COMPARATIVE ANALYSES OF WURNO, DANGE AND WEPPA GYPSUMS IN NIGERIA FOR PLASTER AND CERAMIC PRODUCTION

By

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DEPARTMENT OF INDUSTRIAL DESIGN
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DECLARATION

I, Grace Kokoeka Soyinka, hereby declare that the work in this dissertation entitled "Comparative Analysis of Wurno, Dange and Weppa Gypsum for Ceramic Production in Nigeria" was conducted by me in the Department of Industrial Design, Ahmadu Bello University, Zaria. In the course of this study, some research works were conducted at National Steel Council, Malali Kaduna, Industrial Development Centre, (IDC) Zaria and Multi-user Laboratory of the Department of Chemistry, Ahmadu Bello University, Zaria. The information derived from literature has been duly acknowledged in the texts and in the list of references provided. No part of this dissertation was previously presented for another degree or diploma at any University.

_____	_____	_____
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CERTIFICATION

This thesis entitled "Comparative Analysis of Wurno Dange and Weppa Gypsum for Ceramic Production in Nigeria" by Soyinka Grace K meets the regulations governing the award of the degree of Doctor of Philosophy in Industrial Design of Ahmadu Bello University, Zaria. It has this day been approved for its contribution to knowledge and literary presentation.

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DEDICATION

This work is, first and foremost, dedicated to God the Father of our Lord Jesus Christ for His steadfast faithfulness. Also for bringing me a parcel of joy in my dear husband who has been a wonderful support in this research endeavour. Secondly, this work is dedicated to my three awesomely patient and understanding children in NinuJesu Ikanniwa, Ooreofeoluwabo and Edidioluwa Soyinka.

Finally, this work is dedicated to the evergreen memory of my late parents who layed a good and early foundation for my educational advancement.

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DEFINITION OF SPECIAL TERMS

Alpha, Beta and Gamma: The three types of calcined gypsum or plaster of Paris.

Anhydrite: A variation of gypsum rock without two moleculeS of water of crystallization.

Ball clay: A highly plastic clay, usually greyish in colour, it forms the basic of many potting bodies.

Beneficiation: A process of conditioning obtained sample(s) to suit the use for use, in this case, for experiment.

Biscuit/Bisque: A porous unglazed fired pottery.

Boiling: The periods that plaster in the oven turns into semi-gaseous states usually referred to as first and second boiling.

Blend: A systematic mixture of two or more materials.

Blunger: A machine used for stirring and mixing ceramic materials, usually the ingredients of a clay body and water thoroughly in form of a slip.

Body: The natural clay of which a pot is formed, materials mixed together for the formation of pottery, or the main part of the pot as distinct from the glazed.

Calcine: Purification and preparation of materials by the action of heat, usually in the region of 700^0C to 900^0C but in the case of plaster between 120^0C and 180^0C.

Ceramic: A word formed from the Greek word Keramos, meaning clay. A word for all pottery processes. Also used currently in connection with such as glass and cement.

Cottle: A wall of stiff expendable card, plastic, wood or clay to contain liquid plaster until it sets.

Deflocculation:	The addition of electrolytes (e.g. soda ash and sodium silicate) to clay slip reducing the amount of water required to make it pourable.
Earthenware:	Low temperature ware. Pottery made of porous body usually covered with glaze (about 1000°C).
Electrolyte:	Alkaline substance, usually soda ash or sodium silicate introduced into a clay slip to increase fluidity and prevent settling.
Fettling:	Smooth trimming of rough edges or seams of cast wares and finishing of greenwares.
Feldspar:	A group of minerals that contain alkalis plus silica and alumina.
F.G.D:	Flue gas desulphurization. A process whereby a synthetic gypsum is produced.
Fluidity:	The property of a liquid to be able to flow or move freely without the hindrance of friction.
Former:	A natural mould which is usually a one piece mould.
Glaze:	Ceramic glaze is a special form of glass in its lower thermal expansion alumina content.
Greenware:	Unfired ceramic piece.
Gypsum:	The raw material for producing plaster of Paris - (or $CaS0_4.2H_20$).
Kaolin:	Pure clay also known as China clay - ($Al_20_3.S_I0_2.2H_20$).
Kiln:	A furnace made of refractory clay materials for ceramic production.
Levigation:	The process of passing a thick slurry through a mesh where the heavier particles are strained while the finer particles overflow.
Marking:	Traditional way of determining when liquid plaster is ready for pouring

Mesh: Interwoven threads or wire producing theoretically square holes in between and measured by the number of threads per linear inch, used for sieving ceramic materials.

Model: A prototype shape to be copied or reproduced, usually in clay or plaster.

Mould: A hollow form, usually made of plaster of Paris, containing a hollow negative form.

P.O.P: Otherwise known as Plaster of Paris, is a semi-hydrated calcium sulphate.

Ratio mix: The normal ratio of plaster and water

Seam: A line of junction between two edges of a cottle.

Settle: The periods after the two boiling periods during plaster production usually referred to as first and second settle.

Slaking Time: Soaking period given plaster in water before stirring usually between two to five minutes.

Slip: A homogenous mixture of ceramic raw materials and water used for casting.

Stoneware: A high-fired ware with slight or no absorbency (temperature is about 1200°C to 1300°C).

Synthetic gypsum: Gypsum not naturally mined but a derivation from industrial production. For example, F.G.D.

Thixotropy: The property of slip to change fluidity, if left undisturbed becomes more viscous.

Throwing: The action of making pots on a rotary potters' wheels,

Temperature: A means of describing heat and is measured in degree centigrade (°C) or degree fahrenheit (°F).

Undercut: A part of a casting mold that may prevent the cast piece from dropping out of the mould after draining

Viscosity: Resistance to flow of liquid due to internal friction

Vitreous: Like glass or glassy

Abstract

The raw mineral used world-wide in the production of plaster of Paris is gypsum. This is available in Nigeria. Constant importation of plaster of Paris constitutes a drain on national economy and an added cost on ceramic mass production. Its availability has prompted the central direction of this project to analyse, in a comparative way and to see the possibility of local usage which is likely to cut down cost of ceramic production.

Gypsum was obtained from three locations, Wurno and Dange in Sokoto State and Weppa in Edo state. The three samples obtained were labelled and transported from sites to the Ceramic Section, Department of Industrial Design, Ahmadu Bello University, Zaria. They were beneficiated and calcined at the Industrial Development Centre (IDC) at the temperature of 170^0C. This was done for the three samples from these locations. Various tests were carried out and they included absorbency, shrinkage, compressive and density tests. These tests aided in determining performance of processed plaster of Paris for ceramic mass production. Results showed that the ratio mix of 75-85 plaster/water ratio mix were suitable for ceramic slip casting process. 65 and 70 plaster/water ratio mix were better used for casting models and press moulding process. The processed plaster of Paris from the three locations; Wurno, Dange and Weppa were used in the production of ceramic moulds which were in turn used for production of ceramic wares. The wares were fired and glazed. This showed that gypsum obtained from the three locations can be used for ceramic application. However, Weppa sample was the best of the three locations in performance and colour followed by Wurno. Dange was good but showed early signs of wearing fatique. Wurno and Dange samples were identified as anhydrites $CaSO_4 2H_2O$ while Weppa sample was identified as gypsum $CaSO_4.2H_2O$.

CHAPTER ONE

INTRODUCTION

1.0 Background to the Study

The normal production processes involved in ceramic design and practice is often times tedious and demanding. The necessities for faster mode of production such as the use of plaster of Paris mould have become imperative for ceramic production process in recent times. The production and use of moulds in their various forms seem to bring relief and solution to initial production methods that seem more strenuous at accomplishing mass production. Although there are various types of moulds, the plaster of Paris mould appears to be the type common with most ceramic mass productions. Ceramic moulds are made from plaster of Paris produced from gypsum. It was first developed in Montmartre, France around 1770, which gave rise to its name plaster of Paris and the discovery revolutionised mass production by slip casting. Gypsum is a rock or natural mineral that is mined from the earth, usually referred to as hydrous calcium sulphate, or ($CaSO_4.2H_2O$). When gypsum is exposed to heat treatment, 75% of water loss is recorded. This changes it to hemihydrates ($CaSO_4.½H_2O$). In other words, gypsum + heat = plaster (and ¾ water which evaporates). The making of plaster is done by grinding gypsum, and then heating it until the chemically-bonded water partially evaporates from 100^0C to about 180°C.

It is evident from past studies that gypsum is available in Nigeria. Researchers and writers alike have tried to establish this fact from their findings. Examples of such reports can be found with the Ministry of Solid minerals Development 2000, and Mohammed-Sani, 2002. According to Akpakip, (1993), moulds may come in various forms but the medium which can best be adapted for ceramic production is gypsum plaster. This is so because moulds made from plaster have the ability or tendency to absorb large quantity of water from casting slip,

thus making it a cheap and reliable medium for ceramic production that cannot for now, be substituted with other media.

Generally, moulds may be produced using other media such as metal, cement, wood, earthenware, silicone gel, polyvinyl and so on, (Sullayman, 2006, Akomolafe, 1991). Moulds of different types are often used to execute both small and large projects. These could work well for non-clay processes and allied disciplines but not with ceramic mass production due to the peculiarity of its production. This may be due to the fact that most ceramic productions use clay in wet state (plastic or slurry) or such materials (ceramic bodies) that form or dry on plaster moulds. An exception is when other mechanical processes like dust / granular pressing, isostatic pressing, tape casting, and rain pressing, and so on are involved.

However, the fact remains that whether mould is made in a single one piece, two-piece, three-piece or multiple-piece form, its production helps in replicating art, craft or other industrial products exactly, either in relief or in-the-round. In other words, to attain accuracy, uniformity in weight, size and finishing, mould-making and its usage are desirable. Akpakip, (1996) worked on using gypsum in the area of plaster of Paris production. The major thrust of this study is to analyse the composite nature of gypsum which can be used as guide toward producing ceramic plaster.

There are some undeniable facts about the importance of mould in ceramic production that should be addressed and discussed at this stage. The first is that moulds are good for precision and replication for the production either in general art/craft works (in the form of household utensils or decorative wares), or in the minute scientific ceramic productions such as dentures and other medical body part replacement. Secondly, availability of mould and its use reduces time spent on the job and conserves energy which can be directed toward production of more ceramic wares. Lastly, the introduction and use of moulds in ceramic

production process must be acknowledged as it has made an otherwise unaffordable ceramics available and affordable to everyone. These facts, among others, are the reasons why production and use of plaster, ipso facto moulds should be encouraged and sustained.

The constant requirement and demand for gypsum for production as raw material by cement and other allied industries in Nigeria served as a parameter for investigation of the availability and location of gypsum within the nation. However in 1995, during the reign of General Sani Abacha, the military Head of State from 1993 to 1998, placed a ban on the importation of gypsum into the country. This was due to the fact that some geological surveys revealed that Nigeria is rich in gypsum mineral (Mohammed-Sani, 2002). The availability of gypsum in commercial quantity made it imperative for industrialists to harness the information to the advantage of cement manufacturing, classroom board chalks and plaster of Paris.

Holding this thought in mind, it is pertinent to state that one of the focal points of this study is to locally source and mine gypsum. The three locations namely; Wurno and Dange in Wurno and Dange Local Government Areas of Sokoto State, and Weppa in Agenebode Local Government Area of Edo State have provided the sites. There is need to further investigate the samples collected, ascertain their properties such as their chemical and mineralogical constituents and then process them into plaster of Paris that can be suitable for ceramic purposes. A comparative analysis of the various types of gypsum obtained and plaster of Paris processed will be assessed to determine their comparative strengths in the making of mould formed with selected ratio mixes in line with the existing acceptable standards (USG, 2002; British Geological Survey, 2006; Guler and Cigdem 2012 and Singh, 2013).

The study is expected to contribute towards broadening the scope for creative interest and knowledge of material exploration, processing and analyses. It is also evident from available

literature that there are ample amount of gypsum deposits at Dange and Wurno areas of Sokoto State, and Weppa in Agenebode of Edo State as contained in Mohammed-Sani (2002), Ojo, (1995), and Ministry of Solid Minerals Development, (2000). According to Sullayman, (1991), Arayela, (2006) in the factories and industries where various ceramic wares are produced, moulds are used as convenient mode of production, whether slip casting, jiggering, jolleying or dry pressing. It answers the question of standardisation, uniformity, time and material management in factories.

Although the studio potter and the traditional practitioners of pottery may be sufficient with throwing and their making standards, industrial mass production can only suffice with moulds. It is also production from factory or industrially-produced wares that may facilitate exportation of indigenous ceramics, thereby improving Nigeria's foreign exchange capacity and giving the country a chance to compete favourably with other countries of the world in production and exports of ceramic goods. Therefore mould making is important in the mass production process of ceramics for profitability, cost effectiveness and time management.

1.1 Statement of the Problem

Ceramic ware production at traditional level within local communities in Nigeria are both historical and immemorial. Contemporary concept of production in Nigeria started from the early twentieth century to the mid-20th century when Mr. Micheal Cardew introduced the contemporary concept of ceramic practice along with an already existing traditional method at Suleja near Abuja. Ahuwan, (2003) and Fawowe, (2004) posit that coupled with introduction of ceramics into school curriculum, eventually, industries emerged amidst the very favourable production environment such as access to importation of raw material, functional infrastructural facilities and strong monetary base (Naira) that could be used for importation of goods and services.

With the political dispensation of the civilian administration in the 1980s there emerged an upsurge in the economic stability of the country which had a significant negative implication on Nigeria's economy and industrial growth. The outcome of all these are, inflation, unstable exchange rates, difficulty in importation, power instability and collapse of infrastructures. All these affected industrial developments including ceramic manufacturing.

It is true that imported ceramics have purchase advantage over locally made ones, which might have been due to method of production. The methods of production overseas are through mass production which encourages industrial production and growth. The consistency in design with perfect use of gypsum plaster (plaster of Paris) arising from emphasis put on product design of pro-to-types before working moulds are produced fro models. This aspect of ceramic production is a shortcoming to local ceramic production in Nigeria which invariably translates to higher cost of overhead in production cost.

It is obvious that Nigeria has large geological deposits of Gypsum which could be used to produce plaster of Paris but this has been undermined significantly when compared to other countries like Canada, United States of America and Mauritania. These countries have developed their gypsum for the production of plaster of Paris. The importation of this commodity into Nigeria as a country has become very expernsive for cottage level utilization hence the need for local research like this to boost local use of Nigerian gypsum for industrial use. This study there is to inquire into the chemical and mineralogical properties of gypsum deposits obtained from Wurno, Dange and Weppa as raw materials and their comparative suitability for the production of ceramic plaster. The study will also endeavour to establish the characteristics of plaster made from the gypsum obtained, determining the water plaster ratio suitable for ceramic mould making and carrying out the comprehensive comparative

analyses required from the three deposits, using approved or acceptable parameters for the analysis.

1.2 Aim of the Study

The aim of this study is to process and assess the suitability of local gypsum deposits from Wurno, Dange, and Weppa areas with a view to apply them in ceramic mould-making. This will be done through the processing of the gypsum into plaster and undertaking comparative analyses of gypsum samples from the three sites mentioned above.

1.3 Objectives of the Study

The objectives of the study are to:

1. sample and assess the chemical properties of gypsum obtained from Wurno, Dange and Weppa areas by means of x-ray fluorescence spectrophotometer (XRF).
2. assess the mineralogical properties of Wurno, Dange and Weppa plaster by means of x-ray diffraction (XRD)
3. process Wurno, Dange and Weppa gypsum into plaster of Paris for ceramic mould making, using available standard process,
4. produce test bars of various plaster / water ratio from samples of processed plaster for expected tensile strength and absorbency tests
5. carry out physical performance tests such as; shrinkage test, absorbency test, compressive strength test, density test and mould release test.
6. produce moulds from the best water / plaster ratio mix from test bars, and make comparative analysis of the behaviour of plasters obtained from the three sites along the path of slip casting, press moulding and so on to ascertained and establish their performance variations.

1.4 Research Questions

1. Using X-ray Fluorescence Spectrophotometer (XRF), what are the chemical properties differentia among gypsum obtained from Wurno, Dange and Weppa?
2. Using X-ray Diffraction (XRD), what are the mineralogical properties of plasters obtained from Wurno, Dange and Weppa gypsum samples?
3. What is the casting performance of Wurno, Dange and Weppa plaster when standard water/plaster ratio is applied?
4. How responsive is the plaster/water ratio mixes in relation to total water absorbency of the processed plaster from the above mentioned three locations?
5. How effective are the shrinkage, absorbency and compressive strength capacity of the plaster performance to mould release?
6. Are there any significant differences in the outcome of the final cast wares from the mould produced using plaster of Paris processed from the three study areas for this study?

1.5 Justification for the Study

The absence of locally processed plaster has resulted in high cost of ceramic production in Nigeria because of high cost of purchasing imported plaster of Paris. This study seeks to harness the locally available gypsum for manufacturing of indigenous plaster of Paris such that the dependence on importation could be drastically reduced and subsequently impact positively on the cost of ceramic mass production in Nigeria. It is also to provide a good data base for all findings such as information on gypsum locations, chemical composition of gypsum and procedure to produce plaster with the material from the varied locations.

1.6 Delimitation

The study is delimited to gypsum obtained around Wurno, Dange and Weppa in Sokoto and Edo States of Nigeria, for ceramic plaster processing.

1.7 Limitation of the Study

There are materials in terms of literature on this study area but detailed information on the processing of local gypsum for the three sites in this project are non-existent. Availability and accessibility of places for analyses of mineral or material are limited, thereby time consuming. The present security situation in Nigeria posed a big challenge, especially in selecting the locations of gypsum to access within Nigeria, especially in the North Eastern Nigeria.

1.8 Significance of the Study

The significant of the study is to provide a comprehensive data base for practical information on all tests and analyses carried out within the study. It expanded the horizon of indigenous technology into development of locally processed plaster of Paris for pottery and ceramic purposes. The study revealed the potentials of the local gypsum mined in Nigeria with potentials in contributing to the economic development of the nation. It would provide local substitutes for plaster which could be made by indigenous industries and investors who may want to turn the results of the research into wealth and useable products.

1.9 Scope of the Study

The study covers the identification of the different properties of gypsum, process of plaster production technology and comparative analyses of the various gypsums obtained and eventual utilization of processed plaster of Paris for mould-making for ceramic manufacture.

1.10 Basic Assumption

This study assumes that the raw gypsum rock obtained from these locations would display sufficient characteristics that will make them suitable for all the processing into plaster moulds and also pass the intended tests suitable to accomplish this study. It is also assumed that sufficient electricity supply would be available during the cause of plaster processing.

CHAPTER TWO

LITERATURE REVIEW

2.0 Introduction

A study of this nature necessitates the need to investigate and establish existing studies already carried out and available in the area of raw material sourcing and plaster of Paris manufacturing. The existing literatures on useable gypsum vary due to the various fields or discipline of study. It is fact that native or raw gypsum is useful at different presentable stages, either as raw mineral gypsum rock, crushed or calcined as each case may be. However, literature review on gypsum will also be in areas such as various natures and forms of gypsum and on plaster of Paris. A general discourse to address the topic proficiently is reviewed under the list of sub – topics drawn as follows:

- Introducing gypsum
- History and process of making plaster of Paris.
- Information on various tests necessary for this study
- General information on slip and casting.

2.1 Conceptual Framework

From all that has been said, gypsum plaster or plaster of Paris is hemi-hydrate of calcium sulphate or ($CaCO_4$ ½ H_2O) a product from the natural mineral gypsum ($CaSO_4.2H_2O$). Gypsum rock is peculiar in nature when compared with other earth minerals in that, when gypsum is crushed and heated to temperature 120^0C and above but not beyond 200^0C, 75% or ¾ loss of water of formation is recorded. This process of heating gypsum powder is called calcinations. However, when this calcined powder is mixed with water, the water of formation lost is replaced as plaster reverts to the solid state before calcinations. It is this attribute of gypsum that makes it valuable for mass production in ceramics.

Researchers who have experimented with gypsum have done so with intent to achieve specific ends. Gypsum use are vast and extensive as available from various literatures, USG (2002), OPC (2014), Kumar, Saini and Sameer (2013); BSG (2006) and Elsen (2006) etc. Gypsum is useful in general and art production, building, construction/architectural engineering, agricultural and medical use e.g. orthopaedic, denture and pharmaceuticals. Be this as it may, the concern of this study is the use of gypsum plaster in mould-making for ceramic production.

Pusukumtana and Watanopas, (2001), Guler and Cigdem, (2010) agree there are three types of gypsum plaster namely alpha, beta and gamma or α, β, γ variations. According to this sources alpha plaster is produced at 160^0C. It is said to be high in natural strength, low in porosity and low in expansion. Beta plaster was said to be high in porosity, low expansion and low in strength.

Pfeifer, Wang and Eyerer, (2013) and Lokuliyana, Perera and Gunawardena, (2013) produced ceramics from moulds of different types. Pfeifer *et al* (ibid) used a mixture to achieve porosity in mould for ceramic products whereas Lokuliyana *et al* (ibid) studied and produced plaster of Paris from discarded moulds. Plaster of Paris when mixed with water, reverts to previous state of $CaSO_4.2H_2O$ or gypsum chemically and structurally. Therefore, this attribute of used moulds was exploited in that investigation by Lokuliyana *et al* (2013). Used plaster moulds were crushed and calcined in temperature 180^0C for two hours in an open atmosphere.

A similar study was also carried out by Chandara, (2007), used ceramic casting mould was also exploited in this study. In the "Role of Catalyst in Effecting Morphological and Mechanical Characteristics of High Strength Alpha Gypsum in Plaster" by Singh (2013) synthetic plaster was used for the study by the name phosphogypsum. In this report U.S.

Gypsum Company and Hydrocal, Gardener (these are gypsum manufacturing companies) are also mentioned to have exploited the use of synthetic gypsum for the production of plaster of Paris. The experiment was carried out in an open kiln for 30m – 2hrs. The aim was reducing calcinations period from the normal 6-7hours to about two hour period.

In Akpakip (1993) and Oren Plastering Company (2014), natural gypsum was used in producing plaster of Paris for ceramic production and as building/construction material respectively. Oren plastering (2014) is silent on temperature range but agrees with Akpakip (ibid) on calcinations for roasting of gypsum rock for plaster of Paris. Akpakip (ibid) exploited the use of kiln with temperature ranging from 120^0C to 180^0C for three hours. The thrust of this study is to exploit the experience garnered from literature and such as presented by Akpakip (1993) to achieve the expected aims and objectives of this investigation.

2.2 History and Process of Making Plaster of Paris

Plaster according to Oren Plastering Company, (2014) is one of the most ancient media used by ancient men in handicrafts as well as building. Historical evidences show that primitive man plastered mud over a framework of sticks and reeds to enclose a protective structure to keep out the natural elements e.g. rain, sun, etc. Another instance is that of the Pharaoh of Egypt who used plaster surfaces in their palaces and pyramids. It has been observed that the plaster work and the decorations on it were done more than four thousand years ago. The plaster surfaces still appear in hard and durable state today, even after so many years. The ancient hieroglyphics were often painted on smooth plaster surfaces, according to Oren Plastering Company (2014). An example is in old Greek architecture where stone and plaster were used in the interior of an ancient building. Another ancient architectural structure is the Weis Church built in 1754.

The study of ancient Greek architecture reveals that plaster was used by the Greeks at least 500 years before the birth of Christ. Incidentally, it is from the Greek that the word "plaster" came. The Greek word translated in English would means "to daub on" which is the same meaning with "to plaster" in English. However research has shown or indicated that, principal tools, process of developing plaster and various use of it is practically identical to those of modern day applications. Study has also shown that the finest plasterwork accomplished by the ancient Egyptian was made of plaster produced from calcined gypsum (gypsum made into powder by heat action) or roasted gypsum, very much like the way plaster of Paris is made in today's world. (Oren Plastering Company, 2014).

According to Singer and Singer (1971) plaster is used in ceramic industry for three related purposes as stated here; (a) for model making, (b) for making moulds for pressing and jiggering shapes from plastic clay, and (c) Making moulds for slip casting wares. This author favours 78 parts by weight water to 100 parts plaster and 90 parts by weight water to 100 parts plaster for casting moulds, or better put in this way 100:78 and 100:90. In this presentation, it is important to note that plaster is stable at 100 part whereas water is varied. It is interesting to note that Singer and Singer (ibid), Hamer (1975) both agree to this principle. Hamer believes that ratio mix affects setting time, final strength and absorbency of the set plaster after it has dried. Hamer (1975), Singer and Singer (1971), USG (2002), Akpakip (1993) all agree that the closer the water rate to plaster, the more porous and weaker the mix. While Hamer (1975) says a fresh plaster in good condition should give 3 (three) to 6 (six) minutes of pouring time, Singer and Singer (1971) suggests from 15-30 minutes of setting time.

Nelson (1978) says plaster setting time is from 3 to 4 minutes while plaster hardens in about 20 minutes. Whatever the setting time, these authors record is correct because Hamer (1975)

and Singer and Singer (1971) advise on first testing plaster to determine setting time. The dry mould has twice the strength of a wet plaster (Singer and Singer, 1971). It is also noted that using hot or high temperature to dry damp or wet moulds is not advisable but mould should be dried in moderate temperature not exceeding 45^0C. This way mould life can be prolonged. Therefore drying of moulds each time they are used is advisced.

