

**EVALUATION OF LOCAL CERAMIC MATERIALS FOR HIGH VOLTAGE
INSULATION APPLICATION**

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**A Dissertation Submitted in Partial Fulfilment of the Requirements for the Degree of
Master's in Material Sciences and Engineering of the Nelson Mandela African Institution
of Science and Technology**

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ABSTRACT

This study investigated the Tanzanian local ceramic material for their potential as raw materials for production of high voltage insulators based on the mix proportion of raw materials and firing temperatures. The raw materials used were: Pugu kaolin, quartz supplied by the Kilimanjaro Industrial Development Trust (KIDT), and Kilimanjaro feldspar.

The study characterised both individual raw materials and the blends produced from these raw materials. A total of six batches were prepared by varying the proportion of raw materials. Cylindrical samples of 10 and 60 mm diameter were produced by slip casting method and fired at the maximum temperature of 1100, 1200 and 1300 °C. Fired samples were tested for insulation resistance, bending strength, and bulk properties.

The X-ray diffraction (XRD) analysis showed that on heating Pugu kaolin produces mullite phase which is very important for mechanical and dielectric properties. Furthermore, the highest trend in insulation resistance and bending strength is revealed to samples fired at 1200 °C with lower values for those fired at 1100 and 1300 °C.

The optimum mix proportion and firing temperature, which compromise both insulation resistance and bending strength is with 33 wt% Pugu kaolin, 11 wt% Kilimanjaro quartz, and 56 wt% Kilimanjaro feldspar at the firing temperature of 1200 °C. With this mix proportion and firing temperature at maximum insulation resistance of 205800 MΩ at the injection of 1000 V is achieved. The bending strength and water absorption for this mix proportion and firing temperature is 65.358 MPa and 0.01 %, respectively. These results correspond to the standard specifications for ceramic insulators.

Keywords: Insulation resistance, bending strength, kaolin, quartz, feldspar, mullite

DECLARATION

I, **Moses George Moyo** do hereby declare to the Senate of Nelson Mandela African Institution of Science and Technology that this dissertation is my own original work and that it has neither been submitted nor being concurrently submitted for degree award in any other institution.

Moses George Moyo Ph.D.

December, 2014

Name and signature of candidate

Date

The above declaration is confirmed by

Prof. Eugene Park



December, 2014

Name and signature of supervisor

Date

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CERTIFICATION

This is to certify that the dissertation entitled “*Evaluation of Local Ceramic Materials for High Voltage Insulation Application*” submitted by Mr. Moses George Moyo (M135/ T.12) in partial fulfillment of the requirements for the award of **Master of Science degree in Material Sciences and Engineering** at Nelson Mandela African Institution of Science and Technology, Tanzania is an authentic work carried out by him under my guidance.



December, 2014

.....
Prof. Eugene Park
(Principal supervisor)

.....
Date

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DEDICATION

This dissertation is dedicated to my family and NM-AIST for helping me during the undertaking of this work.

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LIST OF ABBREVIATIONS AND SYMBOLS

MPa	Megapascal
Wt	Weight
XRD	X-ray diffraction
SEM	Scanning Electron Microscope
DC	Direct Current
Lw	Wet length
Lf	Final length
Fs	Load at failure
LOI	Loss on ignition
KIDT	Kilimanjaro Industrial Development Trust
JIS	Japan Industrial Standards
Ws	Soaked weight
Wd	Dry weight
NM-AIST	Nelson Mandela African Institution of Science and Technology
AEMC	American Energy and Manufacturing Competitiveness
C1	Mix proportion for batch one
C2	Mix proportion for batch two
C3	Mix proportion for batch three
C4	Mix proportion for batch four
C5	Mix proportion for batch five
C6	Mix proportion for batch six

CHAPTER ONE

1.0 Introduction

This chapter describes the general introduction of the study. It mainly focuses on the background information of the study, the problem statement and justification of study, objectives, research questions and significance of the research.

1.1. Background information

Local developed technology is considered as an essential step in exploring and promoting underutilized resources for the economic potential of any nation. Tanzania is endowed with geological environment which is very rich in hosting different mineral resources, including gold, diamond, base metals and various industrial minerals including ceramic raw material (SEAMIC, 2008). Most of the local ceramic materials are found to be applied in production of earthenware and low voltage electrical insulators.

Electrical insulators are essential components in power transmission networks playing an important role in preventing or regulating current flow in electrical circuits (Islam *et al.*, 2004). Insulators used for power transmission are made from glass, porcelain or composite polymer materials. Porcelain insulators are ceramic materials composed primarily of clay, feldspar, and quartz (Carty & Senapati, 1998).

In the ceramic mix each of these raw materials plays its important role. Clay [$\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$] provides plasticity to the ceramic mixture; quartz (SiO_2) maintains the shape of formed article during firing; and feldspar ($\text{K, Na Al Si}_3\text{O}_8$) saves as flux. The porcelain is known as “silicious porcelain” if quartz is used as a filler and an “aluminous porcelain” if bauxite is used (Carter & Norton, 2007). For an insulator to qualify in application, international electrotechnical commission (IEC) specified up to twenty standard property tests, but the most important property is dielectric strength, and mechanical strength.

Because of the complexities of the porcelain system and despite the substantial amount of research already conducted within the field, there remain significant opportunities for research and study, particularly in the areas of raw material understanding, processing science, and phase and microstructure evolution (Carty & Senapati, 1998).

Electricity demand in Tanzania is increasing yearly; by March 2013 the generation capacity was 1,564 MW expected to reach 48,000 GWh by the year 2035 (Ministry of Energy and Minerals report-April 2013). In spite of the enormous broad range of application and availability of raw materials, mostly used insulators are still imported due to manufacturing of electrical insulators in small quantities and majority restricted to the low voltage power supply. Currently; in Tanzania these ceramic raw minerals (i.e. quartz, feldspar and kaolin) are employed in the production of low voltage insulators, but limitation of information exists about the materials characteristics and whether they can be employed for production of high voltage insulators.

The aim of this research is to investigate the applicability of local ceramic materials for their potential in the production of electrical insulators for high voltage application based on mix proportion of raw materials and firing temperatures. The study involves ceramic raw materials from some selected Tanzanian deposits which include; Pugu kaolin, quartz supplied by the Kilimanjaro Industrial Development Trust (KIDT), and Kilimanjaro feldspar.

1.2. Problem statement and justification of study

Abundant of ceramic raw minerals which can be used to produce ceramic insulators are available in Tanzania (SEAMIC, 2008). The current practice indicates that these local ceramic materials are being used for production of low voltage insulators which is produced by the KIDT industry in the Same - Kilimanjaro region. But literature shows that these ceramic minerals can also be employed for high voltage insulators if their characteristics are controlled (Islam *et al.*, 2004).

On the other hand; information on material characteristics and whether they can be employed for the production of high voltage insulators is inadequate, in this case; there is a need to investigate their applicability for production of high voltage insulators.

1.3. Objectives

1.3.1. General objective

The general objective of this study is to investigate the applicability of local ceramic materials for production high voltage electrical insulation.

1.3.2. Specific objectives

- (i) To determine the chemical composition and physical properties of selected local ceramic minerals.
- (ii) To fabricate the porcelain body from the locally available materials based on mix proportions and firing temperatures.
- (iii) To determine the electrical insulation, mechanical and bulk properties of finished porcelain body.

1.4. Research questions

- (i) What are the chemical and physical properties of locally sourced ceramic materials?
- (ii) What is the optimum mix proportion and the firing temperature of porcelain body produced by local ceramic materials for high voltage application?
- (iii) Is the fabricated porcelain body complying with the minimum requirement of mechanical and dielectric properties for high voltage application?

1.5. Significance of the research

This study provides scientific information for advancement to the production of high voltage insulators which are not currently produced in our country. Electricity demand is increasing from year to year, by March 2013 the generation capacity was 1,564 MW expected to reach 48,000 GWh by the year 2035 (Ministry of Energy and Minerals report-April 2013). This shows that the demand of electrical accessories such as insulators is also increasing while most of these products are imported. It is better to make use of our natural resources into useful goods so as to promote our economy.

CHAPTER TWO

Ceramic Raw Materials in Tanzania – Structure and Properties for Electrical Insulation Application¹

Abstract

Characterization of ceramic raw materials from Tanzanian deposits is carried out in order to investigate their potential as raw materials for production of electrical insulators. The raw materials utilized in this study are: Pugu kaolin, quartz supplied by the Kilimanjaro Industrial Development Trust (KIDT), and Kilimanjaro feldspar.

The results shows increasing Pugu kaolin beyond 48 wt% while reducing Kilimanjaro quartz below 6 wt% is leading to the reduction of electrical insulation and bending strength. The mix with constituent's percentage composition of 48 wt% Pugu kaolin, 46 wt% Kilimanjaro feldspar and 6 wt% quartz is possessing the highest strength of 53.525 MPa. Moreover, this mix proportion has an insulation resistance of 34812 MΩ at injection of 1000 V. In this regard, it is observed that high quality electrical insulators can be achieved from Tanzania originated ceramic materials.

Keywords: Ceramic raw materials, electrical Insulation, bending Strength, porcelain, mullite

2.1. Introduction

Electrical insulators are materials which prevent the current flowing in electrical circuits from being inserted as a barrier between conductors (Islam *et al.*, 2004). In transmission and distribution system the overhead conductors are generally supported by towers or poles which are properly grounded. For this reason there must be an insulator between tower or pole body and conductors to prevent the flow of current from the conductors to the earth through grounded supporting towers or poles (Electrical4u, 2014). Electrical insulation is important for preventing damage and reliability of electrical systems and equipment. On the other hand, it is a basis for

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protecting individuals from electric shocks and assures safety of personnel while carrying out electrical works. Generally, the properties required for material being an insulator are high resistivity, high dielectric strength, good mechanical properties and protection of a conductor from a severe environment like humidity and corrosiveness (Islam *et al.*, 2004). Dielectric strength and mechanical strength are two most important properties which must be taken into consideration. Electrical insulation resistance is a test which can be carried instead of dielectric strength test.

According to the international electrical testing association the minimum recommended value of insulation resistance of electrical systems and equipments is 1000 M Ω at the injection of 1000 V however, there is specific values of insulation resistance for a particular injected voltage ((NETA), 2007). In the consideration of mechanical strength the Japanese Industrial Standards and their counterpart require a minimum mechanical strength of 65 MPa.

In application the insulation can be group to that for either high voltage or low voltage depending on the voltage involved. The International Electrotechnical Commission and its national counterpart terms low voltage as the voltage between 120 to 1500 DC voltage and high voltage as the voltage above 1500 DC voltage (Mecano, n.d). Dielectric strength is the measure of materials to withstand high voltage. The higher the dielectric strength the better is the electrical insulation (Islam *et al.*, 2004). For high tension electrical insulation the dielectric strength has to be above 3kV/mm (Buchanan, 1986).

