SUBSTITUTION OF RICE HUSK ASH FOR QUARTZ IN CERAMIC WALL TILES FORMULATION

BY

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DECLARATION

I hereby declare that the thesis entitled "Substitution of Rice Husk Ash for Quartz in Ceramic Wall Tiles Formulation" has been written by me in the Department of Industrial Design of the Faculty of Environmental Design, Ahmadu Bello University, Zaria, Nigeria under the supervision of Professor A.M. Ahuwan, Dr. A.D. Garkida and Dr. I.H. Mshelgaru. The information derived from the literature has been dully acknowledged in the text, and the list of references provided. No part of this dissertation has been previously presented for another degree or diploma at any university.

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CERTIFICATION

This thesis entitled "Substitution of Rice Husk Ash for Quartz in Ceramic Wall Tiles Formulation"by Opoku, Eric Vernon meets the regulations governing the award of Doctor of Philosophy in Industrial Design of Ahmadu Bello University, Zaria, Nigeria, and it is approved for its contribution to knowledge and literary presentation.

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DEDICATION

This work is dedicated to the memory of my late father Andrews Bonsra, mum Victoria OhenewaBonsra, wife, Faustian Opoku and daughters, Sarah and Sylvia Opoku.

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Abstract

The processing of rice has been cited as a source of waste that contaminates and pollutes the environment. Waste addition into production cycles represents an alternative form of restoration, which is interesting from both environmental and economic standpoints. Technological development trends towards utilization of waste as a means of cost reduction in industrial processing has attracted the use of rice husk ash (RHA) as a value added material. Rice husk which is an agricultural waste was investigated for its SiO_2 content and was utilized as a replacement for quartz for the formulation of wall tile bodies. In this study, the development of ceramic wall tile formulations containing silica from rice husk ash as a replacement for quartz was investigated. Six formulations using the raw materials, kaolin, ball clay, whiting, and quartz/rice husk ash were selected and used in the mixture design. The elemental analysis of the RHA generated from the open air burning of rice husk and the other materials used for the study was carried out using X-ray Fluorescence Spectrophotometer (XRF) and the result indicated that the RHA had a high concentration of SiO_2 (82.1%) with a very low impurity content. A process was developed to utilize the RHA in ceramic wall tile formulation. The experimental samples were fired in an electric kiln at the sintering temperature ranges of 1100^{o}C, 1140^{o}C and 1160^{o}C and characterized. The drying and firing shrinkage values were measured according to ASTM C 326 – 82, water absorption, apparent porosity and apparent specific gravity were determined by ASTM C 373 – 88 and flexural strength was determined by ASTM C 648. The morphological characterization of the formulations was performed by scanning electron microscopy (SEM) on fracture surfaces of the samples. At the optimum temperature of 1160^{o}C shows the typical sequence of enhanced densification. The sintering temperature of 1160^{o}C was found suitable for the wall tile formulations containing RHA. It was concluded that the ash generated from the burning of

rice husk can be utilized in the basic formulation for wall tile production as a partial or complete replacement of the raw material quartz.

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Glossary

Beneficiation The process of refining raw materials in order to optimize their performance or make them useful in a manufacturing process.

Calcite A crystalline variety of limestone containing not more than 5% magnesium carbonate.

Characterization The process of evaluating substances of which a material is made or composed.

Ceramic Tile A thin surfacing unit composed of various clays fired to hardness. The face may be glazed or unglazed

.

Cottoforte It is a Glazed ceramic tile, moulded by pressing, is produced by the method of double-fired at low temperatures. This material differs from the majolica in terms of mechanical strength that is derived from the compound in the mass

Body Ceramic made of one or more material designed for a special purpose.

Earthenware A low fired form of pottery or objects made from clay, which is porous and permeable.

Extrusion A process in which clay material is forced through a mould for the desired shape.

Leather Hard The condition of a clay body or paste when it has become firm but not dry.

Majolica It is a glazed earthenware pottery often associated with certain regions of Italy, although it also is produced in other parts of the world. They are usually decorated with brightly coloured lead glazes and best known for naturalistic style.

Modulus of Rupture It is a measure of the cohesive characteristics of the material forming the tile and relating to the degree of compactness and consistency.

Monoporosa These tiles are single-fired tiles with higher porosity and water absorption levels

Mosaic Tiles Ceramic mosaic tiles are defined by their size, generally less than 6 inches square. They may be glazed or unglazed.

Plasticity - The properties of a material that allow it to be shaped and to retain its shape and moisture.

Red Stoneware Tiles. These are tiles composed of typically Italian raw materials that produce a red vitrified tiled body suitable for indoor or outdoor use. Generally they are not glazed.

Porosity This is the **v**olume of pores in a fired tile relative to volume of tile body that is capable of absorbing moisture.

Shrinkage The decrease in the size of a clay object due to drying and firing.

Single-Fired Tiles Glazed tiles produced by the single-firing method in which the raw tile body and the glaze undergo a single pass through the kiln at high temperature.

Sintering Process of compacting and forming a solid mass of material by the action of heat which compels interstitial silica in a ceramic body to melt and help in fussing the entire body.

Slake To moisten dry clay with water in order to weaken bondage and enhance true chemical combination.

Terracotta Raw material used to produce unglazed, red body tiles generally extruded and 1/2” thick or more. Surface may be rustic, smooth, polished, or waxed for lustre.

Tile Density The amount, weight or mass of solid materials contained in a tile in relation to the volume of the tile. This has direct bearing on the amount of moisture the tile can absorb.

Water Absorption The quantity of water a tile can absorb expressed as a percentage of the dry tile weight. High water absorption corresponds to a porous structure, while compact, vitrified structures feature low water absorption.

White-body Earthenware Tiles. This type of tile is composed of raw materials producing a white tile body that can be coated with a transparent glaze and takes colour easily. They are normally used for wall tiles.

WarpageDimensional defect of a tile's surface flatness, a condition where one corner of a tile is not in the same plane as the other three.

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background

There have been remarkable increases in agricultural, industrial and economic growths in the recent decades that havecontributed to an improved quality and well-being for citizens. However, these production systems create by products and wastes which affect the environment. These effects may occur in the course of the production cycle, either during the initial phase of obtaining raw materials, during the transformation and production phase, during product distribution or when the consumer must dispose of products which are no longer required.

A greater proportion of the waste generated is still far being used in its totality as a product or by-product, making technological alternatives important in order to reduce its possibleimpact on the environment such as the leaching of potentially toxic substances into soils and groundwater, reductions in plant establishment and growth due primarily to adverse chemical characteristics of the waste, changes in the elemental composition of vegetation growing on the waste, and increased mobility and accumulation of potentially toxic elements throughout the food chain.

Social concerns about the problem of waste management in recent years have been rising due to the growing quantity of agricultural, industrial, construction and demolition wastes generated, despite the measures which are taken at national and regional levels aimed at controlling and regulating waste management, in accordance with sustainable development policies. Sustainable development, according to Junkes, Carvalho, Segadaesand Hotza (2011) requires a reduction in consumption of natural raw materials that are not renewable for the generation of new products from recycled waste.

In addition to helping to protect the environment, there are many reasons why theincreased amount of waste ought to be utilized or re-utilized. Firstly, disposal costs are minimized. Secondly, less area is reserved for disposal, thus enabling other uses of the land and decreasing disposal permitting requirements. Thirdly, there are financial returns from the sale of the by-product that could offset the processing and disposal costs: and fourthly, the use of such waste offers a series of advantages such as a reduction in the use of other natural raw materials, contributing to an economy of resources. (Ahmaruzzaman,2010).

Wastes such as ashes from coal, municipal solid waste, wood, and so on, have exhibited good potential in the production of ceramic wares. Similarly, various ceramic systems have shown their suitability for making products that are thermally and mechanically stable and also exhibit good chemical durability. The studies by Mishulovich and Evanko (2003)and Zimmer & Bergmann (2010)are examples of ceramic tiles raw materials obtained from fly ash of the mineral coal. The results obtained by these researchersshows that fly ash, when mixed with traditional raw materials, has the necessary requirements to be used as a raw material for the production of ceramic tiles. Attempts have also been made by El-Fadaly, Baka, andBreka (2010) and Junkes, et al., (2011) with regards to the recycling of ceramic industry and industrial wastes for ceramic tile formulations.

Torres, Fernandez, Agathopoulos, Tulyagannov and Ferreira, (2004) in the area recycling have investigated the potential profit of incorporation of granite waste as raw material in porcelain-tile industrial formulations, particularly with regards to environmental and economic aspects, while Freire and Halanda (2006) characterized avian eggshell waste with the aim of using it in a ceramic wall tile paste. In an attempt to find alternative ways to reuse of waste for ceramic productions, Okunna (1983) characterized available ashes around Zaria, Nigeria for the development of celadon, temmoku and chun glazes.

The ceramic industry covers a diverse range of compounds and several products that can be manufactured by different methods. This industry therefore, presents favourable conditions for the implementation of waste recovery systems. Ceramic products made from natural (conventional

ceramics) and technological (technical ceramics) using biogenic materials like rice husk ash are known to havebeen studied (Zakharov and Belyakov 1995).

It has been found for example that when quartz is replaced with rice husk ash (RHA) which is an agricultural waste in the traditional whiteware composition, it drastically reduces both the maturing temperature, percentage of thermal expansion and increases strength marginally due to the significant reduction of the quartz phase and the simultaneous increase of the glassy phase (Prasad, Maitiand Venugopal 2001). Such a reduction in the maturing temperature will help to reduce the consumption of energy.

If we consider the fact that enormous quantities of various kinds of waste that is generated in the country possesses similar characteristics that are needed for building materials like bricks and tiles, one needs not worry about the paucity of raw materials because some of these waste materials are consistent in providing the same results for some ceramic minerals. This factor among others gives the motivation for this research.

1.1 Statement of the Problem

During the production of rice, huge amounts of rice husk are left as waste product. The husk is usually disposed of by burning, thus giving rice husk ash (RHA). This by product is produced in many rice producing places world-wide and constitutes an environmental problem due to air and water pollution. On the other hand, chemical analysis shows that a white variety of RHA contains up to 80-90% silica by weight. It is therefore, not unusual that this cheap source of value added product that is siliceous may be of interest for numerous industrial applications including the production of whitewares. It has also been found that the replacement of quartz with rice husk ash reduces the maturing temperature and also increases the strength of the tile marginally (Prasad, et al.,2001).

The problem of this research is the exploration of Rice Husk Ash [RHA] as a replacement for quartz in the formulation of a whiteware body for the development of ceramic wall tiles bodies.

1.2 Aim and Objectives of the Study

The aim of this study is to use rice husk ash as a replacement for quartz with a view of producing ceramic wall tiles.

The study intends to:

i. find a sustainable source of rice husk ash.

ii. find ways of utilizing the ash from the burning of rice husk for the production of ceramics tile.

iii. develop a whiteware body that has good strength properties and can mature at a lower temperature for the production of ceramics tiles.

iv. determine the fired strength of the matured specimens of all the body mix soas to find their suitability for ceramic tiles production.

v. determine the physical, chemical and mechanical properties of the body mix and their effects on the quality of the tiles produced.

1.3 Justification of the Study

The increasing demand of ceramic tiles for both the floor and wall in recent times has led the researchers to become interested in developing this field particularly, fashionable but low cost ceramic tiles that are very much lucrative for interior designing as well as for real estate housing developments. In fact, construction industry is now widely using ceramic tiles because of its variety of impressive colours, textures and design.

Consequently, cost-effective and acceptable methodologies for the manufacture of ceramic tiles are highly desirable. Specifically, utilisation of low cost, easily available natural resources like clay, feldspar, quartz and carbonates as raw materials for ceramic tiles production has no doubt made a significant step forward in this regard (Jahan, Parveen, Ahmed and Zaman, 2008).

Nowadays, industries of all kinds are looking for alternative, less expensive, raw material and have optimized their processes in order to reduce the amount of waste produced and reduce the corresponding environmental impact. Amin, et al (2011) states that for some time now the ceramic industry has been the target for utilization of other wastes and industrial rejects. Today, it is clear that some wastes are close in their composition to the natural materials required for the traditional ceramic industry.

In addition, some wastes contain materials that are not only compatible but also beneficial in the fabrication of ceramics. Recently, wastes have been constructively applied to resources in recycling research, and different authors have investigated the incorporation of different types of wastes and quarry residues in the manufacture of traditional ceramic materials.

Traditional ceramics, such as bricks, or roof and floor tiles, generally feature high heterogeneity due to the wide range of the composition of natural clays used as raw materials in their manufacture. There is therefore a high tolerance for incorporating large amounts of suitable wastes as raw materials (Simpson, et al, 1998). This fact attracts further importance since ceramic industry, which is classified as heavy industry, consumes huge amounts of diminishing mineral resources. Recycling of wastes has unambiguously beneficial environmental and economic impacts, while high temperature firing can turn wastes, which contain hazardous components, into inert and safe healthy products.

The rice industry produces large amounts of rice husk wastes worldwide, which are expected to increase owing to the fact that the world production of rice has been increasing annually world-wide in the recent years. Evidently, deposition of huge amounts of rice husk wastes and open burning are generating environmental concerns.

The giant amount of waste rice husk generated is still far from being used in its totality as a product or by-product, making technological alternatives needed in order to reduce its possible environmental impact; where, the major potential impacts of its disposal on terrestrial ecosystems include: leaching of potentially toxic substances into soils and groundwater; reductions in plant establishment and growth due primarily to adverse chemical characteristics of the waste; changes in the elemental composition of vegetation growing on the waste; and increased mobility and accumulation of potentially toxic elements throughout the food chain.

The presence of silica in rice husk ash (RHA) generated from the burning of rice husk ash according to Prasda, et al (2001) has been known since 1938. The ash is largely composed of 87 – 97% silica with small amount of alkalies and other trace elements. The use of RHA as a replacement of silica in the field of ceramics has been studied by several authors. Prasda, et al (2001) investigated the effect of RHA in traditional whiteware compositions by completely replacing the quartz phase and the results were that there was no substantial modification to the physical and mechanical properties of the products.

In the light of waste utilization, waste presents a potential source of profit by incorporating RHA as a raw material in ceramic tiles formulations, particularly with regards to economic and environmental aspects. Maximum use of rice husk ash in ceramic tile production would reduce both environmental impact and the cost of production. Waste utilization often results in substantial savings through reduced purchasing costs and efficient practices. It can also reduce waste disposal cost.

In addition, waste utilization has environmental benefits, including reduced energy consumption and pollution, conservation of natural resources and extension of valuable landfill capacity. Practicing waste prevention can also improve customer relations by demonstrating a company's concern about the environment.

1.4 Scope and Delimitation of the Study

Based on the preliminary tests conducted on the sampled population with regards to the available raw materials, the study was delimited to the utilization of kaolin (Kankara Clay), quartz, calcium carbonate, rice husk ash and ball clay (Bomo Clay) to formulate a whiteware body for the production of wall tiles.

1.5 Limitation of the Study

Material characterization through analysis of characteristic decomposition patterns, studies of degradation mechanisms and reaction kinetics, determination of organic content and determination of inorganic content in a sample is useful for corroborating predicted material structures. Sensitive equipment for the Thermogravimetric (TG) and Differential Thermal Analysis (DTA) is available outside Nigeria. Access this equipment is limited to the availability of funds. The quality of the tiles would therefore be dependent on the property tests like water absorption and mechanical strength.

1.6Significance of the Study

The production of tiles, which comprises a substantial portion of the whiteware manufacturing, is exemplary of the improvements which have been realized throughout the industry as a result of research and development endeavours.Mishulovichand Evanko, (2003) for example have conducted pilot scale experiments and successful runs on the actual mass production with industrial base equipment that indicated that tile bodies produced from high-

carbon fly ash are superior to those required for wall tile applications and comparable to those required for floor tiles.

The uses of rice husk ash (RHA) as silica replacement in the ceramic field have also been studied at the laboratory level by several authors. Prasad, et al (2000) investigated the effect of RHA in traditional whiteware compositions by completely replacing the quartz phase with RHA silica without substantial modification to the physical and chemical properties of the products. Amin, Youssef, El–Mahllawy and, El–Abd (2011) have reported the use of Attaqa quarry waste in the manufacture of single fast firing ceramic wall tiles while,Okunna (1983) reported the use of RHA for the production of ceramic glazes.

The evaluation of the possible uses of this waste is not only important from a scientific point of view. In fact Bondioli, Barbieri, Ferrairi and Manfredini (2009) reaffirm that considerable efforts are being taken worldwide to minimize the cost of raw materials that are very incidental on the total price of tiles. Waste should therefore be looked at as a resource that can be extracted, useable and the recyclable.

Waste prevention according to the Environmental Protection Agency, U.S.A. 2005 often results in substantial savings through reduced purchasing costs and more efficient practices. Garkida (2002) also asserts that waste prevention has economic benefits and reduces pollution. Practicing waste prevention can as well conserve natural resources and reduce energy consumption as well as improving customer relations by demonstrating a company's concern about the environment.

With the current stringent legislations regarding the environment and the market demand for environmentally friendly products, manufacturers are concerned with developmental studies aimed at reducing the environmental impacts. This is often done through lowering the amount of residue generated or treating those that are inevitably generated during the production processes.

The high costs associated with raw material extraction, as well as the damage that the extraction causes to the environment are also important reasons to motivate the use of industrial process wastes. Depletion of reliable, trustable raw material reserves and the conservation of non-renewable sources are also contributing factors to the reuse of waste materials. This corroborates with the view of Ahuwan, (1999) that the contemporary approach to ceramic production starts as experiments to test locally available materials and their suitability for tableware production. The use of the waste generated by the rice paddy therefore fits into this scenario.

In developed countries, where the mills are large, disposal of the husk is a big problem. Burning the heapsin the open is not acceptable on environmental grounds and so the majority of the husk is currently going into landfills. The cost of this landfilling according to the Rice Husk Market Study of 2003 erodes the profit of the milling company and this has led to many research programmes into the potential uses of boththe husk and the ash.

Considering the size of the tile industry, a considerable fraction of the RHA produced globally can be utilized to prepare ceramic tiles. Asraw materials and energy contribute to the major costs in running a tile plant, replacement of quartz with RHA would be attractive to the tile manufactures. Such utilization is also environmentally attractive and the nation will benefit from such an undertaking.

CHAPTER TWO

LITERATURE REVIEW

2.1 Ceramic Tiles and their Historical Background.

Tiles were one of the earliest “technological” materials that have been made by mankind. From the beginning, they had artistic, decorative as well as hygienic functions. Among the oldest examples of glazed decorated products were the tiles that covered the Ishtar gate of Babylonia’s King Nebuchadnezzar II (575 B.C), that has now been rebuilt in the Pergamon in Berlin.

The development of clay tiles began with the introduction of roofing tiles, followed by flooring and wall tiles,that can be traced as far back as the fourth millennium BC (4000 BC) when tiles were used to decorate various houses in Egypt (Designboom, 2010). In those days, clay bricks were dried beneath the sun or baked and the first glazes were blue in colour.

Ceramic tiles thathad decorations which were white and blue stripped and later possessed more varied patterns and colourswere also found in Mesopotamia as early as 4,000 BC. In China, the centre of ceramic art, a fine white stoneware tiles with the earliest Chinese glaze was produced during the Shang – Yin Dynasty (1523-1028).

Tile decoration has been improved upon as were methods of tile manufacture over the centuries. During the Islamic period for example, all methods of tile decoration were brought to perfection in Persia. Throughout the world, ceramic tile production and decoration have reached great heightsin various countries and cities. The development of the tile mosaics of Spain and Portugal, the floor tiles of Renaissance Italy, the fiancés of Antwerp, the tile iconography in the Netherlands, the ceramic tiles of Germany and the Far East (China) are all prominent landmarks in the history of ceramic tiles.

The tiles were handmade in the early days and this made it a labour intensive business. Each tile was hand formed and hand painted, thus each tile was a work of art on its own right. The technique of tile and its secrets of trade were safely guarded and orally handed from father to sonand master to student: thus rarely have designs, patterns and details of technique been documentedand few complete treatises exist on the art of tile work in the past. Each tile was hand-formed and hand-painted, thus each was a work of art in its own right (Kristen, 2012)

The Industrial Revolution forever changed the fabricationtechnologyanddecorativepotentialofceramictiles. The industry have beengradually improvedandperfected. Manufacturing of ceramic tile on a large scale throughout the world

today is not hand-made or hand painted for the most part. Except in some few cases, automated manufacturing techniques are used and the human hand does not enter into the production until it is time to install the tile.

The reasons for this considerable diffusion ofceramicfloorandwalltilecanbe found ontheonehand,inthewiderangeofcolours, decorative possibilities and shapes which allowthedesignerandarchitectavarietyof design solutionsdistinctlysuperiortothose possiblewithanyothermaterial,andonthe other hand, the superior technical characteristics of ceramic tile (Clark, 2002).

Technical tests carried out in various laboratories according to Kristen, (2012) confirm that ceramic tile generallyhave a higher resistancetochemicalattackthanmarble andgranite. Theyarenoteasily scratchedlikewood, linoleum and carpet,andneitherburnnoraredamagedby direct contact with fire orincandescent bodies, such as cigarettes. In addition, ceramictilesareeasytoclean,arenoteasily punctured and have a hardness and resistancetoabrasionwhich,ingeneral,are not found in othermaterials like polymeric tiles. In fact they are used in an almost infinite number of ways by both the rich and the poor. Most modern houses throughout the world use ceramic tiles for their bathrooms and kitchens and in every vital area of the premises. Ceramic tiles are also the choice of industries, where walls and floors must resist chemicals.

The technical dynamism of the tile industry has been a major factor in promoting the on-going evolution of processes and materials that has resulted in shift from traditional double firing to fast double and single firing. This success has also culminated in an increasingly large sized tiles having high aesthetic and decorative content.

We are now living in an era where high quality and volume of tiles produced allow the tile user to appreciate tiles for their natural beauty, their decorative possibilities and

functional utility. With modern technology providing an ever increasing variety of tile types and styles, more and more people are discovering the artistic tradition of ceramic tiles.

2.2 Classification of Ceramic Tiles

There are many types of ceramic tiles available on the market and they differ according to the condition of the surface, colour of the body (white or red), manufacturing technology, raw materials and end use. The differences between the “red” and “white” tiles for example depend on the amount of iron minerals contained in the body. By reacting with other body components, they can give more or less colouration and modify the behaviour of the body during firing.

Many classifications have been proposed to define tilesbased on either the characteristic of the material used for production or the firing cycle. It is therefore very difficult to have a complete and exhaustive classification due to the extreme heterogeneity of tile products, their processing, and subsequent characteristics (Manfredini and Pellacni, 1999). In this study, the European (EN) and the American Society for Testing andMaterials (ASTM) standards are considered in view of the fact that they are based on the current international norms (ISO13006), such as the level of water absorption and shaping method which are significant factors in defining the performance specifications of the different products.

The European (EN) standards classify ceramic tiles as a function exclusively of water absorption (which directly correlates to the porosity) and shaping method (extrusion or pressing) as shown in Table 1. The shaping methodsare classified as:

- Shaping process A - (extruded floor tiles). This process includes split tiles and individually extruded tiles.
- Shaping process B - (dry-pressed floor and wall tiles)
- shaping process C - (cast floor and wall tiles)

The water absorption, E, is expressed as a mass fraction given as a percentageand classified as follows:

- Group I (low water absorption) where $E \leq 3\%$
- Group II (medium water absorption) where $3\% < E \leq 6\%$ (Group IIa) or where $6\% < E \leq 10\%$ (group IIb)
- Group III (high water absorption) where $E > 10\%$

Table 1: Classification of Ceramic Tiles in Compliance with EN 87.

SHAPING METHOD	**WATER ABSORPTION, E (%)**			
	(Group I) $E \leq 3$	(Group IIa) $3 < E \leq 6$	(Group IIb) $6 < E \leq 10$	(Group III) $E > 10$
Extruded tiles (A)	A I	AIIa	AIIb	AIII
Pressed tiles (B)	BI	BIIa	BIIb	BIII
Cast tiles (C)	CI	CIIa	CIIb	CIII

Source: Tile Whiteware- Manfredini and Pellacni, (1999).

These parameters indicated above are directly related to the physical, chemical, mechanical, and micro-structural properties such as modulus of rupture, chemical resistance and linear coefficient of thermal expansion and frost resistanceof fired tiles (Manfredini and

Pellacni, 1999).In addition to defining and classifying tile whiteware as well as outlining sampling procedures, the EN standard provides thirteen additional specifications concerning the testing method for the different products and eight concerning the requirement of the products as well as the reference limits for the different properties.

Drews (1989) however, emphasizes that the various characteristics without any exception must be measured according to the specified testing methods so that a correct comparison can be made with the requirements established in the respective product standard. These standards are found in Table 2.

Table 2: European (EN) Standards for Ceramic Tiles

Standard Type	**Standard No.**	**Description/ Classification**
General	EN 87	Definition, classification.
	EN 163	Sampling.
Testing Methods	EN 98	Format, appearance
	EN 99	Water absorption
	EN 100	Bending strength
	EN101	Mohs hardness
	EN 102	Wear resistance of unglazed tiles
	EN 103	Linear thermal expansion
	EN 104	Thermal shock resistance
	EN 105	Crazing resistance
	EN 106	Chemical Resistance of unglazed tiles
	EN 122	Chemical Resistance of glazed tiles
	EN 154	Resistance to Abrasion
	EN 155	Moisture Expansion
	EN 202	Frost Expansion
Product Requirements	EN 121	Group AI
	EN 159	Group BIII
	EN 176	Group BI
	EN 177	Group BIIa
	EN 178	Group BIIb
	EN 186	Group AIIa

	EN 187 EN 188	Group AIIb Group AIII

Source: Tile Whiteware- Manfredini and Pellacni, (1999).

The tile properties measured according to the existing ASTM methods are also provided in Table 3.

Table 3: Properties of Ceramic Tiles Measured according to ASTM Methods

Property	**Type of Tile**			
	Flat Glazed Wall Tiles	Flat Ceramic Mosaic Tiles	Flat Quarry Tiles	Flat Paver Tiles
Non-Destructive Tests				
Thickness	x	xxx		
Facial Dimension	x	xxx		
Spacers	x			
Warpagex	xxx			
Wedging	x	xxx		
Colour Uniformity	x			
Electrical Properties		x		

Destructive Tests			
Water Absorption	x	xxx	
Crazing	x	xxx	
Thermal Shock	x	xxx	
Bond Strength	x	xxx	
Breaking Strength	x	xxx	
Abrasive Hardness	x		xx

Source: Tile Whiteware- Manfredini and Pellacni, (1999).

2.3 Technical Properties

Technical properties are the properties that tiles must possess in order to adequately and dependably perform their function as floor or wall covering. They are therefore critically important properties, on which the "functionality" and "durability" of the tiled surface depend. It is equally important as opined by Drews (1989) for the users to be familiar with these properties, and to keep them in mind for the purposes of identification and selection of tiles that meet one's needs.

The most important technical properties that distinguish the various types of ceramic tiles, and the individual products within a single type, are grouped into several categories:

- regularity
- structural

- mechanical/mass
- mechanical/surface
- thermo hygrometric
- chemical
- Safety

2.3.1 Property of Regularities

Theproperty of regularitiesdetermine the suitability of a batch of tiles for the creation of a “regular” tiled surface, one that is free of “irregularities” like troughs or bulges, steps between adjacent tiles and irregular course of joints. Ceramic tiles are not like other ceramic products which are used individually. A ceramic tile is considered a “module”, whose regular repetition on a surface generates a tiled surface containing tens or thousands of tiles laid out next to each other. In order for the tiled surface to be visually pleasing, the tiles must have specific size and appearance properties. The tiles of a single lot may contain small differences of size and appearance, or small deviations from flatness. Therefore, these differences and deviations Carlos, (2008) states must be controlled to ensure that they do not compromise the regularity of the tiled surface to be constructed.

The degree to which the various types of tiles can fulfil the demand for size regularity must be associated to the EN ISO classification parameter that is related to the shaping method and water absorption levels.

The appearance of the tiled surface is usually affected by this property of regularity. Extruded tiles like terracotta and clinker generally give a floor or wall a more “rustic” look, while pressed tiles yield a smoother and a more uniform surface. Shaping by extrusion according to Drews (1983) usually allows for a less accurate control of size and of surface finishing, as compared to pressure shaping. Drews(1983) however, state that the differences

presented here does not constitute a value judgment in regard to quality, but only clarify a distinction regarding production techniques. The “rustic look” of some extruded products (like certain terracotta tiles) for example is not considered a defect, but is to the contrary a highly desirable effect for many environments. As regards the role of water absorption, the compactness of the structure created in tiles with vitreous support (Groups AI and BI) is a result achieved with special raw materials and by firing at relatively high temperatures.

The firing process in these products entails the substantial formation of a liquid phase, which then leads to an equally ample and compact vitreous phase during cooling (Porcelanosa, 2010). The formation of liquid phases during firing and size shrinkage entails some risks like deformations, irregularity in the sizes of tiles produced at different times or in different areas (hence at different temperatures) of the kiln. There is therefore the riskof having several “production sizes” for the same product. These risks Porcelanosa(2010) states, are typical of vitreous products (red stoneware tiles, porcelain, clinker tile, single-fired tile with low water absorption) that tend to diminish gradually as porosity increases and eventually to disappear for those highly porous products like majolica, cottoforte, and monoporosa tile whose raw material and lower firing temperatures allow for a negligible liquid phase during firing and hence a very low degree of shrinkage.

2.3.2 Structural Properties

The structural properties describespecifically the “structure” of the tile in terms of porous structure. The most important of these properties is water absorption which constitutes a measure of porosity as well as a classification property that several other important properties are dependent on. Porcelaineous stoneware tilesfor example, are product that with low levels of water absorption. Porcelain stoneware products available on the market possess water absorption levels of under 0.5% and in most cases, water absorption levels are under

0.1%. Whitish and red single-fired tiles, clinker tiles and red stoneware tiles are also products with very low levels of water absorption. Products like monoporosa, majolica and cottoforte tiles are identified by the high levels of their water absorption.

In practical terms, a tile's water absorption level significance lies in the fact that many of the tile's properties are critically determined by it, since water absorption is an indicator of a material's microstructure. It is for this reason that water absorption has been selected as a classification parameter for tiles (Byrne, 2008).

2.3.3 Massive Mechanical Properties

These properties concern resistance to loads (for instance the weight of people and of furniture on a floor) to which the tiled surface will be subjected, and which it must withstand without breaking. These properties are called "massive" because they concern the tile in its entirety and also distinguish them from the "surface" properties that concern only the working surface of the tile. They are significant especially in the case of floors tiles.

The mechanical properties of the tiles are measured in terms modulus of rupture and breaking strength. The modulus of rupture is a property of the material that constitutes the tile, and is generally inversely proportional to the level of water absorption. For example, it is much greater in porcelain stoneware, whose water absorption level is below 0.5%, than it is in monoporosa tile, whose water absorption level exceeds 10%. The breaking strength (from bending) is a property of the tile based on its own structure and size. It is therefore not only a function of its water absorption level but also of its thickness. The thicker the tile, the greater it's breaking strength.

2.3.4 Surface Mechanical Properties

This property pertains to the working surface of the tile. They concern the resistance to scratching, trampling, and deterioration from the movement of hard bodies/materials across the surface. This property too is especially significant for floors on which one walks, drags chairs, furniture, carts, and the like. The most important surface mechanical property is abrasion resistance, which measures a tile's tendency to wear (in the case of unglazed tiles) or to alter its physical appearance (in the case of glazed tiles) as a result of the aforementioned actions and conditions.

As regards this property, the observations and considerations pertaining to glazed tiles are quite different from those pertaining to unglazed tiles. For unglazed tiles, abrasion resistance tends to increase as water absorption decrease, reflecting increase in the structural compactness. Tiles with the best surface mechanical properties performance areporcelain, red stoneware and clinker tile which have low levels of water absorption.

Porcelanosa (2010) however, states that the method for measuring abrasion resistance for unglazed tiles does not consider the question of visual effects. The mechanical surface properties depend not only on the type of product but also, to a significant extent, on any specific surface treatment to which tiles may have been subjected either during production or after installation.

2.3.5 Thermo-Hygrometric Properties

Thermo-hygrometric properties are the properties of resistance to specific temperature ("thermo") and humidity ("hygrometric") conditions and include thermal shock and frost resistanceand in the case of glazed tiles, crazing resistance. Abrupt shifts in temperature for instance which occurs when a hot pan is placed on a tiled kitchen counter and exposure to frost which occurs with floors and wall coverings in cold climate must not damage tiles at all. Crazing is the fine cracks that may appear in a glaze due to some manufacturing or

environmental conditions. These properties also include thermal expansion and moisture expansion, which measure how much a tile may expand if exposed to higher levels of temperature and humidity.

Frost resistance is substantially influenced by the open porosity measured by water absorption. On the basis of the frost mechanism, the tiles with least water absorption, like porcelain and other equally compact products of other types, are certainly the most reliable in terms of frost resistance. Since water cannot penetrate inside these materials, the process that leads to frost damage cannot even begin. It must be noted, though, extruded products such as various terracotta tiles that are neither glazed nor treated and are among the most porous products with porosity as high as 10% are often resistant to frost, while pressed products of comparable porosity are rare. This difference in performance is due to the particular distribution of the shape and sizes of the pores, which results from the extrusion method (Porcelanosa, 2010). The resistance to crazing basically depends on the composition of the glaze: some types of glazes, used to achieve various effects, run a higher risk of crazing (Bridges, 2010). For this reason, norms have included resistance to crazing among the standardized properties.

With regards moisture expansion, this generally correlated to water absorption, although Bridges, (2010) states that considerable variations exists presumably due to the effects of other micro structural properties like the type, composition and distribution of the various phases included.

2.3.6 Chemical Properties

These are the properties of resistance of tiles to the chemical aggressiveness or staining action of other substances that may come into contact with thesurface of the tile. Chemical property measurements are stain resistance, resistance to household chemicals, acid

and alkali resistance. The compactness of the surface layer of the tile is extremely important in this regard. The greater its porosity, the greater the specific surface or interface that comes into contact with the chemical agent (Timellini and Palmonari, 2000). A greater porosity therefore promotes the absorption of the substances.Furthermore, the pores, because of their shape, constitute areas of penetration of the substance itself, which can thus become irremovable by even the most aggressive cleaning procedures.

As for glazed products, the glaze layer that covers the working surface makes it compact and non- absorbent. A good practical measure of this compactness Timellini and Palmonari (2000) further states is the tile's resistance level to stains: obviously, a high resistance reflects a non-absorbent surface, while a low resistance level reflects the presence of micro porous veins/channels through which stains and chemical substances may penetrate the interior of the glaze. Glazes also usually possess a high level of chemical resistance, despite the fact that some colourations and decors are quite sensitive to acids and other chemical agents.

In unglazed products, measurement of chemical resistance is generally excellent for all products. Stain and soil resistance is in this case more directly correlated to compactness: hence the best performance can be expected from porcelain stoneware and other types of products with similar compactness. These materials are also subject to a higher firing temperature, which creates not only compactness but a correspondingly greater chemical inertia. Porcelanosa (2010) however opines that though the surface porosity which is important in containing the risk of chemical attack, according to the mechanism described, is not always precisely correlated to the more macroscopic porosity which determines the degree of water absorption. In other words, even those unglazed products which are of extremely low levels of water absorption can still be micro porous on the surface and admit some certain amount of staining substances or absorption of chemical.

In the case of unglazed products of greater porosity, like terracotta, the initial disadvantage of the porosity and the surface porosity is corrected with the application of the impermeability agents mentioned earlier. Properly treated terracotta floors require greater caution on the user's part than other types of floors, but their chemical resistance and ease of maintenance are more than adequate for many residential environments.

2.3.7 Safety Properties

These properties are especially significant for the safe use of tiled areas with regards to hygiene for the user or against risk of accidents. The main safety property is slip resistance, which is very important for tiles intended for floors in certain exterior public and industrial areas. Safety properties also include lead and cadmium release from some glazes, which is especially monitored in the case of tiles whose usage brings them into contact with substances associated with food.

The most common use of this sort is for kitchen working tops. As regards slipperiness, certain types of ceramic tiles can ensure excellent safety performance. Apart from the smooth-surfaced glazed ceramic tiles with a low-to-medium friction coefficient that are not lower than that of other non-ceramic floor materials, there are many commonly used types of glazed tiles with a rough surface, and unglazed tiles with surface reliefs. They both meet the anti-slip requirements for all residential and industrial environments. Generally, floors for which anti-slip safety requirements are most important are public and industrial environments. These same environments also have the strictest requirements for resistance to surface mechanical stress like abrasion, chemical resistance, and hygiene.

Flooring materials which can meet these special and severe demands are to be found mostly among unglazed tiles with low water absorption levels, like porcelain stoneware. There are in fact various products of this type, made with special surface reliefs that combine

a compliance with the anti-slip requirements with excellent mechanical and chemical resistance.

In conclusion, all the properties cited are considered in the technical standards on tiles. Some properties refer only to tiles for certain specific applications (for instance for floors, or for exterior environments). For example,frost resistance is a relevant property only for tiles to be used for exterior floors or walls (in climates with the risk of frost). It is negligible for tiles for interior areas. Abrasion resistance is a property relevant only to tiles to be used for floors, namely for areas that will be walked on.

2.4 Types of Tiles

Due to the diverse nature of ceramic tiles in terms of its decorative qualities, vast usage, and a lot more cost-efficient than other forms of tilling mediums like marble and stone, the following types of tiles and descriptions take into account the purpose for their usage, technical information, as well as the most commonly used commercial terms.

2.4.1 Majolica Tiles

This is typically an Italian tile but can also be found in other countries with the suitable raw materials. It is manufactured from quarry clay containing not only clay-like and sandy powders but also substantial amounts of carbonates and ferrous oxides. Majolica tiles are always glazed with a non-transparent glaze, covering the rusty colour of the body (“bisque”). This product is typically used for interior wall coverings, and the most common sizes are 15x15cm, 15x20cm and 20x20cm. Its physical properties are a good level of mechanical resistance despite its high porosity and an excellent resistance to glaze crazing. The production cycle includes double firing or more recently single firing in rapid-firing kilns. The development of this new technology has contributed to the revival of majolica tile,

which had somewhat declined in the 1980s following the increased popularity of single-fired tile likemonoporosa tiles (Clark, 2002).

2.4.2 Cottoforte Tile

This is also a typical Italian product produced almost exclusively from the Emilia-Romagna region. The tiles are always glazed with a non-transparent glaze and are used mainly for interior floors, though wall tiles of this type also exist as well. This product enjoyed its greatest popularity in the 1960s-70s thanks to the great variety and improvement in the types of decoration and perfected production techniques. Typical sizes are 15x25cm, 20x20cm and 20x30cm. Cottoforte tile is double-fired and their properties include adequate to good mechanical resistance, easy application of glazes and decoration.Due to the high energy costs of this technique, cottoforte has largely been replaced by red single-fired tile. Thanks to the use of specially formulated bodies and recent advances in glazing and silk screening decoration techniques, single-fired products now offer the same technical properties and aesthetic appeal as cottoforte tiles, at a lower production costs.

2.4.3 Earthenware - White Body Tiles

This type of tile now makes up only a modest percentage of tiles production, due to the high costs of the double-firing technique, and of the 'precious' raw materials like white firing clays, sands and fluxes. The distinction between the "soft earthenware" and "hard earthenware" has to do with the types of fluxes used. In the former case, calcium is used while in the lattermagnesium carbonates and feldspar are used. The tiles obtained are white and this allows for direct decoration on the bisque surface and a single transparent glaze. The classical size is 15x15cm and mainly used for interior wall coverings.

2.4.4 Red Single-Fired and Monporosa Tile

The technique used for the production this type of tiles is the simultaneous firing of body and glaze (single firing). It covers a broad range of glazed materials with a great variety of physical properties, due to the diversity of the body's water absorption ranging from slightly above zero to about 15%. All these types of tiles are produced from clays containing iron oxides. Feldspar-based fluxes are generally used to obtain vitrified materials, while clays containing carbonates are used for porous materials. Depending on the proportions of the various component materials used, Clark (2002)states that the tiles can either be vitrified with low water absorption suitable for exterior floors, or more a porous material that is suitable for interior floors or interior wall covering. Available sizes range from the 10 x 20 cm and 20 x 20cm to as large as 40 x 40 cm.

2.4.5 Whitish Single Fired and Monoporosa Tile

This type of product is distinguished from red single-fired tile by the colour of the body that is white in colour due to the use of clay without iron. Other materials used for the body composition include siliceous sands of considerable purity and feldspars. Most of the products in this category are floor tiles, used for both exterior and interiors. The larger sizes (30 x 30 cm, 40 x 40 cm) are more common than the traditional 20 x 20 cm. Presently, there has been an increased production of the porous tiles with water absorption of above 10% that are used for interior wall covering.

2.4.6 Terracotta Tiles ("Cotto")

Terra-cotta tiles are either handmade or machine made for pavers. The machine made terra-cotta tiles are much denser than the handmade tiles and can usually be installed outdoors as they are frost resistant. This tile is also known as "Rustic terracotta" "Tuscan terracotta" or

“Florentine terracotta”. It usually comes in larger sizes such as 25 x 25 cm, 30 x 30 cm, 20 x 40 cm and 40 x 60cm. Unlike cottoforte or red single-fired tile, terracotta tiles are usually not glazed although recently some partially or totally glazed terracotta tiles have been introduced. The colours range from terra-cotta to yellow to brown. Where additional colours are desired,ceramic stains are used.

Unglazed terracotta tiles used forinterior floors are generally treated with special substances to enhance their colour and cleanability, The exterior terracotta floorsare not treated in order to avoid the risk of frost damages.

This product is traceable to ancient times and has been updated for modern use thanks to its popularity among architects and designers for its warm and characteristic red colour. Technically speaking, terracotta is halfway between brick, which in the past was the most common flooring for non-luxury type housing, and the modern cottoforte and red single-fired tiles (Benningtontiles, 2009).

2.4.7 Red Tile

This is also a typically Italian product. It is an unglazed vitrified product and the most common sizes are 7.5 x 15 cm and 10 x 20cm. Red stoneware tiles are suitable for interior and exterior residential floors, industrial environments and areas of heavy traffic.The wider range of uses is made possible by the product’s unique physical properties like good frost resistance and excellent resistance to both breakage and abrasion. Like other tile products that are either partially or totally vitrified during firingBenningtontiles (2009) states that the red stoneware tile is quite sensitive to the variations of temperature in industrial kilns.

The terms “stoneware” or “red stoneware” are sometimes used for types of tiles that are not vitrified and therefore do not have the properties detailed above. Red stoneware tile can be distinguished by its water absorption level, which is below 3 or 4%.

2.4.8 Porcelain Tiles

Porcelain tilesare made from a blend of fine-grain clays and other minerals to produce a very dense body that makes it highly resistant to moisture, staining and wear. Because of these features, porcelain tile is able to withstand years of heavy foot traffic in both interior and exterior applications, while maintaining its colour and beauty. They come in a wide range of colours which are achieved by mixing pigments with the clay or glazes. The special clays allow the product to be fired at extreme temperatures of 1350^{O} C resulting in a denser and harder body than most other tiles.

The product is almost completely vitrified and this makes it completely impervious and with excellent mechanical properties. The traditional 5 x 10cm and 10 x 10cm sizes have been disappearing, and larger sizes (20 x 20 cm, 30 x 30cm and 40 x 40cm) have grown increasingly popular. Usually unglazed, porcelain stoneware is used for floors of all kinds but recently, large slabs (100 x 60cm) have been introduced to be used for exterior wall covering.

Porcelain stoneware originated as a product for technical use, such as for floors in heavily trafficked public and industrial areas. Research and development efforts have however, gradually produced new products that combine porcelain's unique technical strengths with ever greater aesthetic properties, for areas that have design requirements like malls, offices, hotels and restaurants. The body is often coloured by introducing colouring oxides into the body mix which blend extremely well, with the matrix during firing. This type of tile , obtained by mixing raw materials of various colouration to obtain a surface and support with a granular look, is sold under various denominations such as "ceramic granite" or "ceramic porphyry" (Benningtotiles, 2009).

Porcelain stoneware tiles with a polished, glossy surface are used for interior and exterior wall covering. Besides the glazed products described above, other variations have

also been emerging that feature various types of surface treatments. For example, application of penetrating salts, or glazes, which creates a sophisticated look, is in increasing demand. Porcelain stoneware with surface decors is also growing in popularity.

2.4.9Quarry Tile

Quarry tiles are unglazed tiles made from natural clays. Their colours are usually limited to earth tones, ranging from red to light tan, although some manufacturers add oxides to the clay to offer a wider range of colours. Their body is both thick and dense, making them a popular choice for both heavy commercial and residential installations. Their surfaces generally have good slip resistant qualities.

2.4.10 Mosaic Tiles

Mosaic tiles are small tiles, less than 6 inches square in size. They are usually very dense products that are tough and stain resistant. Mosaics are ideal for shower floors because their small size gives them the flexibility to follow the contour of the floor as it slopes to the drain. The toughness and stain resistance of this type make them ideal for countertops.

2.4.11 Wall Tiles

Wall tiles are thin tiles with a porous body and a white or coloured glossy or matte glaze. They are produced in a wide variety of shapes and sizes are available but the majority of the tiles are square or rectangular.

Wall tiles are used in kitchens, bathrooms, washrooms and swimming pools as well as in decorative work such as table top and fireplaces. Wall tiles are not used in places that are exposed to frost attack or severe abrasion. Their aesthetic appeal is the

most important feature of wall tile products used for interior decoration. As far as the technical characteristics are concerned Clark, (2002) states that **tiles for wall decoration should match the following criteria:**

- **Excellent dimensional stability in the manufacturing process, usually obtained using body compositions with very low firing shrinkage (less than 0, 5%). All wall tile products must meet these requirements.**
- **Very narrow dimensional range to ensure easy setting and vertical application.**
- **The glaze must be easy to clean with no dust retention because one of the most important requirements in bathroom or kitchen application is hygiene.**

2.4.12 Floor Tiles

These tiles, glazed or unglazed, have the sufficient strength, impact, and abrasion resistance to withstand weight and foot traffic. They are usually thicker, denser, and heavier than wall tiles. If the substrate is strong enough to support the weight, floor tiles may also be used on walls and counter tops. Floor tiles range in size from less than one square inch with mosaics up to 24" x 24". Although some of the most popular sizes for floor tile are 13" x 13" and 12" x 12", there is a growing trend towards large tiles.