Like in some other profession, there are risk/hazard factors in practice of plaster production, fine gypsum powder constitutes health hazard when a worker is unprotected. It is useful and advisable for all producers and users of plaster to be armed with knowledge on how best to handle plaster of Paris (http://www/inchen.org/doc.) This especially is true during the period of plaster processing and use because during the stirring/turning plaster from one container into another, loose plaster powder fill the air. Plaster is not to be inhaled. Sensitive skins and eyes are not to be exposed to plaster. Smoking ought not to be engaged when plaster dust is presented. If inhaled, it could induce cough, redness of the eyes, if eyes are exposed to plaster powder. For this reason, use of protective gloves, wearing of safety goggles/spectacles, breathing protection/nasal cover and facial masks are strongly advised when working with plaster of Paris.

2.3 Gypsum Rock, Types, Uses and Theories of Formation

Several studies have been carried out on both the physical and chemical environment that favours natural formation of gypsum and anhydrite, the replacement of one mineral rock by another. Anhydrite is a mineral very much like gypsum but without the water of crystallisation, and with chemical formula $CaSO_4$, it is found most frequently in evaporate deposits with gypsum. The name anhydrite was given by A. G. Werner in 1904 because of the absence of water of crystallization in contrast to the presence of water in gypsum. (en.wikipedia.org/wiki/Anhydrite) Based on these studies, many theories or concepts have

been put forward in an attempt to try and explain the various thick deposits of gypsum found around the world.

According to available literature, the formations of earthy or non-metallic minerals take a period of thousands and hundreds of years. However, it is assumed by some writers that the word gypsum is from the Greek word "gypso" meaning "chalk or plaster" Gypsum (calcium sulphate) generally occurs as sedimentary deposits and this is common in much of the geological records evident from literature. Gypsum is rampant among carbonate rocks especially dolomite and do occur in saline lakes. In fact, gypsum is commonly formed in the marine environment of sea-water as opined by Deer, Howie and Zussman (1962), Palache, (1951), Wright, Hastings, Jones and Williams (1985) and also around oxide portion of ore deposits. However, two of the three authors state that gypsum could be formed in volcanic environments associated with metamorphic rocks. In another submission, Gypsum Supply Co.(2014) States that early use of plaster dates back to 4000 B. C. when the Egyptians applied gypsum plaster-like substance to the interior and exterior of pyramids. It was also noted from this same source that, one Benjamin Franklin brought gypsum to the United State in the mid – 1700s as soil additives.

Read and Srebrodolsky as reported in Akpakip, (1993)] acclaim that there some varieties of native or raw gypsum, namely; selenite or crystallized form, alabaster which is a fine-grained, compact snow-white or light-coloured massive variety. The third and last variety is the satin spar or fibrous variety which has a silky lustre. In colour, the hue of gypsum ranges from colourless to white, and sometimes from yellowish hue to red. The hardness of gypsum rock in light of the various opinions of most writers ranges between 1.5 and 2. This hardness can be scratched with an adult finger-nail (Akpakip, 2004).

It is important at this time that the existence of another type of gypsum be established in order to balance the discussion on the different types of gypsum. Euro: GYPSUM (2007), the factsheet on gypsum says, that gypsum industry is working towards building value for the society by offering safe, economic and recyclable products for the home owner using substitutes to natural gypsum whenever possible. This is in order to reduce pressure on natural resources. According to this source, an intelligent alternative is the gypsum derived from the flue gas desulphurisation (FGD). This is the end product of wet purification procedure with natural lime that essentially forms according to the same laws as natural gypsum, but this time, in a speeded – up process taking only a few hours as opposed to natural gypsum that is formed over two to four million year period of time according to British Geological Survey BGS, (2006), United States Gypsum USG, (2002).

It is purported also that the FGD or synthetic gypsum has a higher purity of gypsum content of 96 percent than most natural gypsum of 80 percent. Apart from flue gas desulphurisation (FGD), there are other types of synthetic gypsum namely, Phosogypsum, Titanogypsum, Atrogypsum. It is however important to observe that the just mentioned synthetic gypsum may not be the only known synthetic gypsum, as it is also acclaimed by the same source that some amount of gypsum are recovered by the production of some organic acids e.g. Tartaric, Lactic, Formic, and Oxalic Acids, BGS (2006) and USG (2002).

Even though the issue that has to do with synthetic gypsum has just been discussed, it is an exercise done to bring variety in knowledge so as to enrich the study. It is an academic exercise; therefore this study will in no way deal more extensively in the area of synthetic gypsum other than what has been discussed so far. This is so because synthetic gypsum is not essential to the core subject of this study.

Gypsum as posited by Kumar, (2013) is one of the important industrial minerals. It is hydrous calcium sulphate ($CaSO_4.2H_2O$) having a composition of 79% calcium sulphate and 21% water. Gypsum in its natural form also finds applications in the manufacture of hydraulic cements, ammonium sulphate fertilizer, sulphuric acid and soil reclamation for agricultural purposes. When crushed and calcined into powder form it is plaster of Paris and if enriched with additives like accelerators, retarders, fillers and binders, the use of gypsum are varied and extensive. The calcined gypsum or plaster of Paris is used in building / engineering / architecture, pottery, ceramics and orthopaedic medicine according to plaster grades (British Geological Survey, 2006).

However in Nigeria, according to available publications, Ministry of Solid Minerals Development, (2000) and Mohammed-Sanni, (2002) gypsiferous shales are reported from the upper Cretaceous Dukamaje Formation and the Paleocene Dange Formation is available in the Sokoto area. The 1.46-million tonne gypsum deposit at Wurno also in Sokoto State is currently being mined by small-scale miners to supply the Sokoto cement plant. Other gypsum prospects are reported from Nafada/Bajoga in Gombe State, at Fika in Yobe State, Weppa area of Agenebode in Edo State, and at Guyuk/Gwalura in Adamawa State (Ministry of Solid Minerals Development 2000). Gypsum is a natural mineral which has the formula $CaSO_4.2H_2O$. Gypsum is a soft, transparent or translucent mineral composed of crystallized calcium sulphate ($CaSO_4$), found naturally primarily in sedimentary deposits. Gypsum is used for the manufacturing of various items e.g. wall boards, paper, paints, plasters, cement etc.

According to Gypsum Recycling International (2007), in total 80 million tons of plasterboard, wallboard and dry wall is produced every year. According to literature there is gypsum in Nigerian soil, Mohammed-Sani, (2002) claims there is in an occurrence of gypsum in Mutwe area of Yobe State, Nigeria. In yet another development, it is also stated by Aribisala and

Adegbesan; in (Ministry of Solid Minerals Development 2000). The reserve estimate of the Sokoto phosphate deposit is about 5 million tonnes. The Raw Materials Research and Development Council (RMRDC) has therefore set up pilot plants for the mining and processing of phosphate rock in Sokoto State (Ministry of Solid Minerals Development 2000). Mining is currently ongoing according to this source.

Many researchers and writers, USG, (2014), Naturesway Resources, (2014), Office of the Deputy Prime Minister, (2006) including Mohammed-Sanni, (ibid) and Akpakip, (ibid) agreed that plaster usage could be multi – purpose for the production of plaster of Paris which can be used for diverse purposes, ranging from orthopaedic, dental, wall/roof spraying, plastering or decorations, and production of ceramic moulds for mass production purposes as presented by Akpakip, (1993), supports the use of plaster of Paris in ceramic mould making for mass production, while Okojie, (1987) favours the use of plaster moulds for slip casting; Sullayman, (1991) used plaster moulds for jiggering and jolleying techniques and Lee *et al*, (2012), used plaster moulds for a modified slip casting methods as in the case of denture casting (Giordano and Mclaren, 2010). Therefore there seems to be a consensus on the use of plaster of Paris for ceramic mould making.

The industrialised nations account for the largest share of gypsum production in the world, while the United States of America is rated the highest consumer of gypsum. There are other nations that are engaged in the exportation of gypsum, chief among them is Canada. There are other Countries that also export gypsum in substantial amount includes Spain, Poland and Germany. The annual consumption of gypsum in the United States of America ranges from three to five million tonnes. This is procured from fifty – two mines where gypsum occurs mainly in Silurian strata, in the eastern states. The Silurian strata is a geologic period and system of reading the time of geological findings, this time period is said to be that which

extends from about 443.4 ± 1.5 million years ago (mya). (en.wikipedia.org/wiki/Silurian). Canada is said to exports gypsum in the form of crude rock. (Mohammed-Sanni, (2002), (Euro: Gypsum, 2007).

Sikombo in Mohammed-Sanni, (2002) reports that; Zambia obtains a form of gypsum by neutralizing the sulphuric acid effluence from the electrolytic copper refineries with calcium carbonate ($CaCO_2$). Such a by-product of gypsum is only suitable for cement manufacturing. In Nigeria however, gypsum consumption and production for industrial application is in the range of between 6,000,000 to 8,000,000 tonnes per year. The Nigerian gypsum production capacity in comparison with the available record of production in the United States of America is very low. The US out-put is to the tune of 20,000,000 to 150, 0000,0000 tonnes per year (Afric Mining Co, 1996). In some places however, gypsum is used as a by-product of scrubbers used in coal-fired power stations. In North America, an extra 0.7 metric tonnes of by-products of gypsum is consumed. On the other hand, all of 6.0 metric tonnes of gypsum is procured as by-products from other industries and interestingly, in Eastern Europe, gypsum consumption follows the similar pattern. The type of gypsum introduced (synthetic gypsum) is relevant to the discussion, in that, it will present an occasion for the researcher to determine the desired nature of gypsum suitable for this study.

Holding this thought in mind, it is pertinent to state that one of the focal points of this study is to procure locally mined gypsum from two locations namely; Wurno, Dange area of Sokoto State and Weppa in Agenebode Local Government Area of Edo State. This is to further investigate the samples so collected, to ascertain property constituents as gypsum and process them into plaster of Paris that can be suitable for ceramic moulds. It is the intention of this study to also make comparative analysis of these various types of plaster of Paris processed from the two chosen locations within the study. This is to determine their individual and

comparative strengths at selected ratio mixes within available and acceptable parameters and produce plaster moulds from these two locations to determine their suitability for use in Ceramic mass production. Akpakip (1993) and Mohammed-Sani, (2002), both claim availability of gypsum in Sokoto and Yobe States of Nigeria.

This study aims at investigating these claims, processing the gypsum for ceramic plaster moulds, and by effecting comprehensive comparative analysing of the findings originating from the study. This will establish and justify the claims and contribute to knowledge in the area of ceramic materials sourcing, processing and production. Gypsiferous shales are reported from the upper cretaceous Dukamaje formation and the paleocene Dange Formation in the Sokoto area. The 1.46-million tonne gypsum deposit at Wurno in Sokoto State is currently being mined by small-scale miners to supply the Sokoto cement plant. Other gypsum prospects are reported from Nafada/Bajoga in Gombe State, at Fika in Yobe State, and at Guyuk/Gwalura in Adamawa State (Ministry of Solid Minerals Development, 2000).

It is also evident from available literature that there are ample amount of gypsum deposits in Dange/Wurno areas of Sokoto State and Weppa in Agenebode of Edo State as contained in Mohammed-Sanni (2002) and Akpakip (1993), while in Mohammed-Sanni, (ibid) the location is called Fika site, Akpakip (ibid) calls it Grujaja-Fika. Obviously, from investigations, the general name for the larger area of where the gypsum is naturally deposited and located is called Fika while the specific mining site or base from where gypsum collected was mined was located at Grujaja area of Fika situated in Yobe State of Nigeria. Afric Mining Corporation was the company or body that mines gypsum rock from this specific area according to both sources.

Another area of investigation will be on the authenticated claims of different types of gypsum either as crystallites, selenites, gypsite, alabaster, etc. It will be expected of this investigation

that the character of gypsum rock obtained for the study from these two locations be ascertained before production of plaster of Paris for this study commences. There are mainly two sources of gypsum according USG (United States Gypsum), (2002), Euro:Gypsum, (2007); and British Geological Survey, (2006), in Factsheet Natural Environment Research Council,. These other sources are, the naturally mined gypsum and the synthetic gypsum, the latter was briefly mentioned or introduced above. Even though this synthetic gypsum is not the focus of the study, the mined or naturally mined gypsum is the material selected for the study, attempt shall be made to briefly discuss the synthetic gypsum so as to enrich and bring the study abreast with topical issues relative to discussion within this study.

As earlier mentioned, the United States of America and most industrialized nations of the world appear to be the highest consumers of gypsum. The gypsum industry as contained in Euro: Gypsum, (2007) is working towards building value for society by offering economic and recyclable products for the home owner using substitutes to natural gypsum whenever possible, in order to reduce pressure on natural resources. An intelligent alternative (according to this source) is the gypsum that comes from the flue gas desulphurisation plant otherwise, known as FGD. It is the end product of a wet purification procedure with natural lime, which essentially forms gypsum. This is according to the same laws that form natural gypsum but this one occurs in a very fast period or speeded-up process taking only a few hours. It is an important supplement to the supply of natural gypsum.

The origin of gypsum has been debated for over a period of over fifty years, an incorrect assumption of gypsum origin for an example was that found in Van't Hoff *et al* (1903) in Germany which presented the idea that perhaps calcium sulphate is primary in its origin. This presentation, according to further investigations was found to be based on an incorrect assumption, due to the fact that hemihydrates which is calcium sulphate with a ½ (half)

molecule of water or ($CaSO4.½ H_2O$) is actually formed at a four-phase equilibrium point at 42°C (forty - two– degree) centigrade. A situation like the one above demands careful investigation in respect of all intellectual topics or discourse, so that an incorrect fact might not be taken for a fact, throwing a large population of people to believe in error.

According to Sullayman, (1991), Arayela, (2006) in the factories and industries where various ceramic wares are produced (or manufactured) moulds are used as a convenient mode of production, whether slip casting, jiggering, jolleying or dry casting, moulds answer the question of standardisation, uniformity, time and material management in the factory. Though the studio potter and the traditional practice may be sufficient with throwing and making standards, industrial production can only suffice with moulds. Generally speaking, mould making is important in the production process of ceramics. The importance of gypsum in making moulds cannot be overlooked because naturally mined gypsum is the universally accepted material used in the production of plaster of Paris and with plaster, ceramic moulds are made.

Arayela, (2006) in a lead paper at the conference of Ceramic Association of Nigeria (CerAN) stated that in ceramics of today, forming techniques have grown in leaps and bounds, from the traditional forming and throwing techniques to forming by using (plaster) moulds. Ceramic forming techniques are actually ways of making objects using ceramic materials and this has been employed in the making of everyday utilitarian wares, engineering ceramics, medical ceramics and ceramic fibre optics in the telecommunication industry etc.

Apart from these mentioned areas, ceramics is also useful in automobile application because of their unique heat, wear, and corrosion resistance, light weight, with electrical and heat insulation properties. Spark plugs, insulators and glass windows in cars, numerous ceramic electric motors are used in modern cars as well. Ceramic composite brake rotors which are

standard equipment on formula-One race cars are now being offered on selected high-performance passenger vehicles. The composite brake rotors reduce vehicle weight by more than ten pounds; resist heat-induced fading and lasts as long as the vehicle. In electronics, the use of ceramics is becoming popular as ceramic super conductors allow the flow of electricity with little or no resistance and no heat loss. All these products of ceramics are standardized in production and therefore use moulds as a matter of necessity. Some high temperature ceramics are either very tiny or delicate and therefore difficult to handle during the forming process; or the material mix or combination is very low in plasticity, and as such, requires the use of moulds in formation process.

The growth of ceramic practice in forming shapes is such that different techniques have evolved over the years. From forming with hands (otherwise known as hand building), which includes pinching, coiling or slabbing methods to throwing on the potters' wheel. Sullayman, (1991), Akpakip, (1993) and Arayela, (2006) present a ceramic technique used in the production of mass quantities of commercial quality, this process or technique is the use of plaster mould as presented in Akpakip, (ibid) and Sullayman (ibid), slip casting in Arayela, (ibid). The process though presented differently by both researchers is one and the same thing, which is the use of mould for slip casting technique, a process of mass production in ceramics. This process relies on the use of plaster moulds to cast with slip, otherwise called slip casting. By this, the slip could be liquid clay or a mixture or blend of different ceramic materials e.g. feldspar, ball clay, kaolin, etc, in water in specified percentage. For high temperature ceramic e.g. porcelain and general mass production for commercial purposes, the use of plaster mould is expedient. It is important to mention that apart from the process of using moulds for slip casting, moulds are also used for pressing or ram extension, jiggering and jolleying and granular pressing.

It is therefore imperative to highlight that gypsum is the universally or generally accepted raw material for plaster of Paris, which in turn is the material necessary for the production of mould for ceramic mass production. This is considered important piece information especially seeing that ceramic is gradually becoming more inter-disciplinary. More so, now that ceramic production techniques are used even in dental replacement art or denture as evident in Guler and Cigdem, (2012); Lee, Lee, and Lee, (2012).

As there is greater need for ceramic products, there is an equal demand on the ceramist to supply those demands through new approaches and targets. One way of achieving new targets is acquiring necessary information and using such information for sustainable development, in the areas of material development and processing of available raw materials. This study is a follow-up to an earlier research work that had been done in the area of development of plaster of Paris with gypsum from (Grujaja-fika of Yobe State of) Nigeria by Akpakip, (1993). There is need to equally carry out a research on the possibility of developing plaster of Paris from other places such as Sokoto gypsum. Earlier studies by Mohammed-Sani, (2002), Harem, (2011), and Ojo, (1995) indicate that there is gypsum in Wurno and Dange areas of Sokoto State. As already mentioned, it is one of the focal points of this study that gypsum will be obtained, investigated and processed into plaster of Paris for ceramic production and findings from this study will be analysed and compared with that done on Grujaja-fika gypsum sample from Yobe State by Akpakip, (1993).

2.4 Temperature Options and Heat Treatment in Plaster Processing

Different categories of temperature suggestions have been made by some authors and researchers, the different suggestions may be due to the end use of the plaster produced. For an example, as earlier mentioned in this study, gypsum could be calcined for various end uses like cement production, orthopaedic casts, and building or construction purposes. In fact,

Singh, (1992) posits that plaster of Paris or gypsum plaster is widely used for a number of things beginning from building, manufacturing of sulphuric acid, pottery/ceramics and Portland cement. According to this source, there are three crystalline forms of α, β and γ or alpha, beta and gamma gypsum plaster. The most commonly used is β – form which is made by heating gypsum or $CaSo_4.2H_2O$ at about 130°C - 160°C in an open kettle or rotary kiln. The making of alpha plaster is made by which consists of heating gypsum in a saturated steam atmosphere for six hours, drying and then grinding. Additives to produced plaster which accelerates or slows down setting time include gum Arabic (1-2%), soda ash, lime or litharge.

Pusukumtana and Wattanopas, (2010) calcined used plaster moulds at 160°C; British Geological Survey (BSG), (2006) posits from 150°C to 165°C, Chandara, (2007) suggests that plaster could be dehydrated at a temperature as low as from 100°C to 160°C. While Mohammed, (2010) subjected ground or milled gypsum to elevated temperature exposure for the purpose of the study on residual compressive strength tests subjecting gypsum to temperature ranging from 100°C, 200°C, 300°C, 500°C, 700°C and 900°C. Most writers agree on the importance of appropriate temperature manipulation and controlled humidity of immediate calcinations environment within the kiln compactment. In a similar situation, Akpakip, (1993) chose to use 120°C and 180°C as desired temperatures. It is the aim of this study to evaluate all suggested temperature to establish the particular one which is best suited and reasonable temperature that may assist in achieving desired and expected results.

A more recent publication as presented in British Geological Survey (BGS), (2006) that gypsum ($CaSO_4.2H_2O$) is first ground to powder and heated at 150°C to 165°C. Kumar, Saini and Sameer, (2013) suggest around 120°C to 160°C. Euro Gypsum (2013) agrees with BGS, (ibid) on grinding of gypsum before calcinations or heating and on temperature choice.

Kaplan, (2013) advices that plaster should be stored in airtight food service container to keep away moisture. No specific mention of temperature is made by this author.

Some literature acknowledge the periods of boiling and settling of plaster. For example, Euro Gypsum, (2013), BSG, (2013), agree on the fact that plaster, during calcinations boils during the hours in the kiln or oven. These writers also attest to the fact that after each period of boiling the plaster settles before another period of boiling resumes. As advised by Akpakip, (1993) that calcination of each batch or feed of plaster should be discharged. In fact, Singer and Singer in Akpakip, (1993) posit that the continuous stirring of the plaster charge, the size of the charge, the ventilation system of the calciner to keep the humidity of the air surrounding the material sufficiently high is capable of producing good plaster. Therefore it could be understood that, production of plaster does not depend on temperature alone but on the efficiency of stirring and ensuring that the surrounding of the charge in the kiln or oven has adequate water vapour pressure.

Gypsum is important for the production of plaster moulds, wall/ceiling decorations, sculpture, and cement production. It is used for the production of Plaster of Paris (P.O.P) and classroom chalk, etc. A strategy for large-scale mining of gypsum used in the cement industries is urgently required to sustain existing plants and meet future expansion. Current cement production is put at 8 million tonnes per annum while the national requirement is 9.6 million tonnes. About one billion tonnes of gypsum deposits are spread over many states in Nigeria. (Mohammed-Sani, 2002).

There is no record of first or ancient use of plaster in Nigeria, but early works of arts, such as some sculptural works of Enwonwu and Demas Nwoko may be close enough evidence of plaster use in Nigeria. If there was any ancient use of plaster in Nigeria, evidence could have been left for posterity. The fact that there is no excavation or ancient historical finding

pointing towards such is indicative of absence of ancient plaster use in Nigeria. That being as it may classroom chalk has been in Nigerian schools as early as when the colonial/imperial system of education came into the country.

Another evidence of plaster use could be traced to the birth of contemporary art practice as mould making was part and parcel of contemporary ceramic art practice. Therefore, apart from plaster featuring in mould making either in sculpture production or classroom chalk production, there is also that possibility of early use of plaster in mould making in the early days of contemporary ceramics in Nigeria (Ahuwan, 2003 and Fowowe, 2003).

It is gathered from various literatures that hydrate calcium sulphate ($CaSO_4.2H_2O$) or raw gypsum when heated to 117^0C looses 75% (seventy-five percent) water of formation during calcinations to become $CaSO_4.½H_2O$ which otherwise is known as plaster of Paris. it is also understood from literature that gypsum from site is crushed to finer particles at different stages, until when gradually plaster is ground into what appears to be flour-like texture. Knaauf, (2013) suggests first, drying it out (that is ground gypsum otherwise called land plaster) before it is fed into the kettle or rotating cylinder for calcinations.

During the period of calcination, the powder is constantly mixed by huge rotors or sweeps within kettle structure, such that temperature is kept uniform. This process is carefully monitored to ensure the correct or exact water removal from gypsum was sustained and achieved. Otherwise, should the kettle or container and plaster get overly heated or prolonged beyond 200^0C. The end product may result in what is known or termed as "dead plaster" not good enough for plaster of Paris but maybe for cement, e.g. Keene cement. Singer and Singer, (1979) says, in plaster production a lot depends on temperature control as well as efficiency in stirring and water vapour pressure management within the kiln during calcinations. The kilns available in the Department of Industrial Design, Ahmadu Bello

University, Zaria has no thermocouple for temperature control as mentioned in Singer and Singer, (ibid) kilns in a Federal Government parastatal (Industrial Development Centre, I. D. C.) in Samaru, Zaria was sourced for and permission granted the researcher to use the kiln available there for this study.

2.5 Information on Various Tests Suitable for this Study

Various tests that are suitable for this study are as follows:

- XRD (X – ray Diffraction)
- XRF (x – ray Fluorescence Spectrophotometer)
- Compressive strength test
- Absorbency test
- Shrinkage test
- Density test
- Practical Casting and Mould Release Test

There are different types of scientific tests recommended or suggested as capable tests for materials and minerals of all types, here are some topical tests; XRD (otherwise known as x-ray Diffractions), XRF or x-ray Fluorescence. Of these tests, the first two are selected, along with modulus of rupture, porosity/absorption test and mould release test, for use and completion of this study.

2.5.1 X-ray Diffractions (XRD)

The chemical properties of a material can often be linked back to the arrangement of atoms in its crystal structure. X-ray diffraction (XRD) is a non-destructive analytical technique which can yield unique fingerprint associated with a crystal structure of such material according to Bragg equation. According to Brauwer, (2010) and Gauglitz *et al*, (2004) a crystal structure can be regarded as something being built of layers, or planes, of which each acts as semi –

transparent mirror. X-rays with a wave-length similar to the distances between these planes can be reflected such that the angle of reflection is equal to the angle of incidence. This behaviour is normally called, “diffraction”, and it is described by Bragg’s Law.

Analysis of the diffraction pattern allows the identification of phases with a given sample. With that achieved, it may be possible to quantify each phase present, the “crystallinity” of a sample, the crystal structure and their lattice parameters, crystal size and strain and all information that can be vital in material characterisation and quanlity control.

X-ray Scattering Technique: This is an x-ray diffraction pattern formed when x-rays are focused on a crystalline material. X-ray scattering techniques are a family of non-destructive analytical techniques which reveals information about the crystal structure, chemical composition, and physical properties of materials.