The insulators are made from nonconducting materials such as porcelain, glass and composite polymers. Each of these materials has a number of advantages over the other, for instance, glass insulators are considered to have very high resistivity and strength comparing to porcelain and composite polymer insulators. However, the problem associated with glass insulators is the tendency of allowing the path to leakage current once the air dust is deposited to the wet glass surface (Electrical4u, 2014). Polymer or composite polymer insulators have light weight compared to ceramic equivalents. The problem associated with polymer insulators is the tendency to exhibit electrical failure if there is any unwanted gap between core and weather sheds (Holtzhausen, n.d)

Porcelain is the most commonly used material for overhead insulators. Porcelains are resistant to high temperature, electricity, and harsh environment than polymers (Callister & Rethwisch, 2011). Therefore, the use of porcelain insulators is highly recommendable for these reasons. Porcelain is a ceramic material primarily composed of clay, quartz, and feldspar. Each of these materials plays its respective role in the properties of the blend formed from green to the fired body. Clay $[Al_2Si_2O_5(OH)_4]$ gives plasticity to the ceramic mixture; flint or quartz (SiO_2) maintaining the shape of the formed article during firing, and feldspar $[K_xNa_{1-x}(AlSi_3)O_8]$ serves as a flux to lower the melting point of the mixture. These three constituents place electrical porcelain in the phase system $[(K, Na)_2O-Al_2O_3-SiO_2]$ in terms of oxide constituents hence the term triaxial porcelain (Carty & Senapati, 1998; Olupot, 2006).

These raw materials are readily available at lower cost compared to other types of insulators which are based on industrially processed materials. Under controlled heating and cooling rates as well as firing cycles, it is possible to influence the properties of the final products which originate from the blend produced from these raw materials (A.O. Oladiji, 2010). Also, different properties can be achieved by varying the constituent's percentage composition of materials.

Over a period of time different researchers came up with a number of alternatives in researching material performance in terms of both mechanical and dielectric properties. Quartz as a filler material has been pointed out to have a detrimental effect on the performance of ceramic product. Excessive amount of quartz may lead to the initiation of non-coherent interface in structure, micro crack formation and decrease of mechanical properties (Maity & Sarkar, 1996; Ramaswamy *et al.*, 2005; Sedghi *et al.*). Different studies have been made in an attempt of minimizing the use of quartz as filler materials. Some of the studies are: replacement of quartz with alumina (Maity & Sarkar, 1996; Sedghi *et al.*); substitution of fly ash for quartz (Dana *et al.*, 2004); and the substitution of quartz by rice husk ash and silica fume (Prasad *et al.*, 2001).

The dielectric and mechanical properties of ceramics are affected by the phases which are developed on sintering. Phases which are important for proper electrical insulation and mechanical strength are mullite and glassy phase. Mullite phases particularly the one which is needle like shaped proved to perform well in electrical insulation property. On the other hand, the existence of excessive amount of glassy phase promotes free movement of ion hence poor electrical insulation properties (Islam *et al.*, 2004). In alumina based insulators corundum phase

proved to have great contribution to electrical insulation and mechanical properties (Sedghi *et al.*, 2014).

Evaluation of the structure and properties of raw materials is the primary step which leads to proper design of a particular structural component for intended performance. Through characterization of materials, chemical components of interest and harmful ingredients can well be quantified and checked if they comply with the requirements. The aim of this work is to characterize the local ceramic materials from some selected Tanzanian deposits to evaluate their potential as raw materials for production of electrical insulators.

2.2. Materials and Methods

2.2.1. Raw Materials

Analysis of raw materials involved feldspar from Kilimanjaro and quartz supplied by the Kilimanjaro industrial Development Trust (KIDT). After crushing and grinding the raw materials to powder form, pellets of respective raw materials were formed by pressing. Prepared pellets were fired at the temperatures ranging from 600 to 1400 °C. The crystalline structural phases for each firing temperature are determined by X-ray diffractometer of Rigaku Corporation/D/max 2200 V/PC having a 3 kV X-ray generator with Cu-target. Physical properties such as water absorbance, apparent porosity, apparent density and bulk density of samples sintered at different temperatures were determined by Archimedes method.

2.2.2. Blends formed from Raw Materials

For investigation of electrical insulation and mechanical strength cylindrical samples of 60 mm length and 10 mm diameter from six blends of Pugu kaolin, quartz, and feldspar (see Table 1) are fabricated by slip casting followed by air drying. Then samples are bisque fired at 800 °C before being sintered at the maximum temperature of 1300 °C in an electric furnace at the approximate rate of 100 °C/h with one hour soaking time. The insulation resistance of the fired samples was measured by (Megohmmeter AEMC instrument model 1060) by subjecting the test samples to 1000 DC voltage for 1 minute and recording the displayed value of insulation resistance. Bending strength of samples was determined by three point bending test strength machine manufactured by MFG CO.LTD model No. 704372. The load at failure in Kgf was recorded for

ten test samples. The span of 50 mm was used in testing the samples. The failing load F_s was determined from the test and then the calculation of bending strength was done by using the formula below.

$$\text{Bending Strength} = \frac{F_s * L}{\pi R^3}$$

R = Radius of sample

L = Span (center to center from the support)

F_s = Load at Failure

Table 1: Batch Compositions (wt. %)

	C1	C2	C3	C4	C5	C6
Al ₂ O ₃	25	30	35	40	45	50
SiO ₂	80	75	70	65	60	55
K ₂ O	15	15	15	15	15	15

Values from theoretical batch calculations (Combination of Pugu kaolin, Quartz and Feldspar)

2.3. Results and Discussion

2.3.1. Properties and Structure of Raw materials

Chemical composition of Raw Materials

The chemical composition of raw materials used in this study is shown in Table 2. The composition purity requirements of raw materials for insulators application are: in clays (Fe₂O₃, TiO₂) < 3.5 %; in quartz; SiO₂ > 99 %; and in feldspar Fe₂O₃ < 1 %; (K₂O, Na₂O) > 12 % (Meng *et al.*, 2012). This chemical composition analysis is showing that the content of the components is closely related to the requirements for insulator manufacturing. Kilimanjaro Feldspar composed of relatively small amounts of detrimental oxide, which meets the percentage purity requirements of raw materials for electrical insulator application as reported by (Meng *et*

al., 2012). The amount of flux (K_2O Na_2O) in feldspar is 14.94 wt%, which complies with the composition purity requirement for raw materials since (K_2O Na_2O) has to be greater than 12 %.

Crystallographic Structure of Raw Materials

The XRD results showed that in feldspar the raw materials contains albite, and microcline, tridymite and quartz as other phases developed on sintering as indicated in Fig. 1. The feldspar major components of interests are: potash feldspar ($K_2O.Al_2O_3.O.6SiO_2$); soda feldspar ($Na_2O.Al_2O_3.O.6SiO_2$); and lime feldspar ($CaO.Al_2O_3.O.6SiO_2$). Potash feldspar is usually crystallized in monoclinic form as orthoclase or in triclinic form as microcline where as lime and soda feldspar crystallizes in triclinic form as anorthite and albite (Iqbal & Lee, 2000).

Table 2: Chemical Composition of Raw Materials in wt%

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O	LOI
Feldspar	53	19.1	0.24	-	0.09	0.01	2.04	12.9	12.62
Quartz	97.7	-	-	-	-	-	-	-	2.3

The results indicate that feldspar under study contains high content of potash feldspar compared to soda feldspar since the chemical composition by XRD shows K_2O is 12.9 % while Na_2O is only 2.04 wt%.

The XRD pattern shown in (Fig.1) started to change beyond 1200 °C, this is because, beyond 1200 °C there was a phase change from crystalline to amorphous this is well demonstrated by the sharp peaks below between 600 and 800 °C and spread with almost no sharp peaks above 1200 °C. With amorphous phase: X-rays will be scattered in many directions leading to a large bump distributed in a wide range (2 Theta) instead of high intensity narrower peaks.

In the XRD- crystallographic study of quartz the maximum peak is revealed around 25 (2θ-degree) which is typical diffraction angle for quartz. The 2.3 % loss on ignition in quartz raw materials in Table 1 demonstrate that quartz content was not 100 %, which implies there was some water contained in quartz.

The study made to Pugu kaolin by (H. Hamisi, et al) is revealed the development of mullite phase when fired at 1400 °C. Mullite is very important phase for promoting the mechanical and dielectric properties.

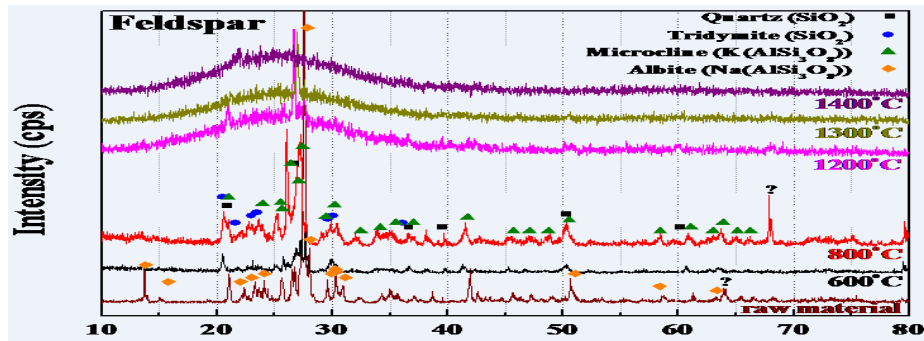


Figure 1: XRD Patterns of feldspar at different firing temperature

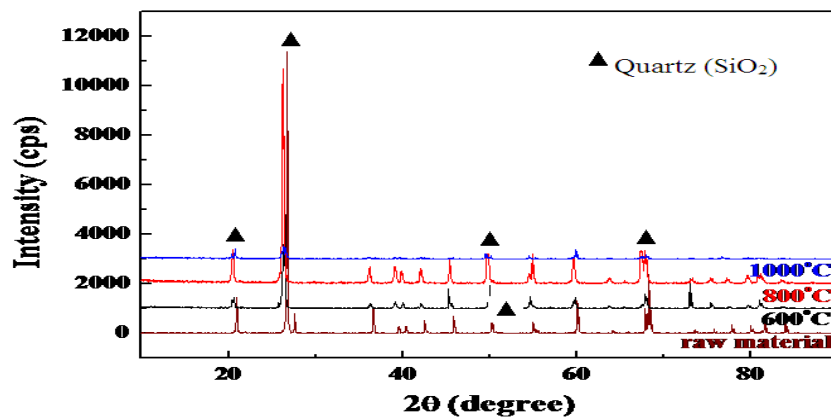


Figure 2: XRD Patterns of quartz at different firing temperature

Bulk Properties of Raw Materials

On sintering feldspar samples, linear shrinkage continued to increase up to 800 °C and in further increase in the sample's firing temperature decrease in linear shrinkage was noted as indicated in (Fig.3). Water absorption of sintered feldspar samples is found to increase above 1200 °C and started to drop abruptly beyond 1300 °C. In connection with the results in Table 3 the maximum densification with low level of apparent porosity was observed at 1200 °C and above this temperature densification decreased with increase in apparent porosity which implies that the optimum sintering temperature for feldspar is 1200 °C.