2.5 TILE RAW MATERIALS

A variety of raw materials are used in the production of tiles. These raw materials or minerals can generally be broken down into three categories, which are clays, fluxes and “fillers” (or non-plastics). Each material has a specific function. The clay is the component that provides plasticity or cohesion for forming, while the flux promotes vitrification. The

filler is typically inert, but it also helps to modify the glass viscosity during vitrification, which effectively extends the range of firing temperatures of the composition.

2.5.1 Ball Clay.

Clay is the component that receives the most attention in traditional ceramic compositions. Ball clays are characterized by moderate kaolinite content, some organic material such as lignite, silica (SiO_2) in the form of quartz (sand) in varying degrees of fineness, illite, montmorillonite ($(Al, Mg)Si_4O_{10}(OH)_2$,) and other minerals such as mica.

As a group, Velde, 1992 states that ball clays tend to be fine grained. This characteristic, together with the organic and mica content, makes these clays plastic and workable with good strength when dry. Ball clays are always sedimentary clays, that is, they have been formed in one location and transported, usually by water, to another location where they settle, along with sand, vegetation, and other materials. As the deposit ages, the decaying vegetation forms complex organic compounds which, when dispersed in the clay, contribute significantly to the properties of the clay. A variety of beneficiation processing and refining steps are available to remove the impurities. The organic impurity however, plays a role in the plasticity of the body, but must be controlled for some fast firing applications (Worral, 1986). Particle size distribution of ball clays is also a critical characteristic that affect its behaviour in a ceramic body.

Ball clays, depending on their mineral composition, burn from a cream to brown or greycolour and therefore can be used only to a limited extent in bodies where whiteness and translucency are critical (Singer and Singer,1984). Many ball clays can be used as mined. Producers today however, are offering blends and combinations of clay (either in dry or slurry form) which have more stable and predictable properties in addition to convenience in handling when in the slurry state. Table 4 is a summary of some clays and kaolin found in Nigeria.

Table4: Summary of Some Clay and Kaolin Occurrences in Nigeria

LOCATION	STATE	QUALITY	QUANTITY(MILLION OF TONS)
Ozubulu	Anambra	Kaolin	4.2
Enugu	Anambra	Clay	19.0
Enugu	Anambra	Fire Clay	110
Nus	Imo	Kaolin	0.015-0.1
Umuahia	Imo	Kaolin	Not Determined
Ibere	Imo	Kalolin	2.0
Ogbuebelu	Imo	Kaolin	Not Determined
Giri	Sokoto	Kaolin	2.0
Illo	Sokoto	Kaolin	Large Deposit
MarabaRido	Kaduna	Brick Clay	5.250
FarinKasa	Kaduna	Brick Clay	Not Large
BakinKusa	Kaduna	Brick Clay	Not Large
Kankara	Katsina	Kaolin	3.400
Kachia	Kaduna	Kaolin	Large
IkotEkpene	AkwaIbom	Ball Clay	Small
Obom	AkwaIbom	Ball Clay	Small
Mbiafor	AkwaIbom	Ball Clay	Small
Itu	AkwaIbom	Kaolin	Large
Obomkpa	Delta	Ball Clay	Not Determined
Ukwu-Nzu	Delta	Brick Clay	Not Determined
Ibusa	Delta	Kaolin	3.0
Ossiomo	Edo	Kaolin	0.5
Okpella	Edo	Kaolin	3.0
Igoriakhi North	Edo	Secondary Clay	4.0
OkeEri	Ogun	Kaolin	2.0
Imope	Ogun	Kaolinitic	1.0
Ilefun Hill Meko	Ogun	Kaolinitic	Large Reserve
Onibode	Ogun	Kaolinitic	0.81
DawakinTofa	Kano	Brick Clay	Not Determined
Tsanyawa	Kano	Kaolinitic	Not Determined
Nafuta	Plateau	Kaolinitic	6.0
Worrom	Plateau	Kaolinitic	6.0
Major Porter	Plateau	Kaolinitic	3.0
SabonGida	Plateau	Kaolinitic	3.0
Okitipupa	Ondo	Swamp Clay	0.075
Shagbon	Ondo	Kaolin	0.45
Alkakeri	Bauchi	Kaolin	Not Determined

Source: Guide to the Non-Metallic Mineral Industrial Potentials of Nigeria, Raw Materials Research and Development Council, Nigeria (1992).

2.5.2 Kaolin.

Kaolin also known as China clay is made up primarily of the minerals kaolinite and mica, and occasionally significant amounts of halloysite with little if any organic content. Kaolins (Al_2 (Si_2O_5) $(OH)_4$) are classified as primary clays, that is, they are found in the location where they are formed(Worral, 1986).

Unlike ball clays, most commercial deposits of kaolin have not acquired significant amounts of other minerals (other than silica) or organic matter during their formation: occasionally small amounts of mica and montmorillonite$(Al, Mg)Si_4O_{10}(OH)_2$ are present. As a result they have a white or cream fired colour (Worral, 1986).

Kaolins because of the absence of organic matter and minor contents of "binding" minerals such as montmorillonite, have very little strength when dry, and their contribution to a tile body is more of that of a filler which improves the whiteness of the fired article and serves as the starting point for the desired crystalline makeup of the fired body (Wood, 1990).

Since fired colour is typically a concern with kaolin, the level iron content (Fe_2O_3) and titanium (TiO_2) content are very important. As the iron content increases, the fired colour has more of a red shade, while increasing titanium content imparts a buff or tan colour. For products where extreme whiteness is desired, the clay selected must have minimum level of these impurities.

The Kankara clay used for this study is of a good quality and compare favourably with the foreign kaolin in terms of its quality. This comfirms the assertion by Sullayman (2006) that Kankara clay is a high quality kaolin that appears to be more superior to that of the worlds famous China clay in Cornwall, Britain. See Appendix 2.

2.5.3 Non-plastic Materials

The non-plastic or filler portion of the whiteware composition usually consists of flint (ground quartz)(SiO_2), but it may include alumina (Al_2O_3) or pyrophillite ($Al_2Si_4O_{10}(OH)_2$)depending on desired function of the filler. Much of the silica in a ceramic body is supplied by the clays and fluxing materials, but in most instances the formula for the body will call for direct quartz additions (Wood, 1991). Occasionally alumina is added to the body either partially or totally replacesquartz, when properties such as thermal expansion and strength must be enhanced. The functions of quartz in the body acts asa filleras well as a modifier of fired-body properties such as thermal expansion.

It also helps to control the firing warpage of the body by forming a relatively non-fusible network of grains, counteracting to some extent the effect of feldspar during sitering (Worral, 1986).

2.5.4 Fluxing Materials

Fluxes promote the formation of a glassy bond during vitrification. Fluxes provide alkaline (Na_2O and K_2O) or alkaline earth (CaO or MgO) to the composition, which promote glass formation and reduce glass viscosity during firing which serves to enhance vitrification. The level of fluxing components Worral, (1986) states must be optimized to achieve the desired fired property in the selected firing range.

There are several types of feldspar that are used commercially. These include soda feldspar (albite, $NaAlSi_3O_8$), potash feldspar (microcline or orthoclase, $KAlSi_3O_8$) and lime feldspar (anorthite, $CaAl_2Si_2O_8$). Other fluxing materials include such materials as nephelinesyenite($K_2Na_6Al_8Si_9O_{34}$) andspondumene$(Li,Al)SiO_{3.}$. Nephelinesyenite is seldom used because it is relatively expensive, but it provides greater fluxing power and a wider

firing range than other forms of feldspar in porcelain compositions. Additionally, nephelinesyenite does not usually have significant quartz contamination, although it may contain trace impurities of mica (Worral, 1986).

2.6 Rice Husk Ash

Rice Husk Ash (RHA) is obtained by burning the rice husk and it is produced in plants where rice milling is performed. Generally, the rice husk ash is dumped as a waste.

Rice is the primary source of food for billions of people around the world and according to the statistical data of Food and Agriculture Organization (FAO, 2003), the world annual paddy production is approximately 600 million tons. In Nigeria, rice is cultivated virtually in all the agro-ecological zones (Figure 1) covering an estimated area of 1.6 million hectares. This, according to the estimates of 2005 reveals an annual yield of 2.3 million tons (Daramola, 2005).

The ricehuskistheshell produced during de-huskingof paddy. 25 mass% of the rice grain is husk and, therefore, about 150 million tons of rice husks residue are produced world-wide annually (RHA Market Study, 2003). The paddy on an average consists of about 72 % of rice, 5 % –8 % of bran, and 20 % – 25 % of husk. It is also estimated that every tonne of paddy produces about 0.20 tonne of husk and every tonne of husk produces about 0.18 tonne to 0.20 tonne of ash.

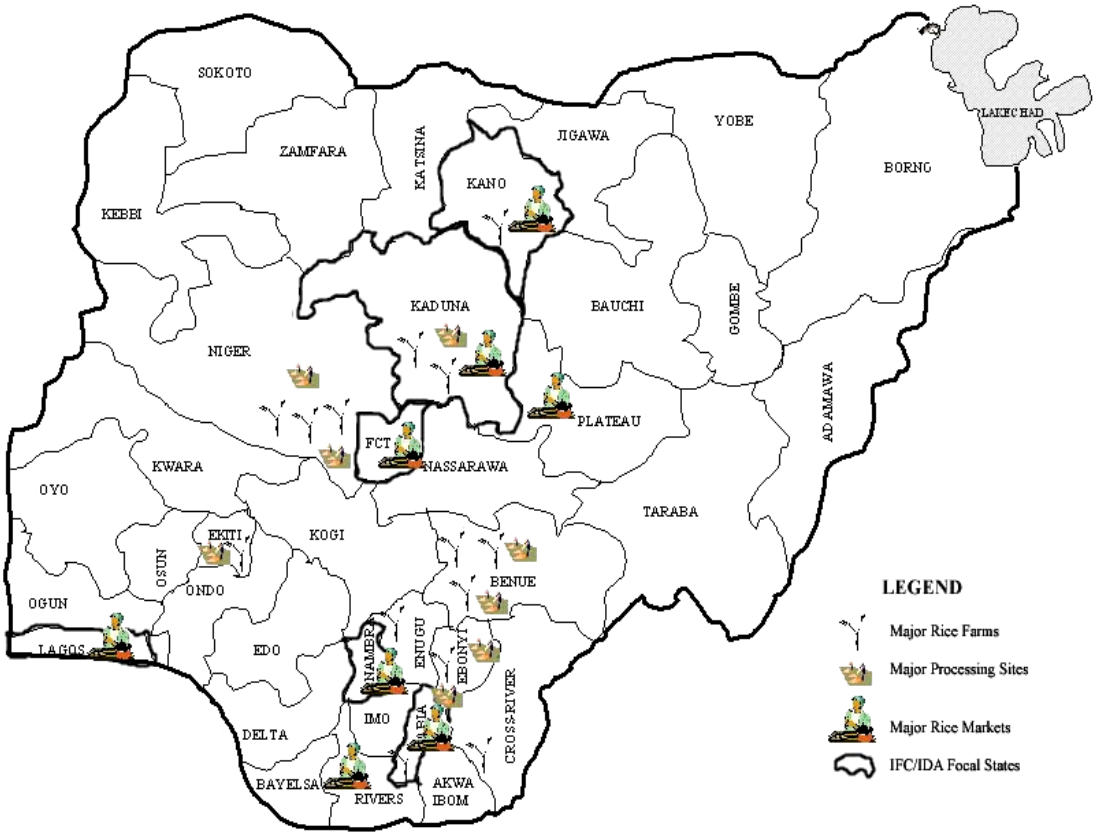


Figure 1: Map of Nigeria showing Rice-growing Area.
Source: Daramola 2005.

The chemical constituents of the RHA from different countries as indicated in Table 5 vary from sample to sample and this according to the RHA Market Study, 2003 may be due to the different geographical conditions type of paddy, climatic variation, soil chemistry and fertilizers used in the paddy growth.

Due to its high silica and lignin content, rice husks are insoluble in water, tough, woody and abrasive in nature with low nutritive properties and resistance to weathering (Muthadhi, Anitha and Kothandaraman, 2007).

Table 5. Comparisons of Chemical Properties of RHA from Various Locations.

Constituents	Malaysia	Brazil	Netherlands	India	Iraq	USA	Canada
	Weight, %						
Silica (SiO_2)	93.10	92.90	86.90	90.70	86.80	94.50	87.20
Alumina (Al_2O_3)	0.21	0.18	0.84	0.40	0.40	Trace	0.15
Iron (Fe_2O_3)	0.21	0.43	0.73	0.40	0.19	Trace	0.16
Calcium (CaO)	0.41	1.03	1.40	0.40	0.40	0.25	0.55
Potassium (K_2O)	2.31	0.72	2.46	2.20	3.84	1.10	3.68
Magnesium (MgO)	1.59	0.35	0.57	0.50	0.37	0.35	0.35
Sodium (Na_2O)	*	0.02	0.11	0.10	1.15	0.78	1.12
Sulphur (SO_3)	*	0.10	*	0.10	1.54	1.13	0.24
Loss on Ignition	2.36	*	5.14	4.48	3.30	*	8.55
Note: * not reported							

Source: Rice Husk – Properties and Its Uses: A Review (Muthadhi, Anitha and Kothandaraman, 2007).

The nature of silica is mainly amorphous and has been termed opaline silica. It is well known that the rice husks have a low calorific value (3585 kcal kg–1) and a high (20–22 mass %) ash content (Rice Husk Market Study, 2003). The ash contains nearly 95 mass % of silica and is an important renewable source of silica.

Burning is a cheap method of extracting the silica from rice husks for possible commercial use, but it brings up the associated problems of uncontrolled particle size and variable impurity levels, mainly in the form of intimately mixed carbon. Due to the growing environmental concern and the need to conserve energy and resources, efforts have been made to burn the husks under controlled conditions and to utilize the residual ash in a variety of end products. The controlled burning of the rice husks in air leads to production of white rice husk ash (WRHA) or 'white ash' containing almost pure about 95% silica in a hydrated amorphous form, similar to silica gel, with high porosity and reactivity (Muthadhi, Anitha and Kothandaraman, 2007).

2.7 Fabrication Technology

The highperformanceofceramic tile according to Wood, (1991)is theresultof extensively studiedfabrication technology, always in evolution, whose key feature is the very high firing temperature which varies, depending on type of product, from 950^{o}C to more than 1200°C. The raw materials forthe productionofceramictilearewidelyavailablenaturalmaterials and thetilebodyisformedfromamixtureofclay(togive plasticity), quartz(the non-plastic fractiontogivestructuralstrength) andcarbonates or feldspars (to provide a fluxing action). The glazes are formed from sand, kaoliniticclay, frit and oxide-based pigments to provide the colour.

Depending on whether the product made is to be glazed or not and whether single fire, twice fire or third fire is involved, the tile will or will not be glazed in a given process, the order of the glazing and firing stages has to be suitably rearranged.

The successive stages of ceramic tile manufacturing process can be summarized as follows:

- Raw materials preparation
- Pressing/Extrusion and drying of the green body
- Firing, with or without glazing
- Additional treatments

2.7.1 Raw Materials Preparation

The tile manufacturing processes starts by selecting the raw materials required for the body composition, which are mainly clays, feldspars, sands, carbonates and kaolins. In the traditional tile industry, the raw materials for the tile body are carefully mixed and ground, in

order to obtain a perfectly homogeneousmaterialforshaping. The preliminary homogenization is required in most cases to ensure consistent characteristics.

2.7.1.1 Dry or Wet Milling

The mixture of body components is usually dry milled with either a hammer or pendulum mill or wet milled in a continuous or batch ball mill. The resulting milled material exhibits different characteristics depending on whether dry or wet milling is used. In dry milling, fragmentation occurs and particle aggregates and agglomerates remain, with larger particle size being larger than 300 microns. Materials that are wet milled exhibit particles sizes that is smaller than 200 microns. According to the Applied Processes and Techniques, 2005a decisive factor in selecting the type of milling to be used is dependent on the capital outlay required in each case.

2.7.1.2 Wet milling and Spray Drying

The wet milling and subsequent spray drying processes are currently the most widely implemented methods in ceramic floor and wall tile manufacture by the single-fire processes because of the important technical improvements they provide. In wet milling, the raw materialsare either wholly or partially fed into the ball mill as it is usually done or they can be directly dispersed. Part of the water contained in the resulting suspension (slip) is removed by spray drying to obtain a product with the required moisture content for each of the processing stage.

In this drying process, the fine drops of sprayed suspension come into contact with hot air to yield a solid with low water content. The moisture content of the body slip usually ranges from 0.0-0.45 kg water/kg dry solid. The spray-drying process reduces the water content to 0.05-0.07 kg water/kg dry solid (Applied Processes and Techniques, 2005).

The spray drying takes place according to the scheme shown in Figure 1:

- Pumping and spraying the slip.
- Hot gas generation and feed.
- Drying by contact of hot gas-slip drops.
- Separation of spray dried powder from the gases.

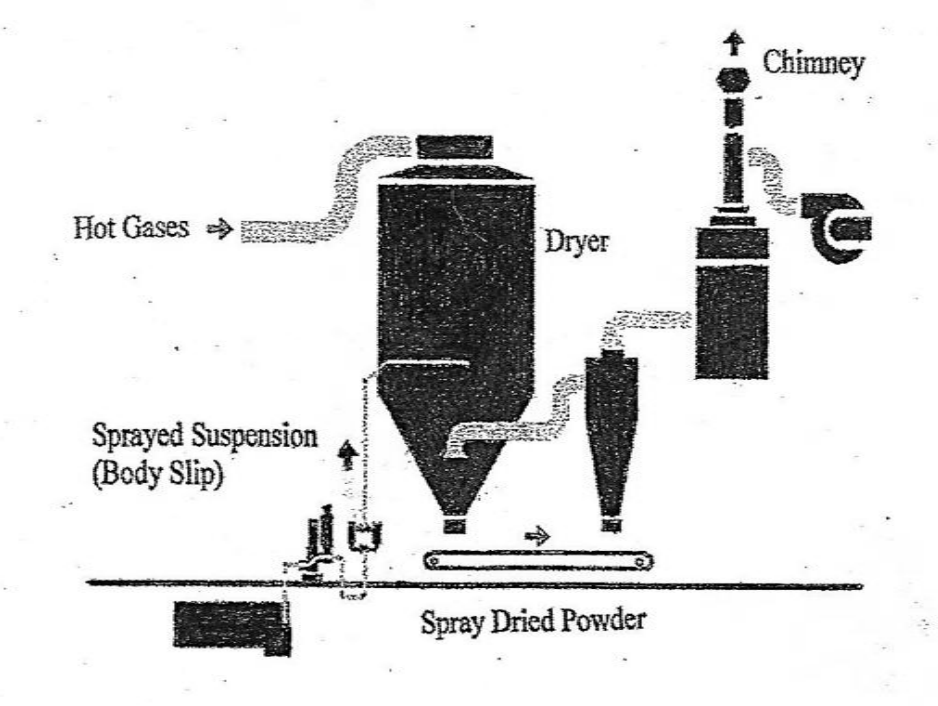


Figure 2: Schematic Illustration of the Spray Drying Processes
Source: Applied Processes and Techniques (2005).

The slip from the milling facility storage tanks, with a 60-70 % solid contents and appropriate viscosity is fed into the spray dryer by reciprocating pumps. The slip is sprayed as a fine mist, which dries on coming into contact with the hot gas stream. The gases come from a conventional air-natural gas burner or are exhaust gases from a cogeneration turbine. Thegranulate, with a moisture content of between 5.5 and 7%, is discharged onto a conveyor belt and conveyed to the silos form the dryer for subsequent pressing.

The use of the spray-drying process to obtain the raw material for the body (spray-dried powder) provides important advantages that favour the development of subsequent manufacturing process stages. One of the most important advantages of the spray-drying process is the production of highly uniform and less spherical hollow granules that provide the spray-dried powder with high flow ability that facilitates press die filling and the pressing of large-size tiles.

Another advantage worth mentioning is that it allows performing two operations, namely drying and granulation, simultaneously with same facilities. Additionally, the continuous nature of the process allows automation. The low side of the spray dry process is the cost of energy for the drying process.However, the energy efficiency can be raised by heat recovery from the gases and electricity generation by installing cogeneration turbines(Applied Processes and Techniques, 2005).

2.7.2 Tile Forming Processes

The processes specifically used for ceramic tile manufacture include dry (dust) pressing, extrusion, and plastic moulding.

2.7.2.1 Dry pressing

The dry pressing process at 5-7% moisture content with hydraulic presses is the most common tile forming method. Forming of the tiles takes place by mechanically compressing the paste in the die and is one of the most cost-efficient forming methods for making ceramic ware with a regular geometry. In pressing, the oil-hydraulic press system drives the rams into the powder bed in the die. Characteristically, the main hydraulic press has a high compaction force, high productivity, easy adjustment and consistency in holding the set pressing cycle

schedule. The pressing facilities have been developed significantly in the last few years with very sophisticated, easily adjustable and highly versatile programmers.

2.7.2.2 Extrusion

Extrusion is a plastic forming method that has been used extensively for the fabrication of ceramics for bricks, insulators, pipe and tiles. The extrusion process consists of forcing a highly viscous, dough-like plastic mixture of ceramic powder plus additives through a shaped die.

Tile forming by the extrusion process basically consists of putting the plastic body through a die that produces a constant tile cross section. The equipment involved is made up of three main parts: a driving system, the die and the cutter. The most common driving system is the auger.

2.7.3 Drying of the Green Ceramic Bodies

After forming, the tile body is dried to reduce the moisture content (0.2-0.5 %) to appropriately low levels for the firing and eventual glazing stages. In the dryers that are commonly used in the ceramic industry, heat is transferred mainly by convection from hot gases to the tile surfaces, and also slightly by radiation from these gases and from the dryer walls to the tile surfaces. The air used for the processmust be sufficiently dry and hot, because it does not only serve to remove the water from the solid but also to provide energy in the form of heat to evaporate the water.

Presently, the bodies are dried either in vertical or horizontal dryers. After shaping, the bodies are placed in the dryer where they face a hot gas countercurrent. The hot gases come from a natural gas burner or from the kiln cooling stack. The main heat transfer mechanism between the air and the bodies is the conventional method. In the vertical dryers,

the pieces are fed into baskets consisting of several decks of rollers and moved upward through the dryer, where they come into contact with the hot gases. The temperature in this type of dryer is normally less than 200ºC and the drying cycles range from 35-50 minutes.

The horizontal dryers are designed like the rollers kilns. The items are fed onto different decks inside the dryer, and conveyed horizontally on the rollers. Burners located on the sides of the kiln produce acounter current of hot drying air. The maximum temperature in these types of facilities is usually higher than in the vertical dryers (around 350ºC) and the drying cycles are shorter, between 15 and 25 minutes.

Overall, the Applied Processes and Techniques, 2005 reveals that horizontal dryers have a lower energy consumption compared to the vertical dryers due to a better arrangement of the items inside the dryer and a lower thermal mass. The resulting emission from the drying stage is a gaseous stream with a temperature of about 110ºC, with a very low concentration of suspended particulates from the tile surfaces being drawn along in the exhaust stream.

2.7.4 Glazing

Glazing involves applying one or more coats of glaze with a total thickness of 75-500 microns onto the surface of the tile by different methods. Glazing is done to provide the fired product with a series of technical and esthetical properties such as impermeability, cleanability, gloss, colour, surface texture, and chemical and mechanical resistance. The nature of the resulting glaze coating is essentially vitreous, although in many cases the glaze structure contains crystalline elements.

2.7.5 Firing

Firing is one of the most important processes in the tile manufacturing stages in view of the fact that most tile characteristics depend on it. These include mechanical strength, dimensional stability, chemical resistance, water absorption and fire resistance. The main variables considered in the firing stage are the firing cycle with regards to temperature/time and kiln atmosphere, which must be related to each composition and manufacturing technology as well as the ceramic product to be made. In the firing operation, the tiles are subjected to a firing cycle during which a series of reactions take place in the piece, generating changes in the microstructure and providing the desired final properties.

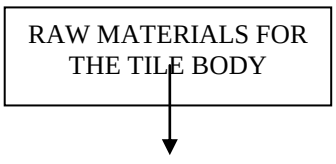
2.7.5.1 Single and Twice Fire

Ceramic tiles can undergo one, two or more firings. The unglazed ceramic tiles are fired once while the glazed tiles can be fired once after applying the glaze to the green tile in the single -firing process.With regards to the body may be fired first, followed by glaze application and subsequent second firing. There may sometimes be an additional drying stage after glazing. This occurs just before the material is placed in the kiln to reduce tile water moisture content to low enough levels for the firing stage to be carried out properly.

2.7.5.2 Fast firing

This is currently the prevailing ceramic tile firing method and is done in single -deck roller kilns. The fast firing method Jones, (1996) states has contributed to reducing firing schedules to less than 40 minutes, due to the heightened coefficients of heat transmission to the tiles, as well as their uniformity and flexibility. In the single -deck roller kilns, the tiles travel over rollers and the heat required for firing is provided by natural gas–air burners fitted at the sides of the kiln.

The main heat transmission mechanisms are convection and radiation. As non-muffled kilns are involved, gas comes into direct contact with the tiles. This heightens the heat transmission coefficients, reduces the firing cycle and energy consumption and increases kiln flexibility compared to the kilns that were formerly used (Jones, 1996).



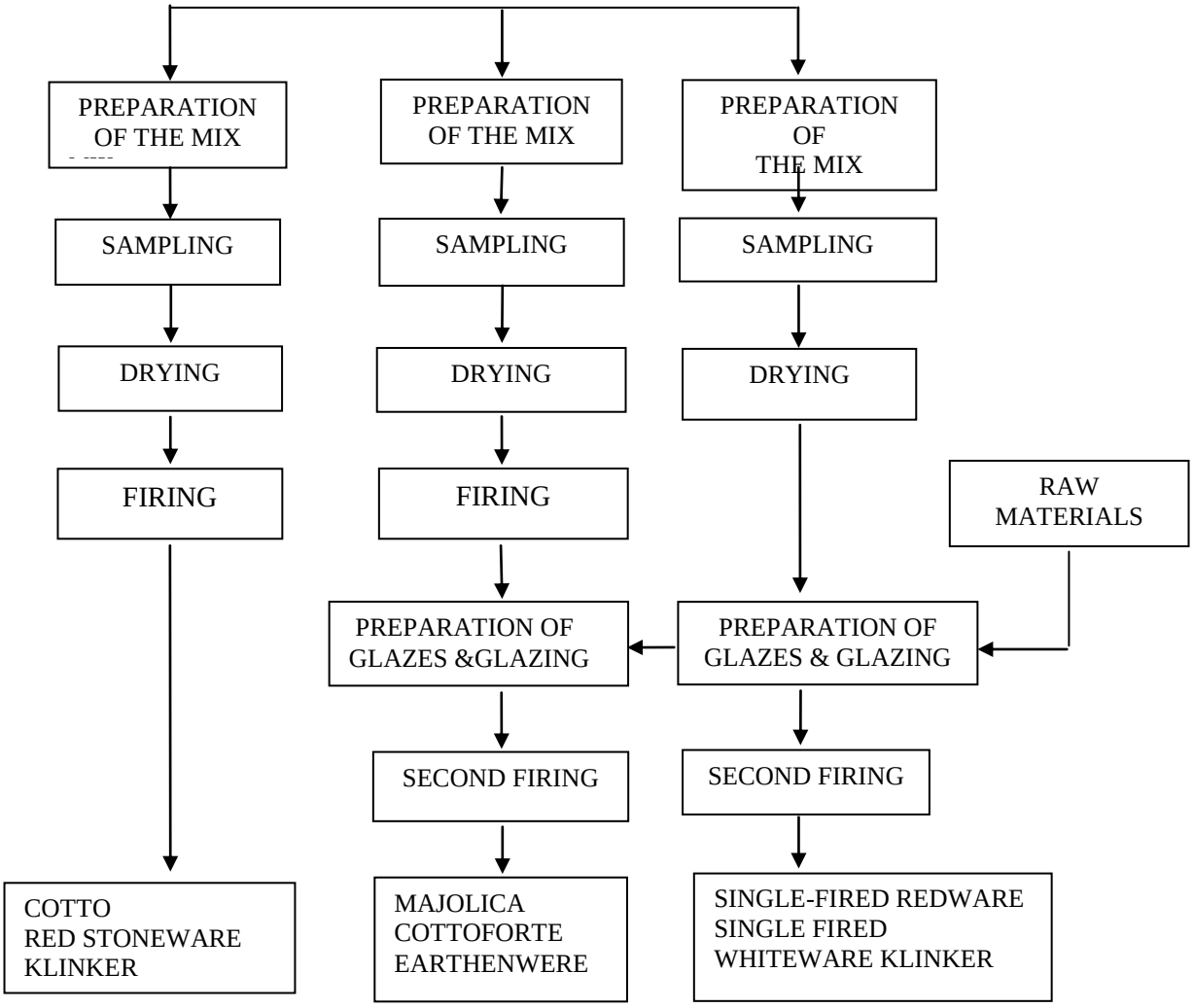


Figure 3: Technological Cycles for the Production of Ceramic Wall and Floor Tiles.
Source: Applied Processes and Techniques, (2005).

2.8 Standard/Methods of Testing Properties of Tiles

Ceramic tiles are expected to possess certain property which informs their suitability for some installation than others. A good knowledge of the characteristics and behaviour of ceramic wall and floor tiles according to Byrne (2008) is a fundamental prerequisite for the production and as well as achieving the desired and anticipated value of the tile. The following are some of the important attributes/tests of ceramic tiles as recommended by the American Society for Testing and Materials (ASTM) and European (EN) Standards, some of which are examined in this study: (a) Drying Shrinkage (b) Firing Shrinkage (c) Water Absorption, (d) Thermal Shock Resistance, (e) Chemical Resistance, (f) Modulus of Rapture.

2.8.1 Drying Shrinkage

Ceramic tiles are usually dried at high relative humidity after forming, especially if the wet method is used. Drying, which takes several days, removes the water at a slow rate to prevent shrinkage cracks and warpage.

ASTM C-326 describes the standard test method for determining the percentage shrinkage of the green ceramic tile. The shrinkage properties is determined by initially measuring the original length of the green tile along the centre and re-measuring the lengths after drying and firing of the tiles, using a venire calliper. The linear drying shrinkage is determined as a percentage of the plastic length using the following relationships:

$$S_d = \frac{L_p - L_d}{L_p} \times 100$$

Where:

S_d= Lineardrying shrinkage, %.

L_p = Plastic length of test specimen.

L_d= Dry length of test specimen.

2.8.2 Firing Shrinkage

The firing shrinkage of ceramic tiles can be determined by finding the percentage decrease in the dimensions of the dried unfired tile. This is done by calculating the total linear shrinkage after drying and firing of the tile specimen as a percentage of plastic length as follows:

$$Si = \frac{L_p - L_f}{L_p} \times 100$$

Where:

S_i = Total linearshrinkage after drying and firing, %.

L_p = Plastic length of test specimen.

L_f = Fired length of test specimen.

2.8.3 Water Absorption

Water absorption is the quantity of water which a ceramic tile can absorb under particular experimental conditions expressed as percentage in weight of the dry tile. The percentage of water absorption by the tile body determines whether ceramic tile is impervious, vitreous, semi-vitreous or non-vitreous. Since this absorption takes place through the pores of the material which are in communication with the external surface,Byrne (2008) states that waterabsorptionservesasameasureofthequantityofsuchpores in the material.

Thewater absorption,therefore,providesimmediateindicationofthestructureofthematerial. Highwater absorption corresponds to a porous structure, while compact, vitrified structures have a low water absorption.

ASTM C-373 describes the standard test method for determining the percentage of water absorbed by the body of a ceramic tile. Results are expressed as a percentage of the weight of water absorbed to the weight of dry tile. Impervious tile has water absorption of 0.5 % or less: vitreous tile more than 0.5 %, but not more than 3.0 %: semi-vitreous: more than 3.0 % but not more than 7.0 %: and non-vitreous tile has water absorption of more than 7.0 %. The body of glazed wall tile is normally non-vitreous with water absorption often exceeding 15%. The body of glazed floor tile is normally vitreous or semi-vitreous with water absorption between 2.0 and 6.0 %. There are however different designations for dust-pressed, extruded, and other body forms as stated as follows:

Impervious	**Vitreous**	**Semi-Vitreous**	**Non-Vitreous**	
	≤0.5%	3-0.5%	3-7%	7-20%
Forming Method				
Pressed	P1	P2	P3	P4
Extruded	E1	E2	E3	E4
Other	O1	O2	O3	O4

The procedure for determining the rate of absorption is as follows:

The dry and cold test specimens are initially weighed and subsequently submerged in clear water preferably soft, distilled or rain water at room temperature for 24 hours. The specimens are then removed and the water on the surface of the test specimens are wiped off and weighed again within 5 minutes after removing from the bath as specified in the ASTM C-373 standard.

The percentage water absorption of the specimen is calculated using:

Absorption $\% (W) = 100 (W_s - W_d) / W_d$

Where: W_d= dry weight of the specimen

W_s = saturated weight of the specimen after submerging in cold water.

2.8.4 Chemical Resistance

Chemical resistance refers to the behaviour of the ceramic surface when exposed to chemicals which, due to their composition and properties, corrode the ceramic surface so as to penetrate it permanently or at least alter its aesthetic appearance. The acid or base chemicals may include various processed liquids like milk in a cheese factory, lubricating grease and oils in an automotive repair shop, blood in a slaughterhouse, and laboratory chemicals. Others are materials that may come accidentally into contact with floor or wall tiles like foods, ink and the like in a private residence, as well as detergents for ordinary, special cleaning and maintenance.

Deterioration of a ceramic tile surface caused by the above cited chemicals according to Bradytiles, (2009) generally includes two distinct mechanisms effects: an actual chemical action, whereby the alteration of the ceramic surface is due to a reaction between the chemical and some component of the surface itself and a physical action of absorption, according to which the chemicals penetrates to the interior of the surface so as to lodge there permanently or at least firmly enough to be extremely difficult to remove. Bradytile, (2009) therefore statesthat glaze compositions should be properly formulated so as to be very resistant to chemical attack and easy to clean in order to achieve a better tile resistance to chemicals and stains.

The chemical resistance of the tiles are categorised into groups (A to E) and are determined by ASTM test C 650 based on the based on resistance to staining, with samples unaffected by any of the chemicals rated A, while samples affected by 4 or more chemicals are given an E rating.

Resistance Class	Maximum number of affected samples
A	0
B	1
C	2
D	3
E	4 or more

2.8.5 Modulus of Rapture (Flexural Strength)

The modulus of rupture is a parameter characteristic of a test specimen and indicates the maximum stress that a test specimen, subjected to an increasing stress from being bent under specific conditions and with specific procedures, can withstand before breaking. The modulus of rupture is measured in Newton per square millimetre (N/mm^2). The modulus of rupture is the mechanical characteristic to which reference is most generally made in regard to the quality control of ceramic tile. This parameter is used on the one hand to determine the correctness of the production process in relation to having obtained the desired degree of compactness and consistency, and on the other hand to determine whether or not the tile is capable, as far as its compactness is concerned, to support the static and dynamic loads to which the ceramic floor or wall may be subjected.

As its definition indicates, the breaking strength is a performance property of the tile, and is determined by both the structural properties of the material, and the size of the tile. The breaking strength tends to increase with the increase of the material's modulus of rupture and of the thickness of the tile.

A measure of the breaking strength of tiles according to ASTM C648 test values is expressed in pounds force and the minimum value for glazed wall tiles is 90 pounds.

The procedure for modulus of rapture (flexure test) that usually consists of not less than five specimens is determined as follows:

The test specimen is supported flatwise making sure that the support for the test specimens is free to rotate in the longitudinal and transverse direction and adjusted in such a way that they will exert no force in these directions. A load is then applied to the upper surface of the specimen at the centre to break it.

The modulus of rapture of each specimen is calculated as follows:

$$R = 3W\,(l/2 - X)/bd^2$$

Where:

W= maximum load at which the specimen failed

b = width if the specimen in (mm)

l = distance between the supports in (mm)

b = net width of the specimen at the plane of flexure in (mm)

d = thickness of the specimen at the plane of failure in (mm)

x = average distance from the mid-span of the specimen to the plane of failure measured in the direction of the span along the centreline of the bed surface subjected to tension, in (mm).

2.8.6 Thermal ShockResistance

Thermal shock refers to the sudden and unexpected variations of temperature of floor or wall tiles due to accidental or intentional contact with hot or cold bodies such as from the fall of a boiling liquid, for a cleaning with steamer machines, or, in the case of exterior floors or walls, from sudden changes in temperature. Thermal Shock resistance is the characteristic which indicates whether or not the tile is capable of withstanding such rapid changes in temperature without being damaged. There is no test value. The sample either passes or fails.

ASTM C-484 describes the standard test method for determining a ceramic tile's ability to withstand exposure to high temperature and rapid cooling without glazes showing

failure, or the body of the tile showing evidence of disintegration. The method for determining the resistance to thermal shock is applicable to all ceramic tiles under normal conditions of use and must be performed on at least five tiles free from defects at the beginning of the tests.

The procedure consists heating the glazed tiles in an electric oven to a temperature of 150^{o} C, removed from the oven and quickly placed on a thin sheet of aluminium at room temperature, The surface of the tiles are then inspected with a magnifying glass for a form of surface damage or cracks.

This procedure is repeated ten times so as to create a thermal cycling fatigue cycle's operations. After each operation, the surface of the tile is examined for visible defects.

2.9 X-ray Fluorescence (XRF)

X-ray fluorescence (XRF) spectrometer is an x-ray instrument used for routine, relatively non-destructive chemical analysis of rocks, minerals and fluids. X-ray fluorescence (XRF) spectrometry is an elemental analysis technique with broad application in science and industry. XRF is based on the principle that individual atoms, when excited by an external energy source, emit X-ray photons of a characteristic energy or wavelength. By counting the number of photons of each of the energy emitted from a sample, the elements present may be identified and quantitated.

Modern XRF instruments are capable of analysing solid, liquid, and thin-film samples for both major and trace components. The analysis is rapid and usually sample preparation is minimal or not required at all.

Preparation of samples for XRF analysis is highly variable depending on the matrix and goals of the analysis. For example most of the materials that areanalysedlike obsidian, metals, and ceramic paints do not require any sample preparation. The choice of sample

preparation depends on the nature of the X-ray beam relative to the sample. A piece of obsidian that is 1 cm thick and has a clean, flat surface will provide ideal results. As sample gets smaller, thinner, or less homogenous, Wirth, (2010) states that it is necessary to understand the nature of the X-ray beam and how it interacts with the sample.

2.10 X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis is an analytical technique designed to provide more in-depth information about crystalline compounds, including identification and quantification of crystalline phases. X-ray diffraction is a way of determining the structure of molecules and what substances are present in a material. The diffraction tells the distances within the crystalline structure. Since different molecules have different separations, one can distinguish different substances by the diffraction pattern they create.

PrimarilyX-Ray Diffraction analysis is used for the identification of crystalline phases for a large variety of powders and thin-film samples. This includes the analysis of corrosion products, ceramics, clays, oxide or nitride coatings and many others.

The information obtained from X-ray diffraction analysis includes types and nature of crystalline phasepresent, structural make-up of phases, degree of crystallinity, amount of amorphous content, micro strain as well as the size and orientation of crystallites.X-ray diffraction is a useful tool when trying to positively identify a contaminant or corrosion product, and for identification of foreign phases for purity analyses of crystalline powders.

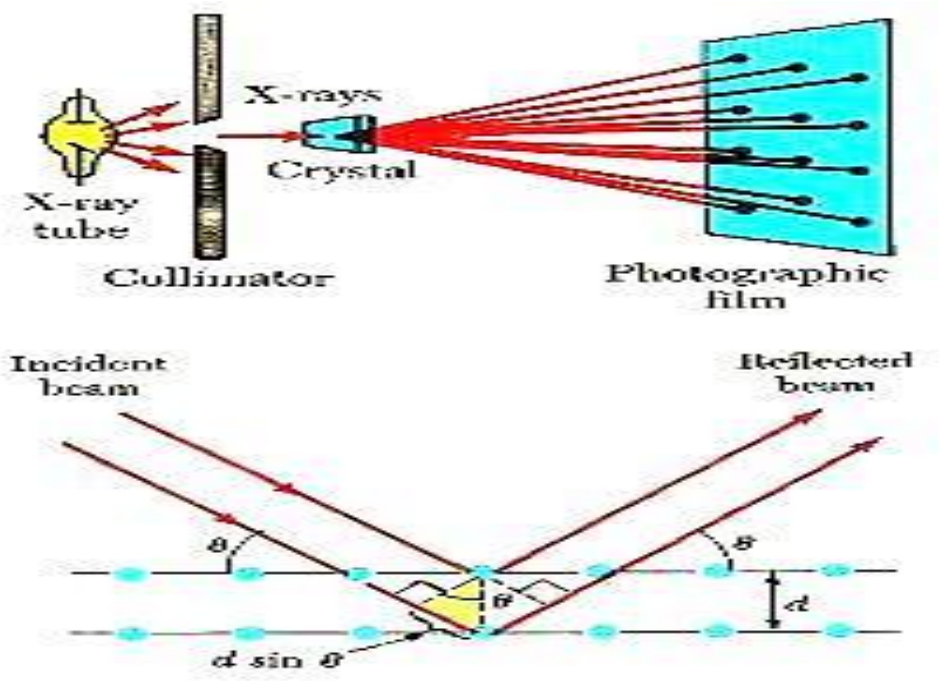


Figure 4: Schematic Diagram of X-ray diffraction.
Source: X-Ray Diffraction (XRD), Wirth (2010).

The XRD Analysis if performed by focusing X-Ray beam that is shot at the sample at a specific angle of incidence as illustrated in Figure 4. The X-Rays deflect or "diffract" in various ways depending on the crystal structure (inter-atomic distances) of the sample. The locations (angles) and intensities of the diffracted X-Rays are measured. Every compound has a unique diffraction pattern.

In order to identify a substance, the diffraction pattern of the sample is compared to a library database of known patterns. In addition to identification of crystalline phases, peak shapes and intensities can be used to gather information about percentagecrystallinity and crystalline size. Because the technique is structure based, XRD can differentiate between compounds with the same elements, yet different properties such as Fe_2O_3 and Fe_3O_4.

2.11 Scanning Electron Microscope

The scanning electron microscope (SEM) is a microscope that uses electrons instead of light to form an image. Since their development in the early 1950's,Schweitzer states that the scanning electron microscopes have developed new areas of study in the medical and physical science communities. The SEM has allowed researchers to examine a much bigger variety of specimens.

The scanning electron microscope has many advantages over traditional microscopes. The SEM has a large depth of field, which allows more of a specimen to be in focus at one time. The SEM also has much higher resolution, so closely spaced specimens can be magnified at much higher levels. Because the SEM uses electromagnets rather than lenses, the researcher has much more control in the degree of magnification. All of these advantages, as well as the actual strikingly clear images, make the scanning electron microscope one of the most useful instruments in research today.

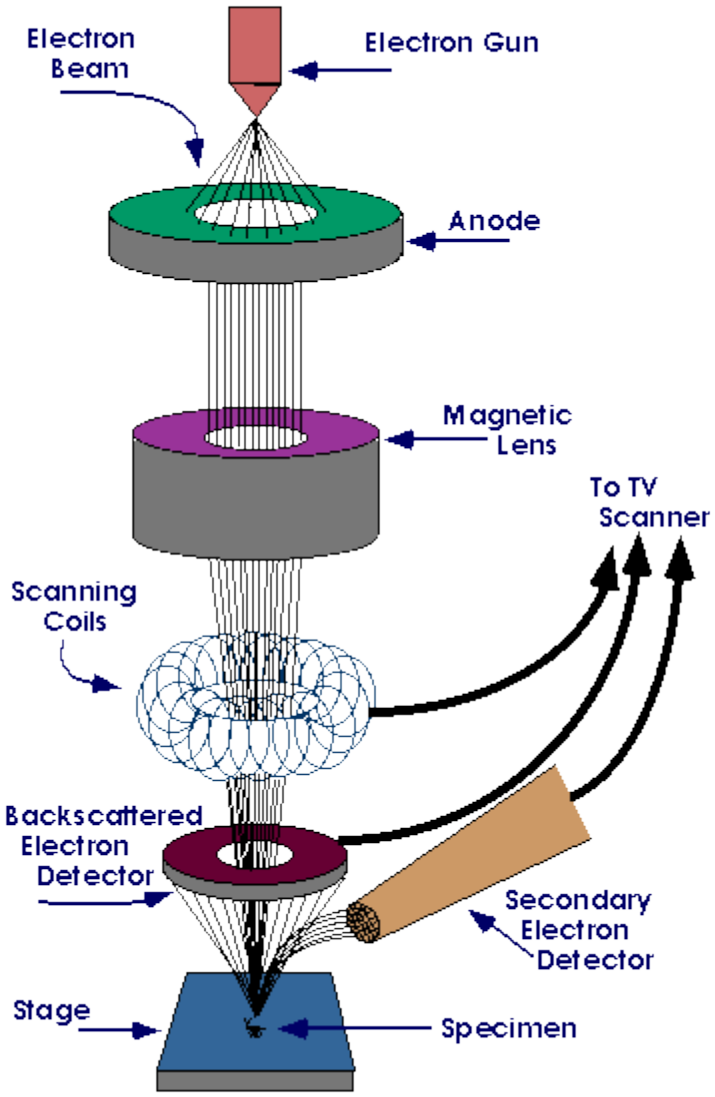


Figure 5: Diagram of Scanning Electron Microscope.
Source: TheScanning Electron Microscope (SEM), Schweitzer, (2014).

The SEM is an instrument that produces a largely magnified image by using electrons instead of light to form an image. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum. The beam travels through electromagnetic fields and lenses, which focus the beam down toward the sample. Once the beam hits the sample, electrons and X-rays are ejected from the sample.

