ADDIS ABABA UNIVERSITY

ADDIS ABABA INSTITUTE OF TECHNOLOGY

School of Multidisciplinary Engineering

Centre for Materials Engineering

Investigating the Effect of Partial Substitution of Feldspar by Alkaline-Rich Material on the Processing Temperature of Locally Produced Electrical Porcelain Insulator

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Presented in the Partial Fulfillment of the Requirement for the Degree of Masters of science in Materials Engineering

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SCHOOL OF MULTIDISCIPLINARY ENGINEERING

CENTRE FOR MATERIALS ENGINEERING

This is to certify that the thesis entitled "Investigating the Effect of Partial Substitution of Feldspar by Alkaline-Rich Material on the Processing Temperature of Locally Produced Electrical Porcelain Insulator". Submitted in partial fulfillment of the requirements for the degree of Masters of Science in Materials Engineering, the graduate program of Centre for Materials Engineering complies with the regulations of the University and meets with the standard quality.

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Advisor	Signature	Date

Declaration

I hereby declare that this MSc Thesis entitled "Investigating the Effect of Partial Substitution of Feldspar by Alkaline-Rich Material on the Processing Temperature of Locally Produced Electrical Porcelain Insulator" has not been submitted in any form for another degree, diploma or an award at any university or other institution of the tertiary education. I confirm that appropriate credit has been given within this thesis where reference has been made to the work of others and has been duly acknowledged. The work was under the guidance of Dr. Sintayehu Nibret instructor in Addis Ababa University, Centre for Materials Engineering.

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Student	Signature	Date

Approval of Board of Examiners

We, the undersigned, members of the board of examiners of the final open defense by Eaba Beyene have read and evaluated this Thesis entitled "Investigating the Effect of Partial Substitution of Feldspar by Alkaline-Rich Material on the Processing Temperature of Locally Produced Electrical Porcelain Insulator" and examined the candidate. This is, therefore, to certify that the thesis has been accepted in the partial fulfillment of the requirement of the Masters of Science degree in Materials Engineering.

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Internal examiner	Signature	Date
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External examiner	Signature	Date
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Chairman	Signature	Date
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Advisor	Signature	Date

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Table of Contents

List of Tables

List of Figures

Abbreviations

ASTM: American Standard for Testing Materials

EDS- Energy dispersive spectroscopy

FE-SEM- Field emission scanning electron microscope

SEM: Scanning Electron Microscope

XRD: X-ray diffraction

Abstract

The demand for electric power supply has been significantly increasing in the world and particularly in Ethiopia. In the power generation sector, it is crucial to develop a high quality insulation material to transmit and distribute the generated power, such as porcelain insulator. Therefore, in this study, we produced an electrical porcelain insulator from locally available clay (Bombowha clay), feldspar (Arero feldspar) and quartz (Arero quartz) and achieved reduced processing temperature without compromising insulator properties. The physical and dielectric properties such as apparent porosity, water absorption, bulk density, and dielectric strength of the as prepared samples were performed. Furthermore, XRD and FE-SEM analysis were utilized to analyze the phase formation and morphology of the phases after firing. The Arero feldspar was partially substituted with alkaline rich materials (sodium carbonate, sodium chloride and glass powder). These preliminary samples were then fired at 1000 ^{o}C and the best substitute and processing technique were compared and the one with good dielectric strength was chosen for further study. Reasonable dielectric strength was achieved from the samples containing sodium carbonate as a substitute which was chosen for further study. Experiments were performed using different concentration of sodium carbonate (5%, 7.5% , and 10% of the feldspar composition in the porcelain), and firing temperature of 1000 ^{o}C, 1100 ^{o}C, and 1200 ^{o}C. The XRD and FE-SEM results showed that sufficient amount of the required phase, i.e., mullite phase, formed at 1100 ^{o}C and 1200 ^{o}C with the addition of the sodium carbonate as a substitute. Furthermore, relatively good dielectric strength values for porcelain insulator were achieved by the samples prepared at a lower temperature of 1200 ^{o}C compared to previous studies. The maximum dielectric strength which was achieved by substituting 10% of the feldspar with Na_2CO_3 is 7.51 kV/mm; the standard for low voltage overhead line porcelain insulator being in the range of 6.1 - 13 kV/mm. Moreover, substituting values of 5% and 7.5 % at 1200 ^{o}C exhibited comparable dielectric strength values with the 10 % substitute.

1. INTRODUCTION

1.1 Background

The demand for electric power supply has been significantly increasing in the world. Especially developing countries like Ethiopia are at the forefront of such high demand. The reason for such an increase is that the fast growth of population and industrialization in most sectors. From the start of the millennia to 2012 the demand for electricity increased by 45% in sub-Saharan countries and yet the increase in demand is expected to continue with a rate of 4% a year for the next 20 years. This is because nearly 600 million people in Sub Saharan Africa lack access to electricity (Khan & Singh, 2017). Being one of the fastest growing economies in Africa, Ethiopia invested millions of dollars to assure the demand of such industries and population is met and satisfied. Ethiopia has a renewable energy potential around 60,000 MW and the energy sector of Ethiopia has shown a tremendous growth over the past twenty years and the current installed capacity of Ethiopia is 5000 MW. Even though the power generating capacity is yet far behind the demanded power, there is a huge progress and development that are being undertaken to address such problems. Around 23 power plants in Ethiopia are generating electricity using hydropower, wind energy, geothermal and diesel sources. Many more are under construction including the Great Ethiopian Renaissance Dam. The GERD is expected to raise the power capacity of the country from its current state to more than 10,000 MW in the coming years (Mondal et al., 2018).

The production of electric power is not an easy task. It demands a lot of worth money, resources and manpower. Therefore, it is very important to conserve and use it efficiently. The careful distribution and utilization of the generated power is vital. Thus, it is crucial to develop a high voltage and long distance transmission and insulation materials for such power industries. This is because most of the electric power generated is lost due to poor transmission and insulation. Many factors contribute to the loss of the generated energy one of which being improper insulation. In order to transmit and distribute the electric power, it is important to have a well suited insulator which can prevent the flow of current from the wire to the earth through ground supporting tower or pole (Ezenwabude & Madueme, 2015). Though many insulators are commercially available, porcelain insulator is the most commonly used material for insulation in electric power transmission and distribution system (Ovri & Onuoha , 2015). This is because porcelain insulators show outstanding properties like high mechanical strength, high electrical stability and corrosive resistance even in humid environment (Meng et al., 2012). In addition, the availability of raw materials used for porcelain production in makes it a potential candidate for such applications.

Tabor ceramics, the pioneer in the production of porcelain insulators in Ethiopia, has stopped producing porcelain insulators due to lack of import substitute material and failure to meet the demanded quality; the factory used to import ball clay from China which is used as a precursor in the production of porcelain insulators. Recently, the development of porcelain insulators from locally available raw materials in Ethiopia has been studied and has a promising outcome (Merga et al., 2019). However, high temperature has been employed in the preparation of the porcelain insulator. Consequently, it is beneficial if such materials can be processed at lower temperature, hence lower energy consumption. This study focuses on the reduction of the processing temperature for the production of a porcelain insulator, to be used as an overhead power line insulator, from locally available raw materials without compromising the dielectric strength of the porcelain insulators.

1.2 Statement of the Problem

The increase in the population size and industrialization in Ethiopia requires sustainable supply of electric power. In order to meet the demand of the people and industries, the government has tried to use the available resources for the production of electricity. Yet, there is a huge gap between the generated and delivered power. This accounts mainly to inefficient transmission and insulation in the power grids. One of the major demands for better transmission of the grids is efficient insulation materials, mostly porcelain insulators.

In 1997, the total imported porcelain insulators in Ethiopia was 78.2 tonnes and it was tripled in 2006 being 252.4 tonnes. In 2017, the projected total imported porcelain insulators demand was increased to 2075 tonnes signifying the government's determination to satisfy the demand of the population and industries via multiple projects in the country (ethiopianreview, n.d.). The production of porcelain insulators for overhead lines was halted by tabor ceramics, the pioneer in the production of porcelain insulators in Ethiopia. This is mainly because of lack of import substitution, as the company had been importing ball clay from china and shortage of foreign currency which makes the production even more difficult.

On the other hand, there are no researches regarding porcelain insulators in Ethiopia except a recent work on preparation of porcelain insulators from locally available raw materials (Merga et al., 2019). Lack of research works in porcelain materials in Ethiopia has several disadvantages. First, the potential of the country will be hidden leading to misuse of its resources to produce such insulators. Second, it makes the energy sector dependent on other countries supply potential. To solve the aforementioned problem, one promising research has been conducted to substitute the imported ball clay by locally available raw materials during the time when tabor ceramics used to produce porcelain insulators. The research found out that electrical porcelain insulators can be produced locally by replacing the ball clay, which was imported, by locally available Bombowha clay. However, there are no any other researches performed to enrich and improve the pioneer research work in Ethiopia.

The pioneer research work on porcelain insulators in Ethiopia faces a drawback of having high firing temperature of 1300 ^{o}C; hence high processing temperature that leads to higher processing cost. One estimated reason for the high firing temperature is the low alkaline content of the

Arero feldspar of Ethiopia. The Arero feldspar in Ethiopia has an alkaline content of approximately 7% which is much lesser than the standard alkaline content in feldspar (12%) used in porcelain insulators (Merga et al., 2019) (Moyo & Park, 2014). In the making of porcelain insulator, the feldspar is used as a fluxing agent to melt the clay, feldspar, and quartz mix at lower temperature (usually starting from 1000 ^{o}C) and form a glassy phase. Since the Arero feldspar has lower alkaline content, higher temperature was employed to obtain the glassy phase which helps the nucleation and formation of the required phase, mullite phase. In this study, materials with sufficient alkaline constituent were employed by partially substituting the Arero feldspar to achieve higher alkaline content and reduce the processing temperature without compromising the dielectric properties of the electrical porcelain insulators.

1.3 Objective

General Objective

- The general objective of this work is to investigate the effect of partially substituting feldspar by alkaline rich materials to reduce firing temperature in the making of locally produced porcelain insulator.

Specific Objective

- ✓ To prepare three different batches of pellets by integrating individual glass powder, sodium carbonate and sodium chloride substitutes in the clay, feldspar and quartz mix for the formation of porcelain insulator by substituting 10% of feldspar.
- ✓ To choose the best substitute for feldspar among glass powder, sodium carbonate and sodium chloride in the porcelain mix by comparing their dielectric strength.
- ✓ To investigate the extent to which the substitute that outperformed the other substitutes can be added to the porcelain mix to lower the processing temperature without compromising the dielectric strength.
- ✓ To analyze the dielectric strength, physical property and phase of the final product.

1.4 Significance of the study

This study will be of great importance to our country, Ethiopia. First, it proposes a very cost effective production of electrical porcelain insulators. Costs associated with importing raw materials and the cost of the energy required to process such materials will be reduced. Thus, ceramic industries interested in producing porcelains can easily process their desired material. Second, efficient distribution of electricity will be achieved in that the produced electricity will be utilized and distributed with good electrical porcelain insulators. Finally, it is also helpful for researchers engaged in dielectric materials to use the findings of this study for further research and development of porcelain insulators.

1.5 Scope of the study

The scope of this paper focuses on the reduction of the firing temperature and attaining the required dielectric strength and physical property for overhead line porcelain insulators. This work is limited to preparation pellets of porcelain insulator using locally available raw materials and achieving standard values of dielectric strength and physical properties - improvement of both dielectric strength and physical properties of porcelain insulators is not covered in this study.

In addition, mineralogical properties of the raw materials used in this study were directly taken from analysis performed by a previous study since the raw materials used in both studies were from the same origin, Tabor Ceramics.

2. LITERATURE REVIEW

2.1 Power line insulators

A material that hinders the flow of electric current is termed as an insulator. It separates electrical conductors without passing current through itself (Adnan et al., 2004). There are many materials that can be identified as insulators, ceramics being one of the major groups of materials. Ceramics can be defined as compounds between metallic and nonmetallic elements; they can be oxides, nitrides, and carbides. Common ceramic materials include aluminum oxide (or alumina, Al_2O_3), silicon dioxide (or silica, SiO_2), silicon carbide (SiC), silicon nitride (Si_3N_4), and traditional ceramics, those composed of clay minerals (i.e., porcelain), as well as cement and glass. Ceramics exhibit stiff and strong mechanical properties which is comparable to those of metals. They also are very hard. Ceramics exhibit extreme brittleness and are highly susceptible to fracture. Furthermore, ceramic materials have low electrical conductivities; they are resistant to the passage of electricity. They also are resistant to high temperatures and harsh environments than metals and polymers. Ceramics possessing such properties are used for many electrical applications (William D. Callister, 2009). Insulators are also essential parts of overhead power lines distribution systems. A power line transmission possesses different kinds of insulators to distribute electricity. They are used to hinder the flow of current from the wires to the ground through the supporting poles. Power line insulators are mainly known as high voltage and low voltage power line insulators. The types of materials used as power line insulators vary and are available in different kinds. Of all the available materials for power line insulators, the three major insulating materials used in power line transmission lines are porcelain, glass and composite insulators.