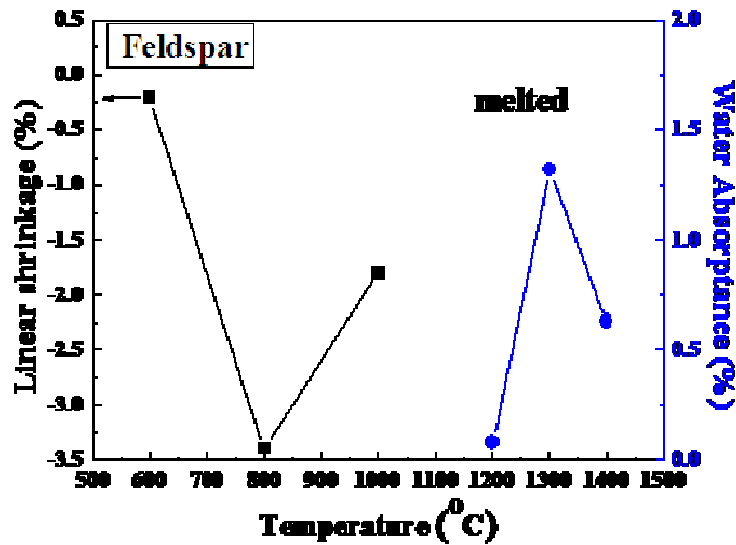


Figure 3: Linear Shrinkage and Water absorption (Feldspar)

Table 3: Bulk density and apparent density (Feldspar)

Heat treatment temperature (°C)	Apparent density (g/cm ³)	Bulk density (g/cm ³)	Apparent Porosity (%)
1200	2.318	2.313	12.3
1300	2.299	2.232	11.5
1400	2.284	2.251	12.7

2.3.2. Properties of Blends formed from Raw Materials

Bending Strength

The highest bending strength of 53.525 MPa is achieved by samples composed of 48 wt% Pugu kaolin, 46 wt% Kilimanjaro feldspar, and 6 wt% Kilimanjaro quartz. Strength is found to increase with an increase in content of Pugu kaolin while reducing the content of Kilimanjaro quartz. Decreasing quartz beyond 6 wt% and increasing Pugu kaolin beyond 48 wt% is leading to the reduction of strength as indicated in Table 5, and (Fig.4).

Table 4 : Mix proportion of raw materials for batches of table 1(Normalized wt. %)

	C1	C2	C3	C4	C5	C6
Pugu kaolin	9	22	33	41	48	54
Quartz	19	14	11	8	6	4
Feldspar	72	64	56	51	46	42

The samples with high content of Kilimanjaro quartz are found to be weaker in bending strength. Low strength is accredited by the development and propagation of cracks which is due to quartz and glass thermal mismatch (Iqbal & Lee, 2000). Similarly, increasing Pugu kaolin is leading to the increase in alumina content, hence increase in strength this argument is well supported by the chemical composition study whereby Pugu kaolin is found to possess the highest amount of 34.7 % alumina content.

Table 5: Insulation Resistance and Mechanical Strength of Prepared Batches

Batches	Insulation Resistance at the injection of 1000V (M Ω)	Linear	Bulk density (g/cm ³)	Water	Strength (MPa)
		Shrinkage (%)		absorption (%)	
C1	25592	11.917	2.855	0.21	33.52
C2	42750	13	2.213	0.11	9.421
C3	11720	12	2.288	0.32	36.72
C4	10404	11	2.028	0.22	37.86
C5	34812	9.5	1.988	0.77	53.53
C6	16844	10.167	1.823	1.24	46.88

Average taken from ten samples per each Mix Proportion

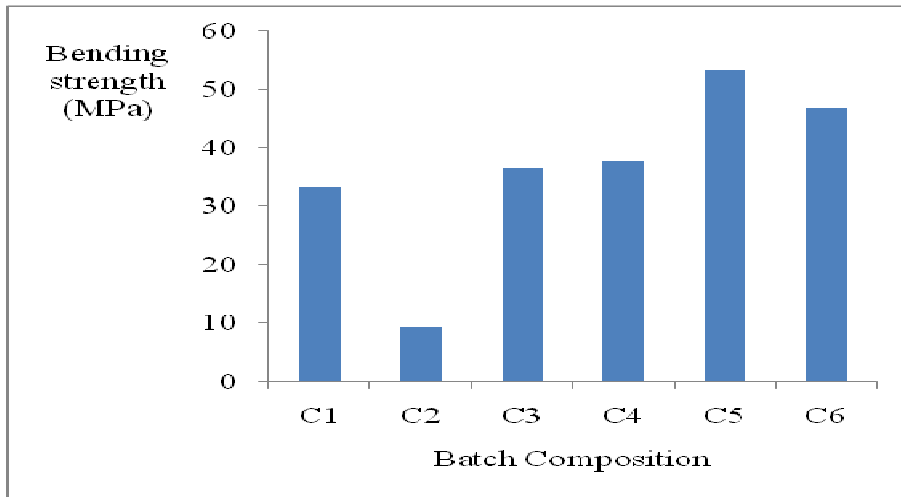


Figure 4: Bending Strength of Samples from different Mix Proportion

Insulation Resistance

The samples of mix proportion “C2” with 14 wt% Pugu kaolin, 22 wt% Kilimanjaro quartz, and 64 wt% Kilimanjaro feldspar revealed the highest value of insulation resistance of 42750 MΩ at the maximum injection 1000 V but this sample was characterized by very poor bending strength. The lower strength is attributed by the cracks and flaw developed to samples of this mix proportion. For this reason, to take care of both the mechanical strength and insulating property mix proportion “C5” is acceptable so long as it has shown good performance in both mechanical strength and electrical insulation and the samples of this mix proportion were free from cracks and flaws (Fig.5) and Table 5. The International Electrical Testing Association recommends the minimum insulation resistance of 500 MΩ at the injection 1000 V. With this test sample “C5” the insulation resistance of 34812 MΩ at the injection of 1000V is achieved.

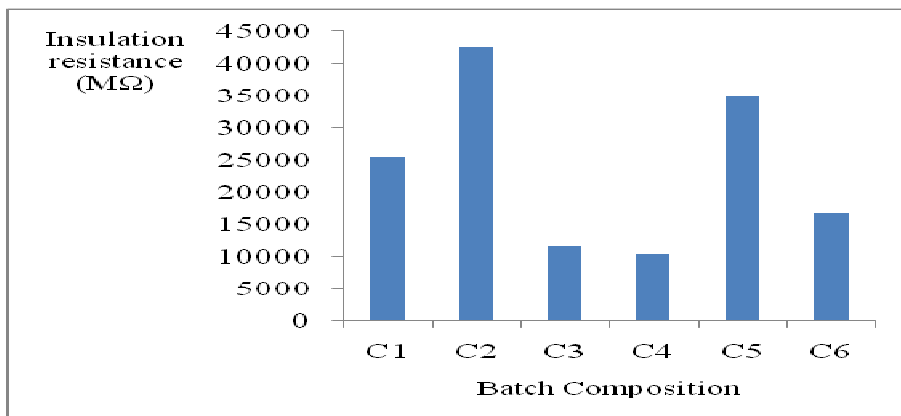


Figure 5: Insulation Resistance of Samples of different mix proportion at injection of 1000 Volts

Linear Shrinkage

The linear shrinkage percentage of porcelain is found to decrease with increase in kaolin content while reducing the content of quartz and feldspar as indicated in Tables 5 and (Fig.6). This is attributed by the nature of kaolin as a clay material. The experienced shrinkage is due to drying and firing of the porcelain. The samples with low shrinkage values are accompanied with low bulk density and high strength comparing to those with high values of shrinkage.

Bulk Density

The bulk density was decreasing with a decrease in the content of Kilimanjaro feldspar. This is because feldspar is low melting minerals and serves to lower the temperature at which viscous liquid forms. The liquid phase reacts with other components and fills the pores leading to its densification (Iqbal & Lee, 2000). Thus, with high content of Kilimanjaro feldspar density is high which continued to decrease as the content of Kilimanjaro feldspar is reduced (Fig.7) and Table 5. This trend in bulk density is also accompanied with increasing the content of Pugu kaolin while reducing the content of Kilimanjaro quartz. On the other hand, bulk density is found to increase with increase in linear shrinkage which suggests that the interparticles distance between particles has been reduced and pores have been minimized leading to increase in density.

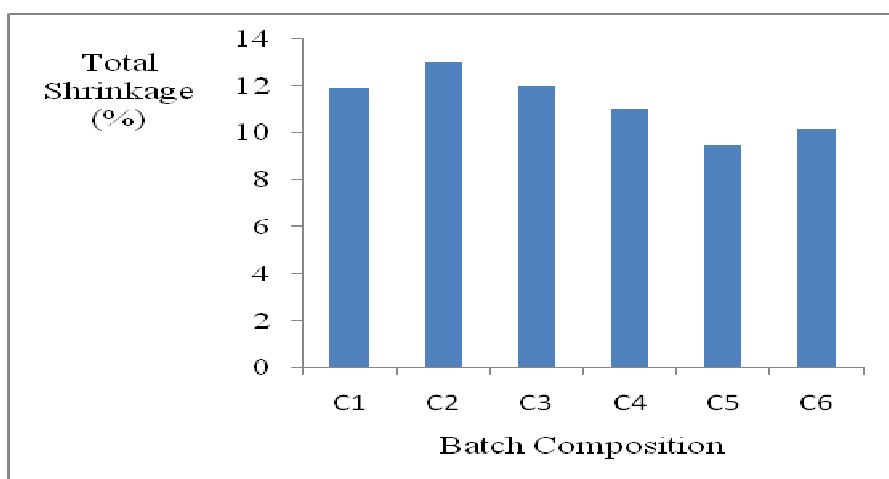


Figure 6: Linear Shrinkage of Samples from different Mix Proportion

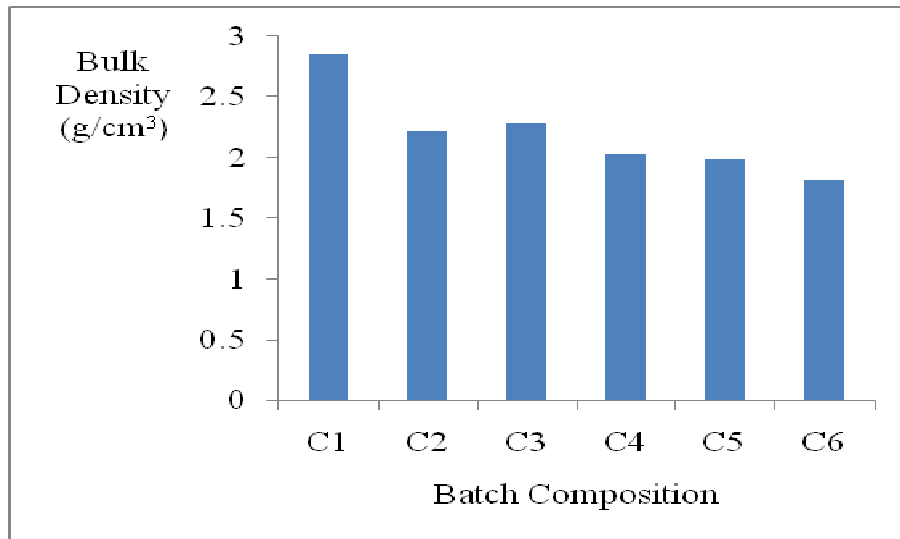


Figure 7: Bulk density of samples of different mix proportion

Water Absorption

Water absorption is observed to decrease with the increasing kaolin content and a reduction of feldspar as indicated in Tables 5 and (Fig.8). Feldspar as a flux form viscous liquid which permits better flow of the mixture by doing so it bridges the pores and reduces water absorption. In this regard, reducing the content of feldspar creates more porosity hence higher water absorption.