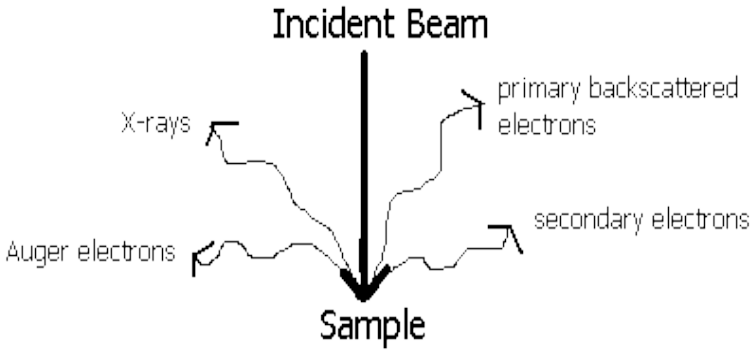


Figure 6: Diagram of Effects of Electron Beam on Sample in a Scanning Electron Microscope
Source: The Scanning Electron Microscope (SEM), Schweitzer, (2014).

Detectors then collect these X-rays, backscattered electrons, and secondary electrons and convert them into a signal that is sent to a screen similar to a television screen. This produces the final image.

2.12 Apparent Porosity

The pores in a material which is either open or closed type controls the density can be apparent or true. Apparent porosity is the percentage relation between the volume of the open pore space and the total volume of the sample. The percentage apparent porosity is calculated thus:

$$Apparent\ Porosity = \frac{W - D}{W - S}\ x\ 100$$

Where:

D = Dry Weight of Sample

W = Saturated Weight of Sample in Air

S = Weight of Sample Suspended in Water

2.13 Bulk Density

The bulk density which is the weight per unit volume of a material is responsible for the over-all weight including its pores. Bulk density was calculated using a direct volume measurement method and is determined by dividing the mass of the brick by the external volume of the tile.

$$\text{Bulk Density} = \left(\frac{D}{W-S}\right) \text{g/cm}^3$$

Where:

D = Dry Weight

W = Saturated Weight

S = Suspended Weight in Water

2.14 Thermal analysis (DTA-TG)

When a material is heated its structural and chemical composition can undergo changes such as fusion, melting, crystallization, oxidation, decomposition, transition, expansion and sintering. Using Thermal Analysis such changes can be monitored in every atmosphere of interest. The obtained information is very useful in both quality control and problem solving.

There are two types of thermal analysis; TG (Thermogravimetric) analysis and DTA (Differential Thermal Analysis).Differential thermal analysis has long been applied in the study of minerals such as clay minerals, carbonates, sulphates and zeolites, sulphides and arsenides, graphite.In Differential Thermal Analysis, the temperature difference that develops between a sample and an inert reference material is measured, when both are subjected to

identical heat - treatments.Thermogravimetric(TG) analysis on the other hand determines the weight gain or loss of a condensed phase due to gas release or absorption as a function of temperature.This method is useful for determining sample purity and water, carbonate, and organic content; and for studying decomposition reactions.

The instruments used for the thermal analysis are predominantlythermocouples, platinum resistance thermometers, and thermistors. When the sample undergoes a transformation, it will either absorb (endothermic) or release (exothermic) heat.Sharp Endothermic physicalchangesin crystallanity or fusionusually result in endothermic curves. Chemical reactions are exothermic.

CHAPTER THREE

MATERIALS AND METHODS

3.0 Research Design

This study sought to experimentally investigate the possibility of incorporating rice husk ash as a material for the production of ceramic wall tiles. In order to accomplish this study properly, detailed investigation was focused on:

i. Choice of raw material and their mineralogical composition

ii. Formulation and preparation of five different batches of tile bodies.

iii. Studies of the physico – chemical properties

iv. Comparison with local tiles

3.1. Methodology

In this study, the following steps were adopted: mineralogical analysis of the materials, beneficiation and characterization of the raw materials, mould design and fabrication, formulation of specimens, drying and sintering of the specimens and property tests of the prepared tiles.

3.2. Population of the Study

In order to fulfil the objectives of the present study, the choice of locally available raw materials was very important. The selection of Rice Husk (RH) was paramount as it was the source of silica for the replacement of quartz in the tile body composition due to its huge availability.

The additional raw materials used in the study were Quartz, Bomo Clay (Ball Clay) Kankara Clay (Kaolin) and Calcium Carbonate (Whiting).

3.2.1. Sourcing of Raw Materials

The Rice Husk (RH) used for the study was obtained from a local rice mill in Zaria City. Bomo Clay was manually mined at Bomo, a village in Zaria, while the quartz was handpicked from the dried river bed at the same Bomo Village. Kaolin was bought from the local clay miners at Kankara in Katsina State and Calcium Carbonate from Ikepeshi – Akoko in Edo State was purchased from the local market in Zaria.

3.3 Beneficiation of Raw Materials

The rice husk, quartz, kaolin and Bomo clay were subjected to a series of beneficiation processes. The calcium carbonate was not beneficiated because it had already been processed into powder form.

3.3.1 Rice Husk

In order to convert the rice husk (RH) to rice husk ash (RHA), the rice husk collected from the rice mill in Zaria City was burnt. Before the burning, the RH was heaped in an enclosure made of clay bricks so that the RH will not be blown away due to its light weight. Fire was then set on it in the open air.

By this controlled burning of the RH in the open air, it was possible to reduce the presence of carbon due to incomplete combustion as much as possible, thus obtaining a whitish rice husk ash. The RH took five days to completely burn into ash in the open air.



Plate 1(a)



Plate 1(b)

Plate I (a) and (b): Various Stages of the Burning of the Rice Husk into Ash.

Source: Research Photograph,Opoku (2012).



Plate 1(c)



Plate 1(d)

Plates I (c) and (d): Various Stages of the Burning of the Rice Husk into Ash.
Source: Research Photograph,Opoku(2012).

3.3.2. Kankara Clay (Kaolin)

The Kankaraclay was beneficiated by first soaking it in water for two days in plastic containers so that the clay can be well slaked. It was then blunged and sieved through a 200 Tyler mesh (75μm). The slurry was then allowed to sediment.

The water that had collected on top of the slurry was then decanted and poured away while the remaining material was poured into a clean plaster mould to further reduce the water content. On the second day, the Kankara clay was removed from the plaster mould and dried in the open air before crushing it into powder.

3.3.3. Bomo Clay (Ball Clay)

The Bomo Clay underwent the same processes as the Kankara Clay. It was first soaked in water to slake before blunging. It was then sieved through the 200 tyler mesh and allowed to sediment.

The water was then decanted, poured into a plaster mould to further reduce the water content before it was removed and dried in the open air. When it was well dried, it was crushed into powder.

3.3.4 Quartz.

The quartz collected from the river bed near Bomo village was thoroughly washed with water and dried before it was fired to a temperature of 700^{o}C. This calcination process facilitated the breakdown of the cleavages of the quartz and made it easy to crush.

The calcined quartz was then crushed into smaller sizes with a crusher before charging them into a horizontal ball mill for further wet grinding into finer particles. The milling of the quartz was done for 6 hours each day for two days before it was discharged into a bin.

After two days, the water that had collected on top of the milled quartz was decanted and left to dry in the open air.

3.4 Characterization of the Materials

Testing for the chemical and mineralogical composition of materials used for ceramics is important to be known because these chemicals and minerals have a great influence on the technological behaviour and the final properties of the fired product (King, 2002). In this respect, specific effort was made to determine the chemical compositions of the raw materials used for the study using the X-ray Fluorescence (XRF) analysis as well as the mineralogical crystals of the fired samples using X-ray Diffraction (XRD) analysis.

The X-ray Fluorescence (XRF) analysis was conducted at the Multi User Laboratory of the Department of Chemistry, Ahmadu Bello University, Zaria, Nigeria using the Oxford Instrument X Supreme 8000 while X-ray Diffraction (XRD) analysis was conducted at the National Steel Raw Materials Exploration Agency (NSRMEA) Kaduna, Nigeria with a Computerized X-ray Diffraction Equipment that uses Cu Kx radiation at a scan speed of 4^{o}/min 2O.

3.5 Mould Fabrication

In order to make many tiles with the same design and consistency of thickness with less distortion around the edges, Carols (2008) is of the opinion that the use of cavity moulds (Plates 2 (a) and (b) should be considered. The cavity mould technique for producing tiles was therefore adopted for the production of the tile specimens.

An original tile model 14.7 x 5.5 x 0.7$cm^3$was made out of clay avoiding undercuts. Water was then rubbed on the bottom of the tile to create a thick slip and stuck onto a board. Plaster retention boards were then assembled around the template (the original tile that has

been stuck on the board) allowing 2.5 inches of space around all the sides. The mould boards were then fastened to keep the plaster from leaking when poured onto the template. For this purpose, a wad of day was put at the base of each wall to keep the form secure.

Before mixing the plaster, measurement of the water and plaster was made at a ratio of 0.7 parts of water to I part of plaster (Frith, 1992). The plaster for the mould was prepared by first sifting the weighed plaster into water of the determined quantity in a container and allowed to slake for about a minute without agitation. The plaster was then mixed with the fingers until a heavy creamy consistency was achieved.

When the mixture of plaster and water had reached a proper consistency, it was poured slowly onto the template placed in the middle of the assembled mould boards in the form of a steady stream. The table used for the preparation of the mould was then agitated slightly to bring any air bubbles in the plaster mixture to the surface and to settle the plaster out making the top completely flat. After the plaster had set, the mould was then taken apart from the plaster retention wall.



Plate 2(a): Cavity Moulds used for the Production of Tiles.
Source: Research Photograph,Opoku (2012).



Plate 2(b): Cavity Moulds used for the Production of Tiles.
Source: Research Photograph,Opoku (2012).

3.6.0 Preparation of the Tile Body

In view of the fact that the study was based on the incorporation of rice husk ash as a replacement for quartz in a wall tile body, it became apparent that a tile body had to be formulated.

Upon inquiry from the internet, Dr Alberto Silva a Ceramic Consultant for Digitalfirein 2010 suggested a reference wall tile recipe that could be further studied to develop a basic wall tile body maturing at 1160^{o}C. This body, he further stated would have the allowable recommended tile properties such as compressive strength, maximum percentage water absorption and maximum percentage total shrinkage as specified by ASTM standard for bricks and clay tiles (ASTM C 67-00). The recipe suggested by Dr Alberto was:

Ball Clay	20%
Kaolin	30%
Quartz	22%
Calcium Carbonate (Whiting)	20%

3.6.1 Preparation of the ExperimentalTile Bodies

Guided by the information gathered from the Ceramic International Journal (2010) that the basic raw materials used for ceramic tiles are appropriate clays, carbonates, feldspars, Kaolins and quartz, an industrial wall tiles body elaborated from Dr. Alberto Silva recipe served as the standard wall tile body mix. This typical industrial body was prepared from a mixture of plastic clay, kaolin, quartz and calcium carbonate.

The beneficiated raw materials (Bomo clay, kaolin, calcium carbonate, quartz and rice husk ash) were formulated into compositions of five different batches of wall tile bodies according to predetermined amounts. The standard tile body mix S1 was selected as the starting composition.

To produce tiles with body samples S2, S3, S4, S5, S6 for the study, quartz was varied with 5% over the range of 22%, 17%, 12%, 7%, 2%, 0% (5% decrement) and a corresponding substitutionof 5% increment of 0%,2%,7%,12% 17% 22% of RHA. Sample 1 (S1) was the basic tile body composition developed from Dr. Alberto Silva's recipe.

Table 6: Body Composition with Progressive Replacement of Quartz with Rice Husk Ash in a Standard Wall Tile Composition (mass %).

Mix No.	China Clay (Kankara Clay)	Whiting	Bomo Clay (Ball Clay)	Quartz	Rice Husk Ash
S1	30	20	28	22	0
S2	30	20	28	17	2
S3	30	20	28	12	7
S4	30	20	28	7	12
S5	30	20	28	2	17
S6	30	20	28	0	22

Table 6 shows the matrix planning with six mixtures of independent components (Kankara Clay, Whiting, Bomo Clay, and Quartz) and the reference material (RHA). For example, to produce tiles with sample S2, S3, S4, S5 and S6 the percentage of quartz was varied over the range of 22%, 17%, 12%, 7%, 2% and 0% to 5% progressive increment of RHA content(0%, 2%, 7%, 12%, 17% and 22%).

Following the proportions in Table 6, five kg of each batch composition was prepared and subjected to ball milling in the dry state for homogenization ensuring that no grinding media were introduced. The ball milling took 1 hour for each batch before discharging. After discharging the mixture, 15% water (by weight) was added to each batch and kneaded until it

became plastic and workable. The body mixtures were then rolled into ball shapes, wrapped in polythene bags and stored away for two weeks to age.

3.7 Production of the Test Tiles

After seven days the composite mixtures were ready for the preparation of the tile specimens using the cavity mould and hydraulic press. A slab of clay from the prepared body was cut slightly larger than the mould (Plate 3) and placed on top of the mould. It was then covered with a block of wood (Plate 4) before pressing. Rectangular specimens measuring 14.7 x 5.5 x 0.7cm^{3}were prepared by pressing with a hydraulic press (Plate 5) at a pressure of 15MPa. This was intended to guarantee that the degree of compaction for all the tile specimens was always the same. After the pressing, a wire cutter was used to slice off the excess clay (Plate 6) before a metal straight edge was used to scrape and level the top of the tile (Plate 7).



Plate 3: Slicing of the prepared body before placing on the mould.
Source: Research Photograph,Opoku (2012).



Plate 4: Mould with Clay Covered with a Block of Wood before Pressing.
Source: Research Photograph,Opoku (2012).



Plate 5: Pressing of the tiles with the Hydraulic Press
Source: Research Photograph,Opoku (2012).



Plate 6: Slicing off the excess clay after the pressing
Source: Research Photograph,Opoku (2012).



Plate 7(a): Scraping and levelling the top of the Pressed Tile.
Source: Research Photograph,Opoku (2012).



Plate 7 (b): Scraping and levelling the top of the Pressed Tile.
Source: Research Photograph,Opoku (2012).



Plate 8: Removal of the Pressed Tile from the Mould
Source: Research Photograph,Opoku (2012).

3.8 Drying

After the pressing of the tile, they were removed from the mould (Plate 8).They were placed directly onto a rigid metal rack in a room to dry (Plate 9). This process allowed air to circulate on all sides of the tile and as such dried evenly without warping. This drying process according to Carlos (2005) is an important process that affects the product yield and requires a longer time. Inappropriate drying may cause the tile to cut or get broken, and as such it was necessary to use the appropriate conditions of placement, temperature, humidity and time.

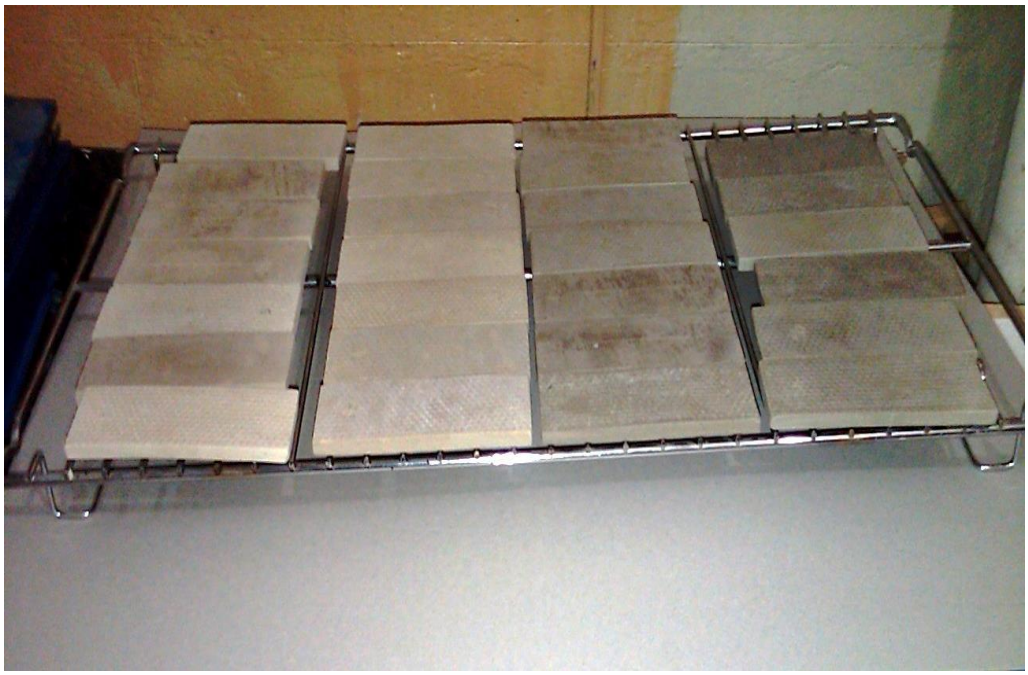


Plate 9 (a): Pressed Tiles Assembled on a Rack to Dry.
Source: Research Photograph,Opoku (2012).



Plate 9(b): Pressed Tiles Assembled on a Rack to Dry.
Source: Research Photograph,Opoku (2012).

3.9 Sintering

A set of pressed tiles from each of the six matrix groups were fired at 1100^{o}C, 1140^{o}C and 1160^{o}C using an electric kiln at the Industrial Development Centre (IDC), Zaria (Plate 10).

Each set of tiles were loaded into the kiln, sealed and fired gradually over three firing stages. These stages included pre-heating for one hour, full firing for six to ten hours, thirty minutes and soaking for thirty minutes. The firings were thus completed in between eight to twelve hours.



Plate 10: The Tiles loaded into the Electric Kiln for Firing.
Source: Research Photograph,Opoku (2012).



Plate 11: Tiles Fired at Different Temperatures
Source: Research Photograph,Opoku (2012).



Plate 12: Glazed Tiles
Source: Research Photograph,Opoku (2012).

3.10 Property Tests

A number of tests based on the American Society for Testing and Materials (ASTM C67-00) were carried out on the tiles produced for some properties required to determine the status of tile specimens. These tests included: Drying Shrinkage, Firing Shrinkage, Water Absorption, Modulus of Rapture (Flexural Strength) and Thermal Shock Resistance.

3.10.1 Drying Shrinkage

ASTM C-326 describes the standard test method for determining the percentage shrinkage of the green ceramic tile. The shrinkage properties was determined by initially measuring the original length of the green tile along the centre and re-measuring the lengths after drying and firing of the tiles, using a venire calliper. The linear drying shrinkage was determined as a percentage of the plastic length using the following relationships:

$$S_d = \frac{L_p - L_d}{L_p} \times 100$$

Where:

S_d= Lineardrying shrinkage, %.

L_p = Plastic length of test specimen.

L_d= Dry length of test specimen.

3.10.2 Firing Shrinkage

The firing shrinkage of ceramic tiles was determined by finding the percentage decrease in the dimensions of the dried unfired tile. This was done by calculating the total linear shrinkage after drying and firing of the tile specimen as a percentage of plastic length as follows:

$$Si = \frac{L_p - L_f}{L_p} \times 100$$

Where:

S_i = Total linearshrinkage after drying and firing, %.

L_p = Plastic length of test specimen.

L_f = Fired length of test specimen.

3.10.3 Water Absorption

This parameter is measured to determine the quantity of water which a ceramic tile can absorb under particular experimental conditions expressed as percentage in weight of the dry tile. The percentage of water absorption by the tile body determines whether ceramic tile is impervious, vitreous, semi-vitreous or non-vitreous.

ASTM C-373 describes the standard test method for determining the percentage of water absorbed by the body of a ceramic tile. Results are expressed as a percentage of the weight of water absorbed to the weight of dry tile.

The fired and cold test specimens was initially weighed and subsequently submerged in clean water at room temperature for 24 hours. After this period, the specimens were then removed and the water on the surface of the test specimens wiped off and weighed again within 5 minutes after removing from the bath.

The percentage water absorption of the specimen was calculated using:

Absorption $\% (W) = 100 (W_s - W_d) / W_d$

Where:

W_d=dry weight of the specimen

W_s = saturated weight of the specimen after submerging in cold water.

3.10.4 Modulus of Rapture (Flexural Strength)

Modulus of rupture is a mechanical characteristic to which reference is usually made concerning the quality control of ceramic tiles. MOR is utilized for two reasons.Firstly, it is used to determine the correctnessof the production process in relation to having obtained the desired degree of compactness and consistency. Secondly, it is used to determine whether or not the tile is capable ofsupporting the loads that will be imposed on it.

As its definition indicates, the breaking strength is a performance property of the tile, and is determined by both the structural properties of the material, and the size of the tile. The breaking strength tends to increase with the increase of the material's modulus of rupture and of the thickness of the tile.

The thickness of the specimens is also taken into account when determining the limits of breaking strength and modulus of rupture. In other words, both water absorption and thickness of the specimen are the two criteria that are used todetermine the limits of breaking strength and modulus of rupture of the specimens (Hanan and El Nouhy, 2013).

A measure of the breaking strength of tiles according to ASTM C-648 test values is expressed in Newton's per square millimetre (N/mm^2) pounds force and the minimum value for glazed wall tiles is 90 pounds.

The procedure for modulus of rapture (flexure test) that usually consists of not less than five specimens is determined as follows:

The test specimen is supported flatwise making sure that the support for the test specimens is free to rotate in the longitudinal and transverse direction and adjusted in such a way that they will exert no force in these directions. A load was then applied to the upper surface of the specimen at the centre to break it.

The modulus of rapture of each specimen was calculated as follows:

$$M.O.R. = 3WL/2bt^2$$

Where:

W= maximum load at which the specimen failed (N)

L = distance between the supports in (mm)

b = width of the specimen in (mm)

t= thickness of the specimen at the plane of failure in (mm)

3.10.5 Characterization of the Sintered Samples

The morphological characterization of the formulations was performed by scanning electron microscopy on the fractured surfaces of the samples sintered at 1160oC

Scanning Electron Microscopy images of porosity evolution during sintering were obtained with a JEOL JSM 6360LV microscope in fracture mode for samples 1 – 6 sintered at 1160oC at the Department of Chemical Engineering, Ahmadu Bello University, Zaria, Nigeria.

CHAPTER FOUR

4.0 Results and Discussions

This chapter exhibits and discusses the results of the study. It contains the all the test results and treatments applied to the tile body based on specific methods applied in the study.

The summary of items in this chapter includes results of beneficiation and characterization of the materials as well as property test results on the ceramic tile samples. The property tests include linear and firing shrinkage, water absorption, and flexural strength.

4.1 Beneficiation of Raw Materials

The rice husk, quartz, kaolin and Bomo clay used for this study went through various beneficiation processes. Through these processes, it was possible to wash out the mineral salts in the clays and other impurities like organic and carbonaceous matter. The process of soaking, sieving and levigationalso helped in washing out the mineral salts as well as the removal of the organic matters.

Through the burning of the rice husk in the open air, it was possible to achieve a whitish rice husk ash with negligible amount of carbon traces.

4.2 Chemical Analysis

The distinctive role of ceramic raw materials used for processing ceramic tiles is significantly influenced by their mineralogical compositions as well as their physical and chemical properties (Worrall, 1986). Table 1 presents a summary of the chemical composition of the raw materials that were used for the study by means of X-ray Fluorescence Spectrophotometer (XRF). The analysis of the rice husk ash (RHA) revealed that silica (SiO_2) was the most dominant mineral of about 82% after complete burning. Other minerals like MgO, Al_2O_3, P_2O_5, FeO, K_2O and CaO are present in the RHA but forms only 1 - 6% of the total composition. Thus the RHA waste material can be considered from the chemical view point as a pure natural material because its composition is similar to quartz.

The other raw materials like whiting, clay and kaolin contained all their mineralogical components like CaO (Lime), SiO_2 (Silica) and Al_2O_3 (Alumina) that had effect on the processing and the compaction of the tile samples. However, these minerals influenced the sintering variables that produced modifications of linear shrinkage and porosity as well as the mechanical properties of the tiles. The MgO, K_2O and Na_2O_3 acted as fluxes and had a strong effect on the lowering of the sintering temperature. Other minor components like the Fe_2O_3 in addition to being a flux, mostly affected the colour of the sintered bodies.

The presence of Na_2O, K_2O and CaOalso enhanced the liquid phase during sintering by forming low melting eutectic compounds (Pontikes et al, 2009). Their presence helped also in reducing the sintering temperature of the tiles body by fusing at lower temperatures and dissolving materials like quartz which melt at higher temperatures. This Karaman et al, 2006 states leads to the glassy phase formation that promotes strength and fracture toughness.

Table 7: Chemical Composition of Raw Materials Used for the Study (%)

Material / Compound	Quartz	Kaolin	Calcium Carbonate	Ball Clay	Rice Husk Ash
Al_2O_3	-	42.8	0.1	13.38	-
SiO_2	98.6	54.8	0.2	55.90	82.14
K_2O	0.37	0.961	0.01	2.89	5.35
CaO	0.18	0.35	55.7	1.96	2.26
MnO	-	0.011	0.01	0.19	0.332
Fe_2O_3	0.084	0.409	0.05	10.60	1.03
TiO_2	0.046	0.046	-	1.0	0.15

NiO	0.010	0.0078	-	-	0.009
CuO	0.062	0a.059	-	-	0.070
ZnO	-	0.019	-	-	0.070
Ga_2O_3	-	0.021	-	-	-
Rb_2O	-	0.0082	-	-	0.043
Y_2O_3	0.020	0.026	-	-	0.013
ZrO_2	-	0.020	-	-	0.024
RuO_2	0.479	0.409	-	-	0.28
Re_2O_7	-	0.008	-	-	0.01
OsO_4	0.018	0.01	-	-	-
Cr_2O_3	0.018	0.019	-	-	0.016
Au	0.031	0.019	-	-	-
Gd_2O_3	0.057	-	-	-	-
MgO	-	-	0.01	-	-
P_2O_5	-	-	0.11	0.22	11.0
Na_2O	-	-	-	1.47	0.350
SO_3	-	-	-	-	0.05
H_2O_5				9.35	

Source: The Multi User Laboratory of the Department of Chemistry, Ahmadu Bello University, Zaria, Nigeria

4.3 Preparation of the Experimental Bodies

The ball mill was used for the mixing of the formulated tile test bodies ensuring that no grinding media were introduced. Mixing the formulated bodies in the ball mill produced homogeneous batches.

A uniform moisture distribution of the batches was achieved when the desired quantity of water was added, kneaded and stored away in polyethylene bags. This mode of storing allowed within the matrix, a uniform distribution of moisture. It also aided the batches to gain excellent particle inter locking bonding.

The homogeneous mixing, in addition to the ageing period of seven days helped significantly in the production, drying and handling of the test tiles before sintering without cracks.

4.4 Press Forming of the Bricks

Rectangular specimens measuring 14.7 x 5.5 x 0.7cm^3 were successfully prepared by pressing with a hydraulic press at a pressure of 15MPa in a plaster of Paris mould. This mode of production guaranteed that the degree of compaction for all the tile specimens was the same.

4.5 Drying and Sintering of Test Tiles

The pressed tiles were placed directly onto a rigid metal rack in a room to dry. This process allowed air to circulate on all sides of the tile and as such they dried evenly without warping. This confirmed Carlos (2005) assertion with regards to the use appropriate conditions of placement, temperature, humidity and time during the drying of tiles since inappropriate drying may cause the tile to cut or brake.

The electric kiln at the Industrial Development Centre, Zaria, Nigeria was used for the sintering of the tiles because it had a temperature monitoring device like a thermocouple and a meter for reading the actual temperature. It was observed that the formulated tile bodies underwent some physical, chemical and mineralogical changes according to the various temperatures at which they sintered (1100^{o}C, 1140^{o}C and 1160^{o}C). The fired colour of the tile for example were creamer and dense at 1160^{o}C than those fired at 1100^{o}C that were light brick red and porous.

The effects of the firing temperatures are further discussed under linear shrinkage (LS), water absorption (WA), X-ray diffraction (XRD) analysis and Flexural tests (FS).

4.6 Linear Firing Shrinkage

Table 8: Percentage Linear Shrinkage of Experimental Bodies

Sample	Plastic Length	Dry Length	Fired Length	Fired length	Fired length	Drying Shrinkage	Firing Shrinkage	Firing Shrinkage	Firing Shrinkage

	(cm)	(cm)	1100°C (cm)	1140°C (cm)	1160°C (cm)	(%)	1100°C (%)	1140°C (%)	1160°C (%)
S1	14.7	14.35	14.32	14.24	14.20	2.38	2.59	3.13	.4
S2	14.7	14.35	14.31	14.24	14.18	2.38	2.65	3.13	3.54
S3	14.7	14.35	14.31	14.24	14.18	2.38	2.65	3.13	3.54
S4	14.7	14.35	14.31	14.22	14.17	2.38	2.65	3.27	3.61
S5	14.7	14.35	14.30	14.22	14.16	2.38	2.72	3.27	3.61
S6	14.7	14.35	14.30	14.21	14.16	2.38	2.75	3.43	3.67

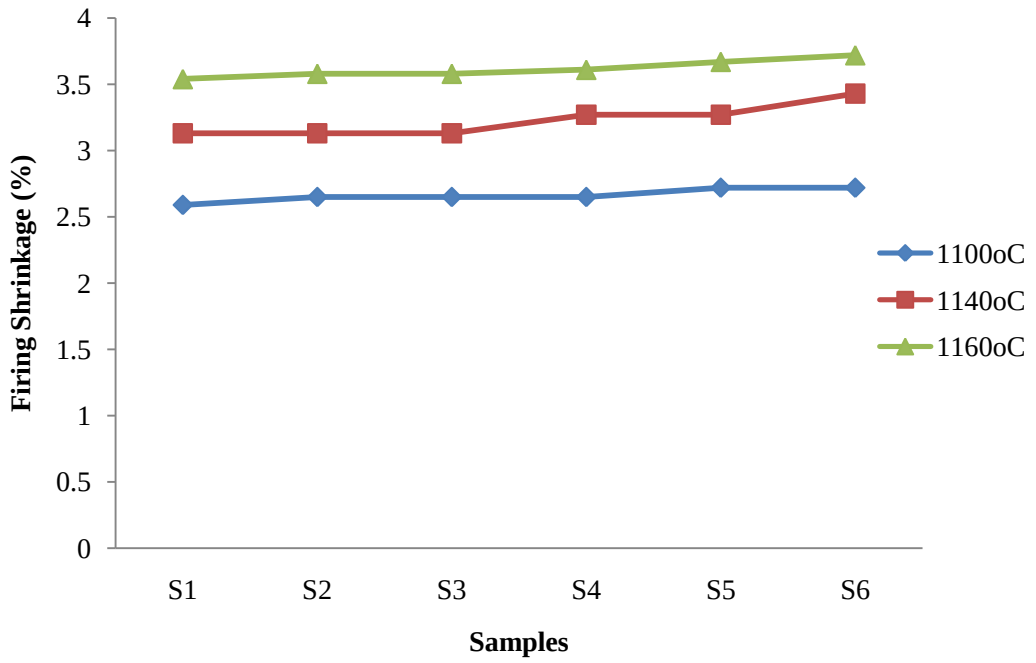


Figure 5: Variation in Linear Firing Shrinkage with temperature.

Figure 5 present the behaviour of the linear shrinkage as a function of the sintering temperatures for the prepared tile samples. No significant increase in shrinkage was observed with increase in firing temperature. It can be seen that they all showed similar behaviour with a comparatively greater linear shrinkage at 1160°C that can be attributed to the formation of the liquid phase that fills the pores and promotes approximation of the particles.

It is also clear from the line chart that the tile specimens exhibited low firing shrinkage which is well within the acceptable limit for industrial production of wall tiles. Linear shrinkage is an important criterion representing the quality of tile and a lower value of firing shrinkage is always desired to researchers. In the industrial processing of ceramic bodies such as wall and floor tiles, due to the sensitivity of the raw materials and processing changes that can be determined in the laboratory, linear firing shrinkage is frequently used as quality and process control parameter in the development and manufacturing stages (Correia, Hotza, and Segadães, 2004)).

Linear firing shrinkage is also used as a restricting and quality control parameter during the production processes of tiles as the desired values are used to specify the range of acceptable compositions.

4.7 Water Absorption

Table 9: Percentage water Absorption at 1100^{o}C

Sample	Dry Weight (g)	Soaked Weight (g)	Water Absorption (%)
S1	101.3	122.8	21.2
S2	101.3	122.5	21.0
S3	101.5	121.4	20.7
S4	101.5	120.7	20.0
S5	98.8	118.8	19.6
S6	98.8	118.0	19.4

Average Water absorption at 1100^{o}C is 20.3%

oTable 10: Percentage Water Absorption at 1140^{o}C

Sample	Dry Weight (g)	Soaked Weight (g)	Water Absorption (%)
S1	104.8	124.5	18.8
S2	104.8	124.2	18.5
S3	104.5	123.8	18.4
S4	103.8	122.4	18.0
S5	103.5	121.8	17.7
S6	103.5	121.2	17.1

Average Water Absorption at 1140^{o}C is 16.8%

Table 11: Percentage Water Absorption at 1160^{o}C

Sample	Dry Weight (g)	Soaked Weight (g)	Water Absorption (%)
S1	106.0	12.5	16.5
S2	106.2	123.5	16.3
S3	106.1	123.4	16.3
S4	106.3	123.5	16.1
S5	107.8	124.7	15.7
S6	108.0	124.2	14.8

Average Water Absorption at 1140^{o}C is 16%

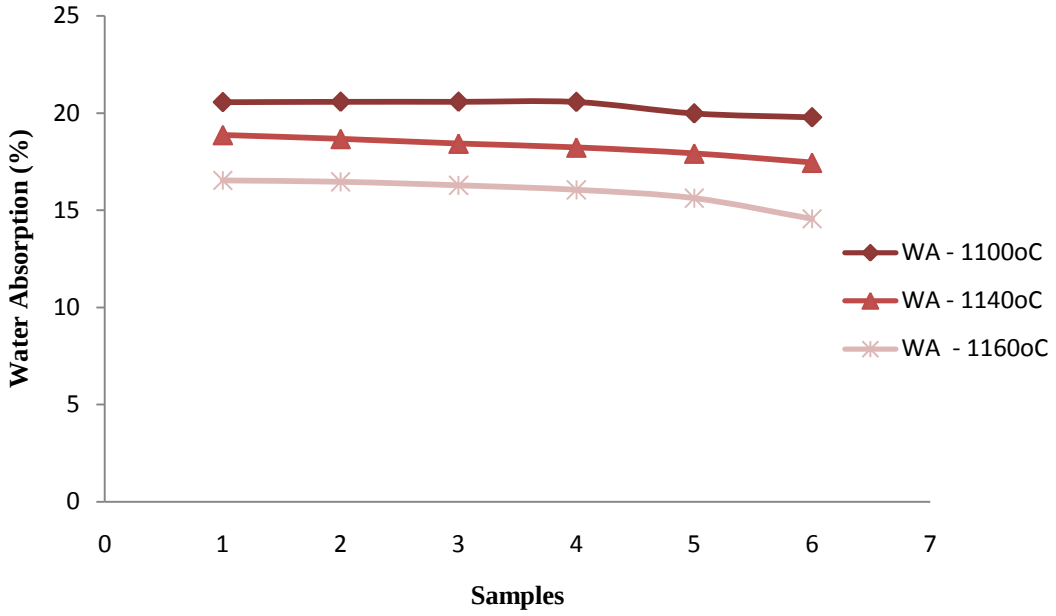


Figure 6: Variation in Water Absorption with Temperature.

Figure 6 presents the water absorption in relation to the firing temperature for all the prepared tiles. Water absorption is a means that can be used to evaluate the sintering process of ceramic tiles. In Figure 6, it was observed that all the formulations showed a decrease in water absorption with increasing temperature. This is as a result of the reduction in the open porosity due to the approximation of the particles associated with both solid state and liquid phase formation during the sintering process. Such behaviour is expected of ceramic bodies because of densification caused by the vitreous phase sintering process.

After sintering at 1100^{o}C, Figure 3 showed apparent porosity around 12%. When sintered at 1140^{o}C the test samples showed a decrease in water absorption of 5.5%. At 1160^{o}C, there was a further decrease in water absorption around 5%. The water absorption of the experimental tile formulations are above 14.8% which is within the range specified for ceramic wall tiles (WA □ 10%).

While there was a reduction in water absorption with progressive sintering temperature for all the tile formulation at the different sintering temperatures (1100^{o}C,

1140°C and 1160°C), it was obvious that a substantial decrease in water absorption occurred at 1140°C and continued to the final temperature of 1160°C.

4.8 Flexural Strength

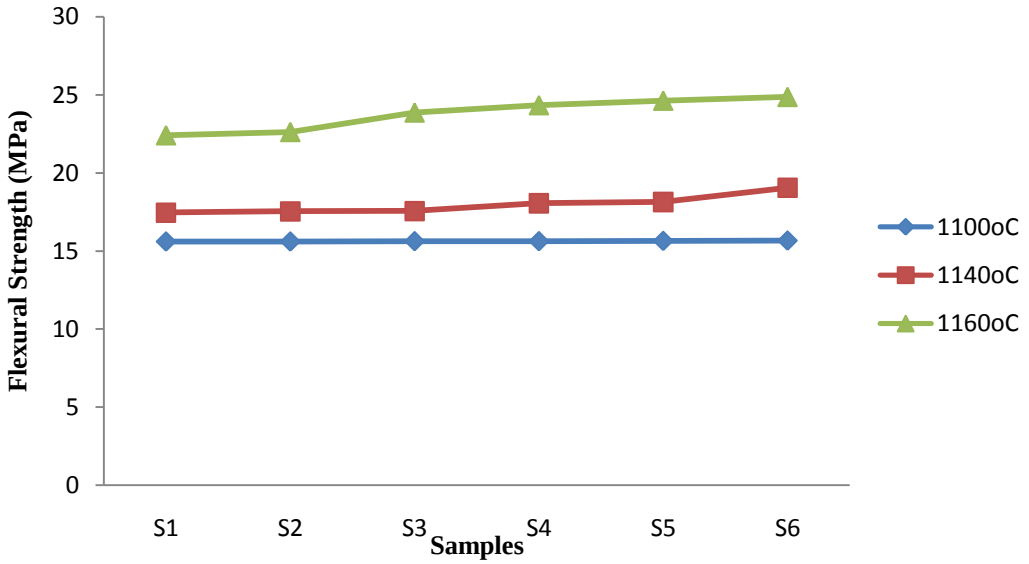


Figure 7: Variation in Flexural Strength with Temperature

The flexural strength of the tile bodies as a function of the formulation type and sintering temperature can be seen in figure 7. In figure 7, it can be observed that the flexural strength increased with sintering temperature. The temperature at which the flexural strength was maximal was at 1160°C for the formulations. From the results, it can be seen that the flexural strength increased with increasing densification and decreasing water absorption as shown in figures 1 and 2 respectively. The mechanical strength as indicated isalso strongly dependent on the microstructure especially defects such as pores and cracks. Together with water absorption, the flexural strength has an important role in the classification of tiles.

The flexural strength of the tile formulations fired at 1100°C, 1140°C and 1160°C (figure 7.) was between 15.60-24.87 MPa. The formulations sintered at 1160°C are within the

range required by ISO 13006 for the manufacture of ceramic tiles. This means that it should be possible to obtain tile products that fit the high quality standards from these formulations as the fired bodies presented flexural strength values in conformity to wall tiles for firing temperatures from 1100^{o}C.

The strength of the specimens fired at 1160^{o}C was comparatively higher than those sintered at 1100^{o}C and 1140^{o}C due to better densification. There was no marked difference in strength values between the tile samples sintered at 1100^{o}C and 1140^{o}C with temperature.

The effect of temperature was to increase the flexural strength by means of densification which is attributed to the formation of calcium based crystalline phases (calcium silicate) of higher mechanical strength (Sousa andHolanda, 2005).

4.9 Modulus of Rapture

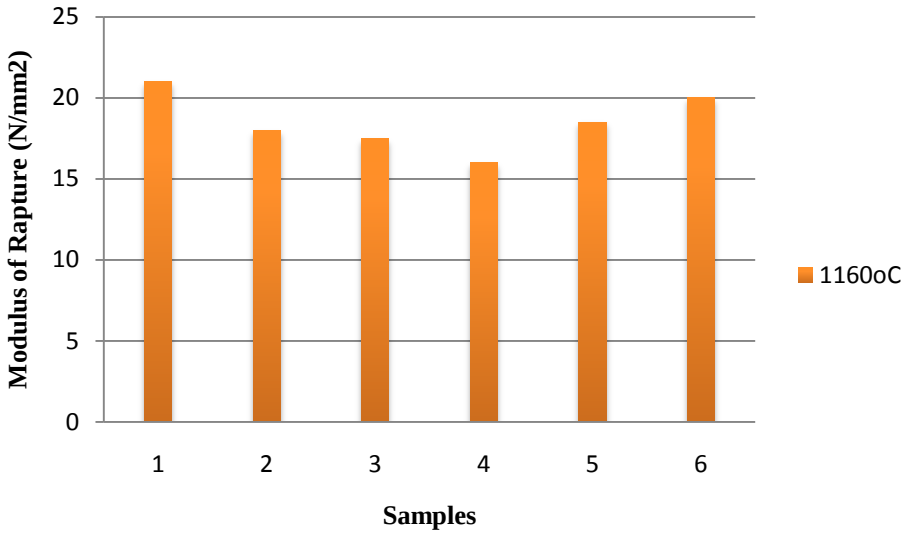


Figure 8: Modulus of Rapture Results of Tile Specimens Sintered at 1160^{o}C.

The values of the modulus of rapture of the experimental tiles samples as shown in figure 8 and are specified according to ISO 13006. The results obtained range from 21.52 to 17.34 N/mm^2 and satisfies the minimum requirement of the modulus of rapture of ceramic tiles.

In determining the modulus of rapture specimens, the thickness of the specimens was taken into account. Both water absorption and modulus of rapture are the two important criteria that determine the limits of the breaking strength and modulus of rapture (Hanan and El Nouhy, 2013). Regardless of their application, ceramic bodies are expected to satisfy the two most important requirements of water absorption and flexural strength.

4.10 XRD Investigation of the Fired Samples

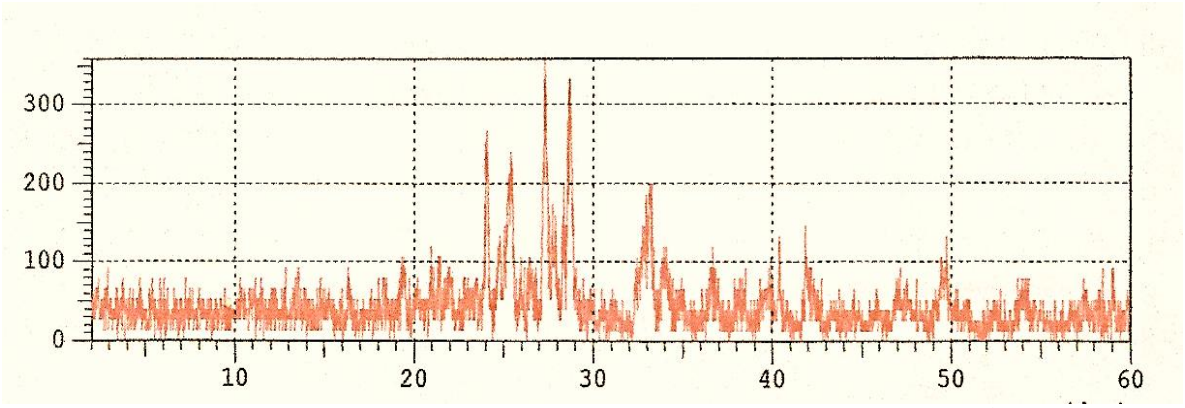


Figure 9: X-ray Diffraction Pattern for Formulation S1 Sintered at 1160^{o}C

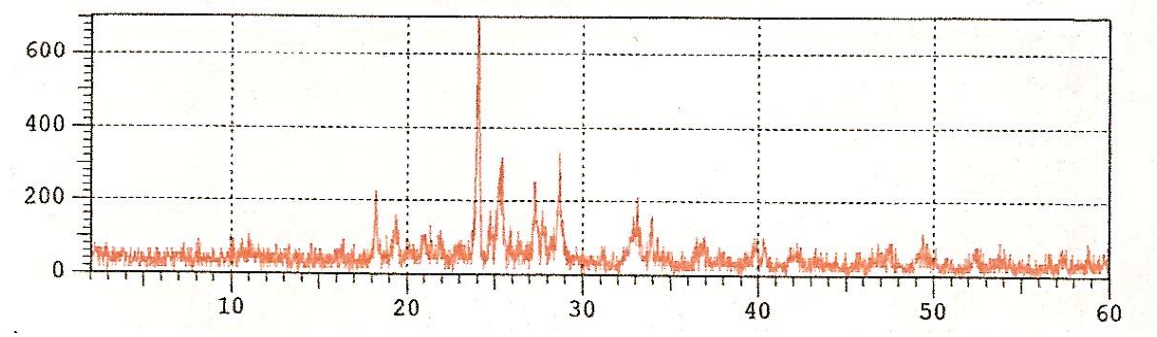


Figure 10: X-ray Diffraction pattern for Formulation S2 Sintered at 1160^{o}C

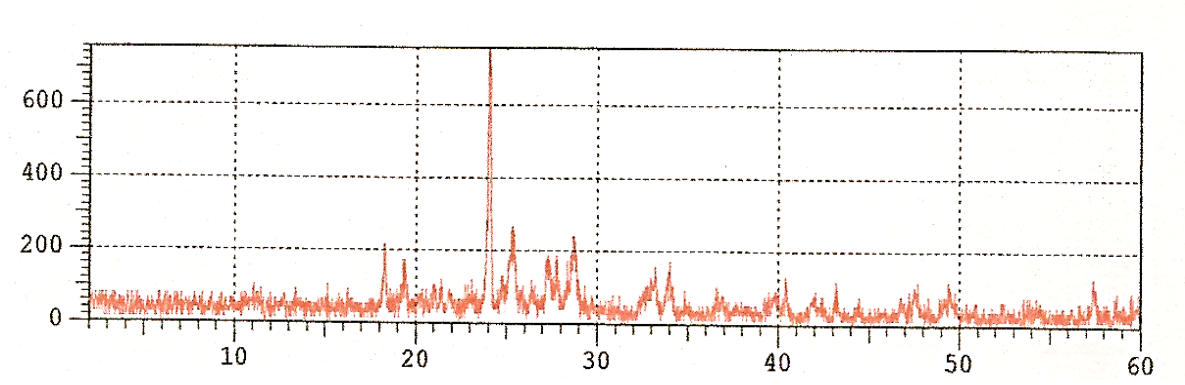


Figure 11: X-ray Diffraction pattern for Formulation S3 Sintered at 1160^{o}C

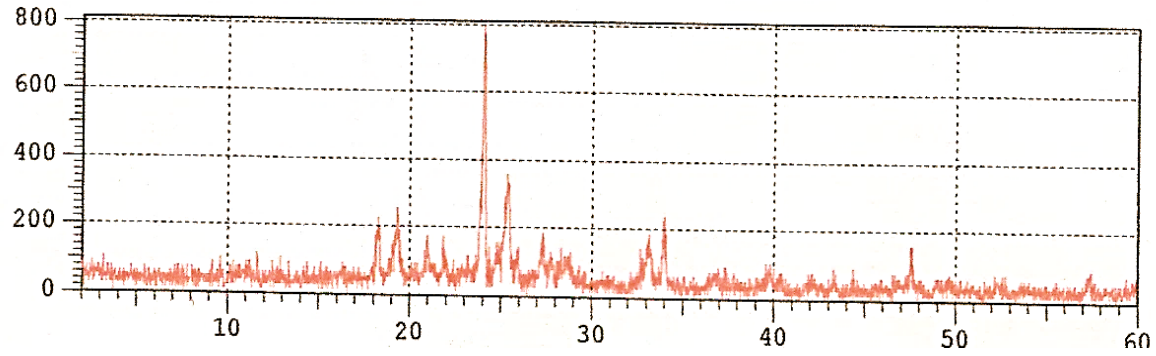


Figure 12: X-rayD pattern for Formulation S4 Sintered at 1160°C.

