influence on the property of absorption, conductivity, chemical corrosion, refractoriness, strength, hardness and many others. The strength of a ceramic material greatly depends on the pore size and the size of the particles (Kingery, 1976). Pores generally decrease the cross-sectional area on which the load is applied but also acts as stress concentrator hence weakening the tile. For an isolated spherical pore the stress concentration is doubled. The strength of a porous ceramic material decreases exponentially with porosity. The breaking strength of a ceramic material is related to porosity according to the equation 2.8 (Olupot, 2006)

$$S = S_0 e^{-bp} \quad (2.8)$$

Where S_0 is the maximum strength with zero porosity, p , is the volume fraction of pores and b is a constant which is 5-8 for porcelains. Experimentally porcelain is found

to have a porosity of about 3% and earthenware is found to have a porosity of 10 to 15% (Kingery, 1976). This difference in the porosity is responsible for the difference in the magnitude of the strength of the two materials. From equation 2.8, strength of ceramic products is exponentially related to porosity.

2.2.2 Water Absorption

Grimshaw (1971), defines water absorption of a body as the percentage increase in the weight of the samples on saturation with water. The amount of water absorbed by a ceramic tile is the best indicator of its quality and class (SANS, 2008 and Cristina. et.al 2001). Water absorption reflects the density of the body of the tile whereby tightly packed particles experience a low water absorption compared to loosely held particles. Low water absorption therefore signifies improved mechanical strength of the samples.

Determination of water absorbed of ceramic tiles is very important in assessing its densification and strength. There are two methods of impregnating tiles with water namely boiling and vacuum method. Boiling is done to impregnate open pores that are easily filled with water. These pores are small, irregularly shaped and interconnected with others. Impregnation by boiling involves placing tiles vertically in a heating apparatus with no contacts between them, heating is then carried out for two hours. The source of heat is removed, the samples cooled and wiped before measurement of their masses is accurately carried out using an electronic beam balance (ISO 10545-3, 1995). In the calculation, the assumption made is that 1cm^3 of water weighs 1g. This is true within about 3% for water at room temperature. For each tile, the water absorption expressed as a percentage of the dry mass, is calculated using equation 2.9

$$W.A = \frac{m_2 - m_1}{m_1} \times 100\% \quad (2.9)$$

Where m_1, m_2 is the mass of dry and soaked tiles respectively.

According to Abadir and Sellam. et.al (2003), feldspar decreases the water absorption of samples by forming a glassy matrix. Feldspar forms molten glass at about 980⁰C (Stathis and Ekonomaku. et. al 2004), the content of glassy phase increases with temperature. The glassy phase tends to fill the voids created hence decrease in water absorption.

According South African National Standards, class B₁ tiles have water absorption of less than 3%, B₂ tiles absorb between 3 to 6% water, B₃ floor ceramic tiles absorb between 6 to 10% and finally class B₄ ceramic tiles absorb less than 20% water content. Quarry floor tiles manufactured by Uganda clays absorb 10 to 12% water by weight (Uganda Clays Limited, 1994).

The density of tiles is measured by the amount of water it absorbs. Non-vitreous tiles absorb 7% or more water by weight. Semi-vitreous tiles absorb 3 to 7% of water by weight. Both non vitreous and semi vitreous tiles are suitable for interior use. Vitreous tiles absorb 0.5 to 3% by weight of water and impervious tiles absorb less than 0.5% by weight of water. Both vitreous and impervious tiles are suitable for both interior and exterior used because they are frost resistant ([http://www.nationalfloor covering alliance/tile](http://www.nationalfloorcoveringalliance/tile)).

2.2.3 Shrinkage

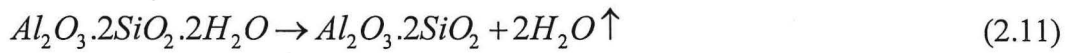
Kingery (1976), reported that pressed green ware contains between 25-50% porosity. This amount depends on particle size, particle size distribution and forming methods. During the firing process this porosity is removed causing linear firing shrinkage. The firing shrinkage can be reduced by addition of a non shrinking material such as sand or flint to the mix. If firing is carried out to complete densification a linear shrinkage of 12-15% can be recorded (Kingery, 1976). A research on mechanical properties of stoneware tiles (Criatina.et.al. 2001) presented a linear shrinkage of approximately 7% which value is comparable to the industrial range of 5-7.5%. Values of shrinkage by Cristina and Federica.et.al (2001) were calculated basing on an average of six samples at each peak temperature.

Linear shrinkage and water absorption are directly related to open porosity, these properties are used to represent the verification curve. Obwoya, (2004) investigated the mechanical properties of Ugandan clays, his findings showed that most samples which exhibited high MOR also exhibited high shrinkage. According to Martin and Maringo et. al. (2007) the linear shrinkage LS (%) of fired stoneware samples can be calculated from equation 2.10.

$$L.S = \frac{l_s - l_e}{l_s} \times 100\% \quad (2.10)$$

Where l_s, l_e are the dimensions (mm) of the green and fired specimen respectively

Kaolinite is one of the minerals used in manufacturing ceramic tiles, its chemically illustrated as $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$. Senapati and Cathy (1998), reported that the crystal structure of kaolinite contains hydroxyl group. Firing kaolinite to a temperature of approximately $550^{\circ}C$ causes dehydroxylation of these groups hence forming metakaolin. The chemical equation representing this process is 2.11



Shrinkage is caused by dehydroxylation. However, the percentage of shrinkage of samples increases with increasing content of kaolinite clay. This is because of increase in dehydroxylation (Cristina and Federica. et . al.2001).

Warping or distortion is caused by variation in the amount of firing shrinkage at different parts of the samples, this sometimes causes cracking. The major cause of warping is density variations in the green body. There is high shrinkage for parts of the samples with low density than those parts with higher density in the green body. For pressed samples pressure variations in the die causes density variations, usually shrinkage at the centre of the samples is higher than at the ends (Kingery, 1976). The presence of temperature gradient also causes warping. If a tile is laid on a flat surface and heated from above, there is a temperature difference between the top and bottom parts of the samples. This causes greater shrinkage at the top of the samples hence warping (Kingery, 1976).

2.2.4 Strength of Ceramics

The strength of ceramic materials is associated with its ability to withstand pressure being applied to it without rupturing. Strong ceramic materials have a capacity to be stretched or compressed without fracture. Martin and Ma-Rinco. et.al. (2007), reported that the average bending strength of porcelain stoneware samples fired at 1260°C-1280°C is

34 ± 2 Mpa. Furthermore Cristina and Federica. et. al (2001) recorded values of 30-50MPa at 1140-1240°C. The results were beyond 27MPa required by EN 100.

Modulus of rupture is used to determine the strength of ceramic samples using three point loading test. According to ISO 10545-4(1994), this method involves two cylindrical rods made of metal are in contact with the test specimen with a length, l , projecting from either sides of the specimen, one rod is slightly pivotal and the other rod rotatable as seen in Figure 2.5. The central cylindrical rod of the same diameter is used for transmission of the load force, F . Breaking load is the force in newtons necessary to cause the test specimen to break.

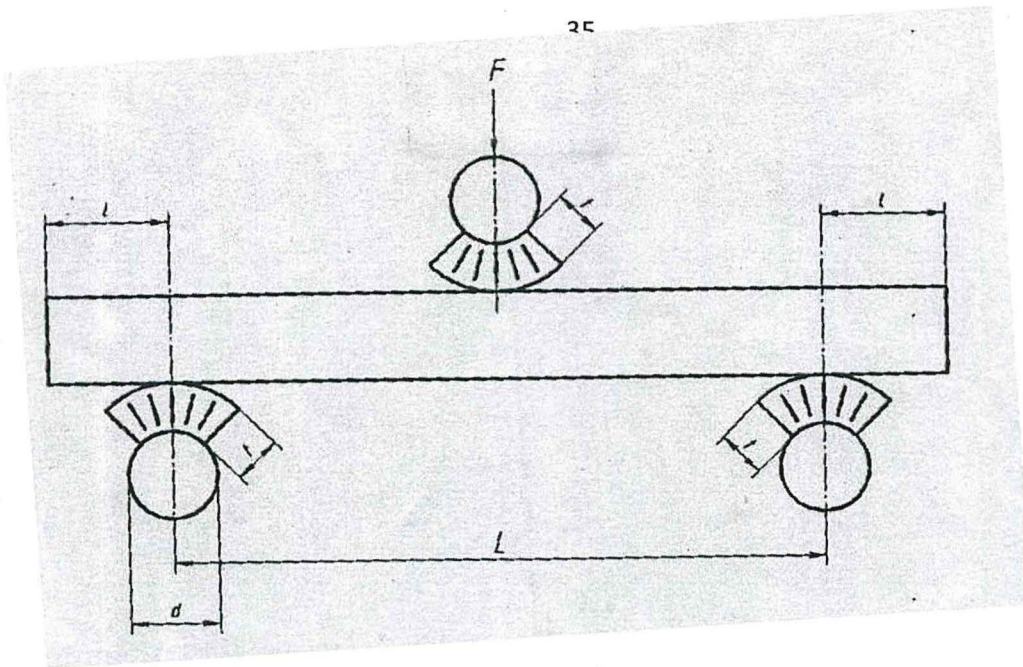


Figure 2.3: Schematic Diagram of the Three Point Loading test (Source ISO 10545-4)

The breaking strength, S , expressed in Newton's is calculated by means of equation 2.12

$$S = \frac{FL}{b} \quad (2.12)$$

Where F the breaking load in Newton's, L is the span in millimeters between the span rods and b is the width of the test specimen in millimeters.

The modulus of rupture (MOR) expressed in newtons per square millimeters is calculated by means of equation 2.13

$$MOR = \frac{3FL}{2bt^2} \quad (2.13)$$

Where t is the minimum thickness of the test specimen in millimeters. A minimum of five acceptable results is necessary to calculate the average value (ISO 10545-4, 1994).

The strength of ceramic materials can also be expressed in terms of Young's. The strength, ϕ , depends on the Young's modulus of elasticity, ε , of the ceramic material, its effective surface energy for fracture initiation, γ_i , and the size of the flaw, δ , as seen in equation 2.14. From the same equation strength decreases with increase in flaw size. (Anura, 1999).

$$\phi = \text{cons} \tan t \left(\frac{\varepsilon \gamma_i}{\delta} \right)^{0.5} \quad (2.14)$$

The terms ε and γ_i in equation 2.14 both depend on the porosity in the ceramic, as porosity increases they decrease. The constant depends on the shape, position and orientation of the fracture-initiating flaws. The term $\varepsilon \gamma_i$ determines the ceramic and fracture toughness represented by equation 2.15.

$$k_{ic} = (2\varepsilon \gamma_i)^{0.5} \quad (2.15)$$

Flaws are regions within the sample from which the material is missing and through which mechanical load or force cannot be transmitted. When the mechanical load is increased on a ceramic body, the stress adjacent to the flaws also increases. The stress is high which then pulls the atoms apart. When the bonds between the atoms are ruptured the flaws grow in size causing the sample to split or break.

According to Ashby (1986), ceramics can be fibre-strengthened just like concrete in order to improve on its toughness and modulus of elasticity. A material is stretched more when it's porous than when it's fully dense, a porous material therefore has low modulus of elasticity (Anura, 1999). Ceramic stoneware containing 50% kaolinitic

clay, 40% feldspar and 10% quartz with a firing temperature between 1260⁰C to 1280⁰C has a young's modulus of 72 ± 0.5 , this value is comparable with the commercially accepted value of 70 ± 1 (Martin and Ma-Rinco et. al. 2007).

Crushing strength is to some extent dependent on the size and shape of the article. Usually larger samples need more power to be crushed. However, Shape, size and distribution of the particles has a direct impact on the hardness and strength. Maximum strength is obtained by use of irregularly shaped particles. This is because they interlock tightly than regular shapes (Grimshaw, 1971). Generally in many fired ceramic materials, tensile and crushing strength are roughly inversely proportional to porosity.

The formation of mullite as a result of increase in temperature improves on the strength of the ceramic body (Olupot, 2006 and Senapati and Cathy, 1998). Abadir and Sallam et.al. (2003) reported that large amount of kaolinite is fundamental in the mullite formation thus obtaining the desired strength of the ceramic tile. The presence of alumina is very useful in improving the bending strength of the body. It improves on the micro-hardness of the glassy phase and decreases the brittleness of the ceramic samples. Talc also has a considerable effect on the ceramic body because its amount in the raw material increases both the mullite content and the modulus of rupture of the article.

According to Cristina and Federica et. al. (2001) and Senapati and Cathy (1998), the strength of stoneware can be explained by developing three major theories that can be summarized in the mullite hypothesis. Thus interlocking of the fine mullite needles is

responsible for the stoneware strength. The matrix reinforcement thus “thermal” compressive stress due to thermal expansion leads to strength improvement. Finally the dispersion-strength hypothesis thus dispersed particles limit the size of Griffith flaws leading to increased strength..