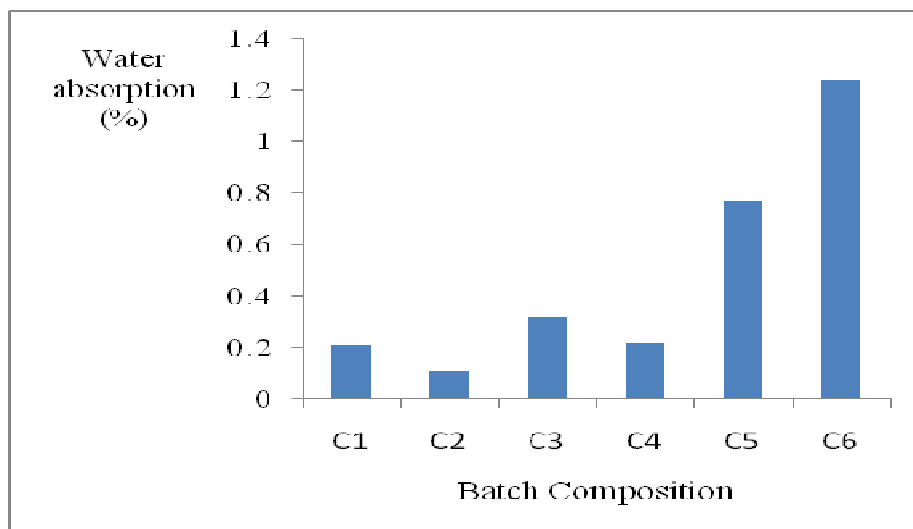


Figure 8: Water absorption of samples of different mix proportion

2.4. Conclusion

Electrical insulation and bending strength are increasing with reduction of Kilimanjaro quartz content while increasing the content of Pugu kaolin. Essentially, increasing Pugu kaolin beyond 48 wt% and reducing Kilimanjaro quartz content below 6 wt% is leading to reduction of electrical insulation and bending strength. In addition, the mix with constituent's percentage composition of 48 wt% Pugu kaolin, 46 wt% Kilimanjaro feldspar and 6 wt% Kilimanjaro quartz is possessing the highest strength of 53.525 MPa. This mix proportion shows the electrical insulation resistance of 34812 M Ω at injection of 1000 V where the required minimum insulation resistance at the injection of 1000 DC voltage is 500 M Ω . To this end, the use of Pugu kaolin for production of electrical insulators is encouraged since the formation of mullite phase improves the mechanical strength and insulation resistance. In this aspect, these results are indicating that a high quality electrical insulator can be achieved from Tanzanian originated ceramic materials.

2.5. Acknowledgements

With much pleasure, I extend my sincere thanks to all those who assisted me in all the activities leading to this work both in their individual and institutional capacities. Firstly, I acknowledge the financial support from the government of The United Republic of Tanzania by funding my scholarship. My gratitude goes to the management of the Nelson Mandela African Institution of Science and Technology (NM-AIST). I also thank the staff of the Department of Material Sciences for their maximum cooperation rendered to this work.

CHAPTER THREE

The Effect of Firing Temperature on Electrical Insulation Resistance and Bending Strength of Tanzanian Local Ceramic Materials²

Abstract

This study focused on the investigation of influence of firing temperature on electrical insulation resistance and bending strength of Tanzanian originated ceramic materials. For exploring the bending strength and electrical insulation resistance of porcelain, different mix proportions and firing temperatures are adopted in this study. Six formulations of three raw materials, namely: Pugu kaolin, quartz supplied by the Kilimanjaro Industrial Development Trust (KIDT), and Kilimanjaro feldspar are used in experimental design. The green body preparation is done by slip casting, dried, and fired at the maximum temperatures of 1100, 1200, and 1300 °C at the rate of 100 °C/h with one hour holding time.

The highest electrical insulation resistance and bending strength is revealed to samples fired at 1200 °C. Test sample with 33 wt% Pugu kaolin, 11 wt% Kilimanjaro quartz, and 56 wt% Kilimanjaro feldspar fired at 1200 °C experienced highest insulation resistance of 205800 MΩ at the injection of 1000 V. This test sample corresponds to the strength of 65.358 MPa and water absorption of 0.01 %.

Keywords: Tanzania, electrical insulation resistance, bending Strength, kaolin, quartz, feldspar

3.1. Introduction

Electrical conduction is important to allow the passage of electricity to potential user of electricity from the generating point, but electrical insulation is strongly recommended to prevent electrical shocks and assure safety of the personnel in carrying out electrical works (AEMIC, 2014). The insulators must have high dielectric strength, high resistivity, low loss factor, and good mechanical properties (Islam *et al.*, 2004). The most important properties which must be taken into consideration are mechanical strength and dielectric strength. Sometimes the insulation resistance test is carried out instead of dielectric strength test ((NETA), 2007). In the

² (Manuscript)

consideration of mechanical strength the Japan Industrial Standard and their counterpart require a minimum strength of 65 MPa. On other the hand, the International Electrical Testing Association recommended minimum values of insulation resistance at specific direct current test voltages.

Porcelain is the most commonly used material for overhead insulator in recent years (Electrical4u, 2014). It is the triaxial mixture of kaolin, quartz and feldspar which is heat treated to obtain final hard and glazed porcelain insulator materials (A.O. Oladiji, 2010; Bouzidi *et al.*, 2013; Bragança & Bergmann, 2003; Callister & Rethwisch, 2011; Carty & Senapati, 1998; Das & Dana, 2003; Ece & Nakagawa, 2002; Olupot, 2006). The firing temperatures for porcelain usually range from 1200 to 1400 °C (A.O. Oladiji, 2010).

Electrical and mechanical property is influenced by the mix proportional of raw materials. It has been proven that increasing quartz content leads to decrease of mechanical strength and dielectric property. Current researches pointed out the improvement of strength and electrical properties by replacing some percentage of quartz with alumina (Ramaswamy *et al.*, 2005; Sedghi *et al.*). In the present work, the theoretical batch calculation is carried out with the intention of reducing the quartz content while maximizing the content of alumina.

Apart from thinking about mix proportion of raw materials, the final performance of ceramic products like insulators is governed by firing temperatures. Desired microstructure and phased, responsible for a particular purpose is determined by firing temperature adopted. Firing process serves three fundamental functions: to substantially reduce the number of pores in ceramic; to increase the density; and to bond together the individual materials grains into a strong hard mass (No III, 1996). Heating rate and soaking time are important factors to be taken into consideration, however, in this study only single heating rate and soaking time is adopted.

Thermal behaviour of silica and alumina shows that mullite phase ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) starts to form at 1100 °C. At a temperature above 1100 °C mullite crystals continue to grow and the needles appear. Increasing the temperature to 1200 °C partial dissolution of quartz phase occurs. At 1350 °C there is a considerable amount of glassy phase, which is formed (Islam *et al.*, 2004).

The aim of this study is to investigate the applicability of local ceramic materials for their potential in the production of electrical insulators for high voltage application based on mix proportions of raw materials with the major intention of minimizing quartz content so as to come up with an optimum mix proportion of raw material which will produce a porcelain with high

electrical insulation and high mechanical strength. On the other hand, this work is focussing on determination of required firing temperature of the blends formed from the materials under study.

3.2. Materials and Methods

The materials used in this study are: Pugu kaolin, quartz supplied by the Kilimanjaro Industrial Development Trust (KIDT), and Kilimanjaro feldspar.

Table 6: Mix proportion of Raw Materials (wt%)

	C1	C2	C3	C4	C5	C6
Pugu kaolin	9	22	33	41	48	54
Quartz	19	14	11	8	6	4
Feldspar	72	64	56	51	46	42

Quartz and feldspar were separately crushed by a Jaw crusher of type JC-4 and made in powder form by edge runner of size 12008 type N4S. A sleep casting technique was used to produce test samples. In each batch 10 spherical samples of 60 mm length and 10 mm diameter were prepared. A sleep of from a 3Kg batch was produced by adding to it 3 litres of water, followed by wet milling in a pot mill for 24 hours to obtain a uniform mixture.

After milling the mixture was passed through sieve No.200 and then magnetic filtered to remove iron. Then the slurry was put into a gypsum pot for two days to suck water so as to obtain a filtered cake. The filtered cake was made in a plastic form by adding a small amount of water to favour the desired consistency for forming using the prepared moulds to give the cylindrical shape of 60 mm length and 10 mm diameter. Prepared samples were air dried for two days followed by drying at 60 °C before bisque firing at 800 °C. The dried samples were then fired at the maximum temperature of 1300 °C in an industrial electric furnace at the approximate rate of 100 °C per hour with 1 hour holding time.

Linear shrinkage was determined by taking the percentage difference between the initial green body length and fired length of samples.

$$\text{Total linear shrinkage} = \frac{L_w - L_f}{L_w}$$

L_w = Wet length of a sample

L_f = Fired Length of a sample

Bulk density was determined by measuring the mass and volume of samples.

$$\text{Bulk density}(\rho) = \frac{\text{Mass}}{\text{Volume}}$$

Water absorption of fired bodies was determined by soaking method whereby the weight of the test samples before soaking and measuring the weight of samples after 24 hours soaking.

$$\text{Water absorption} = \frac{W_s - W_d}{W_d}$$

W_s = Soaked weight of sample

W_d = Dry weight of sample

Insulation resistance of fired bodies was determined by Megohmmeter AEMC instrument model 1060 by subjecting the test samples to 1000 DC voltage for 1 minute and recording the displayed value of insulation resistance.