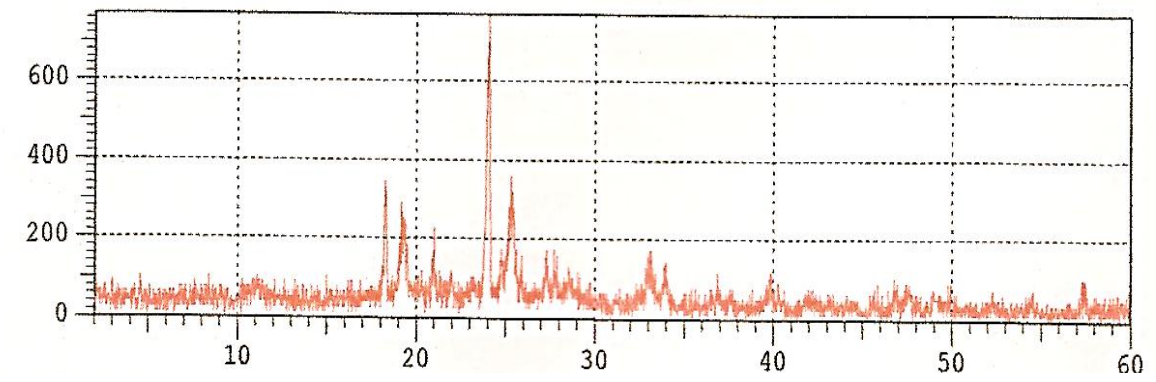


Figure 13: X-ray Diffraction pattern for Formulation S5 Sintered at 1160°C.

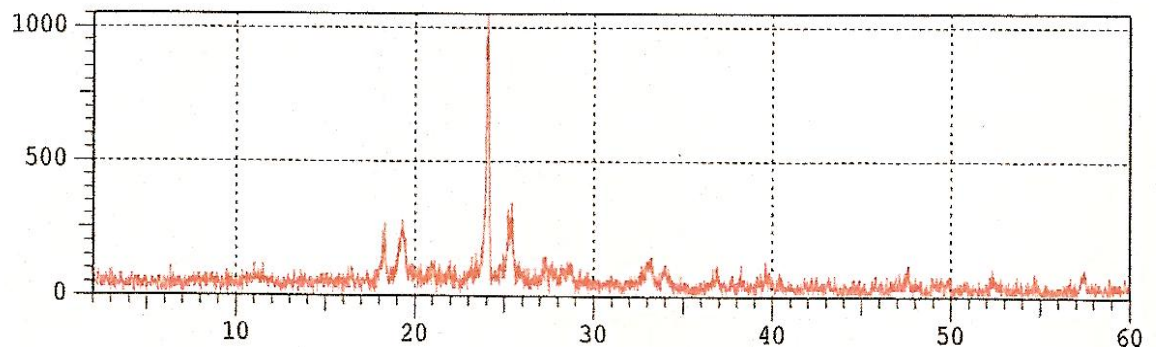


Figure 14: X-ray Diffraction pattern for Formulation S6 Sintered at 1160°C.

To identify the mineralogical phases for the sintered tile formulations X-ray diffraction was used. Figures9, 10, 11, 12, 13 and 14 shows the X–ray diffraction patterns of

the formulations S1, S2, S3, S4, S5 and S6 sintered at 1160^{o}C. The phase transformation at 1100^{o}C and 1140^{o}C are found in the appendices.

Comparing the XRD patterns that were conducted on the six experimental tiles sintered at 1160^{o}C at both the edges and core of each tile, the results showed limited differences regarding the formed phases pertaining to edge and core respectively. The XRD patterns for both the edges and cores of the tested specimens are shown in Figures9, 10, 11, 12, 13 and 14. This reflects that during the sintering, the temperature was uniform.

The XRD analyses in this study indicated that certain mineralogical phases like Antigorite ($Mg_3Si_2O_5.OH_4$), Anorthite ($CaAl_2Si_2O_8$), Kaolinite ($Al_2Si_2O_5.OH_4$), Halloysite ($Al_2Si_2O_5.OH_4$), Xonolite ($Ca6Si_6O_{17}.OH_2$), Orthoclase ($KAlSi_3O_8$), Xonotlite ($Ca6SiO_{17}(OH)_2$), Wollastonite ($CaSiO_3$) and Talc ($Mg_3Si_4O_{10}.OH_2$) were formed during the sintering. These minerals exhibited various peaks of siliceous phasescombined with other elements which according to Woods, (1991) influence a reduction in shrinkage, thermal expansion and a higher flexural strength due to verification at a lower firing temperature.

In view of the fact that the ceramic systems are non-equilibrium systems, different phases inevitably develop, with each phase having its own structure, composition, and properties as the sintering temperature varies both in time duration and degree (El Nouhy, 2013). This assertion by El Nouhy, (2013) is evident in the XRD analysis of the experimental tile specimens that revealed various phase transformations at different sintering temperatures (see appendices 6 - 72). Consequently, it is difficult to predict the exact final phases of the end products.

4.12 Scanning Electron Microscopy (SEM)

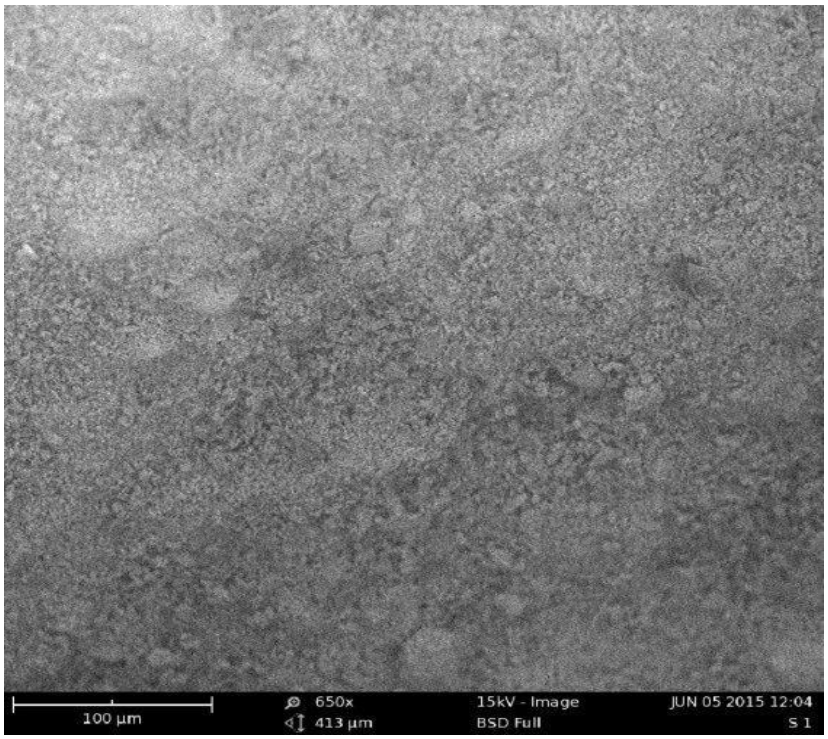


Figure 15: SEM Micrograph of Sample 1 Sintered at 1160^{o}C

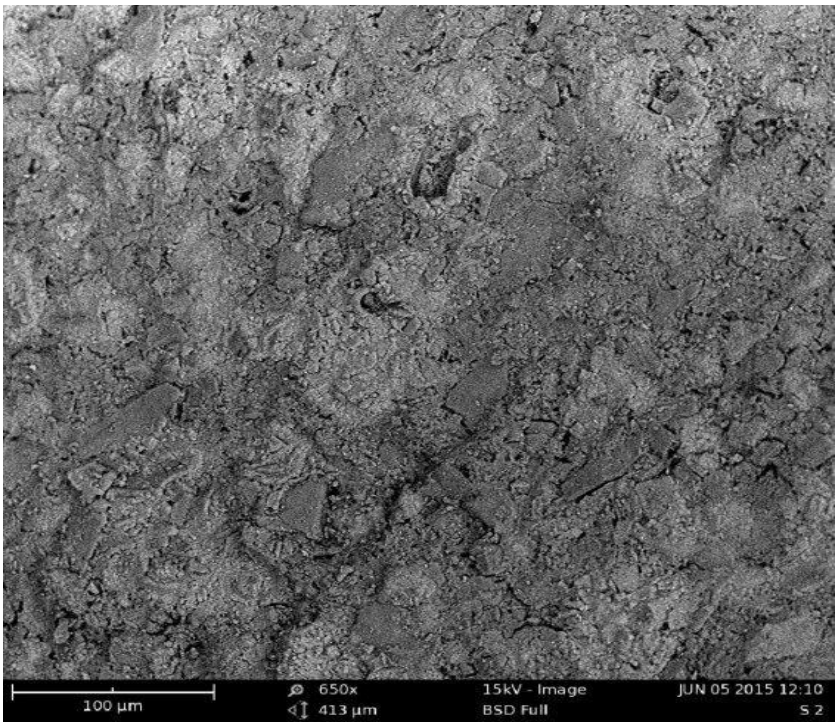


Figure 16: SEM Micrograph of Sample 1 Sintered at 1160^{o}C

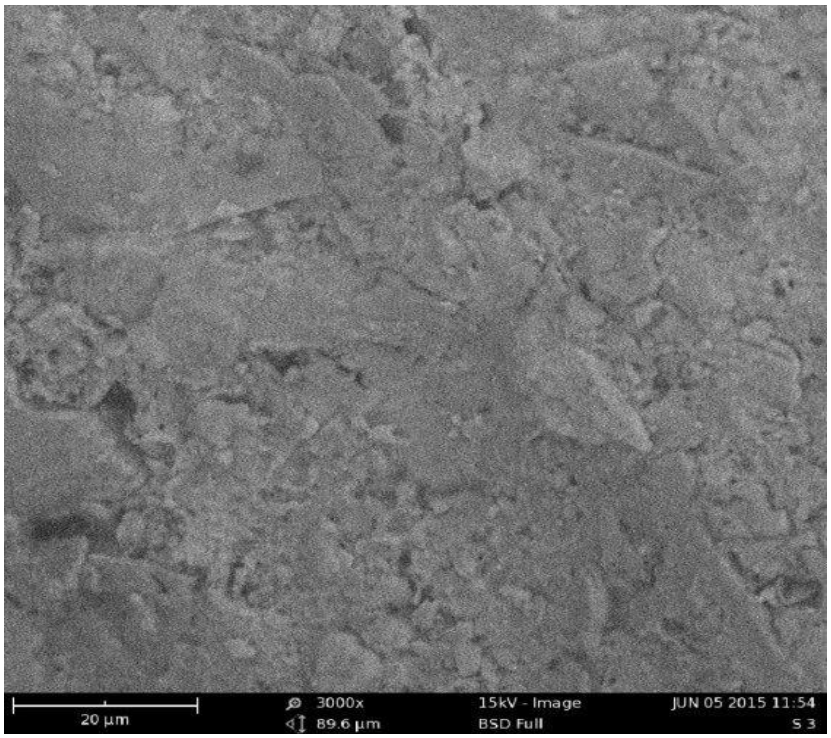


Figure 17: SEM Micrograph of Sample 1 Sintered at 1160^{o}C

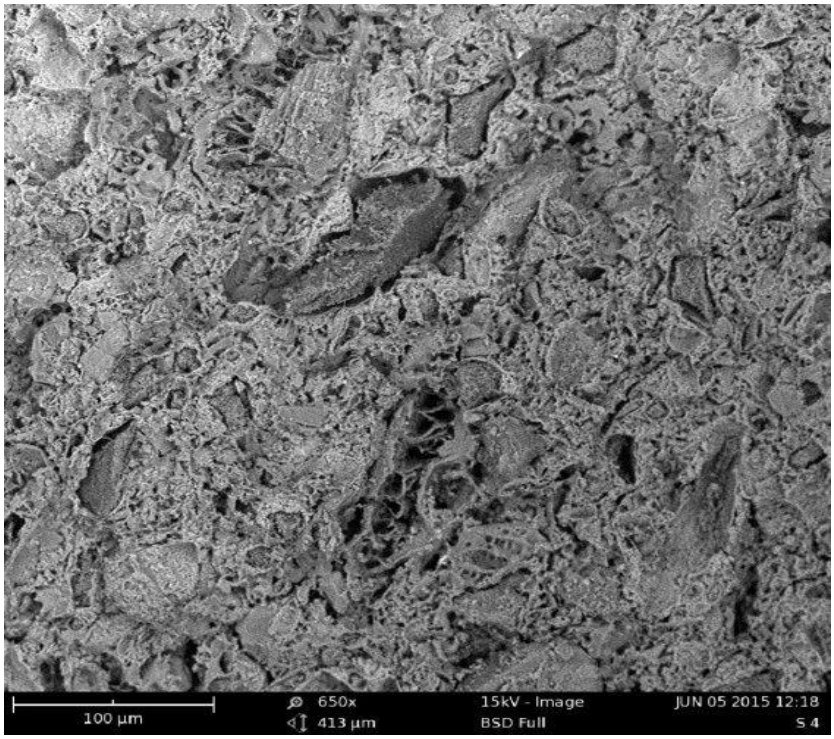


Figure 18: SEM Micrograph of Sample 1 Sintered at 1160^{o}C

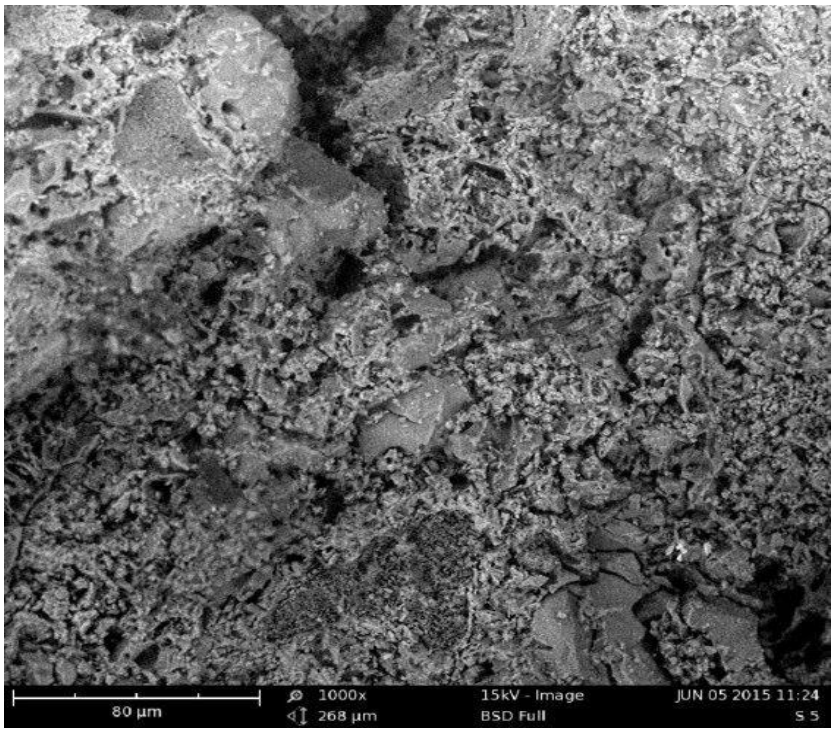


Figure 19: SEM Micrograph of Sample 1 Sintered at 1160^{o}C

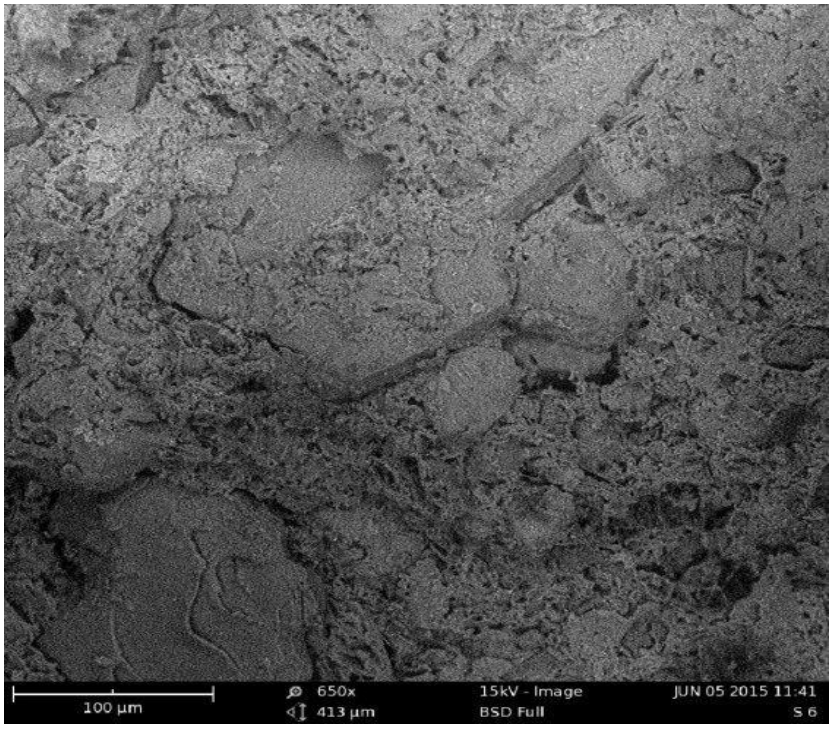


Figure 20: SEM Micrograph of Sample 1 Sintered at 1160^{o}C

The morphological characterization of the formulations was performed by scanning electron microscopy on fracture surfaces of the samples. Figures 15, 16, 17, 18, 19 and 20 shows the micrographs of samples sintered at 1160^{o}C. At the optimum temperature of 1160^{o}C shows the typical sequence of enhanced densification.

Examination of the microstructure confirmed that with increased rice husk addition, the samples revealed a dominance of high porosity connected with dense zones. Samples 4, 5 and 6 presented a higher feature of porosity than samples1, 2 and 3 in the form of sponge-like pores as a result of the decomposition of the $CaCO_{3.}$

The large amount of porosity however, did not affect the mechanical properties (Figure 7) as well as the visual aspects of the tiles. This confirms Alcântara et al (2007) assertion that raw materials for ceramics containing $CaCO_3$and alumina silicates are prompt to form crystalline phases at relatively low temperatures (from 950^{o}C – 1000^{o}C). As a consequence, even with high porosity, the experimental tiles presented good strength.

4.11 Physical Characteristics of the Formulated Tile Samples and Comparison with Commercial Tiles

In order to assess the quality of the tiles prepared from the formulated bodies for this study, it was necessary to make a comparative study with the commercially available tiles. It should be noted that the commercially available tiles were collected in the fired and finished form and as such it was not possible to compare the firing shrinkage behaviour of the prepared tiles with the commercial tiles.

In view of the unavailability of data regarding the raw materials used (constituents and nature), the manufacturing processes, and the curing methods employed, the mechanical

properties (namely flexural strength) and physical properties (namely water absorption and apparent porosity) were the essential criteria employed for the evaluation.

Table 12: Physical and Mechanical Properties of the Prepared Tiles and Comparison with Commercial Tiles.

Characteristics	1100°C	1140°C	1160°C	Tile A (VIAENY)	Tile B (PNT)
Water Absorption (%)	11.82	12.45	13.72	12.20	14.20
Bulk Density (gm/cc)	1.75	1.92	1.98	1.68	1.69
Apparent Porosity (%)	14.38	24.74	3.47	20.68	20.71
Apparent Specific Gravity	2.53	2.53	2.63	2.12	2.14

Results of the various comparative property tests are reported in Table 12.The numerical values of water absorption of all the compositions are within the range of all the tiles (15-20%). The apparent porosity of the specimens was higher than commercial tiles while the bulk density of the formulated tiles compositions are in the same range with a small variation.

The apparent porosity and density of the specimens were higher than those observed for the commercial tiles in spite of the fact that increasing sintering temperature in most cases contributes to a decrease in porosity as a result of densification of the specimens. This observed density trend can be understood by the formation of anorthite ($CaAl_2Si_2O_8$) in the experimental formulations during the sintering. Anorthite provides a mechanical strength to the body and its presence according to Khalil et al, (1989) compensates the higher porosity.

Measurement of water absorption, bulk density, porosity,and specific gravity are the tools for determining the degree of maturity or vitrification of a ceramic body (Jahan, et al,

2008). It is evident from Table 12 that the numerical values of water absorption of all the compositions sintered at 1160^{o}Care withinthe range of that of walltiles.

Raw materials for ceramic formulations containing $CaCO_3$and alumina silicates (Al_2O_3 SiO_2) usually form crystalline phases at relatively low temperatures (from 950^{o}C – 1000^{o}C) that promote densification. Both admixtures according to Vodova, Sokolar and Hroudova, 2014 can be described as melting agent at accelerates the sintering at lower temperatures. Notwithstanding the high porosity, the experimental tiles presented good strength.The value of the flexural strength(Figure 7) are within the range specified by ISO 13006 and justifies the good quality of the experimental tiles.

Taking into cognizance the physical properties, comparing with the locally available tiles, the experimental tiles sintered to 1160^{o}C satisfies the desired qualities of ceramic wall tiles.So in view of the physical properties, comparing with the commercially available available tiles, the prepared tiles sintered to 1160^{o}Csatisfies the desired quality of ceramic wall tiles.

CHAPTER FIVE

5.0 SUMMARY, CONCLUSION AND RECOMMENDATIONS

5.1 Introduction

In the light of recycling of waste as a value added product, this study investigated the potential gains of incorporating rice husk ash (RHA) as a raw material in ceramic body formulations for the production of wall tiles. This chapter is the embodiment of the observations made on the various experiments carried out in the course of the studytowards achieving the aim and objectives of the study. Recommendations for further research and general recommendations are also highlighted in this chapter.

5.2 Summary

The first stage of this investigation was directed towards the collection and various beneficiation processes of the raw materials used for the study. The rice husk, quartz, kaolin and Bomo clay used for this study went through various beneficiation processes. Through these processes, it was possible to wash out the mineral salts in the clays and other impurities like organic and carbonaceous matter. The process of soaking, sieving and levigation helped in washing out the mineral salts as well as the removal of the organic matters. Through the burning of the rice husk in the open air, it was possible to achieve a whitish rice husk ash with negligible amount of carbon traces.

All the raw materials were also characterized in terms of their chemical composition through X-ray fluorescence (XRF) while X-ray diffraction (XRD) was later used for the identification of the mineralogical phases resulting from the sintering processes of the

specimens. It was confirmed through the XRF analysis that the RHA was 82.14% rich in silica (SiO_2).

Six experimental body formulations named S1, S2, S3, S4, S5 and S6 were prepared varying the quantities of RHA and quartz. While the quartz was reduced in quantity the RHA was increased. Rectangular specimens measuring 14.7 x 5.5 x 0.7cm^3prepared by uniaxial pressing technique in a plaster mould at 15MPa and dried at room temperature. The sintering was carried out in an electric kiln at temperatures varying from 1100^oC to 1160^oC.

The densification behaviour of the tiles was described in terms of linear shrinkage, water absorption and flexural strength. Linear shrinkage values upon drying and firing were evaluated from the variation of the length of the rectangular specimens. Water absorption values were determined from weight difference between the fired and water saturated samples immersed in rain water for five hours. The flexural strength was determined by three point bending test.

To assess the quality of the tile bodies, it was necessary to compare them with commercially available tiles. Because the commercially available tiles were in fired and finished form, it was only possible to determine and compare the physical properties of the commercial tiles with the experimental tiles. The results are documented in the text

5.3 Conclusion

Based on the results of the investigation carried out in the course of this study, the following conclusions were drawn:

i. The rice husk ash (RHA) generated from the burning of rice husk which is an agricultural waste can be utilizedin the basic mixture used for the manufacture of ceramic wall tiles by adding it as a partial or total replacement of the raw material quartz. This means that it should be possible to obtain quality tile products that fit the standards similar to those of wall tiles.

ii. The firing curves which correlates to the water absorption (LA), linear shrinkage (LS) and flexural strength (FS) with firing temperatures of the tile bodies formulated with rice husk ash can be seen in Figures 1, 2, and 3. These curves show that the linear shrinkage and water absorption decreased with increased sintering temperature. Such result was expected because of the densification of the tile bodies.

iii. The flexural strength of the sintered tile formulations as a function of sintering temperature can be seen in Figure 3. The increased flexural strength in this case is attributed to increased densification an as a result of the vitreous phase sintering process.

iv. The incorporation of the rice husk ash had negligible effect on the properties during the entire production processes anticipating that there will be no costly modification if used in the industrial production line. This agricultural waste can be used as a product with higher added value with respect to environmental and economic issues. It is therefore an attractive alternative and renewable source of ceramic raw material.

v. Beyond reducing the environmental impact of deposition of waste and lowering the cost of production of wall tiles, it is worth noting that rice husk ask contain no hazardous chemicals. It is a true natural material that is environmentally safe to recycle.

vi. It is feasible to process the formulation by pressing without the addition of any additives to improve plasticity. The shrinkage, waste absorption and mechanical strength tests indicated that the best firing cycle to be used with the alternative raw materials is at 1160^{o}C.

5.4 Recommendations

i. It is recommended that rice husk ask should be explored as a principal raw materials for ceramic tile production.This recommendation is based on the fact that the rice husk ash used in the production system for this study have shown to be suitable for ceramic products that are thermally and mechanically stable and exhibit good chemical stability.

ii. It is recommended that communities where rice production is going on should be encouraged to set up business for the production of rice husk ash with a view of utilizing the rice husk that is dumped as waste. The reuse approach is environmentally appealing and can contribute to the provision of low cost raw material, conservation of natural resources and the management of a pollutant waste. The use of the RHA as raw materials for industries will also helpin boosting the farm economy and rural development.

iii. The utilization of rice husk ash for advanced applications should be subjected to further studies because of its intrinsic nature.

iv. The differential (DTA) and gravimetric (TG) thermal analysis of the formulations should should be studied in order to identify the probable water consuming reactions and decomposition of carbonaceous materials with a view to determining their effect on the formulations.

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APPENDICES

Appendix 1:XRF Analysis of Bomo Clay

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

ANALYSED: 12/08/2013 4:37:07 PM

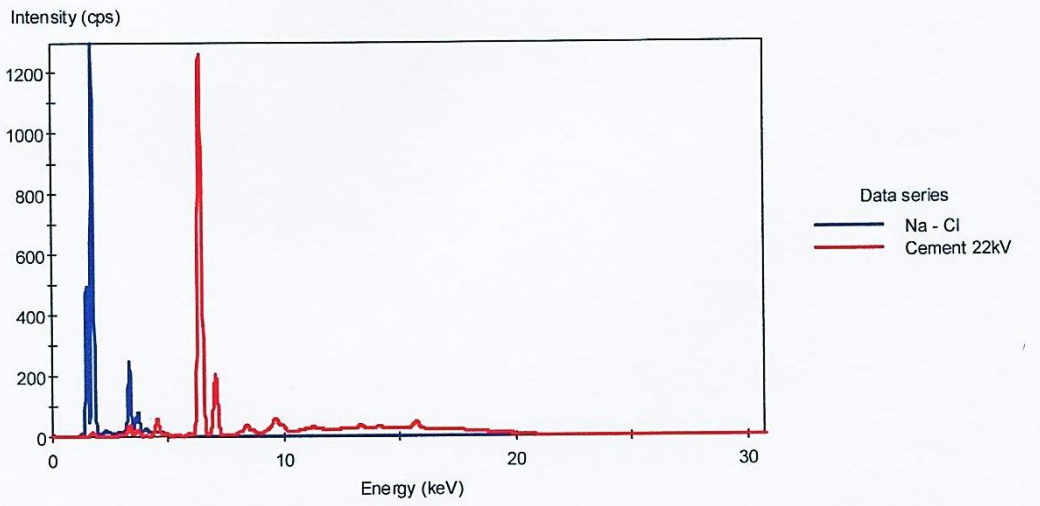
SAMPLE: BOMO CLAY

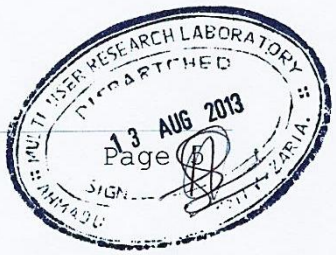
Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	0.090 Wt %
MgO	0.866 Wt %
Al2O3	19.569 Wt %
SiO2	67.336 Wt %
P2O5	0.000 Wt %
SO3	0.411 Wt %
Cl	0.017 Wt %
K2O	2.241 Wt %
CaO	1.186 Wt %
TiO2	1.750 Wt %
Cr2O3	0.011 Wt %
Mn2O3	0.060 Wt %
Fe2O3	6.408 Wt %
ZnO	0.025 Wt %
SrO	0.028 Wt %

Spectra for all conditions





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Page 1

Appendix 2:XRF Analysis of Kankara Kaolin.

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

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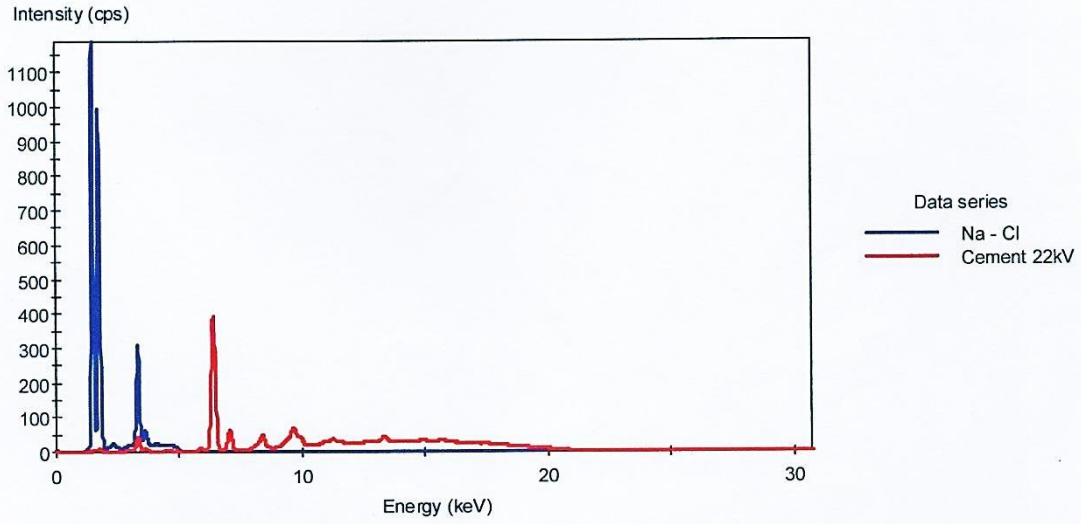
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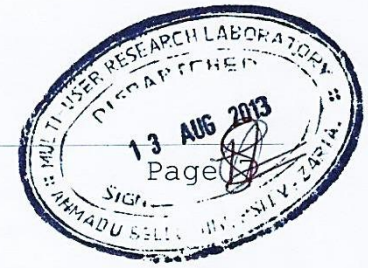
Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	0.050 Wt %
MgO	0.759 Wt %
Al2O3	46.441 Wt %
SiO2	48.223 Wt %
P2O5	0.000 Wt %
SO3	0.476 Wt %
Cl	0.004 Wt %
K2O	2.146 Wt %
CaO	0.227 Wt %
TiO2	0.163 Wt %
Cr2O3	0.000 Wt %
Mn2O3	0.058 Wt %
Fe2O3	1.448 Wt %
ZnO	0.003 Wt %
SrO	0.003 Wt %

Spectra for all conditions





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Appendix 3:XRF Analysis of BomoQuartz

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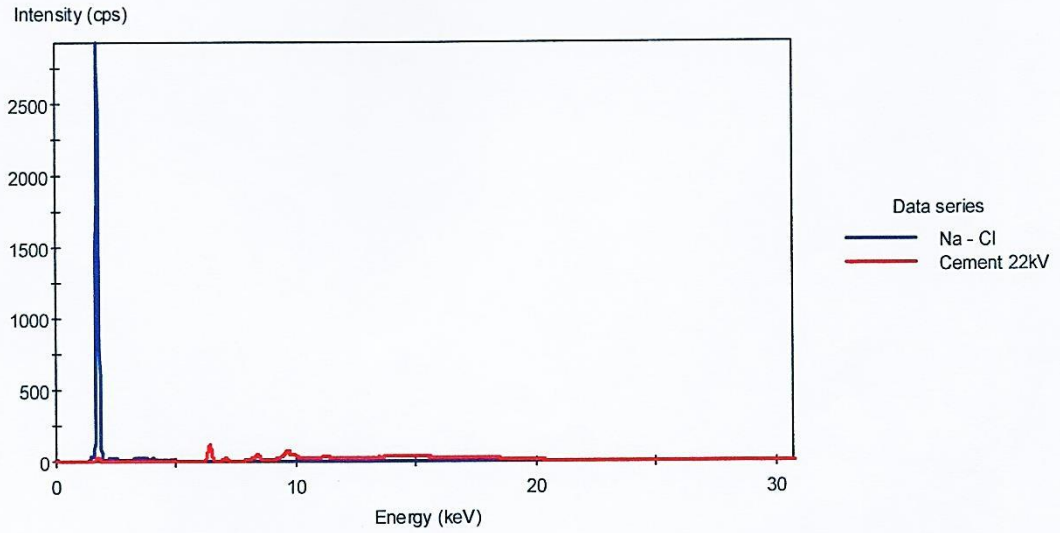
SAMPLE: QUARTZ

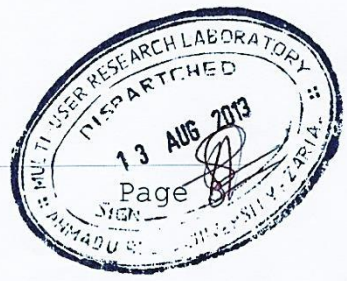
Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	0.000 Wt %
MgO	0.087 Wt %
Al2O3	0.497 Wt %
SiO2	98.061 Wt %
P2O5	0.000 Wt %
SO3	0.724 Wt %
Cl	0.077 Wt %
K2O	0.029 Wt %
CaO	0.119 Wt %
TiO2	0.014 Wt %
Cr2O3	0.000 Wt %
Mn2O3	0.007 Wt %
Fe2O3	0.384 Wt %
ZnO	0.000 Wt %
SrO	0.000 Wt %

Spectra for all conditions





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Page

Appendix 4:XRF Analysis of Zaria Rice Husk Ash

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

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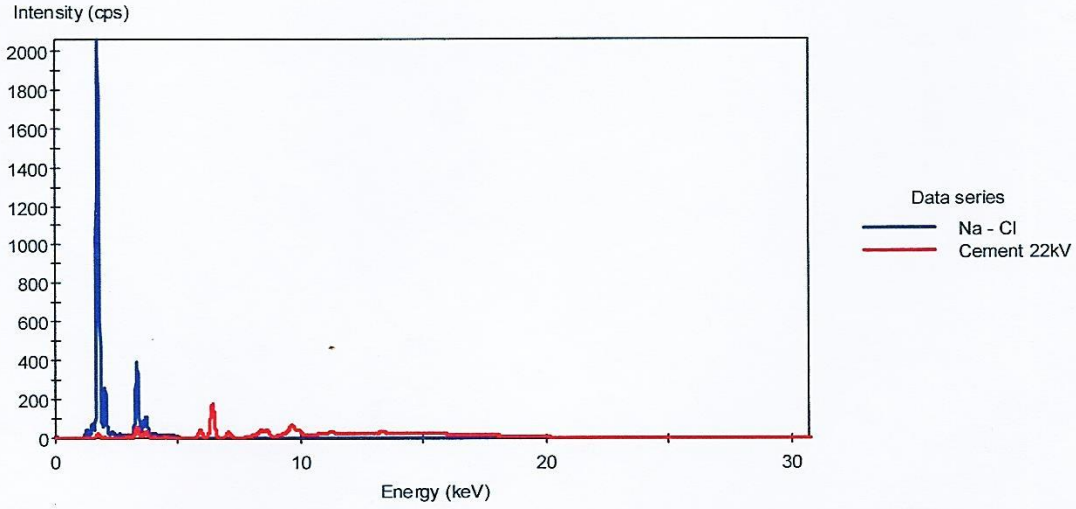
SAMPLE: RICE HUSK ASH

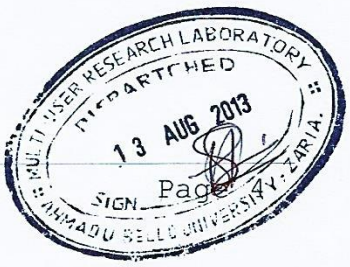
Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	0.350 Wt %
MgO	2.348 Wt %
Al2O3	1.311 Wt %
SiO2	82.145 Wt %
P2O5	6.890 Wt %
SO3	0.728 Wt %
Cl	0.077 Wt %
K2O	3.195 Wt %
CaO	1.697 Wt %
TiO2	0.161 Wt %
Cr2O3	0.002 Wt %
Mn2O3	0.294 Wt %
Fe2O3	0.745 Wt %
ZnO	0.051 Wt %
SrO	0.007 Wt %

Spectra for all conditions





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Appendix 5:XRF Analysis of IkepeshiWhiting

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

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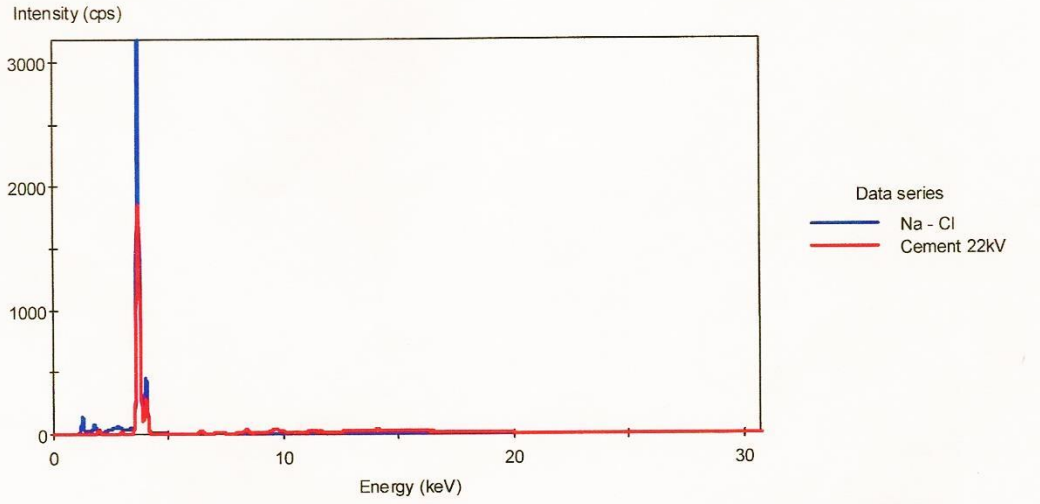
SAMPLE: WHITING

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	0.000 Wt %
MgO	21.498 Wt %
Al2O3	1.416 Wt %
SiO2	4.462 Wt %
P2O5	0.063 Wt %
SO3	0.332 Wt %
Cl	0.236 Wt %
K2O	0.118 Wt %
CaO	71.487 Wt %
TiO2	0.018 Wt %
Cr2O3	0.003 Wt %
Mn2O3	0.011 Wt %
Fe2O3	0.282 Wt %
ZnO	0.000 Wt %
SrO	0.074 Wt %

Spectra for all conditions



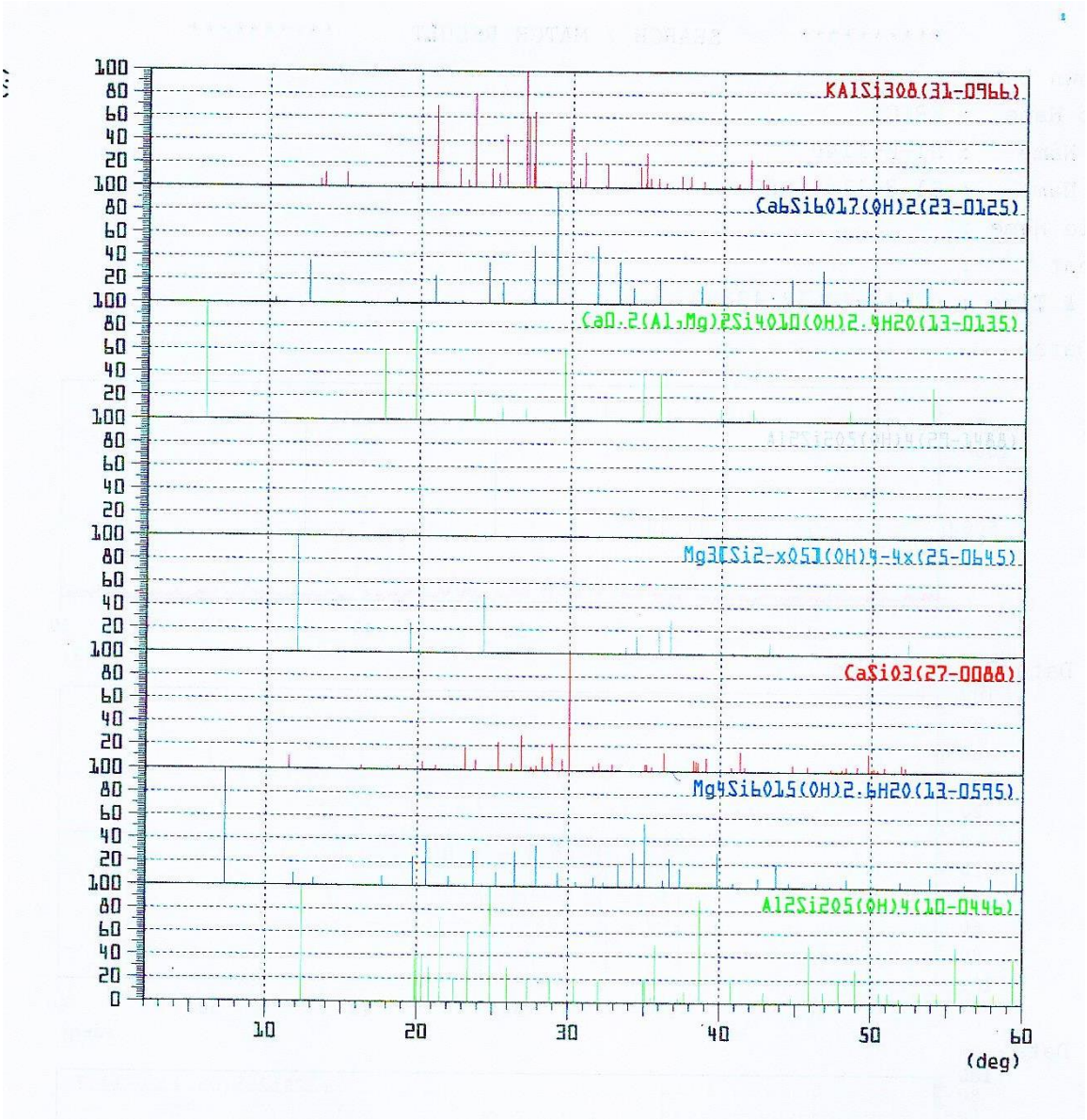


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Appendix 6: XRD Analysis of Sample 1 at 1140^{o}C.



Appendix 7: XRD Analysis of Sample 1 at 1140^{o}C (continuation).



Appendix 8: XRD Analysis of Sample 1 at 1140^{o}C (continuation).