According to Senapati and Cathy (1998), the presence of cristobalite instead of sand in porcelains often produces an increase in strength. This is mainly achieved in porcelain bodies with excess sand content. The transformation of sand to cristobalite is accelerated by using small particles of about 25 μ m. Increase in strength of cristobalite porcelain bodies can be explained basing on three perspectives: cristobalite grains tends to be much smaller than sand particles hence smaller grains always form stronger bodies, cristobalite is formed at the expense of sand hence reducing the quartz content and finally cristobalite inversion temperature is low 225-250⁰C compared to 573⁰C for sand, this leads to lower strain development during cooling process hence forming less crack.

Kingery (1976), reported that development of cracks within a ceramic body is caused by sand content and their particle sizes. A glassy matrix with sand, a single sand particle experiences both radial and tangential stress due to difference in thermal expansion coefficients. Therefore the total stress ,P, on the particle is given by equation 2.16

$$P = \frac{\Delta\alpha\Delta T}{\frac{1+\nu_m}{2E_m} + \frac{1-2\nu_p}{E_p}} \quad (2.16)$$

Where $\Delta\alpha$ is the difference in expansion coefficients between the glassy matrix and the particle, ΔT the cooling range of the glass-particle system, ν_m and ν_p are Poisson ratios, E_m and E_p are elastic moduli and subscripts m and p the matrix and particle respectively. The nature of cracks is dependent on the expansion coefficient of the matrix and particle. If the particle contracts more than the matrix, P , is negative resulting into circumferential cracking around the particles. This is true for sand particles in a felspathic glass matrix. The stress generation and associated cracking due to the presence of sand particles is severe because of rapid displacive phase transformation during cooling (Senapati. et. al. 1998).

2.3.1 Modulus of Rupture and Water Absorption Rating

The density of ceramic tiles is determined by its ability to absorb water and strength. Ceramic floor tiles can be manufactured by either extrusion or pressing method (SANS 2008). Extruded tiles are formed by a process in which wet clay or plastic raw material is forced through a mould and then cut into shape before firing. They are categorized as group A₁, A₂, A₃ and A₄. As shown in Table 2.3, A₁ is the strongest and A₄ weakest. Pressed tiles are formed by applying pressure to the raw material placed in a mould. They are categorized as B₁, B₂, B₃ and B₄. (<http://www.builddirect.com>). The property and physical requirement of extruded and pressed tiles is shown on Tables 2.3 and 2.4.

Table 2.3: Physical requirements for pressed (group B) tiles

Property	Tile group	Physical requirements(%wt)
Waterabsorption,% (by mass)	B ₁	Average ≤ 3
	B ₂	3 ≤ Av ≤ 6
	B ₃	6 ≤ Av ≤ 10
	B ₄	≤ 20
Modulus of rupture,MPa,	B ₁	Average 30
	B ₂	20
	B ₃	18
	B ₄	15

Table 2.4: Physical requirements for extruded (group A) tiles.

Property	Tilegroup	Physicalrequirements
Waterabsorption,% (by mass)	A ₁	Average ≤ 3
	A ₂	3 ≤ Av ≤ 6
	A ₃	6 ≤ Av ≤ 10
	A ₄	≥ 10
Modulus of rupture,MPa,	A ₁	17
	A ₂	17
	A ₃	15
	A ₄	7

SOURCE: (SANS, 2008)

2.3.2 Tiles

A tile is a flat surfaced piece of hard-wearing material made of ceramic, stone, glass, plastic and even rubber (<http://www.tile.com>). Tiles are generally used for covering roofs, floors, walls and other surfaces such as table tops. Roof tiles are specifically designed to keep rain out and are traditionally made from ball clay (UCL, 1994). However, materials such as concrete and plastic are also used. Floor tiles are commonly made from ceramic or stone although recent technological advances have resulted into rubber, plastic or glass tiles. Rubber floor tiles are more useful in areas which require protection from easily breakable floor. Their common uses include; flooring of workshops, garages and gyms. Plastic floor tiles are the most recent innovation and these are used in areas vulnerable to contamination by oils, grease and chemicals. The common use of these tiles can be in chemical factories, shops and even school laboratories.

Ceramic floor tiles are made from clay mixed with other ingredients such as feldspar and sand, they can be glazed which makes them more slippery. They are commonly used in showers, living rooms, bed rooms, kitchens, laboratories, industries and so on. They are easy to clean, attractive, durable and replaceable incase of any damage.

A report by the South African National Standard, (2008) emphasizes that the type of traffic if pedestrian and type of shoe heel or sole if rubber or metal must be considered before using a specific ceramic tile group. Other factors to consider are the amount and frequency of traffic. Ceramic floor tiles classified as class one (A_1 or B_1) are suitable for low traffic domestic areas such as bathrooms and toilets. Classes two (A_2 or B_2) are

used in domestic areas where normal foot wear is worn, such as the living and bed rooms. Tiles which are suitable for entire domestic areas such as kitchens, bathrooms, living and bedrooms and also commercial areas subjected to light traffic are classified as class three (A₃ or B₃). Class four tiles (A₄ or B₄) are suitable for use in high traffic areas such as hospitals, public buildings, shopping malls, schools and restaurants.

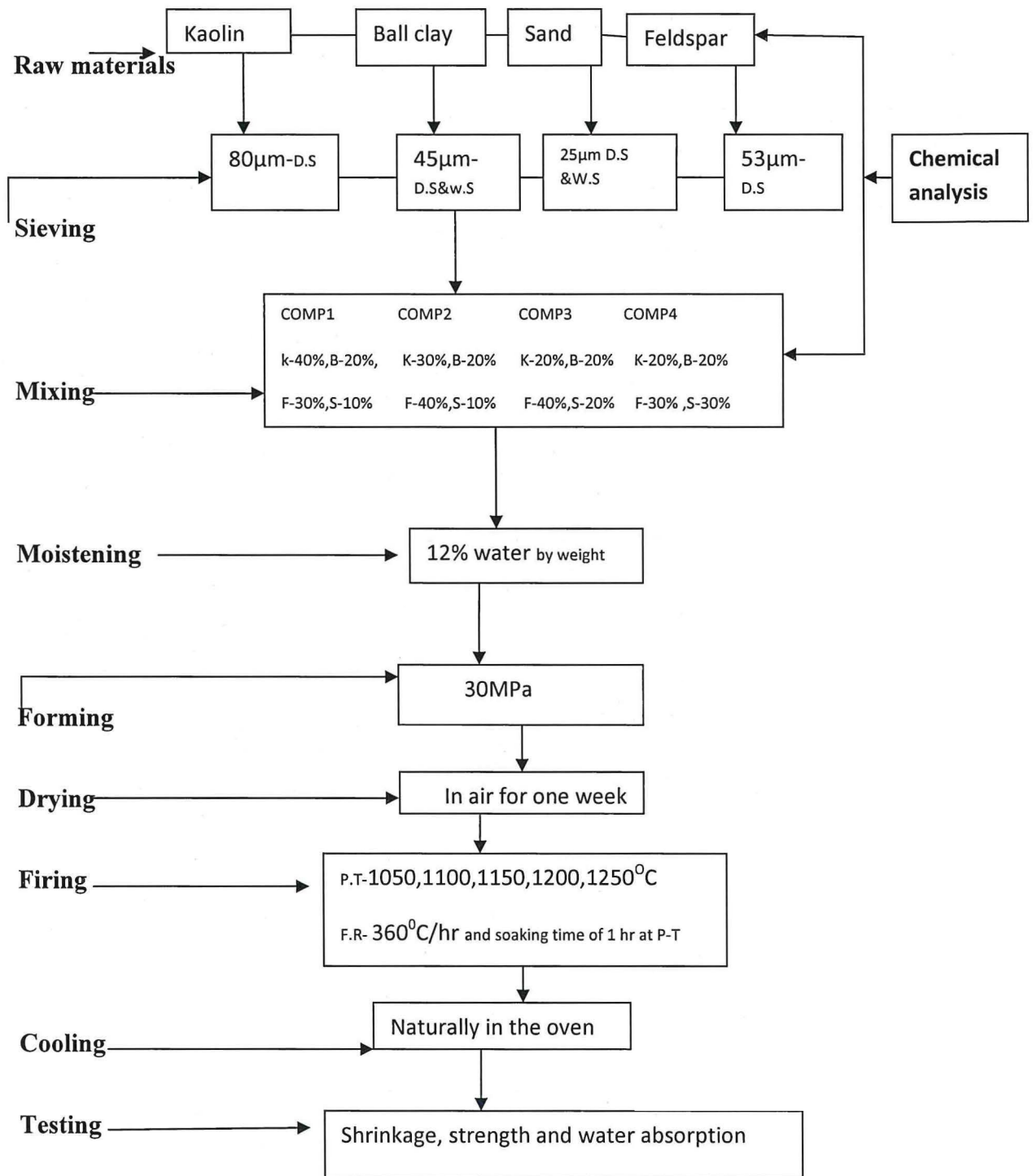
CHAPTER THREE: METHODOLOGY

3.1 Research Design

This research was concerned with quantifying the relationship between dependent and independent variables of ceramic floor tiles produced using local minerals. The dependent variables were experimentally measured while the independent variables were varied. Dependent variables included shrinkage, strength and water absorption while the independent variables were temperature of tile firing and composition of minerals.

Four compositions of minerals were formulated as shown in Table 3.1. This was done to find out the effect of composition on the properties of tile samples. Peak firing temperatures were set at 1050, 1100, 1150, 1200 and 1250°C. Measurement of dependent variables was taken for samples that were fired to these temperatures. Forming pressure for the different samples was kept at 30MPa for particles sizes of 80µm kaolin, 53µm feldspar, 45µm ball clay and 25µm sand.

The entire research was based on the experimental layout shown in Figure 3.1. To the top, the raw materials, particle sizes and compositions of formulated samples is found. The middle indicates the forming pressure, water content and the drying process of the green body. Finally at the bottom, is the firing schedule, cooling process and the major properties of interest in the fired product.



D.S-dry sieving, w.s-wet sieving, K-kaolin, B-ball clay, F-feldspar, S-sand, P.T-peak temperature, F.R-firing rate

Figure 3.1: Experimental Layout

3.2 Preparation of Raw Materials

The materials used in the production of the tiles were kaolin and feldspar from Mutaka deposit in Bushenyi district, ball clay from Ntawo deposit in Mukono district and sand from Lido beach in Wakiso district. The materials were milled using a ball mill and sieved to the required particle size ranges. This was done to obtain particle sizes of 80, 53, 45 and 25 μm for kaolin, feldspar, ball clay and sand respectively.

3.2.1 Ball Clay

Ball clay from Mukono was dug in its raw form, impurities such as stones, grass, roots, and metallic bodies were removed by hands. The clay was then broken down into smaller pieces and put in a pail containing water. It was soaked in a large volume of water for five days to allow heavier particles such as sand and other metallic objects to settle at the bottom of the pail. The floating particles were then drawn off as a liquid of clay and water. The clay was wet sieved using 200, 100, 60 and finally 45 μm sieve mesh. Using sieves of different sizes was meant to avoid blockage of their holes. The 45 μm liquid clay was then poured onto a plaster of paris mould and left for a week so as to suck away water. It was then put in an oven at a temperature of 105⁰C for four hours to dry. The dry clay was crushed using a mortar and pestle and put in a ball mill for one hour. The milled clay was then sieved to form power of 45 μm .

3.2.2 Kaolin

Kaolin after mining was dried under sun heat for four days. The foreign bodies such as stones, grass, roots and metallic objects were removed by hand from the dried kaolin. Ball-milling was not necessary because it had small particles, however it was dry sieved

using mesh sizes of 300, 100 and then 80 μm . The powder was put in buckets and covered to avoid contamination.

3.2.3 Feldspar

Feldspar was dry milled for five days using a ball mill running for ten hours each day at a speed of 50 rev/min. Porcelain milling stones got from lakes were used as the milling medium. After milling, the materials which were crushed to much smaller particle sizes were put in plastic buckets. The milled material was sieved through 200 μm , 100 μm and then 53 μm to form white powder. The ball mill and milling stones were cleaned with water before milling different minerals such as sand.

3.2.4 Sand

Sand was wet milled using a ball mill for five days running for ten hours each day at a speed of 50 rev/min using porcelain milling stone from the lake as a milling medium. It was then wet sieved through 100, 53 and finally 25 μm . The wet sand was then poured onto the plaster of paris mould and left for four days. The semi-dry material from the plaster of paris mould was later dried in the sun. The dried material was subsequently sieved to 25 μm to form powder. The powder samples were then stored in plastic buckets and covered to avoid dust and moisture contamination.

3.3 Composition Ratios of Raw Materials in the Samples

Four composition ratios of ball clay, kaolin, feldspar and sand were formulated. The percentage by weight for each composition is shown in Table 3.1

Table3.1: Composition Ratios of Formulated Tile Samples (wt %).