The bending strength of samples was determined by three point bending test according to JIS standard. Samples dimensions and the load at failure in Kgf were recorded for ten test samples.

$$\text{Bending Strength} = \frac{F_s * L}{\pi R^3}$$

R = Radius of sample

L = Span (center to center from the support)

F_s = Load at Failure

3.3 Results and Discussion

3.3.1. Bending Strength

Bending strength of porcelain is remarkably increased by firing at higher temperature (Fig.9). The lowest trend in bending strength is noted for samples fired at 1100 and 1300 °C. On the other hand, the highest trend in bending strength is revealed to samples fired at 1200 °C. Sharp increase in bending strength is observed for samples fired at 1200 °C which dropped at 1300 °C. At 1100 °C the bending strength of samples was low with the maximum of 48.199 MPa to samples with mix proportion “C2” of 22 wt% Pugu kaolin, 14 wt% Kilimanjaro quartz, and 64 wt% Kilimanjaro feldspar. The highest strength of 72.677 MPa is revealed to samples fired at

1200 °C with a mix proportion “C1” of 9 wt% Pugu kaolin, 19 wt% Kilimanjaro quartz, and 72 wt% Kilimanjaro feldspar. At 1300 °C the maximum strength is found to be 53.528 MPa to samples with mix proportion “C5” of 48 wt% Pugu kaolin, 6 wt% Kilimanjaro quartz, and 46 wt% Kilimanjaro feldspar.

The sharp increase in bending strength from the samples firing temperature of 1100 °C to 1200 °C is attributed by enhanced vitrification which is effectively happened at 1200 °C. Water absorption is found to be minimized at this particular firing temperature, which is accompanied by higher insulation resistance, making the implication that amount of closed pores have been minimized to a greater extent. The bending strength is found to decrease at 1300 °C which implies that the deterioration of the samples took place. This is well agrees with the results of water absorption, which was increasing at this particular temperature.

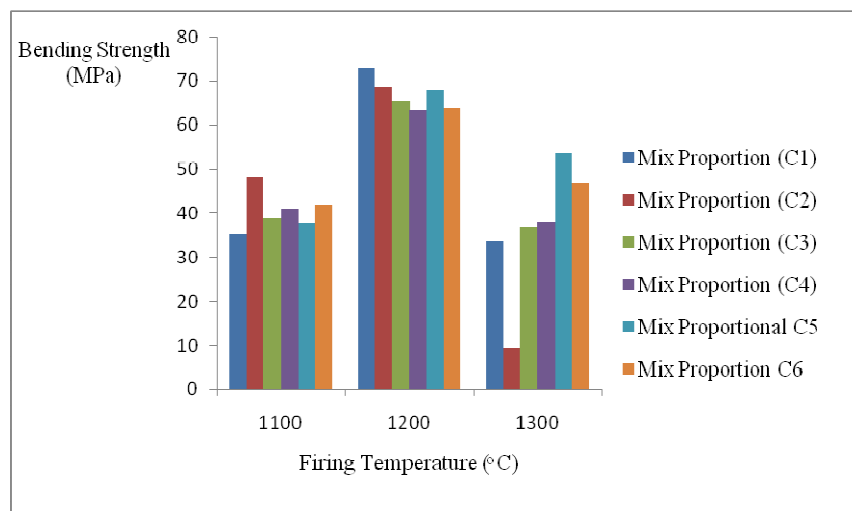


Figure 9: Bending strength of samples of different firing temperature and mix proportion

3.3.2. Insulation Resistance

The insulation resistance of samples fired at 1100 °C and 1300 °C was found to be low. High trend in insulation resistance is noted to samples fired 1200 °C with the highest value of 205800 MΩ at the injection of 1000 DC volts to samples with mix proportion “C3” (Fig.10) and Table 7. According to the acceptance test specification for electrical power distribution equipment and system set by the International Electrical Testing Association the minimum of 500 MΩ is required for a minimum test voltage of 1000 DC voltage .

Table 7: Insulation resistance in (MΩ) with respect to firing temperatures and mix proportion

	C1	C2	C3	C4	C5	C6
Firing Temperature (°C)						
1100	2375	11230	819	4230	2720	13880
1200	48400	19410	205800	38220	70700	152200
1300	25592	42750	11720	10404	34812	1684

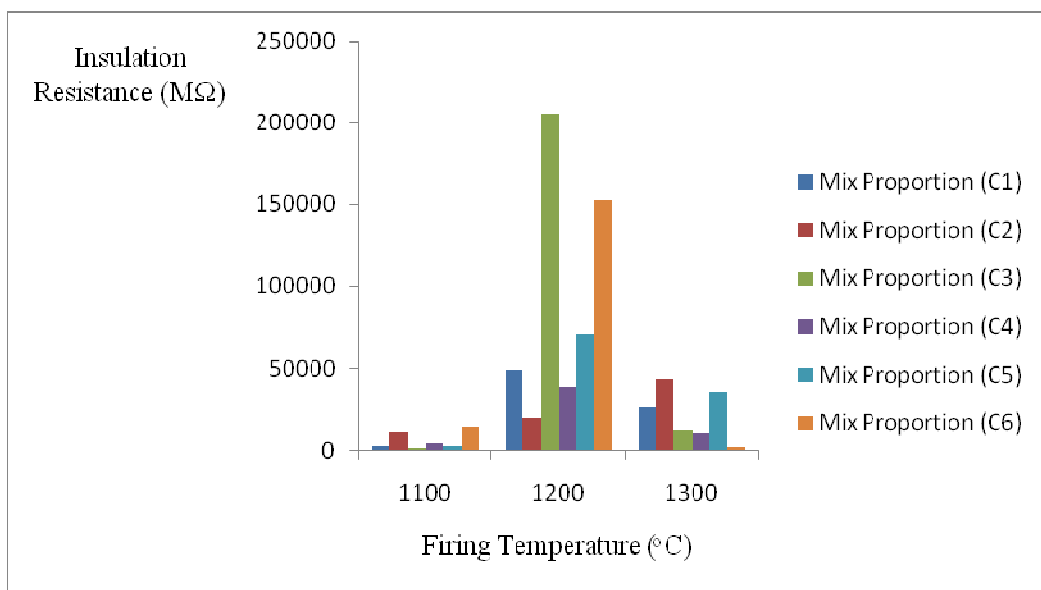


Figure 10: Insulation Resistance of samples from different firing temperature and mix proportion

3.3.3. Linear shrinkage

Linear shrinkage of samples is found to increase with firing temperature refer (Fig.11). Shrinkage occurs during drying as liquid between the particles is removed and the interparticle distance decreases. Taking into consideration the stages in sintering process which involve vapour transportation, surface diffusion, lattice volume diffusion grain growth diffusion and dislocation motion Linear shrinkage to increase with firing temperature is attributed by densifying mechanism such as grain boundary diffusion, lattice diffusion from the grain boundary and plastic flow by dislocation motion which remove materials from the grain boundary region leading to shrinkage.

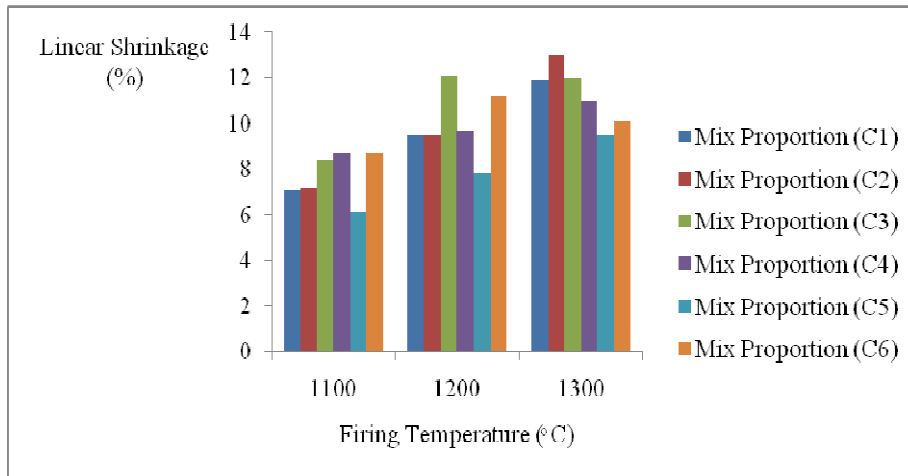


Figure 11: Linear shrinkage of samples from different firing temperature and mix proportion

3.3.4. Water absorption

The results show at the temperature above 1200 °C water absorption is approximately equal to zero and started to increase at 1300 °C. This abnormality to samples fired at 1300 °C is caused by increase in closed pores which resulted from the release of oxygen which was entrapped in the pores and bloating. The bending strength and insulation resistance is lower at 1300 °C compare to those samples fired at 1200 °C. This is attributed by the abnormality which occurred to the samples fired at 1300 °C. Furthermore, the highest water absorption at 1100 °C is due to excessive amount of closed and open pores as viritification process didn't take place at this firing temperature. Water absorption is the criterion to indicate the reduction of porosity. The lower water absorption is associated with the highest strength and electrical insulation resistance.

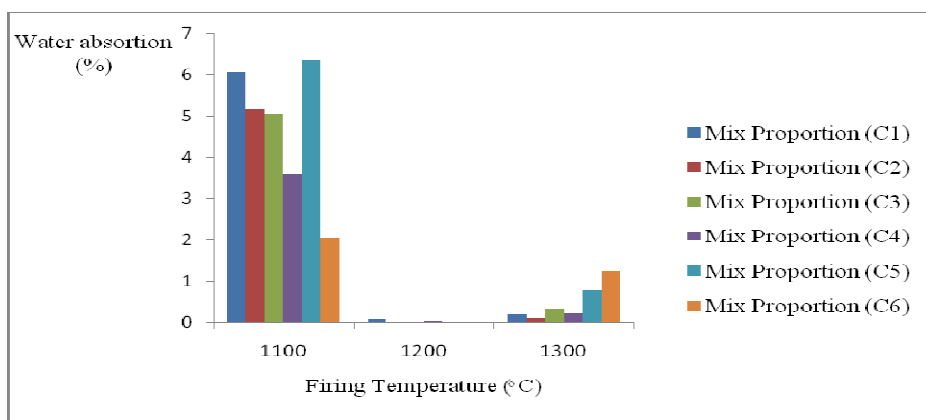


Figure 12: Water absorption at different mix proportion and firing temperature

3.3.5. Bulk Density

There is no great variation in bulk density noted to samples of different mix proportion fired at 1100, 1200 and 1300 °C. The highest trend in bulk density is revealed to samples fired at 1300 °C with a maximum 2.855 g/cm³. This trend of bulk density is resulted from the decrease in volume of samples as a consequence of increased linear shrinkage (Fig.13).