Glass insulators are manufactured using lime, quartz and other materials which are mixed and processed at higher temperature and are quenched and cooled in a mould. Glass insulators have good mechanical resistance and have good dielectric strength. They also have low thermal expansion coefficient and it is resistant to deformation which occurs as a result of temperature variation. Even though glass insulators have such good qualities they also have drawbacks in having less mechanical durability against bending forces compared to that of porcelains. They should also be processed in a controlled manner to avoid impurities because impurities lead to failure of the insulator in an environment with considerable temperature variations. Another drawback of glass insulators is that they easily absorb contaminations and moisture which will lead to the corona discharge effect (Taherian, 2018).

Another insulator used for overhead power lines are the composite insulators. Composite insulators are made up of two or more insulating materials. Each individual material contributes to the better performance of the composite. One of the materials is responsible for providing dielectric strength while the other provides mechanical resistance. Composite insulators have advantages of having good performance in contaminated environments, low weight, are hydrophobic, less susceptibility to different weather condition and have long shelf life. But they still have drawbacks such as low temperature inefficiencies and inadequate weight loss (Taherian, 2018).

2.2 Porcelain insulators

Many ceramic materials are now being used in the electronic industry one of which being electrical porcelain insulators. The electrical porcelain insulators are produced from natural ceramic raw materials; clays (kaolin), feldspar and quartz (Ngayakamo & Park, 2018). These ingredients go through different physical changes and react together under thermal condition to produce the final product. Of all the over head ceramics used for high and low tension insulation electrical porcelain insulators are the most complex multiphase ceramic materials used as overhead insulator.

2.2.1 History of porcelain insulators

The invention of electricity changed the world for good. At first human dependence on electricity increased and second many inventions and innovation raised after it. Because of this reason the production and transmission of electricity became important. The transmission of electricity not only required good conductors to transmit electricity but also good insulators that prevent power loss due to inefficient overhead power lines. Due to this reason insulating materials have been developed to prevent energy loss. History tells us that telegraph insulators prior to electrical insulators were of demand during the 1850’s and 1860’s. Though local potteries began making telegraph insulators, the production was in lower quantities than glass insulators because they were cheaper than porcelain insulators. But all of this changed after the invention and growth of electricity. This was because the demand for a better, in quality and quantity, material capable of carrying higher voltage power lines increased dramatically. Fred M. Locke from New York was the first person to be convinced that porcelains are better insulators. He started experimenting by mixing clays to produce finer insulators. He was also designing new insulators capable of holding up high voltage even in harsh weather conditions (*A Brief History of Porcelain Insulators*, n.d). The demand for porcelain insulators for power line insulation has increased dramatically since then. Currently most power line cables are held and insulated by porcelain insulator (Tod, n.d.).

2.2.2 Constituents of porcelain insulators

Processing of raw materials is one of the major steps for the production of a well performing porcelain insulator. It involves the selection of raw materials, preparing them to a certain quality, mixing and processing them through different treatments to attain the desired properties. The desired properties for insulators such as dielectric strength and dielectric permittivity are greatly influenced by the initial processing and preparation steps. This is because dielectric properties of porcelain are dependent on the amount and quality of different phases (Paul et al., 2014) which are formed due to the existence of certain minerals and elements when they pass through different processing mechanisms at high temperature (Liang et al., 2017). Porcelain is made up of three major constituents: clay, feldspar and quartz.

I. Clay

Clay is a naturally occurring material that is composed of fine grained minerals that have plastic nature. They have certain water content that will dry and harden when fired. It is primarily inorganic material (excluding peat, muck, some soils, etc) containing some amount of organic materials (Guggenheim et al., 1995). Plasticity is defined as the tendency of a material to be molded into a certain shape when a certain stress is applied on it and keep the shape after the stress is removed. Plasticity is affected by many factors such as chemical composition and particle aggregation. Four types of clays can be distinguished for industrial applications this are

Bentonites, Kaolins containing kaolinite, Palygorskite and sepiolite, and Common clays containing illite/smectite mixed-layer minerals (Bergaya et al., 2006).

Clay minerals can be characterized by certain properties.

1. They have a layer structure with one dimension in the nanometer range;

2. They exhibit anisotropy, meaning there is difference in physical properties along different molecular axes for the layers or particle.

3. Several types of surfaces such as external planar and edge surfaces as well as internal surfaces exist.

4. Modification of both external and internal surfaces is simple. This can be done by adsorption, ion exchange, or grafting.

5. Plasticity, which is the major character of clay

6. Hardening on drying or firing, which is common to most clay minerals (M.S. Prasad, 2018).

- **Kaolin**

Kaolins are white raw materials essentially containing white clay which makes them open to beneficiation, (improving the economic value of the ore by removing the gangue), making them ideal materials for most ceramic industries. They originally were obtained from China in the 15th century. There are two types of kaolin deposits, primary (residual) and secondary (sedimentary). The main constituent of kaolin is kaolinite. It is a hydrous aluminum silicate of the approximate composition $2H_{2}0\text{-}A1_{2}0_{3}\text{-}2SiO_{2}$. There is a stack of alternating alumina octahedral sheet and silica tetrahedral sheet structure in kaolinite. A perfectly ordered kaolinite crystal maintains a shape that is pseudo-hexagonal. This might be ordered in high order manner or low order manner (M.S. Prasad, 2018). Primary kaolin may be refined and processed to extract pure kaolin. A kaolin that is processed as such mostly have a well ordered crystallinity. Kaolin is one of the primary constituent of porcelain insulator. Kaolin can be formed in two ways one is by the action of weathering and the other is by hydrothermal alteration. Both ways of formation are common in Ethiopia though the latter exists abundantly than the former. Many Geological works indicated the existence of Kaolin in many localities within Ethiopia (Tadesse et al., 2003).

Clay provides plasticity and green strength during the forming stages of porcelain. It also contributes substantially to easy shape formation and color of the fired ware. It also serves as a binder for the other constituents at green state.

II. Quartz

Quartz, a silica mineral, is abundantly found in the Earth's crust, occurring in large amounts in igneous, metamorphic and sedimentary rocks. They are highly stable enriched in all siliciclastic sediments and rocks. Quartz exhibits excellent chemical and mechanical properties because of its interesting structure. The chemical formula of quartz is SiO_2 because in three dimensional networks, the α-quartz crystal structure is composed of $[SiO_4]^{4-}$ tetrahedral joined with all the Oxygen atoms. Quartz can be used in many sectors as a bulk product for high-purity quartz sands or quartzite and perfect natural crystals, as well as a Si ore (Götze, 2009). Quartz is included to for the formation of porcelain insulator and accounts for around 10% of the makeup. It serves as a filler material which helps the insulator maintain its shape both at green state and during firing. Quartz is one of the most abundant mineral resources of Ethiopia. It occurs in rocks like granite, sand of beaches, the gravel of rivers etc. The main sources of quartz are quartzite and sandstone which are regarded as the source of silica sand. Both rock units exist abundantly in Ethiopia (Tadesse et al., 2003).

III. Feldspar

Feldspars are among the most important groups of rock- forming minerals in the Earth's crust, alumino-silicate minerals, containing varying proportions of potassium, sodium or calcium. They result from the cooling of magma and the crystallization of igneous rocks as intrusive or extrusive rocks. Feldspars are also present in many types of metamorphic and sedimentary rocks. Their origin contributes to their widely varying range of compositional and structural characteristics (Balić-Žunić et al., 2013). There are three common feldspars named on the basis of potassium, sodium and calcium content; Potash feldspar, soda feldspar and lime feldspar. The potash feldspar includes orthoclase ($KAlSi_3O_8$) and microcline ($KAlSi_3O_8$). Orthoclase is more common of the potash feldspars which show striations rarely on cleavage surfaces and is stable between 500 ^{o}C and 900 ^{o}C. Microcline, which are also known as "small incline", show lamellar twinning as its common feature. Between 400 ^{o}C and less, microcline is the stable structure for potash feldspar. The key difference between orthoclase and microcline is that the aluminum and silicon atoms are differently oriented in their structure. The positions of the aluminum and silicon become more disordered at high temperatures and result the monoclinic symmetry of orthoclase. Soda feldspar is another group of feldspars and is also called albite ($NaAlSi_3O_8$) (Aliyu et al., 2016). Lime feldspar might include anorthite ($CaAl_2Si_2O_8$) or plagioclase. Plagioclase is a solid solution of the end members albite and anorthite with minor substitution of K for Na (Bersani et al., 2018). The Potash feldspar and soda feldspar are the most important members of the feldspar group. Feldspars are used in the glass industry, in ceramic industry and industries of rubber, paint, plastic, welding electrode serving as a flux and filling material. The main importance of alkali containing feldspar is that it acts as a fluxing material. The main role of fluxes in ceramics is to melt at low temperature and bind particles to each other. This happens as fluxing agents start melting (or softens), before the ceramic body, which will collapse the porosity and bonds the un-melted ceramic particles to each other which will lead to the binding of the ceramic body. The liquid melt will fill the voids in the microstructure level which will result densification of the body. In order to have a dense and properly sintered body the role of fluxes is critical (Aliyu et al., 2016). Especially for porcelains the amount of alkaline content in the feldspar is crucial so that the feldspar could perform well. Most of the feldspars that are available in Ethiopia are connected with pegmatite. That is the reason the feldspar that occurs in Ethiopia shows projection of basement rocks with pegmatites. Feldspar exists in a number of locations in Ethiopia making it abundantly available (Getaneh, 2015).

IV. Alkaline-rich materials

Alkalis are metal elements found at group 1 of the periodic table excluding hydrogen. It includes elements like Lithium (Li), Sodium (Na), Potassium (K), Cesium (Ce), Rubisium (Rb) and Francium (Fr). These metals release hydroxide ions (OH^-) when dissolved in water and have a pH value more than 7 (Ropp, 2020). They are soft and have lower melting point due to the fact that their atomic size is large with only one valence electron. This single valence electron is relatively far from the nucleus, there is only weak metallic bonding, which results in a soft consistency and low melting point. This property of alkali metals made them to be preferable constituents in fluxing agents. Alkali metals are one of the constituents of feldspar. Feldspars containing such elements are suitable for the production of good quality porcelain insulators. In order to produce porcelain insulators the amount of alkaline content in the feldspar should not be less than 12%. The Arero feldspar which is used as a flux in the production of porcelain insulator in Ethiopia has very low alkali content (Merga et al., 2019). On the other hand, in Ethiopia we can find alkaline rich materials such as sodium carbonate and sodium chloride abundantly. Sodium carbonate (Na_2CO_3) or soda ash is a salt containing two sodium atoms and carbonate molecule. It is a white powder and has light weight. Carbonic acid and sodium hydroxide will result if sodium carbonate dissolves in water (National Center for Biotechnology Information., 2018). Sodium carbonate (Na_2CO_3) is also used to activate kaolinite as solid state reaction occurs between those two at 350-800℃ which made the kaolin go through different phase changes (Yan et al., 2017). Sodium chloride, NaCl also known as table salt is a commonly available, inexpensive commodity. It can be found in saline lakes or as underground deposits as halides. Sea water consists of a lot of salt making it the main constituent of dissolved solids found in seawater. It has a natural structure of cubic crystalline form. Sodium chloride produced commercially can exist in various size ranges be it fine granules of powder, compressed pellets or blocks. Depending on the impurities present in the salt, color of salt varies that it can be white, gray, reddish, or even brownish (Feldman, 2008).