********** SEARCH / MATCH RESULT **********

Group Name : ERIC
Data Name : S1-@-1140
File Name : S1-@-1140.PSE
Sample Name :
Comment :

<Entry Card>

No.	Card	Chemical Formula / Chemical Name (Mineral Name)	S	L / Dx	d / WT%	I / S.G.	R
1	29-1487	Al2Si2O5(OH)4	0.073	0.667(4/ 7)	0.794	0.589	0.312
		Aluminum Silicate Hydroxide (Halloysite-7	-----			-------	
2	31-0794	(Mg,Al)9(Si,Al)8O20(OH)10.4H2O	0.188	0.800(12/17)	0.671	0.532	0.285
		Magnesium Aluminum Silicate Hydroxide Hydr	-----			-------	
3	13-0259	Na0.3(Al,Mg)2Si4O10(OH)2.xH2O	0.465	0.571(4/ 7)	0.530	0.641	0.194
		Sodium Magnesium Aluminum Silicate Hydroxi	-----			-------	
4	35-0463	LiAlSi4O10	0.761	0.524(22/42)	0.727	0.479	0.182
		Lithium Aluminum Silicate (Petalite-1\ITM	-----			-------	
5	21-0963	Mg3Si2O5(OH)4	0.123	0.583(14/27)	0.647	0.467	0.176
		Magnesium Silicate Hydroxide (Antigorite-	-----			-------	
6	13-0558	Mg3Si4O10(OH)2	0.186	0.600(12/30)	0.557	0.427	0.143
		Magnesium Silicate Hydroxide (Talc-2\ITM\	-----			-------	
7	31-0966	KAlSi3O8	0.198	0.405(17/42)	0.683	0.496	0.137
		Potassium Aluminum Silicate (Orthoclase)	-----			-------	
8	23-0125	Ca6Si6O17(OH)2	0.236	0.464(13/39)	0.670	0.374	0.116
		Calcium Silicate Hydrate (Xonotlite, syn	-----			-------	
9	13-0135	Ca0.2(Al,Mg)2Si4O10(OH)2.4H2O	0.123	0.538(7/17)	0.595	0.357	0.115
		Calcium Magnesium Aluminum Silicate Hydrox	-----			-------	
10	29-1488	Al2Si2O5(OH)4	0.089	0.500(7/21)	0.644	0.342	0.110
		Aluminum Silicate Hydroxide (Kaolinite-1\	-----			-------	
11	25-0645	Mg3[Si2-xO5](OH)4-4x	0.331	0.714(10/18)	0.695	0.214	0.106
		Magnesium Silicate Hydroxide (Chrysotile	-----			-------	
12	27-0088	CaSiO3	0.620	0.571(24/42)	0.603	0.295	0.102
		Calcium Silicate (Wollastonite-2\ITM\RG)	-----			-------	
13	13-0595	Mg4Si6O15(OH)2.6H2O	0.247	0.611(22/39)	0.612	0.269	0.101
		Magnesium Silicate Hydroxide Hydrate (Sep	-----			-------	
14	10-0446	Al2Si2O5(OH)4	0.186	0.486(18/39)	0.579	0.357	0.101
		Aluminum Silicate Hydroxide (Dickite-2\IT	-----			-------	

Appendix 9: XRD Analysis of Sample 1 at 1140^{o}C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S1-@-1140

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated (Counts)
1	15	24.0900	3.69130	100	0.14650	138	1217
2	26	28.8000	3.09743	40	0.12000	55	331
3	25	28.6600	3.11224	25	0.14000	35	250

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated (Counts)
1	2.1150	41.73754	3	0.11000	4	34
2	2.5150	35.10018	3	0.15000	4	55
3	5.5700	15.85362	3	0.12000	4	45
4	7.5850	11.64593	4	0.05000	6	26
5	7.9150	11.16110	3	0.07000	4	17
6	9.2600	9.54277	3	0.08000	4	36
7	12.1550	7.27566	3	0.07000	4	24
8	13.6450	6.48434	3	0.05000	4	23
9	16.4233	5.39312	3	0.11330	4	54
10	18.3183	4.83926	12	0.18330	16	152
11	18.5133	4.78872	6	0.06670	8	37
12	19.4100	4.56947	4	0.10000	5	43
13	21.4216	4.14470	8	0.18330	11	117
14	21.8933	4.05645	3	0.09330	4	39
15	24.0900	3.69130	100	0.14650	138	1217
16	24.7866	3.58912	4	0.05330	5	22
17	25.2950	3.51812	8	0.11000	11	89
18	25.5400	3.48492	7	0.07000	9	42
19	25.8216	3.44755	4	0.06330	5	22
20	26.4400	3.36830	8	0.12000	11	80
21	26.5600	3.35336	7	0.12000	10	62
22	27.3900	3.25360	5	0.12000	7	53
23	27.9100	3.19415	3	0.10000	4	33
24	28.5200	3.12720	8	0.08000	11	56
25	28.6600	3.11224	25	0.14000	35	250
26	28.8000	3.09743	40	0.12000	55	331
27	28.9600	3.08068	8	0.09000	11	85
28	33.1000	2.70421	4	0.08000	6	38
29	33.8150	2.64865	4	0.13000	5	53
30	34.0366	2.63191	11	0.15330	15	117
31	34.2366	2.61699	5	0.11330	7	44
32	34.4133	2.60396	4	0.06670	5	20
33	34.8600	2.57161	6	0.16000	8	73
34	35.0000	2.56164	3	0.04000	4	14
35	36.7033	2.44658	5	0.08670	7	54
36	36.8800	2.43526	4	0.08000	6	38
37	37.8000	2.37808	3	0.08000	4	39
38	40.3860	2.23157	14	0.13200	19	161
39	41.8400	2.15732	4	0.20000	6	88
40	43.4000	2.08332	3	0.12000	4	30
41	44.5900	2.03044	3	0.10000	4	23
42	47.5650	1.91015	10	0.19000	14	155
43	47.8450	1.89963	3	0.05000	4	11
44	48.0000	1.89385	3	0.04000	4	18
45	48.3500	1.88096	3	0.06000	4	22
46	49.4216	1.84265	12	0.14330	17	140
47	49.6586	1.83441	7	0.13070	9	67
48	49.9700	1.82371	4	0.06000	5	17
49	50.3000	1.81252	3	0.12000	4	33
50	57.1650	1.61008	3	0.07000	4	16

Appendix 10: XRD Analysis of Sample 1 at 1140^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	57.3333	1.60575	7	0.09330	9	65
52	59.7308	1.54690	5	0.11170	7	58

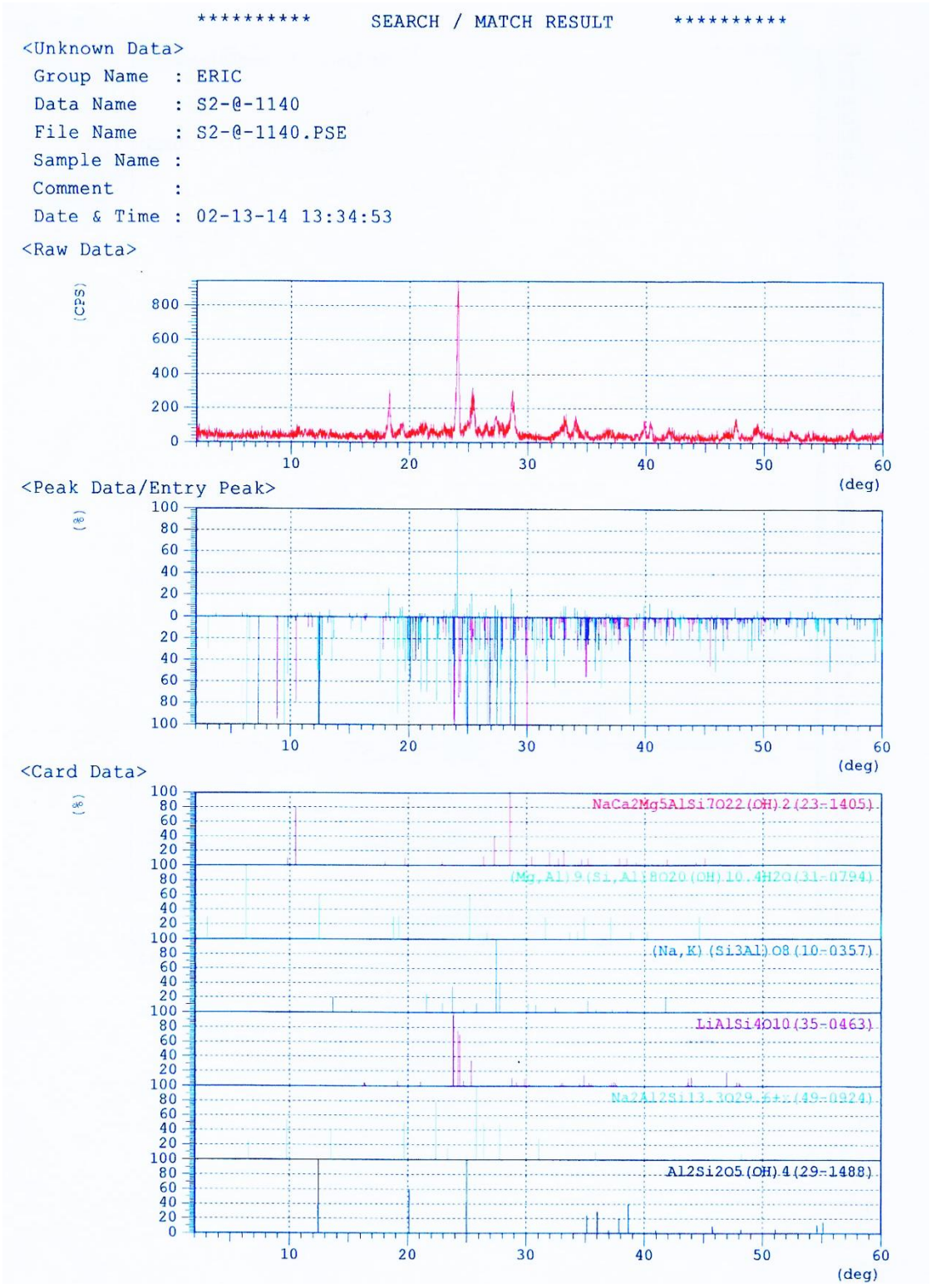
```
          ***  Basic Data Process  ***

 # Data Infomation
          Group                 : ERIC
          Data                  : S1-@-1140
          Sample Nmae           :
          Comment               :
          Date & Time           : 02-13-14 13:13:52

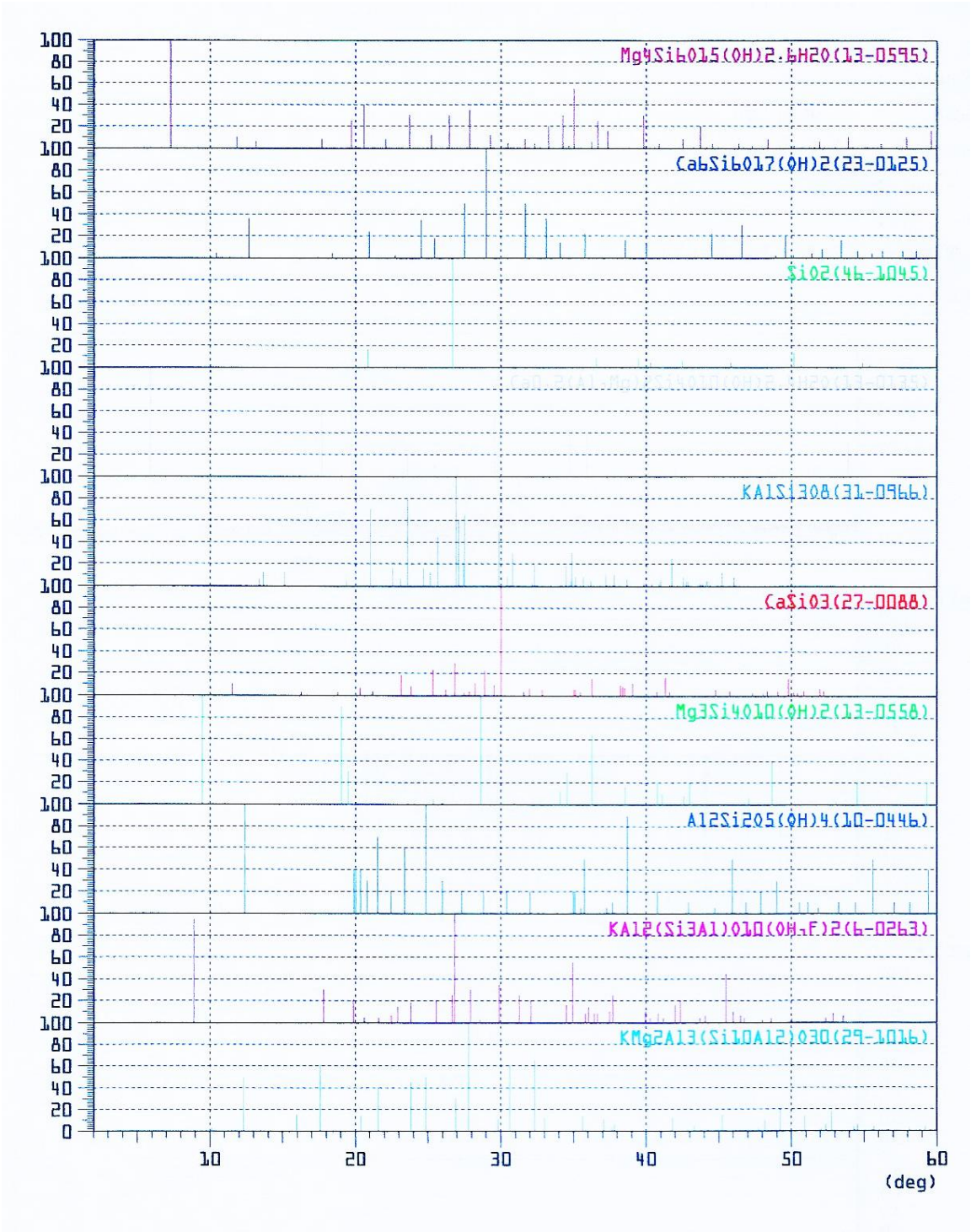
 # Measurement Condition
     X-ray tube
          target                : Cu
          voltage               : 40.0 (kV)
          current               : 30.0 (mA)
     Slits
          Auto Slit             : not Used
          divergence slit       :   1.00000 (deg)
          scatter slit          :   1.00000 (deg)
          receiving slit        :   0.30000(mm)
     Scanning
          drive axis            : Theta-2Theta
          scan range            :    2.0000 - 60.0000 (deg)
          scan mode             : Continuous Scan
          scan speed            :    8.0000 (deg/min)
          sampling pitch        :    0.0200 (deg)
          preset time           :    0.15 (sec)

 # Data Process Condition
     Smoothing                  [ AUTO ]
          smoothing points      : 9
     B.G.Subtruction            [ AUTO ]
          sampling points       : 9
          repeat times          : 30
     Ka1-a2 Separate            [ MANUAL ]
          Ka1 a2 ratio          : 50 (%)
     Peak Search                [ AUTO ]
          differential points   : 9
          FWHM threhold         : 0.050 (deg)
          intensity threhold    : 30 (par mil)
          FWHM ratio  (n-1)/n   : 2
     System error Correction    [ YES ]
     Precise peak Correction    [ NO ]
```

Appendix 11: XRD Analysis of Sample 2 at 1140^{o}C.



Appendix 12: XRD Analysis of Sample 2 at 1140^{o}C (continuation).



Appendix 13: XRD Analysis of Sample 2 at 1140^{o}C (continuation).

********** SEARCH / MATCH RESULT **********

Group Name : ERIC
Data Name : S2-@-1140
File Name : S2-@-1140.PSE
Sample Name :
Comment :

<Entry Card>

No.	Card	Chemical Formula / Chemical Name (Mineral Name)	S	L / Dx	d / WT%	I / S.G.	R
1	23-1405	NaCa2Mg5AlSi7O22(OH)2 Sodium Calcium Magnesium Aluminum Silicate	0.339	0.783(18/23) -----	0.772 -----	0.468 -------	0.283
2	31-0794	(Mg,Al)9(Si,Al)8O20(OH)10.4H2O Magnesium Aluminum Silicate Hydroxide Hydr	0.195	0.867(13/17) -----	0.711 -----	0.442 -------	0.272
3	10-0357	(Na,K)(Si3Al)O8 Potassium Sodium Aluminum Silicate (Sanid	0.215	0.765(13/17) -----	0.705 -----	0.482 -------	0.260
4	35-0463	LiAlSi4O10 Lithium Aluminum Silicate (Petalite-1\ITM	0.625	0.619(26/42) -----	0.758 -----	0.481 -------	0.226
5	49-0924	Na2Al2Si13.3O29.6+x Sodium Aluminum Silicate (Mordenite, syn	0.101	0.500(7/14) -----	0.692 -----	0.621 -------	0.215
6	29-1488	Al2Si2O5(OH)4 Aluminum Silicate Hydroxide (Kaolinite-1\	0.109	0.571(8/21) -----	0.726 -----	0.494 -------	0.205
7	13-0595	Mg4Si6O15(OH)2.6H2O Magnesium Silicate Hydroxide Hydrate (Sep	0.313	0.694(25/39) -----	0.655 -----	0.437 -------	0.199
8	23-0125	Ca6Si6O17(OH)2 Calcium Silicate Hydrate (Xonotlite, syn	0.293	0.714(20/39) -----	0.699 -----	0.389 -------	0.194
9	46-1045	SiO2 Silicon Oxide (Quartz, syn)	0.235	0.769(10/42) -----	0.728 -----	0.341 -------	0.191
10	13-0135	Ca0.2(Al,Mg)2Si4O10(OH)2.4H2O Calcium Magnesium Aluminum Silicate Hydrox	0.195	0.769(10/17) -----	0.700 -----	0.345 -------	0.186
11	31-0966	KAlSi3O8 Potassium Aluminum Silicate (Orthoclase)	0.236	0.643(27/42) -----	0.709 -----	0.398 -------	0.182
12	27-0088	CaSiO3 Calcium Silicate (Wollastonite-2\ITM\RG)	0.558	0.619(26/42) -----	0.686 -----	0.427 -------	0.181
13	13-0558	Mg3Si4O10(OH)2 Magnesium Silicate Hydroxide (Talc-2\ITM\	0.227	0.750(15/30) -----	0.517 -----	0.438 -------	0.170
14	10-0446	Al2Si2O5(OH)4 Aluminum Silicate Hydroxide (Dickite-2\IT	0.169	0.595(22/39) -----	0.673 -----	0.400 -------	0.160
15	6-0263	KAl2(Si3Al)O10(OH,F)2 Potassium Aluminum Silicate Hydroxide (Mu	0.386	0.619(26/42) -----	0.657 -----	0.390 -------	0.159
16	29-1016	KMg2Al3(Si10Al2)O30 Potassium Magnesium Aluminum Silicate (Os	0.225	0.606(20/33) -----	0.681 -----	0.380 -------	0.157

Appendix 14: XRD Analysis of Sample 2 at 1140°C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S2-@-1140

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	26	24.1138	3.68771	100	0.18700	90	785
2	15	18.3526	4.83029	26	0.19000	23	247
3	37	28.7051	3.10746	26	0.24000	23	281

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	3.9229	22.50558	3	0.04000	3	15
2	6.0749	14.53707	3	0.04000	3	13
3	6.4562	13.67935	3	0.06000	3	21
4	10.6944	8.26583	3	0.04000	3	22
5	11.3105	7.81693	3	0.12000	3	33
6	11.6386	7.59729	3	0.06000	3	23
7	11.8126	7.48577	3	0.05000	3	16
8	12.5241	7.06205	4	0.04000	4	21
9	13.3705	6.61684	6	0.06000	5	38
10	15.0747	5.87242	3	0.04000	3	19
11	15.6980	5.64063	3	0.05000	3	18
12	16.3664	5.41174	3	0.05000	3	17
13	16.5211	5.36141	3	0.06000	3	13
14	18.1080	4.89498	4	0.14000	4	40
15	18.3526	4.83029	26	0.19000	23	247
16	19.3249	4.58940	8	0.08000	7	54
17	19.5043	4.54759	9	0.14000	8	74
18	20.9701	4.23291	3	0.06000	3	18
19	21.1396	4.19935	3	0.04000	3	13
20	21.4189	4.14522	3	0.08000	3	29
21	22.9951	3.86452	6	0.12000	5	46
22	23.2346	3.82523	3	0.04000	3	13
23	23.5672	3.77199	4	0.06670	4	13
24	23.7834	3.73819	7	0.10000	6	35
25	23.9331	3.71514	22	0.12000	20	154
26	24.1138	3.68771	100	0.18700	90	785
27	24.7116	3.59984	3	0.10000	3	31
28	24.9561	3.56512	8	0.11000	7	52
29	25.1907	3.53245	12	0.14660	11	72
30	25.3770	3.50694	22	0.33330	20	268
31	25.9294	3.43346	4	0.20000	4	46
32	26.3886	3.37475	4	0.14000	4	29
33	26.5683	3.35233	8	0.12000	7	56
34	27.3204	3.26173	11	0.17330	10	97
35	27.5268	3.23774	7	0.08000	6	30
36	27.8613	3.19962	4	0.09000	4	32
37	28.7051	3.10746	26	0.24000	23	281
38	28.8848	3.08853	13	0.12000	12	87
39	32.8900	2.72100	6	0.10000	5	40
40	33.1197	2.70265	10	0.13340	9	59
41	33.2596	2.69160	10	0.12000	9	56
42	34.0379	2.63181	9	0.22830	8	95
43	34.2686	2.61462	6	0.06000	5	21
44	34.8980	2.56889	7	0.12000	6	42
45	35.0778	2.55614	3	0.00000	3	0
46	36.6864	2.44766	4	0.10000	4	21
47	36.9012	2.43391	3	0.11000	3	28
48	37.1860	2.41592	3	0.06000	3	16
49	37.6806	2.38534	3	0.07000	3	23
50	37.8455	2.37532	3	0.06000	3	15

Appendix 15: XRD Analysis of Sample 2 at 1140^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	39.2744	2.29213	4	0.16000	4	45
52	39.5292	2.27794	4	0.05000	4	22
53	39.8690	2.25930	11	0.15000	10	98
54	40.3836	2.23169	13	0.12660	12	95
55	41.9326	2.15277	9	0.12000	8	68
56	42.2524	2.13721	7	0.08000	6	30
57	42.9619	2.10354	3	0.06000	3	19
58	43.2318	2.09103	3	0.04000	3	25
59	44.4111	2.03821	4	0.08000	4	24
60	45.6104	1.98736	3	0.08000	3	19
61	46.0402	1.96980	3	0.10000	3	28
62	47.5727	1.90986	10	0.15330	9	91
63	49.1686	1.85154	3	0.16000	3	35
64	49.4735	1.84084	7	0.15000	6	50
65	49.7533	1.83114	3	0.11000	3	19
66	49.9283	1.82513	3	0.04000	3	11
67	52.2738	1.74863	7	0.13330	6	46
68	53.7416	1.70428	6	0.11000	5	35
69	54.1265	1.69306	6	0.08000	5	32
70	54.3714	1.68602	3	0.09000	3	20
71	57.0054	1.61421	3	0.04000	3	15
72	57.3852	1.60442	4	0.20000	4	56
73	59.7894	1.54552	4	0.11000	4	30

Appendix 16: XRD Analysis of Sample 2 at 1140^{o}C (continuation).

```
         ***  Basic Data Process  ***

# Data Infomation
          Group                : ERIC
          Data                 : S2-@-1140
          Sample Nmae          :
          Comment              :
          Date & Time          : 02-13-14 13:34:53

# Measurement Condition
    X-ray tube
          target               : Cu
          voltage              : 40.0 (kV)
          current              : 30.0 (mA)
    Slits
          Auto Slit            : not Used
          divergence slit      :   1.00000 (deg)
          scatter slit         :   1.00000 (deg)
          receiving slit       :   0.30000(mm)
    Scanning
          drive axis           : Theta-2Theta
          scan range           :    2.0000 - 60.0000 (deg)
          scan mode            : Continuous Scan
          scan speed           :    8.0000 (deg/min)
          sampling pitch       :    0.0200 (deg)
          preset time          :    0.15 (sec)

# Data Process Condition
    Smoothing                  [ AUTO ]
          smoothing points     : 11
    B.G.Subtruction            [ AUTO ]
          sampling points      : 13
          repeat times         : 30
    Ka1-a2 Separate            [ MANUAL ]
          Ka1 a2 ratio         : 50 (%)
    Peak Search                [ AUTO ]
          differential points  : 11
          FWHM threhold        : 0.050 (deg)
          intensity threhold   : 30 (par mil)
          FWHM ratio  (n-1)/n  : 2
    System error Correction    [ YES ]
    Precise peak Correction    [ NO ]
```

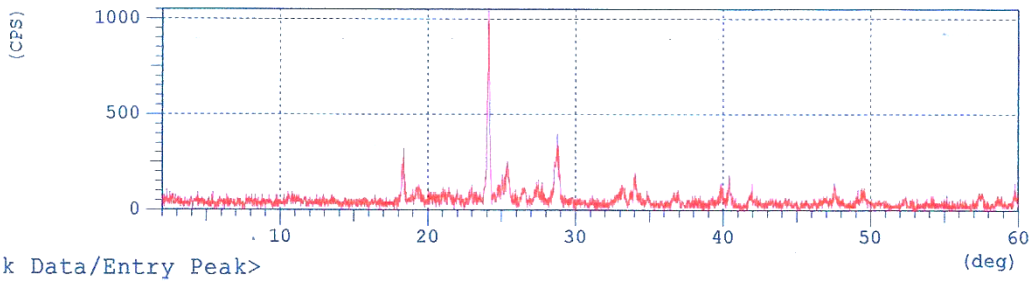
Appendix 17: XRD Analysis of Sample 3 at 1140^{o}C

********** SEARCH / MATCH RESULT **********

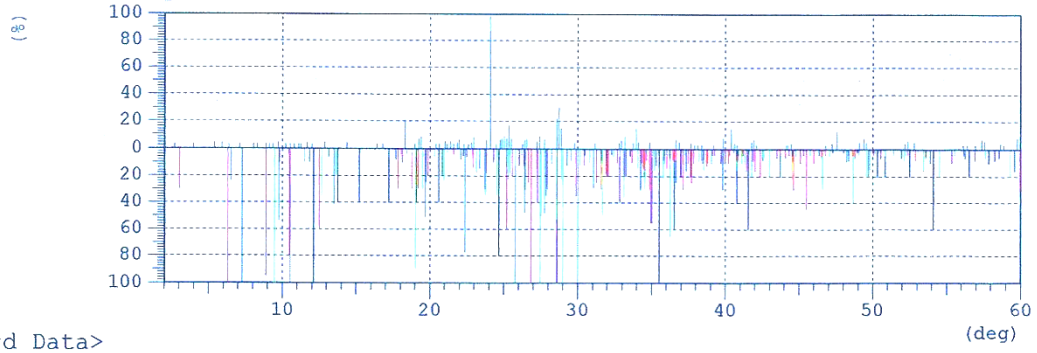
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Group Name : ERIC
Data Name : S3-@-1140
File Name : S3-@-1140.PSE
Sample Name :
Comment :
Date & Time : 02-13-14 12:54:29

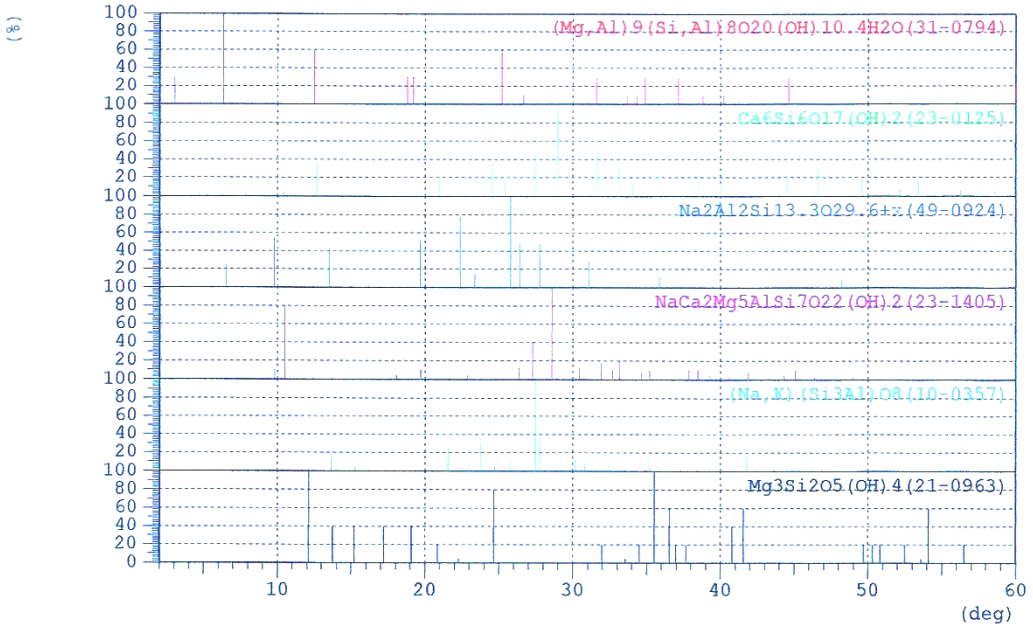
<Raw Data>



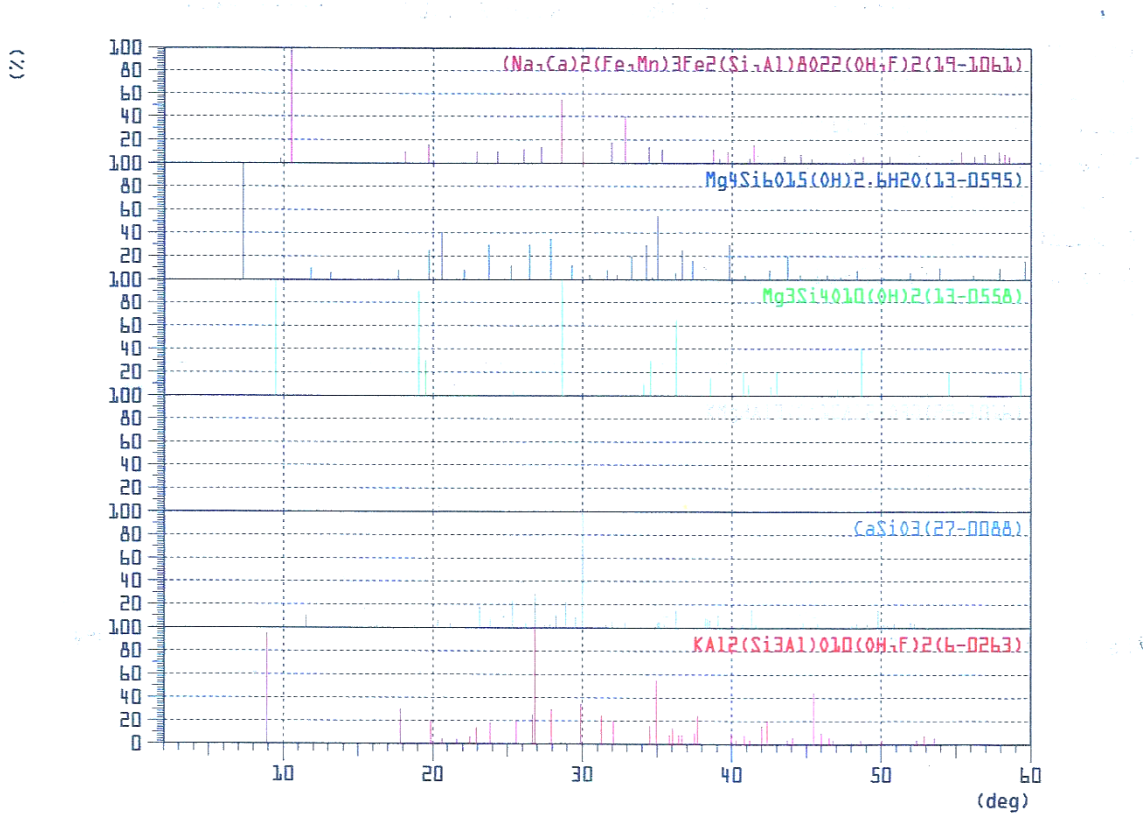
<Peak Data/Entry Peak>



<Card Data>



Appendix 18: XRD Analysis of Sample 3 at 1140^{o}C (continuation).



Appendix 19: XRD Analysis of Sample 3 at 1140^{o}C (continuation).

********** SEARCH / MATCH RESULT **********

Group Name : ERIC
Data Name : S3-@-1140
File Name : S3-@-1140.PSE
Sample Name :
Comment :

<Entry Card>

No.	Card	Chemical Formula / Chemical Name (Mineral Name)	S	L	d	I	R
				Dx	WT%	S.G.	
1	31-0794	(Mg,Al)9(Si,Al)8O20(OH)10.4H2O	0.161	1.067(16/17)	0.612	0.487	0.318
		Magnesium Aluminum Silicate Hydroxide Hydr	-----	-----		-------	
2	23-0125	Ca6Si6O17(OH)2	0.239	0.750(21/39)	0.803	0.497	0.300
		Calcium Silicate Hydrate (Xonotlite, syn	-----	-----		-------	
3	49-0924	Na2Al2Si13.3O29.6+x	0.112	0.643(9/14)	0.702	0.614	0.277
		Sodium Aluminum Silicate (Mordenite, syn	-----	-----		-------	
4	23-1405	NaCa2Mg5AlSi7O22(OH)2	0.291	0.826(19/23)	0.786	0.416	0.270
		Sodium Calcium Magnesium Aluminum Silicate	-----	-----		-------	
5	10-0357	(Na,K)(Si3Al)O8	0.243	0.824(14/17)	0.644	0.500	0.265
		Potassium Sodium Aluminum Silicate (Sanid	-----	-----		-------	
6	21-0963	Mg3Si2O5(OH)4	0.130	0.667(16/27)	0.708	0.536	0.253
		Magnesium Silicate Hydroxide (Antigorite-	-----	-----		-------	
7	19-1061	(Na,Ca)2(Fe,Mn)3Fe2(Si,Al)8O22(OH,F	0.330	0.774(24/38)	0.651	0.486	0.245
		Sodium Iron Silicate Hydroxide (Riebeckit	-----	-----		-------	
8	13-0595	Mg4Si6O15(OH)2.6H2O	0.258	0.778(28/39)	0.626	0.482	0.235
		Magnesium Silicate Hydroxide Hydrate (Sep	-----	-----		-------	
9	13-0558	Mg3Si4O10(OH)2	0.150	0.850(17/30)	0.581	0.430	0.212
		Magnesium Silicate Hydroxide (Talc-2\ITM\	-----	-----		-------	
10	29-1016	KMg2Al3(Si10Al2)O30	0.184	0.697(23/33)	0.697	0.423	0.206
		Potassium Magnesium Aluminum Silicate (Os	-----	-----		-------	
11	27-0088	CaSiO3	0.429	0.714(30/42)	0.695	0.408	0.203
		Calcium Silicate (Wollastonite-2\ITM\RG)	-----	-----		-------	
12	6-0263	KAl2(Si3Al)O10(OH,F)2	0.300	0.690(29/42)	0.727	0.377	0.189
		Potassium Aluminum Silicate Hydroxide (Mu	-----	-----		-------	

Appendix 20: XRD Analysis of Sample 3 at 1140°C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S3-@-1140

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	41	24.1369	3.68423	100	0.16050	100	872
2	56	28.7849	3.09902	31	0.22400	31	258
3	55	28.6252	3.11595	22	0.10400	22	159

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	3.0286	29.14883	3	0.06000	3	18
2	4.2052	20.99536	3	0.02000	3	5
3	5.5735	15.84367	4	0.11000	4	34
4	6.0457	14.60722	4	0.10000	4	33
5	6.5378	13.50879	3	0.02670	3	7
6	7.2512	12.18127	3	0.04000	3	15
7	8.1581	10.82905	3	0.04000	3	14
8	8.4149	10.49915	3	0.02000	3	4
9	9.0035	9.81405	3	0.03000	3	8
10	9.2065	9.59810	3	0.04000	3	17
11	9.4641	9.33742	3	0.02000	3	12
12	9.8309	8.98984	4	0.06000	4	22
13	10.3320	8.55494	3	0.03000	3	7
14	10.6149	8.32756	3	0.04000	3	23
15	10.8980	8.11186	3	0.05000	3	16
16	11.1912	7.89999	3	0.04000	3	8
17	11.3304	7.80324	3	0.04000	3	13
18	11.7679	7.51410	4	0.04000	4	16
19	12.0862	7.31692	3	0.04000	3	10
20	12.9621	6.82439	4	0.08000	4	25
21	14.4566	6.12208	4	0.08000	4	20
22	16.4961	5.36948	3	0.05000	3	10
23	16.8703	5.25121	3	0.04000	3	9
24	18.3667	4.82661	21	0.17170	21	213
25	19.0508	4.65481	3	0.03000	3	9
26	19.2966	4.59607	7	0.12330	7	45
27	19.4620	4.55738	8	0.11500	8	53
28	20.4515	4.33906	3	0.04000	3	9
29	20.6859	4.29042	3	0.03000	3	9
30	21.0947	4.20818	5	0.07000	5	19
31	21.2344	4.18081	3	0.05000	3	10
32	21.4488	4.13950	5	0.06000	5	31
33	21.6533	4.10087	3	0.05000	3	13
34	21.9176	4.05201	4	0.08000	4	18
35	22.0274	4.03206	6	0.14000	6	57
36	22.3616	3.97255	3	0.09000	3	19
37	22.6559	3.92161	3	0.04000	3	13
38	22.8704	3.88531	4	0.13000	4	26
39	23.0076	3.86245	8	0.10500	8	57
40	23.8932	3.72126	6	0.08000	6	41
41	24.1369	3.68423	100	0.16050	100	872
42	24.5452	3.62386	3	0.02670	3	7
43	24.7715	3.59127	6	0.08000	6	28
44	24.8962	3.57356	7	0.05000	7	21
45	25.0809	3.54766	9	0.10000	9	72
46	25.2705	3.52147	7	0.00000	7	0
47	25.4053	3.50309	17	0.19000	17	147
48	25.5500	3.48358	7	0.10660	7	54
49	26.3087	3.38482	3	0.08000	3	17
50	26.4485	3.36724	6	0.08000	6	27

Appendix 21: XRD Analysis of Sample 3 at 1140^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	26.5916	3.34945	7	0.12670	7	53
52	27.2921	3.26505	5	0.07000	5	29
53	27.4602	3.24544	9	0.13330	9	69
54	27.7764	3.20921	7	0.10000	7	44
55	28.6252	3.11595	22	0.10400	22	159
56	28.7849	3.09902	31	0.22400	31	258
57	28.9248	3.08435	15	0.12000	15	92
58	29.8236	2.99341	4	0.08000	4	27
59	31.2068	2.86381	4	0.09000	4	26
60	32.8500	2.72422	4	0.08000	4	28
61	32.9999	2.71219	6	0.08000	6	28
62	33.2221	2.69455	9	0.14500	9	91
63	33.7691	2.65214	4	0.10000	4	23
64	34.0143	2.63358	15	0.11900	15	84
65	34.1387	2.62427	5	0.08000	5	30
66	34.9030	2.56854	5	0.09000	5	32
67	36.6864	2.44766	7	0.08000	7	40
68	36.9195	2.43274	5	0.11330	5	43
69	37.6956	2.38442	4	0.12000	4	27
70	37.9537	2.36880	3	0.09670	3	23
71	38.1902	2.35467	3	0.03000	3	9
72	38.4450	2.33965	3	0.10000	3	21
73	39.2144	2.29550	3	0.04000	3	15
74	39.8889	2.25822	8	0.13000	8	76
75	40.4311	2.22918	15	0.10500	15	97
76	40.6434	2.21803	4	0.08000	4	15
77	40.8383	2.20789	3	0.03000	3	10
78	41.7977	2.15940	5	0.11000	5	36
79	41.9492	2.15195	7	0.07330	7	27
80	42.1474	2.14229	3	0.07000	3	18
81	44.1512	2.04960	3	0.04000	3	11
82	44.2711	2.04433	3	0.12000	3	21
83	44.4910	2.03473	4	0.08000	4	15
84	46.8298	1.93841	3	0.04000	3	19
85	47.2695	1.92140	4	0.08000	4	21
86	47.5794	1.90961	13	0.10000	13	91
87	49.0986	1.85402	5	0.10000	5	40
88	49.4035	1.84329	8	0.15000	8	52
89	49.5784	1.83719	5	0.14000	5	44
90	52.1973	1.75101	4	0.10000	4	25
91	52.3772	1.74542	6	0.14000	6	52
92	52.8070	1.73222	5	0.04000	5	31
93	56.1057	1.63794	3	0.04000	3	12
94	56.4256	1.62941	3	0.04000	3	15
95	56.9454	1.61577	3	0.04000	3	12
96	57.3752	1.60468	7	0.14000	7	61
97	57.5752	1.59958	6	0.14000	6	56
98	58.6781	1.57212	4	0.17330	4	43
99	58.8697	1.56745	3	0.07000	3	19
100	59.7927	1.54544	8	0.12330	8	48

Appendix 22: XRD Analysis of Sample 3 at 1140^{o}C (continuation).