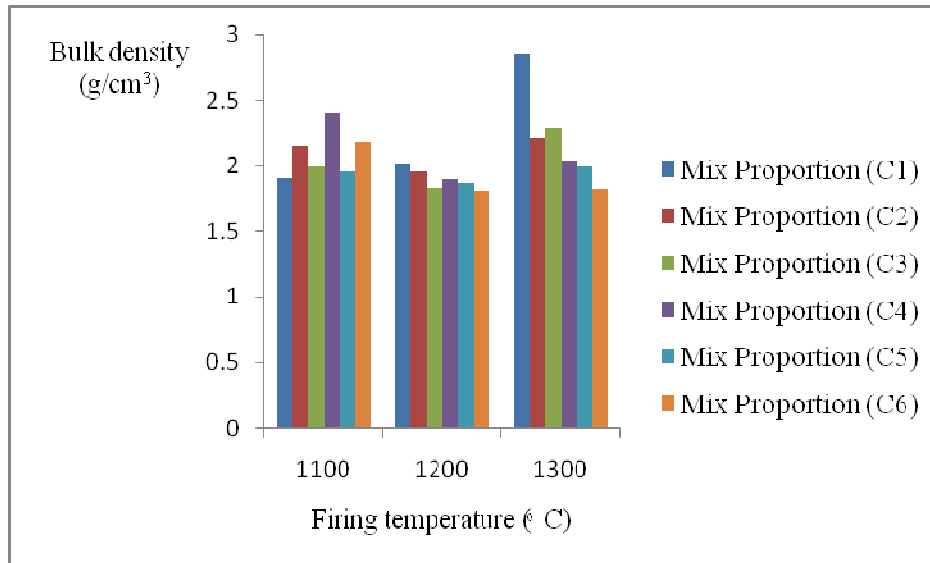


Figure 13: Bulk density of samples from different firing temperature and mix proportion

3.4. Conclusion

The analysis of data showed that electrical insulation resistance and bending strength were related to water absorption. Samples fired below 1200 °C were characterized by high water absorption indicating the presence of open porosity which lead to lower electrical insulation resistance and bending strength. Additionally, samples fired at 1300 °C possessed lower electrical insulation and bending strength compare to those fired 1200 °C. The optimum firing temperature the porcelain body studied is 1200 °C using the heating rate of 100 °C/h and 1 hour holding time. On the other hand, the optimum mix proportion for materials studied is that of 33 wt% Pugu kaolin, 11 wt% Kilimanjaro quartz and 56 wt% Kilimanjaro feldspar fired at 1200 °C. At this optimum mix proportion and firing temperature the highest insulation resistance of 205800 MΩ is achieved at the injection of 1000 DC volts. This test sample corresponds to the bending strength of 65.358 MPa and water absorption of 0.01 %. According to the International

Electrical Testing Association a minimum electrical insulation resistance of 500 MΩ is required at the injection of 1000 DC voltage. The minimum strength and water absorption of 65 MPa and 0.01 % respectively is recommended.

3.5. Acknowledgements

I wish to express my deepest appreciation to those who have provided me encouragement and moral support towards the completion of this work. I would like to convey my thanks to the management of Kilimanjaro Industrial development trust (KIDT) for giving access to their industrial processing equipments. Lastly, I extend my appreciation to the Government of United Republic of Tanzania for funding my studies and this research work.

CHAPTER FOUR

4.1 GENERAL DISCUSSION

For production of insulators the composition purity of raw materials requires, in clays (Fe_2O_3 , TiO_2) <3.5 %; in quartz; SiO_2 > 99 %; and in feldspar Fe_2O_3 < 1 %; (K_2O , Na_2O) > 12 % (Meng *et al.*, 2012). Chemical composition of raw materials used in this study meets the composition purity requirement for raw materials see the result in (Table 2). Kilimanjaro Feldspar composed of relatively small amounts of detrimental oxide, which meets the percentage purity requirements of raw materials for electrical insulator application as reported by (Meng, 2012) (Meng *et al.*, 2012). The amount of flux ($\text{K}_2\text{O}.\text{Na}_2\text{O}$) in feldspar is 14.94 %, which complies with the composition purity requirement for raw materials since ($\text{K}_2\text{O}.\text{Na}_2\text{O}$) has to be greater than 12 %. After being beneficiated quartz contains is revealed to contain 97.3 % of SiO_2 which is close to the required composition purity of 99 % SiO_2 .

Moreover, the X-ray diffraction (XRD) results showed that in feldspar the raw materials contains albite, and microcline, tridymite and quartz as other phases developed on sintering as indicated in Fig. 1. In the XRD- crystallographic study of quartz the maximum peak is revealed around 25 (2θ -degree) which is typical diffraction angle for quartz. The 2.3 % loss on ignition in quartz raw materials in Table 1 demonstrate that quartz content was not 100 %, which implies there was some water contained in quartz. On the other hand, the study made to Pugu kaolin by (Hashimu Hamisi, 2014) has revealed the development of mullite phase when fired at 1400 °C. Mullite is very important phase in promoting the mechanical and dielectric properties.

In general the result showed electrical insulation resistance and bending strength was increasing with increase in samples firing temperature. Firing the samples at 1300 °C is leading to reduction of insulation resistance and bending strength. The maximum electrical insulation resistance and bending strength is revealed for the samples fired at 1200 °C. This phenomenon is in line with the trend of water absorption of samples. Water absorption of the samples fired at 1200 °C was low and started to rise for the sample firing temperature of 1300 °C. Higher temperatures lead to an increase in water absorption. This increase can be explained by the decomposition of oxides allowing the release of free oxygen similar to the study made by (Bragança & Bergmann, 2003).

Bloating also occurred at higher temperatures and leads to rise of water absorption and decrease the insulation resistance and bending strength as a result of increase in pore size.

On the other hand, samples fired at 1300 °C with a constituent's percentage composition of 48 wt % Pugu kaolin, 46 wt% Kilimanjaro feldspar and 6 wt% quartz possessed the highest strength of 53.525 MPa, however, this value of strength is less than the minimum recommended strength of 65 MPa as per JIS standard. This low strength compared to the minimum recommended value is caused by flaw and cracks developed at this particular firing temperature. It is therefore necessary to investigate the impact of heating rate and holding time on firing the porcelain samples produced from the raw material studied. Moreover, this mix proportion showed electrical insulation resistance of 34812 MΩ at injection of 1000 V. Comparing these results with standard specification, insulation resistance is in line with the recommendation of the International Electrical Testing Association, which requires 500 MΩ at the injection of 1000 DC voltage.

Additionally, the analysis of data showed that samples fired below 1100 °C were characterized by high water absorption indicating the presence of open porosity which lead to lower electrical insulation resistance and bending strength. On the other hand, samples fired at 1300 °C possessed lower electrical insulation and bending strength compared to those fired 1200 °C. This phenomenon for samples fired at 1300 °C is accredited by the formation of more closed pores formed as a result of oxygen release from decomposition of oxides.

Moreover, samples fired at 1200 °C showed the highest value of insulation resistance and bending strength. Water absorption of samples fired at this temperature is very low. This implies that the optimum firing temperature of porcelain studied is 1200 °C using the heating rate of 100 °C/h and 1 hour soaking time, however, good performance can be achieved from the temperature ranging from 1200 and 1300 °C.

The electrical insulation resistance of 205800 MΩ is attained at the injection of 1000 DC volts to samples with 33 wt% Pugu kaolin, 11 wt% Kilimanjaro quartz, and 56 wt% Kilimanjaro feldspar fired at 1200 °C using the heating rate of 100 °C/h and 1 hour holding time. According to the International Electrical Testing Association a minimum electrical insulation resistance of 500 MΩ is required at the injection of 1000 DC voltage. This test sample corresponds to the highest strength of 65.358 MPa and water absorption of 0.01 %. According to JIS standard the minimum strength and water absorption of 65 MPa and 0.01 % respectively is recommended.

4.2 CONCLUSION

The ceramic materials under study are processing essential properties of electrical insulation application. This is proved by the test results obtained from the blends produced from raw materials under study. The study made to explore the properties of raw materials pointed out that Pugu kaolin contains mullite phase, which is an essential phase for upholding electrical insulation property of porcelain.

The results of test samples fired at 1300 °C indicate that increasing the content of Pugu kaolin while reducing the content of the quartz is leading to increase in electrical insulation resistance and bending strength. In addition, the optimum mix proportion at this firing temperature is that of 48 wt% Pugu kaolin, 46 wt% Kilimanjaro feldspar and 6 wt% Kilimanjaro quartz. The strength highest strength of 53.525 MPa and the electrical insulation resistance of 34812 MΩ at the injection of 1000 DC voltage is achieved at this mix proportion and firing temperature.

Furthermore, samples fired at 1200 °C achieved the highest electrical insulation resistance and bending strength compare to those fired at 1100 and 1300 °C. The highest insulation resistance of 205800 mega ohms at the injection of 1000 DC test voltage and bending strength of 65.358 MPa is attained by samples with mix proportion of 33 wt% Pugu kaolin, 11 wt% Kilimanjaro quartz, and 56 wt% Kilimanjaro feldspar fired at 1200 °C. In addition, this test samples correspond to 12.1 % linear shrinkage, density of 1.829 g/cm³, and water absorption of 0.01 %.

To conclude, good results is achieved by samples with 33 wt% Pugu kaolin, 11 wt% Kilimanjaro quartz, and 56 wt% Kilimanjaro feldspar. This mix proportion corresponds to the firing temperature of 1200 °C using the heating rate of 100 °C/h. In this regard, the results are indicating that high quality electrical insulators can be achieved from Tanzanian originated ceramic materials.

4.3 RECOMMENDATIONS

There are a number of factors to be taken into account for the achievement ceramic products for different applications. This includes optimization of particle sizes of raw materials, the effect of heating and cooling ramp, and the effect of holding time, microstructure and structural phases which are produced, and fabrication method adopted. This dissertation is focused on the effect of mix proportion of raw material and firing temperature only. With this study the following are the recommendations.

Focusing on this study, effective performance in both mechanical and insulation property is observed to samples fired at 1200 and 1300 °C. Further study has to be done for the temperature ranging between 1200 and 1300 °C. Within this range of firing temperature unique properties can be achieved.

The following are recommendations for future works.