2.2.3 Production steps of porcelain insulator

Steps that should be followed for production of porcelain insulators are the following. First the raw materials are dried, crushed and grinded. This is the primary size reduction step before mixing. The second step is sieving which is performed to have a uniform particle size distribution. The grinded powder is then sieved using 40-75μm sieves. This step is essential to mix the raw materials according to their respective percentage. One method of mixing is ball milling (Belhouchet et al., 2019). Ball milling is a technique of mixing and homogenizing powder mix. It is the most widely used technique for crushing and grinding powders to an extremely fine form. It is a rotating hollow cylindrical shell which is filled with balls that are capable of crushing powders into a micro or nano sized particles (Sumanth Kumar et al., 2018).

There are two ways in which ball milling can be performed. These are the dry ball milling method and the wet ball milling method. The two differ in the medium in which the powder is milled. For wet ball mill a liquid medium in which the powder is poured is used while there is no liquid used to mill for dry ball milling process. In order to increase the rate of grinding of powders, the grinding medium should be small. This is because the rate of grinding depends on how many interactions are occurring between the balls and the powder. The interaction points, on the other hand, are greatly dependent on the size of the grinding medium and the surface area of the balls. Their relation is that as the grinding medium size and the balls surface increases the grinding rate decreases. Grinding rate also relies on the size of the particles. As the particle size decreases the grinding rate also decrease as it is more difficult to crush fine particles (Rahaman, 2013).

After ball milling; molding the powder mix by applying constant pressure on it using hydraulic press is performed. Finally the green body is fired at temperature in a furnace (Belhouchet et al., 2019).

2.2.4 Formation and effect of different phases formed during sintering

Firing porcelain ceramics makes the green body pass through different phase transitions essential for the properties of the final product. Most of the phases formed are essential and necessary for the performance of the insulator.

During the first stages of sintering, at around 550 ^{o}C, the crystal structure of kaolinite which contains hydroxyl groups began to be removed by dehydroxylation process and then transforms to amorphous metakaolin. This process ranges between the temperature ranges of 420 ^{o}C to 660 ^{o}C. The temperature range depends on the amount and nature of the alkali content or other impurities present in the flux (Iqbal, 2008). Dehydroxylation is affected by the surface area of kaolin, they have direct proportional relation. The process takes place according to the following chemical equation

$$Al_2O_3.2SiO_2.2H_2O \longrightarrow Al_2O_3.2SiO_2 + 2H_2O \text{ ……………… Eqn. 2.1}$$

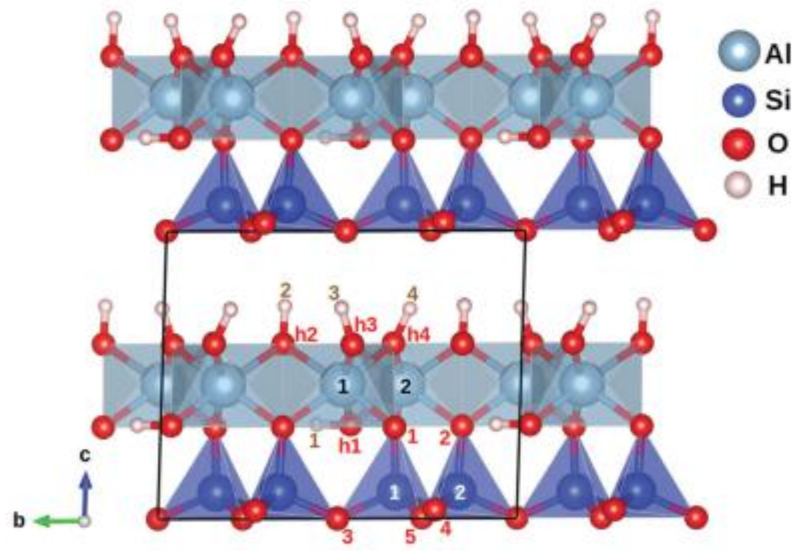

Fig 2.1 layered crystal structure of kaolinite ($Al_2O_3.2SiO_2.2H_2O$) (Source- Weck et al., 2015)

At around the same temperature, at 573 ^{o}C, α to β quartz inversion takes place. After the metakolin forms, γ-Al_2O_3 spinel starts to nucleate within the metakaolin at around 900 ^{o}C. Then at 950 ^{o}C to 1000 ^{o}C the metakaolin transforms into a spinel-type structure and amorphous free silica (Carty, 2020).

$$3(A1_2\ O_3.2SiO_2) \longrightarrow 0.282A1_8 + 6SiO_2 \text{……………………….. Eqn. 2.2}$$

The amorphous silica which resulted from the decomposition of metaklaolin support eutectic melt formation. This eutectic melt forms at around 990 ^{o}C for potash feldspar and at around 1050 ^{o}C for soda feldspar. Reduction of melt formation temperature assists in lowering porcelain firing temperature. This can be achieved by using potash feldspar or albite containing potash feldspar(Carty, 2020). At around 1000 ^{o}C parts of the clay that survived its decomposition began to form primary mullite (Iqbal & Lee, 2000). This primary mullite forms at the lower temperature. Further increase in temperature results in an elongated needle-shaped mullite which crystallizes from the feldspar-rich melt termed as secondary mullite (Lee & Iqbal, 2001).

$$SiAl_2O_3 + SiO_2 \longrightarrow 1/3(3Al_2O_3.2SiO_2) + 4/3SiO_2 \text{……………… Eqn. 2.3}$$

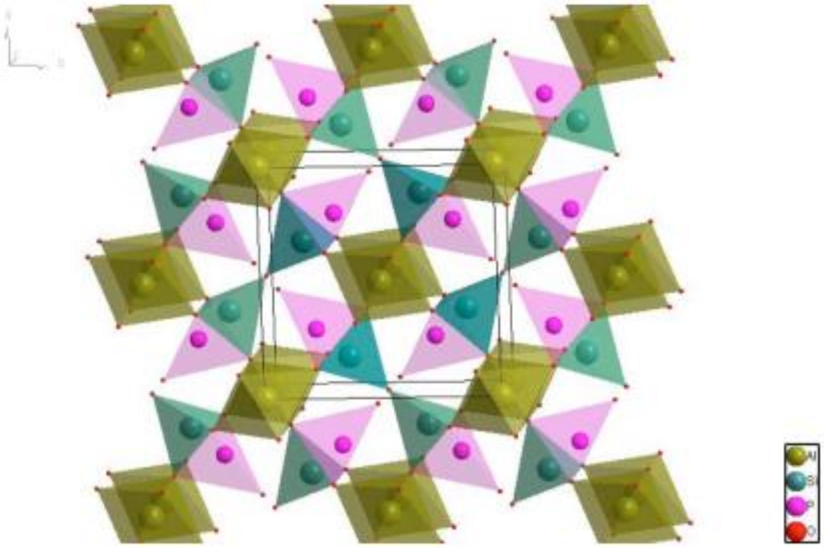

Fig 2.2 crystal structure of mullite (Source- Kaduk, 2009)

At around 700 ^{o}C to 1000 ^{o}C sanidine nucleates. Sanidine is high temperature and homogeneous mixed alkali feldspar. The temperature range difference for the formation of sanidine is as such because it depends on the sodium to potassium ratio (Carty, 2020).

Commercial porcelain possesses a microstructure of coarse quartz grains and matrix system containing primary and secondary mullite. This matrix contains glass, primary mullite and alkali(Na, K) aluminosilicates containing larger and elongated secondary mullite (Lee & Iqbal, 2001).

A. **Metakaloin**

As we fire kaolinite, different microstructural development in kaolin occurs. The first microstructural change is the conversion of kaolin to metakaolin (Carty, 2020). Metakaolin can be explained as an amorphous aluminosilictae intermediate phase which demonstrates two-dimensional regularity in the kaolin layers. The kaolinite transforms to metakaolinite by removing the hydroxyl groups above the temperature of 400 ^{o}C. The metakaolinite then transforms to a spinel structure or a Si-containing $Al_2O_3$and amorphous silica at a temperature around 980 ^{o}C (Sperinck et al., 2011).

B. Mullite

Mullite ($3Al_2O_3 \cdot 2SiO_2$) is an alumina silicate intermediate phase that occurs in porcelains when they are fired at high temperature. Mullite based ceramics exhibit a wide range of properties such as high thermal stability and excellent thermal shock, low thermal expansion and conductivity, together with a suitable strength and fracture toughness which makes them suitable for many applications. This is mainly because of the crystal structure of mullite which possesses two characteristic features. First they possess parallel octahedral bond chains reinforced by tetrahedral double chains, and second there is a structural disorder of Al and Si on tetrahedral sites with Oxygen vacancies required for charge compensation. The edge-sharing AlO_6 octahedral are the strength of the characters in mullite structures. These octahedral chains have part of the Si^{4+} replaced by Al^{3+}. In-order to compensate the charge deficit, oxygen atoms from the tetrahedral (Al^{3+}, Si^{4})$_2O_5$ are removed creating oxygen vacancies. A disturbed structural channels running parallel to the crystallographic c-axis are produced due to the combination of octahedral chains with tetrahedral clusters and O vacancies. This Al, Si atoms and/ or of O vacancies distribution scheme in mullite is very stable which persists up to the melting point near to 1900 ^{o}C (Schneider et al., 2015).

Mullite phase appears in two distinct types, the primary and secondary mullite. The primary and secondary mullite differ both in their size and shape and their role within the porcelain insulator (Schuller, 1964). Further increase in temperature transforms the metakaolin into a primary mullite. This primary mullite is dense and short. On contrary the secondary mullite is formed by the reaction between clay and feldspar relicts. It has needle-like shape individuals that are interlocked to each other. As firing time increases primary mullite aggregates dissolve in the melt which latter recrystallize as secondary mullite (Schuller, 1964). Mullite formation is most affected by the concentration gradients and diffusion rates of alkalis. As alkali diffuses out of feldspar at higher temperatures, secondary mullite nucleates and grows (Carty, 2020). The interlocked structure of mullite, that is the secondary mullite, affects properties of porcelain insulator. The more mullite phase nucleates the more porosity and cavity is reduced due to increase in volume. This is because porosity or cracks formed due to shrinkage are filled up due to increase in volume (Meng et al., 2012).

C. Glassy phase

The glassy phase is the uncrystallized leftover of a liquid phase present during densification which leads to reduction in porosity and enhanced densification of the body. The liquid phase can be formed to densify the green body or due to phase formation by impurities (Clarke, 1991). Densification leads to improvement in the mechanical and electrical properties. Early densification is also helpful in lowering the sintering temperature of porcelain products (Lee & Iqbal, 2001). Mass growth in solid state reaction is facilitated by the existence and extent glassy phase. Crystalline growth is also accelerated if sufficient amount of glassy phase is present (Amigó et al., 2005).

2.2.5 Properties of porcelain insulator

I. Electrical properties

The maximum voltage an insulating material can withstand before break down is known as dielectric strength. It is measured as the maximum voltage that produces a dielectric breakdown through a material per the thickness of the material, expressed as Volts per unit thickness. It depends on the nature and thickness of the material and on the method and conditions of the test. Higher dielectric strength corresponds to better insulation properties(Yuan & Mueller, 2017). The standard value of dielectric strength porcelain insulators should acquire to be used as overhead insulators is in the range between 6.1 - 13KV/mm (Olupot et al., 2010).

II. Physical properties

Apparent porosity is a parameter that relates the percentage relation between open pores volume with the exterior volume of a specimen and it affects both the functional and structural applications of ceramics (Lacroix, 2019). Pore existence and distribution in porcelains can only be reduced and cannot totally be omitted. This is mainly because decomposition of organic and inorganic compounds always takes place when firing takes place at elevated temperature. Pores might be advantageous if their size and shape is maintained. If spherical pores with less than 20µm size are present they have a positive effect on the bending strength of porcelains. Larger and non uniform pores will have an impact on the mechanical and electrical properties as they act the place for stress concentration and origin for fracture (Meng et al., 2012). Another parameter is water absorbance that is the measure of the relation between the water absorbed and the dry mass of the specimen.

2.3 Characterization techniques

2.3.1 X-ray diffraction

X-ray diffraction is a non-destructive characterizing technique based on Bragg's law ($n\lambda = 2d\sin\theta$) for crystalline materials in which a monochromatic beam of X-rays scattered at specific angles from each set of lattice planes in a sample are allowed to incident on a sample. The incident X-rays are reflected back and produce peaks which then are detected by a detector (Patel & Parsania, 2017). These peaks are produced by constructive interference of a monochromatic beam of X-rays. The atomic positions within the lattice plane produce peak intensities which provide information on structures, phases, preferred crystal orientations (texture), and other structural parameters. The XRD pattern is unique for different atomic arrangements for different materials (Kohli, 2019). The diffractometer operates at known voltages and current using a Cu Kα X-rays ($\lambda = 0.15406$ nm) over the 2θ range (Patel & Parsania, 2017).