Raw materials	Composition of samples (%)			
	1	2	3	4
Ball clay	20	20	20	20
Kaolin	40	30	20	20
Feldspar	30	40	40	30
Sand	10	10	20	30
Total	100	100	100	100

3.4 Chemical Analysis of Minerals

The chemical analysis was determined using XRF machine (Philips X'Unique II).

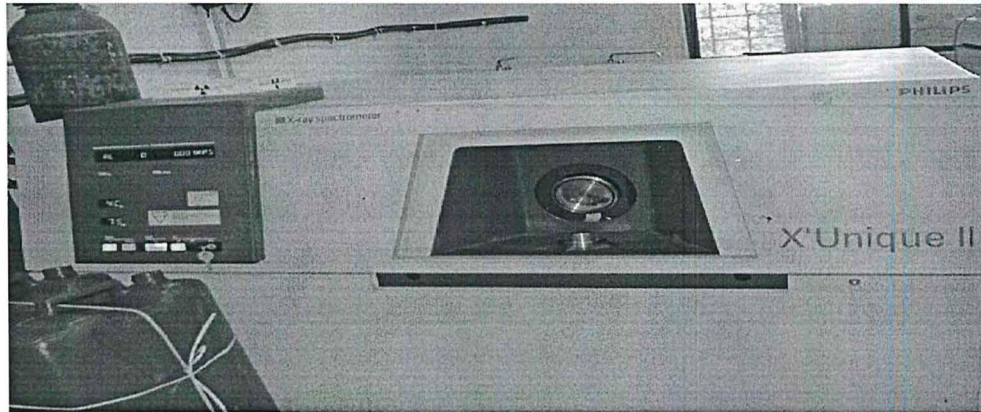


Figure 3.2: XRF machine used to analyze Chemical Compositions

The raw materials were sieved to powder of particles size 25, 45, 53 and 80 μm for sand, ball clay, feldspar and kaolin respectively. The powder for each mineral was

made into pellets and then inserted into the machine. The XRF machine was then calibrated to detect and record to percentage the oxide of interest in the raw materials

3.5 Mixing of Raw Materials

Mixing of the raw materials was done in a mortar mixer shown in Figure 3.3. The mixer is made of a cylindrical can with a rotating plate at its centre. Powdered materials were poured into the cylindrical can and the mixer was switched on. The plate rotating at a constant fixed speed was to blend the materials. Mixing of the materials was done in two phases. Phase one had dry powder mixed for ten minutes while in phase two 12% water by weight was added to the powder and mixed for another ten minutes. For each composition 500 grams were measured for mixing. This was done to avoid spillage of materials due to the size of the cylindrical can, spillage occurred while using 800 grams. The aim of mixing was to blend particles of different sizes as shown in Figure 2.1, so as to attain a dense and strong body after firing.

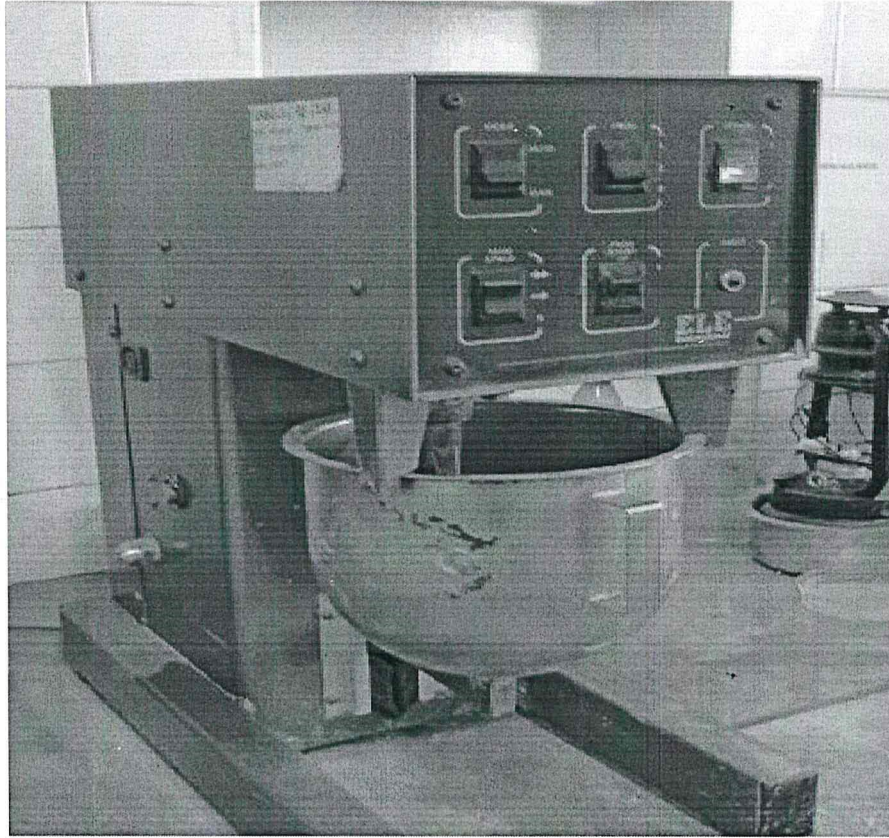


Figure 3.3: Picture of the Automatic Mixer.

3.6 Production of Sample Tiles

Fifty specimen tiles of 106mm x 50mm x 9.6mm of the green body were produced from each compositions indicated in Table 3.1. This was done by pressing compositions containing a moisture content of 12% by weight at a pressure of 30MPa as per Figure 3.4 and Figure 3.5. The pressed specimens were subsequently left to dry in open air for one week. Firing was then done in a kiln at sintering temperatures of 1050, 1100, 1150, 1200 and 1250⁰C.

3.6.1 Forming

Semi-dry pressing was applied with a water content of 12% by weight. A mass of 110g of the mixture was weighed and poured into a rectangular steel mould of dimensions 110mmx50mm x 10mm. The mould was then placed on a jigger table for two minutes to level the content as shown in Figure 3.6. The mould was transferred to a compact compression machine shown in Figure 3.5 where a force of 165kN was applied on the body, this is equivalent to a pressure of 30Mpa. The sample was carefully removed from the mould to avoid breaking and left to dry in air for one week. The mould was cleaned before formulating another sample, this was done to avoid sticking of the green body on the sides of the mould.

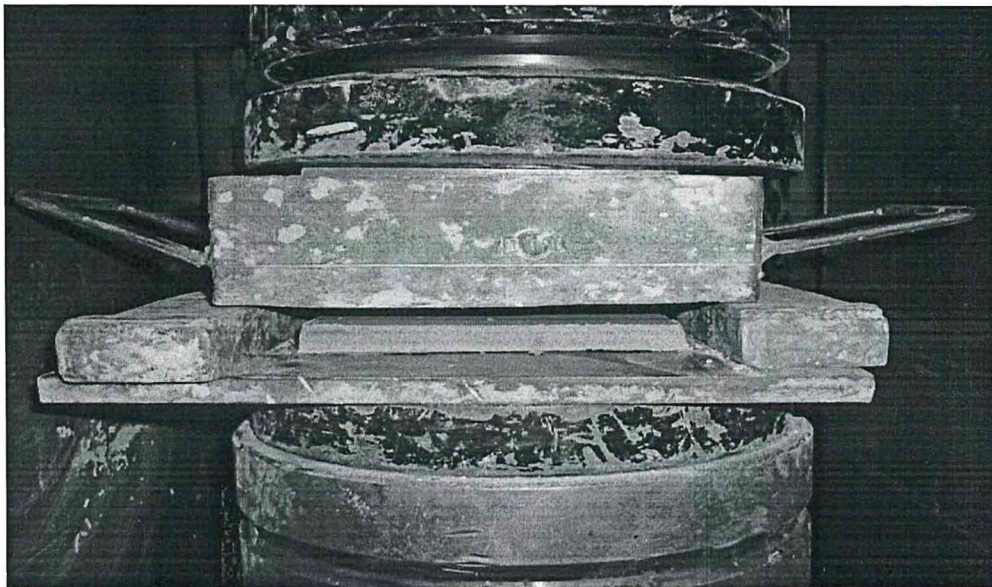


Figure 3.4: Picture of a Green Body (tile) after Ejection from the Mould



Figure 3.5: Picture of the Forming Process using a Compact Compression Machine.

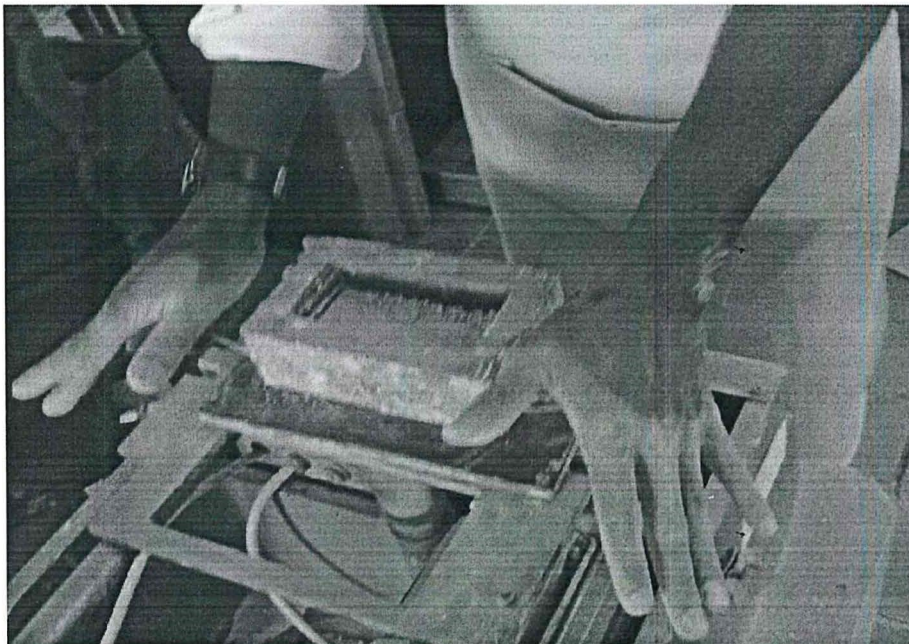


Figure 3.6: Picture of a Body on a Jigger Table ready for Leveling.

3.6.2 Firing

Firing was aimed at improving densification of the body in order to form a strong sample. Firing was done in a kiln as shown in Figure 3.7 at peak temperatures of 1050, 1100, 1150, 1200 and 1250⁰C. The dry samples were heated from room temperature to 105⁰C at a heating rate of 100⁰C per hour and holding at 105⁰C for 2 hours, this was done to drive away the moisture. After 105⁰C, the heating rate was increased to 360⁰C per hour to peak temperature and soaked for 1hour before switching off the firing kiln. The body was left to cool in the kiln for 16 hours before removing. For each sample composition shown in Table 3.1, ten specimens were fired at each peak temperature.

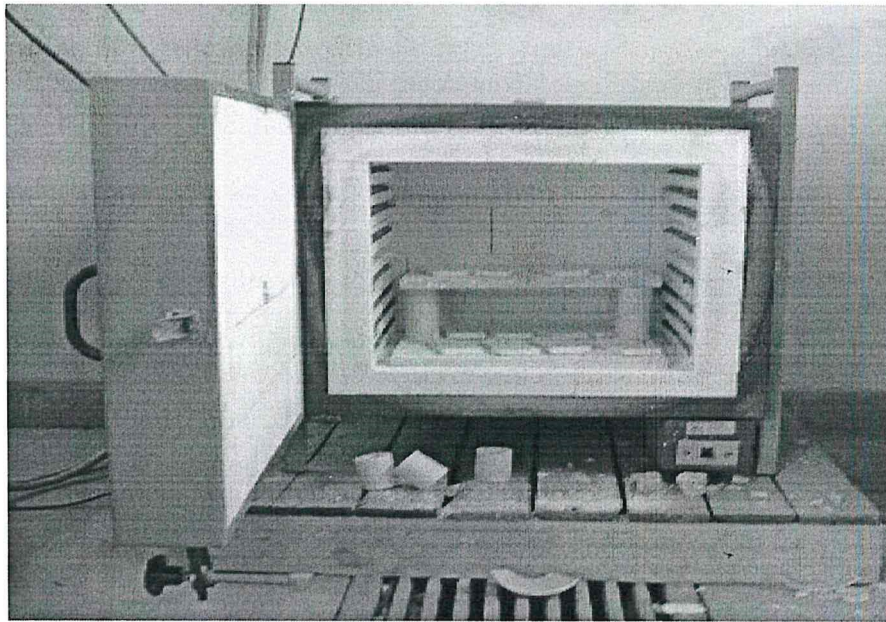


Figure 3.7: Dry Samples in an Automatic Electronic Kiln ready for Firing.