- Investigation of the effect of heating ramp on mechanical strength and dielectric properties.
- The study on the effect of holding time.
- The study on the microstructural and different phases which are produced on firing the mix produced from these raw materials.
- To carry out dielectric strength to know exactly the breakdown voltage of porcelain produced from these materials
- Effect of particle size on electrical insulation and mechanical strength

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APPENDICES

Appendix 1: Batch calculations

Step: 1

- Identification of raw materials involved
 - (i) Pugu kaolin : $\text{Al}_2\text{O}_3(\text{Si}_2\text{O}_5)(\text{OH})_4$
 - (ii) Quartz : SiO_2
 - (iii) Feldspar : KAlSi_3O_8

Step: 2

- Establish the chemical equation for each raw material
 - (i) Pugu kaolin: $\text{Al}_2\text{O}_3(\text{Si}_2\text{O}_5)(\text{OH})_4 \longrightarrow \text{Al}_2\text{O}_3 + 2\text{SiO}_2 + \text{H}_2\text{O}$
 - (ii) Quartz : SiO_2
 - (iii) Pugu kaolin: $\text{KAlSi}_3\text{O}_8 \longrightarrow \text{K}_2\text{O} + \text{Al}_2\text{O}_3 + 6\text{SiO}_2$

Step: 3

- Calculation of number of moles from each raw material
 - Important data : - Chemical components of interest (SiO_2 , Al_2O_3 & K_2O)
 - Molar mass of each component
 - Chemical composition of raw materials

Table 8: Chemical Composition of raw materials (For Pugu kaolin-study made by (Hashimu Hamisi, 2014)

	Feldspar (%)	Quartz (%)	Pugu Kaolin (%)
SiO_2	53	97.7	48.9
Al_2O_3	19.1	-	34.7
Fe_2O_3	0.24	-	2.22
TiO_2	-	-	0.6
CaO	0.09	-	0.01
MgO	0.01	-	0.11
Na_2O	2.04	-	0.07
K_2O	12.9	-	0.34
Loss on ignition (LOI)	12.62	2.3	13.05

• Component of interest	Molar mass
SiO ₂	60.09
Al ₂ O ₃	101.96
K ₂ O	94.196

Table 9 : Moles of components from each raw material

Raw Materials	Moles of chemical component of interest		
	SiO ₂	Al ₂ O ₃	K ₂ O
Pugu kaolin	0.815	0.3403	0
Quartz	1.6258	0	0
Feldspar	0.882	0.1873	0.1369

Notes: Each chemical component of interest are represented in $\frac{\text{moles}}{\text{litre}}$ or $\frac{\text{grams}}{\text{litre}}$

Step: 4

- Required number of moles of raw material

(a) SiO₂

Pugu kaolin: 1M (Pugu kaolin) \longrightarrow 2M (SiO₂)..... From chemical equation
 ? (Pugu kaolin) \longleftarrow 0.815M (SiO₂)..... From the raw materials
 = 0.4075M

Quartz: 1M (Quartz) \longrightarrow 1M (SiO₂)..... From chemical equation
 ? (Quartz) \longleftarrow 1.6258M (SiO₂)..... From the raw materials
 = 1.6258M

Feldspar: 1M (Feldspar) \longrightarrow 6M (SiO₂)..... From chemical equation
 ? (Feldspar) \longleftarrow 0.882M (SiO₂)..... From the raw materials
 = 0.294M

(b) Al₂O₃

Pugu kaolin: 1M (Pugu kaolin) \longrightarrow 1M (Al₂O₃) From chemical equation

$$\begin{aligned} &? \text{ (Pugu kaolin)} \longleftarrow 0.3403\text{M (Al}_2\text{O}_3)\text{..... From the raw materials} \\ &= 0.3403\text{M} \end{aligned}$$

$$\text{Quartz: } = 0$$

$$\begin{aligned} \text{Feldspar: } 2\text{M (Feldspar)} &\longrightarrow 1\text{M (Al}_2\text{O}_3) \text{From chemical equation} \\ ? \text{ (Feldspar)} &\longleftarrow 0.1873\text{M (Al}_2\text{O}_3)\text{..... ..From the raw materials} \\ &= 0.3746\text{M} \end{aligned}$$

(c) K₂O

$$\text{Pugu kaolin} = 0$$

$$\text{Quartz: } = 0$$

$$\begin{aligned} \text{Feldspar: } 2\text{M (Feldspar)} &\longrightarrow 1\text{M (K}_2\text{O)}\text{..... From chemical equation} \\ ? \text{ (Feldspar)} &\longleftarrow 0.1369\text{M (K}_2\text{O)}\text{..... From the raw materials} \\ &= 0.2739\text{M} \end{aligned}$$

2.1.1 Equations

Let:

$$\text{Moles of Pugu kaolin} = x$$

$$\text{Moles of Quartz} = y$$

$$\text{Moles of Feldspar} = z$$

$$0.4075x + 1.6258y + 0.294z = \text{Total moles of "SiO}_2\text{" required in a batch}$$

$$0.3403x + 0.y + 0.3746z = \text{Total moles of "Al}_2\text{O}_3\text{" required in a batch}$$

$$0. x + 0. y + 0.294z = \text{Total moles of "K}_2\text{O" required in a batch}$$

2.1.2 Molar mass

$$\text{Pugu kaolin: Al}_2(\text{Si}_2\text{O}_5)(\text{OH})_4 = 258.12\text{g}$$

$$\text{Quartz: SiO}_2 = 60.09\text{g}$$

$$\text{Feldspar: KAlSi}_3\text{O}_8 = 278.34\text{g} \quad : \text{ Note: Mole} = \text{Mass/Molar mass}$$

Step: 5

Table 10 : Determination of batches

Batch	Components	Wt% of components	Number of moles of components
C1	SiO ₂	80	1.3313
	Al ₂ O ₃	25	0.2452
	K ₂ O	15	0.1592
C2	SiO ₂	75	1.2482
	Al ₂ O ₃	30	0.2924
	K ₂ O	15	0.1592
C3	SiO ₂	70	1.1648
	Al ₂ O ₃	35	0.3433
	K ₂ O	15	0.1592
C4	SiO ₂	65	1.0817
	Al ₂ O ₃	40	0.3923
	K ₂ O	15	0.1592
C5	SiO ₂	60	0.9985
	Al ₂ O ₃	45	0.4414
	K ₂ O	15	0.1592
C6	SiO ₂	55	0.9153
	Al ₂ O ₃	50	0.4904
	K ₂ O	15	0.1592

For batch C1

$$0.4075x + 1.6258y + 0.294z = 1.3313$$

$$0.3403x + 0.y + 0.3746z = 0.2452$$

$$0. x + 0. y + 0.294z = 0.1592$$

$$x = 0.0807$$

$$\text{Mass} = 20.83 \quad \text{wt\%} = 9 \quad \text{Pugu kaolin}$$

$$y = 0.6935 \quad = 41.67 \quad = 19 \text{ Quartz}$$

$$z = 0.5812 \quad = 72.13 \quad = 72 \text{ Feldspar}$$

For batch C2

$$0.4075x + 1.6258y + 0.294z = 1.2481$$

$$0.3403x + 0.y + 0.3746z = 0.2942$$

$$0. x + 0. y + 0.294z = 0.1592$$

$$x = 0.2247 \quad \text{Mass} = 56.79 \quad \text{wt\%} = 22 \quad \text{Pugu kaolin}$$

$$y = 0.6063 \quad = 36.43 \quad = 14 \quad \text{Quartz}$$

$$z = 0.5812 \quad = 161.77 \quad = 64 \quad \text{Feldspar}$$

For batch C3

$$0.4075x + 1.6258y + 0.294z = 1.1648$$

$$0.3403x + 0.y + 0.3746z = 0.3433$$

$$0. x + 0. y + 0.294z = 0.1592$$

$$x = 0.3690 \quad \text{Mass} = 95.25 \quad \text{wt\%} = 33 \quad \text{Pugu kaolin}$$

$$y = 0.5189 \quad = 31.18 \quad = 11 \quad \text{Quartz}$$

$$z = 0.5812 \quad = 161.77 \quad = 56 \quad \text{Feldspar}$$

For batch C4

$$0.4075x + 1.6258y + 0.294z = 1.0817$$

$$0.3403x + 0.y + 0.3746z = 0.3923$$

$$0. x + 0. y + 0.294z = 0.1592$$

$$x = 0.5130 \quad \text{Mass} = 132.42 \quad \text{wt\%} = 41 \quad \text{Pugu kaolin}$$

$$y = 0.4316 \quad = 25.93 \quad = 8 \quad \text{Quartz}$$

$$z = 0.5812 \quad = 161.77 \quad = 51 \quad \text{Feldspar}$$

For batch C5

$$0.4075x + 1.6258y + 0.294z = 0.9985$$

$$0.3403x + 0.y + 0.3746z = 0.4414$$

$$0. x + 0. y + 0.294z = 0.1592$$

$$x = 0.6573 \quad \text{Mass} = 169.66 \quad \text{wt\%} = 48 \quad \text{Pugu kaolin}$$

$$y = 0.3443 \quad = 20.69 \quad = 6 \quad \text{Quartz}$$

$$z = 0.5812 \quad = 161.77 \quad = 46 \quad \text{Feldspar}$$

For batch C6

$$0.4075x + 1.6258y + 0.294z = 0.9153$$

$$0.3403x + 0. y + 0.3746z = 0.4904$$

$$0. x + 0. y + 0.294z = 0.1592$$

$$x = 0.8013$$

$$y = 0.2570$$

$$z = 0.5812$$

$$\text{Mass} = 206.83$$

$$= 15.44$$

$$= 161.77$$

$$\text{wt\%} = 54 \quad \text{Pugu kaolin}$$

$$= 4 \quad \text{Quartz}$$

$$= 42 \quad \text{Feldspar}$$

Appendix 2 : Data for samples fired at 1100, 1200 and 1300 °C

Table 11: Properties of Porcelain Samples Fired at 1100 °C

Mix Proportion	Linear Shrinkage (%)	Bulk density (g/cm ³)	Water absorption (%)	Bending Strength (MPa)
C1	7.083	1.903	6.07	35.148
C2	7.167	2.151	5.16	48.199
C3	8.417	1.99	5.06	38.88
C4	8.75	2.413	3.59	40.982
C5	6.167	1.957	6.36	37.617
C6	8.75	2.185	2.06	41.831

Average taken from 10 samples

Table 12: Properties of Porcelain Samples Fired at 1200 °C

Mix Proportion	Linear Shrinkage (%)	Bulk density (g/cm ³)	Water absorption (%)	Bending Strength (MPa)
C1	9.5	2.011	0.09	72.677
C2	9.5	1.954	0.03	68.644
C3	12.083	1.829	0.01	65.358
C4	9.7	1.9	0.04	63.379
C5	7.833	1.864	0.01	67.688
C6	11.19	1.81	0.02	63.794

Average taken from 10 samples

Table 13: Properties of Porcelain Samples Fired at 1300 °C

Mix Proportion	Linear Shrinkage (%)	Bulk density (g/cm ³)	Water absorption (%)	Bending Strength (MPa)
C1	11.917	2.855	0.21	33.517
C2	13	2.213	0.11	9.421
C3	12	2.288	0.32	36.719
C4	11	2.028	0.22	37.859
C5	9.5	1.988	0.77	53.528
C6	10.167	1.823	1.24	46.884

Average taken from 10 samples

Average values of samples fired at 1100 °C (Table 14 – 19)

Table: 14

Batch C1						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	57	5.000	2.038	8.254	33.239
2	60	56	6.667	1.694	5.101	24.981
3	60	55	8.333	2.015	3.052	41.472
4	60	54.5	9.167	1.803	3.644	29.275
5	60	56	6.667	1.961	4.815	39.050
6	60	55	8.333	1.957	4.621	45.376
7	60	55	8.333	1.839	4.355	39.050
8	60	56	6.667	1.886	8.929	32.554
9	60	57.5	4.167	1.793	9.299	32.897
10	60	55.5	7.500	1.977	8.596	33.582
Average			7.083	1.903	6.07	35.148

Table: 15

Batch C2						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	55	8.333	2.123	4.259	39.033
2	60	54	10.000	2.271	5.405	51.849
3	60	54	10.000	2.216	5.542	51.849
4	60	57	5.000	2.430	5.253	60.450
5	60	56	6.667	2.190	5.508	58.265
6	60	56	6.667	2.110	5.219	38.545
7	60	56	6.667	1.972	5.045	46.351
8	60	56	6.667	2.029	5.079	43.912
9	60	57	5.000	1.916	5.282	39.033
10	60	56	6.667	2.255	5.018	52.701
Average			7.167	2.151	5.16	48.199