The XRD characterization was performed using Shimadzu XRD 7000 X-ray diffracatometer. It has X-ray protective housing with a high precision vertical goniomter. The goniometer has a speed rate of 1000^{o}/min and high precision angle reproducibility (0.0002^{o}) making it attain reliable data.

2.3.2 Scanning Electron Microscope

Scanning electron microscope (SEM) is an electron microscope used for imaging the microstructure and morphology of materials. SEM functions by emitting an electron beam on a sample where several different interactions between the electron and the sample occur. As the beam reaches the material photons and electrons are emitted from the sample surface. The interactions are detected and their signals are used to form an image. The interactions will emit secondary electrons, backscattered electrons, electron channeling and Auger electron each having different signals to form the image. SEM consists of several components such a tungsten gun, electro-magnetic lenses and a high-vacuum environment (Omidi et al., 2017).

The surface analysis was performed using COXIEM-30 SEM. It can magnify up to 100000X with a spatial resolution of 15nm. It uses a pre-centred tungsten filament with an acceleration voltage of 1-30kV.

2.4 Previous Works

A paper made by Adnan et al., 2004 tried to analyse a porcelain insulator fired up to 1350 ^{0}C. It used SEM to characterize the microstructure and tried to understand the interdependence between the structure and property of the ceramic insulator. The porcelain insulator was inspected its dielectric strength for its electrical performance. The formation of mullite phase starts from 1000 ^{o}C and continues to grow with increasing firing temperature. Slow heating rate and cooling rate were both advantageous for mullite formation. It was also essential to minimize the formation of glassy phase due to the fact that they promote conduction by giving path for mobile ions which later hinders insulation. Glassy phase will likely appear if the amount of silicondioxide is high. The work concluded that sufficient amount of mullite phase formation and less amount of glassy phase will contribute to the good quality of porcelain insulator.

The effect of waste glasses on the physical and mechanical properties of soft porcelain has been studied resulting in a reduction of water absorption and porosity. An increase in the bulk density and a better sintering temperature was also achieved Owoeye et al., 2019. Researches also showed that the use of recycled waste glass powder as a fluxing agent replacing feldspar led to a lower sintering temperature of porcelain samples Belhouchet et al., 2019. The research made by Luz & Ribeiro, 2007 showed glass powder waste to be an efficient fluxing agent in porcelain stoneware. This was made possible by firing the porcelain stoneware at 1000-1250 ^{o}C. They saw that the glass powder accelerated the densification process resulting in a lower porosity and water absorption green body. It was therefore concluded that the use of waste glass powder with feldspar had a promising outcome. Recent publication by Merga et al., 2019 showed that it was possible to manufacture porcelain insulators in Ethiopia using locally available raw materials. Merga et al., 2019 used Bombowha clay, Arero feldspar and Arero quartz from Oromia region of Ethiopia to substitute the imported ball clay totally by locally available materials. The raw material mineralogy showed that the Bombowha clay contains enough amount of aluminium oxide required for the formation of mullite phase. The Arero feldspar which is used as a flux lacks enough amounts of alkaline content which is probably the reason for high processing temperature. The optimum property was achieved by using 45% of Bombowha clay, 45% of Arero feldspar and 10% of quartz fired at 1300 ^oC. Properties of the final fired porcelain were characterized and observed that increase in temperature at around 1300 ^oC resulted in the decrease of physical properties like water absorption and apparent porosity. The reason behind this was assumed to be the melting of feldspar at higher temperature around 1100 ^oC. The feldspar is responsible for densification process because it melts and reaches its glassy phase which fills the voids in the microstructure. It was found that there was an increase in dielectric strength as temperature was increased. This was due to an increase in vitrification with increase in temperature. At the maximum temperature which is 1300 ^oC, the sample showed a dielectric strength, 8 kV/mm, which was suitable to be used as overhead power line insulators.

Benchmarking previous studies, this study tries to partially substitute the Arero feldspar by alkaline materials to possibly reduce the sintering temperature of the porcelain insulator. This work also tries to analyze if the required phases, which are essential for the properties of the insulator, are formed at lowered firing temperature when feldspar is substituted by materials with good alkali content.

3. MATERIALS AND METHODOLOGY

3.1 Raw materials

The major raw materials used in this study for porcelain body formation were Bombowha clay, Arero feldspar and quartz which were all collected from Oromia region, Ethiopia. Merga et al. analysized mineralogical properties of the aforementioned raw materials and this study referred the analysis results from the previous study (Merga et al., 2019). The Bombowha clay contains sufficient amount of aluminum oxide to silicon dioxide ratio for the formation of a mullite phase. Similarily, the Arero feldspar showed less alkaline content, 7wt%, than the desired alkaline content from feldspar to be used in porcelain insulator formation. Thus, in this study the Arero feldspar was partially substituted with potential substitute materials. The other raw materials used were the substitute materials which include sodium carbonate, sodium chloride and glass powder. They are all industrial scale manufactured materials purchased from chemical markets in Addis Ababa, Ethiopia. The quartz is also good grade quartz containing low amount of impurity. The substitutes used were processed chemicals purchased from a chemical store except the glass powder. Glass powder was prepared by collecting scrambled glass, crushing them and sieving them to the required size.

3.2 Experimental procedure

Fig 3.1 illustrates the general experimental procedure followed throughout the study for the formation of porcelain insulator. The raw materials were first crushed and sieved before mixing them. Crushing to reduce the particle size was done using centrifugal mill. Sieving was made by using 75µm sieve for Bomboha clay, Arero feldspar and all the substitutes. Quartz was passed through 45µm sieve. The powders were then mixed and milled using both dry and wet ball milling techniques. The milled powder was then oven dried and molded using hydraulic press. The compacted pellet was then fired at varying temperature using high temperature box furnace to get the final porcelain insulator samples.

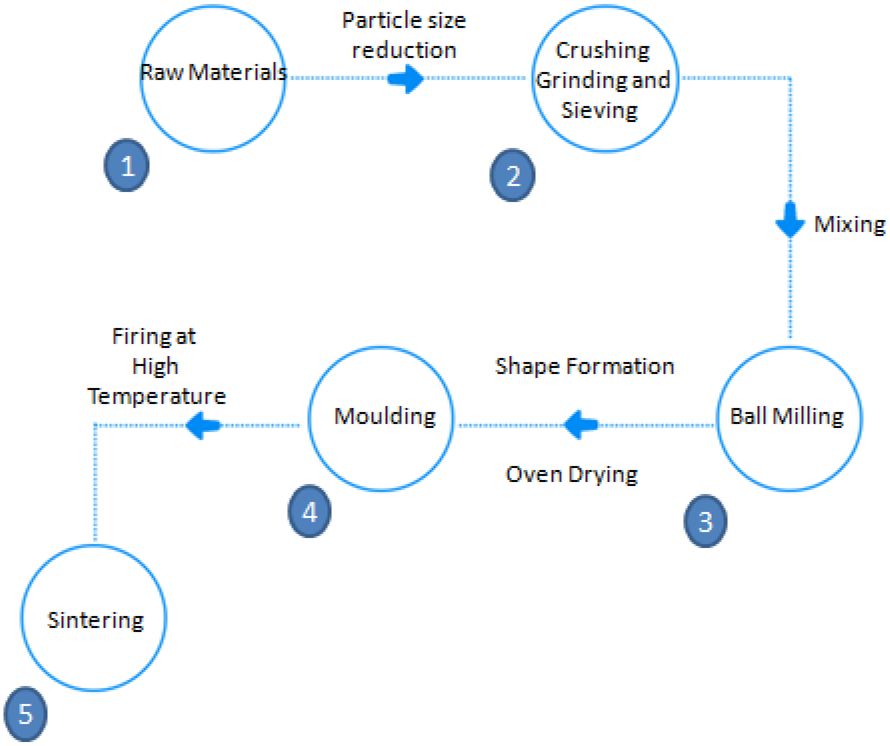

Fig.3.1 Procedures followed for the preparation and formation of porcelain insulator

There were different potential substitutes that can partially replace feldspar. Among potential candidates, three substitutes (sodium carbonate, glass powder and sodium chloride) were selected based on their fluxing ability as proposed by other literatures (Yan et al., 2017) (Al-hilli & Al-rasoul, 2010) (Su et al., 2012). The three potential substitutes (sodium carbonate, sodium chloride, glass powder) were substituted and each was homogenized with both dry and wet ball mill process. Here we tried to compare not only suitable substitute material for feldspar but also a better homogenizing technique for the preparation of porcelain insulator. The comparisons were made before selecting the final substitute material based on their dielectric strength.

The mixture of the raw materials was exposed to both dry and wet ball mill. Both milling processes lasted for 14hrs. After milling, the sample in the wet ball mill container was oven dried. The powders from both the wet and dry milling container was extracted and molded separately using hydraulic press. The molded samples were then fired using high temperature furnace.

3.3 Formation of porcelain insulator

Formation of porcelain insulator for the 1st step comparison was made according to table 3.1

Table 3.1 Percent composition of raw materials for the formation of porcelain insulator (Preliminary Substitute Sample (PSS))

Percentage (wt%)	Sample 1 (PSS1)	Sample 2 (PSS2)	Smaple 3 (PSS3)
45%	Bomboha Clay	Bomboha Clay	Bomboha Clay
35%	Arero Feldspar	Arero Feldspar	Arero Feldspar
10%	Arero Quartz	Arero Quartz	Arero Quartz
10%	Sodium Carbonate(Na_2CO_3)	Sodium Chloride(NaCl)	Glass Powder(gp)

The percent composition of each constituent was chosen by looking at a previous study's optimum dielectric strength achieving composition (45% clay, 45% feldspar and 10% quartz) and feldspar was partially substituted on the optimum composition to achieve the required amount of alkaline content. The powders were mixed according to Table 3.1 which shows the percentage of each constituent added to form the porcelain mix. The mix was then molded using a hydraulic press. A 20 MPa pressure was applied for 10 minutes to form the green body. The substitute containing pellets were then fired at 1250 ^{o}C and 1000 ^{o}C with heating and cooling rate of 6 ^{o}C /min and soaking time of 3hrs. These materials then were checked for their electrical performance. The best performing pellet with its processing technique was chosen to proceed to the next step.

Using the preliminary result, better substitute and processing technique was chosen to investigate the extent to which the processing temperature can be lowered and substitute can be added. Substitute percentage in the insulator was performed according to table 3.2.

Table 3.2 Percentage of substitute for formation of porcelain insulator (final substitute)

Component	Overall content (wt%)		
	Batch 1 (S1)	Batch 2 (S2)	Batch 3 (S3)
Bomboha Kaolin	45	45	45
Arero feldspar	40	37.5	35
Substitute(Na_2CO_3)	5	7.5	10
Quartz	10	10	10

Table 3.2 shows the percentage each powder was mixed to form the porcelain mix. The mix was then molded using a hydraulic press. A 20 MPa pressure was applied for 10 mins to form the green body. The green bodies were then fired according to table 3.3 which shows the firing temperature through which the good performing green body was passed through to get the final porcelain insulator. The firing process took place using firing and cooling rate of 6 ^{0}C /min and soaking time of 3hrs.

Table 3.3 Firing temperature of substitute (S1, S2 and S3 stands for Batch 1, 2, and 3, respectively, and T1, T2, T3 in turn stands for firing temperature 1, 2, and 3, respectively)

Firing Temperature	Batch (sample name)		
1000 ^{o}C (T1)	S1(S1T1)	S2(S2T1)	S3(S3T1)
1100 ^{o}C (T2)	S1(S1T2)	S2(S2T2)	S3(S3T2)
1200 ^{o}C (T3)	S1(S1T3)	S2(S2T3)	S3(S3T3)

3.4 Sample Characterization

Two basic characterization techniques were used to evaluate the final product, X-ray Diffraction (XRD) and Field Emission Scanning Electron Microscope (FE-SEM). XRD was performed to analyze the crystal structure and phase formation. In order to be characterized by XRD, the fired pellets were crushed until they became fine powders. FE-SEM was also performed to analyze the morphology of the fired structure. Before performing FE-SEM tests, the samples were grinded and etched in HF(48%) for 30 seconds (Al-hilli & Al-rasoul, 2010). XRD and FE-SEM were performed in Adama Science and Technology University, Department of Materials Science and Engineering and Sungkyunkwan University, South Korea.