2.5.2 X – ray-Fluorescence (XRF)

When a primary X-ray excitation source from an X-ray tube or a radioactive source strikes a sample, the X-ray can either be absorbed by the atom or scattered through the material. The process in which an x - ray is absorbed by the electron by transferring all of its energy to an innermost electron is called the “photoelectric effect.” During this process, if the primary x-ray had sufficient energy, electrons are ejected from the inner shells, creating vacancies. These vacancies present an unstable condition for the electron. As the electron returns to its stable condition, electrons from the outer shells are transferred to the inner shells and in the process giving off a characteristic x-ray whose energy is the difference between the two binding energies of the corresponding shells. The emitted x-rays produced from this process are called “X- ray Fluorescence,” or XRF. The process of detecting and analyzing the emitted x-rays is called “X - ray -Fluorescence Analysis.” The XRF method is widely used to measure the elemental composition of materials. Since this method is fast and non-destructive

to the sample, it is the method of choice for field applications and industrial production for control of materials. Depending on the application, XRF can be produced by using not only x-rays but also other primary excitation sources like alpha particles, protons or high energy electron beams.

2.5.3 Compressive Strength

The compressive strength test is done to ascertain the compressive strength of a material. This is especially administered on samples of earthern minerals, and ceramics especially minerals that lack tensile strength and capacity. Plaster of Paris fall into this description.

2.5.4 Shrinkage Test

Some minerals are known to shrink or reduce in size from wet to dry state, clay and other earth mineral inclusive. The shrinkage level in plaster should be minimal and therefore inconsequential to the size of final product.

2.5.5 Density Test

The density of an object is the state or quality of compactness or closely, set of that object or mineral. In calculations, it is expressed as mass per unit volume. The density test is the enquiry to know the compactness and how closely set processed plaster is.

2.5.6 Absorbency Test

The capacity or ability of a material to absorb moisture and to what percentage, is the reason absorbency test is carried out. Processed plaster from the three locations were subjected to absorbency tests and the results is presented here.

2.5.7 Practical Casting and Mould Release Test

As indicated by the topic, processed plaster will be used to produce ceramic slip and dry pressing moulds. This exercise should determine how castable the produced moulds from processed plaster is.

2.6 General Information on Mould Making and Slip Casting

Mattison, (2006) specifies hump moulds, two-part moulds and multi-part moulds and defines a mould as a plaster of Paris former in which clay can be pressed or slip cast to create forms. Mattison, (ibid) and Wensley, (2002) state that slip casting was developed for mass production of pottery wares but contemporary work shows increasingly inventive interpretation. Slip casting utilizes the ability of plaster to absorb water from liquid clay slip when it is poured into a plaster mould. As water is absorbed from the clay slip, it begins to form a layer of solidifying clay on the inside surface of the mould. When this layer has reached the required thickness, excess slip is poured out, leaving the formed or deposited layer to harden or stiffen and shrink away from the mould. Prepared slip for casting should be without lumps, it must be smooth. If lumps are present, then it is recommended that slip should be sieved or a blunger be used in bending slip smooth.

According to Zakin, (2001) the mould forming process has many similarities with print making, just as printmaking first became popular as an economic way to create two dimensional images, slip casting began as an economic way to create three-dimensional forms. Agreeing with this concept, Akpakip, (1993) stated that mould is important in the production process of ceramics. The mould comparatively is to the potter what lino-cut or film negative is to the print-maker or photographer. Therefore, the researcher concluded, the advent of mould, film negative and lino-cut not only made more people able to purchase the now accessible wares but also made production quicker, easier and cheaper. Zakin, (2001)

speaks of sprig as a form of mould unlike other authors who choose to discuss sprig as another form of decoration.

Many authors and researchers agree that in evaluating the existing mould types, plaster mould made from gypsum still performs the best for ceramic slip casting. Zakin, (2001) and Gukas, (1985) in Akpakip, (1993) posit that clay mixed to a homogenous consistency may produce slip but that will make a poor slip for ceramic casting.. The normal clay and water mixture cannot be used as a casting slip because it will quickly saturate the mould, bringing slip casting to sudden stop. In addition to this problem of saturated mould, the clay particles within the slip will settle, causing heavy base and cracks. Therefore the ceramist uses a slip that is specially formulated for the slip casting process. From various literatures, ceramic casting slip should contain deflocculant, it (the deflocculant) breaks the natural bonds that hold the clay together and changes a stiff clay or clay body into a liquid or fluid slip. For this study, Kankara and Bomo clays are considered for use in slip casting. Kaplan (2010) suggests the purchase of slip for casting, believing it is difficult to formulate. A simple composition as contained in Akpakip, (1993) will make a good start. It is as follows; 50 percent Kankara, 50 percent Bomo and 50 percent weight of dry material for water. For the deflocculant, it is with a ratio of 1:3 sodium silicate and sodium carbonate.

CHAPTER THREE

MATERIALS AND METHODS

3.0 Research Design and Procedure

It can easily be deduced from literature that gypsum is the raw material or rock used in developing, manufacturing or processing plaster of Paris. The method employed for this investigation is experimental approach which involves controlling and manipulating conditions that will prevail in the study.

This chapter is sub-divided into stages in order to systematically carry out the research as shown below:

- Field trips and collection of material (gypsum) samples.
- Physical property tests of gypsum
- Laboratory tests and characterization of gypsum
- Pilot study
- Comparative analysis of materials (gypsum).

3.1 Preparations for Field Trip

The journey embarked upon for the purpose of the field trip was guided by various literature dwelling on the on locations of raw gypsum at large-scale quantity. Mohammed – Sani (2002), Federal Government Report (2000) and Rock for Crops (2013) indicates that gypsum could be found at Wurno, Dange and Weppa. Grujaja-fika was the source of gypsum used by Akpakip, (1993) for the Development of plaster of Paris from gypsum for ceramic mould making in Nigeria. The two count reasons that the location of Grujaja-fika was exempted from the chosen locations for this study are as follows;

- Those two studies have been carried out on Fika or Grujaja-fika gypsum site by several researchers from different areas of specialization. They are Akpakip, (1993) and Mohammed-Sani, (2002), who are Ceramist and Geologist respectively.

- The current security situation in the country (Nigeria), especially in the north eastern part of Nigeria made it unsafe for both the indigene and outsiders to visit the sites for research purposes as they face life threatening risks constantly.

Due to the above stated reasons, Grujaja-fika was not considered as one of the study location. Dange and Wurno in Sokoto State and Weppa in Agenebode of Edo State were selected as locations from where raw gypsum were collected.

In Akpakip, (1993) it is reported that an unstructured interview was carried out to elicit respondents' directives on how to access the mineral deposit and their responses, gave a lead to locations where gypsum could be found and possibly collected. According to this report, there were five (5) prospective locations and nine (9) out of fifteen (15) respondents selected Yobe State, they were closely followed by four (4) people who named Sokoto State as (another) location from where raw gypsum could be procured or collected from. As was discussed earlier, because of the reasons given above, Sokoto State was preferred and selected as preference over Yobe State. Therefore Sokoto gypsum located at Wurno and Dange were used. Another location of interest was in Edo State, Weppa in Agenebode local government area. These three locations mentioned above were selected for obtaining gypsum for this study.

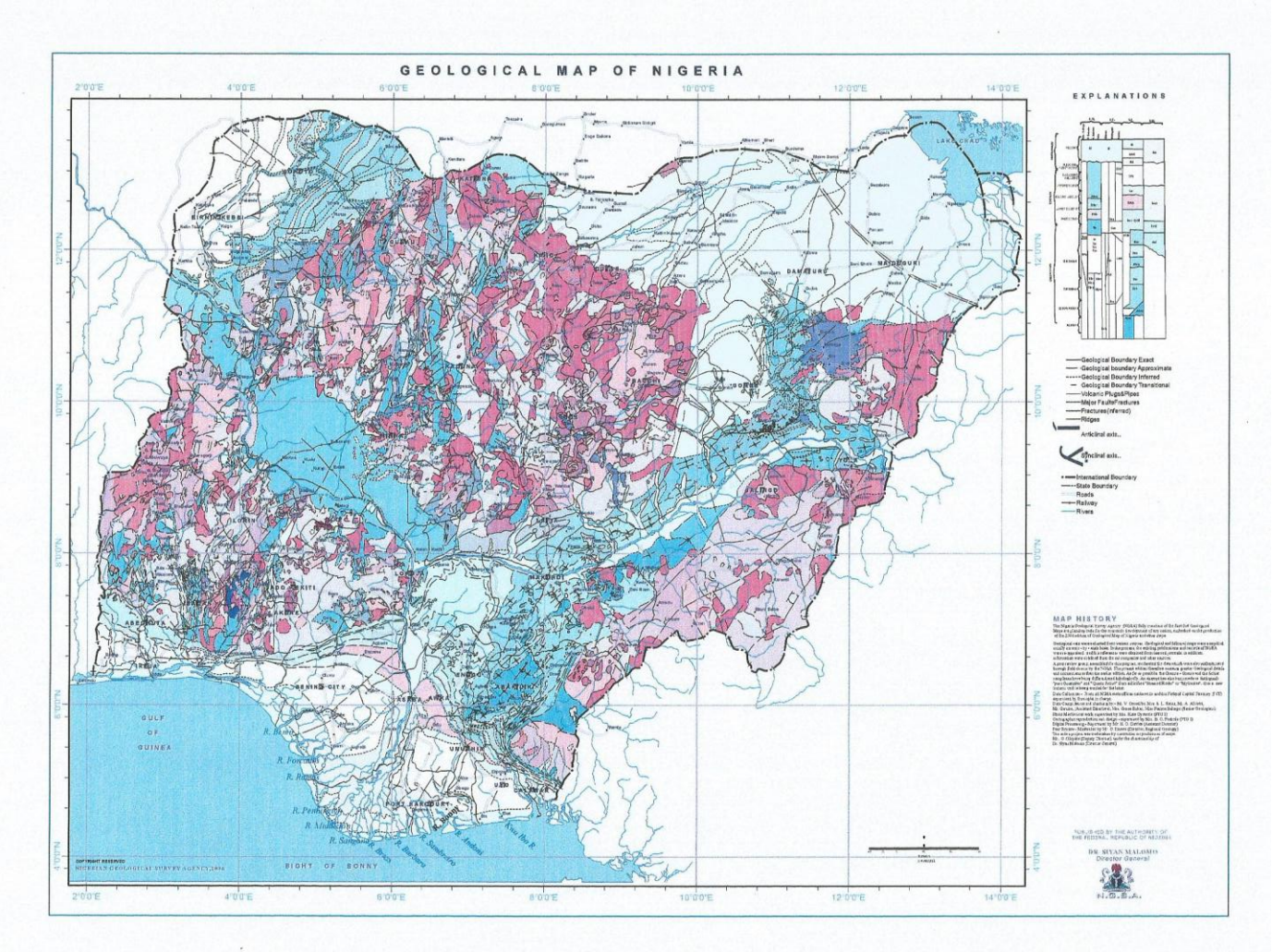


Fig 1: Geological map of Nigeria

Source: Department of Geology, Ahmadu Bello University, Zaria

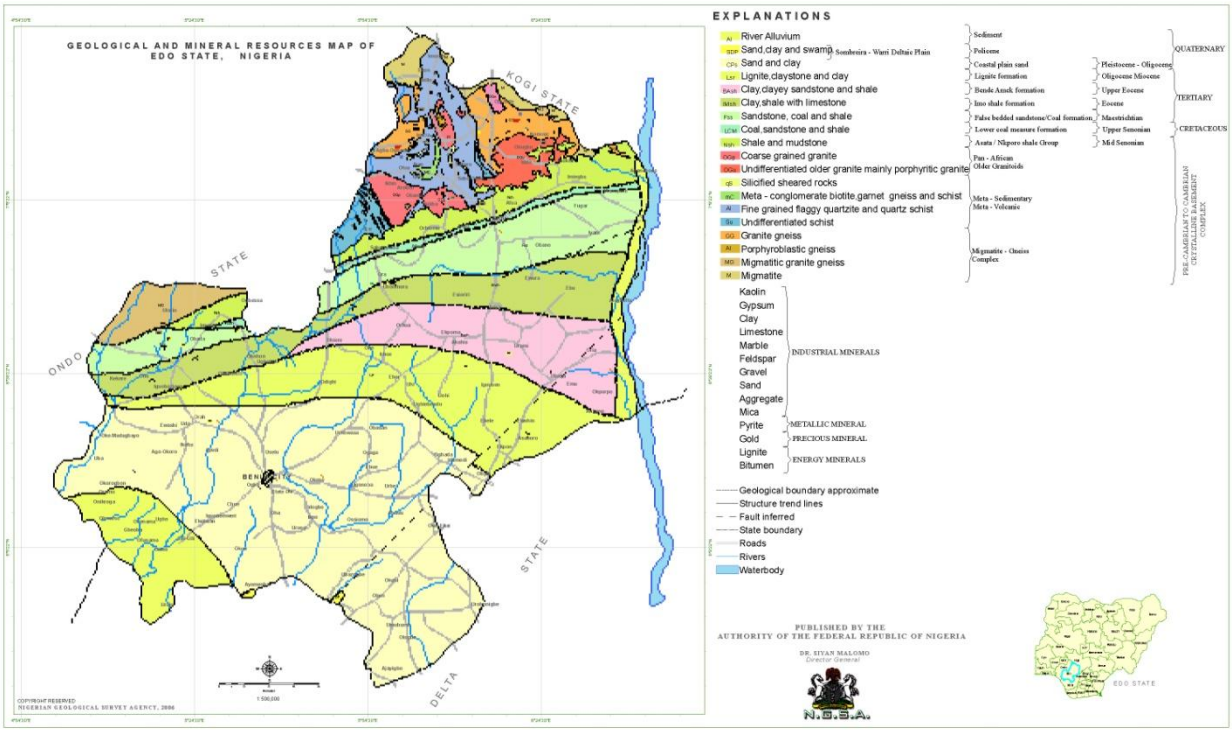


Fig. 2: Geological and mineralogical map of Edo state showing the study area

Source: Department of Geology, Ahmadu Bello University, Zaria

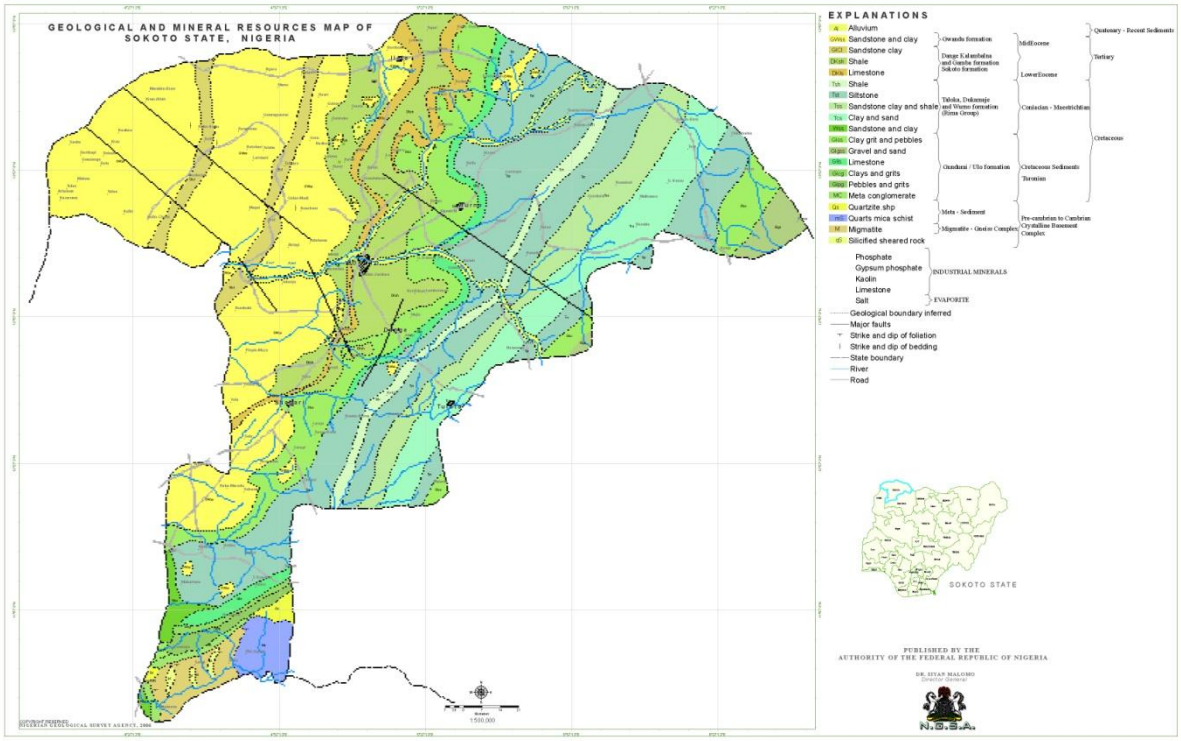


Fig 3: Geological and Mineralogical map of Sokoto State showing study areas

Source: Department of Geology, Ahmadu Bello University, Zaria



Plate I: The Researcher at Wurno site



Plate II: The Researcher at Dange site



Plate III: Washing of gypsum



Plate IV: Obtained gypsum from site

3.2 Field Trips and Prospecting of Material

The process of mining gypsum in Wurno as well as Dange sites was quite similar to the process reported by Akpakip, (1993). In that account, it is the bull – dozers doing the digging at the mining pit exposing the raw massive gypsum. The excavator was then used to extract raw gypsum (with other soil dirt, for example stones, fossils, soil clay, and so on). Raw gypsum mixed thus with stones and soil dirt, was then left in a heap to be manually sorted by trained labourers. The labourers were employed hands, who were constantly there on site to sort out the gypsum.

The gypsum sample was obtained from the location as the researcher made her interest in the mineral (gypsum) known to the interpreter which was translated in Hausa to the contact persons in both locations. Once substantial quantity of mineral was obtained, each bag was labelled with a marker. This was done so that the gypsum from these two locations (Wurno and Dange) would not be mixed up in transit from Sokoto to Zaria. This was how the gypsum was obtained and transported back from Sokoto to the Department of Industrial Design, Samaru Campus of Ahmadu Bello University, Zaria. Some samples were then selected for the Physical Laboratory Tests at the Department of Geology, Ahmadu Bello University, Zaria, with assistance of Dr. Alagbe who is a lecturer in that Department. The first assessment of gypsum obtained from the two locations in Sokoto, there was much sand or (or) earth in the gypsum from these two locations, Wurno and Dange. Before any crushing activities were conducted, gypsum was washed and spread out to dry. Sample at Weppa (gypsum) was obtained with much ease for this study.

The importance of the physical tests for minerals cannot be over-emphasized. This is because these tests are necessary for the identification of the minerals and in this context, gypsum. The physical tests include streak, cleavage, hardness, colour, tenacity; form, or shape,

specific gravity, and lustre. Although the physical tests could be more than these, Akpakip, (1993) in her report had seven (7) as against the eight done in this study. The tests done by Akpakip, (ibid) is the normal expected physical tests carried out on mineral for identification. The inclusion of tenacity test increased the physical test done by the researcher by one, making it eight (8) in the decision of this researcher.

3.3 Physical Gypsum Tests

3.3.1 Streak Test

This is one of the simplest physical test and yet equally very important. The streak of a mineral is the colour of its powder which may be very different from that of the mineral in massive form. This means that the streak of a mineral is the colour of the fine powder of that mineral produced when it is rubbed or scrapped against a streak plate. A streak plate usually is a whitish unglazed porcelain flat tile (plate) used in determining the streak of (powder colour) of any mineral.

Experiment:

Raw gypsum was used to carry out this experiment.

Test Instruments:

The instrument used was Streak Plate (From Department of Geology, A. B. U. Zaria.). In this experiment, the streak plate was obtained from the Department of Geology, Ahmadu Bello University, Zaria. The gypsum sample was rubbed against the surface of the streak plate until fine collection of gypsum powder was achieved. In the experiment, the streak produced was white as indicated in available literature. The streak test was carried out for gypsum from the three locations of Wurno, Dange and Weppa one after the other.

3.3.2 Cleavage Test

The cleavage of a mineral is the clean, smooth and easy parting of layer in that mineral. A cleavage plane therefore is the line of natural weakness in mineral structure along which the layer of mineral could be removed. The experiment and instruments for the experiment are as listed below.

1. Raw gypsum

2. Razor blade, or finger nail

With either the razor or finger nail, the procured gypsum was subjected to cleavage test by using both to attempt tracing the top layer of gypsum from the lower layer. This came off peeled off the lower layer. Procured gypsum sourced were both of selenite and fibrous varieties with perfect cleavage.

3.3.3 Hardness Test

Akpakip, (1993) and Gribble, (2004) state that hardness of one mineral differs from one another significantly. The determination of a mineral's hardness is considered as one of the important tests in identifying mineral characteristics. The hardness of gypsum was measured on the Moh's scale of mineral hardness.

3.3.4 Moh's Scale of Mineral Hardness:

1. Talc.
2. Gypsum.

   2.5 Adult fingernail
3. Calcite.
4. Fluorspar.
5. Apatite.

   5.5 Window glass and Pen knife blade

6. Orthoclase Feldspar

   6.5 metal file.

   Quartz.

7. Topaz
8. Corundum
9. Diamond.

   Sources: Hamer, (1975), Akpakip, (1993); Gribble, (2004)

The samples used for these tests are talc and calcite. The Fingernail was used by the researcher.

The hardness of gypsum was determined by "elimination" in line with above given procedure. The fingernail of the researcher was successfully used to scratch the surface of gypsum repeatedly. Gypsum is second (2nd) on Moh's Scale of Mineral Hardness, so gypsum was rubbed against the surface of talc and it scratched it. On the other hand, when gypsum was used to rub against the surface of \calcite, it could not scratch that surface, instead calcite dented the surface of gypsum, from both locations. To further establish gypsum\'s hardness on Moh's Scale of Mineral Hardness, calcite is number three (3) while talc is number one (1) and the softest mineral on Moh's Scale of Material Hardness. This procedure is known as "elimination". Therefore, gypsum through this experiment of elimination occupies the 2^{nd} position on Moh's Scale of Mineral Hardness.

3.3.5 Colour/Optical Properties

Colour of a mineral remains one of its most striking features and quality, Akpakip, (1993) posits that relying on mineral colour could be misleading. Sometimes gypsum is transparent and at other times to translucent in colour. They can also vary from colourless to white and

influenced by impurities. The colour of gypsum may also vary from yellow, tan, pink to blue and brown. Other colours are reddish brown, grey black or colourless in transmitted light.

3.3.6 Lustre

The lustre of a mineral is the character of light reflected by it. The lustre of gypsum is vitreous on examination. For this experiment, gypsum sample used, (still in its massive form) was first washed with water to reduce impurities picked from site. It was then dried until the water on its surface dried up. This was done as the water on the washed gypsum surface may influence or hinder expected result in any way. The washed and dried massive gypsum was held in the hand and slowly rotated both clockwise and anti-clockwise. This observation revealed that the surface of gypsum was shimmering almost like water surface that is, the surface of a chipped and broken glass.

There are two identified varieties of gypsum available from the locations, namely selenite or crystalline and fibrous gypsum samples. Crystalline gypsum sample has a pearly or vitreous lustre while the fibrous variety has silky lustre, (BSG, 2006).

3.3.7 Habit, Form or Shape

The habit of any given mineral is the form, or shape by which such mineral occurs naturally. The question usually asked of mineral occurrence is, is it in granules, cubic or massive form? Gypsum is commonly in tabular and massive forms. Natural minerals such as stones, sand and gravels are examples of minerals which occur in granule forms as oppose to gypsum in larger (massive or tabular) forms. Massive in this regard indicates that gypsum does not occur in aggregate or grains. All gypsum samples used for this study were sourced from Wurno, Dange and Weppa areas and all are either in massive or tabular form.

3.3.8 Specific Gravity

The specific gravity test was conducted on the raw gypsum. The experiment and instruments for the experiment are as listed below.

1. A graduated measuring cylinder
2. Water
3. Glass cylinder
4. Weighing balance
5. A thin rubber band
6. Dry napkin.

The specific gravity of an object (mineral) is the actual weight of the mineral in air, divided by the weight of same mineral in water level displaced (which is increase in volume). The graduated measuring cylinder was filled with water to the level marked 500ml. A lump or massive raw gypsum rock was tied with thin rubber band and suspended into the cylinder halve filled with water. It was observed that the water level rose to 600ml, so the displaced water level recorded was 100ml. The dry napkin was used to dry the gypsum sample and the same gypsum was weighed on a weighing balance. The reading was 230grammes.

The specific gravity of gypsum in this given circumstance equals the weight of mineral in air divided by the weight of displaced water level or increase in volume. Where one millilitre of water is equivalent to 1 gram and M1 is given as mass of gypsum in air, while m2 equals mass of gypsum given by displaced water level. The working is as shown below:

$M_1 \div M_2$

$= 230 \div 100$

$= 23 \div 10$

$= 11.5 \div 5$

= 2.3

Specific gravity of Wurno gypsum equal 2.3

Calculation for Dange gypsum is as shown here;

$M_1 \div M_2$

=99 ÷ 43

=2.3023

=2.3

Specific gravity of Dange gypsum is 2.3, the calculations as shown above.

The calculation for Specific gravity of Weppa gypsum is shown below;

$M_1 \div M_2$

=115 ÷ 50

=2.3

Specific gravity of Weppa gypsum is also 2.3

Specific gravity is one of the physical tests done to identify minerals. Each mineral has its peculiar specific gravity. The result from this experiment has proved that the investigated mineral is gypsum.