3.7 Experimental Measurements

The measurement of properties of the tile samples was done at the Uganda National Bureau of Standards and at Ministry of Works and Transport-Kireka laboratory. The properties measured included linear shrinkage, strength and water absorption

3.7.1 Linear Shrinkage

The dimensional measurements taken were thickness (t), breadth (b) and length (L). Five samples of the green body were measured, with each dimension measured three times and the average recorded. For each composition fired at a particular peak temperature, five samples were used with each dimension measured three times and their average recorded. A vernier caliper was used to measure dimensions of specimen. Percentage linear shrinkage by thickness, breadth and length was calculated using formulae in equations 3.1 to 3.3.

$$\% \text{ shrinkage by thickness} = \frac{t_d - t_f}{t_d} \times 100\% \quad (3.1)$$

Where t_d, t_f are thicknesses of green and fired samples respectively.

$$\% \text{ shrinkage by breadth} = \frac{b_d - b_f}{b_d} \times 100\% \quad (3.2)$$

Where b_d, b_f is breadth of green and fired samples respectively.

$$\% \text{ shrinkage by length} = \frac{l_d - l_f}{l_d} \times 100\% \quad (3.3)$$

Where l_d, l_f is length of green and fired samples respectively.

The average of the three shrinkage values in the expression in equation 3.1 to 3.3 was taken as the final linear shrinkage of the tile.

3.7.2 Measurement of Modulus Of Rupture (MOR)

Three point loading test was done. The machine used was Multiflex Electronic loader Figure 3.8. It was adjusted by moving two lower cylindrical rods so that they are of equal length from both sides of the lower plate with dimensions. Three points were marked on each sample where the cylindrical rods were placed. The span distance, L , between two lower cylindrical rods was adjusted to 70mm. The loading force was applied at a rate of 2mm/min, it was directed to the centre of the fired sample. The force was applied until the sample was broken, the proving ring reading on the scale at this point was recorded. After each reading the scale was re-set to zero before another measurement was taken. The proving ring reading was multiplied to a constant 0.0043 to obtain loading force, F , in kN. The modulus of rupture was calculated from the equation 3.4.

$$MOR = \frac{3 FL}{2 bt^2} \quad (3.4)$$

Where F is loading force, b is the breadth and t is the thickness of the sample. At every peak temperature, six tiles were tested and their average used to obtain the overall MOR.

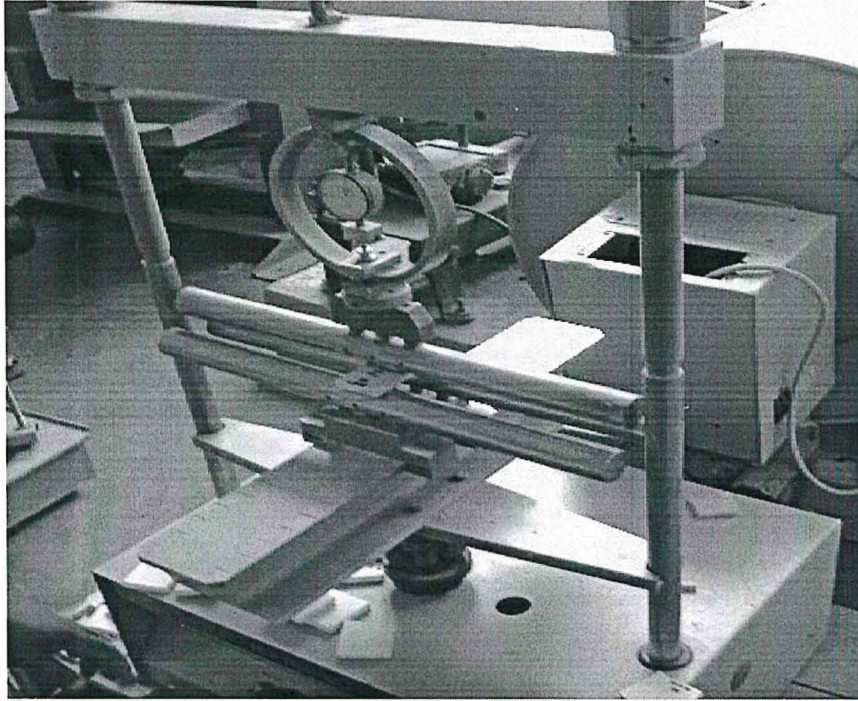


Figure 3.8: Picture Showing Strength Test using a Three Point Loading Machine

3.7.3 Measurement of Water Absorption

The samples were immersed in a water bath, with no contact between them. The water was heated to a temperature of 92°C for two hours, the temperature of the water was determined using a thermometer. After two hours the heating was stopped and the specimens were left soaked in water for 24hrs. The mass, m_1 , of the dry body and m_2 , of the soaked samples were then determined using an electronic balance. The percentage water absorption was evaluated using equation 3.5.

$$\%W.A = \frac{m_2 - m_1}{m_1} \times 100 \quad (3.5)$$

At every peak temperature the average mass of dry and soaked samples was calculated from a total of five specimens.

CHAPTER FOUR: RESULTS AND DISCUSSIONS

4.1 Introduction

This chapter covers results and discussions obtained from chemical analysis and measured quantities such as shrinkage, MOR and water absorption. These results are expressed as graphs of measured quantities against temperature. Features of graphs are described at various peak temperatures. Effects of firing temperatures and compositions on quantities have been discussed.

4.2 Results of Chemical Composition of Minerals

The chemical composition of SiO_2 , Al_2O_3 and Fe_2O_3 in the raw materials used is shown in Table 4.1.

Table 4.1: Chemical composition of the minerals used in the present study

Mineral	Al_2O_3 (wt%)	SiO_2 (wt%)	Fe_2O_3 (wt%)	Total (wt%)
Kaolin	24.9	40.3	0.11	65.31
Ball clay	15.4	57.8	3.20	76.40
Feldspar	13.2	52.8	0.14	66.14
Sand	0.391	95.4	0.22	96.01

The raw materials used in the present investigation were kaolin, ball clay, feldspar and sand. Representative samples were collected from the deposits and processed for use in

the study. Alumina and silica content was analyzed because of their influence in strength of porcelains. Furthermore, the color of fired samples is due to the presence of iron oxide in ball clay. The overall percentage of oxides in Table 4.1 is less than 100%. This is because other elements (oxides) as seen in Table 2.1 were not considered for tests simply because they are of less influence to the mechanical properties of the fired samples.

4.3 Discussion of Chemical Composition of Minerals

Chemical analysis revealed that kaolin had the highest alumina content and sand had the least as shown in Table 4.1. On the other hand sand had the highest silica content and kaolin had the least. This is in agreement with earlier studies carried out by Olupot (2010) and Kirabira (2005). However, difference in percentage composition (wt%) of alumina and silica in the present study to earlier studies as seen in Tables 4.1 and 2.1 respectively is due to difference in the exact location of the mines where the minerals were obtained. The chemical composition of iron oxide in the present study was higher than that recorded by Olupot (2010). This is because samples in earlier studies by Olupot and Kirabira were passed through a magnet hence reducing the iron content as seen in Table 2.1.

In the present study only the chemical composition of alumina, silica and iron oxide was determined. This is because both alumina and silica have a high influence on strength of porcelains (Kaahwa 1999, Obwoya 2004 and Senapati et. al 1998). According to Weems (1988) and BGS (2011) iron oxide contributes significantly to the color of both fired and unfired ceramic samples. Iron oxide combines with alumina and silica to form greyish samples at high temperature

4.4 Results of Shrinkage

The linear shrinkage of the formulated compositions at different firing temperatures is shown in Figure 4.1.

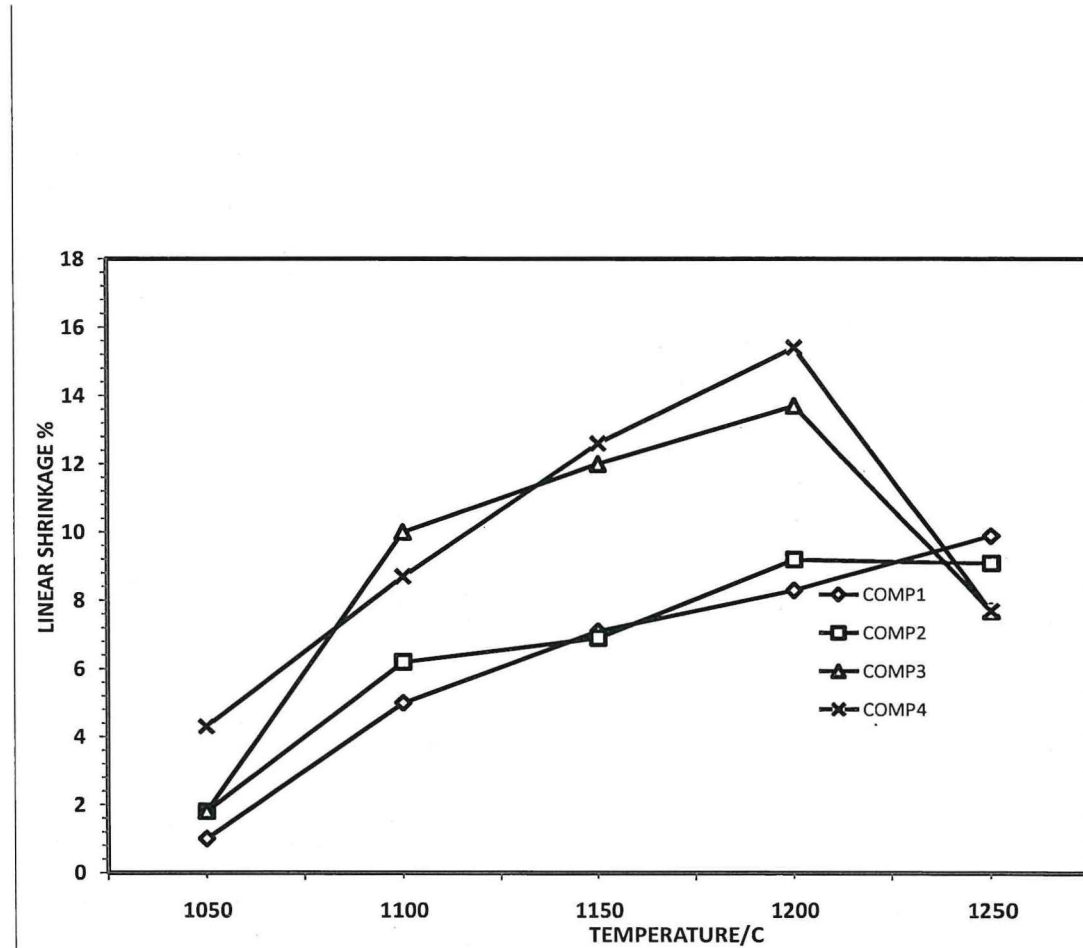


Figure 4.1: Linear Shrinkage (%) of the Studied Tiles

All samples exhibited increasing linear shrinkage as temperature increased. Compositions 3 and 4 exhibited the highest shrinkage at 1200°C, for same samples firing at 1250°C resulted in a decrease in shrinkage. The shrinkage experienced at 1200°C for compositions 4 of about 16% is the highest for all the samples. Still from Figure 4.1 composition 3 and 4 experienced greater shrinkage compared to 1 and 2 for temperature

range of 1050 to 1200⁰C with composition 1 experiencing least shrinkage. Compositions 1 and 2 on the other hand exhibited progressive increase in linear

4.5 Discussion of Shrinkage

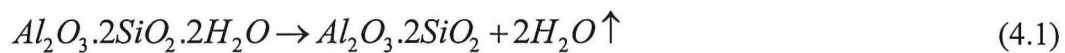
Firing samples containing moist clay eliminates moisture at different temperatures. The removal of moisture creates voids within the specimen and on cooling there is shrinkage. According to Rayan (1978), at a temperature of 100⁰C moisture left in the samples after drying in open air is driven off. Between 400 to 700⁰C, chemically held water and other organic compounds are eliminated. Escape of moisture and other organic compounds causes contraction of the spaces created within the specimen on cooling. Partial fusion of finer particles occurs at 900⁰C, fused substances formed fills up the voids created hence reducing the number of pores hence causing shrinkage.

Low shrinkage for all the compositions at 1050⁰C as shown in Figure 4.1 is due to low rate of particles fusion, moisture escape and glassy phase. Glassy phase results from firing samples containing feldspar (Kirabira, 2005). At 1050⁰C samples experienced less glassy surfaces compared to those at 1200 to 1250⁰C as shown in Figure 4.2. Glassy matrix acts to densify the body and reduce porosity hence increasing shrinkage of the fired specimen (Obwoya, 2004).

Compositions 1 and 2 exhibited high shrinkage at 1200-1250⁰C as seen in Figure 4.1. However, compositions 3 and 4 exhibited low shrinkage at the same temperature range, this was due to the amount of sand in the samples as seen in Table 3.1. Hutchings and Xu. et. al. (2006), reported that sand increases porosity which results from cracks within fired specimen hence decreasing shrinkage of the body.