3.5 Physical and Electrical properties

Physical properties including apparent porosity, water absorption and bulk density were inspected using ASTM standard ASTM C20 – 00. The samples were first measured for their dry weight (D) and then immersed in a container containing distilled water, which is then let to boil for 2 hrs. The samples should be hanged so that there is no contact between the samples and the surface of the container. After boiling the water, the samples were left immersed in the water for 12 hrs and then their suspended weight (S) was measured while in water. Finally, the samples were dried using clean towel and their saturated weight (W) was measured. The apparent porosity, water absorption, and bulk density were later calculated using Equations 3.1 to 3.4 and all the calculations are made assuming 1g=1cm^3 according to ASTM standard ASTM C20 – 00.

Exterior volume V (cm^3) = W - S 3.1

Apparent porosity P (%) = [(W - D)/ V] ×100 ....3.2

Water absorption A (%) = [(W - D)/D] ×100 .... 3.3

Bulk density B (g/cm^3) = D/V... 3.4

The dielectric strength of the porcelain insulator was investigated using TERCHO-HV which is available in Addis Ababa Institute of Technology, Addis Ababa University. TERCHO-HV is a high voltage machine that displays the break down voltage of the material. The sample was first immersed in an oil medium (because air breaks at low voltage) and exposed to high voltage. The dielectric strength values were later calculated using equation 3.5.

Dielectric strength = Break down Voltage (kilovolts) / Thickness of the pellet (mm) 3.5

4. RESULTS AND DISCUSSION

The process followed to produce porcelain insulator samples is illustrated on Fig 3.1. As shown in Fig 3.1, the raw materials were first crushed and sieved with their respective particle size requirement. The sieved powders were then mixed according to their respective proportion and milled for 14 hrs. The homogenized mixtures were then oven dried and molded to have a cylindrical shape with appropriate thickness. Finally, the molded samples, green body, were fired at different temperatures. The final pellets were characterized with FE-SEM and XRD to understand the morphology and phase formation within the pellets. Moreover, the pellets were inspected for their electrical and physical properties.

4.1 Preliminary Results

At first, preliminary experiments were carried out to select the better substitute among sodium carbonate (Na_2CO_3), sodium chloride (NaCl) and glass powder. The three were separately processed. Each substitute was integrated with the powder mix of Bombowha clay, Arero feldspar and quartz by substituting 10% of feldspar from the optimum performing mix as proposed by (Merga et al., 2019). The process was performed using different milling processes at different temperatures. Digital image of the as prepared samples processed at a temperature of 1250 ^oC for both milling techniques is shown in Fig. 4.1a. As shown in Fig. 4.1a, the wet ball milled samples that contain NaCl showed visible porous structure, certain volume expansion as well as lost the cylindrical shape of the original green body. As for the sample containing Na_2CO_3, the pellet melted, shrank and lost the green body's cylindrical shape and exhibited an oval one. Other than losing the green body's shape, the Na_2CO_3 based sample was compact and dense unlike the one containing sodium chloride while the glass powder based pellet maintained the original green body shape. In the case of the dry ball milled samples fired at 1250 ^oC, the sample containing NaCl still displayed a porous structure but maintained its shape unlike the one processed by wet ball milling process. The sample containing Na_2CO_3 also melted and lost its shape but maintained its dense structure. The glass powder based sample again maintained its shape regardless of the milling techniques. In order to proceed to the next step, the substitute NaCl was left out because the NaCl based sample displayed a visible porous structure, the NaCl substitute was not helpful in making the pellets denser. The samples that lost the original green body shape were not suitable to test their dielectric strength because they have lost their shape.

As a result, the samples were fired at lower processing temperature to compare the effect of the substitutes in the final porcelain insulator.

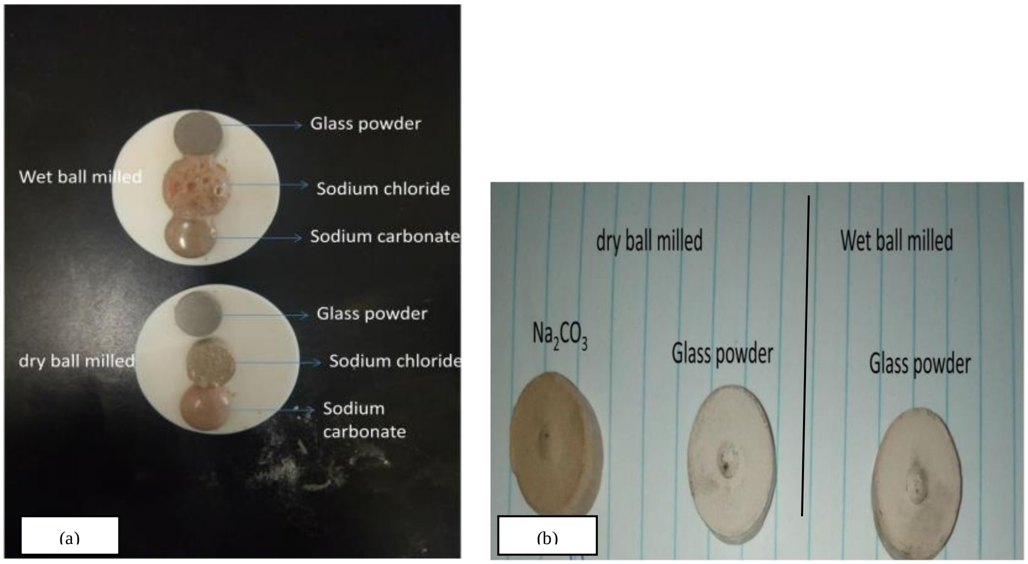

Fig 4.1 (a) Wet and dry ball milled porcelain pellets fired at 1250 ^{o}C containing different substitutes (glass powder, NaCl and Na_2CO_3) and (b) Wet and dry ball milled porcelain pellets fired at 1000 ^{o}C containing different substitutes (glass powder and Na_2CO_3)

Before firing the wet ball milled mixes using Na_2CO_3 substitute, a separate accumulation of each raw material that makes up the porcelain was observed due to the less dense behavior of the Na_2CO_3. It was observed that the dense materials like quartz and feldspar accumulated at the bottom of the container while the less dense Na_2CO_3 accumulated at the top. The mix was not homogenized well and because of this reason the wet milling process for the mix containing Na_2CO_3 substitute was dropped. The wet ball milled mix containing glass powder was homogenized well. Both the dry ball milled mixes containing Na_2CO_3 and glass powder were homogenized well. The three samples, wet and dry ball milled glass powder based samples and dry ball milled Na_2CO_3 based sample, were fired at 1000 ^{o}C. The pellets formed are as shown on fig 4.2 (b). The three samples were then inspected for their dielectric strength by testing their break down voltage. Table 4.1 describes the values of the breakdown voltage, thickness, and their respective dielectric strength.

Table 4.1 Break down voltage, thickness and dielectric strength of dry and wet ball milled glass powder substitute containing pellet; and dry ball milled Na_2CO_3 substitute containing pellet fired at 1000 ^oC

Sample	Break Down Voltage (kV)	Thickness (mm)	Dielectric Strength(kv/mm)
Glass Powder (Wet milled)	18.11	6.55	2.76
Glass Powder (Dry milled)	30.21	8.33	3.62
Na_2CO_3 (Dry milled)	31.1	8.25	3.76

As shown the preliminary result, Table 4.1, the dry ball milled mix that contains Na_2CO_3 have a better dielectric strength than the glass powder based substitute using both milling techniques. Due to this result, Na_2CO_3 was selected as the best substitute of the three. Then the Na_2CO_3 was added in different percentages in the mix and fired at three different temperatures to understand the phase transformation and the physical and electrical properties of the processed porcelain insulator. Table 3.2 and 3.3 show the amount of Na_2CO_3 that has been added in the mix and the firing temperature at which each mix with its respective substitute content, respectively.

The three batches were mixed, dry ball milled, molded and fired at different temperatures separately. All of the fired samples maintained their shape and were suitable to test the dielectric strength and all other characterization and performance testing techniques. The pellets were then characterized using XRD and FE-SEM techniques.

4.2 Characterization of phases

As a material is fired from a certain temperature to a higher one it is obvious that there is a phase change. Especially ceramics go through a very complicated phase change when they are processed through a very high temperature. These phases are essential and necessary for the property and performance of the final material. As the green body made up of the mixes of clay, feldspar, quartz and sodium carbonate is sintered, there will be an interaction between individual raw materials to create intermediate phases and finally transform into phases that determine the final property of the porcelain. Mullite phase, which forms within 1000 ^{o}C to 1200 ^{o}C, is one of the important phases required for the good electrical and physical performance of porcelain insulator. The samples were investigated to see if there is a possible formation of such phases.

4.2.1 X-ray Diffraction

X-ray diffraction analysis was performed on each sample to study the effect of change in substitute concentration and firing temperature on the required phase formation.

The first, second and third batches were crushed and powdered to make them suitable to be investigated in the XRD. Each batch is plotted according to its firing temperature. Fig 4.2 shows the XRD patterns of the three batches fired at 1000 ^{o}C.

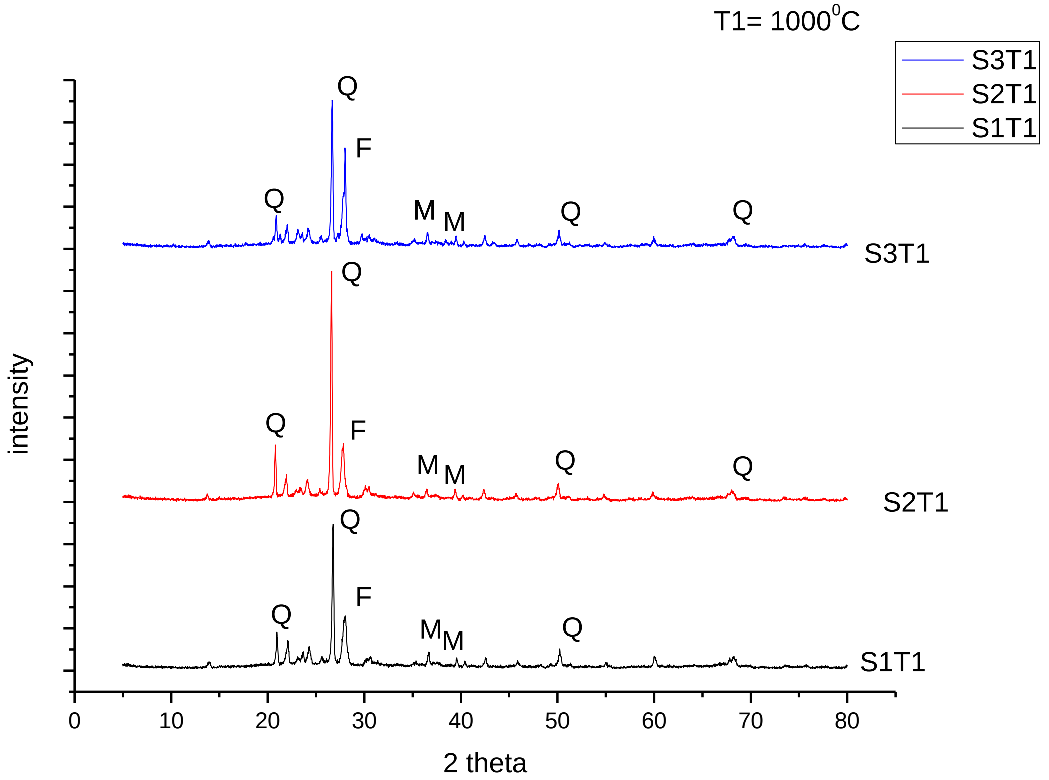

Fig 4.2 Powder XRD patterns of porcelain having different Na_2CO_3 concentration fired at 1000 ^{0}C. The diffraction peaks are identified as Q- quartz, F- feldspar and M-mullite

The presence of quartz can be observed from the peaks at 22^{o},26^{o}, 50^{o} and 70^{o} on Fig 4.2. Feldspar is also present at 1000 ^{o}C as can be seen from the peak at 27^{o}. The existence of free silica attributes to glassy phase formation which has an amorphous structure and cannot be displayed in the XRD pattern because it has an amorphous structure. The γ-Al_2O_3 spinel also nucleates at around 900 °C. However, it is controversial that what kind of crystal structure γ-Al_2O_3 possesses but it will transform to a mullite crystalline structure between 1000 °C to 1100 °C (Carty, 2020). It can be observed that at around peak 36° and 38° mullite peaks appeared showing mullite phase might have nucleated at a lower temperature.