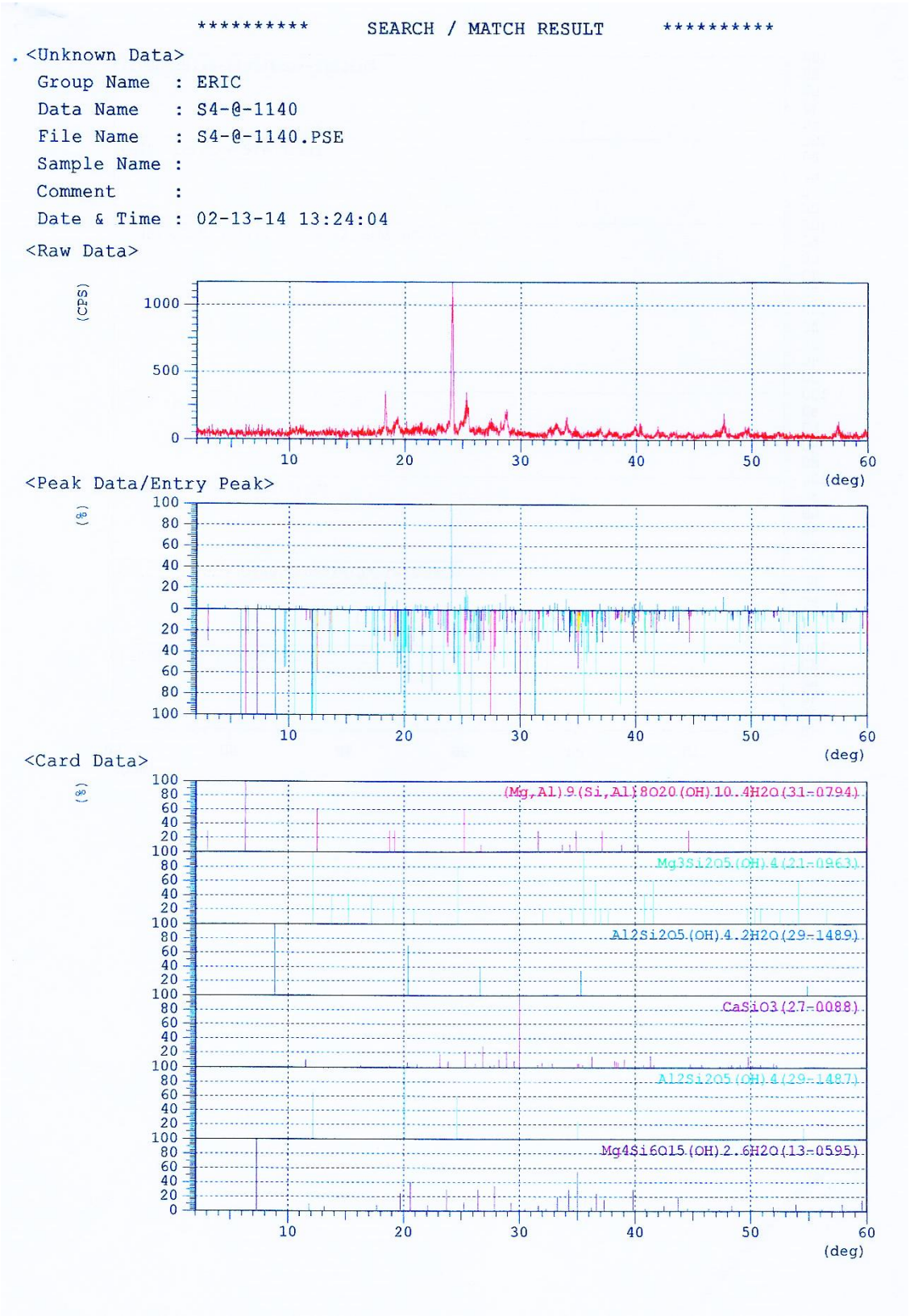
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         ***  Basic Data Process  ***

# Data Infomation
          Group                : ERIC
          Data                 : S3-@-1140
          Sample Nmae          :
          Comment              :
          Date & Time          : 02-13-14 12:54:29

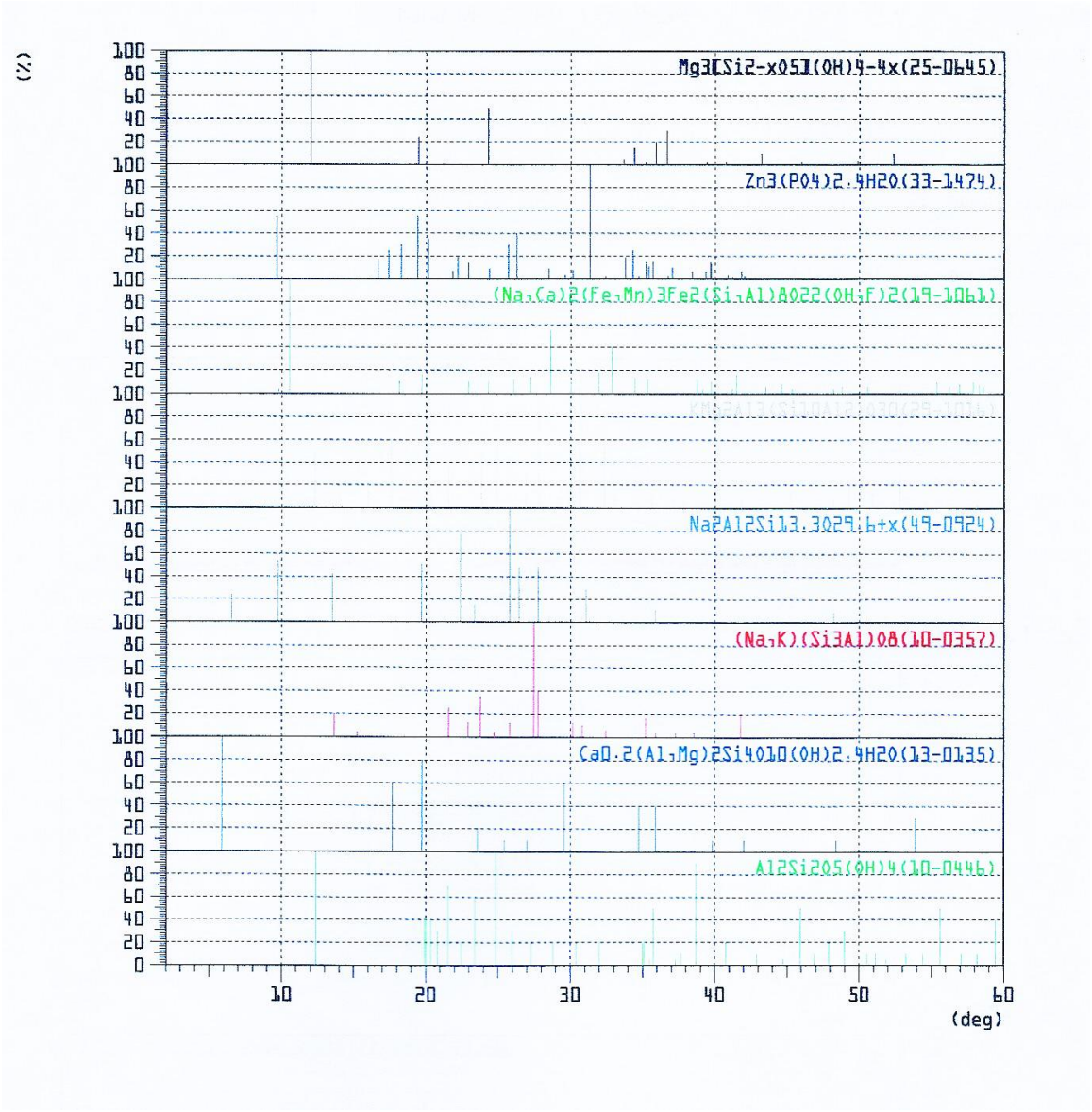
# Measurement Condition
    X-ray tube
          target               : Cu
          voltage              : 40.0 (kV)
          current              : 30.0 (mA)
    Slits
          Auto Slit            : not Used
          divergence slit      :   1.00000 (deg)
          scatter slit         :   1.00000 (deg)
          receiving slit       :   0.30000(mm)
    Scanning
          drive axis           : Theta-2Theta
          scan range           :    2.0000 - 60.0000 (deg)
          scan mode            : Continuous Scan
          scan speed           :    8.0000 (deg/min)
          sampling pitch       :    0.0200 (deg)
          preset time          :    0.15 (sec)

# Data Process Condition
    Smoothing                  [ AUTO ]
          smoothing points     : 9
    B.G.Subtruction            [ AUTO ]
          sampling points      : 11
          repeat times         : 30
    Ka1-a2 Separate            [ MANUAL ]
          Ka1 a2 ratio         : 50 (%)
    Peak Search                [ AUTO ]
          differential points  : 9
          FWHM threhold        : 0.050 (deg)
          intensity threhold   : 30 (par mil)
          FWHM ratio  (n-1)/n  : 2
    System error Correction    [ YES ]
    Precise peak Correction    [ NO ]
```

Appendix 23: XRD Analysis of Sample 4 at 1140^{o}C



Appendix 24: XRD Analysis of Sample 4 at 1140^{o}C (continuation).



Appendix 25: XRD Analysis of Sample 4 at 1140^{o}C (continuation).

********** SEARCH / MATCH RESULT **********

Group Name : ERIC
Data Name : S4-@-1140
File Name : S4-@-1140.PSE
Sample Name :
Comment :

<Entry Card>

No.	Card	Chemical Formula / Chemical Name (Mineral Name)	S	L	d	I	R
				Dx	WT%	S.G.	
1	31-0794	(Mg,Al)9(Si,Al)8O20(OH)10.4H2O	0.179	0.933	(14/17)	0.706	0.515	0.340
		Magnesium Aluminum Silicate Hydroxide Hydr	-----			-------		
2	21-0963	Mg3Si2O5(OH)4	0.135	0.708	(17/27)	0.657	0.565	0.263
		Magnesium Silicate Hydroxide (Antigorite-	-----			-------		
3	29-1489	Al2Si2O5(OH)4.2H2O	0.043	0.600	(3/ 7)	0.517	0.750	0.233
		Aluminum Silicate Hydroxide Hydrate (Hall	-----			-------		
4	27-0088	CaSiO3	0.543	0.643	(27/42)	0.678	0.520	0.227
		Calcium Silicate (Wollastonite-2\ITM\RG)	-----			-------		
5	29-1487	Al2Si2O5(OH)4	0.043	0.500	(3/ 7)	0.766	0.546	0.209
		Aluminum Silicate Hydroxide (Halloysite-7	-----			-------		
6	13-0595	Mg4Si6O15(OH)2.6H2O	0.226	0.667	(24/39)	0.656	0.478	0.209
		Magnesium Silicate Hydroxide Hydrate (Sep	-----			-------		
7	25-0645	Mg3[Si2-xO5](OH)4-4x	0.279	0.786	(11/18)	0.656	0.404	0.208
		Magnesium Silicate Hydroxide (Chrysotile	-----			-------		
8	33-1474	Zn3(PO4)2.4H2O	0.328	0.571	(24/42)	0.778	0.426	0.190
		Zinc Phosphate Hydrate (Hopeite, syn)	-----			-------		
9	19-1061	(Na,Ca)2(Fe,Mn)3Fe2(Si,Al)8O22(OH,F	0.270	0.613	(19/38)	0.639	0.471	0.185
		Sodium Iron Silicate Hydroxide (Riebeckit	-----			-------		
10	29-1016	KMg2Al3(Si10Al2)O30	0.172	0.636	(21/33)	0.693	0.414	0.183
		Potassium Magnesium Aluminum Silicate (Os	-----			-------		
11	49-0924	Na2Al2Si13.3O29.6+x	0.121	0.500	(7/14)	0.571	0.629	0.180
		Sodium Aluminum Silicate (Mordenite, syn	-----			-------		
12	10-0357	(Na,K)(Si3Al)O8	0.270	0.647	(11/17)	0.693	0.386	0.173
		Potassium Sodium Aluminum Silicate (Sanid	-----			-------		
13	13-0135	Ca0.2(Al,Mg)2Si4O10(OH)2.4H2O	0.142	0.692	(9/17)	0.710	0.333	0.164
		Calcium Magnesium Aluminum Silicate Hydrox	-----			-------		
14	10-0446	Al2Si2O5(OH)4	0.161	0.622	(23/39)	0.595	0.434	0.161
		Aluminum Silicate Hydroxide (Dickite-2\IT	-----			-------		

Appendix 26: XRD Analysis of Sample 4 at 1140^{o}C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S4-@-1140

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	32	24.1041	3.68917	100	0.19070	102	978
2	20	18.3576	4.82898	26	0.14800	27	246
3	36	25.3504	3.51056	19	0.00000	19	0

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	3.3325	26.49132	4	0.06000	4	13
2	4.6955	18.80409	3	0.03000	3	9
3	6.4072	13.78385	3	0.04000	3	12
4	6.6423	13.29650	3	0.04000	3	15
5	7.4530	11.85189	4	0.13000	4	34
6	7.7387	11.41497	3	0.03000	3	12
7	8.1581	10.82905	3	0.04000	3	18
8	8.4545	10.45006	3	0.04000	3	22
9	9.2411	9.56224	3	0.03000	3	9
10	10.7093	8.25437	3	0.07000	3	15
11	11.0024	8.03512	3	0.06000	3	23
12	11.2061	7.88952	3	0.03000	3	10
13	14.2622	6.20509	3	0.05000	3	18
14	14.6958	6.02297	3	0.04000	3	16
15	15.3938	5.75140	4	0.08000	4	24
16	16.8204	5.26668	3	0.06000	3	12
17	17.0350	5.20081	3	0.03000	3	9
18	17.2895	5.12483	3	0.04000	3	14
19	17.8684	4.96007	3	0.04000	3	14
20	18.3576	4.82898	26	0.14800	27	246
21	19.0657	4.65120	3	0.06000	3	15
22	19.4196	4.56723	9	0.11000	9	108
23	20.1673	4.39956	3	0.07000	3	19
24	21.0100	4.22496	5	0.06000	5	23
25	21.1995	4.18762	4	0.08000	4	20
26	21.4638	4.13665	5	0.17000	5	59
27	21.8678	4.06113	3	0.06000	3	16
28	22.0174	4.03387	3	0.04000	3	11
29	22.6210	3.92758	3	0.05000	3	17
30	22.9053	3.87947	6	0.22000	6	92
31	23.8134	3.73355	7	0.08000	7	57
32	24.1041	3.68917	100	0.19070	102	978
33	24.4005	3.64503	3	0.03670	3	8
34	24.8513	3.57992	5	0.04000	5	31
35	25.2506	3.52420	12	0.22000	12	190
36	25.3504	3.51056	19	0.00000	19	0
37	25.4901	3.49163	14	0.16000	14	186
38	26.4235	3.37037	3	0.09000	3	28
39	26.8129	3.32230	3	0.05000	3	11
40	27.0425	3.29461	4	0.11000	4	19
41	27.3271	3.26094	5	0.12000	5	53
42	27.6266	3.22627	5	0.08000	5	44
43	28.3056	3.15040	7	0.08000	7	32
44	28.7625	3.10138	15	0.27500	15	229
45	29.1644	3.05955	3	0.04000	3	13
46	29.4673	3.02879	3	0.08670	3	26
47	32.0209	2.79284	3	0.04000	3	11
48	32.5903	2.74533	5	0.06000	5	26
49	33.0248	2.71020	7	0.13000	7	51
50	33.2396	2.69317	6	0.24000	6	76

Appendix 27: XRD Analysis of Sample 4 at 1140^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	33.9889	2.63549	11	0.14000	11	96
52	34.2436	2.61647	3	0.03000	3	11
53	36.9112	2.43327	6	0.11000	6	57
54	37.6756	2.38564	4	0.12000	4	34
55	37.8737	2.37362	3	0.05670	3	12
56	38.2951	2.34846	3	0.04000	3	20
57	38.8747	2.31477	3	0.04000	3	13
58	39.7141	2.26776	3	0.04000	3	13
59	39.8989	2.25768	6	0.15000	6	54
60	40.3586	2.23302	8	0.09000	8	39
61	40.5568	2.22256	5	0.04670	5	16
62	41.8926	2.15473	6	0.10000	6	52
63	43.2418	2.09057	4	0.14000	4	39
64	43.5016	2.07869	4	0.10000	4	20
65	43.7115	2.06919	4	0.04000	4	15
66	44.5510	2.03213	3	0.04000	3	19
67	45.8103	1.97915	3	0.04000	3	12
68	46.9147	1.93510	4	0.11000	4	40
69	47.5694	1.90999	13	0.14000	13	120
70	49.4285	1.84241	5	0.08000	5	27
71	49.6984	1.83304	5	0.10000	5	35
72	50.3181	1.81191	3	0.06000	3	20
73	52.2872	1.74821	4	0.12000	4	33
74	52.6971	1.73557	3	0.06000	3	17
75	57.3952	1.60417	8	0.18000	8	96
76	59.0547	1.56299	3	0.06000	3	21
77	59.8044	1.54517	5	0.12000	5	39

Appendix 28: XRD Analysis of Sample 4 at 1140^{o}C (continuation).

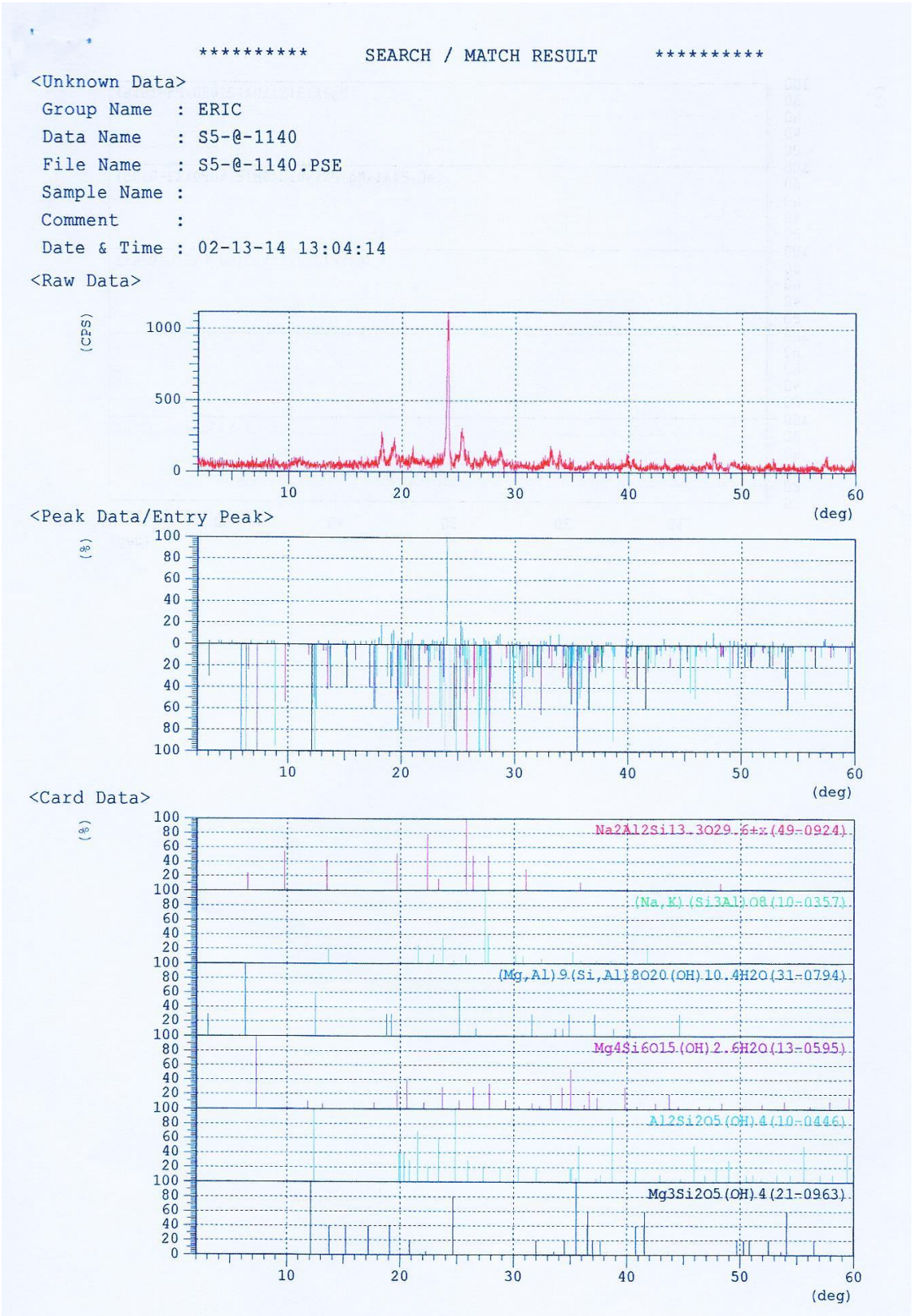
```
          ***  Basic Data Process  ***

# Data Infomation
          Group                 : ERIC
          Data                  : S4-@-1140
          Sample Nmae           :
          Comment               :
          Date & Time           : 02-13-14 13:24:04

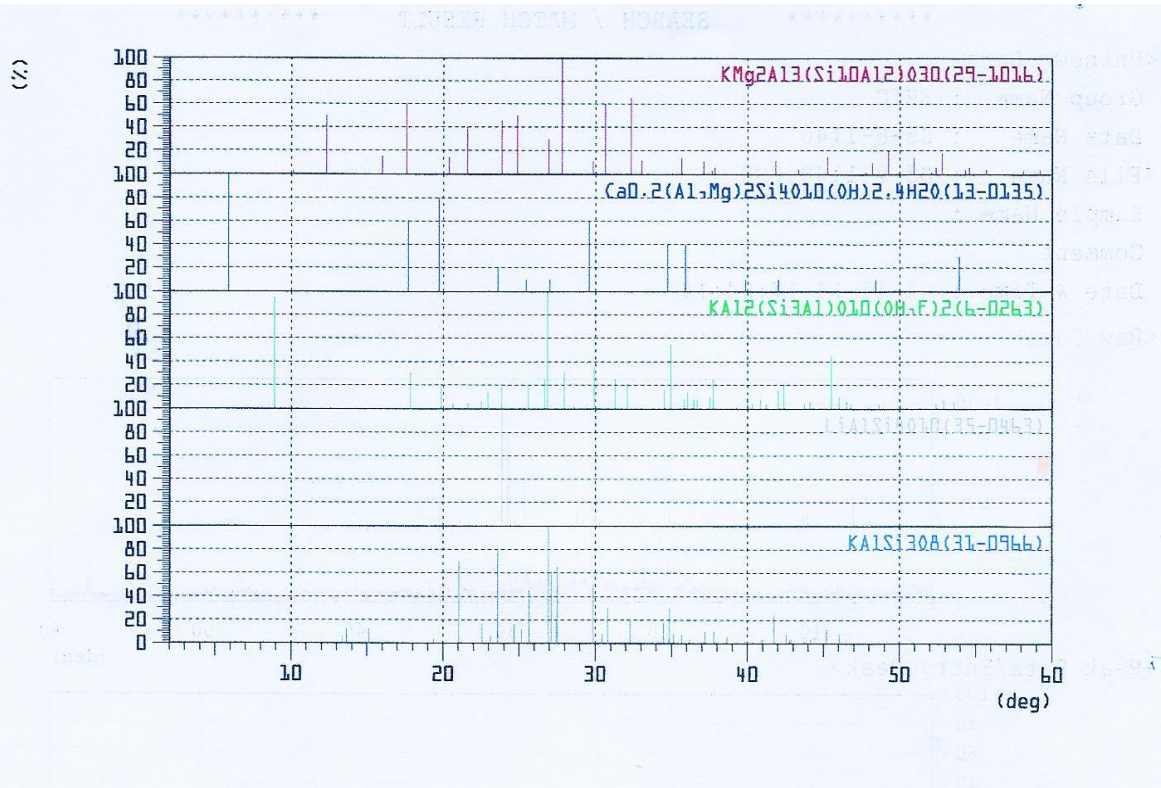
# Measurement Condition
    X-ray tube
          target                : Cu
          voltage               : 40.0 (kV)
          current               : 30.0 (mA)
    Slits
          Auto Slit             : not Used
          divergence slit       :   1.00000 (deg)
          scatter slit          :   1.00000 (deg)
          receiving slit        :   0.30000(mm)
    Scanning
          drive axis            : Theta-2Theta
          scan range            :    2.0000 - 60.0000 (deg)
          scan mode             : Continuous Scan
          scan speed            :    8.0000 (deg/min)
          sampling pitch        :    0.0200 (deg)
          preset time           :    0.15 (sec)

# Data Process Condition
    Smoothing                   [ AUTO ]
          smoothing points      : 11
    B.G.Subtruction             [ AUTO ]
          sampling points       : 11
          repeat times          : 30
    Ka1-a2 Separate             [ MANUAL ]
          Ka1 a2 ratio          : 50 (%)
    Peak Search                 [ AUTO ]
          differential points   : 11
          FWHM threhold         : 0.050 (deg)
          intensity threhold    : 30 (par mil)
          FWHM ratio  (n-1)/n   : 2
    System error Correction     [ YES ]
    Precise peak Correction     [ NO ]
```

Appendix 29: XRD Analysis of Sample 5 at 1140^{o}C.



Appendix 30: XRD Analysis of Sample 5 at 1140^{o}C (continuation).



Appendix 31: XRD Analysis of Sample 5 at 1140^{o}C (continuation).

********** SEARCH / MATCH RESULT **********

Group Name : ERIC
Data Name : S5-@-1140
File Name : S5-@-1140.PSE
Sample Name :
Comment :

<Entry Card>

No.	Card	Chemical Formula / Chemical Name (Mineral Name)	S	L	Dx	d WT%	I S.G.	R
1	49-0924	Na2Al2Si13.3O29.6+x Sodium Aluminum Silicate (Mordenite, syn	0.079	0.714	(10/14) -----	0.746	0.622 -------	0.332
2	10-0357	(Na,K)(Si3Al)O8 Potassium Sodium Aluminum Silicate (Sanid	0.181	0.882	(15/17) -----	0.740	0.465 -------	0.303
3	31-0794	(Mg,Al)9(Si,Al)8O20(OH)10.4H2O Magnesium Aluminum Silicate Hydroxide Hydr	0.166	0.933	(14/17) -----	0.589	0.439 -------	0.241
4	13-0595	Mg4Si6O15(OH)2.6H2O Magnesium Silicate Hydroxide Hydrate (Sep	0.258	0.694	(25/39) -----	0.726	0.477 -------	0.240
5	10-0446	Al2Si2O5(OH)4 Aluminum Silicate Hydroxide (Dickite-2\IT	0.135	0.784	(29/39) -----	0.629	0.468 -------	0.231
6	21-0963	Mg3Si2O5(OH)4 Magnesium Silicate Hydroxide (Antigorite-	0.131	0.708	(17/27) -----	0.576	0.487 -------	0.199
7	29-1016	KMg2Al3(Si10Al2)O30 Potassium Magnesium Aluminum Silicate (Os	0.134	0.667	(22/33) -----	0.687	0.425 -------	0.195
8	13-0135	Ca0.2(Al,Mg)2Si4O10(OH)2.4H2O Calcium Magnesium Aluminum Silicate Hydrox	0.139	0.769	(10/17) -----	0.715	0.331 -------	0.182
9	6-0263	KAl2(Si3Al)O10(OH,F)2 Potassium Aluminum Silicate Hydroxide (Mu	0.209	0.643	(27/42) -----	0.685	0.397 -------	0.175
10	35-0463	LiAlSi4O10 Lithium Aluminum Silicate (Petalite-1\ITM	0.557	0.548	(23/42) -----	0.754	0.380 -------	0.157
11	31-0966	KAlSi3O8 Potassium Aluminum Silicate (Orthoclase)	0.175	0.571	(24/42) -----	0.736	0.362 -------	0.152

Appendix 32: XRD Analysis of Sample 5 at 1140°C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S5-@-1140

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	35	24.0175	3.70228	100	0.19910	103	1123
2	37	25.2400	3.52566	22	0.16000	23	230
3	19	18.2385	4.86025	18	0.22700	19	218

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	3.0850	28.61606	3	0.03000	3	14
2	3.9200	22.52222	3	0.04000	3	11
3	4.1350	21.35164	3	0.03000	3	7
4	5.0550	17.46756	3	0.03000	3	7
5	6.7550	13.07491	3	0.03000	3	8
6	8.2000	10.77381	3	0.04000	3	15
7	8.5600	10.32150	3	0.04000	3	14
8	8.7750	10.06908	3	0.03000	3	11
9	12.7200	6.95373	3	0.04000	3	10
10	13.5700	6.52001	3	0.06000	3	16
11	14.8300	5.96876	3	0.06000	3	18
12	15.0500	5.88200	3	0.06000	3	17
13	15.8550	5.58512	3	0.03000	3	6
14	16.4100	5.39746	3	0.06000	3	14
15	16.8850	5.24667	3	0.03000	3	10
16	17.3750	5.09980	3	0.03000	3	8
17	17.6450	5.02237	4	0.09000	4	30
18	18.0000	4.92411	6	0.12000	6	48
19	18.2385	4.86025	18	0.22700	19	218
20	19.1400	4.63331	10	0.12000	10	81
21	19.3000	4.59526	13	0.20000	13	129
22	19.7333	4.49533	3	0.06670	3	13
23	20.0250	4.43050	4	0.07000	4	23
24	20.3600	4.35835	3	0.04000	3	18
25	20.5766	4.31296	4	0.07330	4	27
26	20.8000	4.26714	4	0.00000	4	0
27	20.9466	4.23760	11	0.10670	11	105
28	21.8000	4.07360	4	0.12000	4	29
29	21.9300	4.04975	4	0.06000	4	13
30	22.7200	3.91069	4	0.08000	4	21
31	22.9000	3.88036	3	0.10000	3	27
32	23.1350	3.84147	3	0.07000	3	14
33	23.5000	3.78262	3	0.05340	3	23
34	23.7200	3.74804	7	0.08000	7	60
35	24.0175	3.70228	100	0.19910	103	1123
36	24.7600	3.59291	6	0.08000	6	60
37	25.2400	3.52566	22	0.16000	23	230
38	25.4000	3.50381	16	0.15000	16	135
39	25.6200	3.47422	4	0.04000	4	18
40	25.8766	3.44035	4	0.20670	4	43
41	26.3916	3.37437	6	0.08330	6	29
42	26.5550	3.35398	3	0.03000	3	6
43	27.2750	3.26705	7	0.11000	7	45
44	27.4400	3.24778	4	0.12000	4	35
45	27.7200	3.21561	3	0.04000	3	17
46	28.3750	3.14285	4	0.07000	4	15
47	28.5200	3.12720	8	0.07000	8	29
48	28.7000	3.10800	10	0.20000	10	123
49	29.4300	3.03254	3	0.10000	3	31
50	29.7350	3.00213	3	0.07000	3	12

Appendix 33: XRD Analysis of Sample 5 at 1140^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	30.1900	2.95791	3	0.06000	3	20
52	30.4500	2.93324	3	0.06000	3	20
53	31.2350	2.86129	3	0.07000	3	13
54	32.2850	2.77059	4	0.09000	4	22
55	32.5800	2.74618	4	0.08000	4	37
56	32.7550	2.73190	3	0.03000	3	7
57	33.1300	2.70183	9	0.18000	9	98
58	33.9050	2.64182	10	0.11000	10	63
59	35.0950	2.55492	3	0.07000	3	12
60	36.7850	2.44133	4	0.11000	4	32
61	38.0650	2.36213	3	0.05000	3	13
62	38.2383	2.35182	3	0.09670	3	17
63	38.4700	2.33819	3	0.06000	3	17
64	39.8316	2.26134	7	0.11670	7	56
65	40.3500	2.23347	4	0.14000	4	43
66	43.1550	2.09458	6	0.11000	6	36
67	44.4900	2.03477	3	0.06000	3	19
68	46.8850	1.93626	4	0.13000	4	39
69	47.5250	1.91167	12	0.19000	12	129
70	48.0900	1.89052	3	0.06000	3	18
71	48.9800	1.85823	5	0.20000	5	77
72	49.3200	1.84621	5	0.12000	5	45
73	49.6700	1.83402	4	0.06000	4	20
74	50.3800	1.80982	3	0.04000	3	12
75	51.2900	1.77983	3	0.06000	3	16
76	52.7650	1.73350	4	0.09000	4	19
77	53.7000	1.70550	3	0.04000	3	14
78	57.2400	1.60815	5	0.14000	5	36
79	57.3500	1.60532	7	0.14000	7	63
80	57.9950	1.58900	3	0.07000	3	14
81	59.7500	1.54645	4	0.10000	4	28

Appendix 34: XRD Analysis of Sample 5 at 1140^{o}C (continuation).

```
          ***  Basic Data Process  ***

# Data Infomation
          Group                : ERIC
          Data                 : S5-@-1140
          Sample Nmae          :
          Comment              :
          Date & Time          : 02-13-14 13:04:14

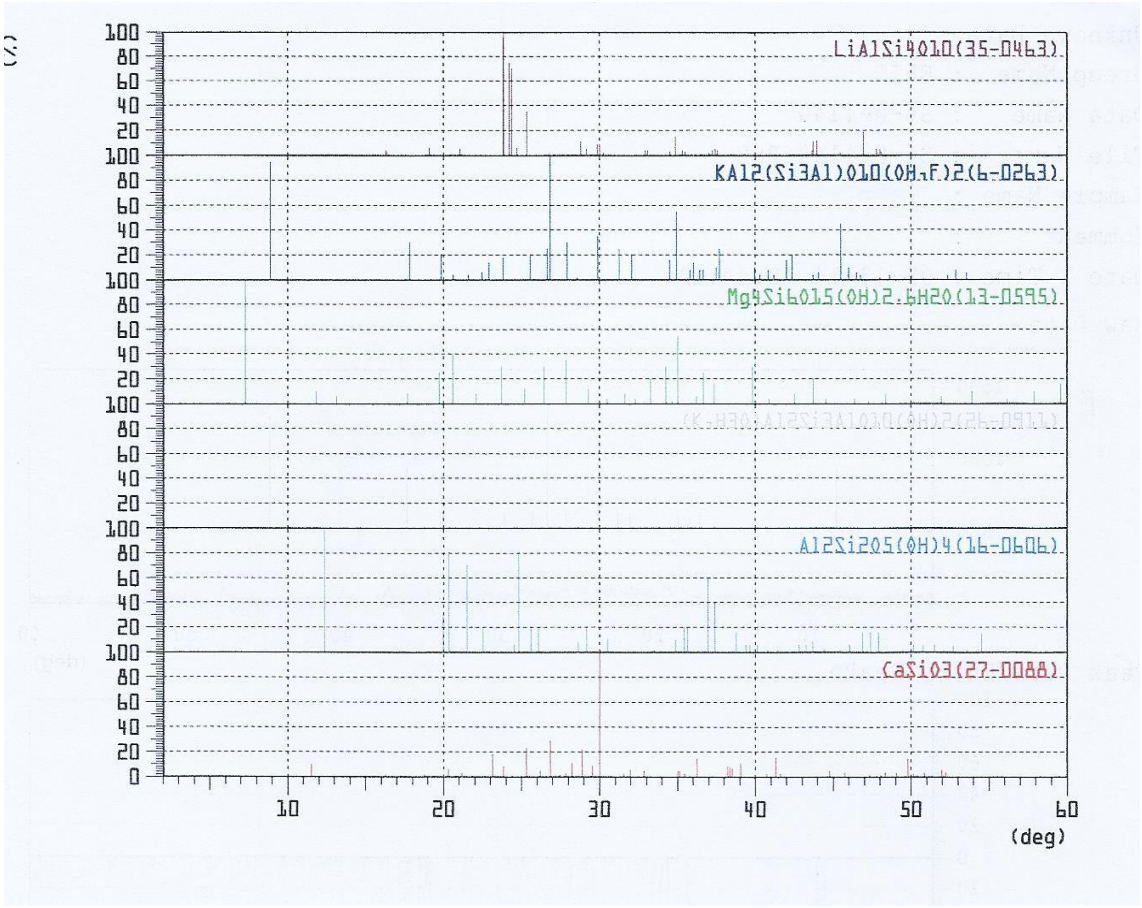
# Measurement Condition
    X-ray tube
          target               : Cu
          voltage              : 40.0 (kV)
          current              : 30.0 (mA)
    Slits
          Auto Slit            : not Used
          divergence slit      :   1.00000 (deg)
          scatter slit         :   1.00000 (deg)
          receiving slit       :   0.30000(mm)
    Scanning
          drive axis           : Theta-2Theta
          scan range           :    2.0000 - 60.0000 (deg)
          scan mode            : Continuous Scan
          scan speed           :    8.0000 (deg/min)
          sampling pitch       :    0.0200 (deg)
          preset time          :    0.15 (sec)

# Data Process Condition
```

Appendix 35: XRD Analysis of Sample 6 at 1140^{o}C.



Appendix 36: XRD Analysis of Sample 6 at 1140°C (continuation).



Appendix 37: XRD Analysis of Sample 6 at 1140^{o}C (continuation).

********** SEARCH / MATCH RESULT **********

Group Name : ERIC
Data Name : S6-@-1140
File Name : S6-@-1140.PSE
Sample Name :
Comment :

<Entry Card>

No.	Card	Chemical Formula / Chemical Name (Mineral Name)	S	L	d	I	R
				Dx	WT%	S.G.	
1	10-0495	KMg3(Si3Al)O10(OH)2	0.692	0.500	(12/26) 0.776	-----	0.388
		Potassium Magnesium Aluminum Silicate Hydr	-----			-------	
2	13-0135	Ca0.2(Al,Mg)2Si4O10(OH)2.4H2O	0.179	0.462	(6/17) 0.639	-----	0.295
		Calcium Magnesium Aluminum Silicate Hydrox	-----			-------	
3	25-0645	Mg3[Si2-xO5](OH)4-4x	0.605	0.500	(7/18) 0.494	-----	0.247
		Magnesium Silicate Hydroxide (Chrysotile	-----			-------	
4	23-0125	Ca6Si6O17(OH)2	0.309	0.393	(11/39) 0.628	-----	0.247
		Calcium Silicate Hydrate (Xonotlite, syn	-----			-------	
5	46-1045	SiO2	0.553	0.385	(5/42) 0.622	-----	0.239
		Silicon Oxide (Quartz, syn)	-----			-------	
6	13-0558	Mg3Si4O10(OH)2	0.134	0.350	(7/30) 0.635	-----	0.222
		Magnesium Silicate Hydroxide (Talc-2\ITM\	-----			-------	
7	35-0463	LiAlSi4O10	0.492	0.238	(10/42) 0.825	-----	0.196
		Lithium Aluminum Silicate (Petalite-1\ITM	-----			-------	
8	6-0263	KAl2(Si3Al)O10(OH,F)2	0.261	0.310	(13/42) 0.598	-----	0.185
		Potassium Aluminum Silicate Hydroxide (Mu	-----			-------	
9	13-0595	Mg4Si6O15(OH)2.6H2O	0.275	0.333	(12/39) 0.533	-----	0.178
		Magnesium Silicate Hydroxide Hydrate (Sep	-----			-------	
10	26-0911	(K,H3O)Al2Si3AlO10(OH)2	0.392	0.353	(6/18) 0.485	-----	0.171
		Potassium Aluminum Silicate Hydroxide (Il	-----			-------	
11	16-0606	Al2Si2O5(OH)4	0.237	0.286	(12/42) 0.580	-----	0.166
		Aluminum Silicate Hydroxide (Nacrite-2\IT	-----			-------	
12	27-0088	CaSiO3	0.484	0.286	(12/42) 0.579	-----	0.165
		Calcium Silicate (Wollastonite-2\ITM\RG)	-----			-------	

Appendix 38: XRD Analysis of Sample 6 at 1140^{o}C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S6-@-1140

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	10	24.0953	3.69050	100	0.17500	154	1425
2	2	18.2977	4.84466	19	0.16400	29	279
3	11	25.3420	3.51170	12	0.31670	18	317

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	18.0880	4.90035	4	0.06000	6	32
2	18.2977	4.84466	19	0.16400	29	279
3	19.1853	4.62248	5	0.20000	8	93
4	19.4112	4.56919	8	0.10670	13	83
5	19.6289	4.51900	4	0.07000	6	28
6	20.0178	4.43208	3	0.05000	5	43
7	21.0000	4.22695	5	0.08000	7	47
8	21.4222	4.14458	5	0.12670	8	65
9	23.7934	3.73664	5	0.10000	8	73
10	24.0953	3.69050	100	0.17500	154	1425
11	25.3420	3.51170	12	0.31670	18	317
12	27.3004	3.26407	5	0.14670	7	75
13	28.6551	3.11276	3	0.10000	5	37
14	28.8399	3.09324	3	0.19000	5	54
15	32.9999	2.71219	3	0.08000	5	36
16	33.1597	2.69948	8	0.10000	12	82
17	33.9814	2.63606	9	0.15500	14	162
18	36.9462	2.43105	5	0.12000	7	56
19	39.8390	2.26094	6	0.13000	9	74
20	40.3577	2.23307	4	0.10170	6	35
21	47.5444	1.91093	7	0.23000	11	163
22	52.4072	1.74449	3	0.12000	5	64
23	57.3952	1.60417	5	0.10000	7	55

Appendix 39: XRD Analysis of Sample 6 at 1140^{o}C (continuation).

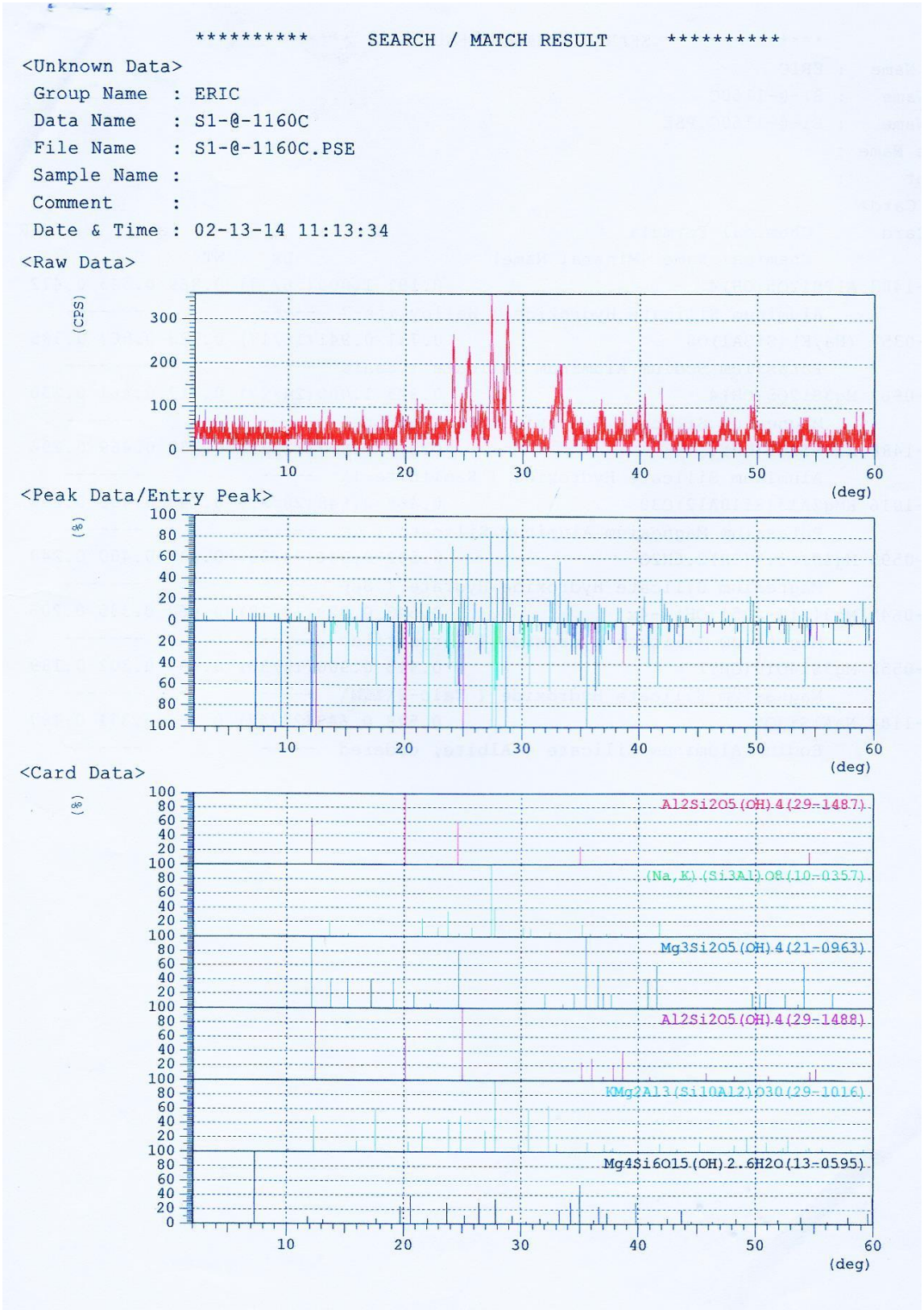
```
          ***  Basic Data Process  ***

 # Data Infomation
           Group                 : ERIC
           Data                  : S6-@-1140
           Sample Nmae           :
           Comment               :
           Date & Time           : 02-13-14 12:44:21

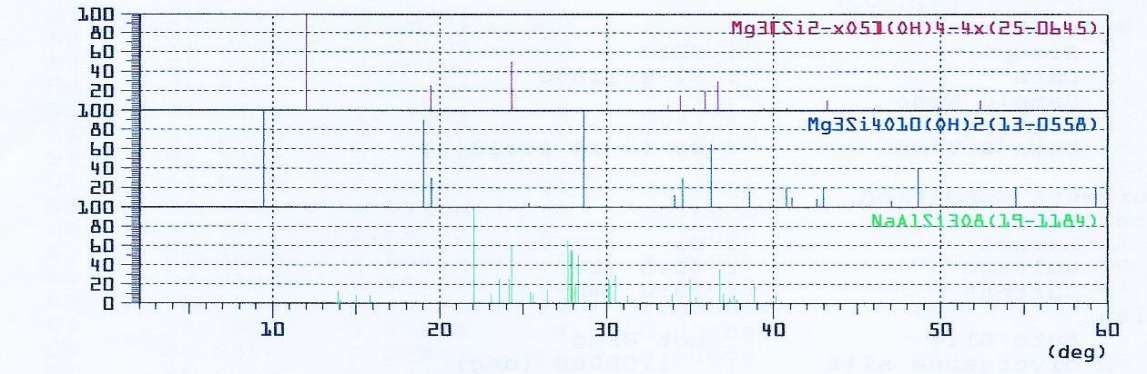
 # Measurement Condition
     X-ray tube
           target                : Cu
           voltage               : 40.0 (kV)
           current               : 30.0 (mA)
     Slits
           Auto Slit             : not Used
           divergence slit       :  1.00000 (deg)
           scatter slit          :  1.00000 (deg)
           receiving slit        :  0.30000(mm)
     Scanning
           drive axis            : Theta-2Theta
           scan range            :   2.0000 - 60.0000 (deg)
           scan mode             : Continuous Scan
           scan speed            :   8.0000 (deg/min)
           sampling pitch        :   0.0200 (deg)
           preset time           :   0.15 (sec)

 # Data Process Condition
     Smoothing                   [ AUTO ]
           smoothing points      : 11
     B.G.Subtruction             [ AUTO ]
           sampling points       : 11
           repeat times          : 30
     Ka1-a2 Separate             [ MANUAL ]
           Ka1 a2 ratio          : 50 (%)
     Peak Search                 [ AUTO ]
           differential points   : 11
           FWHM threhold         : 0.050 (deg)
           intensity threhold    : 30 (par mil)
           FWHM ratio  (n-1)/n   : 2
     System error Correction     [ YES ]
     Precise peak Correction     [ NO ]
```

Appendix 40: XRD Analysis of Sample 1 at 1160^{o}C



Appendix 41: XRD Analysis of Sample 1 at 1160^{o}C (continuation).