3.4 Preparation of Gypsum for Calcination (Crushing/Grinding)

It becomes imperative at this stage to mention that the researcher made use of improvisation of tools and equipment at some stages in this study. The need for improvisation was especially necessary during the milling and calcination processes. It was obvious from the onset of the study that there would be need for improvisation, due to the nature of the research. The first stage of preparation of gypsum from site was to wash and dry, break them down into smaller size and chips before crushing them into powder form. The process was carried out for all the gypsum samples (Wurno, Dange and Weppa) used for this study. The

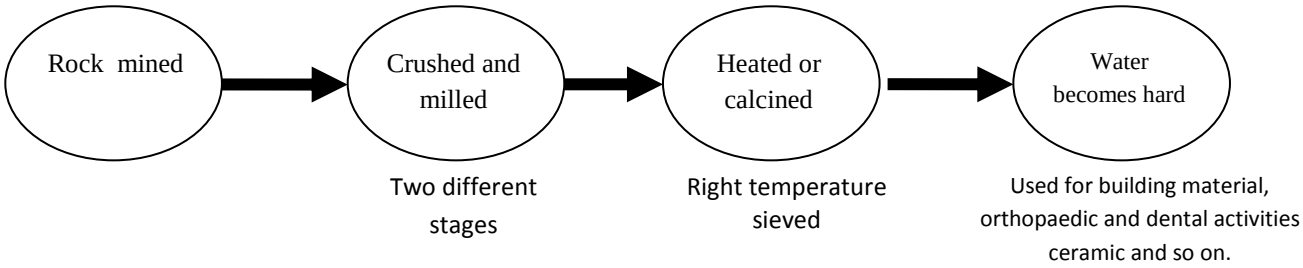
mill needed for grinding gypsum into the required powder size was not available in the Ceramic Section of Industrial Design, Ahmadu Bello University, Zaria., There was need, therefore to look for where this could be carried out. On investigation, it was discovered that there is a ball mill in the Department of Chemical Engineering in the same University where gypsum could be milled into the expected powder size.

It was necessary at this stage that the rock or massive size of gypsum be reduced to smaller particles in readiness for milling. Washed gypsum was taken to the jaw-crusher of Ceramic section of Department of Industrial Design, Ahmadu Bello University, Zaria and reduced to smaller particles before proceeding to the ball mill in the Department of Chemical Engineering. The coarse gypsum (chips or flakes) was then fed into the ball mill for the final milling into what is referred to in literature as land plaster.

The first feed from Wurno (of only 1.5kg) took 9hours to mill but it was observed as unsatisfactorily done. A more effective and faster mill was therefore required at this stage. The researcher was directed to the National Research Institute for Chemical Technology (NARICT) to investigate and ascertain certain the availability of a Rotary Kiln for use and its discovered as accessible for use in the course of this study. The trip to NARICT was also to locate conducive mill for milling the rest of gypsum. On getting to the Institute, Rotary Kiln was available but was out of use because it has broken down for awhile, the situation with the milling machine was handicapped for use due to lack of power supply. Promise on possible repairs on the milling machine by the management, could not materialise two weeks and four days before the bags of gypsum were transferred back to the Ceramic Section, Department of Industrial Design, A. B. U. Zaria.

It was when the bags of gypsum were returned to the Department of Industrial Design, Faculty of Environmental Design, Ahmadu Bello University, Zaria, that the researcher on

further investigation got to know of the mill at the workshop of Department of Metallurgy, Faculty of Engineering, Ahmadu Bello University, Zaria. The machine was discovered to be out of service. The researcher embarked its repair after which the bags of gypsum were transferred there for milling in preparation for calcination. The grinding mill at the Department of Metallurgy was effective and fast. This process was repeated for the gypsum from the three locations namely, Wurno, Dange, and Weppa. The grinding process was done for individual samples and poured back into the labelled sac to avoid samples mix up.



3.5 Calcination of Ground Gypsum into Plaster

In Chandara, (2007), Mohammed, (2010), and BGS, (2006), temperature limits were suggested which range from 100°C to 180°C for the calcination of land plaster. It was however observed that plaster could be processed for a number of uses, even for residual compressive strength test as evident in that of Iraqi gypsum by Mohammed, (2010). Mohammed, (ibid) started his experiment by subjecting gypsum to temperature ranging from 100°C to 900°C for the purpose of Residual Compressive strength. On a closer study it was observed also that the production of plaster for casting and building did not extend to such high temperature as in the case other above researcher. Therefore the temperature limit decided on was informed by that used for the processing of raw gypsum into gypsum plaster for ceramic production as presented in Akpakip, (1993) and of denture replacement art and such similar end product Giordano and Mclaren, (2010); Guler and Cigdem and Singh, (1992).

The temperature range decided on was 120°C, for the first or initial hour and 165°C to 170°C for the second and third hours. This was necessary to retain and control vapour density within the immediate oven environment. The flue of the kiln was also partially shut in line with this objective of vapour control. This procedure of determining how to manipulate or control vapour density within the kiln chamber by the manipulation of temperature and partial shutting of the flue was effective throughout the calcination of plaster of Paris from the three locations.

3.5.1 Determining Temperature

Determining temperature among existing temperatures suggested in literature became a challenge. Akpakip (1993) selected the use of 120^0C for preheating and 180^0C to complete calcination. To support and establish temperature for this experiment, the researcher adopted the preheating temperature of 120^0C and a reduction in the final temperature which was determined at 170^0C which is the normal temperature used for industrial calcination as indicated by (http://cenotech.in/1120-calcination-temperature-of-hymihydrate.php).

3.5.2 Plaster Production Cycle

According to Pasukumtana and Wattanopas (2010), plaster consistency are 60, 65, 70, 75, 80, 85, 90, 95, 100. Beta plaster gives low strength, high porosity low expansion and temperature for calcination is 130^0C. Alpha plaster gives high strength, low porosity and high expansion while temperature for calcination is 160^0C.

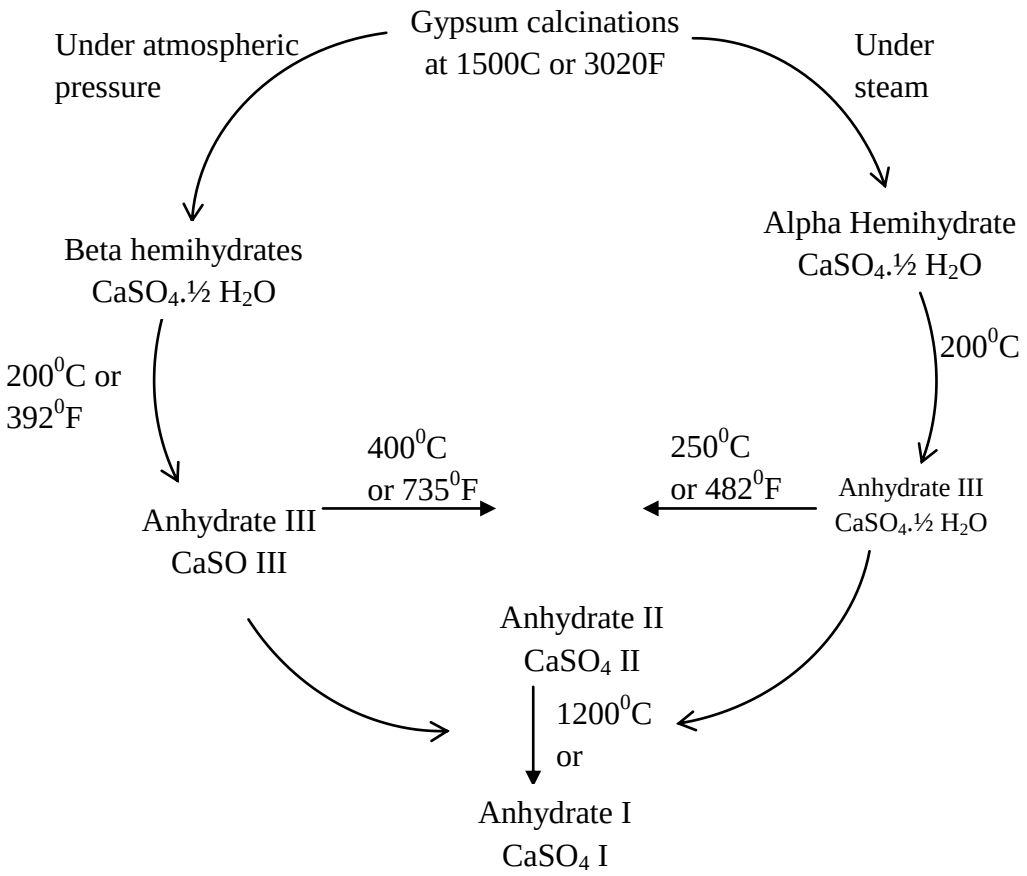


Fig 4: Plaster Production Cycle
Source: Pasumkumtana and Wattanopas (2010)

3.6 Calcination

3.6.1 Kiln for Calcination

The kiln used for the calcinations process is a high temperature electric kiln for producing ceramics wares with maximum temperature of 1400°C. The positive and good thing about it is that the thermal control or thermometric was working effectively. Due to this fact, the kiln was set at 120°C for each first hour and timer was also set for one hour. The temperature was then set at 160°C and 170°C for completion of calcination. The land plaster was weighed before calcination. Calcination period for each process was five hours.

The containers used for this process of calcinations were aluminium two head – pans, two bisque wares, and two Crucibles. Each kiln load of land plaster weighed about 8kg, easy

access to the kilns at the welding section of Industrial Development Centre (IDC) would have made the process fast such that the researcher could have worked to time but for the challenge of power supply. The calcinations process was therefore at the mercy of whenever power (electricity) was available.

It is important to emphasize that due to information garnered from literature, the flue of the electric kiln was partially closed throughout the calcinations of plaster from the three locations mentioned within this study. It is also important to note that at the end of every hour, plaster was structured and turned from one container (pan) into another. By so doing, plaster was kept from local overheating and moderation of temperature of plaster was achieved. This was necessary because from literature, it was observe that in the English open pan system, heavy chains were used in stirring plaster kiln, there are built in stirrers such that plaster is stirred consistently during calcinations. With absence of a working Rotary Kiln and open pan, there was the need for improvisation. The researcher collected bisque plates and crucibles from the ceramic section of the Industrial Design Department of A. B. U. and IDC (Industrial Development Centre) respectively for use in the calcination of plaster within this study and the aluminium head – pan was procured from a building materials shop in Samaru, Zaria.

It was observed during the heating or calcination process of gypsum that when charge is brought down from the kiln for stirring and decanting into another container, the charge was very light when being stirred. The researcher closely observed this behaviour of plaster in the first calcination and therefore monitored subsequent process of gypsum. The behaviour of plaster boiling and settling was commonly observed throughout the period of calcination. It was noted that during the period of calcination, the feed of land plaster being heated in the kiln could boil and settle. In other words, heated land plaster within the process of calcination

tend to become fluid – like in nature, especially if stirred or emptied into another container as was the case during this experiment.

As the third hour gradually concludes, a small scoop of the calcined plaster is taken for a simple test. This involved spreading calcined plaster to cool and mixing it with water. This way, observation is made on how plaster sets. If plaster bat set then it was discharged and another feed or batch was weighed in preparation for a fresh or new calcinations. It was necessary for this test to be carried out at this level because confirmation of maturity of plaster must be ascertained at this stage before the next level of investigation.

To forestall any mix-up of the various gypsum (and subsequent plaster) calcination was done individually. When calcinations or heating of plaster from a location was completed, then a new location was initiated. In other words it was only when Wurno calcinations was completed before Dange sample was calcined, and as earlier said all bags and containers of samples were boldly labelled.

There were two key observations, change in plaster colour from a darker shade to lighter shade. As earlier mentioned, the researcher weighed plaster in batches of 6kg for calcinations and observed that plaster weight 5kg at the end of three hours of calcination. Being that there were two kilns, it made calcinations faster until a condition of power outage was experienced in I.D.C. Calcination process was started 24th of February 2014 but concluded June 18th 2014. Gypsum from Wurno, Dange and Weppa areas were heated or calcined. The results of this experiment were subjected to scientific test as this will be further discussed within the study.



Plate V: Kiln for calcinations at Industrial Development Center, Samaru Zaria.

3.7 Sieving of Processed Plaster

In preparation for the next stages of experiments and tests, it was necessary that produced plaster of Paris be sieved to the standard powder mesh size. Calcined or processed plaster was coarse in particle size when compared with the foreign sample of plaster available. It was then necessary to sieve the newly made plaster, in this situation, 42 micro mesh from the Ceramics Section of Industrial Design A.B.U Zaria was obtained for this purpose. Akpakip (1993) used same mesh size for similar purpose before. The venue for this exercise was the plaster studio of same section. As earlier mentioned, to avoid the mistake or error of mixed-up samples, this exercise also was done by starting with one location before another. Samples were now in plastic containers with locations boldly labelled on them.



Plate VI: Sieving of Processed plaster



Plate VII: Sieving of processed plaster

3.8 General Observations on Calcination Process

During the processes involved at the different stages of calcination necessary for the experiment in this study, there were some observations which the researcher considers useful. These observations are as shown below:

1. Plaster was produced using three different containers namely, aluminium head – pan, bisque wares and crucibles. It was observed that even though plaster was subjected to same temperature limits, time limit, and temperature manipulation and general kiln treatment, there was a noticeable physical change in colour of plaster. Plaster became lighter in colour after calcination.
2. Plaster produced from aluminium head – pan was lighter in weight when compared to that made from the bisque ware and crucible. They also set slightly faster, while the plaster from the head – pan sets in about eight (8) minutes, plaster from crucible sets in about twelve (12) – 15 minutes.
3. It is important that the flue or chimney of the kiln or oven be partially closed. This singular action increases the temperature within the oven environs to help keep it moist. Another step that encourages production of hard plaster is ensuring that temperature increase is gradual for example, beginning with 120^0Cn in the first hour, increasing to about 150^0C to 170^0C toward the concluding hours.
4. Calcination of plaster of Paris with aluminium head – pan is faster but lighter in weight than the one processed from crucible, ipso facto weaker in strength. Plaster of Paris produced from crucible is denser and stronger. Therefore stronger plaster could be produced through the use of crucible
5. The use of aluminium head – pan produces weaker plaster, this grade of plaster could be useful for production of classroom board chalk.

6. A good temperature manipulation and vapour control within the kiln chamber and right choice of container or pan eg crucible can determine the production of good and strong or hard plaster grade.
7. It is important to understand that sudden and sharp increase temperature does not augur well with plaster production it is pertinent that during plaster production, increase in temperature should be gradual, it is because this technique in plaster production contributes to the production of stronger plaster (alpha plaster)
8. It was observed during the plaster processing period there was a change in weight of plaster. Plaster weighed and charged 6kg into the kiln for calcinations and at the end of calcinations process when plaster was re-weighed, the weight dropped to read 5.1kg.
9. Land plaster for calcinations must be monitored closely and diligently, as careless oversight could ruin production, plaster should therefore be closely monitored through these procedures; emptying plaster from one container into another thereby controlling local overheating. Stirring plaster, temperature control and conscious time watch.
10. The process of plaster production does not involve high temperature but an oven or kiln capable to fire up to 200°C or slightly above is good enough for plaster production.

3.9 Production of Metal Moulds for Test Tiles

A metal mould was produced for test cylinders for the experiment conducted to test for compressive strength.

After the production of plaster of Paris, it was sieved to desired smoothness. It was important at this stage to progress to the next level of preparation for the various tests designated for

this study. In pursuance of this objective, a conducive environment was desired by the researcher. A studio or laboratory with the availability of a digital weighing balance and running water was necessary and the desired environment was present in Chemical Engineering Department, Faculty of Engineering, A. B. U. Zaria. The weighing out of plaster powder for the production of test tiles was done in Chemical Engineering Department. Working with plaster of Paris seems to be different from working with clay which is partially due to time frame before setting and shaping becomes a challenge. Another reason is that plaster has the tendency of adhering to containers wherein it is mixed in or poured into. It is therefore imperative that the person working with plaster be ready to mix plaster and pour on already prepared (sized) surface and also be in readiness to wash used container almost immediately. It is important that the model (if any, and in this case moulds) is thoroughly covered with sizing agent, which is simply a thick solution of soft laundry soap and boiling water, to create a shine or separator. The process of using a substance as a separating agent is what is technically known or referred to as "sizing" while the substance is known as "sizing agent". When sizing agent is applied on to surfaces, it makes plaster separates easily from model and surfaces. It is also advisable to mix plaster in plastic or rubber containers than metal ones, washing and cleaning is easier done when container used are either plastic or rubber containers.

There were two types of moulds prepared for this experiment, the rectangular multiple wooden moulds and a cylindrical metal mould made up of a female (Hollow) and male (solid) moulds. Mixed fluid plaster was poured into the hollow part of the mould and allowed time for setting before the solid or positive part of the mould was used to gently push out the cast plaster from the hollow part. The various ratios of plaster and water were weighed out, mixed, poured into the hollow cylindrical part of the mould while time was given for the poured plaster to set firmly and gently extruded from the negative part with the aid of the

positive part. This was done for ratio 65 parts water and 100 parts plaster. Plaster measurement was constant at 100part while water was varied. Therefore the highest measurement of water was 100 grams part to the lowest 65 part grams volume of water; plaster was constant at 100 grams throughout this experiment while water was varied. This is how test tiles and cylinders were determined for Absorbency Test, Shrinkage Test and Compressive Strength Tests were produced.



Plate VIII: Production of metal mould



Plate IX: The produced metal mould

3.10 Production of Metal and Wooden Moulds for Test Tiles

With due consideration to the peculiarity of tests expected to be carried out in this study, it was obvious that the mould must be well thought out. In the light of this, a metal mould was forged with the assistance of the Foundry Section of Department of Metallurgy, for the compressive strength test. A wooden type was also constructed for use in producing test tiles for both the shrinkage and absorbency tests. These tiles were rectangular and different from the compressive strength test tiles which were cylindrical in shape. The metal mould was made in hollow negative and positive solid form. The test tiles and cylinders were made to specifications measuring 20mm in diameter and 22mm in height with a base measuring 2mm.

3.11 The Practical Use of Processed Plaster of Paris for Ceramic Productions

In this chapter, the report on practical use of the processed plaster for mould production and usage were presented. This included plaster moulds for use in slip casting and press moulding (or press mould extension).

For the purpose of discussion, this chapter is divided into five segments as presented below:

- Selecting plaster/water ratio
- Production of models
- Simple moulds: stamp and springs
- Production of various moulds and drying process as well as slip production
- Slip casting and press mould

3.11.1 Determining Plaster/Water Ratio

It was imperative to determine from all the tests carried out, which ratio mix could perform best for the practical exercise of slip casting and pressing extensions. This was bearing in mind that for pressing extension, plaster mould has to be stronger in consistency yet porous

enough for pressing processes. In the case of slip casting, the ratio mix consistency should be more porous yet command the strength and density for the rigours of slip casting. For the processed plaster, it was decided that 100:65 and 100:70 or 70 plaster/water ratio mix would be best for press moulding due to its strength during compressive strength and porosity tendency at that ratio mix. The 65 and 70 ratio mix were considered fit for this exercise.

In the occasion of slip casting, it could present a challenge because although porosity of moulds is important in this process, durability of moulds is equally of strong consideration. Therefore, the plaster/water mix of 100:75 and 100:80 mix were considered proper for the slip casting processes.

For a good comparative study, the imported plaster of Paris used in the Ceramic Section of Department of Industrial Design, Faculty of Environmental Design, Ahmadu Bello University, Zaria was considered and obtained for this exercise. The normal consistency of ratio mix (plaster to water) used in the ceramic section of Department mentioned above that is 100:130 plaster water ratio mix was adopted for the mould which is the commercial (imported) plaster. The aim of this exerciser was to examine and determine the production capacity and efficiency of the two types (local and imported) plaster of Paris. Thereby creating conditions for good comparison between these two plaster of Paris, that is, the locally processed sample and the imported commercial variety.

3.11.2 Preparation and Production of Models

For every successful mass production in ceramics the production begin with a idea and a sketch. Sketches were produced for single mould, two piece mould and three piece mould. The sketches were produced on top of in actual clay work before being prepared for the

process of mould making. As earlier mentioned, moulds were produced as one piece mould, two pieced mould and three-piece mould.

3.11.3 Sprig/stamp simple mould

Sprig and stamps are simple one piece moulds used in ceramic surface decoration. A stylized flower pattern was carved on cast plaster surface. When this was properly finished, the mould of that carved surface was cast or made. This could be used for the purpose of ceramic surface decoration for both thrown and hand-built pieces or cast pieces should there be need.



Plate X: Sprig moulds and Sprig decorative pieces

3.11.4 One Piece Mould

The one piece mould was produced from the commercial plaster. The model was on a flat surface bordered by a wooden frame that measured 30 centimeters length by 60 centimeters breadth. The thickness of the clay slab used was about two inches. This design was made of a centralized canoe image borderd at the four corners by a traditional motif from the researchers home state identified by the name "Nsehe" meaning blossom. At the completion of the single piece mould model, the mould was produced for the one-piece mould,

It is important to state here that due to various tests carried out during the study, the quantity of remaining plaster was not sufficient for big mould such as the model for the one-piece mould. It was therefore important that the situation had to be seriously considered such that the remaining quantity of processed plaster could be judiciously used for specific design that

the plaster could suffice. It was then decided that processed plaster should be used for smaller moulds while the commercial plaster be used for bigger jobs.



Plate XI: One piece mould produced from the study

3.11.5 Two Piece Moulds

The two piece moulds was derived from the traditional Hausa trumpet used to announce the arrival of Emir or other important dignitaries. The instrument shaped like the popular "kakaki" yet smaller in size is known as "pare." This was stylized into art work and made to fit a model for two-piece mould. It was particularly produced on the throwing wheel but finished as hand-built piece. The mould of the piece was then taken in two-piece mould.



Plate XII: Two Piece Mould produced from the study

3.11.6 Three-piece mould

A flower vase designed on paper and thereafter was produced on the throwing wheel. The "excess spare or reservoir mouth" which is used in replenishing casting slip during the casting process was created. This was eventually trimmed off the finished work. The mould for the three-piece mould was then made.

The making of the models were done in two days and being rainy period in August, the modes were given another day to become leather hard before moulds were made. The moulds were given time to dry. This took two week and four days, a total of eighteen days in all. It was noted that the heavier and bigger the mouds took longer time to dry out. This mould was made with processed plaster of Paris.



Plate XIII: Produced three piece mould

3.11.7 Casting Slip

The casting slip was prepared in the Department of Industrial Design (Ceramic section). It was made up of levigated and sieved kaolin and ball clay from the ceramic section. The

mixture of 50% kaolin and 50% ball clay as contained in Gukas, (1985) was used for the casting slip in this study.



Plate XIV: Casting process

3.11.8 Casting with commercial plaster moulds

Attention was not on this plaster mould because of its reliability and competency performance to be used as a standard plaster for the ceramic section. The performance was good, as with this plaster, twelve? works were produced from each mould. According to Speight and Tobi (2006); Singer and Singer (1978) products from plaster moulds range from 90-100 pieces of good casts.

3.12 Preparation/Production of Test Cylinders and Tiles

It is pertinent to note at this stage of the work, the research chose to use a conducive venue due to the sensitive weighing balance which was required to weigh out plaster and water ratio mixes. The Department of Chemical Engineering, (PTDF) Laboratory was duly selected for this exercise because of access to such a weighing balance. The various ratio mixes from

literature ranges from 65 part water and 100 part plaster to 100 part water. It is important to note that in all the ratio mixes preparation for this study, plaster was stable at 100 part, while water was varies within the sizes of 65, 70, 75, 80, and so on. This can be observed in the table as shown below.

Table 3.1: Percentage Composition of Plaster/Water Ratio

Plaster	100	100	100	100	100	100	100	100
Water	65	70	75	80	85	90	95	100

An example of the various ratio mixes selected for this study

The various tests designated for this study are as listed below:

1. Absorbency or Porosity Test
2. Shrinkage Test
3. Compressive strength Test
4. Density Tests
5. X-ray Fluorescence (XRF)
6. X-ray Defraction (XRD)

A wooden template or mould was constructed for the first two tests; Shrinkage Tests and Absorbency Tests. The mould for the Compressive Strength tests tiles was forged in metal. It was a metal mould made of positive or solid and hollow (negative) pieces. Both pieces are lightly sized with sizing agent of laundry soap and water. After which the cast was poured into the hollow cylinder which measured 20mm by 20.2m. When the cast was firmly set, the male or positive part of the mould was used to firmly but gently push the cast out from the negative mould. In this way, all the ratio mixes were extruded from the negative metal mould, the process was carried out for all the three locations with the above stated different ratio mixes of plaster/water in table 3.1.



Plate XV: Test tiles and cylinders from Wurno



Plate XVI: Test tiles and cylinders from Dange



Plate XVII: Test tiles and cylinders from Weppa

3.13 Absorbency Test

Absorbency test is that which is done on materials to determine porosity level of the material. The wooden mould was used in producing test tiles in the various ratio mixes. Test tiles produced were left to dry out for a period of 17 days, that is, two weeks and three days which ensured complete drying out of the physically combined water. The period of drying the plaster test tiles was particularly long due to the fact that it was in the rainy season and the relative atmosphere humid was high. They were further loaded into the oven and dried at 40^0c for 30 minutes to ensure complete drying out of water from the tiles. Plaster tiles of various ratio mixes were then weighed and their weight recorded as shown in the Table 4.1 to 4.3 below. The essence of this experiment was to determine which ratio mix was the most porous of all the mixes. The experiment includes weighing and recording both the dry and saturated weight of the material.