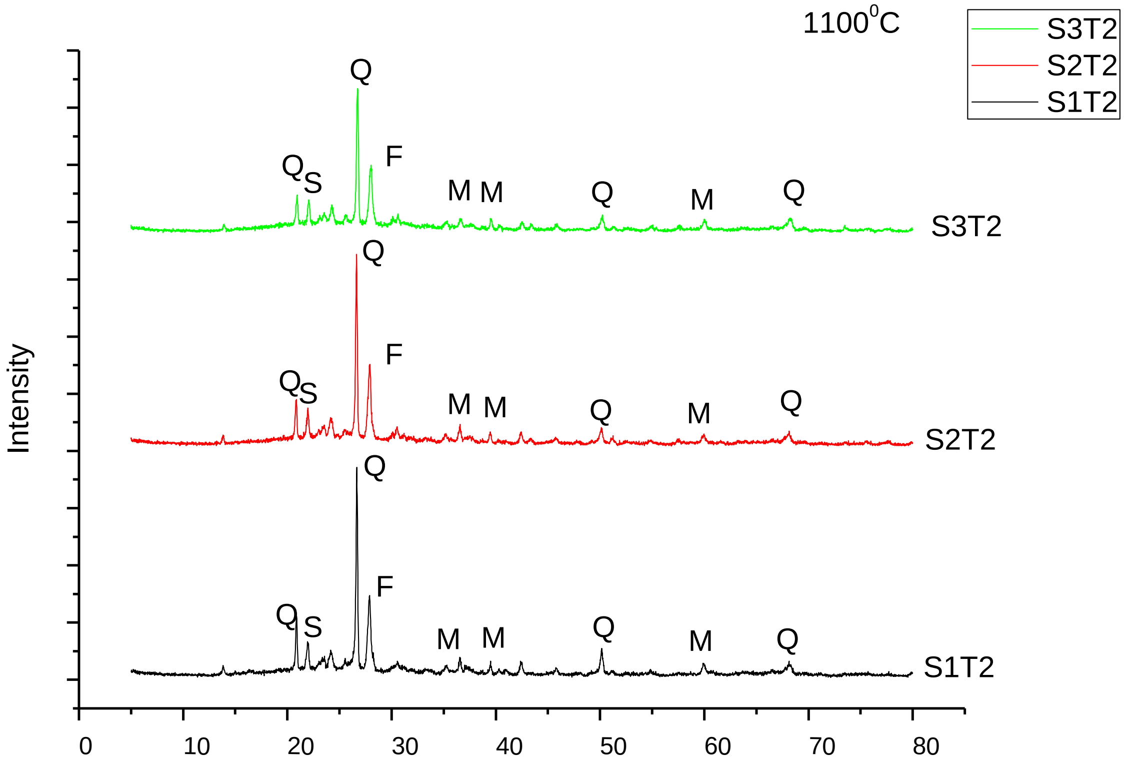

Fig 4.3 Powder XRD patterns of porcelain having different Na_2CO_3 concentration fired at 1100 °C. The diffraction peaks on the diagram are identified as Q-quartz, s-sanidine, F-feldspar and M - mullite

The XRD peaks for batch 1, 2 and 3 fired at 1100 °C are as shown in fig 4.3. Here, it can be observed that quartz and feldspar existed just like the batches processed at 1000 °C. But unlike the first batch sanidine peak was observed (RRUFF, n.d.). This might be attributed to the fact that formation of sanidine phase depends on the sodium to potassium ratio(Carty, 2020). Unlike the first batches fired at 1000 °C, an interesting phase, mullite phase, was observed at 1100 °C as shown in Fig 4.3. This shows that nucleation and growth of primary mullite phase took place above 1000 °C as described by (Iqbal, 2008). If sufficient glassy phase forms around the primary mullite phase, secondary mullite phase can easily nucleate (Meng et al., 2012).

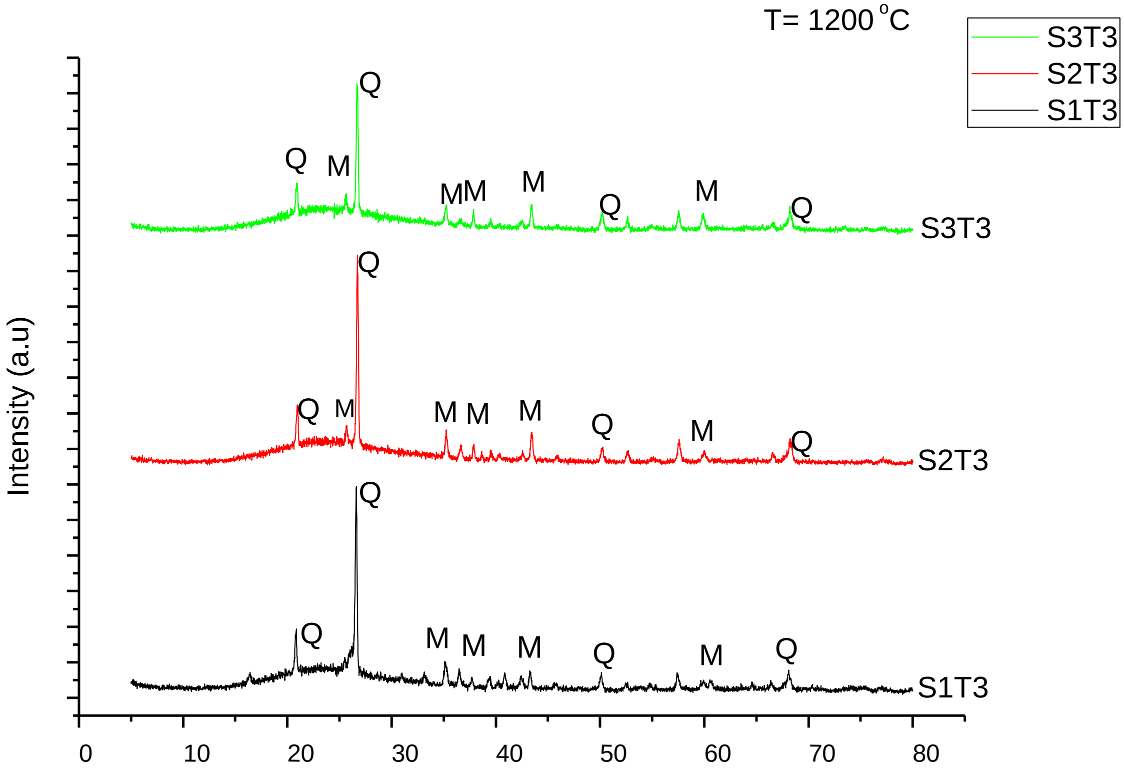

Fig 4.4 Powder X-ray patterns of porcelain having 5%, 7.5% and 10% sodium carbonate containing porcelain insulator fired at 1200 °C. The diffraction peaks on the diagram are identified as Q-quartz and M – mullite.

At the sintering temperature of 1200 °C, as illustrated in Fig 4.4, all the three batches showed quartz peak while the feldspar and sanidine peaks disappeared. Sufficient mullite peaks were also observed showing the required phase was created at this temperature. Batch 1(S1) showed lower mullite peaks than batch 2(S2) and Batch 3 (S3). For instance, at 26^0 mullite phase is observed for S2 and S3 but not for S1. This shows that although mullite phases are observed at all substitute concentration, more mullite phase appears as the substitute concentration has been increased. At around 53^0 a peak was observed which might be attributed to the existence of corundum which is an aluminum oxide that contains traces of iron (Meng et al., 2012). This is better seen on Fig 4.5 which shows the XRD pattern of S3T3.

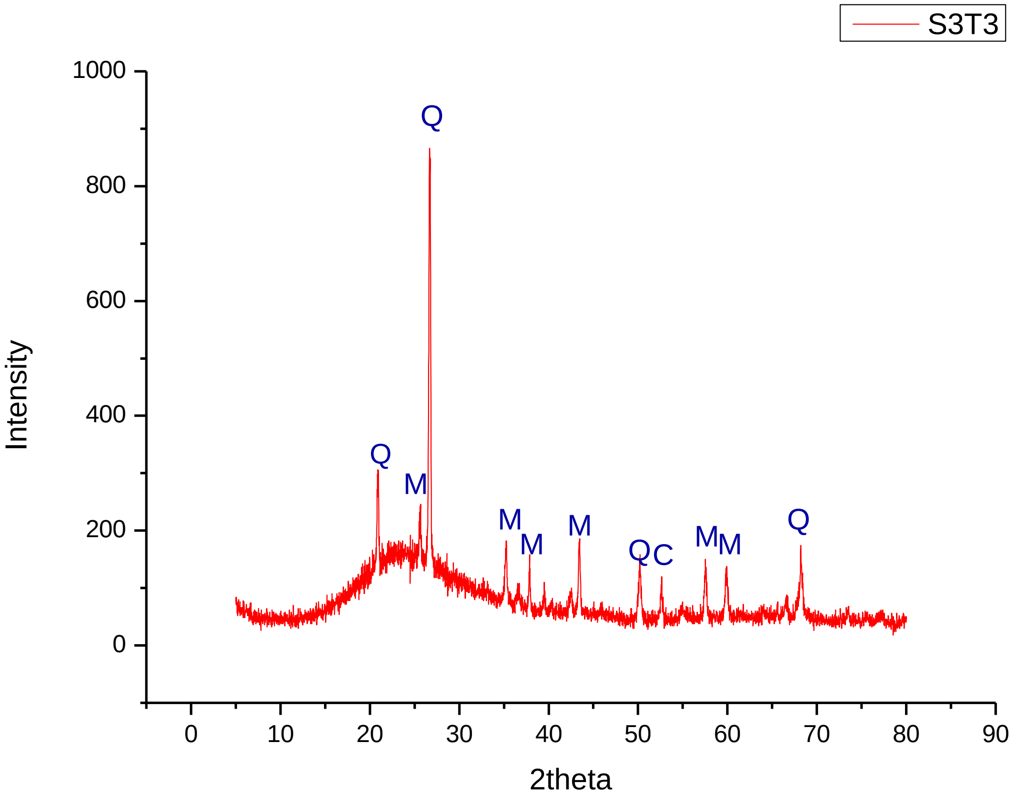

Fig 4.5 XRD pattern of batch 3 fired at 1200 ºC. The diffraction peaks on the diagram are identified as Q-quartz, C- corundum, and M – mullite

The mix with the highest substitute concentration and the highest firing temperature shows more mullite peaks than most of the samples. Peaks at 26º, 34º, 37º, 44º, 58º and 60º 2theta values belong to the mullite phase.