In Triaxial ceramic bodies, high sand content is reported to result in reduced shrinkage (Olupot, 2010). While on the other hand, high kaolinitic content due to high clay content leads to an increase in shrinkage due to the dehydroxylation process of metakaolin formation (Senapati and Cathy. et. al. 1998). In this work, although compositions 3 and 4 had high sand content, their shrinkage at firing temperatures of 1050-1150⁰C were higher than compositions 1 and 2 having low sand content. These trends are contrary to published information highlighted above. The study was limited by experimental equipments, to investigate what could be causing these anomalies especially in form of the resultant phases and microstructures formed.

Compositions 1 and 2 exhibited increasing shrinkage with temperature as shown in Figure 4.1, this is attributed to high clay content as shown in Table 3.1. Clay contains moisture in form of hydroxyl group, firing leads to removal of moisture hence causing an increase in shrinkage according to equation 4.1. Cristina and Federica. et .al (2001), reported that high kaolinitic clay in the samples increases shrinkage of the body.



At 1250⁰C the effect of sand content is pronounced. Samples with higher sand content exhibit low shrinkage values compared to samples with high clay content as shown in Figure 4.1. Sand increases the porosity of the fired specimen due to formation of cracks, this lowers shrinkage of the body (Olupot, 2010 and Hutchings. et .al 2006).

Linear shrinkage of 5-10% was obtained for compositions 1 and 2 for samples fired at temperatures of 1100 to 1250⁰C. This is close to results from similar studies on porcelain stoneware tiles by Cristina and Federica. et. al (2001) of 5-8% at temperatures of 1160 to

1220⁰C. Both values of shrinkage do not deviate much from the commercially recognized values of 5-7.5% (South African National Standards, 2008). In the present work, shrinkage of compositions 3 and 4 of 9-15% for firing temperatures of 1100 to 1200⁰C is not close to the commercially recognized values of 5-7.5%. At 1250⁰C both compositions experienced a decrease in shrinkage to about 8% which is close to values recorded by Cristina and Federica. et. al. (2001) and SANS (2008). In the present work, the strength and water absorption of samples which recorded shrinkage of 5 to 14% is recommended for flooring by SANS (2008). High shrinkage signifies improved densification leading to high strength and low water absorption.

4.6 Results on Appearance of Fired Samples

The color of samples changed from white to brown and then to grayish with temperature as shown in Figure 4.2



Figure 4.2: Samples fired at different temperatures

All compositions appeared to have similar colors at same peak temperature, samples had a smooth surface and edge finish with those fired at 1150 to 1250⁰C being more smooth

and glassy. Surface cracking was not observed for all the samples. However, warping occurred for most of the samples fired at 1050⁰C and a few at 1100⁰C especially those placed at the lower part of the kiln. Samples fired at 1150 to 1250⁰C as per Figure 4.3 did not warp irrespective of their position in the kiln during firing.

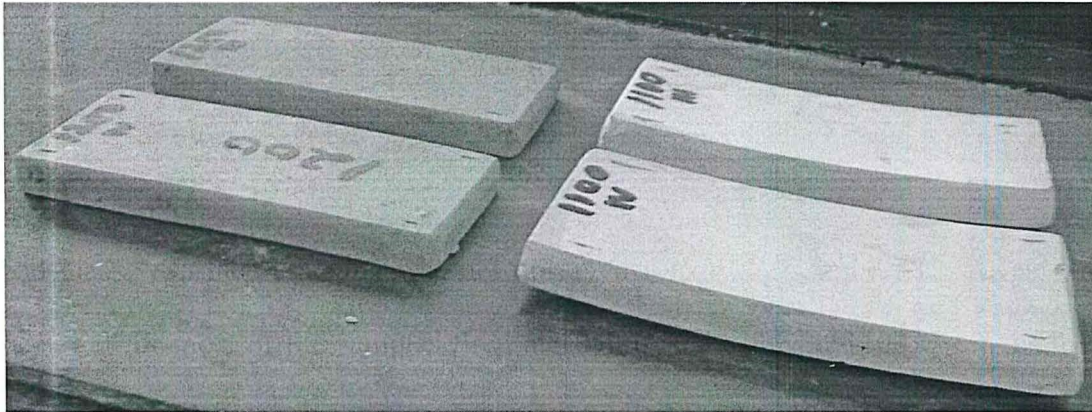


Figure 4.3: Warped Samples (right) and Unwarped Samples (left).

4.6.1 Effects of Firing on Shape

Warping occurred for some samples fired at 1100⁰C and below. This happened mostly for samples placed at the lower part of the kiln during firing. However, all samples fired above 1150⁰C did not warp irrespective of their positioning in the kiln. Warping was caused by temperature gradient between the top and bottom part of the specimen. Samples fired above 1150⁰C did not warp because at very high temperatures there is negligible temperature gradient at different parts of the specimen. Kingery (1976), reported that besides temperature gradient, warping is also caused by density variation in the green body during forming. The effect of density variation was minimized by applying uniform pressure on the powder using a jigger table, steel mould with a die as shown in Fig 3.4 to 3.6.

4.6.2 Effect of Firing on Surface Finish

Surface cracking was not observed for all samples, edges were smooth as shown in Figure 4.2. Cracking is usually caused by using much sand of bigger particle sizes. According to Yaseen (2000), larger quartz grains of about 50-150 μm exhibit continuous peripheral fracture near the grain boundaries hence causing surface cracking. Sand particles between 25-50 μm exhibits less-severe peripheral fracture around the sand grain, this creates small cracks within the specimen. Stathis. et. al (2004), reported that both internal and external cracking is due to stress development and thermal expansion mismatch between the glassy matrix and sand particles during cooling. In the present work, the presence or absence of internal cracks could not be confirmed due to lack of equipment such as Scanning Electron Microscope (SEM).

4.6.3 Effect of Firing on Color

The green body formed was pale grey after drying in open air. This is due to the presence of clay in the samples. The presence of impurities such as iron oxide, magnesia, titania, potassium oxide and calcium oxide among others affect the color of both fired and unfired clay samples. In the present work, samples fired at 1050 $^{\circ}\text{C}$ turned white, those between 1100 to 1150 $^{\circ}\text{C}$ became brown and finally samples fired beyond 1200 $^{\circ}\text{C}$ appeared grey as seen in Figure 4.2. British Geological Survey (2011), reported that the presence of iron oxide in ball clay constitutes the color of the fired samples. Fired bodies made of clay turn orange, red, brown, grey or white (<http://www.pottery.com>). According to Weems (1988), the iron oxide present in clay as seen in Table 4.1 will produce certain

definite colors dependent on the amount of the oxide present . When iron combines with silica and alumina at high temperature, the color becomes greyish or even black.

Similarity in colors for all compositions at each peak temperature is attributed to the amount of ball clay of 20% for all compositions as shown in Table 3.1. Senapati and Cathy (1998), reported that besides providing plasticity and green strength during the forming stages, ball clay also contributes substantially to the color of the fired ware. According to Olupot (2006), if whiteness of the ware is required not more than 15% ball clay should be added. Therefore addition of 20% ball clay in the samples caused different colors of samples at different temperatures.

4.7 Statistical Analysis

Data analysis and interpretation were performed by using Analysis of variance (ANOVA) show in Table 4.2. This was done to find out whether there was any significant effect of temperature on shrinkage, strength and water absorption. ANOVA and F- statistics was used because samples were subjected to different (means) peak temperatures. Finding out the variation of temperature at different peak points with shrinkage, strength and water absorption was necessary.

Table 4.2: ANOVA Table (Source: Montgomery, 2005).

Source of variation	Sum of squares	Degree of freedom	Mean square	F ₀ -value
Temperature	$SS_{Temp} = \frac{1}{n} \sum_i y_i^2 - \frac{y^2}{N}$	$a-1$	$\frac{SS_{Temp}}{a-1}$	$F_O = \left[\frac{SS_{Temp}}{a-1} \right] \left[\frac{N-a}{SS_E} \right]$
Error	$SS_E = SS_T - SS_{Temp}$	$N-a$	$\frac{SS_E}{N-a}$	
Total	$SS_T = \sum_i \sum_j \left(y^2_{ij} - \frac{y^2}{N} \right)$	$N-1$		

Where SS_{Temp} is the sum square of temperature, SS_E is the sum square of errors due to temperature and SS_T is the sum square of the total observations. y_i, y_{ij}, y are observations at a fixed temperature (row), different temperatures (row and column) and total respectively. Also a , is the number of peak temperatures used therefore $(a-1)$ is its corresponding degree of freedom. N and n are total number of observations and compositions respectively. The test statistic F_0 for the hypothesis also known as the F-distribution is given by

$$F_O = \left[\frac{SS_{Temp}}{a-1} \right] \left[\frac{N-a}{SS_E} \right] \quad (4.2)$$

According to Montgomery (2005), if $F_O > F_{\alpha, a-1, N-a}$, then there is a significant effect of temperature on the shrinkage of samples, hence the null hypothesis is rejected.

In the present work the null hypothesis was set as $H_0 : < 3.06$. There is no variation of temperature with shrinkage if the null hypothesis is true. Analysis of shrinkage of experimental results using ANOVA and F- distribution test as shown in Table 4.3 and Appendix table 13 and 14 resulted into $F_o = 61.3$ and $F_{0.05,4,15} = 3.06$ at 0.05 significance levels.

Table: 4.3 ANOVA for Shrinkage

Source of variation	Sum of squares	Degree of freedom	Mean square	F_o
Temperature	1439.6	4	360	61
Error	88	15	5.9	
Total	1527.6			

Since F_o is greater than $F_{0.05,4,15}$ then there was a significant effect of temperature on the shrinkage of the samples and the null hypothesis is rejected.

4.8 Results of Modulus of Rupture (MOR)

The strength of samples at various peak temperatures is shown in Figure 4.4. with a maximum value of about 34MPa which happens to be the highest for all the samples.

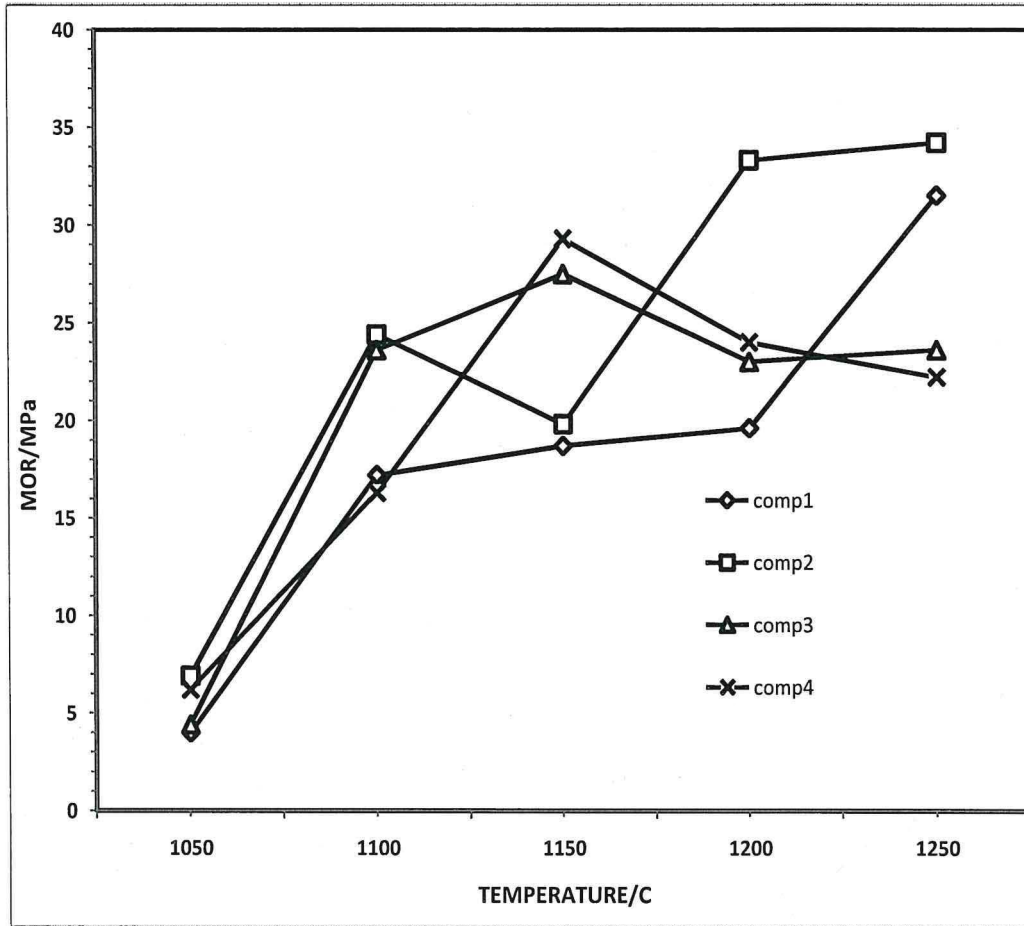


Figure 4.4: MOR of the Sample Tiles at different Temperature

Compositions 3 and 4 demonstrate progressive increase in strength up to a maximum point of nearly 29MPa at 1150⁰C. For the same compositions, further increase in temperature resulted into a decrease in strength with a sharp decline between 1150 to 1200⁰C thereafter a slight alteration in strength was recorded at 1250⁰C, with composition 4 recording the lowest value for all the samples of nearly 22MPa at 1250⁰C. Composition 1 also presented a general increase in strength with temperature, with a slight variation experienced between 1100 to 1200⁰C. Increasing its temperature further resulted into a sharp increase in strength up to a maximum value of about 32MPa at 1250⁰C. Figure 4.3 shows decrease in strength for composition 2 at 1150⁰C to a value of

around 19MPa. However, further increase in temperature for the same composition resulted into a sharp increase in strength. The same composition experienced a slight adjustment in strength at temperatures of 1200 to 1250⁰C. Both compositions 1 and 2 exhibited high values of strength at 1250⁰C with composition 2 recording a maximum

4.9 Discussion of Modulus Of Rupture (MOR)

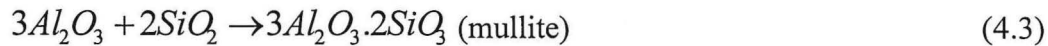
The strength of porcelains stoneware depends on firing temperature, forming pressure and particle size. These factors determine the evolution of microstructure and phase development in porcelains (Branganca and Bargman. et. al 2006). Development of mullite needles due to temperature rise increases the strength of porcelain ware according to Olupot (2010). The use of high forming pressure on fine particles results in decrease of pores in fired samples hence increase in strength.