3.13.1 Experiment:

The absorbency test was conducted on the plaster test tiles

3.13.2 Instrument:

The following instruments were used for the test:

1. Plastic bucket
2. Weighing balance
3. Thin roll of sewing thread
4. Supply of cold water
5. Cotton hand towel

Test tiles of different ratio mix from Wurno were weighed on the scale for dry weight before being immersed completely in water for one hours which was enough time for the test tile completely get saturated in water. Excess water was removed from the test tiles using damp towel and the weight of the saturated (various ratio) tiles was taken again.

3.14 Shrinkage Test and Results

In this experiment, plaster and water mixes of various categories were mixed and poured into the wooden moulds. A line of 5cm was drawn on each tile sample while plaster was still wet and allowed to dry completely. The drawn measurement of 5cm was then re-measured and the observation is as shown on the Tables 4.4 to 4.6 in chapter four.



Plate XVIII: Shrinkage test pieces

3.14.1 Experiment

Instruments

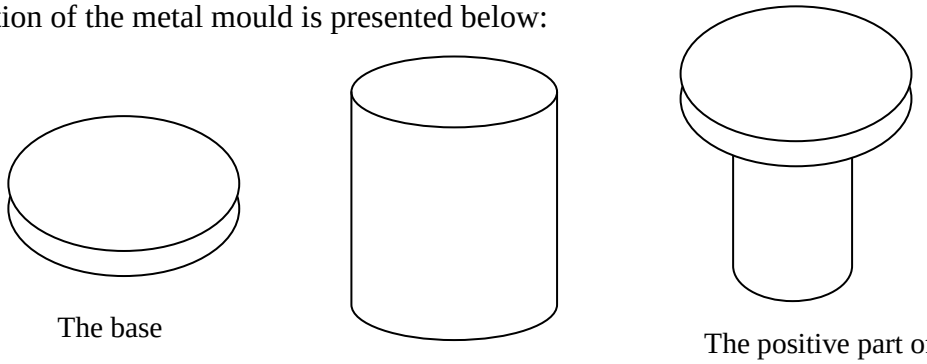
1. Ruler
2. Test tiles
3. HB pencil

On all tiles produced from the wooden mould, a line was drawn on all the different ratio mixes namely 65:100, 70:100, 75:100, 80:100, 85:100, 90:100, 95:100 and 100:100. The straight line measuring 5cm as contained in Akpakip (1993). The tiles were then air dried outside but the weather was not favourable for drying the test tile because it was raining season. However, a period of 17 days was allowed for the tiles to dry properly then weighed and the weight recorded. To ensure that test tiles were thoroughly dried, test tiles were place in the drying oven for further drying in Department of Chemical Engineering, Faculty of Engineering, Ahmadu Bello University, Zaria. This was done for 30 minutes and no change in weight was recorded. The drawn lines on test tiles were now ready for re-measurement.

3.15 Compressive Strength Tests

Compressive strength test is used in determining the strength of materials intended for compression loads. It is used for brittle materials that are not able to withstand tensile deformations. In other words, compressive strength can be determined only for brittle materials that break up when compressed. Test samples for compressive strength test are cylindrical and in the case of this study, a metal mould was devised measuring 22mm by 20mm in diameter. The mould is a set of three piece mould made up of a circular flat base, a hollow or negative cylindrical piece and a positive or solid part for extending the cast test pieces.

The illustration of the metal mould is presented below:



The base

The hollow (negative) part of the mould

The positive part of the mould

The test was carried out in Department of Metallurgical Engineering, Faculty of Engineering, Ahmadu Bello University, Zaria. The equipment used for this experiment is called Hounsefield Tensometre. The Compressive strength test was done with the Hounsefield Tensometre. It was placed on a wooden table and it was manually operated on the machine (Hounsefield Tensometre) where the beam for reading mineral hardness is fixed, lies a thin transparent glass tube containing mercury. When the test sample is placed in the machine and load is applied, the mercury moves within the thin glass tube. The movement of the mercury and the beam rule are together, used in reading mineral compressive strength. The reading is calculated in kilonewton. A graph sheet attached to the machine also recorded a more accurate reading. The compressive strength results was one of the deciding factors that aided in determining which of the eight (8) ratio mixes was the one suitable for use in ceramic mould making for this study.

3.15.1 The Hounsefield Tensometre

The compressive strength test of the produced sample was carried out such that the strengths of various consistencies or mixes of processed plaster could be established. The instruments involved in the process were the hounsefield tensometer and the test cylinders earlier discussed. The machine was operated manually and measures 96cm long, 19cm high and 23.5cm wide. The machine was operated manually with two winding device. The smaller and

slower one is to position the sample such that the machine has proper grip of the sample piece. The second winding device is the one that compresses the test piece according to the load in the machine. It is placed on a table (above waist level).

Beside the beam rule lay a thin glass tube containing mercury. The mercury and the beam rule could be used together in reading mineral compressive strength from the graph sheet wrapped on the beam. A device attached over the beam rule and the glassed mercury leaves punched or slight perforated trail marks on the graph sheet which corresponds with the mercury reading on the beam rule. These marks from the graph sheets are read in kilonewton (kN) and the analyses are as presented in the graphs and tables in this segment.



Plate XIX: Hounsfield Tensometer with compressive strength test pieces

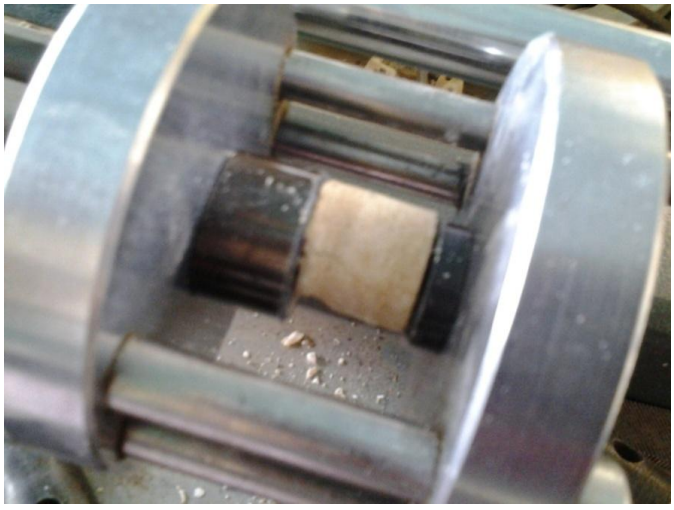


Plate XX: A test piece in Hounsfield Tensometer



Plate XXI: Test pieces after compressive strength test

The practicality of compressive strength test (as earlier mentioned) was carried out in the Department of Mechanical Engineering, Faculty of Engineering, Ahmadu Bello University, Zaria. The test on compression was carried on the samples from the three locations of Wurno, Dange and Weppa. It is also important to state that the various plaster ratio mixes with varied conditions with the compressive strength test was tested. The compressive test was carried out by compressing the test piece to a certain level or height or to such level when such materials were compressed until it ruptured or cracked. The result of the force or load application and deflection are as shown in Tables 4.7 to 4.9 in chapter four.

3.16 Density Test

The weight per unit of volume of a substance is the density of that substance. Plaster is produced in two forms. The Alpha and the beta plaster of Paris. Alpha plaster is said to have a density of 2.76grams per cubic centimetre while the beta has 2.63gram/cm^3. The density of a material is the mass of such divided by the volume, otherwise expressed as indicated below:

Density = $\frac{\text{Mass}}{}$

Volume

In most cases a weighing balance is the instrument used in getting the mass of an object, in this experiment, a digital scale was used to measure the weight of the ratio mix samples from Wurno, Dange and Weppa which were, 100:65, 100:75, 100:80, 100:85, 100:90, 100:95, 100:100. These make up a total of twenty-four sample.

3.16.1 Bulk Density

Seeing that the processed plaster of Paris will be useful both in the dry moulding and when mould would be saturated with wetness from casting, it was necessary that both dry and bulk density of plaster mould be known. The bulk density of a given material is the ratio of the mass of that material (in this case gypsum test piece) to its bulk volume, in other words, the volume of material plus all the pores in that material. This is done by soaking the samples in liquid (water) for a consistent period of time. In this experiment, it was done for three days, This was to achieve complete saturation of all ratio mixes 100:65, 100:70, 100:75, 100:80, 100:85, 100:90, 100:95, 100:100. As with the cases of previous experiments with the study, these ratio mixes were measured and produced from processed gypsum plaster or plaster of Paris for this experiment.

The experiment was done in the soil Mechanics Department of Civil Engineering, Faculty of Engineering, Ahmadu Bello University, Zaria with the kind assistance of Mr. J. Ochepo, a lecturer of that department. Mr. J. Ochepo assisted in both the dry and bulk density tests recorded in this study.

W_1 equals weight of dry test piece

W_2 equals weight of test piece soaked (saturated) in water

W_3 equals weight of test pieces soaked and suspended in air

D equals density of immersion of test.

CHAPTER FOUR

RESULTS AND DISCUSSIONS

4.0 Introduction

This chapter deals with all results of experiments carried out within the study, these results of concluded tests will be presented and discussed in this segment. For a progressive and successful discussion, this section is divided into sub-sections as presented below;

1. Porosity test or Absorbency Test
2. Shrinkage Test
3. Compressive (Stress) Strength Test
4. Density
5. X-Ray Fluorescence Spectrophotometer (XRF)
6. X-Ray Diffraction (XRD)

4.1 Absorbency Test Observation

Table 4.1: Ratio of Plaster/Water Mix of Wurno Sample

	1	2	3	4	5	6	7	8
Plaster	100g	100g	100g	100g	100g	100g	100g	100g
Water	65	70	75	80	90	85	100	95
Dry Weight	100.46	89.93	77.12	97.06	99.01	99.53	100.15	93.12
Saturated weight	127.97	120.44	106.12	131.77	143.37	145.45	149.36	141.26
Observed difference in weight	27.51	30.51	29	34.71	44.36	45.92	49.21	48.14
% porosity	27.38%	33.93%	37.60%	35.76%	44.80%	46.14%	49.14%	51.69%

Table 4.2: Ratio of Plaster/Water Mix of Dange Sample

	1	2	3	4	5	6	7	8
Plaster	100g	100g	100g	100g	100g	100g	100g	100g
Water	65	70	75	80	85	90	95	100
Dry Weight	95.50	97.85	92.21	97.89	100.80	99.54	100.49	98.88
Saturated weight	122.56	130.34	132.55	137.69	146.50	147.45	153.08	154.70
Observed difference in weight	27.06	32.49	40.34	43.8	45.7	47.91	52.59	55.82
% porosity	28.34%	33.20%	43.75%	44.74%	45.47%	48.13%	52.33%	56.45%

Table 4.3: Ratio of Plaster / Water Mix of Weppa Sample

	1	2	3	4	5	6	7	8
Plaster	100g	100g	100g	100g	100g	100g	100g	100g
Water	65	70	75	80	85	90	95	100
Dry Weight	93.05	96.34	98.95	93.58	99.72	100.79	100.20	99.88
Saturated weight	119.50	127.30	133.60	129.33	142.27	147.07	150.12	158.78
Observed difference in weight	26.45	30.96	34.65	35.75	42.55	46.33	49.92	58.9
% porosity	28.43%	32.14%	35.02%	38.20%	42.67%	45.94%	49.82%	58.97%

4.1.1 Discussion

Absorbency test on Wurno, Dange and Weppa Plaster/Water Ratio Mixes

All samples from the three locations were subjected to similar treatment where the samples were soaked in a container for 3hrs.

Wurno – it was observed that the percentage porosity or absorbency increased consistently from 65:100 ratio mix to 100:100 mix. Indicating that porosity or tendency toward

absorbency increases with the increase of water in the ratio mix. Porosity level ranged from 27.38% to 51.69%.

Dange – Performance in terms of absorbency in the Dange sample was better than that of Wurno. Percentage absorbency or porosity started with the 65:100 ratio mix with 28.34% to 100:100 mix with 56.45%.

Weppa – the observation made on Weppa sample was as below. Absorbency level of percentage Weppa samples was observed also within the ranges of 65:100, 28.43% and 100:100 ratio mix to be 58.97%.

The highest percentage absorbency in Wurno plaster ratio mix was noted in the 100:100 ratio mix. This mix when completely dry was higher in weight when compared with the other ratio mixes. It was also observed of this mix, that is 100:100, that is could easily break off when pressed with the finger, especially at wet or damp stage. Even though this sample is highly porous, it lacks in strength to withstand the rigors of slip/press casting procedures. The least porous sample in the ratio mixes is the 100:65 mix of various plaster/water. This mix appears strongest among the eight different ratio mixes. Due to its low percentage absorbency capacity, it may get easily saturated with slip/press casting process and therefore may be a poor choice for this type of casting. The 100/65 mix should be recommended for mother moulds, model making or master mould or any work done directly with plaster.

From the findings of the study, the mixes which fall within 70 and 85 could be said to be both strong and porous enough for ceramic application of slip and press casting production techniques. For the individual who wants a mix for waste or chipped moulds, 85, 90, 95 and 100 mixes could be adviced.

It is important to observe that produced Wurno and Dange plasters had darker colouration, when placed beside Weppa sample. This is mainly due to the high level of impurities present in the gypsum sample from site, even after washing.

The general submission on the absorbency tests carried out is that the plaster of Paris processed from Wurno, Dange and Weppa locations or areas could be used for various ceramic applications according to the test carried out within this study. The samples from the three locations were soaked in water for a period of 1 hour.

4.1.2 Comparative Analysis of Wurno, Dange and Weppa Absorbency Test

The absorbency tests carried out in the Department of Industrial Design is presented as shown below.

Weppa plaster rates with percentage absorbency of 58.95%, Dange had 56.45% while Wurno had 51.69% in the ratio of 100:100 ratio of plaster mix. The plaster mix of 100:65 in the three locations as presented in the tables of absorbency percentage tests are Wurno 27.38%, Dange 28.34% and Weppa 28.45%.

In line with the ratio mixes used in this study, for example 100:65, 100:70, 100:75, 100:80, 100:85, 100:90, 100:95, 100:100. The percentage absorbency reading for each mix from Wurno were as follows 27.38%, 33.93%, 37.60%, 35.76%, 44.80%, 46.14%, 49.14% and 51.69% respectively. The Dange absorbency percentage according to the above mix is as follows, 28.34%, 52.33%, and 56.45% while Weppa's percentage absorbency read thus, 28.43%, 32.14%, 35.02%, 38.20%, 42.67%, 45.94%, 49.82% and 58.97%.

In average percentage consideration of absorbency reading above, Wurno has 49.81%, Dange has 44.05% while Weppa has the total percentage of 41.398%. the average absorbency percentage is the one of Dange, which reads 44.05%. Wurno has the individual lowest

absorbency percentage of 27.38% while Weppa has the highest percentage of 58.97%. this is well represented in the graphs indicating the percentage flow of absorbency.

4.2 Shrinkage Test and Results

Table 4.4 Shrinkage test for Wurno Plaster

Water	65	70	75	80	85	90	95	100
Plaster	100	100	100	100	100	100	100	100
Shrinkage observation	0%	0%	0%	0%	0%	0%	0%	0%

Table 4.5: Dange Plaster/Water Ratio Mix

Water	65	70	75	80	85	90	95	100
Plaster	100	100	100	100	100	100	100	100
Shrinkage observation	0%	0%	0%	0%	0%	0%	0%	0%

Table 4.6: Weppa Plaster/Water Ratio Mix

Water	65	70	75	80	85	90	95	100
Plaster	100	100	100	100	100	100	100	100
Shrinkage observation	0%	0%	0%	0%	0%	0%	0%	0%

4.2.1 Discussion

Shrinkage test on Wurno, Weppa and Dange Plaster/Ratio Mix

Shrinkage test was necessary to ascertain the level of shrinkage of material at any state from prepared or wet state. Although literature states that shrinkage in plaster of Paris is usually no factor to be considered as it is usually very minute. That is to say, that a good ceramic plaster should not exhibit a marginal shrinkage level and if there is any shrinkage it should be minimal or insignificant. Friths (1985) and Singer and Singer (1971) mentioned shrinkage

common with bisquited moulds that shrinkage in plaster if mentioned at all should be of no factor to be considered a threat to mould production.

It was imperative to establish whether the produced plaster had any outstanding or obvious shrinkage difference. It was the reason that informed the preparation and production of test tiles for shrinkage test in this study. On this backdrop or understanding, the shrinkage test was done

It was observed that in the three locations throughout the various ratio mixes there was minimal shrinkage in Wurno and Dange plaster.

4.3 Compressive Strength Tests

4.3.1 Load/Force Reading of Compressive Strength Tests

Table 4.7 Wurno

Ratio	100	100	100	100	100	100	100	100
Mixes	65	70	75	80	85	90	95	100
Force 1st reading	625	680	450	450	325	375	475	350
Force 2nd reading	650	550	525	400	375	275	225	125
Deflection 1st reading	1	1.1	1	.8	1	1	.6	.6
Deflection 2nd reading	1.8	1.6	1.4	2.0	1.4	1.6	1.1	2.4
Average force	637.5	615	487.5	425	350	325	350	237.5
Average deflection	1.4	1.35	1.2	1.4	1.2	1.3	0.85	1.5

Source: Researchers practical test

Table 4.8 Dange

Ratio	100	100	100	100	100	100	100	100
Mixes	65	70	75	80	85	90	95	100
Force 1st reading	750	700	550	550	450	500	425	325
Force 2nd reading	835	700	650	550	500	425	350	175
Deflection	1	1.6	1.4	1.9	1.9	.7	.6	.6
Deflection 2nd reading	1.9	1.4	1.6	1.6	1.4	1.2	1.2	1.4
Average force	792.5	700	600	550	475	462.5	387.5	250
Average deflection	1.45	1.5	1.5	1.75	1.65	0.95	0.9	1

Source: Researchers practical test

Table 4.9 Weppa

Ratio	100	100	100	100	100	100	100	100
Mixes	65	70	75	80	85	90	95	100
Force	1300	1350	985	975	700	500	625	375
Force 2nd reading	950	725	450	425	375	325	275	150
Deflection	1.9	1.8	1.8	1.2	1.2	.8	1	.6
Deflection 2nd reading	2.4	1.4	1.4	1.4	1.4	1.0	1.3	1.6
Average force	1125	1037.5	717.5	700	537.5	412.5	450	262.5
Average deflection	2.15	1.6	1.6	1.3	1.3	0.9	1.15	1.1

Source: Researchers practical test

4.3.2 Comparative Analysis of Wurno, Dange and Weppa Compressive Strength

The compressive strength test is done for earthen materials that lack the pulling strength done for metallic materials and such samples that could withstand the stress of pulling (tensile test). Plaster of Paris falls into the earthen minerals of above description. The compressive strength test was carried out as earlier mentioned in the strength of material laboratory of Mechanical Engineering Department, Ahmadu Bello University, Zaria.

Wurno

In the graph representing plaster of Paris the Paris, the highest compressive strength is below 350 MegaNewton (MN) per M^2 of the 65 ratio mix closely followed by 70 water ratio mix. The next and third ratio mix is 75, 80, 85, 90. The 95 mix has a higher compressive strength than the 90 ratio mix, while 100:100 ratio mix is the one with the lowest compressive strength in the location.

Dange

Dange location's presentation was noted to have the 100 ratio mix in the lowest compressive strength category followed by 80 mix. The highest compressive strength in Dange ratio mix is the 65 ratio mix. A smooth descend can be followed from there through 70, 75 and low record of stress or compressive strength is noted at 80 mix. It rises at 85 mix and again a smooth decrease is again observed through 90, 95 and 100 ratio mix.

Weppa

The presentation of Weppa graph of compressive strength was observed to record then highest compressive strength (stress) at the 65 ratio mix. Unlike the Dange compressive strength graph presentation, the Weppa has an almost smooth decrease of compressive strength (stress) from 65 ratio mix through 70, 75, 80, 85, 90, 95 and 100 mix being the lowest compressive strength and the weakest in this mix.

4.3.3 Compressive (stress) Strength Observation

Compressive strength test was carried out as earlier mentioned at the Department of Mechanical Engineering at the strength of material Laboratory in Ahmadu Bello University, Zaria. For each location, eight samples of various ratio mixes were prepared. That is, Wurno had eight different samples prepared 100:65, 100:70, 100:75, 100:80, 100:85, 100:90, 100:95 and 100:100. These eight ratio mixes were the standard ratio mix for all three locations. Test sample were in cylindrical form instead of the rectangular test tiles prepared for other tests, for example, shrinkage absorbency tests. Compressive test pieces were produced according to recommendation from the strength of materials laboratory.

The said cylindrical test samples were weighed out and prepared from the Department of Chemical Engineering, the mould used in preparation of these test cylinders was produced from the foundary workshop of Department of Metallurgy. This was a metal cylindrical mould as earlier discussed measuring 22mm in weight and diameter of 20mm. To avoid loss of liquid plaster when poured, the mould had a base of 20mm in diameter and an inner protection of 2mm. When the base is in place, poured plaster into the hollow cylindrical mould measured about 20mm. Produced plaster from various mixes 100:65, 100:70, 100:75 and so on were made ready from plaster produced from the three locations Wurno, Dange and Weppa respectively for the compressive strength tests.

4.3.4 Discussion of Compressive Strength Test on Wurno, Dange and Weppa Gypsum Plaster

The compressive strength test is done for earthen materials that lack the pulling capacity and strength needed for metallic materials or such samples that could withstand the stress of pulling (tensile test). Plaster of Paris falls into the category of such earthen mineral of above

description. Even though the preparation of the test cylinders samples were weighed with the digital scale in the Department of Chemical Engineering, faculty of Engineering. The compressive strength tests were carried out in the strength of material laboratory of the Department of Mechanical Engineering Department. The result of the compressive strength test recorded on the table earlier presented on table 4.7 to 4.9.

The compressive strength test was done for each of the eight samples from the three locations, totally twenty samples from the three locations, totalling twenty four samples in all. To be more certain of the test (compressive strength) the test was carried out twice and the average of the tests was determined and used in compressive strength calculations. The readings on the graph sheet from the compressive strength were done by taking the reading of load which is equal to force applied. In other words, the compressive strength or stress of a material is load which equal force applied. In this experiment, for applied was 2.5 kilonewton or 2,500 newton. It is important to note that one(1) kilonewton equals one thousand (1,000) Newton and vice versa. As earlier mentioned in the discussion above, the test samples produced for this test were in small cylindrical (dice) form, therefore, the area of cylinder designated by (A). The cylinders for this test or experiment where in solid form and not hollow. The formula for the Area of a solid cylinder is given as $2\pi r^2 + 2\pi r^2$.

The length of a closed or solid cylinder or dice (L) is the same as the height (h), the measurement of the length (height) of the cylinder was taken. That is to say, each sample of the eight ratio mixes from the three different locations, which totalled twenty-four in all were measure and the height recorded. The Diameter (D) of each sample was also taken and recorded and shown in Table 4.10 to 4.12. The measurement of height (height) and diameter (D) of the twenty-four test cylinders were taken. The compressive test was done twice and the average was taken. This was done in readiness for the next stage.