4.2.2 Field Emission-Scanning Electron Microscope

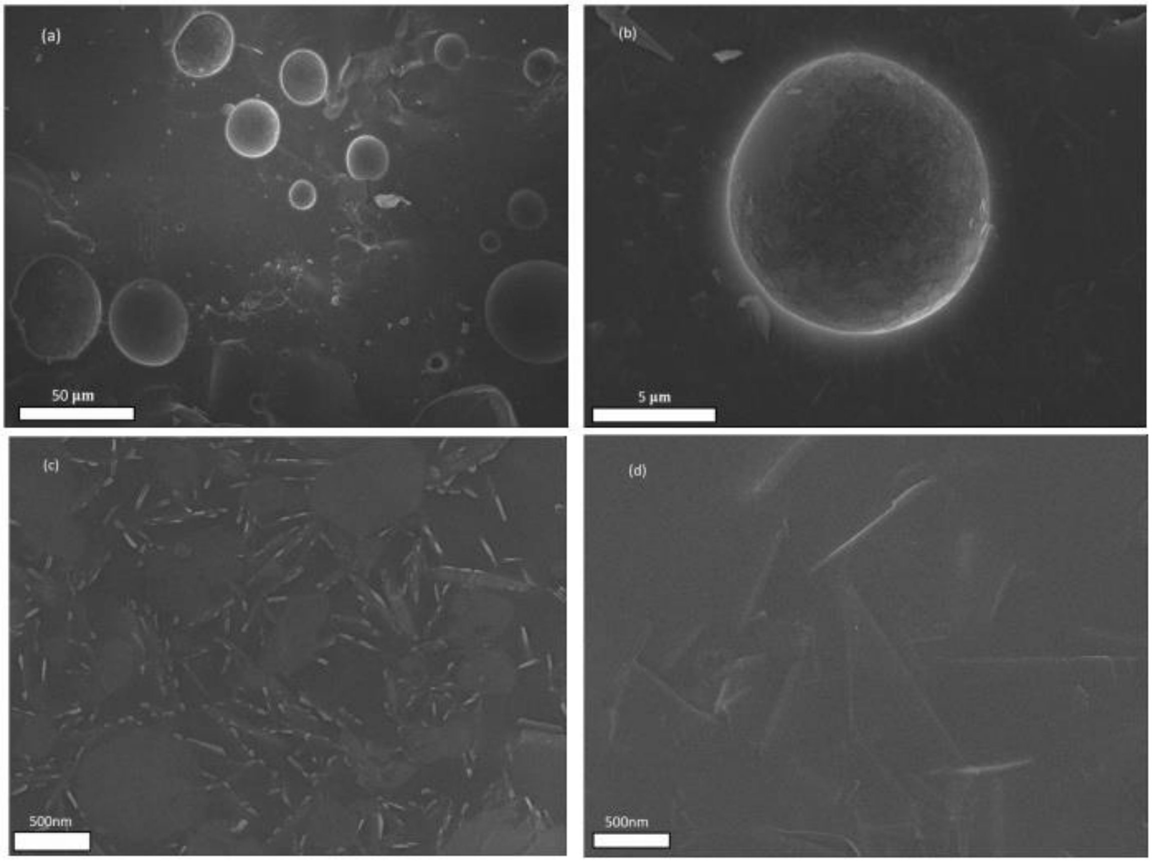

Fig 4.6 SEM image of un-etched porcelain sample (S3T3)

The FE-SEM image in Fig 4.6 shows a buble like structure which is the glassy phase which might be displayed as transparent structure. This shows that the glassy phase has covered the surface of the sample and should be etched before inspecting for possible structures on SEM.

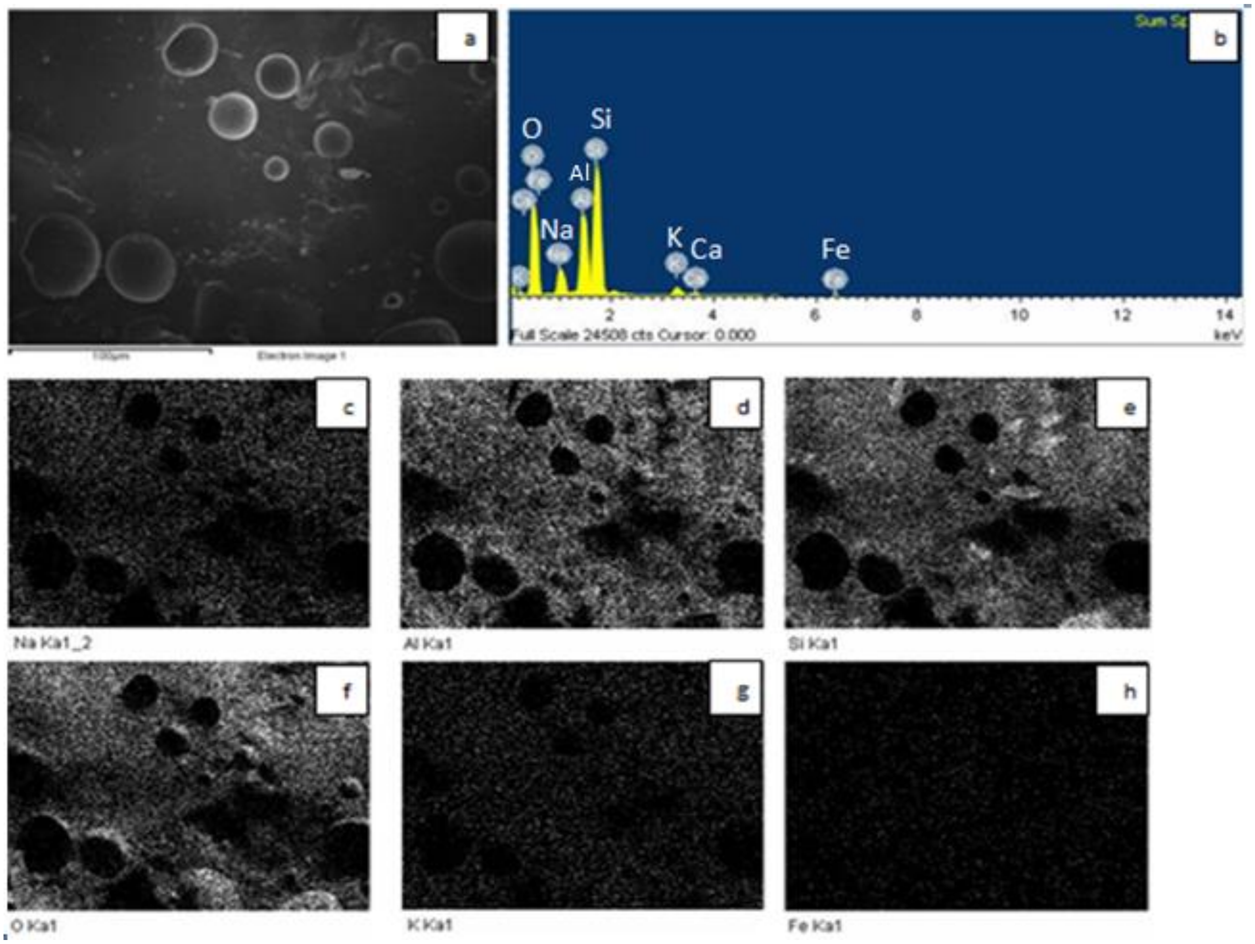

Fig.4.7 EDS and EDS mapping for pellet containing 10% Na_2CO_3 fired at 1200 ºC : (a)SEM image of unetched Na_2CO_3 containing porcelain pellet; (b) EDS spectra of the existing elements; elemental distribution of (c) Sodium (d) Aluminum (e) Silicon (f) Oxygen (g) Potassium (h) Iron

The EDS mapping showing different constituent elements found in S3T3 sample are shown on Fig 4.7. The EDS spectra on fig 4.7(b) shows that all elements (Al, Si, O, K, Na) necessary for mullite phase and glassy phase formation are present which is backed by the EDS mapping in fig 4.7 (c-g) which shows that the individual elements dispersed in the pellet. These elements (Al, Si and O) are essential for the formation of mullite phase ($3Al_2O_3 \cdot 2SiO_2$). The XRD results (Fig 4.3, 4.4 and 4.5) also revealed the appearance of mullite phase, supporting the EDS mapping. In addition it is seen that in Fig 4.7(c) sodium is dispersed on the surface which shows the substitute has increased alkaline content in the porcelain. This might attribute to the formation of the glassy which in turn might have covered the surface. The EDS mapping in Fig 4.7 reveals the bubble like structures, shown in Fig 4.6 (a) and (b), as a dark hole. As etching was performed on the sample, the structures disappeared (as shown on Fig 4.8 and 4.9) showing that they might be glassy phase structures. Iron was also observed in Fig 4.7(h) as it is the common impurity for porcelain insulators.

The pellets fired at 1200 ^{o}C were etched using Hydrofluoric acid (48%). The etching was performed by grinding the samples and immersing them in the acid solution for 30 seconds (Al-hilli & Al-rasoul, 2010). The etched samples were then inspected using SEM.

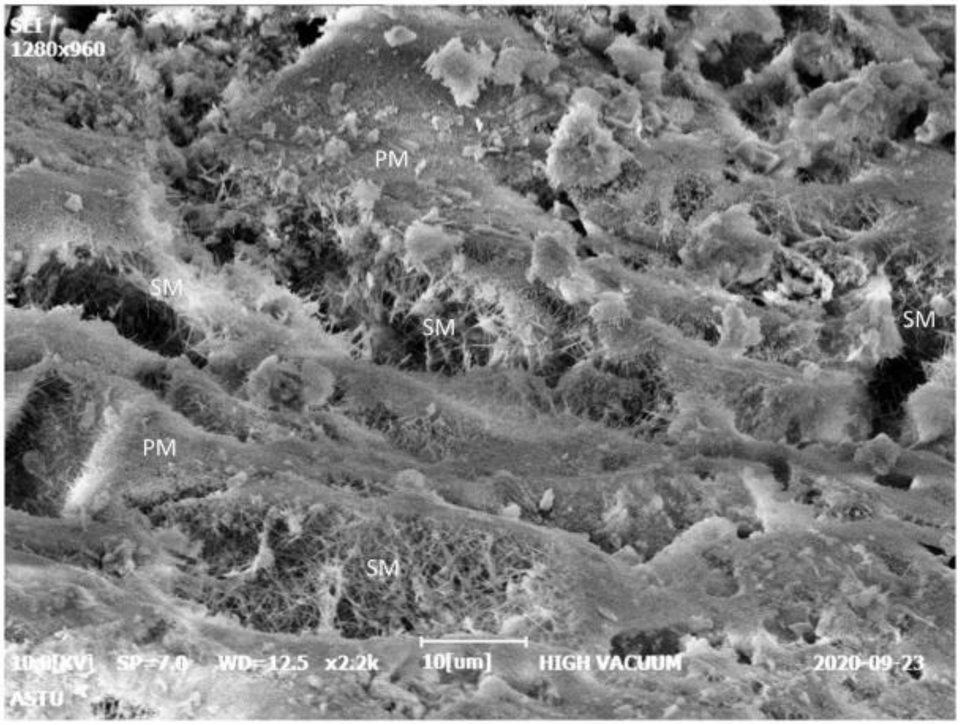

Fig 4.8 SEM image for batch 1 fired at 1200 ^{o}C, indication represents PM- primary mullite, SM- secondary mullite

The FE-SEM image of S1T3 in Fig 4.8 shows the successful formation of primary and secondary mullite phases at 1200 ^{o}C. The one which have a needle like structure entangled together represent the secondary mullite phase. The primary mullite phase is the origin for the secondary mullite phase, as the secondary mullite phase originates from the interface of glassy phase and primary mullite phase (Iqbal & Lee, 2000). The SEM image in fig 4.8 supports the XRD results regarding the formation of mullite phase since both the XRD and SEM results revealed the existence of the mullite phase at 1200 ^{o}C.

Fig 4.9 shows the SEM image of S2T3 and S3T3 after etching. The SEM image reveals that the surplus amount of the formation of the glassy phase as the substitute percentage in the mix was increased. Although S2T3 and S3T3 samples were etched, both the primary and secondary mullite phases were not observed in the SEM images due to the presence and coverage of the glassy phase; it should be noted that the respective XRD pattern revealed the presence of the mullite phase in both S2T3 and S3T3 samples. Based on the XRD and SEM results, it can be said that there is a surplus amount of glassy phase formation at 1200 °C which confirms the substitute used in this study is a good flux that melts and binds the particles at lower temperature.

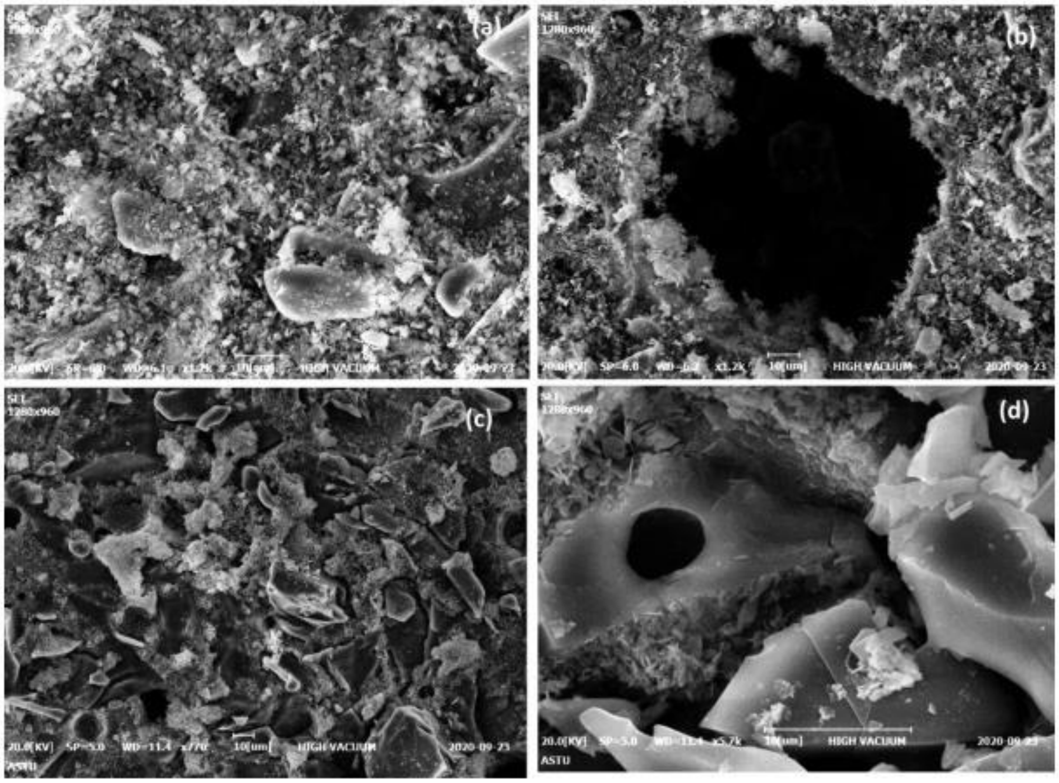

Fig 4.9. FE-SEM images of (a) and (b) etched S2T3 and (c) and (d) etched S3T3

Fig 4.9 (d) clearly shows the glassy phase in S3T3 and the etching time can be further increased to remove the glassy phase effectively for substitute percentages of 7.5 % and above in order to see the mullite phases using SEM.