Senapati and Cathy (1998) and Stathis and Ekonomaku. et. al. (2004), reported that the strength of porcelains is explained basing on three hypotheses. These include mullite hypothesis, matrix reinforcement hypothesis also known as pre-stressing effects of porcelains and dispersion-strengthening hypothesis. These hypotheses are explained basing on microstructure evolution and phase development.

The compositions investigated in this work are bound to produce mullite upon firing at temperatures of 1050, 1100, 1150, 1200 and 1250⁰C. Mullite formation starts at 980⁰C for potash feldspar and 1100⁰C for sodium feldspar (Senapati and Cathy, 1998). The amount of mullite formed increases with temperature. According to the mullite hypothesis, this is responsible for increased strength at high firing temperatures as generally observed in the trends in Figure 4.4. At high temperatures mullite needles split

leading to increases population of finer needles. Fine needles interlock more effectively than large one hence resulting in increase in strength (Stathis and Ekonomaku.et. al.2004).

Clay and feldspar are the major sources of alumina and silica as seen in Tables 2.1 and 4.1. Figure 4.4 shows progressive increase in strength for compositions 1 and 2 with high clay and feldspar content as seen in Table 3.1. The same figure shows overall maximum value of strength of about 34MPa for composition 2. Martin and Ma-Rinco. et. al (2007), reported that the overall bending strength of porcelain stoneware tiles fired at 1260-1280 is 34 ± 2 MPa. According to Kaahwa 1999, the concentration of alumina in the samples increases its strength due to mullite formation. Alumina combines with silica under the influence of heat to form mullite according to equation 4.3 (Obwoya, 2004).



Mullite formation occurs twice in both clay and feldspar minerals. Yassen (2000), reported that primary mullite is formed from clay at low temperature. At high temperatures primary mullite acts as a seed in the formation of secondary mullite from feldspar. Formation of secondary mullite yields strong porcelain ware compared to primary mullite. In the present work, compositions 1 and 2 exhibited high strength at temperatures of 1200 to 1250⁰C this is attributed to formation of secondary mullite from high alumina content in both clay and feldspar minerals as seen in Table 4.1.

Porcelains experience a difference in thermal expansion coefficients between the glassy matrix and dispersed particles such as sand. This difference produces strong compressive stresses on the glassy matrix. Such induced compressive stresses leads to an improvement

in the strength of ceramics according to the matrix re-enforcement hypothesis also known as pre-stressing effect (Stathis and Ekonomaku.et.al, 2004). The pre-stressing effect is greater in samples with high sand content. Figure 4.4 shows high strength for composition 3 and 4 at temperatures of 1050-1150⁰C. This is due to increased pre-stressing effect between sand particles and the glassy matrix.

Excessive amount of sand in porcelains creates a possibility of transformation into cristobalite (Senapati and Cathy, 1998). Using sand particle size of 10-30 μ m has a beneficial influence in strength of porcelain. Sand particle size of 25 μ m was used in the present work and for composition 3 and 4 sand content was 20% and 30% by weight respectively. Cristina and Federica. et. al (2001), recommended a sand content of 10% by weight in porcelains which is constituted in composition 1 and 2 as seen in Table 3.1. Olupot and Byaruhanga et. al. (2006), reported sand converts to cristobalite in a reconstructive phase transformation at temperatures above 1100⁰C. This transformation takes place as a results of bond-breakage in the structure. Figure 4.4 shows high values of strength for composition 3 and 4 compared to 1 and 2 at 1100-1150⁰C, this is attributed to formation of cristobalite. The presence of cristobalite instead of sand in porcelains often produces an improvement in strength.

The strength of cristobalite porcelain bodies is explained basing on three perspectives.(a) Cristobalite grains tend to be finer than sand particles, fine grains form strong bodies as reported by Obwoya 2004. (b) Cristobalite is formed at the expense of sand, decreasing sand content improves on the strength of ceramics according to Hutching and Xu. et. al (2006) Cristobalite inversion temperature is 225-250⁰C compared 573⁰C for sand. This reduces strain development during cooling hence forming fewer cracks.

At temperatures of 1050-1150⁰C most sand particles do not dissolve into the glassy phase. This limits the size of Griffith flaws created within porcelains hence increasing the strength of the body as seen in Figure 4.4 for compositions 3 and 4 at the same temperature. Flaws reduce the strength of porcelain because when a mechanical load is applied on a porcelain body, stress adjacent to the flaws increases. The high stress pulls the atoms apart hence causing the body to rupture easily (Anura, 1994). Senapati and Cathy (1998), reported that dispersed particles such as sand in a glassy matrix limits the size of Griffith flaws. This increases the strength of porcelains according to the dispersion strengthening hypothesis.

Figure 4.4 further shows decrease in strength for compositions 3 and 4 at temperatures of 1200 to 1250⁰C. This is attributed to dissolution of sand into the glassy phase. The dissolution of sand leads to formation of larger flaws as explained by the dispersion strengthening hypothesis. Olupot (2010), reported that at temperatures of 1200⁰C there is dissolution of sand into the glassy phase without increase in the mullite content. This causes a decrease in the strength of the body.

Table 3.1 shows high sand content for compositions 3 and 4 compared to 1 and 2. The strength of compositions 3 and 4 at temperatures of 1200 to 1250⁰C reduced as seen in Figure 4.4, this is attributed to high sand. According to Hutchings and Xu. et. al (2006), sand increases porosity of the body by forming cracks. Samples containing sand on cooling develops pyroplastic deformation and relaxation within the glass phase which prevents the development of residual stresses until the glass transition temperature is reached. Cooling of the samples below the glass transition temperature creates stresses due to thermal expansion mismatch between glass and sand particles. As sand cools

below its inversion temperature of 573⁰C, its particles experiences a volume decrease of about 2% which is enough to produce sufficient strain to cause cracking of the glassy matrix (Senapati and Cathy, 1998). Cracking of the glassy matrix creates weaknesses in the body hence reduction in its strength.

Yaseen and William (2000), further suggested that cracks are experienced in a temperature range of 900-800⁰C for porcelain when cooling from 1200⁰C. In the present work, the strength of samples fired at 1200⁰C to 1250⁰C for compositions 3 and 4 declined as illustrated in Figure 4.4, this is due to many cracks developed within samples after cooling.

Low strength at a temperature of 1050⁰C of approximately 4MPa and 7MPa for compositions (1,2) and (3,4) respectively is attributed to low levels of sintering. At lower temperatures the number of pores in the body is high, this is seen in Figure 4.5 with high water absorption at this point. Increasing temperature reduces the porosity of the samples hence increase in strength. Ashby (1986), reported that high temperature melts fine particles, the liquid formed fill up the voids created hence lowering porosity and increasing strength of the samples Pores reduce the crosssectional area on which the load acts and are also stress concentrators (Kingery, 1976). The pressure exerted on the body is high causing it to rupture easily as per equation 4.2.

$$\text{Applied pressure} = \frac{\text{load}}{\text{area}} \quad (4.4)$$

Samples experience voids called bloats due to less supply of oxygen as a result of poor packing in a firing furnace. When a body is being fired and the gasses formed within

cannot escape, expansion takes place which creates voids (Obwoya, 2004). The creation of voids causes a decrease in strength of the samples as seen in Figure 4.4 for composition 2 at a temperature of 1150⁰C.

Graded materials are stronger due to improved particle compatibility after forming. Porosity of the fired samples is therefore low hence increasing strength. The present work had four particle sizes of 25, 45, 53 and 80µm for sand, ball clay, feldspar and kaolin respectively for grading. This was done to improve strength by reducing porosity of the pressed samples through proper particle mixing. According to Grimshaw, (1971), using equally sized particles creates about 38% voids within the samples. If grading is done only 5.4% of voids are created. Maximum strength is achieved if only three particle sizes are used. The material should have 10% fine, 25% medium and 65% coarse particles. Using four particle sizes compared to three could have a negative impact on strength of the samples due to lower compatibility. Furthermore, properly graded material lessens the possibility of shrinkage and cracking when the samples are subjected to sudden change in temperature (Kingery, 1976). All samples in the present work did not crack as seen in Figure 4.2.

4.10 Comparison of MOR with South African National Standards

All samples fired above 1100⁰C are classified by SANS. Those below 1100⁰C are weak and cannot be used for flooring SANS (2008). Cristina and FRICA. et .al (2001) and SANS (2008) reported that the quality and class of porcelain floor tiles is based on strength and water absorption.

Samples in the present work were formulated by pressing as opposed to extrusion. This is because in pressed samples particles are highly compacted hence creating strong fired samples. SANS (2008), classifies pressed tiles as B₁, B₂, B₃ and B₄. B₁ tiles have high strength and low water absorption whereas B₄ tiles have low strength with high water absorption as shown in Table 2.4.

Table 4.4: Comparison of strength of formulated samples at different firing temperatures with SANS.

Temperature/ ^o C	Composition	Strength/MPa	SANS/MPa	Class
1250	1	31.5	30	B ₁
	2	34.2	30	B ₁
	3	23.6	20	B ₂
	4	22.2	20	B ₂
1200	1	19.6	20	B ₂
	2	33.3	30	B ₁
	3	23.0	20	B ₂
	4	24.0	20	B ₂
1150	1	18.7	18	B ₃
	2	19.8	20	B ₂
	3	27.5	30	B ₁
	4	29.3	30	B ₁
1100	1	17.2	18	B ₃
	2	24.4	20	B ₂
	3	23.6	20	B ₂
	4	16.3	15	B ₄
1050	1	4.0	-	
	2	6.9	-	
	3	4.5	-	
	4	6.2	-	

4.11 Statistical Analysis of Strength

Data analysis was done using ANOVA and F-statistic as seen in Table 4.5. The null hypothesis was set as $H_0 : < 3.06$. Implying that there is no variation of temperature with strength if the null hypothesis is true

Table: 4.5 ANOVA for Strength

Source of variation	Sum of squares	Degree of freedom	Mean square	F_0
Temperature	9619.2	4	2404.8	102
Error	353.8	15	23.5	
Total	9973			

From Table 4.4 it is shown that $F_0=102$ however, $F_{0.05,4,15} = 3.06$ at 0.05 significance levels. Since F_0 is greater than $F_{0.05,4,15}$ then there is a significant effect of temperature on the strength of the samples formulated. Details of statistical analysis are shown in Appendix table 15 and 16.

4.12 Results of Water Absorption

Figure 4.5 shows the percentage of water absorption by weight as a function of different firing temperatures.