Table Showing Measurement of Test Samples for Compressive Strength

Table 4.10 Wurno

	Ratio mix specimen	**Length (mm)**	**Diameter (mm)**
1	100:65	19.7	20.5
2	100:70	19.7	20.5
3	100:75	19.7	20.5
4	100:80	19.3	20.4
5	100:85	19.2	20.4
6	100:90	19.4	20.3
7	100:95	19.6	20.1
8	100:100	19.5	19.7

Table 4.11 Dange

	Ratio mix specimen	**Length (mm)**	**Diameter (mm)**
1	100:65	19.3	20.6
2	100:70	19.5	20.4
3	100:75	19.9	20.5
4	100:80	19.6	20.2
5	100:85	19.5	20.4
6	100:90	19.5	20.4
7	100:95	19.9	20.4
8	100:100	19.6	20.2

Table 4.12 Weppa

	Ratio mix specimen	Length (mm)	Diameter (mm)
1	100:65	19.6	20.5
2	100:70	19.6	20.3
3	100:75	19.5	20.5
4	100:80	19.3	20.6
5	100:85	19.6	20.3
6	100:90	19.8	20.4
7	100:95	19.4	20.2
8	100:100	19.6	20.0

Fig. 5: A graph of stress Vs Ratio mixes for Wurno sample

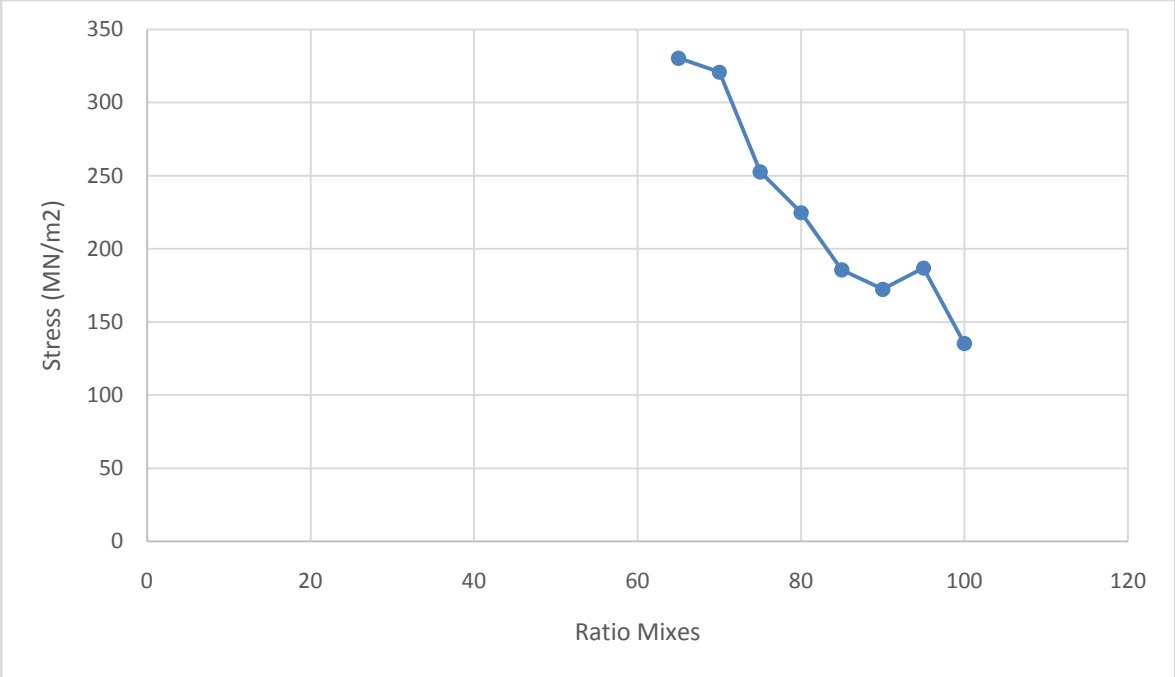


Fig. 6: A graph of stress Vs Ratio mixes for Dange sample

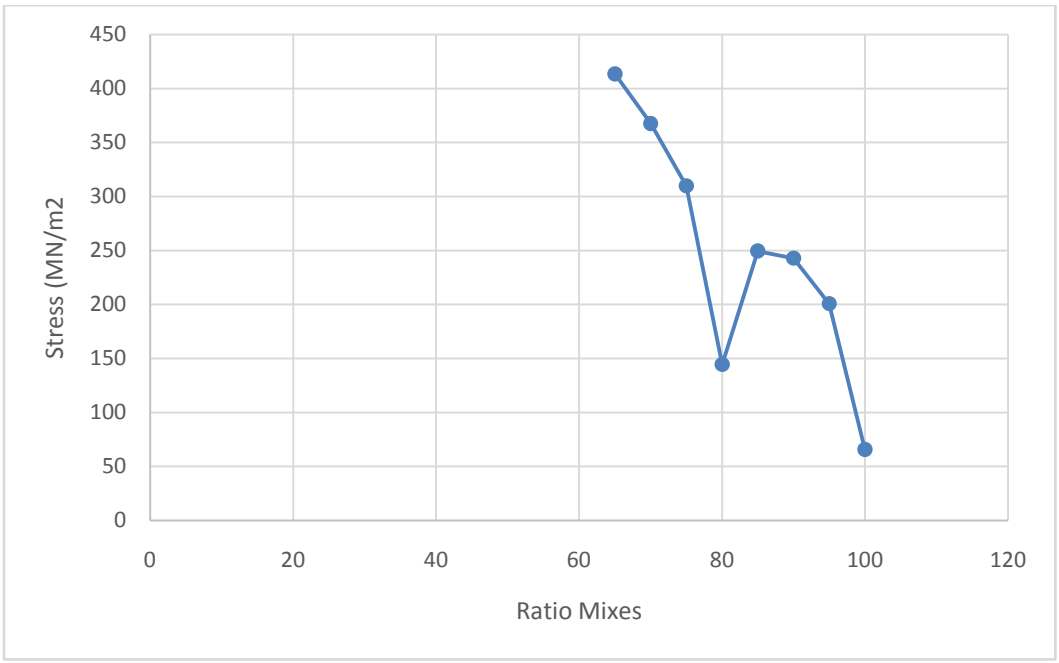


Fig. 7: A graph of stress Vs Ratio mixes for Weppa sample

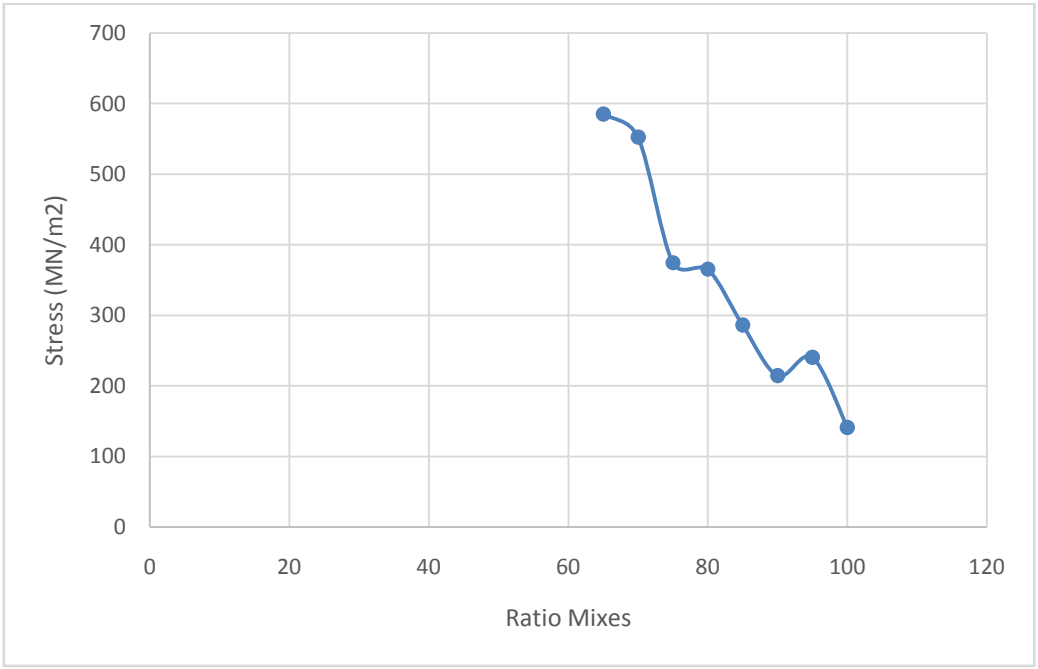
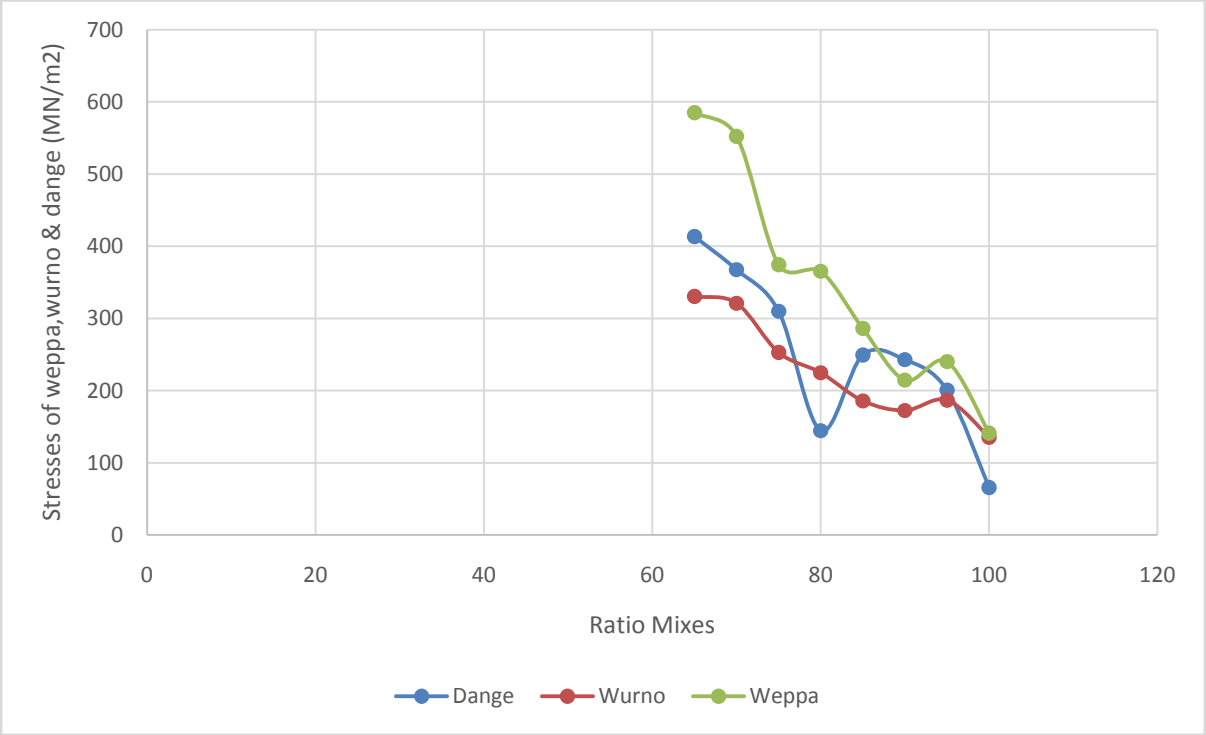


Fig. 8: **A combined graph of stress Vs Ratio mixes for Weppa, Wurno and Dange samples**



There is a mathematical relationship between compressive strength (Stress), force and Area of any given sample and in this case plaster of Paris test cylinders. This relationship is expressed in the formulae as shown below:

$$\text{Stress } (\sigma) = \frac{\text{Force}}{\text{Area}} \quad \text{or} \quad \sigma = \frac{F}{A}$$

Where σ = Sigma

F = Force

A = Area

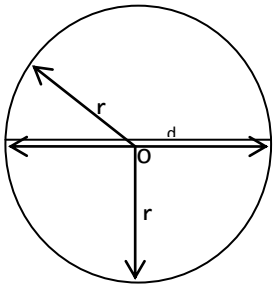
It is important at this stage to be reminded that the area of a solid cylinder is $2\pi r^2 + 2\pi rh$

Where r = Radius

h = height

π = 22/7 or 3.142

The above formulae is 2 x π x r^2 + 2 x π x r x h r = D/2. The Radius of any given object is its diameter divided by two(2) and the radius of any object e.g. a circle is the straight line distance from the centre to any part of the circumference.



Key r = radius

o = center

d = half

In conclusion, for every location in this experiment, 65 ratio mix is the strongest or the mix with the highest stress level compressive strength. 100 mix is the ratio mix with the weakest plaster/water ratio mix. Dange's 100 plaster/water ratio mix and 80 water/plaster ratio mix appear to be the one with unexpected low compressive strength. The general conclusion in this segment of experiment is that Wurno present low compressive strength when placed in comparison with Dange and Weppa. Although Dange is stronger than Wurno in compressive strength analysis, Weppa has the strongest compressive strength of +600 meganewton / m2 as shown in table 4.7 to 4.9.

4.4 Density Test

Table 4.13 Wurno Density Test

Sample Label	L	B	T	Vol	Mass	Density g/cm^3
100/100 Wurno	12.00	4.65	1.65	92.07	99.20	1.08
95/100 Wurno	11.77	4.55	1.50	80.33	92.20	1.15
90/100 Wurno	12.00	4.90	1.50	88.20	98.20	1.11
85/100 Wurno	12.00	4.80	1.70	97.92	98.50	1.01
80/100 Wurno	12.00	4.90	1.50	88.20	96.30	1.09
75/100 Wurno	11.70	4.80	1.30	73.01	76.70	1.05
70/100 Wurno	11.90	4.80	1.37	78.25	89.00	1.14
65/100 Wurno	12.10	5.00	1.40	84.70	99.20	1.17

Key: L = Length B = Breadth T = Thickness

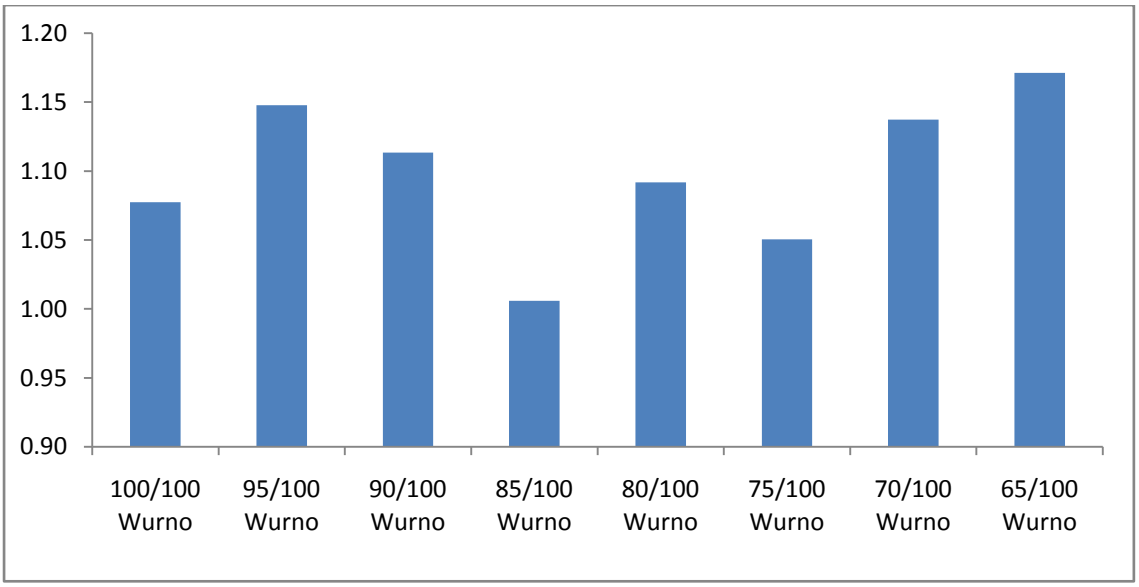


Fig. 9: Chart showing density test recorded from Wurno sample

Table 4.14 Dange Density Test

Sample Label	L	B	T	Vol	Mass	Density g/cm^3
100/100 Dange	12.00	4.90	1.83	107.60	98.00	0.91
95/100 Dange	12.00	5.00	1.80	108.00	99.50	0.92
90/100 Dange	12.00	4.90	1.80	105.84	98.70	0.93
85/100 Dange	12.10	5.00	1.70	102.85	100.00	0.97
80/100 Dange	12.20	4.70	1.60	91.74	97.00	1.06
75/100 Dange	12.10	4.90	1.50	88.94	95.50	1.07
70/100 Dange	12.10	5.00	1.43	86.52	97.20	1.12
65/100 Dange	12.30	4.70	1.35	78.04	95.20	1.22

Key: L = Length B = Breadth T = Thickness

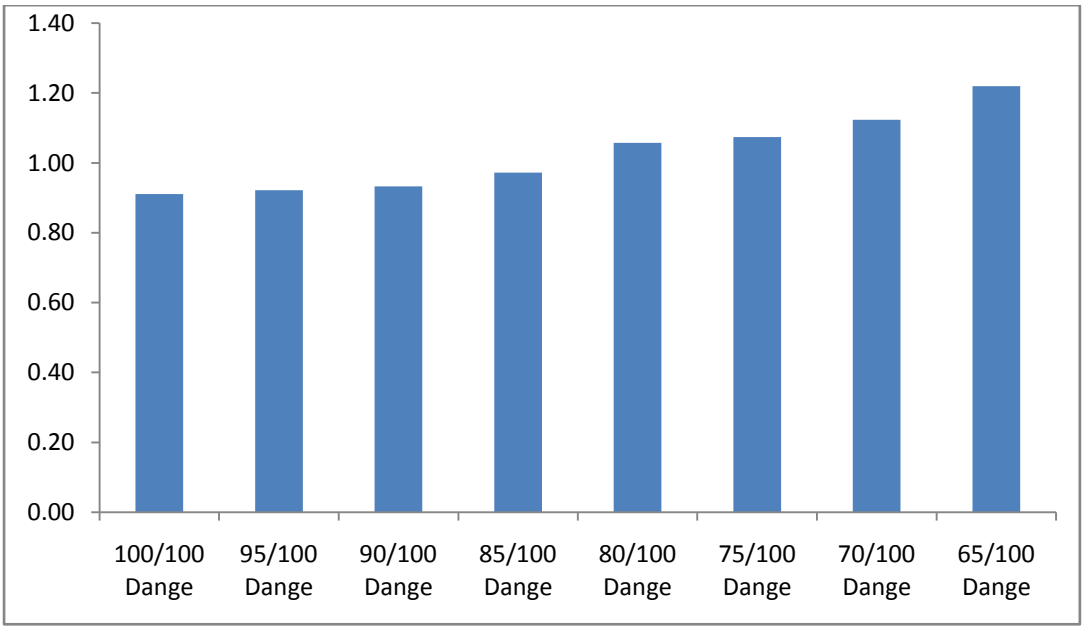


Fig. 10: Chart showing density test recorded from Dange sample

Table 4.15 Weppa Density Test

Sample Label	L	B	T	Vol	Mass	Density g/cm^3
100/100 Weppa	12.10	4.90	1.93	114.43	99.50	0.87
95/100 Weppa	12.10	5.00	1.80	108.90	99.80	0.92
90/100 Weppa	12.00	4.80	1.80	103.68	100.50	0.97
85/100 Weppa	12.00	4.45	1.70	90.78	99.50	1.10
80/100 Weppa	12.00	4.90	1.50	88.20	93.30	1.06
75/100 Weppa	11.67	4.75	1.30	72.06	76.60	1.06
70/100 Weppa	12.00	5.00	1.50	90.00	96.00	1.07
65/100 Weppa	12.00	4.90	1.35	79.38	92.70	1.17

Key: L = Length B = Breadth T = Thickness

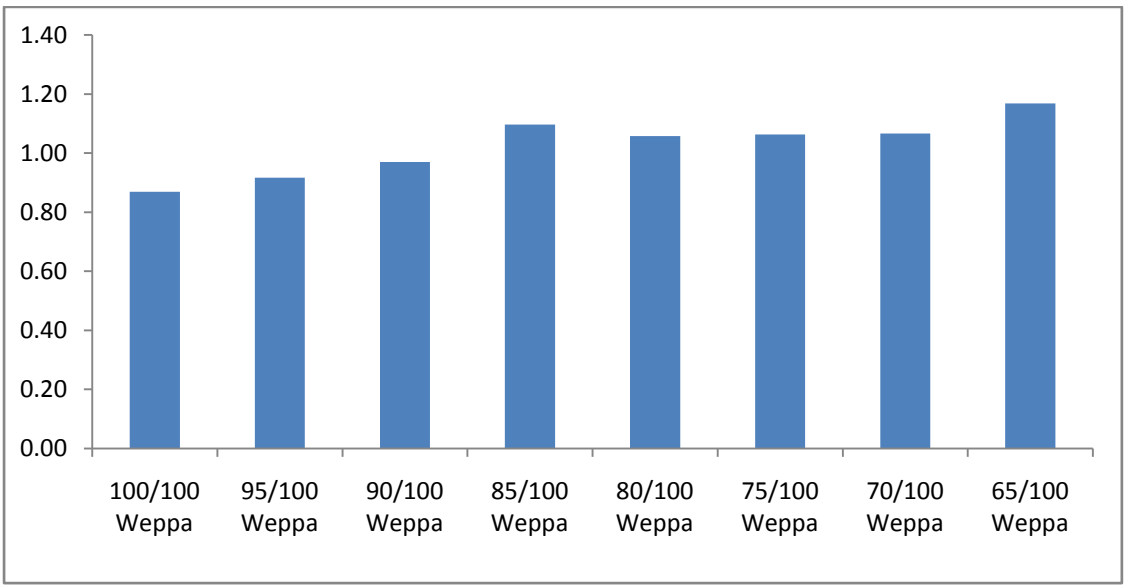


Fig. 11: Chart showing density test recorded from Weppa sample

4.4.1 Observation of Dry Density Test on Wurno, Dange and Weppa Gypsum/Water ratio mix

The shape of the sample pieces in this experiment was different from the ones used for the compressive strength test samples. Even though the ratio mix of plaster/water was exactly the same, the shape was different. The sample test pieces for compressive strength experiment were produced to specifications so also were the sample pieces for density test as they were produced to suit, the type of experiment it was meant for. Density, mass and volume of the specimen, demand that the sample should be sizeable and so this was made to be in rectangular tile form. The plaster tiles or cuboid measurement was administered on individual test sample because of unevenness of edges and walls.

The density tests for Wurno, Dange and Weppa plaster of Paris were carried out at the soil mechanics laboratory of Department of Civil Engineering, Faculty of Engineering, Ahmadu Bello University, Zaria and it is as presented below:

4.4.2 Discussion on Dry Density Test of Wurno, Dange and Weppa Plaster/Water Ratio Mix

4.4.2.1 Density of Wurno Plaster

The density test of Wurno plaster samples 100:65, 100:75, 100:80, 100:85, 100:90, 100:95, 100:100 results is presented here. The plaster/water ratio mix of 100:85 was the one identified with the lowest density which is at 1.01gram per centimetre cube which is expressed as 1.01g/cm^3. The second lowest density was that of 100:75 with the density of 1.05g/cm3, 100:100 follow with 1.08g/cm^3, the density of 100:80 was next with 1.09g/cm^3. The highest density in Wurno location was at 100:65 plaster/water ratio at 1.17g/cm^3. The plaster ratio mix of 100:95 was 1.15g/cm^3, then the 100:70 ratio mix with 1.14g/cm^3. The 100:90 plaster/water ratio mix was the last among the high density recorded in Wurno plaster

with 1.11g.cm^3 was 100:90 plaster ratio mix. Wurno dry density test was the best density with an average density of 1.1g/cm^3.

4.4.2.2 Density of Dange Plaster

The density test of Dange plaster of Paris was noted to descend from plaster/water ratio mix of 100:65 being the highest density recorded in this location, through to 100:100 plaster/water ratio mix. The density for the sample 100:65 plaster/water ratio mix was recorded at 1.22g/cm^3, followed by 100:70 ratio mix with 1.12g/cm^3, then 100:75 had the density of 1.07g/cm^3. This was close followed by 100:80 mix with 1.06g/cm^3. These four plaster/water ratio, 100:85, 100:90, 100:95 and 100:100 recorded density that was below one(1)g/cm^3. The density for these three samples were as follows, 0.97g/cm^3, 0.93g/cm^3, 0.92g/cm^3, and 0.91g/cm^3 respectively. The flow of density recorded in plaster from Dange location was a record of smooth ascents or descent from one end to the order as shown in table

4.4.2.3 Density of Weppa Plaster

Density test of Weppa plaster show that, of the eight samples of plaster/water ratio mixes 100:65, 100:75, 100:80, 100:85, 100:90, 100:95, and 100:100, 65 mix was the one with the highest density at 1.17g/cm^3. This was followed by three recordings that were almost identical in 100:70, 100:75 and 100:80 mixes, and these recordings read thus 1.07g /cm^3, 1.06g /cm^3 and 1.06g /cm3 respectively. 100:85 was the next with 1.10g/cm^3 while the rest were below 1 gram. They are 100:90, 100:95 and 100:100 with 0.97g/cm^3, 0.92g/cm^3 and 0.87g/cm^3.

4.4.3 Comparative Analysis of Dry Density Tests in Wurno, Dange and Weppa Plaster

It should be understood that samples used here in this segment were the rectangular tiles produced for this study in the PTDF laboratory of Chemical Engineering, Faculty of Engineering, Ahmadu Bello University, Zaria. The density of 100:65 in the three locations of

Wurno, Dange and Weppa were all above 1g/cm^3, with Wurno's 100:65 mix reading 1.17g/cm^3, Dange's 65 mix being 1.22g/cm^3 rating the highest density and Weppa's 65 mix ranking the same with Wurno at 1.7g/cm^3. The density level in all the plaster/water ratio mixes in Wurno is good as all the eight samples read above one(1) gram as followed from 65 to 100 mix, 1.17g/cm^3, 1.14g/cm3, 1.05g/cm^3, 1.09g/cm^3, 1.01g/cm^3, 0.17g/cm^3, 1.15g/cm^3 and 1.08g/cm^3 as indicated also on the table and graph.

The Dange and Weppa plaster have four and three sample each below (1) one gram, that is in Dange, from 100:85 plaster/water ratio mix, 100:90, 100:95 and 100:100, it was recorded thus, 0.97g/cm^3, 0.93g/cm^3, 0.92g/cm^3 and 0.91g/cm^3. Weppa plaster as early mentioned has three samples namely, 100:90 mix reading 0.97g/cm^3, 100:95 mix of plaster/water having the density of 0.92g/cm^3 and 100:100 plaster ratio mix reading the lowest among the three locations at 0.87g/cm^3. Dange sample of plaster records the highest density at the plaster/water ratio mix of 100:65 at 1.22 grams/cm^3.

4.4.4 Discussion on bulk density

The bulk density of the processed plaster of Paris from the three locations of this study, namely Wurno, Dange and Weppa are as shown below.

Wurno

Taking into consideration, the concluded dry density test of Wurno sample, as earlier mentioned, processed plaster of Paris will be used in production of ceramic moulds. These moulds will be useful when dried and could still be used even when moulds are wet from casting processes. It is therefore imperative that the bulk density known which involves assessment of plaster mould density when saturated with water.

In the Wurno plaster/water ratio mixes, the 100:95 ratio mix presented the highest bulk density record at 1.83grams per cubic centimeter. The lowest record of bulk density in the Wurno category was the 100:75 ratio mix with 1.53grams per cubic centimeter. Record available for other ratio mixes were as follows; 100:65 recorded 1.61g/cm^3, 100:70 was 1.62g/cm^3, 100:80 was 1.58g/cm^3, 100:85 was at 1.55g/cm^3, 100:90 and 100:100 mixes were 1.69g/cm^3 each and therefore, 95 mix or 100:95 mix was the highest bulk density in Wurno plaster.