```
                **********     SEARCH / MATCH RESULT     **********
 Group Name   : ERIC
 Data Name    : S1-@-1160C
 File Name    : S1-@-1160C.PSE
 Sample Name  :
 Comment      :
<Entry Card>
No.   Card      Chemical Formula                      S        L          d     I     R
                Chemical Name (Mineral Name)                  Dx     WT%    S.G.
 1  29-1487 Al2Si2O5(OH)4                          0.191 1.000( 6/ 7) 0.869 0.543 0.472
             Aluminum Silicate Hydroxide ( Halloysite-7  -----          -------
 2  10-0357 (Na,K)(Si3Al)O8                        0.731 0.941(16/17) 0.723 0.567 0.385
             Potassium Sodium Aluminum Silicate ( Sanid  -----          -------
 3  21-0963 Mg3Si2O5(OH)4                          0.325 1.000(24/27) 0.717 0.461 0.330
             Magnesium Silicate Hydroxide ( Antigorite-  -----          -------
 4  29-1488 Al2Si2O5(OH)4                          0.276 0.857(12/21) 0.735 0.469 0.296
             Aluminum Silicate Hydroxide ( Kaolinite-1\  -----          -------
 5  29-1016 KMg2Al3(Si10Al2)O30                    0.468 0.848(28/33) 0.743 0.409 0.258
             Potassium Magnesium Aluminum Silicate ( Os  -----          -------
 6  13-0595 Mg4Si6O15(OH)2.6H2O                    0.532 0.944(34/39) 0.635 0.400 0.240
             Magnesium Silicate Hydroxide Hydrate ( Sep  -----          -------
 7  25-0645 Mg3[Si2-xO5](OH)4-4x                   0.543 0.929(13/18) 0.662 0.335 0.206
             Magnesium Silicate Hydroxide ( Chrysotile   -----          -------
 8  13-0558 Mg3Si4O10(OH)2                         0.415 0.900(18/30) 0.697 0.302 0.189
             Magnesium Silicate Hydroxide ( Talc-2\ITM\  -----          -------
 9  19-1184 NaAlSi3O8                              0.528 0.643(27/42) 0.786 0.371 0.187
             Sodium Aluminum Silicate ( Albite, ordered  -----          -------
```

Appendix 42: XRD Analysis of Sample 1 at 1160^{o}C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S1-@-1160C

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	51	28.6875	3.10932	100	0.25500	28	360
2	48	27.3200	3.26177	86	0.25000	24	323
3	40	24.0566	3.69635	71	0.19330	20	207

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	2.8400	31.08413	7	0.04000	2	9
2	3.1000	28.47763	4	0.00000	1	0
3	5.3000	16.66063	11	0.04000	3	18
4	5.6400	15.65701	4	0.00000	1	0
5	6.6800	13.22154	7	0.08000	2	15
6	6.9900	12.63586	7	0.06000	2	6
7	7.3000	12.09995	7	0.12000	2	16
8	7.5700	11.66897	7	0.10000	2	15
9	8.0000	11.04270	7	0.12000	2	25
10	8.9200	9.90573	7	0.12000	2	21
11	9.2200	9.58408	7	0.08000	2	12
12	10.1400	8.71649	4	0.00000	1	0
13	10.3100	8.57314	7	0.06000	2	19
14	10.8200	8.17016	4	0.00000	1	0
15	11.0700	7.98621	7	0.10000	2	18
16	11.4600	7.71529	4	0.00000	1	0
17	12.1300	7.29060	7	0.06000	2	8
18	12.3800	7.14393	4	0.00000	1	0
19	13.5400	6.53439	11	0.04000	3	15
20	13.9000	6.36595	4	0.00000	1	0
21	14.8250	5.97077	11	0.05000	3	15
22	14.9800	5.90933	7	0.08000	2	18
23	15.2300	5.81289	11	0.06000	3	26
24	15.7200	5.63278	4	0.00000	1	0
25	16.3700	5.41056	18	0.06000	5	43
26	17.0400	5.19930	4	0.00000	1	0
27	18.2900	4.84668	11	0.06000	3	19
28	18.5250	4.78572	7	0.09000	2	14
29	18.7400	4.73130	4	0.00000	1	0
30	19.4100	4.56947	25	0.18000	7	93
31	20.1350	4.40654	14	0.11000	4	31
32	20.9600	4.23492	18	0.08000	5	30
33	21.4000	4.14883	18	0.10000	5	31
34	21.9200	4.05157	11	0.08000	3	27
35	22.0800	4.02258	14	0.08000	4	23
36	22.5400	3.94151	7	0.04000	2	8
37	22.8950	3.88119	11	0.03000	3	9
38	23.2200	3.82760	7	0.04000	2	8
39	23.4900	3.78421	7	0.10000	2	10
40	24.0566	3.69635	71	0.19330	20	207
41	24.6000	3.61592	14	0.12000	4	27
42	24.7600	3.59291	36	0.16000	10	74
43	25.0600	3.55058	21	0.16000	6	59
44	25.3550	3.50993	64	0.37000	18	310
45	25.9300	3.43339	18	0.10000	5	36
46	26.4700	3.36456	14	0.22000	4	50
47	26.7800	3.32631	4	0.00000	1	0
48	27.3200	3.26177	86	0.25000	24	323
49	27.8450	3.20146	32	0.21000	9	110
50	28.3266	3.14811	39	0.14670	11	96

Appendix 43: XRD Analysis of Sample 1 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	28.6875	3.10932	100	0.25500	28	360
52	28.9600	3.08068	14	0.12000	4	34
53	29.9200	2.98399	7	0.12000	2	27
54	30.5500	2.92387	7	0.30000	2	41
55	31.0400	2.87882	7	0.12000	2	17
56	31.2800	2.85728	7	0.12000	2	24
57	32.3750	2.76309	21	0.13000	6	35
58	32.5600	2.74782	21	0.24000	6	70
59	32.7600	2.73150	39	0.16000	11	117
60	33.1900	2.69708	57	0.22000	16	221
61	33.5600	2.66819	4	0.00000	1	0
62	33.9100	2.64145	21	0.18000	6	97
63	34.1600	2.62268	14	0.00000	4	0
64	34.7600	2.57878	4	0.00000	1	0
65	34.9850	2.56270	11	0.03000	3	15
66	35.3600	2.53638	4	0.00000	1	0
67	36.5350	2.45746	18	0.13000	5	41
68	36.7200	2.44550	14	0.22000	4	55
69	36.9833	2.42869	14	0.04670	4	13
70	38.1300	2.35825	14	0.14000	4	47
71	38.4000	2.34229	14	0.12000	4	36
72	39.8200	2.26197	11	0.08000	3	25
73	40.4050	2.23056	25	0.11000	7	44
74	41.8750	2.15559	32	0.15000	9	78
75	42.1083	2.14419	18	0.11670	5	34
76	42.3500	2.13251	14	0.10000	4	24
77	42.5800	2.12152	7	0.04000	2	6
78	42.7400	2.11395	4	0.00000	1	0
79	43.2700	2.08928	7	0.10000	2	15
80	43.4800	2.07967	7	0.12000	2	21
81	43.9500	2.05851	7	0.10000	2	13
82	44.1400	2.05009	4	0.00000	1	0
83	45.8700	1.97672	7	0.06000	2	16
84	46.2600	1.96096	4	0.00000	1	0
85	47.0000	1.93179	11	0.12000	3	38
86	47.3200	1.91947	4	0.00000	1	0
87	47.5900	1.90921	7	0.06000	2	14
88	47.9800	1.89460	4	0.00000	1	0
89	49.0800	1.85468	7	0.04000	2	10
90	49.4400	1.84201	25	0.16000	7	66
91	49.6933	1.83321	21	0.17330	6	60
92	50.3200	1.81184	4	0.00000	1	0
93	50.8600	1.79386	7	0.04000	2	14
94	51.6800	1.76731	4	0.00000	1	0
95	52.1400	1.75280	7	0.04000	2	7
96	52.4400	1.74347	7	0.20000	2	38
97	53.6100	1.70815	7	0.10000	2	16
98	53.9400	1.69848	11	0.16000	3	46
99	54.2600	1.68921	11	0.02000	3	7
100	54.6000	1.67950	4	0.00000	1	0
101	56.6900	1.62244	7	0.06000	2	14
102	56.8700	1.61773	7	0.06000	2	13
103	57.1000	1.61176	4	0.00000	1	0
104	57.4500	1.60277	14	0.18000	4	43
105	57.7200	1.59591	4	0.00000	1	0
106	58.5100	1.57623	7	0.06000	2	7
107	59.0300	1.56358	21	0.14000	6	53
108	59.3000	1.55710	4	0.00000	1	0
109	59.8100	1.54504	11	0.14000	3	36

Appendix 44: XRD Analysis of Sample 1 at 1160^{o}C (continuation).

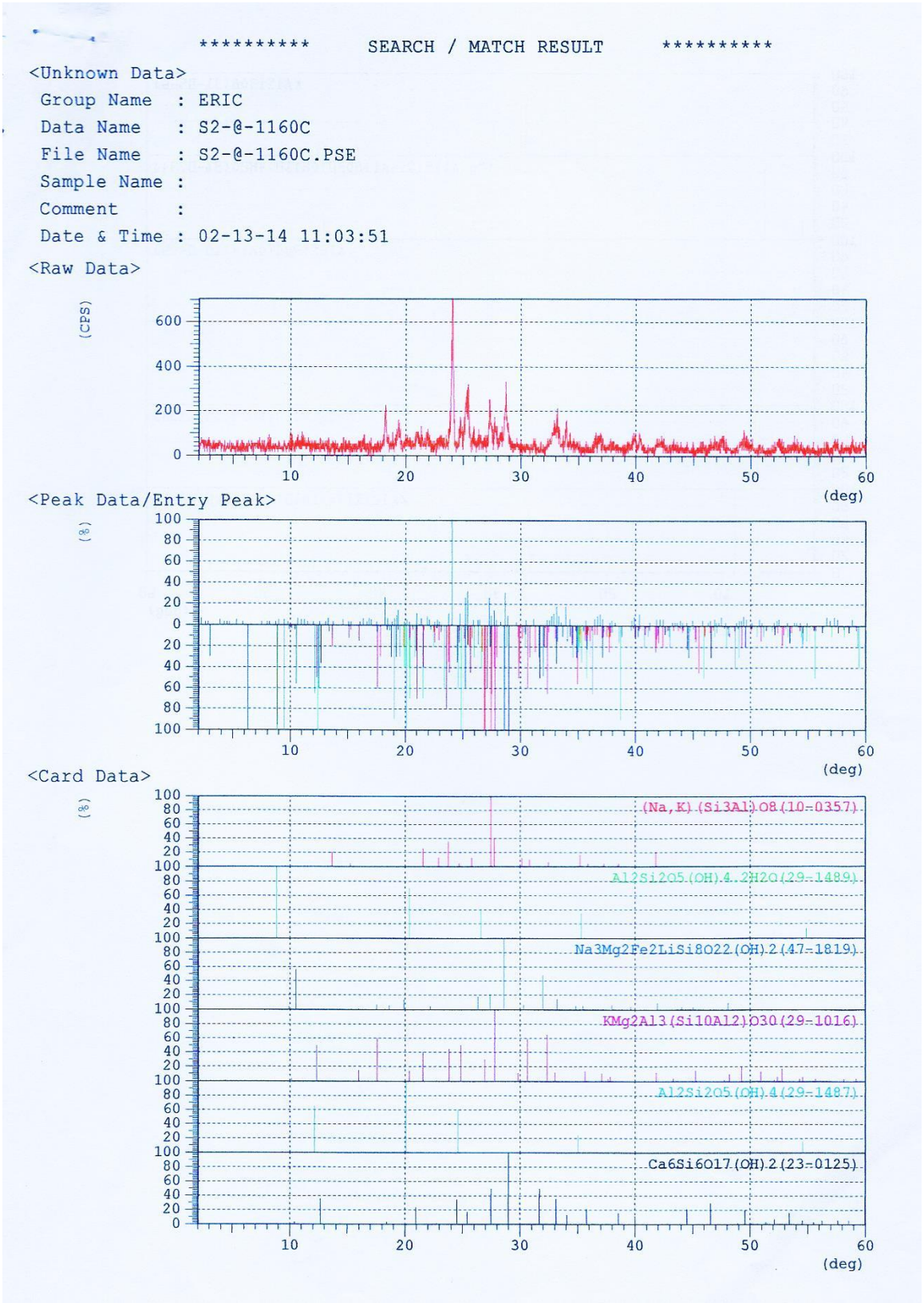
```
          ***  Basic Data Process  ***

# Data Infomation
          Group                 : ERIC
          Data                  : S1-@-1160C
          Sample Nmae           :
          Comment               :
          Date & Time           : 02-13-14 11:13:34

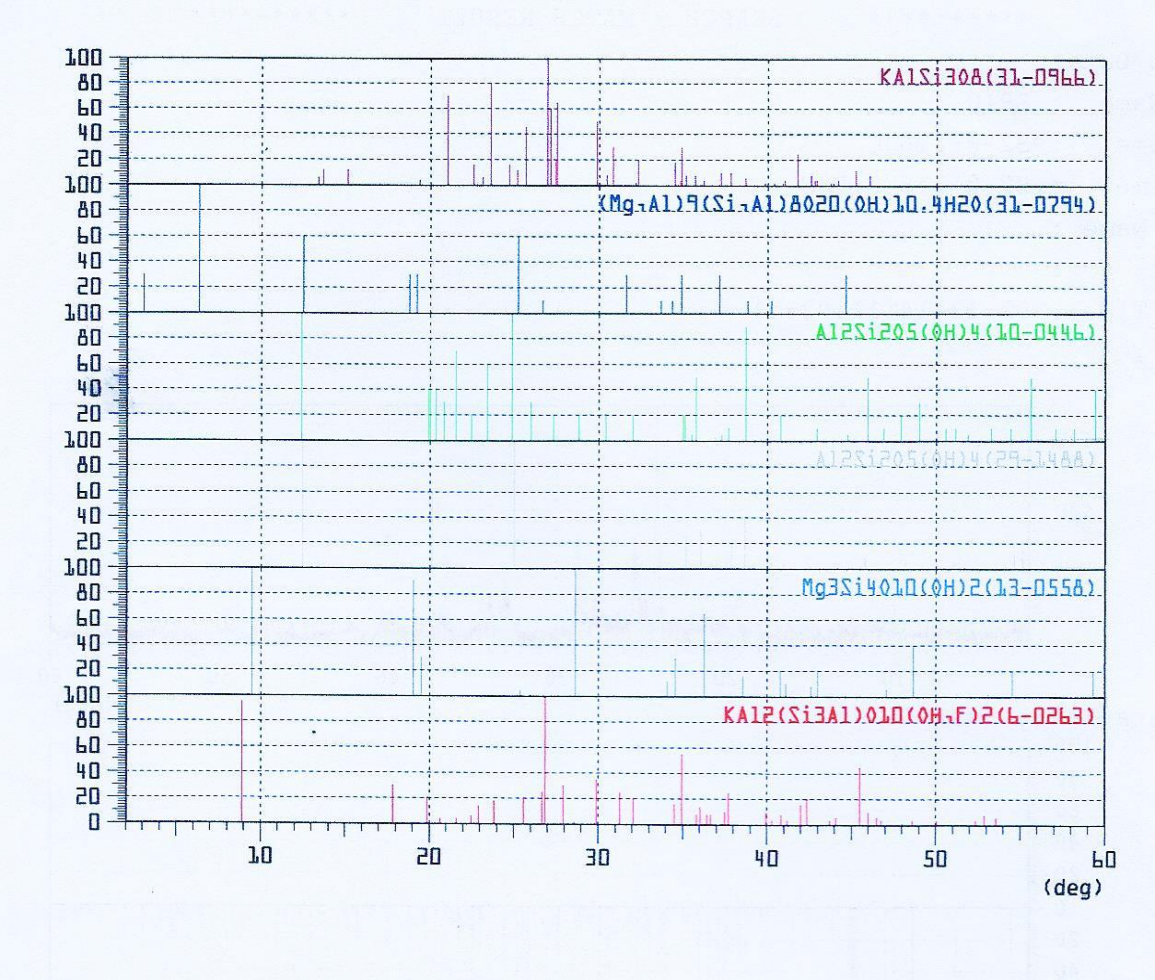
# Measurement Condition
    X-ray tube
          target                : Cu
          voltage               : 40.0 (kV)
          current               : 30.0 (mA)
    Slits
          Auto Slit             : not Used
          divergence slit       :   1.00000 (deg)
          scatter slit          :   1.00000 (deg)
          receiving slit        :   0.30000(mm)
    Scanning
          drive axis            : Theta-2Theta
          scan range            :    2.0000 - 60.0000 (deg)
          scan mode             : Continuous Scan
          scan speed            :    8.0000 (deg/min)
          sampling pitch        :    0.0200 (deg)
          preset time           :    0.15 (sec)

# Data Process Condition
    Smoothing                   [ AUTO ]
          smoothing points      : 15
    B.G.Subtruction             [ AUTO ]
          sampling points       : 17
          repeat times          : 30
    Ka1-a2 Separate             [ MANUAL ]
          Ka1 a2 ratio          : 50 (%)
    Peak Search                 [ AUTO ]
          differential points   : 13
          FWHM threhold         : 0.050 (deg)
          intensity threhold    : 30 (par mil)
          FWHM ratio  (n-1)/n   : 2
    System error Correction     [ YES ]
    Precise peak Correction     [ NO ]
```

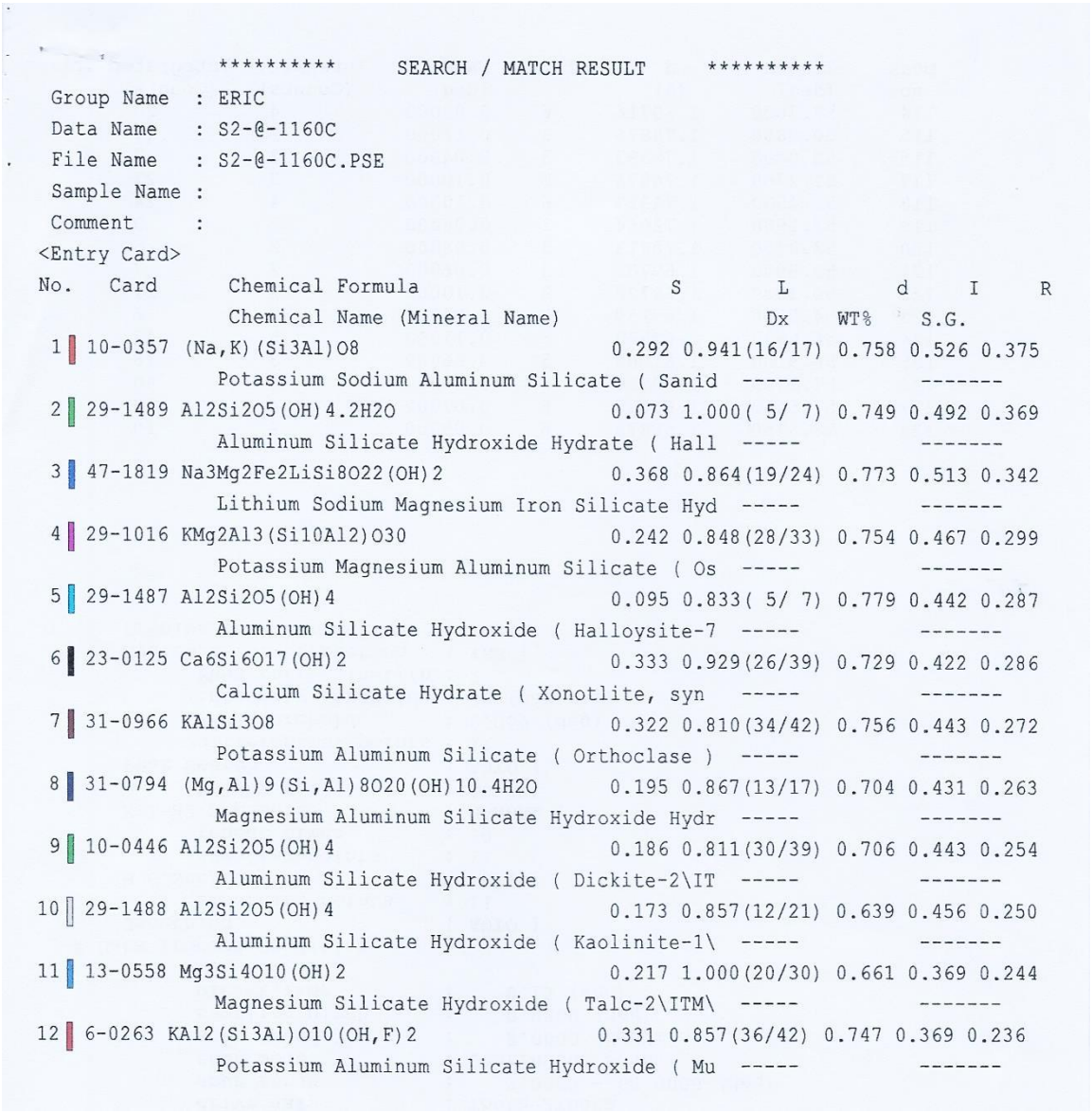
Appendix 45: XRD Analysis of Sample 2 at 1160^{o}C.



Appendix 46: XRD Analysis of Sample 2 at 1160^{o}C (continuation).



Appendix 47: XRD Analysis of Sample 2 at 1160^{o}C (continuation).



```
                **********     SEARCH / MATCH RESULT     **********
 Group Name  : ERIC
 Data Name   : S2-@-1160C
 File Name   : S2-@-1160C.PSE
 Sample Name :
 Comment     :
<Entry Card>
No.   Card      Chemical Formula                        S        L          d     I     R
                Chemical Name (Mineral Name)                    Dx    WT%    S.G.
 1  10-0357  (Na,K)(Si3Al)O8                        0.292 0.941(16/17) 0.758 0.526 0.375
              Potassium Sodium Aluminum Silicate ( Sanid  -----          -------
 2  29-1489  Al2Si2O5(OH)4.2H2O                     0.073 1.000( 5/ 7) 0.749 0.492 0.369
              Aluminum Silicate Hydroxide Hydrate ( Hall  -----          -------
 3  47-1819  Na3Mg2Fe2LiSi8O22(OH)2                 0.368 0.864(19/24) 0.773 0.513 0.342
              Lithium Sodium Magnesium Iron Silicate Hyd  -----          -------
 4  29-1016  KMg2Al3(Si10Al2)O30                    0.242 0.848(28/33) 0.754 0.467 0.299
              Potassium Magnesium Aluminum Silicate ( Os  -----          -------
 5  29-1487  Al2Si2O5(OH)4                          0.095 0.833( 5/ 7) 0.779 0.442 0.287
              Aluminum Silicate Hydroxide ( Halloysite-7  -----          -------
 6  23-0125  Ca6Si6O17(OH)2                         0.333 0.929(26/39) 0.729 0.422 0.286
              Calcium Silicate Hydrate ( Xonotlite, syn   -----          -------
 7  31-0966  KAlSi3O8                               0.322 0.810(34/42) 0.756 0.443 0.272
              Potassium Aluminum Silicate ( Orthoclase )  -----          -------
 8  31-0794  (Mg,Al)9(Si,Al)8O20(OH)10.4H2O         0.195 0.867(13/17) 0.704 0.431 0.263
              Magnesium Aluminum Silicate Hydroxide Hydr  -----          -------
 9  10-0446  Al2Si2O5(OH)4                          0.186 0.811(30/39) 0.706 0.443 0.254
              Aluminum Silicate Hydroxide ( Dickite-2\IT  -----          -------
10  29-1488  Al2Si2O5(OH)4                          0.173 0.857(12/21) 0.639 0.456 0.250
              Aluminum Silicate Hydroxide ( Kaolinite-1\  -----          -------
11  13-0558  Mg3Si4O10(OH)2                         0.217 1.000(20/30) 0.661 0.369 0.244
              Magnesium Silicate Hydroxide ( Talc-2\ITM\  -----          -------
12  6-0263   KAl2(Si3Al)O10(OH,F)2                  0.331 0.857(36/42) 0.747 0.369 0.236
              Potassium Aluminum Silicate Hydroxide ( Mu  -----          -------
```

Appendix 48: XRD Analysis of Sample 2 at 1160^{o}C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S2-@-1160C

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	52	24.0283	3.70064	100	0.18330	65	643
2	55	25.4000	3.50381	32	0.11200	21	154
3	64	28.6483	3.11349	31	0.19670	20	179

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	2.2700	38.88795	6	0.14000	4	44
2	2.6200	33.69373	3	0.04000	2	5
3	2.8700	30.75927	3	0.10000	2	12
4	3.8550	22.90182	5	0.03000	3	7
5	4.2750	20.65271	3	0.07000	2	8
6	4.4900	19.66425	3	0.10000	2	11
7	4.6900	18.82613	3	0.10000	2	14
8	5.3200	16.59804	5	0.04000	3	14
9	5.8700	15.04404	3	0.06000	2	12
10	7.5050	11.76989	3	0.03000	2	7
11	7.9800	11.07033	3	0.08000	2	11
12	8.1900	10.78694	3	0.06000	2	10
13	8.7300	10.12088	3	0.06000	2	9
14	8.9800	9.83968	5	0.04000	3	13
15	9.4600	9.34146	5	0.04000	3	16
16	9.8000	9.01812	3	0.04000	2	5
17	10.6300	8.31577	6	0.14000	4	44
18	10.9400	8.08081	5	0.04000	3	19
19	11.2150	7.88328	5	0.03000	3	9
20	11.5000	7.68854	3	0.08000	2	14
21	12.0100	7.36317	3	0.06000	2	11
22	12.5400	7.05313	3	0.04000	2	8
23	13.0500	6.77862	3	0.06000	2	11
24	13.2900	6.65674	6	0.06000	4	14
25	13.7050	6.45609	3	0.05000	2	10
26	14.5650	6.07676	5	0.05000	3	10
27	15.0800	5.87037	3	0.08000	2	11
28	15.2650	5.79964	3	0.09000	2	11
29	16.0350	5.52283	3	0.05000	2	8
30	16.3500	5.41713	6	0.10000	4	26
31	16.6550	5.31861	3	0.09000	2	9
32	16.8500	5.25749	6	0.06000	4	18
33	17.0250	5.20384	5	0.05000	3	14
34	17.4900	5.06653	3	0.06000	2	15
35	18.2200	4.86514	26	0.14660	17	142
36	18.4400	4.80759	6	0.08000	4	21
37	18.8300	4.70889	3	0.06000	2	9
38	19.0650	4.65137	6	0.07000	4	15
39	19.2400	4.60946	9	0.16000	6	41
40	19.3600	4.58116	14	0.20000	9	77
41	19.9100	4.45583	3	0.06000	2	12
42	20.1200	4.40979	5	0.08000	3	18
43	20.3500	4.36047	3	0.10000	2	15
44	20.9800	4.23093	11	0.12000	7	77
45	21.3800	4.15267	6	0.08000	4	18
46	21.8900	4.05706	8	0.10000	5	35
47	22.0900	4.02078	5	0.10000	3	20
48	22.5400	3.94151	3	0.04000	2	6
49	22.7550	3.90475	5	0.07000	3	17
50	22.9400	3.87368	3	0.04000	2	16

Appendix 49: XRD Analysis of Sample 2 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	23.7600	3.74182	11	0.04000	7	30
52	24.0283	3.70064	100	0.18330	65	643
53	24.7416	3.59554	11	0.18330	7	73
54	25.2000	3.53117	26	0.16000	17	172
55	25.4000	3.50381	32	0.11200	21	154
56	25.8800	3.43991	5	0.04000	3	10
57	26.2800	3.38845	5	0.06000	3	13
58	26.4550	3.36643	5	0.05000	3	16
59	27.2740	3.26717	26	0.13200	17	124
60	27.4400	3.24778	9	0.08000	6	33
61	27.7233	3.21523	14	0.08670	9	47
62	27.9000	3.19527	5	0.04000	3	15
63	28.2400	3.15757	3	0.08000	2	13
64	28.6483	3.11349	31	0.19670	20	179
65	28.8600	3.09113	9	0.10000	6	45
66	29.6500	3.01054	3	0.18000	2	23
67	30.4700	2.93136	5	0.06000	3	18
68	31.1100	2.87250	6	0.10000	4	24
69	32.2900	2.77017	5	0.06000	3	22
70	32.5150	2.75152	5	0.03000	3	6
71	32.7050	2.73596	9	0.11000	6	32
72	32.8800	2.72180	11	0.16000	7	61
73	33.1100	2.70342	18	0.16660	12	100
74	33.3258	2.68640	9	0.11830	6	36
75	33.5300	2.67051	3	0.06000	2	13
76	33.9166	2.64095	18	0.11330	12	80
77	34.2800	2.61378	6	0.08000	4	20
78	35.6800	2.51436	5	0.04000	3	16
79	36.4400	2.46365	6	0.08000	4	35
80	36.7033	2.44658	9	0.11330	6	37
81	36.8933	2.43441	11	0.09330	7	37
82	37.1100	2.42069	6	0.08000	4	16
83	37.7500	2.38111	3	0.10000	2	12
84	37.9200	2.37083	5	0.04000	3	16
85	38.2100	2.35350	3	0.06000	2	16
86	39.6800	2.26963	5	0.04000	3	15
87	39.8700	2.25925	8	0.14000	5	47
88	40.3150	2.23533	11	0.13000	7	61
89	41.2200	2.18832	3	0.04000	2	10
90	41.8000	2.15929	6	0.20000	4	49
91	42.1200	2.14362	6	0.20000	4	46
92	42.4100	2.12963	6	0.06000	4	16
93	43.2000	2.09250	3	0.04000	2	9
94	43.4000	2.08332	3	0.04000	2	5
95	43.6000	2.07422	3	0.04000	2	8
96	43.8800	2.06164	5	0.04000	3	15
97	44.0500	2.05407	3	0.06000	2	10
98	44.4100	2.03825	3	0.14000	2	27
99	44.9400	2.01544	5	0.04000	3	14
100	45.8250	1.97855	5	0.09000	3	22
101	46.3200	1.95856	3	0.08000	2	14
102	46.5000	1.95139	5	0.04000	3	15
103	46.8700	1.93685	6	0.10000	4	24
104	47.0100	1.93140	3	0.10000	2	10
105	47.2100	1.92369	3	0.10000	2	13
106	47.4400	1.91490	8	0.12000	5	54
107	47.8050	1.90112	3	0.07000	2	14
108	48.7600	1.86610	3	0.12000	2	13
109	48.9900	1.85787	5	0.10000	3	26
110	49.1800	1.85114	5	0.04000	3	10
111	49.3850	1.84393	9	0.11000	6	37
112	49.6300	1.83540	6	0.10000	4	22
113	50.0800	1.81996	3	0.04000	2	7

Appendix 50: XRD Analysis of Sample 2 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
114	50.7600	1.79716	6	0.08000	4	20
115	50.9850	1.78976	3	0.11000	2	11
116	52.0400	1.75593	3	0.04000	2	7
117	52.2700	1.74874	5	0.10000	3	22
118	52.4500	1.74317	6	0.10000	4	24
119	53.1900	1.72064	3	0.06000	2	9
120	53.8150	1.70213	3	0.05000	2	5
121	53.9900	1.69702	3	0.06000	2	7
122	54.3300	1.68720	3	0.10000	2	21
123	54.8800	1.67159	3	0.04000	2	6
124	56.5850	1.62520	8	0.11000	5	43
125	56.9100	1.61669	5	0.06000	3	16
126	57.2700	1.60738	8	0.10000	5	40
127	57.5450	1.60035	6	0.07000	4	19
128	58.8150	1.56878	6	0.09000	4	19

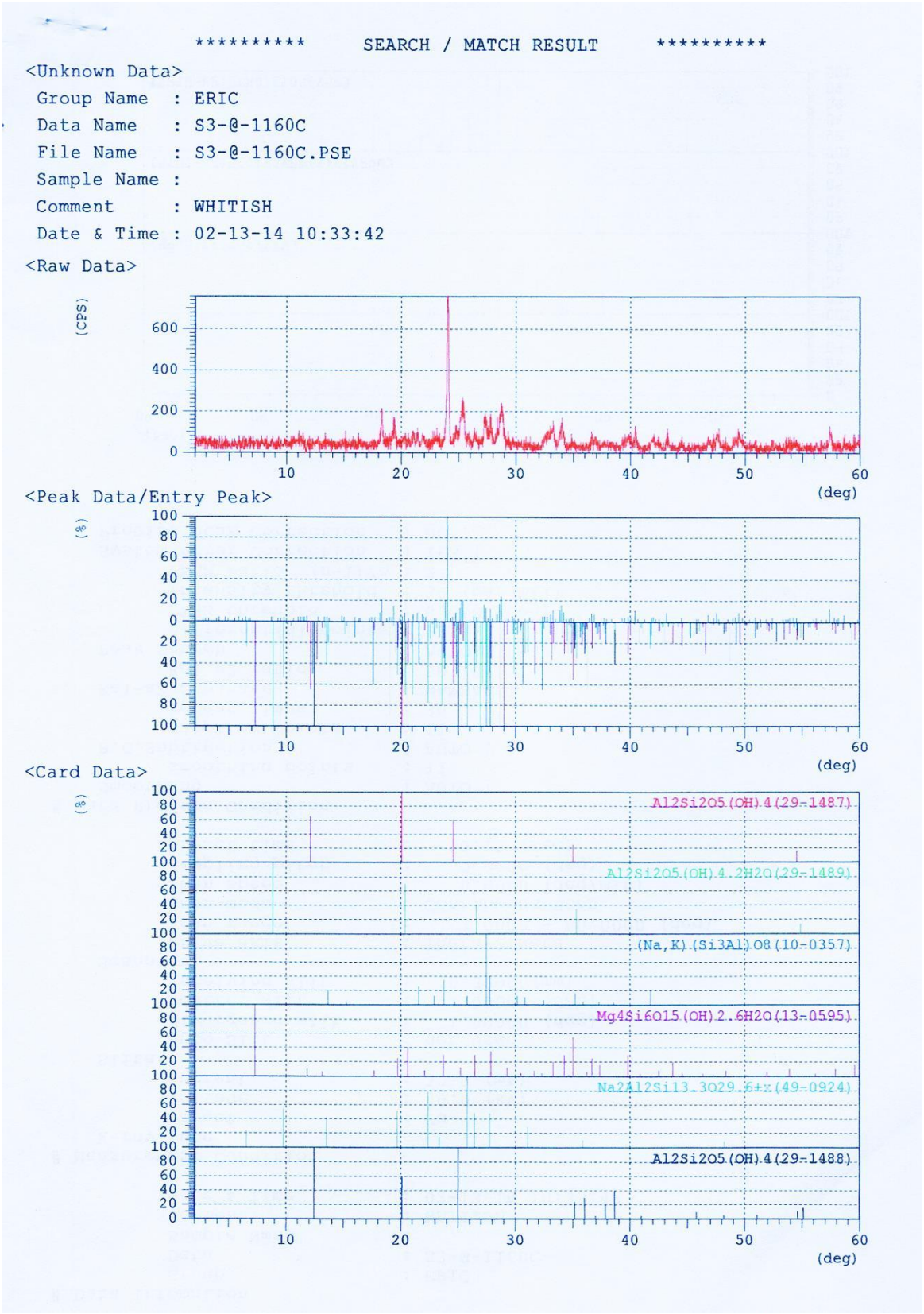
```
          ***  Basic Data Process  ***

  # Data Infomation
          Group               : ERIC
          Data                : S2-@-1160C
          Sample Nmae         :
          Comment             :
          Date & Time         : 02-13-14 11:03:51

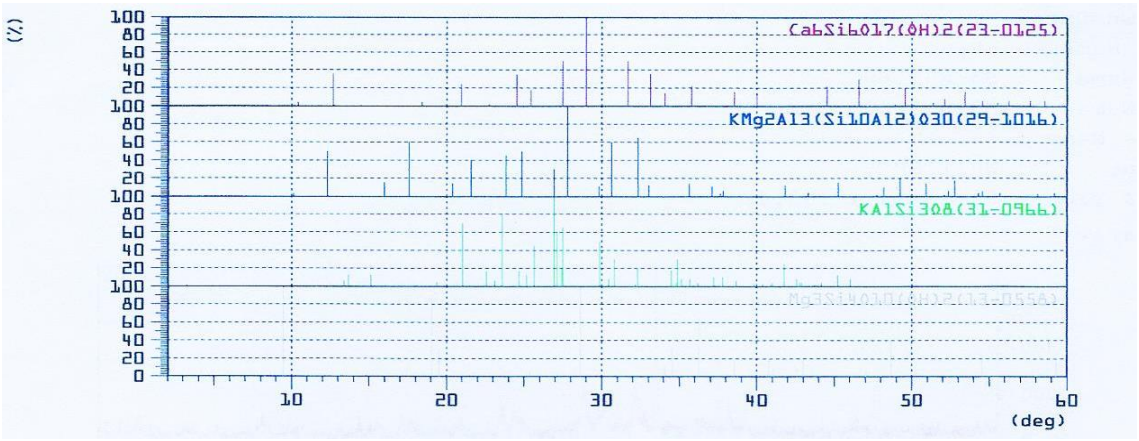
  # Measurement Condition
    X-ray tube
          target              : Cu
          voltage             : 40.0 (kV)
          current             : 30.0 (mA)
    Slits
          Auto Slit           : not Used
          divergence slit     :   1.00000 (deg)
          scatter slit        :   1.00000 (deg)
          receiving slit      :   0.30000(mm)
    Scanning
          drive axis          : Theta-2Theta
          scan range          :    2.0000 - 60.0000 (deg)
          scan mode           : Continuous Scan
          scan speed          :    8.0000 (deg/min)
          sampling pitch      :    0.0200 (deg)
          preset time         :    0.15 (sec)

  # Data Process Condition
    Smoothing                 [ AUTO ]
          smoothing points    : 11
    B.G.Subtruction           [ AUTO ]
          sampling points     : 11
          repeat times        : 30
    Ka1-a2 Separate           [ MANUAL ]
          Ka1 a2 ratio        : 50 (%)
    Peak Search               [ AUTO ]
          differential points : 11
          FWHM threhold       : 0.050 (deg)
          intensity threhold  : 30 (par mil)
          FWHM ratio  (n-1)/n : 2
    System error Correction   [ YES ]
    Precise peak Correction   [ NO ]
```

Appendix 51: XRD Analysis of Sample 3 at 1160^{o}C.



Appendix 52: XRD Analysis of Sample 3 at 1160^{o}C (continuation).




```
                **********     SEARCH / MATCH RESULT     **********
 Group Name  : ERIC
 Data Name   : S3-@-1160C
 File Name   : S3-@-1160C.PSE
 Sample Name :
 Comment     : WHITISH
<Entry Card>
No.   Card      Chemical Formula                    S       L         d     I     R
                Chemical Name (Mineral Name)                Dx    WT%    S.G.
 1 29-1487 Al2Si2O5(OH)4                       0.082 1.000( 6/ 7) 0.829 0.612 0.507
           Aluminum Silicate Hydroxide ( Halloysite-7  -----          -------
 2 29-1489 Al2Si2O5(OH)4.2H2O                  0.089 1.000( 5/ 7) 0.670 0.644 0.431
           Aluminum Silicate Hydroxide Hydrate ( Hall  -----          -------
 3 10-0357 (Na,K)(Si3Al)O8                     0.321 0.941(16/17) 0.732 0.508 0.350
           Potassium Sodium Aluminum Silicate ( Sanid  -----          -------
 4 13-0595 Mg4Si6O15(OH)2.6H2O                 0.293 0.972(35/39) 0.739 0.457 0.329
           Magnesium Silicate Hydroxide Hydrate ( Sep  -----          -------
 5 49-0924 Na2Al2Si13.3O29.6+x                 0.133 0.786(11/14) 0.674 0.606 0.321
           Sodium Aluminum Silicate ( Mordenite, syn   -----          -------
 6 29-1488 Al2Si2O5(OH)4                       0.145 1.000(14/21) 0.629 0.506 0.319
           Aluminum Silicate Hydroxide ( Kaolinite-1\  -----          -------
 7 23-0125 Ca6Si6O17(OH)2                      0.293 0.964(27/39) 0.717 0.450 0.311
           Calcium Silicate Hydrate ( Xonotlite, syn   -----          -------
 8 29-1016 KMg2Al3(Si10Al2)O30                 0.241 0.848(28/33) 0.762 0.440 0.285
           Potassium Magnesium Aluminum Silicate ( Os  -----          -------
 9 31-0966 KAlSi3O8                            0.279 0.810(34/42) 0.741 0.468 0.281
           Potassium Aluminum Silicate ( Orthoclase )  -----          -------
10 13-0558 Mg3Si4O10(OH)2                      0.170 0.950(19/30) 0.737 0.361 0.253
           Magnesium Silicate Hydroxide ( Talc-2\ITM\  -----          -------
```

Appendix 53: XRD Analysis of Sample 3 at 1160^{o}C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S3-@-1160C

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	55	24.0348	3.69965	100	0.16680	75	647
2	71	28.7400	3.10376	24	0.20000	18	188
3	38	18.2833	4.84844	20	0.14000	15	122

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	2.7600	31.98493	3	0.04000	2	8
2	2.9600	29.82422	3	0.04000	2	8
3	3.2300	27.33175	3	0.14000	2	18
4	3.9200	22.52222	3	0.04000	2	7
5	4.1200	21.42934	4	0.04000	3	10
6	4.6600	18.94726	4	0.04000	3	21
7	4.9200	17.94655	3	0.04000	2	9
8	5.5000	16.05523	3	0.08000	2	16
9	5.8200	15.17317	4	0.04000	3	17
10	6.1500	14.35972	3	0.10000	2	19
11	6.4600	13.67131	4	0.04000	3	11
12	6.7000	13.18212	3	0.12000	2	15
13	6.8600	12.87502	3	0.12000	2	22
14	8.7200	10.13247	4	0.04000	3	11
15	9.2900	9.51202	3	0.06000	2	9
16	9.5100	9.29246	3	0.10000	2	12
17	9.8000	9.01812	4	0.04000	3	14
18	10.0000	8.83820	3	0.08000	2	17
19	10.6400	8.30797	3	0.04000	2	9
20	11.0400	8.00784	4	0.04000	3	14
21	11.2400	7.86580	4	0.04000	3	20
22	11.4500	7.72200	4	0.10000	3	25
23	11.5850	7.63232	4	0.03000	3	8
24	11.8100	7.48741	5	0.10000	4	32
25	12.7600	6.93202	4	0.06000	3	17
26	13.3300	6.63685	4	0.06000	3	20
27	13.5250	6.54160	4	0.05000	3	11
28	13.7200	6.44906	3	0.08000	2	16
29	14.8400	5.96476	5	0.08000	4	19
30	15.0200	5.89368	4	0.04000	3	13
31	15.9800	5.54172	4	0.04000	3	8
32	16.3200	5.42702	3	0.16000	2	17
33	16.4400	5.38768	3	0.12000	2	18
34	17.2600	5.13352	3	0.08000	2	16
35	17.4350	5.08238	4	0.03000	3	10
36	17.7100	5.00408	4	0.06000	3	16
37	18.1400	4.88642	8	0.06000	6	27
38	18.2833	4.84844	20	0.14000	15	122
39	18.7450	4.73005	3	0.03000	2	6
40	18.9800	4.67201	4	0.04000	3	9
41	19.2200	4.61421	4	0.10000	3	19
42	19.3700	4.57881	15	0.16000	11	85
43	19.9000	4.45805	3	0.04000	2	7
44	20.1000	4.41414	5	0.04000	4	13
45	20.2850	4.37430	8	0.13000	6	51
46	20.6200	4.30398	3	0.08000	2	11
47	21.0533	4.21637	7	0.06670	5	28
48	21.4100	4.14692	8	0.10000	6	35
49	21.8500	4.06440	7	0.10000	5	26
50	21.9800	4.04065	5	0.12000	4	24

Appendix 54: XRD Analysis of Sample 3 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	22.1600	4.00823	4	0.04000	3	10
52	22.9000	3.88036	3	0.04000	2	7
53	23.0600	3.85379	3	0.12000	2	23
54	23.8600	3.72636	20	0.08000	15	88
55	24.0348	3.69965	100	0.16680	75	647
56	24.3150	3.65765	3	0.03000	2	3
57	24.4800	3.63337	4	0.02000	3	6
58	24.7600	3.59291	8	0.12000	6	42
59	25.1400	3.53946	13	0.12000	10	123
60	25.2800	3.52017	20	0.00000	15	0
61	25.3800	3.50653	20	0.20000	15	166
62	26.3400	3.38087	5	0.00000	4	0
63	26.5800	3.35088	4	0.04000	3	22
64	27.2400	3.27117	16	0.12000	12	72
65	27.3800	3.25476	13	0.10000	10	61
66	27.6200	3.22702	3	0.08000	2	14
67	27.7766	3.20918	12	0.08670	9	46
68	27.9550	3.18911	3	0.03000	2	3
69	28.3600	3.14448	7	0.12000	5	40
70	28.5800	3.12077	17	0.12000	13	87
71	28.7400	3.10376	24	0.20000	18	188
72	29.3550	3.04012	3	0.03000	2	6
73	29.7100	3.00460	5	0.10000	4	20
74	29.8700	2.98887	3	0.06000	2	7
75	30.2300	2.95409	3	0.10000	2	17
76	30.8150	2.89933	3	0.03000	2	3
77	31.2000	2.86442	4	0.08000	3	18
78	32.3100	2.76851	5	0.06000	4	21
79	32.5133	2.75166	5	0.09330	4	22
80	32.7300	2.73393	5	0.16660	4	36
81	33.0200	2.71058	7	0.24000	5	63
82	33.2266	2.69420	11	0.13330	8	50
83	33.4400	2.67749	3	0.04000	2	4
84	33.8400	2.64675	11	0.18000	8	67
85	34.0000	2.63466	16	0.12000	12	76
86	34.1800	2.62120	9	0.08000	7	31
87	34.3800	2.60640	3	0.08000	2	16
88	34.6500	2.58671	3	0.06000	2	9
89	34.8400	2.57304	4	0.12000	3	25
90	35.0500	2.55810	3	0.06000	2	13
91	36.4250	2.46463	4	0.03000	3	11
92	36.6200	2.45195	8	0.10000	6	35
93	36.8400	2.43781	7	0.04000	5	29
94	37.7000	2.38415	4	0.12000	3	34
95	37.9400	2.36962	3	0.08000	2	12
96	38.3500	2.34523	3	0.06000	2	10
97	39.2600	2.29294	4	0.04000	3	11
98	39.4750	2.28094	4	0.07000	3	15
99	39.8150	2.26224	5	0.11000	4	36
100	40.0150	2.25140	4	0.03000	3	6
101	40.4116	2.23021	11	0.12330	8	50
102	40.6400	2.21820	3	0.04000	2	6
103	41.2400	2.18731	3	0.04000	2	9
104	41.8500	2.15682	4	0.14000	3	24
105	42.0000	2.14947	5	0.08000	4	17
106	42.2150	2.13902	3	0.05000	2	9
107	42.5000	2.12533	3	0.04000	2	11
108	43.1700	2.09388	9	0.14000	7	73
109	44.4600	2.03608	4	0.04000	3	18
110	45.7600	1.98121	3	0.08000	2	14
111	45.9400	1.97387	3	0.04000	2	6
112	46.7800	1.94036	7	0.12000	5	42
113	47.2650	1.92158	4	0.09000	3	23

Appendix 55: XRD Analysis of Sample 3 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
114	47.5150	1.91205	9	0.15000	7	66
115	47.7000	1.90506	3	0.00000	2	0
116	48.4950	1.87567	3	0.07000	2	8
117	48.9450	1.85948	4	0.05000	3	14
118	49.1600	1.85185	5	0.08000	4	24
119	49.4600	1.84131	9	0.12000	7	48
120	49.7700	1.83057	4	0.10000	3	20
121	50.3800	1.80982	3	0.04000	2	7
122	50.8850	1.79304	5	0.11000	4	26
123	51.3200	1.77886	3	0.12000	2	20
124	51.8400	1.76223	3	0.04000	2	9
125	52.3800	1.74533	4	0.12000	3	28
126	53.3000	1.71735	3	0.04000	2	6
127	53.5600	1.70963	3	0.08000	2	9
128	53.7200	1.70491	3	0.08000	2	9
129	53.8700	1.70052	4	0.10000	3	19
130	54.2200	1.69037	5	0.08000	4	22
131	54.4900	1.68263	4	0.10000	3	21
132	55.9900	1.64105	3	0.06000	2	9
133	56.1900	1.63568	4	0.10000	3	21
134	56.6250	1.62415	3	0.09000	2	11
135	57.0900	1.61202	3	0.06000	2	10
136	57.3800	1.60456	13	0.16000	10	85
137	57.7550	1.59503	5	0.05000	4	16
138	58.0000	1.58887	3	0.08000	2	15
139	58.1300	1.58563	4	0.06000	3	17
140	58.6200	1.57354	7	0.06000	5	21
141	59.8500	1.54410	3	0.10000	2	14

Appendix 56: XRD Analysis of Sample 3 at 1160^{o}C (continuation).

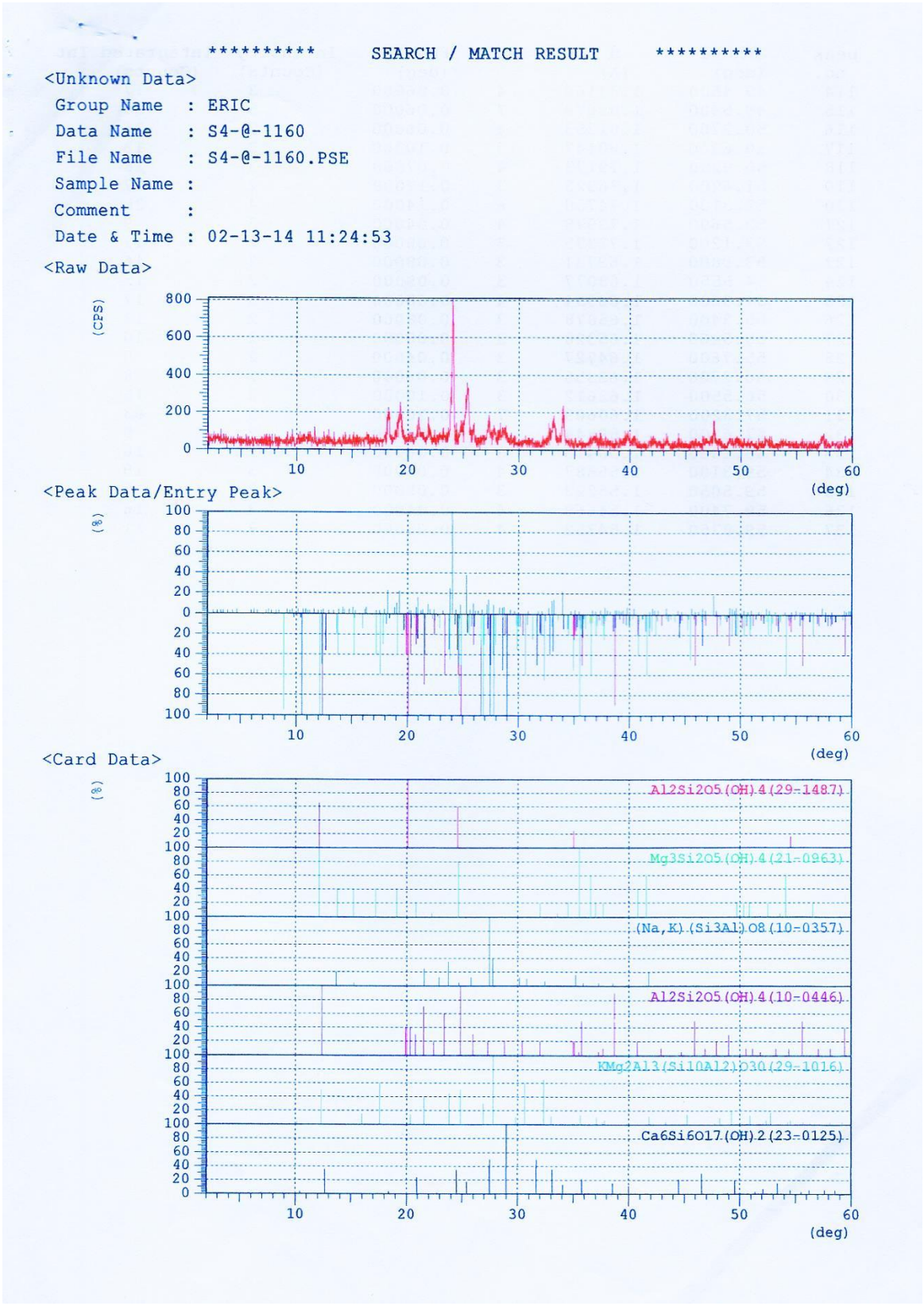
```
          ***  Basic Data Process  ***

 # Data Infomation
          Group                : ERIC
          Data                 : S3-@-1160C
          Sample Nmae          :
          Comment              : WHITISH
          Date & Time          : 02-13-14 10:33:42

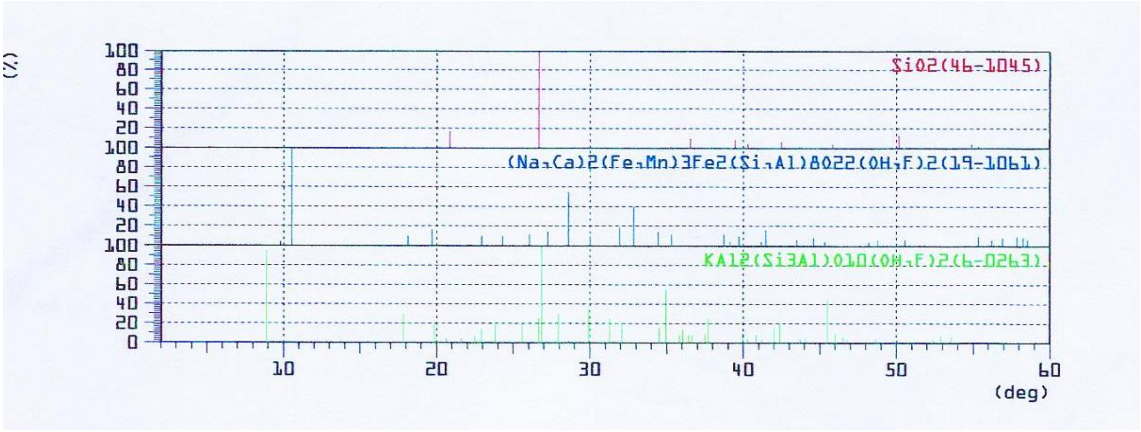
 # Measurement Condition
    X-ray tube
          target               : Cu
          voltage              : 40.0 (kV)
          current              : 30.0 (mA)
    Slits
          Auto Slit            : not Used
          divergence slit      :   1.00000 (deg)
          scatter slit         :   1.00000 (deg)
          receiving slit       :   0.30000(mm)
    Scanning
          drive axis           : Theta-2Theta
          scan range           :    2.0000 - 60.0000 (deg)
          scan mode            : Continuous Scan
          scan speed           :    8.0000 (deg/min)
          sampling pitch       :    0.0200 (deg)
          preset time          :    0.15 (sec)

 # Data Process Condition
    Smoothing                  [ AUTO ]
          smoothing points     : 11
    B.G.Subtruction            [ AUTO ]
          sampling points      : 11
          repeat times         : 30
    Ka1-a2 Separate            [ MANUAL ]
          Ka1 a2 ratio         : 50 (%)
    Peak Search                [ AUTO ]
          differential points  : 9
          FWHM threhold        : 0.050 (deg)
          intensity threhold   : 30 (par mil)
          FWHM ratio  (n-1)/n  : 2
    System error Correction    [ YES ]
    Precise peak Correction    [ NO ]
```

Appendix 57: XRD Analysis of Sample 4 at 1160^{o}C.



Appendix 58: XRD Analysis of Sample 4 at 1160°C (continuation).