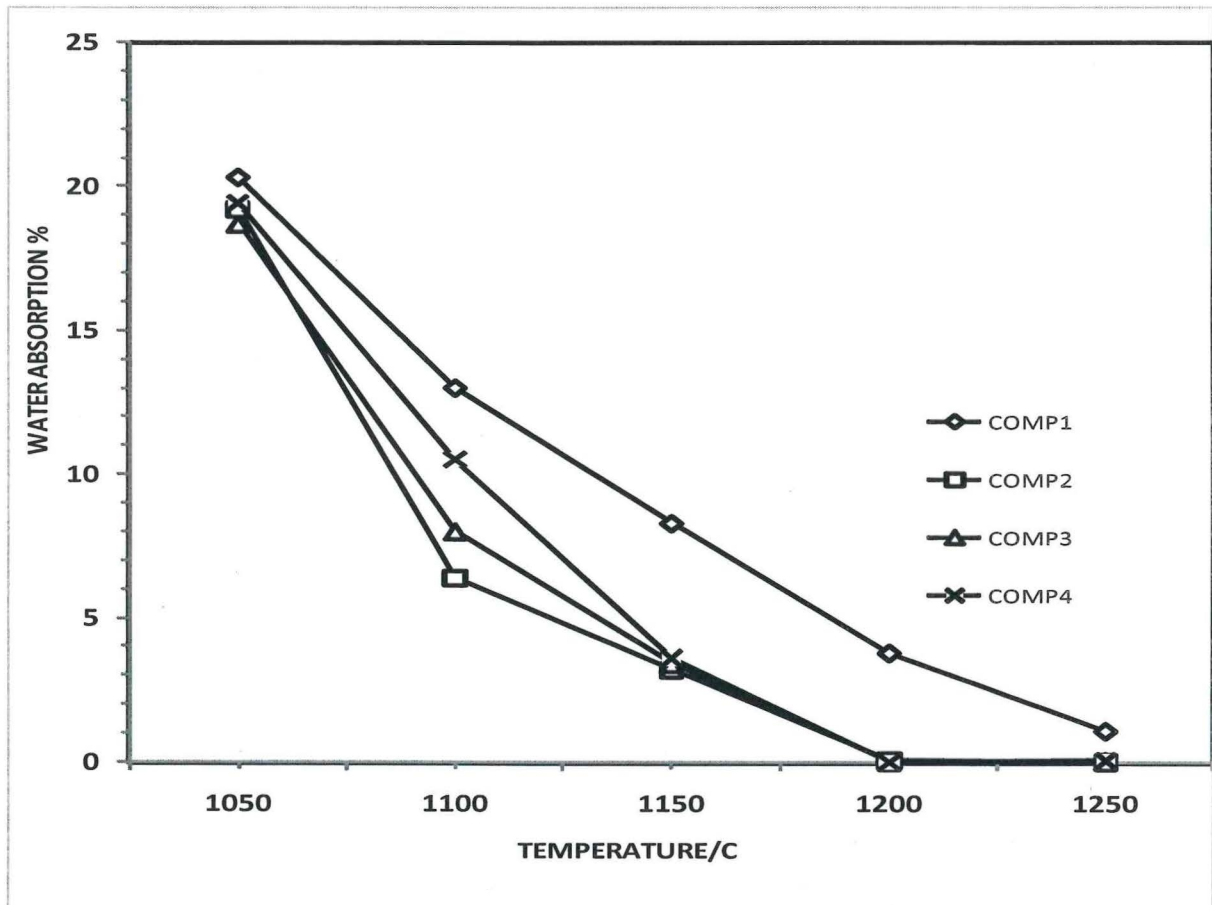


Fig 4.5: Water Absorption of the Sample Tiles

There is a general decrease in water absorption as firing temperature is increased. Composition 1 demonstrated the highest percentage of water absorption compared to all the other compositions at all temperatures. Compositions 2, 3 and 4 experienced slightly similar values of water absorption at temperatures of 1150, 1200 and 1250°C. For the same compositions, firing at 1200 to 1250°C resulted into 0.0% water absorption which was the lowest for all the samples. On the other hand,

4.13 Discussion on Water Absorption

Water absorption of samples determines the number of pores within the body. Samples which absorb more water have high porosity and vice versa. Samples with high water absorption have low strength as seen in Figure 4.4 and Figure 4.5. Low water absorption at 1050⁰C is attributed to high porosity due to low rate of sintering. Decrease in water absorption with temperature as seen in Figure 4.5 is due to increasing rate of sintering. According to Grimshaw (1971) and Ashby (1986), reported that temperature increase causes particle diffusion, sintered substances fill up the voids in samples reducing porosity and increasing strength.

At 1200 and 1250⁰C the percentage of water absorbed is very low with a value of approximately 0.0% with exception of composition 1, this is due to increased formation of glassy matrix due to melting of feldspar. The glassy phase formed fills up the voids reducing porosity. According to Obwoya (2004) and Bergmann and Zimmer et. al (2006), the glassy matrix acts to densify the structure of the specimen and reduce porosity thus reduction in water absorbed. High proportions of feldspar in the samples are responsible for the glassy formation causing a sharp decline in water absorption (Abadir and Sellam. et. al. 2001).

Low water absorption as seen in Figure 4.5 is also due good particle agglomeration of the pressed samples as a result of grading. Applying pressure of 30MPa on graded materials as in the present work, results into tight interlocking of particle hence decreasing the number and size of pores. The decrease in porosity results into low water absorption and improvement in flexural strength.

4.14 Comparison of Water Absorption with SANS

According to SANS (2008), B₁ tiles absorb less than 3% water by weight as shown in Table 2.4, this class of tiles are termed as impervious tiles if their water absorption is less than 0.5%. In the present work compositions 2, 3 and 4 absorbed 0.0% water by weight at temperature of 1200 to 1250⁰C as shown in Figure 4.4. Compositions 2, 3 and 4 further at a temperature of 1150⁰C and composition 1 at 1200⁰C absorbed between 3 to 6% water by weight, SANS classifies this range as B₂ tiles which is also termed as semi-vitreous tiles. B₃ tiles absorb between 6 to 10% water by weight, the present work attained this for compositions 2, 3 and 4 at a temperature of 1100⁰C and composition 1 at 1150⁰C. Finally samples which absorbed between 10 to 20% water are classified under B₄ tiles. This was achieved by all compositions at 1050⁰C as shown in Figure 4.4. A comparison between water absorption (wt %) of samples at different temperatures with SANS is shown in Table 4.6

Table 4.6: Comparison of Water Absorption of formulated samples at different firing temperatures with SANS.

Temperature/ ^o C	Composition	W.A%	SANS %	Class
1250	1	1.1	0– ≤ 3	B ₁
	2	0.0	0– ≤ 3	B ₁
	3	0.0	0– ≤ 3	B ₁
	4	0.0	0– ≤ 3	B ₁
1200	1	3.8	3– ≤ 6	B ₂
	2	0.0	0– ≤ 3	B ₁
	3	0.0	0– ≤ 3	B ₁
	4	0.0	0– ≤ 3	B ₁
1150	1	8.3	6– ≤ 10	B ₃
	2	3.2	0– ≤ 3	B ₁
	3	3.4	3– ≤ 6	B ₂
	4	3.6	3– ≤ 6	B ₂
1100	1	13.0	10– ≤ 20	B ₄
	2	6.4	6– ≤ 10	B ₃
	3	8.0	6– ≤ 10	B ₃
	4	10.5	10– ≤ 20	B ₄
1050	1	20.3	10– ≤ 20	B ₄
	2	19.2	10– ≤ 20	B ₄
	3	18.7	10– ≤ 20	B ₄

4.15 Statistical Analysis of Water Absorption

Data was analyzed using ANOVA and F-statistics as shown in Table 4.7. Detailed analysis of data can be seen in appendix Table 17 and 18. The null hypothesis was set as $H_0 :< 3.06$. There is no variation of temperature with water absorption if the null hypothesis is true.

Table: 4.7. ANOVA for water absorption

Source of variation	Sum of squares	Degree of freedom	Mean square	F ₀
Temperature	1947.3	4	486.8	131.5
Error	56	15	3.7	
Total	2003.3			

From Table 4.7 it can be seen that $F_0=131.5$ however, $F_{0.05,4,15} = 3.06$ at 0.05 significance levels, since F_0 is greater than $F_{0.05,4,15}$ then there was a significant effect of temperature on the water absorption of formulated samples.

CHAPTER FIVE: CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

Porcelain stoneware tiles were formulated from kaolin, ball clay, feldspar and sand. Kaolin and feldspar was mined from Mutaka in Bushenyi district, ball clay from Ntaawo in Mukono and sand from Lido beach in Wakiso district. These deposits had never been exploited to make ceramic floor tiles from selected minerals in Uganda. The raw materials were sieved to produce powder of particle size 25, 45, 53 and 80 μ m for sand, ball clay, feldspar and kaolin respectively.

Chemical analysis by XRF method revealed 24.9% alumina and 95.0% silica content in kaolin and sand respectively which happened to be the highest. These values differ from earlier studies by both Olupot and Kirabira, this is because of the difference in the locations of the mines. A percentage composition of 3.2% iron oxide in ball clay was higher than earlier findings. Oxides of potassium, calcium, sodium, magnesia and magnesium were not analyzed, simply because of their less influence in the mechanical properties of fire samples.

Firing was done in an electric kiln at peak temperatures of 1050, 1100, 1150, 1200 and 1250⁰C. Apart from water absorption, there was a general increase in both strength and shrinkage with temperature, except for compositions with high silica (sand) content at temperatures between 1200 and 1250⁰ C. All samples fired at temperature of 1100 to 1250⁰C were classified by South African National Standards as B₁, B₂, B₃ and B₄ tiles.

Maximum strength of 34MPa was recorded at a temperature of 1250⁰C for samples which contained 20% ball clay, 30% kaolin, 40% feldspar and 10% sand. This figure

obtained is above 30MPa which SANS classified as B₁ tile. At the same temperature minimum strength of 22MPa was attained by samples containing 30% sand, 30% feldspar, 20% kaolin and 20% ball clay, SANS further classifies these tiles as B₂.

There was a general decrease in water absorption with temperature with a minimum value of 0.0% at 1200-1250⁰C. This figure was attained by samples which contained 20% ball clay, 20-30% kaolin, 30-40% feldspar and 10-30% sand. For the samples exhibiting the highest strength, the water absorption attained is range recommended by SANS for class B₁ tiles.

Shrinkage of 5-10% was recorded for samples with 20% ball clay, 30-40% kaolin, 30-40% feldspar and 10% sand at 1100 to 1250⁰C. The value of shrinkage attained in the present work was close to the industrially acceptable range of 5-7.5%.

No cracks were observed and surface finish of all samples was smooth. Warping occurred for most samples fired at a temperature 1050⁰C and some at 1100⁰C. This happened mainly for samples placed at the lower part of the kiln. However, all samples fired at 1150⁰C and above did not warp irrespective of their position in the kiln. The color of samples changed from white at a temperature of 1050⁰C to brown at 1150⁰C and finally to grey at 1250.

In general, a high grade tile class B₁ has been attained using the experimental compositions consisting of 20% ball clay, 30% kaolin, 40% feldspar and 10% sand. Fired at a rate of 360⁰C per hour to a temperature of 1250⁰C. The tile exhibited strength of 34Mpa, water absorption of 0.0% (wt) and shrinkage of 9% (wt). All these are in the

range acceptable by SANS for this standard of tiles. Materials from the deposits studied have been shown to be suitable to produce high grade ceramic floor tiles.

5.2 Recommendations

The following work is suggested for further investigation

- Microstructural and X-Ray diffraction (XRD) analysis should be done to find causes of decrease in strength of samples with high sand content upon firing at 1150 to 1250⁰C. Sand is said to improve the strength of porcelain by reducing the growth of Griffith-flaws and matrix re-enforcement hypothesis. Furthermore sand improves strength by formation of cristobalite especially when particle sizes of 10-30 μ m are used. In the present study sand particle size of 25 μ m was used.
- Effects of material mixing on mechanical properties of ceramic tiles. Mixing determines the arrangement of particles in the body. Proper mixing blends together coarse, medium and finer particles. The fine particles occupy most of the gaps created in the samples hence reduction in porosity. This improves on the strength and lessens the possibility of cracks and warping. In the present work, mixing was done in two phases for ten minute each with 12% water content. Adjusting the mixing pattern by increasing blending time creates a more closed-packed particle arrangement. This improves on the strength of the samples.
- Mechanical properties of ceramic floor tiles made of only ball clay should be analyzed. Ball clay contains alumina and silica which have positive influential in strength of ceramic tiles. In the present study, properties of porcelain tiles were analyzed. Porcelain stoneware tiles are made of kaolin, ball clay, feldspar and sand.

The raw materials used have different functionalities. Clay is used to ease forming and improve strength, feldspar lowers the kiln temperature and sand is used for dimensional stability.