4.3 Electrical and physical properties of the porcelain insulators

4.3.1 Dielectric Strength

Electrical property of a porcelain insulator was measured to understand how much it is capable of resisting electric current passage through it. A parameter that explains the ability of a material to insulate electricity is the dielectric strength. Dielectric strength is the measure of the maximum voltage an insulator is capable of insulating, i.e break down voltage, divided by the thickness of the sample. The breakdown voltage of the samples was measured using TERCO HV 1103 break down voltage testing machine. All tests were performed using an oil medium because air breaks earlier than the oil. Table 4.2 shows the break down voltage, thickness and dielectric strength of each samples. All the samples broke at a certain voltage except the ones fired at T3. For the samples fired at T3, the oil medium was unable to hold to the high voltage it was subjected to and so the oil broke before the samples. This was because the samples were thick and can withstand high voltage. Due to this reason reducing the thickness of the samples was essential. Reducing the sample thickness was performed by molding less amount of powder in the hydraulic press. After reducing the thickness of the samples by repeating the whole molding and firing procedures, the samples were again subjected to high voltage testing machine. The result is shown on table 4.3.

Table 4.2 Break down voltage, thickness and dielectric strength of each batch fired at three different temperatures of 1000 ^{o}C, 1100 ^{o}C and 1200 ^{o}C

Sample	BREAKDOWN VOLTAGE(kV)			THICKNESS(mm)			AVG BDV (kV)	AVG THICK (mm)	DIELECTRIC STRENGTH (kV/mm)
	1	2	3	1	2	3			
S1T1	9.2	10.2	11.2	6.8	6.77	6.87	10.2	6.81	1.5
S1T2	18.2	21	19	6.72	6.81	6.84	19.4	6.79	2.86
S1T3	31	37	38	6.81	6.78	6.83	35.3	6.80	5.19
S2T1	11	10.3	11.3	6.79	6.84	6.8	10.87	6.81	1.6
S2T2	20.6	20	21	6.84	6.88	6.82	20.53	6.84	3.00
S2T3	38.8	40.3	45.3	6.86	6.81	6.79	41.46	6.82	6.08
S3T1	11.5	11.6	11.6	6.77	6.84	6.82	11.56	6.81	1.7
S3T2	29.9	30.3	32.1	6.81	6.81	6.78	30.76	6.8	4.52
S3T3	45.1	45.3	45.1	6.82	6.76	6.83	45.17	6.8	6.64

Table 4.3 Dielectric strength after reducing thickness of the samples fired at T3

SAMPLE	BREAK DOWN VOLTAGE (kV)		THICKNESS (mm)		AVG BDV kV	AVG THICK Mm	DIELECTRIC STRENGTH (kV/mm)
S1T3	23.1	22.8	3	3.2	22.95	3.1	7.40
S2T3	24.8	24.9	3.3	3.4	24.7	3.35	7.42
S3T3	25.2	25.1	3.3	3.4	25.15	3.35	7.51

The result showed on Table 4.2 and 4.3 shows that the dielectric strength of the samples increases with increasing firing temperature. This is an expected result which matches with the XRD results shown on Fig 4.2, 4.3 and 4.4 since the important phase for good electrical performance; i.e mullite phase, appeared in the XRD patterns as the temperature was increased. In addition, this result explains why samples fired at low temperature have lower dielectric strength than those fired at higher temperature. The effect of substitutes and their concentration on dielectric stregnth can also be observed on Table 4.2 and 4.3. As the substitute concentration increases, at the same temperature, there was an increase in the dielectric strength. The increase in substitute concentration might attribute to the formation of sufficient glassy phase to initiate the nucleation and growth of mullite phase that in turn increases its dielectric strength. The maximum dielectric strength achieved in this work is 7.51 KV/mm which is the sample with the highest substitute concentration (10 %) and firing temperature (1200 ^{o}C). As shown in Table 4.3, all the dielectric strength values obtained at 1200 ^{o}C are within the range of the standard for porcelain insulators that is in the range between 6.1-13 KV/mm (Olupot et al., 2010). The optimum sample in this study exhibited a dielectric strength of 7.51 kV/mm at lower temperature of 1200 ^{o}C and is comparable with a previous study that achieved 8 KV/mm at firing temperature of 1300 ^{o}C (Merga et al., 2019).

4.3.2 Physical property

Physical properties of the optimum three samples that showed good dielectric strength was investigated. ASTM standard (ASTM C20 – 00) was followed to test the physical properties of the pellets. The weight of the samples at different conditions is as listed on Table 4.4.

Table 4.4 Dry weight, suspended weight and saturated weight of the samples that have good dielectric strength

Samples	Dry weight (D) Gm			Suspended weight (S) Gm			Saturated weight (W) Gm		
	S1	S2	Average	S1	S2	average	S1	S2	Average
S1T3	3.86	2.75	3.31	2.25	1.61	1.93	3.86	2.75	3.31
S2T3	5.35	4.08	4.71	3.05	2.32	2.67	5.35	4.08	4.71
S3T3	4.41	2.47	3.44	2.33	1.37	1.85	4.41	2.47	3.44

The first step was to determine the volume of the dry mass. The volume of each sample was calculated according to Eqn 3.1. The calculated volume of the dry masses of S1T3, S2T3, and S3T3 was 1.38 cm^3, 2.04 cm^3, and 1.59 cm^3, respectively.

The apparent porosity was also calculated according to Eqn 3.2 showed that all the samples fired at T3 do not have porous structure. This is due to the densification of the fired bodies took place at that temperature. The calculated apparent porosity of S1T3, S2T3, and S3T3 were all zero percent and can be a good indicator that the substitute material was an effective flux in filling pores and making the samples denser. This result out performs the result achieved in the previous report at this temperature which is around 2.9% (Merga et al., 2019).

Water absorption shows the amount of water absorbed by a certain material at a specific condition. The result was the same as the apparent porosity showing densification of the pellets at the lowered temperature.

The last physical property of the porcelain insulators to be evaluated was the bulk density. Bulk density measures the amount of matter in a cubic centimeter volume. Higher bulk density value shows that there is more amount of matter available in that specific volume; meaning the material is dense. The bulk density of each sample was calculated according to Eqn 3.4. The calculated bulk density of S1T3, S2T3, and S3T3 was 2.40 g/cm^3, 2.31 g/cm^3, and 2.16 g/cm^3, respectively. The three batches fired at 1200 ^oC showed a reasonable bulk density showing that the samples were compacted due to the melting of the substitute at lower temperature allowing the porcelain insulator attain good physical property.

It is evident from the observed physical properties in this study that the substitute in the feldspar is a good flux material that contributes to the compact and dense nature of the as prepared porcelain insulators.

The effect of lowering the firing temperature of porcelain insulators in the microstructure evolution and formation of phases was characterized and their effect on the physical property and dielectric strength was as shown on the above section. As the temperature increased the kaolin in the Bombowha clay transformed to metakaolin which latter transformed to γ-Al_2O_3 spinel. This was followed by the transformation of the γ-Al_2O_3 spinel to primary mullite phase. This primary mullite phase acquired sufficient glassy phase to transform to secondary mullite phase (Carty, 2020). This is shown on the XRD and FE-SEM results. The XRD (Fig 4.4) and FE-SEM results (Fig 4.8) obtained by substituting 10%, 7.5% and 5% of the amount of feldspar in the porcelain mix by sodium carbonate shows that sufficient amount of glassy phase and mullite phase formed.

An increase in the strength of the mullite phase will occur when the polycrystalline mullite contains a small amount of glassy phase at the grain boundaries. This occurs due to the stress relaxation and crack healing by the glassy phase at high temperatures (Anggono, 2005). The transformation of primary mullite phase to secondary mullite phase occurs as the alkalines from the feldspar and the substitute diffuse towards the primary mullite phase and grow to form the secondary mullite phase (Meng et al., 2012). The XRD and FE-SEM results show that mullite phase has successfully nucleated. The mullite phase appeared on the pellets processed at 1100 ^{0}C and 1200 ^{o}C as shown on Fig 4.3 and Fig 4.4 (which shows mullite peaks). This result matches with literatures that mullite phase nucleates and grows above 1000 ^{o}C (Carty, 2020). The FE-SEM result on Fig 4.8 reveals a needle like structures that are the secondary mullite phases.

The mullite phase is determinant for the dielectric strength and physical properties of the porcelain insulator (Schneider et al., 2015). This in turn justifies the good dielectric strength results obtained by substitution addition at lowered temperature. The pellets with 5%, 7.5% and 10% substitute showed an excellent dielectric strength as they all have a dielectric strength above 7kV/mm. As mentioned earlier the results are within the range of the standard dielectric strength for porcelain insulators to be used as overhead lines.

It was observed that the increase in the dielectric strength as the substitute concentration increased didn’t show a huge gap. This shows that small amount of addition of Na_2CO_3 is sufficient enough to lower the firing temperature of porcelain insulator with appreciable dielectric strength.

5. Conclusion and Recommendation

5.1 Conclusion

In conclusion, this work was successful in the production of porcelain insulators from locally available raw material at lowered firing temperature. The study used three different kinds of potential alkaline sufficient materials. Among the potential substitutes used in this study, Na_2CO_3 was efficient in providing alkalinity to the mix and was able to lower the sintering temperature of locally produced porcelain insulator, showing the use of alkaline-rich substitute fluxes was advantageous in the formation of necessary phases at lowered temperature and attaining the required properties. The as prepared samples were inspected for their electrical and physical performance. The pellet containing 10% sodium carbonate and fired at 1200 ^oC showed the best result of all the other batches. The best performing sample attained 7.5 KV/mm which falls within the qualification range to be used as low voltage overhead porcelain insulators (Olupot et al., 2010). The physical properties such as the apparent porosity, the water absorption and the bulk density of the pellets also showed that the substitute served in making a dense compact pellet. The characterization results showed that sufficient amount of alkalinity and essential phases responsible for the good electrical and physical performance were also present. The EDX results showed the presence of sufficient amount of alkalinity (Na, K) in the fired pellets which is due to the addition of sodium carbonate. Furthermore, the XRD results showed peaks for mullite phase for all the batches fired at 1000 ^oC, 1100 ^oC and 1200 ^oC. The FE-SEM results also revealed morphological structure of secondary mullite phase. These results were achieved by small percentage (5-10%) substitution of Arero Feldspar by Sodium carbonate which led to earlier densification and necessary phase formation for the locally produced porcelain insulator. In comparison to previous studies, the production temperature was lowered by 100 ^oC. The reduction in temperature will make it easy for local production of porcelain and reduce the energy and cost associated with its production. This study showed that it is possible to process porcelain insulator using locally available materials at lower processing temperature by substituting Arero feldspar by sufficient amount of sodium carbonate.

5.2 Recommendation

This work is helpful to produce porcelain insulator from locally available materials with less processing cost. This paper along with other papers will be helpful to guide through efficient production of porcelain insulators. For researchers in this area I recommend that testing of mechanical properties such as fatigue test is important. In addition it is also recommend to propose the effect of etching time on the SEM image of the 7.5% and 10% sodium carbonate containing pellets to analyze the mullite phase.

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