Dange

The Dange plaster bulk density was the most predictable, owing to the fact that the highest bulk density of this location was highest at 100:65 plaster/ratio mix with 1.68 grams per cubic centimeter. A progressive reduction is observed until the duplicated bulk density at 100:75 and 100:80 at 1,58 grams per cubic centimeter each. The continuous reduction in the bulk density is observed in both 199:85 and 100:90 with 1.50 grams per cubic centime and 1.47 grams per cubic centimeter respectively. 1.50 grams per cubic centimeter is repeated in 100:95 plaster/water ratio mix. The bulk density for 100:100 mix is at 1.51 grams per cubic centimeters. This concludes the bulk density for plaster from Dange location.

Weppa

Weppa plaster recorded the highest bulk density at the third plaster/water ratio mix of 100:75 with 1.95 grams per cubic centimeter or 1.95g/cm3. 150 grams per cubic centimeter was the bulk density of 100:70 and 100:90 ratio mixes. The second highest bulk density is observed at 100:85 water/plaster ratio mix. 100:65 ratio mix was observed with 1.56 grams per cubic centimeter, followed by 100:80 mix with 1.54g/cm^3, 100:95 and 100:100 mixes recorded the bulk densities of 1.45g/cm^3 and 1.42g/cm^3 respectively.

4.4.5 Comparative Analysis of Bulk Density of Wurno, Dange and Weppa Processed Plaster.

In the tables and graphs representation of bulk density, Weppa plaster in this experiment recorded the highest bulk density as well as the lowest bulk density. This could be observed in the graph and table representing bulk density from Weppa. 1.95grams per cubic centimeter is observed at 100:75 ratio mix; this is the highest bulk density record. At the same time, Weppa also records the lowest bulk density at 100:100 plaster/water ratio mix with 1.42grams per cubic centimeter. The highest bulk density in Wurno is at 100:95 plaster/water ratio mix with 1.83grams per cubic centimeter, while Dange's highest bulk density is at 100:65 mix at 1.65grams per cubic centimeters. The lowest bulk density in Dange is in the 100:90 plaster/water ratio mix with 1.47grams per cubic centimeter. The average bulk density in Wurno plaster sample is at 1.64grams per cubic centimeter, Dange is 1.55g/cm^3 and Weppa is at 1.57g/cm^3. Wurno records the highest average bulk density in this study.

4.5 XRF (X-Ray Fluorescence Spectrometer)

The following are elemental composition of Wurno, Dange and Weppa gypsum rocks (%) are presented in Tables 4.17 to 4.22.

Table 4.16: Elemental Composition of Wurno Gypsum Rock

	ELEMENT OXIDE	WURNO 01	WURNO 02	WURNO 03	GYPSUM AVERAGE
1	S_iO_2	11.497	9.089	8.207	9.597
2	$Al_2 O_3$	5.20	3.78	3.589	4.189
3	$Fe_2 O_3$	3.125	3.005	3.164	3.098
4	Mn O	0.211	0.223	0.241	0.337
5	Mg O	1.307	1.176	0.959	1.147
6	CaO	39.834	41.263	42.315	41.137
7	$Na_2 O$	1.185	1.387	1.279	1.283
8	$K_2 O$	0.222	0.182	0.182	0.195
9	T_iO_2	0.339	0.263	0.275	0.292
10	SO_3	36.229	38.632	39.062	37.974
11	Zn O	0.003	0.003	0.002	0.0026
12	Sr O	0.035	0.035	0.003	0.243
13	$Cr_2 O_3$	0.005	0.005	0.006	0.005
14	Cl	0.101	0.025	0.054	0.06
15	$P_2 O_3$	9.704	0.814	0.632	3.626
16	LOI	nd	nd	Nd	Nd
17	Combined (H_2O)	nd	nd	Nd	nd

Note: Detection Limit is 0.001 PPM (part per million) to nearly 100%.
nd - not detected

TABLE 4.17: Elemental Composition of Dange Gypsum Rock

s/n	ELEMENT OXIDE	DANGE 01	DANGE 02	DANGE 03	GYPSUM AVERAGE
1	S_iO_2	10.462	10.587	10.802	10.617
2	$Al_2 O_3$	5.023	5.187	5.145	5.118
3	$Fe_2 O_3$	2.843	2.834	2.779	2.815
4	Mn O	0.190	0.141	0.138	0.156
5	Mg O	1.128	1.219	1.325	1.224
6	CaO	39.96	39.76	38.733	39.484
7	$Na_2 O$	1.188	1.43	1.486	1.368
8	$K_2 O$	0.260	0.252	0.257	0.256
9	T_iO_2	0.288	0.288	0.265	0.280
10	SO_3	37.751	37.572	38.058	37.793
11	Zn O	0.000	0.002	0.000	0.0006
12	Sr O	0.033	0.034	0.030	0.032
13	$Cr_2 O_3$	0.004	0.005	0.005	0.0046
14	Cl	0.113	0.114	0.165	0.130
15	$P_2 O_3$	0.758	0.747	0.812	0.772
16	LOI	nd	nd	nd	Nd
17	Combined (H_2O)	nd	nd	nd	Nd

Note: Detection Limit is 0.001 PPM (part per million) to nearly 100%.
nd - not detected

TABLE 4.18: Elemental Composition of Weppa Gypsum Rock

s/n	ELEMENT OXIDE	WEPPA 01	WEPPA 02
1	S_iO_2	0.06	1.60
2	$Al_2 O_3$	0.42	0.96
3	$Fe_2 O_3$	0.12	0.258
4	Mn O	0.023	0.061
5	Mg O	0.04	Nd
6	CaO	32.4	40.59
7	$Na_2 O$	0.033	Nd
8	$K_2 O$	Nd	Nd
9	T_iO_2	0.013	0.05
10	SO_3	46.43	46.7
11	Zn O	Nd	Nd
12	Sr O	Nd	Nd
13	$Cr_2 O_3$	Nd	Nd
14	Cl	Nd	Nd
15	$P_2 O_3$	Nd	Nd
16	LOI	20.45	Nd
17	Combined (H_2O)	Nd	9.78

Note: Detection Limit is 0.001 PPM (part per million) to nearly 100%.
nd - not detected

Tables of Major Elements Composition in Calcined Gypsum Powder (Plaster Of Paris) from Wurno, Dange and Weppa (%)

Table 4.19: Elemental Composition of Calcined Gypsum Powder from Wurno

s/n	ELEMENT OXIDE	WURNO 01	WURNO 02	WURNO 03	PLASTER AVERAGE
1	S_iO_2	8.01	8.052	7.997	8.019
2	$Al_2 O_3$	2.75	3.16	2.78	2.896
3	$Fe_2 O_3$	2.048	2.103	2.107	2.086
4	Mn O	0.163	0.173	0.149	0.161
5	Mg O	0.809	0.797	0.823	0.809
6	CaO	42.802	42.575	42.449	42.608
7	$Na_2 O$	1.203	1.197	1.372	1.257
8	$K_2 O$	0.131	0.142	0.138	0.137
9	T_iO_2	0.109	0.116	0.113	0.112
10	SO_3	41.194	40.939	41.241	41.124
11	Zn O	0.000	0.000	0.000	0.000
12	Sr O	0.031	0.031	0.031	0.031
13	$Cr_2 O_3$	0.002	0.003	0.004	0.003
14	Cl	0.055	0.051	0.067	0.057
15	$P_2 O_3$	0.681	0.661	0.727	0.689
16	LOI	nd	nd	nd	Nd
17	Combined (H_2O)	nd	nd	nd	Nd

Note: Detection Limit is 0.001 PPM (part per million) to nearly 100%.
nd - not detected

TABLE 4.20: Elements Composition in Calcined Gypsum Powder from Dange

s/n	ELEMENT OXIDE	DANGE 01	DANGE 02	DANGE 03	PLASTER AVERAGE
1	S_iO_2	13.146	12.921	13.075	13.047
2	Al_2O_3	6.021	5.932	6.061	6.004
3	Fe_2O_3	2.687	2.694	2.770	2.717
4	MnO	0.193	0.145	0.171	0.169
5	MgO	1.286	1.882	1.220	1.462
6	CaO	37.548	37.211	37.593	37.450
7	Na_2O	1.051	1.354	0.985	1.13
8	K_2O	0.251	0.267	0.261	0.259
9	T_iO_2	0.245	0.252	0.273	0.256
10	SO_3	36.99	37.576	36.930	37.165
11	ZnO	0.002	0.002	0.0002	0.002
12	SrO	0.034	0.033	0.033	0.033
13	Cr_2O_3	0.006	0.004	0.005	0.005
14	Cl	0.052	0.077	0.051	0.06
15	P_2O_3	0.578	0.649	0.571	0.599
16	LOI	nd	nd	nd	nd
17	Combined (H_2O)	nd	nd	nd	nd

Note: Detection Limit is 0.001 PPM (part per million) to nearly 100%.
nd - not detected

TABLE 4.21: Elements Composition in Calcined Gypsum Powder from Weppa

S/N	ELEMENT OXIDE	WEPPA 01	WEPPA 02
1	S_iO_2	0.06	1.60
2	$Al_2 O_3$	0.42	0.96
3	$Fe_2 O_3$	0.12	0.258
4	Mn O	0.023	0.061
5	Mg O	0.04	Nd
6	CaO	32.4	40.59
7	$Na_2 O$	0.033	Nd
8	$K_2 O$	Nd	Nd
9	T_iO_2	0.013	0.05
10	SO_3	46.43	46.7
11	Zn O	Nd	Nd
12	Sr O	Nd	nd
13	$Cr_2 O_3$	Nd	nd
14	Cl	Nd	nd
15	$P_2 O_3$	Nd	nd
16	LOI	20.45	nd
17	Combined (H_2O)	Nd	9.78

Note: Detection Limit is 0.001 PPM (part per million) to nearly 100%.
nd - not detected

4.5.1 Analysis of XRF (X-ray Fluorescence Spectrometer)

The chemical test for Wurno, Dange and Weppa gypsum and plaster was done at the Multi User Laboratory of Department of Chemistry, Faculty of Sciences, Ahmadu Bello University, Zaria. The instrument used for the chemical analysis is called Oxford X Supreme. According to the User's Manual, this machine (Oxford X Supreme) could be used to measure virtually every element from sodium (Na) to platuinum (Pu) in the periodic table. This can be done in concentration ranging from part per million (PPM) to nearly 100%. The detection limit of this device is 0.00/ppm.

From the Table presented on the chemical composition of Wurno gypsum which is calculated in percentage as shown in the Table above

4.5.2 Discussion on raw gypsum and processed plaster from Wurno

SiO_2

The values of SiO_2 in raw gypsum from Wurno range between 8.207% to 11.49% with an average value of 9.5976%. When compared with the values in plaster of Paris from the same location which range between 7.997% to 8.01 % with an average value of 8.0198%, it shows that there is a decrease of silica after calcination.

Al_2O_3

The values of Al_2O_3 in raw gypsum from Wurno range between 3.589% to 5.20% with an average value of 4.189%. When compared with the average value of plaster of Paris from the same location which range between 2.75% to 2.78%, with an average value of 2.896%, it shows there is decrease in Al_2O_3 after calcination.

Fe_2O_3

The values of Fe_2O_3 in raw gypsum from Wurno range between 3.005% to 3.164% with an average value of 3.098%, and when compared with the value of plaster of Paris from the same location which range from 2.048% to 2.107%, with an average value of 2.086%. This shows there a decrease in Fe_2O_3 after calcination.

MnO

The values of MnO in raw gypsum from Wurno range between 0.211% to 0.241% with an average value of 0.337% and when compared with the value of plaster of Paris from the same location which range between 0.149% to 0.163% with an average value of 0.161%. This shows there is a decrease in MnO after calcination.

MgO

The values of MgO in raw gypsum from Wurno range between 0.959% to 1.307% with an average value of 1.147%, when compared with the average values of plaster of Paris from the same location which range from 0.823% to 0.809% with an average value of 0.809%, it shows there is a decrease in MgO after calcination.

CaO

The values of CaO in raw gypsum from Wurno range between 39.834% to 42.315% with an average value of 41.137%, when compared with the average values of plaster of Paris from the same location which range from 42.449% to 42.802%, with an average value of 42.608%. This shows there is increase of CaO after calcination.

Na_2O

The values of Na_2O in raw gypsum from Wurno range between 1.185% to 1.387% with an average value of 1.283%, when compared with the average values of plaster of Paris from the same location which range from 1.203% to 1.372%, with an average value of 1.257%. This shows there is decrease of Na_2O after calcination.

K_2O

The values of K_2O in raw gypsum from Wurno range between 0.182% to 0.222% with an average value of 0.195%, and when compared with the average values of plaster of Paris from the same location which range from 0.131% to 0.138%, it shows an average value of 0.137%. This shows there is decrease of K_2O after calcination

TiO_2

The value of TiO_2 in raw gypsum from Wurno range from 0.263% to 0.339% with an average value of 0.292%, and when compared with the average values of plaster of Paris from the

same location which range from 0.109% to 0.138%, it shows an average value of 0.112. This shows there is decrease of TiO_2 after calcination.

SO_3

The values of SO_3 in raw gypsum from Wurno range between 36.229% to 39.062% with an average value of 37.974% and when compared with the average values of plaster of Paris from the same location which range from 41.194% to 41.241%, it shows an average value of 41.124%. This shows there is increase of SO_3 after calcination.

ZnO

The value of ZnO in raw gypsum from Wurno range between 0.002% to 0.003% with an average value of 0.0026% and when compared with the average values of plaster of Paris from the same location which all have 0.000% values, it shows an average value of 0.000%. This shows there is no increase nor decrease of ZnO after calcination.

SrO

The average value of SrO in raw gypsum from Wurno range between 0.003% to 0.0035% with an average value of 0.243% and when compared with the average value of plaster of Paris from the same location which range between 0.031% to 0.031%, it shows an average value of 0.031. This shows there is decrease of SrO after calcination.

Cr_2O_3

The values of Cr_2O_3 in raw gypsum from Wurno range between 0.005% to 0.006% with an average value of 0.005% and when compared with the average values of plaster of Paris from the same location which range between 0.002% to 0.004%, it shows an average value of 0.003. This shows there is decrease of Cr_2O_3 after calcination.

Cl

The values of Cl in raw gypsum from Wurno range between 0.025% to 0.101% with an average value of 0.06% and when compared with the average values of plaster of Paris from the same location which range between 0.055% to 0.067%, it shows an average value of 0.057%. This shows there is an increase of Cl after calcination.

P_2O_3

The values of P_2O_3 in raw gypsum from Wurno range between 0.632% to 9.704% with an average value of 3.626% when compared with the average values of plaster of Paris from the same location which range from 0.681% to 0.727%, it shows an average value of 0.689%. This shows there is decrease in P2O3 after calcination.

4.5.3 Discussion on Raw Gypsum and processed plaster from Dange

SiO2

The values of SiO_2 in raw gypsum from Dange range between 10.462% to 10.802% with an average value of 10.617%, when compared with the average values of plaster of Paris from the same location which range between 13.075% to 13.146%, it shows an average value of 13.047%. This shows there is an increase in SiO_2 after calcination.

Al_2O_3

The values of Al_2O_3 in raw gypsum from Wurno range between 5.023% to 5.187% with an average value of 5.118%, when compared with the average values of plaster of Paris from the same location which range between 2.75% to 2.78%, it shows an average value of 6.004%. This shows there is increase in Al_2O_3 after calcination.

Fe_2O_3

The values of Fe_2O_3 in raw gypsum from Dange range between 2.779% to 2.843% with an average value of 2.815%, when compared with the average values of plaster of Paris from the same location which range from 2.107% to 2.048%, it shows an average value of 2,717%. This shows there is decrease in the Fe_2O_3 after calcination.

MnO

The values of MnO in raw gypsum from Dange range between 0.138% to 0.190% with an average value 0.156%, when compared with the average values of plaster of Paris from the same location which range between 0.149% to 0.163%, it shows an average value of 0.169%. This shows there is increase in MnO after calcination.

MgO

The values of MgO in raw gypsum from Dange range between 1.128% to 1.325% with an average value of 1.224%, when compared with the average values of plaster of Paris from the same location which range between 0.809% to 0.823%, it shows an average value of 1.462%. This shows there is an increase in MgO after calcination.

CaO

The values of CaO in raw gypsum from Dange range between 37.211% to 37.593% with an average value of 39.484%, and when compared with the average values of plaster of Paris from the same location which range between 42.449% to 42.802%, it shows an average value of 37.450%. This shows there is decrease of CaO after calcination.

Na_2O

The values of Na_2O in raw gypsu from Dange range between 1.188% to 1.486% with an average value of 1.368%, and when compared with the average values of plaster of Paris

from the same location which range between 1.188% to 1.486%, it shows an average value of 1.13%. This shows there is decrease of Na_2O after calcination.

K_2O

The values of K_2O in raw gypsum from Dange range between 0.252% to 0.260% with an average value of 0.256% and when compared with the average values of plaster of Paris from the same location which range from 0.131% to 0.138%, it shows an average value of 0.259%. This shows there is an increase in K_2O after calcination.

TiO_2

The values of TiO_2 in raw gypsum from Dange range between 0.265% to 0.288% with an average value of 0.280% and when compared with the average values of plaster of Paris from the same location which range from 0.109% 0.113%, it shows an average value of 0.256%. This shows there is decrease in TiO_2 after calcination.

SO_3

The values of SO_3 in raw gypsum from Dange range between 37.572% to 38.058% with an average value of 37.793% and when compared with the average values of plaster of Paris from the same location which range from 41.194% to 41.241%, it shows an average value of 37.165%. This shows there is decrease in SO_3 after calcination.

ZnO

The values of ZnO in the raw gypsum from Dange range between 0.000% to 0.002% with an average value of 0.006% and when compared with the average values of plaster of Paris from the same location which range between 0.000% to 0.000%, it shows an average value of 0.002%. This shows there is decrease in ZnO after calcination.

SrO

The value of SrO in raw gypsum from Dange range between 0.030% to 0.034% with an average value of 0.032%, and when compared with the average values of plaster of Paris from the same location which range from 0.031% to 0.031%, it shows an average value of 0.033. This shows there is increase of SrO after calcination.

Cr_2O_3

The values of Cr_2O_3 in raw gypsum from Dange range between 0.004% to 0.005% with an average value of 0.0046%, when compared with the average values of plaster of Paris from the same location which range from 0.002 to 0.004, it shows an average value of 0.005. This shows there is decrease of Cr_2O_3 after calcination.

Cl

The values of Cl in raw gypsum from Dange range between 0.113% to 0.165% with an average value of 0.130%, and when compared with the average values of plaster of Paris from the same location which range from 0.055% to 0.067%, it shows an average value of 0.06%. This shows there is decrease of Cl after calcination.

P_2O_3

The values of P_2O_3 in raw gypsum from Dange between 0.747% to 0.812% with an average value of 0.772%, and when compared with the average values of plaster of Paris from the same location which range between 0.717%, it shows an average value of 0.599%. This shows there is a decrease of P_2O_3 after calcination.

4.5.4 Discussion on Raw Gypsum and processed plaster from Weppa

SiO_2

The value of SiO_2 in raw gypsum from Weppa shows value of 0.06, and when compared with the average value of plaster of Paris from the same location, it shows an average value of 1.60. This shows there is an increase of SiO_2 after calcination.

Al_2O_3

The value of Al_2O_3 in raw gypsum from Weppa shows value of 0.42, and when compared with the average value of plaster of Paris from the same location, it shows an average value of 0.96. This shows there is an increase of Al_2O_3 after calcination.

Fe_2O_3

The value of Fe_2O_3 in raw gypsum from Weppa showed value of 0.12, and when compared with the average value of plaster of Paris from the same location, it shows an average value of 0.258. This shows there is an increase in Fe_2O_3 after calcination.

MnO

The value of MnO in raw gypsum from Weppa shows value of 0.023, and when compared with the average value of plaster of Paris from the same location, it shows an average value of 0.061. This shows there is an increase in MnO after calcination.

MgO

The value of MgO in raw gypsum from Weppa shows the value of 0.04, and when compared with the average value of plaster of Paris from the same location, it shows zero value. This shows an absence of MgO after calcination.

CaO

The value of CaO in raw gypsum from Weppa shows the value of 32.4, and when compared with the average value of plaster of Paris from the same location, it shows the average of 40.29. This shows there is an increase in CaO after calcination.

Na_2O

The value of Na_2O in raw gypsum from Weppa shows the value of 0.033, and when compared with the average of plaster of Paris from the same location, it shows zero value. This shows an absence of Na_2O after calcination.

TiO_2

The value of TiO_2 in raw gypsum from Weppa shows value of 0.013, and when compared with the average value of plaster of Paris from the same location, it shows an average value of 0.05. This shows there is an increase in TiO_2 after calcination.

SO_3

The value of SO_3 in raw gypsum from Weppa shows the value of 46.43, and when compared with the average value of plaster of Paris from the same location, it shows an average value of 46.7. This shows there is an increase in SO_3 after calcinations.

LO_1

The value of LO_1 in raw gypsum from Weppa shows the value of 20.45, and when compared with the average value of plaster of Paris from the same location, it shows the absence of LO_1 after calcination.

H_2O

The value of combined H_2O in raw gypsum was absent therefore no value was shown or indicated and when compared with the value of plaster of Paris from the same location, it shows the value of 9.78. This shows the presence of combined H_2O after calcination.

4.6 XRD (X-Ray Diffraction)

Mineralogical composition of gypsum obtained from Wurno, Dange and Weppa are presented in Tables 4.23 to 4.28.

Table 4.22 Mineralogical Composition of Gypsum Obtained at Wurno

	Chemical Name	Chemical Formular	Mineral Name
1	Silicon Oxide	SiO_2	Quartz
2	Magnesium Silicate Hydroxide	$Mg_3Si_4O_{10}(OH)_2$	Talc
3	Sodium calcium Magnesium Silicate	$NaCa_2Mg_5AlSi_7O_{22}(OH)_2$	
4	Calcium Sulphite	$Ca(PO_3)_2$	
5	Calcium Sulfite	$CaSO_3$	
6	Calcium Sulfate	$CaSO_4$	Anhydrite
7	Potassium Aluminium Silicate Hydroxide	$KAl_2(Si_3Al)O_{10}(OH,F)_2$	
8	Potassium Aluminium Silicate Hydroxide	$(KiH_3O)Al_2Si_3AlO_{10}(OH)_2$	
9	Calcium Magnesium Silicate Hydroxide	$Ca_2Mg_5Si_8O_{22}(OH)_2$	
10	Calcium Silicate	$CaSiO_3$	Wollastonite

Table 4.23 Mineralogical composition of gypsum obtained from Dange

	Chemical Name	Chemical Formular	Mineral Name
1	Aluminum Silicate Hydroxide Hydrate	$Al_2Si_2O_5(OH)_4.2H_2O$	Halloysite
2	Sodium Calcium Magnesium Aluminum Silicate	$NaCa_2Mg_5AlSi_7O_{22}(OH)_2$	
3	Magnesium Silicate Hydroxide	$Mg_3Si_4O_{10}(OH)_2$	Talc
4	Silicon Oxide	SiO_2	Cristobalite
5	Calcium Sulfite	$CaSO_3$	
6	Calcium Sulphate	$CaSO_4$	Anhydrite
7	Calcium Magnesium Silicate Hydroxide	$Ca_2Mg_5Si_8O_{22}(OH)_2$	Sepiolile

Table 4.24 Mineralogical composition of gypsum obtained at Weppa

	Chemical name	Chemical formular	Mineral name
1	Calcium Sulfate Hydrate	$CaSO_4.2H_2O$	Gypsum

Table 4.25 Mineralogical composition of calcined gypsum poweder obtained from Wurno

	Chemical name	Chemical formular	Mineral name
1	Magnesium Oxide	MgO	Periclase, Syn
2	Calcium Sulphate	$CASO_4$	
3	Magnesium Aluminum Silicate Hydroxide Hydr.	$Mg,Al_9 Si,AL_8O_{20}(OH)_{10}.5H_2O$	Talc
4	Titanium Nitride	Tin	Osbornite (Syn)
5	Calcium Silicate Hydrate	$Ca_5Si_6(O,OH,F)_{18}.5H_2O$	Tobermovite
6	Chromium Oxide	Cr_2O_3	Eskolaite
7	Magnesium Silicate Hydroxide	$Mg_3(Si_2\text{-}Xo_5(OH)_{4\text{-}4x}$	Chrysotile
8	Sodium Aluminum Silicate	$Na_2Al_2Si_{13}.30_{29\text{-}6+x}$	Mordenite (syn)
9	Calcium Silicate Hydrate	$Ca_6Si_6O_{17}(OH)_2$	Xonotlite (syn)
10	Aluminum Silicate Hydroxide	$Al_2S_{i2}O_5(OH)_4$	Wollastonite

Table 4.26 Mineralogical composition of calcined gypsum poweder obtained from Dange

	Chemical Name	Chemical Formular	Mineral Name
1	Calcium Aluminum Iron	$CaO.1Fe_2(Si,Al)40_{10}(OH)_2.4H_2O$	
2	Aluminum Silicate Hydroxide	$Al_2Si_2O_5(OH)_4$	Halloysite
3	Calcium Titanium Oxide	$CaTiO_3$	Talc
4	Sodium Aluminum Silicate Hydrate	$Na_2Al_2Si_4O_{12}.8H_2O$	Faujusi
5	Calcium Silicate	$Ca_3Si_2O_7$	Rankinite
6	Barium Aluminum Silicate	$BaAl_2Si_2O_8$	Celsion, sun
7	Magnesium Silicate Hydroxide Hydrate	$Mg_4Si_6O_{15}(OH)_2.6H_2O$	Sepiolite

Table 4.27 Mineralogical composition of calcined gypsum powder obtained at Weppa

	Chemical Name	Chemical Formular	Mineral Name
1	Calcium Sulfate Hydrate	$Ca(SO_4)\ (H_2O)_{0.5}$	Bassanite

4.6.1 Discussion on XRD Analysis of Gypsum and Plaster from Wurno, Dange and Weppa Areas

X-ray diffraction deals with the mineralogical analysis of minerals. This study includes three locations namely Wurno, Dange and Weppa.