Table: 16

Batch C3						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	55	8.333	2.251	5.302	53.293
2	60	55.5	7.500	2.144	5.184	48.791
3	60	54	10.000	2.041	5.054	48.791
4	60	55	8.333	2.008	5.405	38.545
5	60	55	8.333	1.829	4.844	33.925
6	60	56	6.667	1.687	4.160	17.134
7	60	55.5	7.500	2.276	4.882	43.424
8	60	54	10.000	2.034	6.159	46.839
9	60	54	10.000	1.768	5.208	21.956
10	60	55.5	7.500	1.864	4.423	36.105
Average			8.417	1.99	5.06	38.88

Table: 17

Batch C4						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	56	6.667	2.222	3.824	36.610
2	60	54.5	9.167	1.950	3.648	33.762
3	60	55	8.333	2.251	3.533	36.039
4	60	54.5	9.167	2.386	3.930	39.864
5	60	55.5	7.500	2.778	3.742	45.863
6	60	54	10.000	2.909	3.458	50.924
7	60	54.5	9.167	2.621	3.760	46.351
8	60	54.5	9.167	2.331	2.913	40.677
9	60	54.5	9.167	2.293	3.667	39.864
10	60	54.5	9.167	2.390	3.383	39.864
Average			8.750	2.413	3.59	40.982

Table: 18

Batch C5						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (kN/m ²)
1	60	56	6.667	2.032	6.469	43.912
2	60	56.5	5.833	2.310	5.945	48.791
3	60	56.5	5.833	1.905	6.470	43.912
4	60	56.5	5.833	1.836	6.212	30.155
5	60	56.5	5.833	2.317	5.775	46.351
6	60	57	5.000	1.828	6.486	30.841
7	60	56	6.667	1.658	6.535	29.137
8	60	56.5	5.833	2.080	6.297	34.576
9	60	55	8.333	1.692	6.757	33.925
10	60	56.5	5.833	1.912	6.688	34.576
Average			6.167	1.957	6.36	37.617

Table: 19

Batch C6						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	54.5	9.167	2.014	1.766	40.677
2	60	54.5	9.167	1.993	2.219	35.347
3	60	54.5	9.167	2.358	2.167	41.960
4	60	55	8.333	2.272	2.389	44.400
5	60	54.5	9.167	2.089	2.012	39.660
6	60	54	10.000	1.938	2.189	40.677
7	60	56	6.667	2.160	1.974	46.351
8	60	55	8.333	2.391	2.118	48.303
9	60	54	10.000	2.328	2.373	41.472
10	60	55.5	7.500	2.302	1.379	39.457
Average			8.750	2.185	2.06	41.831

Average values of samples fired at 1200 °C (Table 20 – 25)

Table: 20

Batch C1						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	55	8.333	2.096	0.000	80.957
2	60	53	11.667	2.009	0.050	69.392
3	60	55	8.333	1.903	0.029	69.392
4	60	55	8.333	1.948	0.646	92.522
5	60	54	10.000	2.003	0.053	60.059
6	60	55	8.333	2.000	0.060	69.392
7	60	53	11.667	2.002	0.008	66.404
8	60	54	10.000	2.017	0.035	97.148
9	60	54	10.000	2.207	0.006	63.675
10	60	55	8.333	1.923	0.000	57.826
Average			9.500	2.011	0.09	72.677

Table: 21

Batch C2						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	56	6.667	2.072	0.079	83.270
2	60	54	10.000	2.358	0.000	71.179
3	60	55	8.333	1.903	0.050	57.826
4	60	54.5	9.167	1.882	0.000	78.644
5	60	54.5	9.167	1.642	0.021	64.765
6	60	55	8.333	2.038	0.000	50.030
7	60	54	10.000	1.827	0.064	50.309
8	60	54	10.000	1.886	0.000	80.957
9	60	53	11.667	2.022	0.029	69.392
10	60	53	11.667	1.909	0.025	78.644
Average			9.500	1.954	0.03	68.502

Table: 22

Batch C3						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	52	13.333	1.904	0.028	57.826
2	60	52.5	12.500	1.772	0.000	55.555
3	60	53	11.667	2.022	0.000	70.548
4	60	53	11.667	1.642	0.000	61.296
5	60	54	10.000	1.775	0.033	55.513
6	60	53	11.667	1.980	0.000	76.941
7	60	52	13.333	1.605	0.014	49.731
8	60	53	11.667	1.762	0.027	65.344
9	60	52	13.333	1.850	0.017	66.898
10	60	53	11.667	1.980	0.000	93.928
Average			12.083	1.829	0.01	65.358

Table: 23

Batch C4						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	54	10.000	1.657	0.000	57.826
2	60	54	10.000	1.808	0.000	68.235
3	60	54	10.000	1.827	0.000	54.124
4	60	54.5	9.167	1.824	0.225	64.187
5	60	54	10.000	1.939	0.000	84.426
6	60	53	11.667	1.935	0.044	92.522
7	60	55	8.333	1.691	0.033	75.174
8	60	54.3	9.500	2.286	0.016	43.698
9	60	54	10.000	2.220	0.046	47.340
10	60	55	8.333	1.813	0.000	46.261
Average			9.700	1.9	0.04	63.379

Table: 24

Batch C5						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	55	8.333	1.903	0.000	77.487
2	60	55	8.333	1.936	0.000	79.800
3	60	55	8.333	1.736	0.049	57.826
4	60	56	6.667	1.989	0.012	54.578
5	60	55	8.333	1.813	0.006	55.488
6	60	56	6.667	1.964	0.000	44.063
7	60	55	8.333	1.813	0.007	73.439
8	60	56	6.667	1.882	0.000	76.331
9	60	54.5	9.167	1.830	0.010	78.065
10	60	55.5	7.500	1.772	0.000	79.800
Average			7.833	1.864	0.01	67.688

Table: 25

Batch C6						
Test Piece #	Green Length (mm)	Fired length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	54	10.000	1.919	0.000	52.759
2	60	54.5	9.167	1.798	0.048	69.392
3	60	53	11.667	1.835	0.014	80.957
4	60	53	11.667	1.835	0.007	78.065
5	60	53	11.667	1.775	0.018	69.392
6	60	52.5	12.500	1.832	0.034	61.296
7	60	53	11.667	1.675	0.000	34.696
8	60				0.037	
9	60				0.013	
10	60				0.021	
Average			11.190	1.81	0.02	63.794

Average values of samples fired at 1300 °C (Table 26 - 31)

Table: 26

Batch C1						
Test Piece #	Green Length (mm)	Final length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	52	13.333	2.100	0.182	2.439546862
2	60	54	10.000	1.991	0.658	65.92197743
3	60	53	11.667	2.489	0.000	35.85230351
4	60	53	11.667	2.209	0.302	4.626103679
5	60	53	11.667	2.236	0.299	29.49141095
6	60	52	13.333	1.719	0.000	32.77385405
7	60	53	11.667	2.055	0.325	46.26103679
8	60	52.5	12.500	2.189	0.308	41.63493311
9	60	53	11.667	9.262	0.000	41.8662383
10	60	53	11.667	2.296	0.000	34.30612775
Average			11.917	2.855	0.21	33.517

Table: 27

Batch C2						
Test Piece #	Green Length (mm)	Final length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	54	10.000	1.889	0.154	0.137069739
2	60	51	15.000	2.325	0.000	0.780654996
3	60	51	15.000	3.675	0.189	0.231305184
4	60	52	13.333	2.326	0.000	0.390327498
5	60	53	11.667	2.029	0.146	0.137069739
6	60	52	13.333	1.898	0.000	22.66790803
7	60	52.5	12.500	1.792	0.000	41.63493311
8	60	52	13.333	2.006	0.000	0.462610368
9	60	52.5	12.500	1.797	0.319	6.913256076
10	60	52	13.333	2.394	0.284	20.85396099
Average			13.000	2.213	0.11	9.421

Table: 28

Batch C3						
Test Piece #	Green Length (mm)	Final length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	52	13.333	2.823	0.000	29.13231471
2	60	52.5	12.500	2.109	0.319	-
3	60	54	10.000	2.031	0.000	36.02170271
4	60	53	11.667	2.127	0.535	-
5	60	53	11.667	2.129	0.000	-
6	60	52	13.333	2.388	0.339	-
7	60	53	11.667	2.047	0.556	36.3855583
8	60	52.5	12.500	2.439	0.000	-
9	60	53	11.667	2.282	0.877	45.33581606
10	60	53	11.667	2.502	0.533	-
Average			12.000	2.288	0.32	36.7188

Table: 29

Batch C4						
Test Piece #	Green Length (mm)	Final length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	53	11.667	2.025	0.242	25.12662144
2	60	53.5	10.833	1.877	0.000	62.45239967
3	60	54	10.000	2.069	0.698	55.35139796
4	60	53	11.667	2.123	0.231	29.13231471
5	60	53	11.667	1.956	0.291	68.22292181
6	60	53	11.667	1.961	0.000	1.456615736
7	60	54	10.000	1.857	0.259	38.23616306
8	60	53	11.667	2.196	0.223	26.21908324
9	60	53.5	10.833	2.317	0.210	43.26148735
10	60	54	10.000	1.901	0.000	29.13231471
						37.859
Average			11.000	2.028	0.22	37.859

Table: 30

Batch C5						
Test Piece #	Green Length (mm)	Final length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	54	10.000	2.083	0.314	69.39155519
2	60	55	8.333	2.064	0.312	11.5652592
3	60	54	10.000	2.377	0.794	113.913177
4	60	55	8.333	2.241	1.240	16.98705271
5	60	54	10.000	1.985	0.990	104.0873328
6	60	54	10.000	1.953	0.286	81.86750617
7	60	54	10.000	1.949	0.400	42.04158535
8	60	55	8.333	1.768	2.545	33.30794649
9	60	54	10.000	1.670	0.784	50.55695232
10	60	54	10.000	1.795	0.000	11.5652592
Average			9.500	1.988	0.77	53.528

Table: 31

Batch C6						
Test Piece #	Green Length (mm)	Final length (mm)	Linear shrinkage (%)	Bulk density (g/cm ³)	Water absorption After 24hours (%)	Strength (MPa)
1	60	55	8.333	1.767	1.070	72.83078679
2	60	54	10.000	1.814	1.061	54.62309009
3	60	55	8.333	2.105	1.203	15.12519054
4	60	53	11.667	2.149	2.174	92.52207358
5	60	54	10.000	1.814	1.061	43.69847207
6	60	53	11.667	1.814	1.622	70.64586318
7	60	54	10.000	1.593	0.906	10.92461802
8	60	54	10.000	2.003	0.836	18.19277915
9	60	53	11.667	1.574	0.935	51.2728739
10	60	54	10.000	1.593	1.511	38.600317
Average			10.167	1.823	1.24	46.884