REFERENCES

- Abadir M.F. Sallam E.H and Bakr I.M. (2003). Preparation of porcelain tiles from Egyptian raw Materials. *Ceramics international* .Vol 45 page 303-310
- Agenor D.N. Danchamir H and Vicente C. (2009). Effect of quartz particle size on the mechanical behavior of porcelain tile subjected to different cooling rates. *Journal of European ceramic society*. Vol 29 Page 1034-1046.
- Anura G. (1994). Clay Testing. Intermediate technology publication. London
- Ashby M.F. (1986). Engineering materials 2. Pergamum publishers. New York
- Bergmann C.P and Zimmer A. (2006). Fly ash of mineral coal as ceramic tile raw material. *Ceramic International*. Vol 78 page 456-489
- Branganca S.R. Bergmann C.P and Hubbner H. (2006). Effect of quartz particle size on the strength of triaxial porcelain. *Journal of European ceramic society*. Vol 11 page 789-811
- British Geological Survey (2011). Natural environment research council. Mineral planning factsheet.
- Carneane E. Marilena V. Saulo G and Orestes E. (2001). Microstructural characteristics of ceramic floor tiles with the incorporation of wastes from ceramic tile industries. *Ceramics International*. Vol 67 page 90-107
- Cristina L. Federica B. Paolo V. (2001). Enhancing the mechanical properties of porcelain stoneware tiles: a micro structural approach. *Journal of European ceramic society*. Vol 21Page 785-795.
- Grishaw R.W. (1971). Physics and Chemistry of Clay. Wiley-Interscience. New york

Hutchings S.M.J. Xu Y. Sanchez E and Ibanez M.J. (2006). Porcelain tile microstructure:

Implication for polishability. *Journal of European ceramic society*. Vol 26 Page 1035-1042.

Kaahwa Y. (1999). Introduction to engineering materials. Ate limited. Kampala

Kingery. (1976). Introduction to ceramics. Wiley-interscience. Canada

Kirabira J.B. (2000). Technical viability of manufacturing firebricks using locally available raw materials. Masters thesis, Makerere University.

Kirabira J.B. (2005). Properties of Uganda minerals and fireclay refractories. Doctorial thesis in material science. Royal Institute of Technology (KTH) Stockholm, Sweden

Martin-Marquez J. Ma-Rinco J and Romero M. (2007). Effect of firing temperature on sintering of porcelain stoneware. *Ceramics international*. Vol 55 page 51-69

Montgomery D.C (2005). Design and analysis of experiments. John Willey and sons. Arizona State University

Nyakairu G.W and Koeberl C. (2001). Mineralogical, Chemical Composition and distribution of rare earth elements in clay-rich sediments from central Uganda. *Geochemical journal* Vol35 page 13-28

Obwoya S.K. (2004). Effects of microstructure on mechanical strength of selected Clays from Uganda. Doctorial thesis Makerere University Kampala

Olupot P.W. (2010). Characterization of ceramic raw materials in Uganda for Production of electrical porcelain insulators. Doctorial thesis in material science. Royal Institute of

Technology (KTH) Stockholm, Sweden

- Olupot P.W and Johnson S. Byaruhanga J.K. (2010). Effects of mixing proportions and firing temperature on properties of electric porcelain from Ugandan minerals. Doctorial thesis in Material science (KTH). Stockholm Sweden.
- Olupot P.W. (2006). Assessment of ceramic raw materials in Uganda for electrical Porcelain. Licentiate thesis in material science.(KTH). Stockholm, Sweden.
- Olupot P.W. Johnson S. Byaruhanga J.K. (2006). State of the art paper on development of electrical porcelain insulators from Ugandan raw materials.(KTH) Stockholm Sweden.
- Rahbar N. Aduba B.O. Zimba J. Obwoya S.K. Nyongesa F.W Yakubu I and Soboyejo W.O. (2010). Thermal Shock Resistance of a kyanite based (Aluminosilicate) ceramic. *Ceramics international*. Vol 34 page 456-460
- Raymond H.A. (1994). Properties of engineering materials. Butterworth Heinemann
- Rhodes D. (1973). Clay and Glazes for porters. A and C Black. London
- Ryan W. (1978). Properties of ceramic raw materials. Pergamon press. New York
- Sanchez E. Gaarcia J. Sanz V. Moreno A. (2010). Porcelain tile: Almost 30 years of steady scientific-technological evolution. *Journal of European ceramic society* vol 36 page 831-845.
- Senapati U and Carty W.M. (1998). Porcelain-Raw materials, processing, phase evolution and mechanical behavior. *Journal of American ceramic society* page 3-20
- SingerF. (1963). Industrial ceramics.chapman and hall. New York
- Stathis A. Ekonomaku C.J and Stournaras C. (2004). Effect of firing conditions, filler grain size and quartz content on strength and physical properties of sanitary ware porcelain. *Journal*

of European ceramic society page 2357-2366.

Tucci A. Esposito L. Molmusi L and Rambaldi E. (2007). New body mixes for porcelain stoneware tiles with improved mechanical characteristic. *Journal of the ceramic society*.

Vol 27 page 1875-1881.

Weems J.B. (1988). Chemistry of Clay. Willey-interscience. New York

Yaseen I and William E.L. (2000). Microstructural Evolution in Triaxial porcelain. *Journal of American ceramic society*. vol 2 page 3121-3125

<http://www.enotes.com>. How products are made/ceramic tiles, accessed on 28/11/2010.

Uganda Clays Limited. (1994). ‘ ‘Production of Ceramic ware’’ Kampala Uganda

South African National Standard. (2008). Ceramic wall and floor tiles. Published by standards South Africa. Pretoria.

<http://www.tile.com>. Ceramic tiles, accessed on 7th November 2010

<http://www.azom.com>. Production and processing of engineering ceramics including forming, firing and finishing by dynamic ceramics, accessed on 17th /12/2010

[http://www. Online library.willey.com](http://www.Online library.willey.com).The effect of pressure variation on fired products, accessed on 1/11/2010.

Determination of water absorption, apparent porosity, apparent relative density and bulk density. (1995). ISO 10545-3.

<http://www.builddirect.com>. Ceramic tiles and porcelain tiles-build direct, accessed on 10th /01/2011.

<http://www.nationalfloor covering alliance/tile> .Water absorption of ceramic tiles, accessed on

10th /8/2011.

Determination of modulus of rupture and breaking strength. (1994) ISO 10545-4.

<http://www.lagunaclay.com/support/bloating> and black coring. Bloating and black coring of fired clay, accessed on 3/11/2011.

[http://www.pottery.about.com/understan clay/ht/clay type](http://www.pottery.about.com/understan%20clay/ht/clay%20type). Color of fired clay, accessed on 9th/03/2012.

Table 3: Linear shrinkage of composition 3

T/°C	L/mm	b/mm	t/mm	L.S-L%	L.S-b%	L.S-t%	Av-L.S%
1250	98.4	47.7	9.9	8.7	7.9	6.6	7.7
1200	95.5	45.6	8.7	11.4	11.9	17.9	13.7
1150	97.0	46.6	8.9	10.0	10.0	16.0	12.0
1100	99.2	47.6	9.1	7.9	8.1	14.0	10.0
1050	105.9	50.7	10.4	1.7	2.1	1.8	1.8

Table 4: Linear shrinkage of composition 4

T/°C	L/mm	b/mm	t/mm	L.S-L%	L.S-b%	L.S-t%	Av-L.S%
1250	100.0	48.4	9.6	7.2	6.5	9.4	7.7
1200	95.1	45.7	8.1	11.7	11.7	23.0	15.4
1150	96.2	46.1	8.9	10.8	11.0	16.0	12.6
1100	100.3	48.1	9.3	6.9	7.1	12.2	8.7
1050	105.8	50.4	9.7	1.8	2.7	8.4	4.3

Table 5: MOR of composition 1

T/°C	P.r/div	F/kN	MOR/MPa
1250	232	0.997	31.5
1200	155	0.666	19.6
1150	151	0.645	18.7
1100	150	0.645	17.2
1050	45	0.193	4.0

Table 6: MOR of composition 2

T/°C	P.r/div	F/kN	MOR/MPa
1250	269	1.156	34.2
1200	262	1.126	33.3
1150	170	0.731	19.8
1100	213	0.916	24.4
1050	76	0.327	6.9

Table 7: MOR for composition 3

T/°C	P.r/div	F/kN	MOR/MPa
1250	245	1.054	23.6
1200	264	1.135	23.0
1150	225	0.967	27.5
1100	206	0.886	23.6
1050	55	0.235	4.5

Table 8: MOR values of composition 4

T/°C	P.r/div	F/kN	MOR/MPa
1250	188	0.808	22.2
1200	228	0.982	24.0
1150	237	1.019	29.3
1100	151	0.649	16.3
1050	61	0.262	6.2

Table 9: Percentage of water absorption of composition 1 tiles

T/°C	W.A%
1250	1.1
1200	3.8
1150	8.3
1100	13.0
1050	20.3

Table 10: Percentage of water absorption of composition 2

T/°C	W.A%
1250	0.01
1200	0.07
1150	3.2
1100	6.4
1050	19.2

Table 11: percentage of water absorption of composition 3

T/°C	W.A%
1250	0.03
1200	0.01
1150	3.4
1100	8.0
1050	18.7

Table 12: Percentage of water absorption of composition 4 tiles

T/°C	W.A%
1250	0.05
1200	0.01
1150	3.6
1100	10.5
1050	19.4

APPENDIX B

TABLE OF STATISTICAL ANALYSIS OF RESEARCH

Table 13: ANOVA for shrinkage

Temp/ ^o C	Compositions				y _i (totals)
	1	2	3	4	
1050	1.0	1.8	1.8	4.3	8.9
1100	5.0	6.2	10.0	5.7	29.9
1150	7.1	6.9	12.0	12.6	38.6
1200	8.3	9.2	13.4	15.4	46.3
1250	9.9	9.1	7.7	7.7	34.4
					$\Sigma = 158.1$

$$SS_T = (1.0^2 + 1.8^2 + 1.8^2 + 4.3^2 + \dots + 7.7^2) - \frac{158.1^2}{20} = 1527.6$$

$$SS_{Temp} = \frac{1}{4} [8.9^2 + 29.9^2 + \dots + 34.4^2] - \frac{158.1^2}{20} = 1439.6$$

$$SS_E = SS_T - SS_{Temp} = 88.0 \text{ hence } F_o = 61.3 \text{ and } F_{0.05,4,15} = 3.06$$

Table: 14

Source of variation	Sum of squares	Degree of freedom	Mean square	F _o
Temperature	1439.6	4	360	61
Error	88	15	5.9	
total	1527.6			

Table 17: ANOVA for water absorption

Temp/ ^o C	Composition				y _i (totals)
	1	2	3	4	
1050	20.3	19.2	18.7	19.4	77.6
1100	13.0	6.4	8.0	10.5	37.9
1150	8.3	3.2	3.4	3.6	18.5
1200	3.8	0.07	0.01	0.01	3.9
1250	1.1	0.01	0.03	0.05	1.2
					$\Sigma = 139.1$

$$SS_T = (20.3^2 + 19.2^2 + 18.7^2 + \dots + 0.05^2) - \frac{139.1^2}{20} = 2003.3$$

$$SS_{Temp} = \frac{1}{4} [77.6^2 + 37.9^2 + \dots + 1.19^2] - \frac{139.1^2}{20} = 1947.3$$

$$SS_E = 2003.3 - 1947.3 = 56 \text{ hence } F_O = 131.5 \text{ and } F_{0.05,4,15} = 3.06$$

Table: 18

Source of variation	Sum of squares	Degree of freedom	Mean square	F ₀
Temperature	1947.3	4	486.8	131.5
Error	56	15	3.7	
total	2003.3			

Table 15: ANOVA for strength

Temp/ ^o C	Composition				y_i (totals)
	1	2	3	4	
1050	4.0	6.9	4.5	6.2	21.6
1100	17.2	24.4	23.6	16.3	81.5
1150	18.7	19.6	27.5	29.3	95.3
1200	19.6	33.3	23.0	24.0	99.9
1250	31.5	34.2	23.6	22.0	111.3
					$\Sigma = 409.6$

$$SS_T = (4.0^2 + 6.9^2 + 4.5^2 + \dots + 22.0^2) - \frac{409.6^2}{20} = 9973$$

$$SS_{Temp} = \frac{1}{4} [21.6^2 + 81.5^2 + \dots + 111.3^2] - \frac{409.6^2}{20} = 9619.2$$

$$SS_E = 353.8 \text{ hence } F_0 = 101.9 \text{ and } F_{0.05,4,15} = 3.06$$

Table: 16

Source of variation	Sum of squares	Degree of freedom	Mean square	F_0
Temperature	9619.2	4	2404.8	102
Error	353.8	15	23.5	
total	9973			

**MECHANICAL PROPERTIES OF CERAMIC FLOOR
TILES MADE FROM SELECTED MINERALS IN
UGANDA**

BY

OCHEN WILLIAM

2009/HD/160/MScP

**A DISSERTATION SUBMITTED TO GRADUATE SCHOOL IN PARTIAL
FULFILMENT OF THE REQUIREMENTS OF THE AWARD OF
THE DEGREE OF MASTER OF SCIENCE IN PHYSICS OF
KYAMBOGO UNIVERSITY**

JUNE 2012