```
                **********     SEARCH / MATCH RESULT     **********
 Group Name   : ERIC
 Data Name    : S4-@-1160
 File Name    : S4-@-1160.PSE
 Sample Name  :
 Comment      :
<Entry Card>
No.   Card      Chemical Formula                    S       L          d     I     R
                Chemical Name (Mineral Name)                Dx    WT%    S.G.
 1  29-1487 Al2Si2O5(OH)4                       0.086 1.000( 6/ 7) 0.823 0.432 0.356
            Aluminum Silicate Hydroxide ( Halloysite-7  -----          -------
 2  21-0963 Mg3Si2O5(OH)4                       0.120 0.958(23/27) 0.748 0.473 0.339
            Magnesium Silicate Hydroxide ( Antigorite-  -----          -------
 3  10-0357 (Na,K)(Si3Al)O8                     0.326 0.941(16/17) 0.703 0.473 0.313
            Potassium Sodium Aluminum Silicate ( Sanid  -----          -------
 4  10-0446 Al2Si2O5(OH)4                       0.159 0.919(34/39) 0.773 0.438 0.311
            Aluminum Silicate Hydroxide ( Dickite-2\IT  -----          -------
 5  29-1016 KMg2Al3(Si10Al2)O30                 0.216 0.909(30/33) 0.784 0.414 0.295
            Potassium Magnesium Aluminum Silicate ( Os  -----          -------
 6  23-0125 Ca6Si6O17(OH)2                      0.292 1.000(28/39) 0.698 0.374 0.261
            Calcium Silicate Hydrate ( Xonotlite, syn   -----          -------
 7  46-1045 SiO2                                0.254 1.000(13/42) 0.837 0.305 0.255
            Silicon Oxide ( Quartz, syn )               -----          -------
 8  19-1061 (Na,Ca)2(Fe,Mn)3Fe2(Si,Al)8O22(OH,F 0.318 0.935(29/38) 0.667 0.396 0.247
            Sodium Iron Silicate Hydroxide ( Riebeckit  -----          -------
 9   6-0263 KAl2(Si3Al)O10(OH,F)2               0.256 0.857(36/42) 0.677 0.408 0.237
            Potassium Aluminum Silicate Hydroxide ( Mu  -----          -------
```

Appendix 59: XRD Analysis of Sample 4 at 1160^{o}C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S4-@-1160

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	57	24.0337	3.69982	100	0.17250	69	597
2	60	25.3291	3.51346	38	0.31170	26	395
3	56	23.8400	3.72944	25	0.12000	17	141

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	2.0300	43.48497	3	0.06000	2	6
2	2.5400	34.75477	3	0.12000	2	17
3	2.8900	30.54645	3	0.10000	2	21
4	3.2000	27.58792	3	0.04000	2	6
5	3.4250	25.77606	3	0.13000	2	18
6	3.8250	23.08138	3	0.03000	2	3
7	4.2600	20.72540	3	0.04000	2	6
8	4.6750	18.88650	4	0.03000	3	11
9	5.9600	14.81706	3	0.04000	2	9
10	6.2150	14.20968	4	0.03000	3	11
11	6.4900	13.60818	3	0.06000	2	10
12	7.2800	12.13314	3	0.04000	2	4
13	7.5000	11.77772	4	0.02000	3	4
14	7.8000	11.32539	3	0.04000	2	6
15	8.1950	10.78037	3	0.05000	2	10
16	8.4450	10.46179	3	0.03000	2	5
17	9.1750	9.63098	4	0.03000	3	7
18	9.4400	9.36121	3	0.08000	2	13
19	9.6350	9.17218	4	0.11000	3	25
20	10.1550	8.70365	4	0.03000	3	9
21	10.6400	8.30797	4	0.04000	3	15
22	10.8550	8.14390	4	0.03000	3	7
23	11.0400	8.00784	3	0.08000	2	20
24	11.2000	7.89380	3	0.00000	2	0
25	11.6200	7.60941	4	0.04000	3	13
26	11.9700	7.38769	3	0.06000	2	11
27	12.2600	7.21358	3	0.04000	2	10
28	12.4600	7.09824	4	0.04000	3	9
29	12.9050	6.85445	3	0.11000	2	22
30	13.3700	6.61709	3	0.06000	2	10
31	13.8450	6.39112	4	0.05000	3	20
32	14.1750	6.24306	6	0.11000	4	25
33	14.3500	6.16732	4	0.06000	3	18
34	14.7600	5.99691	4	0.04000	3	15
35	15.0900	5.86650	6	0.10000	4	25
36	15.3200	5.77894	3	0.06000	2	7
37	16.1800	5.47366	4	0.04000	3	9
38	16.3150	5.42867	6	0.11000	4	33
39	17.2250	5.14387	3	0.05000	2	8
40	17.5000	5.06365	3	0.04000	2	8
41	17.9350	4.94181	4	0.03000	3	6
42	18.2358	4.86096	23	0.21830	16	161
43	18.8300	4.70889	6	0.06000	4	17
44	19.0200	4.66228	10	0.09000	7	39
45	19.2950	4.59644	22	0.23000	15	226
46	20.1550	4.40221	3	0.07000	2	10
47	20.3500	4.36047	3	0.10000	2	14
48	20.8000	4.26714	6	0.04000	4	14
49	20.9666	4.23361	16	0.13330	11	97
50	21.2400	4.17972	3	0.00000	2	0

Appendix 60: XRD Analysis of Sample 4 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	21.3950	4.14979	4	0.03000	3	7
52	21.8650	4.06164	9	0.15000	6	47
53	22.0400	4.02979	4	0.12000	3	26
54	22.4000	3.96583	4	0.04000	3	10
55	23.2050	3.83004	3	0.11000	2	11
56	23.8400	3.72944	25	0.12000	17	141
57	24.0337	3.69982	100	0.17250	69	597
58	24.3616	3.65076	4	0.01670	3	3
59	24.7650	3.59220	9	0.15000	6	64
60	25.3291	3.51346	38	0.31170	26	395
61	25.6350	3.47222	4	0.05000	3	10
62	25.9200	3.43469	10	0.12000	7	48
63	26.0950	3.41205	3	0.07000	2	10
64	26.3750	3.37646	3	0.03000	2	5
65	26.7300	3.33242	4	0.06000	3	14
66	27.1800	3.27826	9	0.08000	6	26
67	27.3066	3.26334	14	0.13330	10	62
68	27.7650	3.21050	9	0.09000	6	43
69	28.2750	3.15374	6	0.11000	4	26
70	28.5300	3.12613	6	0.10000	4	31
71	28.7000	3.10800	7	0.04000	5	28
72	29.2800	3.04774	4	0.04000	3	8
73	29.6450	3.01104	3	0.07000	2	10
74	30.8650	2.89474	3	0.03000	2	4
75	31.7900	2.81259	3	0.14000	2	17
76	32.1100	2.78529	3	0.10000	2	21
77	32.7100	2.73556	6	0.18000	4	49
78	33.0000	2.71218	13	0.08000	9	63
79	33.2000	2.69629	14	0.10660	10	91
80	33.9650	2.63729	22	0.20000	15	148
81	34.7200	2.58165	3	0.04000	2	6
82	35.0800	2.55598	4	0.08000	3	15
83	35.4500	2.53015	3	0.10000	2	23
84	36.6400	2.45066	4	0.04000	3	14
85	36.9300	2.43208	3	0.10000	2	21
86	37.3600	2.40507	4	0.04000	3	15
87	37.7650	2.38020	7	0.09000	5	27
88	37.9800	2.36722	4	0.08000	3	19
89	38.2350	2.35201	3	0.11000	2	21
90	38.7000	2.32482	4	0.08000	3	19
91	38.8400	2.31676	4	0.04000	3	10
92	39.0400	2.30535	4	0.04000	3	15
93	39.2300	2.29462	3	0.06000	2	7
94	39.4200	2.28400	4	0.04000	3	9
95	39.7700	2.26470	7	0.10000	5	41
96	39.9800	2.25329	3	0.04000	2	7
97	40.3800	2.23188	6	0.16000	4	40
98	41.8100	2.15879	3	0.06000	2	10
99	42.0650	2.14630	6	0.13000	4	25
100	42.2650	2.13660	4	0.03000	3	14
101	42.5850	2.12128	4	0.13000	3	30
102	43.2900	2.08836	6	0.18000	4	48
103	44.3700	2.04000	4	0.06000	3	22
104	44.7000	2.02570	3	0.04000	2	7
105	45.6000	1.98779	6	0.12000	4	29
106	45.9350	1.97407	3	0.07000	2	9
107	46.2000	1.96336	3	0.04000	2	8
108	46.5900	1.94783	6	0.10000	4	27
109	46.8350	1.93821	4	0.03000	3	10
110	47.5491	1.91076	19	0.11170	13	93
111	47.8100	1.90094	4	0.06000	3	13
112	49.0000	1.85752	7	0.12000	5	42
113	49.2250	1.84955	6	0.11000	4	21

Appendix 61: XRD Analysis of Sample 4 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
114	49.4500	1.84166	4	0.06000	3	17
115	49.5900	1.83679	7	0.06000	5	34
116	50.2700	1.81353	4	0.06000	3	16
117	50.6300	1.80147	3	0.10000	2	13
118	50.9250	1.79173	4	0.07000	3	18
119	51.6200	1.76923	3	0.12000	2	27
120	52.3100	1.74750	6	0.14000	4	28
121	52.5600	1.73978	4	0.04000	3	9
122	53.1200	1.72275	3	0.08000	2	12
123	53.9800	1.69731	3	0.08000	2	16
124	54.5550	1.68077	3	0.09000	2	15
125	54.9600	1.66934	4	0.08000	3	17
126	55.3400	1.65878	3	0.08000	2	13
127	55.5400	1.65328	3	0.08000	2	10
128	55.7600	1.64727	3	0.04000	2	9
129	56.2700	1.63355	3	0.06000	2	8
130	56.5500	1.62612	3	0.10000	2	15
131	57.3200	1.60609	7	0.20000	5	63
132	57.6200	1.59844	3	0.04000	2	9
133	58.1900	1.58413	3	0.06000	2	16
134	59.3100	1.55687	4	0.06000	3	19
135	59.5050	1.55223	3	0.05000	2	5
136	59.7400	1.54668	4	0.04000	3	16
137	59.8750	1.54352	4	0.03000	3	11

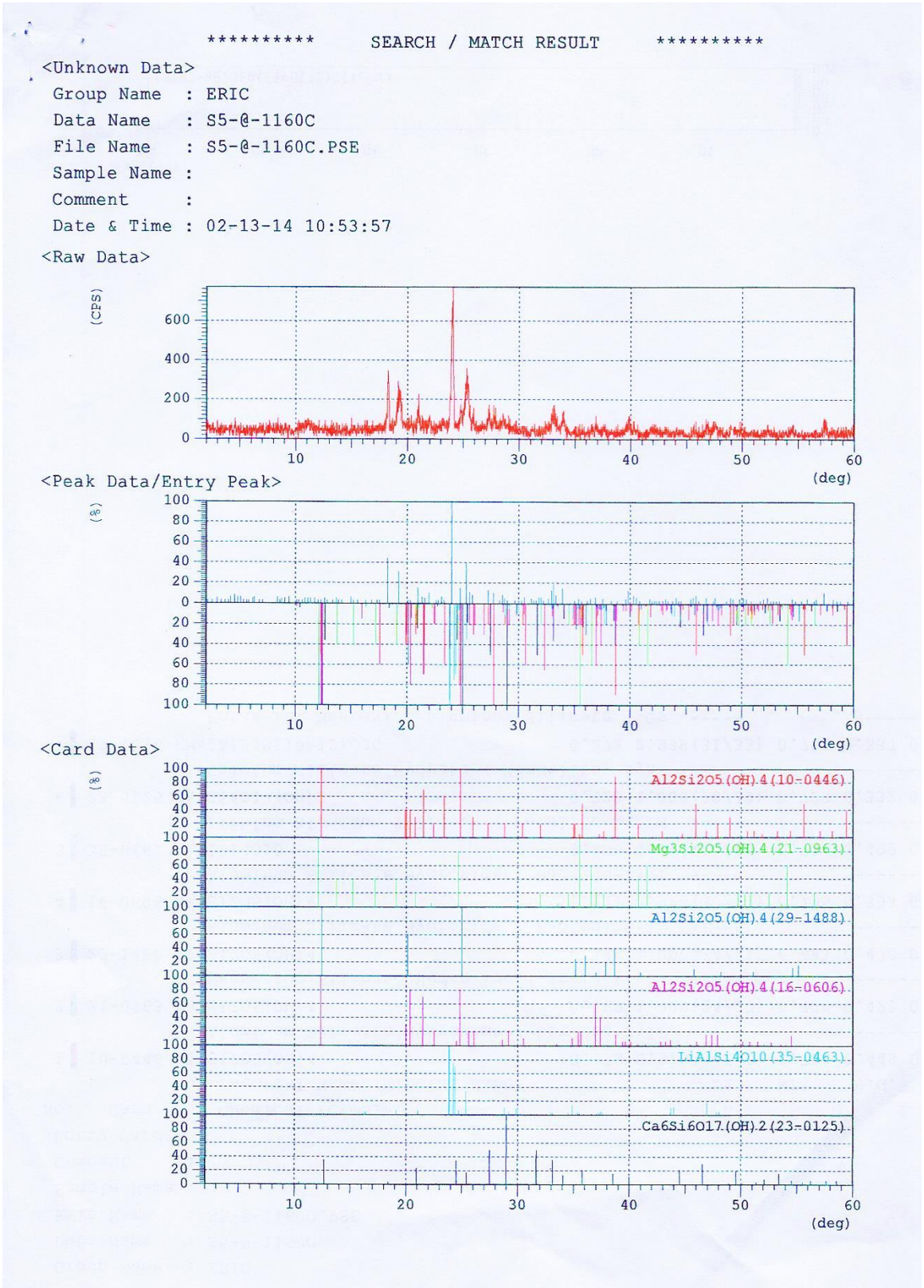
```
        ***  Basic Data Process  ***

# Data Infomation
          Group                 : ERIC
          Data                  : S5-@-1160C
          Sample Nmae           :
          Comment               :
          Date & Time           : 02-13-14 10:53:57

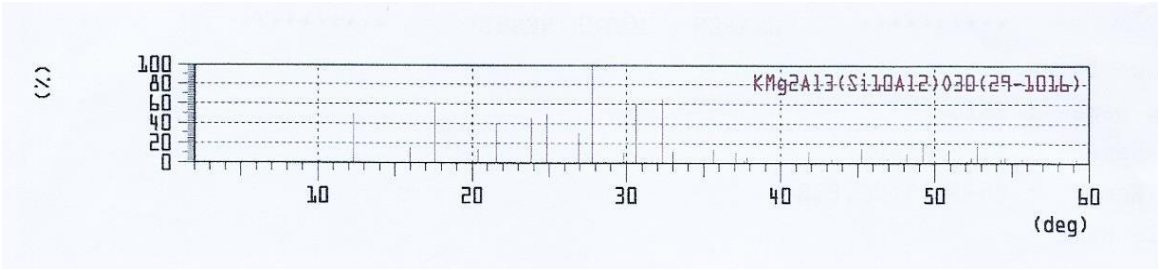
# Measurement Condition
    X-ray tube
          target                : Cu
          voltage               : 40.0 (kV)
          current               : 30.0 (mA)
    Slits
          Auto Slit             : not Used
          divergence slit       :   1.00000 (deg)
          scatter slit          :   1.00000 (deg)
          receiving slit        :   0.30000(mm)
    Scanning
          drive axis            : Theta-2Theta
          scan range            :    2.0000 - 60.0000 (deg)
          scan mode             : Continuous Scan
          scan speed            :    8.0000 (deg/min)
          sampling pitch        :    0.0200 (deg)
          preset time           :    0.15 (sec)

# Data Process Condition
    Smoothing                   [ AUTO ]
          smoothing points      : 11
    B.G.Subtruction             [ AUTO ]
          sampling points       : 15
          repeat times          : 30
    Ka1-a2 Separate             [ MANUAL ]
          Ka1 a2 ratio          : 50 (%)
    Peak Search                 [ AUTO ]
          differential points   : 13
          FWHM threhold         : 0.050 (deg)
          intensity threhold    : 30 (par mil)
          FWHM ratio  (n-1)/n   : 2
    System error Correction     [ YES ]
    Precise peak Correction     [ NO ]
```

Appendix 62: XRD Analysis of Sample 5 at 1160^{o}C.



Appendix 63: XRD Analysis of Sample 5 at 1160°C (continuation).




```
                **********     SEARCH / MATCH RESULT     **********
 Group Name  : ERIC
 Data Name   : S5-@-1160C
 File Name   : S5-@-1160C.PSE
 Sample Name :
 Comment     :
<Entry Card>
No.   Card      Chemical Formula                      S       L         d     I     R
                Chemical Name (Mineral Name)                Dx    WT%    S.G.
 1  10-0446 Al2Si2O5(OH)4                         0.178 0.919(34/39) 0.781 0.449 0.322
            Aluminum Silicate Hydroxide ( Dickite-2\IT  -----          -------
 2  21-0963 Mg3Si2O5(OH)4                         0.169 1.000(24/27) 0.720 0.421 0.304
            Magnesium Silicate Hydroxide ( Antigorite-  -----          -------
 3  29-1488 Al2Si2O5(OH)4                         0.149 1.000(14/21) 0.641 0.470 0.301
            Aluminum Silicate Hydroxide ( Kaolinite-1\  -----          -------
 4  16-0606 Al2Si2O5(OH)4                         0.221 0.929(39/42) 0.795 0.393 0.290
            Aluminum Silicate Hydroxide ( Nacrite-2\IT  -----          -------
 5  35-0463 LiAlSi4O10                            0.803 0.714(30/42) 0.794 0.492 0.279
            Lithium Aluminum Silicate ( Petalite-1\ITM  -----          -------
 6  23-0125 Ca6Si6O17(OH)2                        0.328 1.000(28/39) 0.729 0.332 0.243
            Calcium Silicate Hydrate ( Xonotlite, syn   -----          -------
 7  29-1016 KMg2Al3(Si10Al2)O30                   0.272 0.939(31/33) 0.755 0.337 0.239
            Potassium Magnesium Aluminum Silicate ( Os  -----          -------
```

Appendix 64: XRD Analysis of Sample 5 at 1160^{o}C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S5-@-1160C

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	57	24.0037	3.70438	100	0.22750	62	749
2	45	18.2600	4.85458	44	0.16000	27	250
3	59	25.3133	3.51562	40	0.33330	25	435

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	2.1700	40.67979	3	0.18000	2	18
2	2.5500	34.61850	3	0.06000	2	12
3	2.9900	29.52505	5	0.06000	3	17
4	3.3750	26.15782	3	0.11000	2	19
5	3.7100	23.79658	3	0.06000	2	15
6	4.0000	22.07195	3	0.08000	2	16
7	4.1900	21.07149	6	0.10000	4	23
8	4.5700	19.32019	8	0.14000	5	47
9	4.8450	18.22419	6	0.09000	4	21
10	5.0900	17.34753	6	0.12000	4	23
11	5.3650	16.45893	3	0.21000	2	22
12	5.6350	15.67089	3	0.09000	2	12
13	5.8200	15.17317	3	0.08000	2	15
14	6.2000	14.24403	5	0.04000	3	16
15	6.3600	13.88604	6	0.04000	4	17
16	6.7900	13.00759	3	0.10000	2	30
17	7.0600	12.51073	3	0.04000	2	8
18	8.4000	10.51774	6	0.16000	4	38
19	8.6350	10.23202	3	0.13000	2	16
20	8.7800	10.06336	5	0.04000	3	8
21	9.0800	9.73153	5	0.12000	3	35
22	9.4300	9.37111	6	0.06000	4	14
23	10.1100	8.74228	3	0.06000	2	8
24	10.3150	8.56900	3	0.09000	2	19
25	10.6450	8.30408	3	0.03000	2	7
26	10.8550	8.14390	5	0.09000	3	22
27	11.1500	7.92909	5	0.14000	3	32
28	11.3700	7.77615	5	0.10000	3	24
29	11.7050	7.55434	5	0.05000	3	11
30	12.0500	7.33882	3	0.06000	2	16
31	12.3000	7.19021	3	0.04000	2	10
32	12.6433	6.99574	5	0.04670	3	14
33	12.8600	6.87834	3	0.04000	2	8
34	13.3400	6.63190	3	0.04000	2	12
35	13.6800	6.46783	5	0.04000	3	9
36	13.8800	6.37508	3	0.08000	2	9
37	14.1600	6.24964	3	0.08000	2	17
38	14.9900	5.90541	6	0.10000	4	19
39	15.2000	5.82429	3	0.12000	2	22
40	15.5700	5.68671	3	0.10000	2	10
41	16.3000	5.43363	3	0.04000	2	7
42	16.8400	5.26059	3	0.08000	2	15
43	17.3200	5.11587	3	0.08000	2	13
44	17.5200	5.05792	3	0.04000	2	4
45	18.2600	4.85458	44	0.16000	27	250
46	19.2441	4.60849	31	0.26170	19	321
47	20.0950	4.41522	3	0.05000	2	8
48	20.3000	4.37110	3	0.04000	2	9
49	20.9883	4.22928	15	0.20330	9	87
50	21.3450	4.15940	6	0.11000	4	23

Appendix 65: XRD Analysis of Sample 5 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	21.5950	4.11181	5	0.09000	3	18
52	21.9600	4.04428	10	0.14000	6	52
53	22.6100	3.92947	3	0.06000	2	8
54	22.8000	3.89715	3	0.12000	2	17
55	23.1000	3.84721	6	0.08000	4	23
56	23.3000	3.81464	3	0.08000	2	19
57	24.0037	3.70438	100	0.22750	62	749
58	24.7650	3.59220	15	0.13000	9	75
59	25.3133	3.51562	40	0.33330	25	435
60	25.6000	3.47689	11	0.08000	7	32
61	25.8650	3.44187	8	0.09000	5	37
62	27.2800	3.26647	13	0.16000	8	83
63	27.7500	3.21220	10	0.14000	6	60
64	28.3000	3.15101	3	0.04000	2	11
65	28.5500	3.12398	8	0.10000	5	37
66	28.7300	3.10482	5	0.06000	3	13
67	29.0000	3.07652	3	0.04000	2	6
68	29.6800	3.00757	3	0.04000	2	9
69	30.0100	2.97524	3	0.06000	2	8
70	30.2600	2.95123	3	0.04000	2	7
71	30.5233	2.92637	6	0.07330	4	15
72	31.1300	2.87070	5	0.10000	3	21
73	31.3450	2.85150	3	0.11000	2	14
74	31.5800	2.83082	3	0.08000	2	8
75	31.8033	2.81145	6	0.08670	4	17
76	31.9900	2.79546	6	0.08000	4	15
77	32.2000	2.77771	3	0.08000	2	23
78	32.6700	2.73882	5	0.06000	3	16
79	32.9350	2.71738	13	0.11000	8	59
80	33.1375	2.70124	19	0.11500	12	82
81	33.3350	2.68568	8	0.05000	5	28
82	33.9350	2.63956	15	0.15000	9	99
83	35.0000	2.56164	3	0.08000	2	23
84	35.3700	2.53569	5	0.06000	3	13
85	35.6300	2.51778	3	0.14000	2	24
86	36.1300	2.48407	3	0.06000	2	11
87	36.4400	2.46365	5	0.04000	3	15
88	36.8650	2.43621	6	0.11000	4	44
89	37.5350	2.39425	5	0.11000	3	27
90	38.2700	2.34994	3	0.06000	2	13
91	38.8300	2.31733	3	0.06000	2	12
92	39.6000	2.27403	5	0.10000	3	22
93	39.8283	2.26152	13	0.12330	8	57
94	40.2800	2.23719	8	0.10000	5	37
95	40.5300	2.22397	6	0.10000	4	27
96	40.8150	2.20910	3	0.07000	2	13
97	41.7500	2.16176	5	0.12000	3	26
98	41.9750	2.15069	3	0.09000	2	16
99	42.1700	2.14119	3	0.10000	2	17
100	42.3300	2.13347	3	0.06000	2	16
101	42.5900	2.12105	3	0.06000	2	12
102	42.9150	2.10573	3	0.07000	2	11
103	43.1400	2.09527	5	0.12000	3	25
104	43.3600	2.08515	3	0.08000	2	9
105	43.7800	2.06611	3	0.08000	2	10
106	44.1000	2.05186	6	0.12000	4	22
107	44.3300	2.04175	3	0.06000	2	9
108	44.5300	2.03304	3	0.14000	2	23
109	45.4000	1.99608	5	0.04000	3	10
110	45.6200	1.98697	6	0.16000	4	37
111	45.8750	1.97651	8	0.07000	5	20
112	46.1250	1.96638	3	0.03000	2	5
113	46.7866	1.94010	6	0.13330	4	33

Appendix 66: XRD Analysis of Sample 5 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
114	47.4400	1.91490	3	0.14000	2	16
115	47.5850	1.90940	6	0.13000	4	28
116	48.5950	1.87205	3	0.11000	2	12
117	48.9550	1.85912	6	0.15000	4	34
118	49.2050	1.85026	5	0.07000	3	27
119	49.7800	1.83022	5	0.16000	3	49
120	50.1066	1.81906	6	0.09330	4	22
121	50.3850	1.80966	5	0.05000	3	14
122	50.8300	1.79485	3	0.06000	2	10
123	51.6400	1.76859	3	0.04000	2	8
124	51.9100	1.76002	5	0.06000	3	14
125	52.2800	1.74843	6	0.12000	4	34
126	52.5650	1.73962	3	0.07000	2	8
127	52.8400	1.73121	3	0.08000	2	11
128	53.0600	1.72455	3	0.04000	2	10
129	53.3200	1.71676	3	0.08000	2	8
130	54.4900	1.68263	5	0.10000	3	25
131	54.8000	1.67384	3	0.04000	2	4
132	55.3200	1.65933	3	0.04000	2	8
133	55.8250	1.64551	5	0.05000	3	15
134	56.4500	1.62876	3	0.06000	2	8
135	56.6400	1.62375	5	0.04000	3	12
136	57.3400	1.60558	11	0.20000	7	85
137	58.1700	1.58463	5	0.06000	3	16
138	58.4950	1.57660	3	0.13000	2	15
139	58.8200	1.56866	3	0.08000	2	14
140	59.4900	1.55258	3	0.06000	2	7

Appendix 67: XRD Analysis of Sample 5 at 1160^{o}C (continuation).

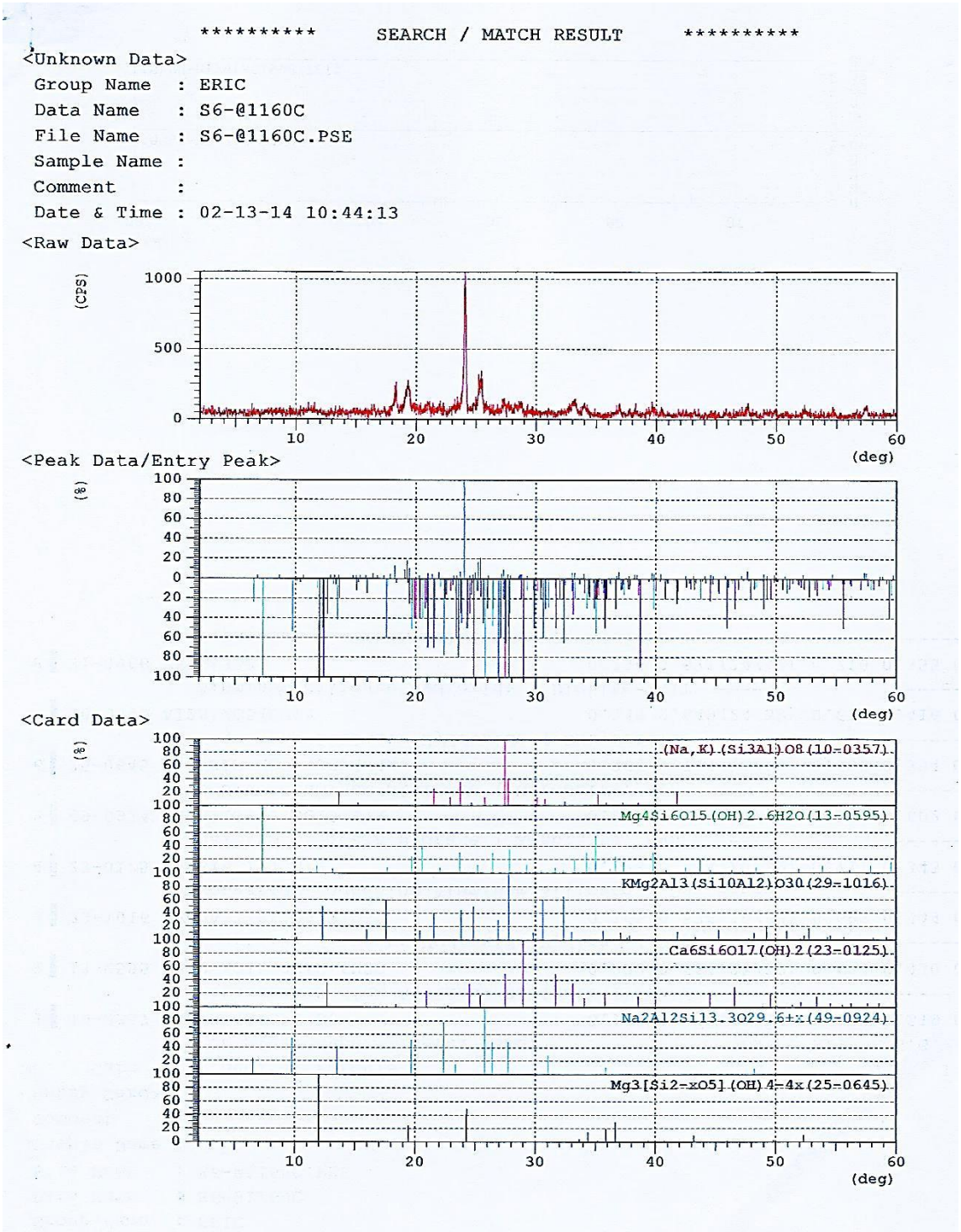
```
      ***  Basic Data Process  ***

# Data Infomation
        Group               : ERIC
        Data                : S5-@-1160C
        Sample Nmae         :
        Comment             :
        Date & Time         : 02-13-14 10:53:57

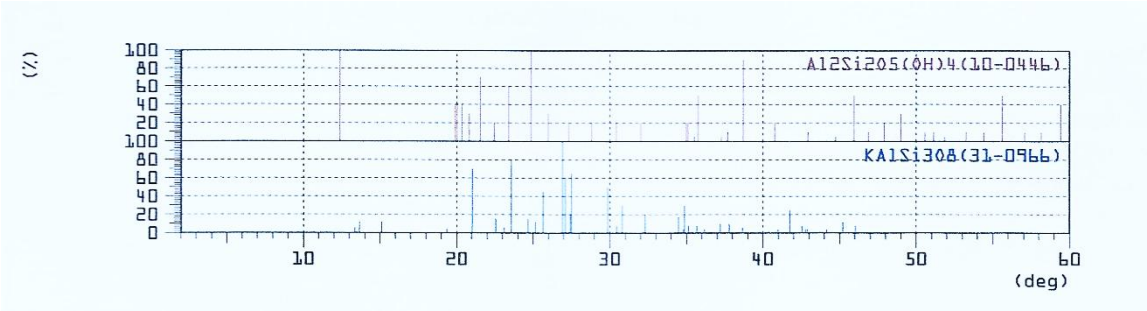
# Measurement Condition
    X-ray tube
        target              : Cu
        voltage             : 40.0 (kV)
        current             : 30.0 (mA)
    Slits
        Auto Slit           : not Used
        divergence slit     :   1.00000 (deg)
        scatter slit        :   1.00000 (deg)
        receiving slit      :   0.30000(mm)
    Scanning
        drive axis          : Theta-2Theta
        scan range          :    2.0000 - 60.0000 (deg)
        scan mode           : Continuous Scan
        scan speed          :    8.0000 (deg/min)
        sampling pitch      :    0.0200 (deg)
        preset time         :    0.15 (sec)

# Data Process Condition
    Smoothing               [ AUTO ]
        smoothing points    : 11
    B.G.Subtruction         [ AUTO ]
        sampling points     : 15
        repeat times        : 30
    Ka1-a2 Separate         [ MANUAL ]
        Ka1 a2 ratio        : 50 (%)
    Peak Search             [ AUTO ]
        differential points : 13
        FWHM threhold       : 0.050 (deg)
        intensity threhold  : 30 (par mil)
        FWHM ratio  (n-1)/n : 2
    System error Correction [ YES ]
    Precise peak Correction [ NO ]
```

Appendix 68: XRD Analysis of Sample 6 at 1160^{o}C.



Appendix 69: XRD Analysis of Sample 6 at 1160°C (continuation).




```
                **********     SEARCH / MATCH RESULT     **********
 Group Name  : ERIC
 Data Name   : S6-@1160C
 File Name   : S6-@1160C.PSE
 Sample Name :
 Comment     :
<Entry Card>
No.   Card      Chemical Formula                      S       L          d     I     R
                Chemical Name (Mineral Name)                 Dx    WT%    S.G.
 1  10-0357 (Na,K)(Si3Al)O8                      0.185 0.647(11/17) 0.687 0.519 0.231
            Potassium Sodium Aluminum Silicate ( Sanid  -----          -------
 2  13-0595 Mg4Si6O15(OH)2.6H2O                  0.222 0.694(25/39) 0.604 0.430 0.180
            Magnesium Silicate Hydroxide Hydrate ( Sep  -----          -------
 3  29-1016 KMg2Al3(Si10Al2)O30                  0.165 0.576(19/33) 0.692 0.444 0.177
            Potassium Magnesium Aluminum Silicate ( Os  -----          -------
 4  23-0125 Ca6Si6O17(OH)2                       0.228 0.714(20/39) 0.711 0.345 0.175
            Calcium Silicate Hydrate ( Xonotlite, syn   -----          -------
 5  49-0924 Na2Al2Si13.3O29.6+x                  0.089 0.500( 7/14) 0.572 0.602 0.172
            Sodium Aluminum Silicate ( Mordenite, syn   -----          -------
 6  25-0645 Mg3[Si2-xO5](OH)4-4x                 0.266 0.714(10/18) 0.680 0.354 0.172
            Magnesium Silicate Hydroxide ( Chrysotile   -----          -------
 7  10-0446 Al2Si2O5(OH)4                        0.148 0.649(24/39) 0.618 0.416 0.167
            Aluminum Silicate Hydroxide ( Dickite-2\IT  -----          -------
 8  31-0966 KAlSi3O8                             0.236 0.571(24/42) 0.710 0.355 0.144
            Potassium Aluminum Silicate ( Orthoclase )  -----          -------
```

Appendix 70: XRD Analysis of Sample 6 at 1160^{o}C (continuation).

*** Basic Data Process ***

Group : ERIC
Data : S6-@1160C

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	26	24.1032	3.68931	100	0.17290	98	942
2	31	25.4502	3.49702	21	0.20000	21	231
3	17	19.3448	4.58472	18	0.28000	18	190

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	6.9760	12.66118	3	0.02000	3	8
2	8.7907	10.05114	3	0.02000	3	6
3	10.7672	8.21011	3	0.02670	3	6
4	13.0069	6.80098	3	0.05000	3	17
5	13.3406	6.63160	3	0.04000	3	15
6	13.6793	6.46816	3	0.04000	3	12
7	14.8952	5.94278	3	0.04000	3	18
8	15.1345	5.84935	3	0.04000	3	13
9	15.4537	5.72924	3	0.04000	3	12
10	15.9274	5.55990	3	0.03000	3	6
11	16.5211	5.36141	5	0.06000	5	29
12	16.8653	5.25276	3	0.05000	3	14
13	17.4392	5.08117	3	0.06000	3	18
14	17.9782	4.93003	4	0.06000	4	15
15	18.3376	4.83421	13	0.24000	13	170
16	19.1654	4.62723	9	0.12000	9	59
17	19.3448	4.58472	18	0.28000	18	190
18	19.5243	4.54297	10	0.08000	10	59
19	20.3618	4.35797	3	0.04000	3	12
20	21.0798	4.21113	5	0.16000	5	55
21	21.4356	4.14202	4	0.06000	4	13
22	21.9875	4.03929	6	0.10000	6	42
23	23.2146	3.82848	3	0.06000	3	16
24	23.4940	3.78358	3	0.04000	3	15
25	23.7735	3.73972	7	0.08000	7	56
26	24.1032	3.68931	100	0.17290	98	942
27	24.5669	3.62071	3	0.03000	3	7
28	24.8314	3.58274	4	0.12000	4	26
29	24.9911	3.56021	6	0.08000	6	22
30	25.2506	3.52420	17	0.13000	17	122
31	25.4502	3.49702	21	0.20000	21	231
32	26.0741	3.41474	3	0.03000	3	16
33	27.2123	3.27444	5	0.07000	5	27
34	27.3710	3.25581	9	0.11200	9	58
35	27.7065	3.21714	4	0.16000	4	36
36	27.9262	3.19233	3	0.08000	3	15
37	28.6451	3.11383	3	0.12000	3	27
38	28.7350	3.10429	4	0.10000	4	18
39	28.8349	3.09376	4	0.10000	4	28
40	29.3042	3.04528	3	0.04000	3	14
41	33.0598	2.70741	6	0.16000	6	44
42	33.2596	2.69160	6	0.22000	6	71
43	34.0238	2.63287	5	0.17000	5	62
44	36.7563	2.44317	3	0.08000	3	20
45	36.9362	2.43168	7	0.10000	7	42
46	37.7655	2.38017	3	0.10000	3	21
47	38.2352	2.35200	4	0.08000	4	22
48	38.4250	2.34082	4	0.06000	4	17
49	39.6841	2.26940	6	0.10000	6	28
50	39.8814	2.25863	5	0.06500	5	19

Appendix 71: XRD Analysis of Sample 6 at 1160^{o}C (continuation).

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
51	40.4436	2.22852	5	0.10000	5	40
52	42.5122	2.12475	3	0.04000	3	9
53	43.1568	2.09449	3	0.07000	3	17
54	44.1112	2.05136	3	0.04000	3	15
55	44.5510	2.03213	4	0.12000	4	36
56	44.7759	2.02244	3	0.03000	3	9
57	45.8203	1.97875	3	0.06000	3	15
58	47.1396	1.92640	3	0.06000	3	13
59	47.6160	1.90823	8	0.13330	8	74
60	48.9837	1.85810	5	0.11000	5	30
61	49.2486	1.84872	3	0.10000	3	25
62	49.8683	1.82719	4	0.08000	4	17
63	50.7479	1.79756	3	0.04000	3	16
64	52.2772	1.74852	4	0.06000	4	32
65	54.6962	1.67677	5	0.10000	5	46
66	57.3453	1.60544	7	0.16000	7	61
67	57.5052	1.60136	6	0.14660	6	57
68	58.6648	1.57244	3	0.04000	3	17
69	58.8947	1.56685	3	0.06000	3	15
70	59.7694	1.54599	4	0.11000	4	21

Appendix 72: XRD Analysis of Sample 6 at 1160^{o}C (continuation).

```
          ***  Basic Data Process  ***

# Data Infomation
          Group                 : ERIC
          Data                  : S6-@1160C
          Sample Nmae           :
          Comment               :
          Date & Time           : 02-13-14 10:44:13

# Measurement Condition
    X-ray tube
          target                : Cu
          voltage               : 40.0 (kV)
          current               : 30.0 (mA)
    Slits
          Auto Slit             : not Used
          divergence slit       :   1.00000 (deg)
          scatter slit          :   1.00000 (deg)
          receiving slit        :   0.30000(mm)
    Scanning
          drive axis            : Theta-2Theta
          scan range            :    2.0000 - 60.0000 (deg)
          scan mode             : Continuous Scan
          scan speed            :    8.0000 (deg/min)
          sampling pitch        :    0.0200 (deg)
          preset time           :    0.15 (sec)

# Data Process Condition
    Smoothing                   [ AUTO ]
          smoothing points      : 11
    B.G.Subtruction             [ AUTO ]
          sampling points       : 11
          repeat times          : 30
    Ka1-a2 Separate             [ MANUAL ]
          Ka1 a2 ratio          : 50 (%)
    Peak Search                 [ AUTO ]
          differential points   : 9
          FWHM threhold         : 0.050 (deg)
          intensity threhold    : 30 (par mil)
          FWHM ratio  (n-1)/n   : 2
    System error Correction     [ YES ]
    Precise peak Correction     [ NO ]
```