4.6.2 Discussion on Raw Gypsum from Wurno, Dange and Weppa Gypsum

The Gypsum from Wurno was identified with ten different mineral/chemicals components. The first is the Quartz (Silicon Oxide) or SiO_2, followed by Magnesium Silicate Hydroxide (Talc) $MgSi_4O_{10}(OH)_2$ and Sodium Calcium Magnesium Aluminium Silicate, that is, $NaCa_2Mg_5AlSi_70_{22}(OH)_2$. Calcium Phosphate $Ca(PO_3)_2$. Calcium Sulphite $CaSO_3$ and Calcium Sulphate Anhydrite $CaSO_4$ were others identified in the analysis. Other chemicals / minerals include Potassium Aluminum Silicate Hydroxide, $KA_{12}(Si_3Al)O_{10}(OH,F)_2$, Potassium Aluminum Silicate Hydroxide, $(K,H_3O)Al_2Si_3AlO_{10}(OH)_2$, Calcium Magnessium Silicate Hydroxide, $Ca_2Mg_5Si_8O_{22}(OH)_2$ and Calcium Silicate (Wollastonite) $CaSiO_3$.

The Dange XRD analysis identified seven (7) minerals in the gypsum analysis namely, Aluminum Silicate, Hydroxite Hydrate, Sodium Calcium Magnesium, Aluminum Silicate, Magnesium Silicate Hydroxide (Talc), Silicon Oxide (Cristobalite), Calcium Sulfite, Calcium Sulfate (Anhydrite) and Calcium Magnesium Silicate Hydroxide.

The sample obtained from Weppa after the XRD analysis was identified as gypsum, $CaSO_4.2H_2O$. The two molecules of water of Crystallization were present in this sample. Other minerals present in this sample analysis may have been insignificant or clearly absent as the mineral presented for analysis was only identified as gypsum, $CaSO_4.2H_2O$.

4.6.3 Discussion on Processed Plaster from Wurno, Dange and Weppa

After calculation, it was observed that most of the minerals/chemicals disappeared while new ones were formed and two were retained. The two that were preserved or retained were Calcium Sulfate ($CaSO_4$) and Magnesium Silicate Hydroxide $Mg_3(Si_2\text{-}xO_5)\ (OH)_{\ 4\text{-}4x}$. Newly formed minerals were Periclase Magnesium Oxide, Magnesium Aluminum Silicate Hydroxide or Hydr, Titanium Nitrate or Osbornite and Calcium Silicate hydrate or Tobermorite. Chromium Oxide or Eskolaite, Sodium Aluminum Silicate or Mordenite, Calcium Silicate Hydrate or Xonotite and Aluminum Silicate Hydroxide or Halloysite were among newly formed mineral. Such minerals as Silicon Oxide, Calcium Phosphate and so on were no more present in the newly formed plaster sample.

The Dange sample after the calcinations process was observed to have formed some other minerals, example of which are, Sepioloite (Magnesium Silicate Hydroxide Hydrate) which was absent in the Dange gypsum sample was present in the calcined plaster. Barium Aluminum Silicate (Celsion) Calcium Aluminum Iron Silicate Hydroxide, Calcium Titanium Oxide, Sodium Aluminum Iron Silicate Hydroxide (faujusi) and sodium Silicate (Rankinite) were newly formed. Calcium Sulfate (Anhydrite) was completely absent in the calcined Dange Plaster.

After calcinations, the material, plaster of Paris processed from gypsum obtained from Weppa in Edo state was subjected to X-ray defraction analysis. The result identified the material as Bassanite (calcium Sulphate Hydrate).

CHAPTER FIVE

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

5.0 Introduction

This chapter comprises of the summary, conclusion and recommendation arising from the study. In respect to the confines of limitations of the study, the aim and objectives of the study were centred on comparative analysis of Wurno, Dange and Weppa gypsum for ceramic production in Nigeria. Embedded in this chapter is therefore a summary of observations based on various tests and experiments carried out during the course of this research in order to achieve the objectives. The specific recommendations for further research work and general recommendations were also proffered in this chapter.

5.1 Summary

The study began with a field trip to the areas that have naturally occurring gypsum. These areas are Wurno, Dange and Weppa where raw gypsum samples were sourced. The material obtained from these three locations were labelled from site and transported down to the Ceramic Section of the Department of Industrial Design, Ahmadu Bello University, Zaria. These samples were characterised and beneficiated. Gypsum samples used for this study were washed, broken with hammer on a swept clean floor. Milled gypsum was then calcined for the purpose of being used for ceramic production.

Calcination was done through partial closing of the kiln flue, manipulations of temperature and close monitoring of sample for time limit, stirring and turning plaster from one container to another to avoid excess over-heating of local areas. Calcination was done in the Industrial Development Centre (IDC) Samaru Zaria. All the three samples were subjected to same treatments.

The tests carried out included absorbency, shrinkage, compressive strength and density tests. Further analysis carried out were the chemical and mineralogical tests such as X-ray Fluorescence Spectrophotometer (XRF) and X-ray Diffraction (XRD).

The absorbency test showed that plaster processed from Wurno, Dange and Weppa were generally absorbent as they could absorb liquid (water). On the average however, Weppa plaster had the average absorbency percentage; followed by Dange and Wurno having the lowest average percentage absorbency at 58.95%, 56.43% and 51.69% respectively. Shrinkage test showed minimal shrinkage except at 100:95 – 100 ratio mixes of Wurno and Dange plaster. On a general note, it is deduced that the compressive strength of Wurno presents the lowest compressive strength when compared with samples of Dange and Weppa. Although Dange appears stronger than Wurno considering the compressive strength analysis, Weppa has the strongest compressive strength of almost +600 meganewton per meters square, i.e. $+600MN/M^2$.

For the density test on (plaster of Paris) or gypsum plaster from Dange and Weppa plaster indicates less density than that of Wurno. This is evident in Dange's 100:85, 100:90, 100:95 and 100:100 mixes with stress or compressive strength of 0.97g/cm3, 0.93g/cm3, 0.92g/cm3 and 0.97g/cm3. Weppa plaster compressive strength test as above mentioned has three samples below one (1) gram namely 100.90 mix reading 0.97g/cm3, 100:95 mix of plaster/water ratio mix with 0.92g/cm3 and 100:100 plaster ratio mix reading the lowest at 0.87g/cm3. However, Wurno and Weppa plaster density read 1.17g/cm3 each at 65 ratio mix while Dange had 1.12g/cm3.

X-ray fluorescence spectrometer was used for chemical analysis, so as to determine the elements of a sample from sodium (Na) to platinum (Pu) in the periodic table. This analysis confirmed the presence of calcium and sulphate in the three samples from Wurno, Dange and

Weppa. Gypsum is called calcium sulphate. It is observed that there is decrease of all the elements analysed from Wurno gypsum (SiO_2, Al_2O_3, MnO, MgO, K_2O, SrO, FeO, Na_2O, TiO_2, LO_1, Cl, Cr_2O_3 and P_2O_3) except CaO and SO_3 which recorded increase after calcinations of raw gypsum from Wurno. The value of ZnO however indicates no change in the raw material and plaster of Paris. The increase of CaO and SO_3 may have resulted from reaction of the other silicate minerals and capturing of calcium from sodium calcium magnesium silicate to form more CaO while the SO_3 also increase by the upgrading of SO_2 to form more of SO_3 and their combination to form more gypsum.

There is increase of SiO_2, Al_2O_3, MnO, MgO, K_2O, SrO while there is decrease of FeO, CaO, Na_2O TiO_2, SO_3, ZnO, Cr_2O_3 and P_2O_3 as observed from gypsum obtained from Dange. The decrease of CaO and SO_3 led to the disappearance of ($CaSO.2H_2O$) gypsum in the calcined product (plaster of Paris) as well as SiO_2. This may have resulted from reaction e.g. dehydration and evaporation of sulphur oxide leaving SiO_2 and CaO to be taken up by the other silicate minerals. The general decrease of contents of the other elements oxide is simply as a result of de-oxidation and dehydration during calcination or evaporation of some of the volatile elements.

In the Weppa area, there is increase of all the elements analysed (SiO_2, Al_2O_3, MnO, MgO, CaO, Fe_2O_3, Na_2O, TiO_2, SO_3 and LO_1,) except for the decrease of Mgo, Na_2O. In the raw gypsum and calcined products – K_2O, ZnO, SrO, Cr_2O_3, Cl and P_2O_3 were found to e below detection limits. The mineral analysis indicates that gypsum calcium sulphate and ($CaSO.2H_2O$) change to Bassanite ($2CaSO_4.H_2O$) by losing part of its water. The lost water in the raw gypsum to give rise to Bassanite is recorded with water content of the plaster of Paris or calcium product in the chemical.

The summary of the major findings of this research work are presented below:

i. Fifteen elements were discovered from the analyses carried out on the gypsum samples obtained from Wurno and Dange, while seven elements were analysed from Weppa gypsum. After calcination fourteen elements were analysed, Wurno plaster and Dange plaster had fifteen elements, while eight elements were analysed in Weppa plaster.

ii. Calcium sulphate was found in Wurno and Dange gypsum and after calcination it disappeared in Dange plaster of Paris. Weppa sample was identified as gypsum, after calcination, it was found to be Bassanite ($CaSO_4$. ½ H_2O).

iii. There was no difference between the imported plaster and the locally processed plaster of Paris. Casted pieces separated from moulds between 20 to 25 minutes.

iv. The ratio mix of 100:75 and 100:85 plaster/water ratio mixes were found to be adequately porous and strong for slip casting. It was also found that 100:65 and 100:70 plaster/water ratio mix were suitable for press moulding production.

v. From the compressive strength test carried out, it was found that the strength of both plaster in moulds produced were strong enough for casting purposes when compared with the behaviour of the foreign plaster.

5.2 Conclusion

The gypsum obtained for the processing of plaster of Paris during this study was from three locations namely Wurno, Dange and Weppa areas of Sokoto and Edo States respectively. The processing of plaster was carried out at the Industrial Development Centre (IDC), Samaru, Zaria. The processing involved partial closing of the flue so as to be able to manipulate the temperature during calcinations process.

However, the obtained gypsum and produced plaster from these locations were submitted for various tests and it was observed that processed plaster from Wurno and Dange showed slight indications of shrinkage during the shrinkage test. This was more at the 95 and 100 plaster, water ratio mix. The X-ray diffraction also indicates presence of calcium sulphate (CaSO4) in the gypsum from Dange which disappeared after calcinations. All plaster processed were used for the production of moulds that can be adopted for ceramic production. However, the plaster from these locations was used for mould production and use. The moulds responded well to ceramic slip casting

5.3 General Recommendation

Gypsum and plaster of Paris is an important earth mineral useful in many areas like the building and construction industry, art, agriculture and the health. The recommendations are made.

1. It is of necessity that a consultancy be established such that some of these research findings could be translated into useable forms. This would allow findings generated translate to development of appropriate sustenance of minerals necessary in promoting further research and development in Section, Department and the University.
2. In the course of this study, there were areas of serious attention. This especially was in the area of electric power supply and test kilns in Ceramic section. This research is an eye opener to the fact that there is need for alternative power supply for research in the laboratories.
3. There is absence of postgraduate work space in the Ceramic section of Department of Industrial Design. Steps should be taken by the head of section to liaise with Head of Department of Industrial Design for Studio and work space for postgraduate students in the section.

4. There is urgent need for equipment to the Ceramic Sections in tertiary institutions in Nigeria, such as crushing and milling equipment which are necessary for minerals beneficiation. On this note, electric and test kilns should also be provided to enhance quality research work in ceramics.
5. Postgraduate studio and laboratory are as a matter of urgency in the Ceramic Section. This will enable conducive environment for accurate measurement of materials and general research work.
6. Ceramic students of all levels need introductory course in the sciences; especially geology and chemistry. This will enhance their understanding of geochemical and mineralogical properties of materials relevant ion ceramics.
7. Property testing standards and facilities should be made available to the Ceramic Section of tertiary institutions across Nigeria. In the same vain, analytical equipments such as X-ray fluorescence spectrometer (XRF) and X-ray diffraction (XRD) should be made available and accessible to research students from diverse disciplines. This will encourage world class findings.

5.4 Recommendation for Further Studies

Having concluded the study, the researcher would like to make the following recommendations for further studies:

1. Various temperature have been suggested for plaster calcination so as to be able to establish each plaster characteristic behaviour intern of what remains and what removed through calcination.
2. Literature reveals the availability of raw gypsum in Gombe State, Anambra State, and Benue State. Similar study should be encouraged in some of these locations so as to enrich the existing literature in the area of Nigerian gypsum and plaster processing.

3. Plaster has been produced from available gypsum from Wurno, Dange and Weppa areas of Sokoto and Edo States respectively. Effort should be made to use the discovery from the physio chemical characters to develop further or better application of these gypsum plaster.
4. There is also need for comparative analysis of other gypsum sites in Nigeria to be able to appreciate their peculiarities and differences in term of physical and chemical properties.

5.5 Contribution to knowledge

The study contributes to knowledge as highlighted below:

1. The study revealed that plaster of Paris when subjected to same temperature limit, time limit and temperature manipulation ranges in the kiln, a noticeable physical change in colour is observed, that is, it becomes lighter after calcinations.
2. Calcinations using ceramic bisque wares and crucibles produce plaster of Paris with slight tendency to set faster than those produced in aluminium or metal containers.
3. The locally beneficiated plaster of Paris revealed similar elemental properties with the imported ones from France and Mauritania (Calcium sulphate and Bassanite). This eventually produced a good water/plaster ratio from which a castable plaster of Paris that has the potentiality of contributing to indigenous economy against imported substitution and export.
4. After calcinations of Weppa, Dange and Wurno plaster of Paris and subjected to XRD test. It was observed that essential mineral component such calcium sulphate in Dange disappeared yet the final plaster of Paris from the material still produced a solid plaster of Paris that subsequently produce ceramic mould which ordinarily may have been thought to be a serious deficiency. This defect did not make the plaster of Paris impotent for ceramic production.

5. The study revealed that the chemical and mineralogical properties of the selected gypsum investigated in Nigeria on which bases further studies can be initiated for an improve plaster of Paris that is indigenous and competitive.
6. The study revealed a process technology that is not very expensive for the adoption of cottage level production which can easily be converted to wealth generation through poverty alleviation and provision of data base for practical information on all test analysis leading to the success of the study.

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htt://www.maragon.co.uk/plaster-guide.htm retrieved 23rd September, 2014: 1:30am

APPENDICES

Appendix 1

RAW GYPSUM OBTAINED FROM THE THREE STUDY AREAS



Plate XXII: Gypsum obtained from the three study areas

Appendix 2

WOODEN AND METAL MOULD USED IN THE STUDY



Plate XXIII: Wooden mould for absorbency, shrinkage and density test used in the study



Plate XXIV: Metal mould for compressive strength test used in the study

APPENDIX 3

XRF (X-Ray Fluorescence Spectrometer)



Plate XXV: Oxford X-Supreme 8000 Machine for XRF Analysis

Source: Singer and Singer (1971).

Appendix 4

PRODUCTION OF MOULD AND SLIP CASTING



Plate XXVI: Production of two-piece mould



Plate XXVII: Slip casting of two-piece mould

Appendix 5

ART WORK AND PRODUCTION OF ONE PIECE MOULD



Plate XXVIII: Art work for one piece mould being examined by Professor Ahuwan



Plate XXIX: Production of one-piece mould for the study

Appendix 6

WEIGHING OF PLASTER FOR TEST PIECES



Plate XXX: Weighing of plaster for test tiles being examined by Professor Suleiman

Appendix 7

GREEN AND BISQUE WARES FROM PROCESSED PLASTER



Plate XXXI: Finished green wares produced from moulds of processed plaster



Plate XXXII: Freshly bisque wares from moulds in the oven

Appendix 8

RESULT OF GLAZED WORKS



Plate XXXIII: A sample of glazed vase



Plate XXXIV: A sample of glazed vase

Appendix 9

RESULT OF GLAZED WORKS



Plate XXXV: Samples of glazed vases



Plate XXXVI: Samples of glazed vases

Appendix 10

RESULTS OF GLAZED WORKS



Plate XXXVII: Samples of glazed bowls



Plate XXXVIII: A collection of glazed bowls and vases

Appendix 11

DEPARTMENT OF INDUSTRIAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA - NIGERIA
Website: www.industrialdesign.org.ng E-mail: did@abu.edu.ng

Vice - Chancellor: Professor Abdullahi Mustapha, B. Sc (Hons) Pharm (ABU) Ph.D (London), FPSN
Head of Department: Dr. Salihu Maiwada, B.A., M.A., Ph.D(ABU) MSNA, MATTN

Tel: 08028185081
069879415

12th February, 2014

The Head
Department of Chemistry
Ahmadu Bello University
Zaria

Dear Sir/Madam,

TO WHOM IT MAY CONCERN

The bearer of this letter is Grace K. Soyinka a Ph.D Student of the Department of Industrial Design, Ahmadu Bello University, Zaria. She is presently set to conduct some tests in regards to her study.

Please kindly assist to enable her use facilities of your Lab to achieve results of tests necessary for her research work. Thanking you in anticipation.

Yours faithfully,

Prof. U. A. Sullayman

Sections: Basic Design • Ceramic Design • Graphics Design • Textile Design • Glass Technology

Appendix 12

DEPARTMENT OF INDUSTRIAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA - NIGERIA
Website: www.industrialdesign.org.ng E-mail: did@abu.edu.ng

Vice - Chancellor: Professor Abdullahi Mustapha, B. Sc (Hons) Pharm (ABU) Ph.D (London), FPSN
Head of Department: Dr. Salihu Maiwada, B.A., M.A., Ph.D(ABU) MSNA, MATTN

Tel: 08028185081
069879415

12th February, 2014

Director
Industrial Development Centre (IDC)
Zaria

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Prof. U. A. Sullayman

Sections: Basic Design • Ceramic Design • Graphics Design • Textile Design • Glass Technology

Appendix 13

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Website: www.industrialdesign.org.ng E-mail: did@abu.edu.ng

Vice - Chancellor: Professor Abdullahi Mustapha, B. Sc (Hons) Pharm (ABU) Ph.D (London), FPSN
Head of Department: Dr. Salihu Maiwada, B.A., M.A., Ph.D(ABU) MSNA, MATTN

Tel: 08028185081
069879415

12th February, 2014

Director General
National Steel Council
Malali
Kaduna

Dear Sir/Madam,

TO WHOM IT MAY CONCERN

The bearer of this letter is Grace K. Soyinka a Ph.D Student of the Department of Industrial Design, Ahmadu Bello University, Zaria. She is presently set to conduct some tests in regards to her study.

Please kindly assist to enable her use facilities of your Lab to achieve results of tests necessary for her research work. Thanking you in anticipation.

Yours faithfully,

Prof. U. A. Sullayman

Sections: Basic Design • Ceramic Design • Graphics Design • Textile Design • Glass Technology

Appendix 14

NIGERIAN GEOLOGICAL SURVEY AGENCY
NATIONAL GEOSCIENCES RESEARCH LABORATORY (NGRL), KADUNA

No. 1 & 2 Aliyu Makama Road,
Barnawa, Kaduna
P.MB 2007, Kaduna South.

E-mail: ngsalabs@ngsa-ng.org
Web Site: www.ngsa-ng.org

NGRL/OP/5334/2705

2nd June, 2014

Grace K. Soyinka,
Dept. of Industrial Design,
ABU,
Zaria.

LABORATORY REPORT NO 2705
RESULT OF ANALYSIS

I am directed to forward our Laboratory Report No 2705. This is in response to your request for Chemical Analysis of one sample using XRD and XRF.

Please acknowledge receipt.

P. B. Otiono
Assistant Director (Lab)

Appendix 15

WURNO GYPSUM XRF (Sample 1)

SAMPLE: WURNO SAMPLE 1

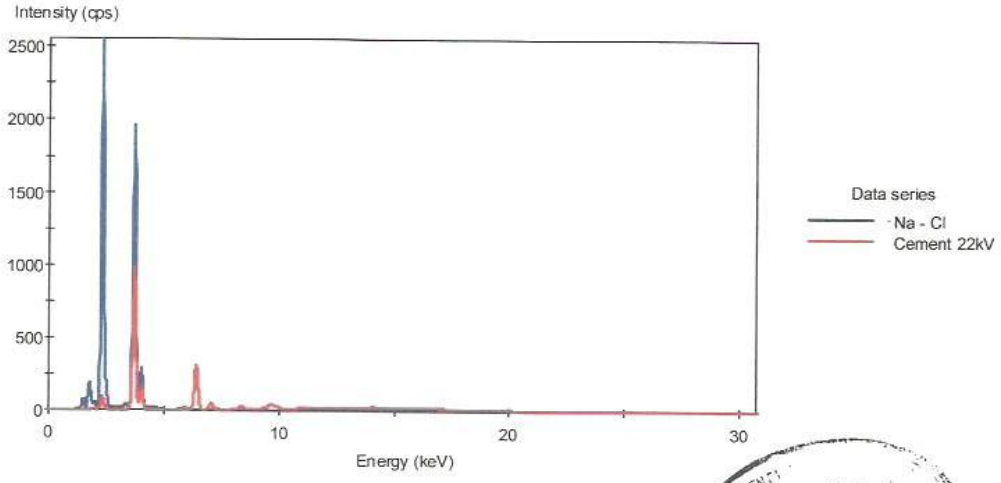
ANALYSED: 17/02/2014 4:20:54 PM

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	1.203 Wt %
MgO	0.809 Wt %
Al2O3	2.762 Wt %
SiO2	8.010 Wt %
P2O5	0.681 Wt %
SO3	41.194 Wt %
Cl	0.055 Wt %
K2O	0.131 Wt %
CaO	42.802 Wt %
TiO2	0.109 Wt %
Cr2O3	0.002 Wt %
Mn2O3	0.163 Wt %
Fe2O3	2.046 Wt %
ZnO	0.000 Wt %
SrO	0.031 Wt %

Spectra for all conditions





Printed on 17/02/2014 4:40:07 PM

Appendix 16

WURNO GYPSUM XRF (Sample 2)

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

ANALYSED: 17/02/2014 4:27:07 PM

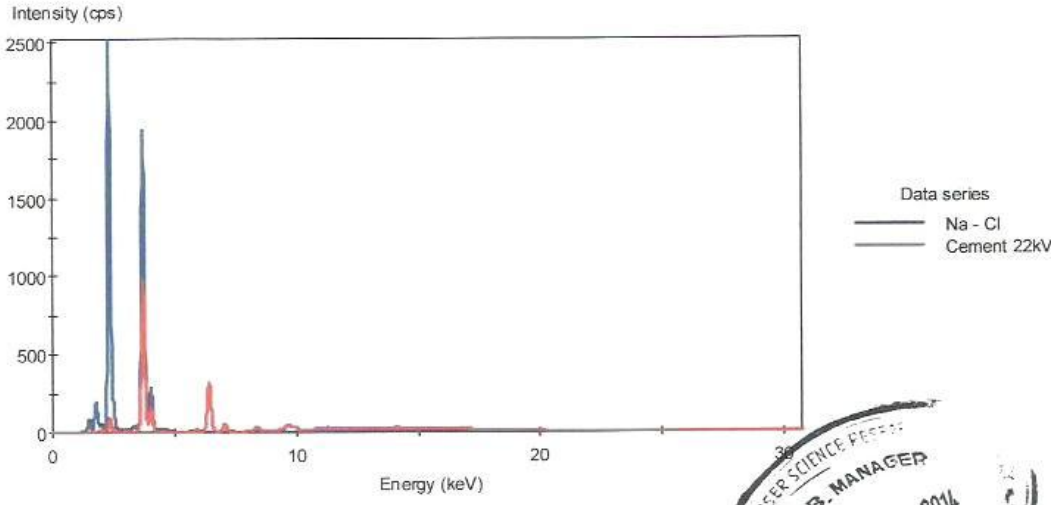
SAMPLE: WURNO SAMPLE 2

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	1.197 Wt %
MgO	0.797 Wt %
Al2O3	3.160 Wt %
SiO2	8.052 Wt %
P2O5	0.661 Wt %
SO3	40.939 Wt %
Cl	0.051 Wt %
K2O	0.142 Wt %
CaO	42.575 Wt %
TiO2	0.116 Wt %
Cr2O3	0.003 Wt %
Mn2O3	0.173 Wt %
Fe2O3	2.103 Wt %
ZnO	0.000 Wt %
SrO	0.031 Wt %

Spectra for all conditions





Printed on 17/02/2014 4:40:07 PM

Appendix 17

WURNO GYPSUM XRF (Sample 3)

ANALYSED: 17/02/2014 4:33:16 PM

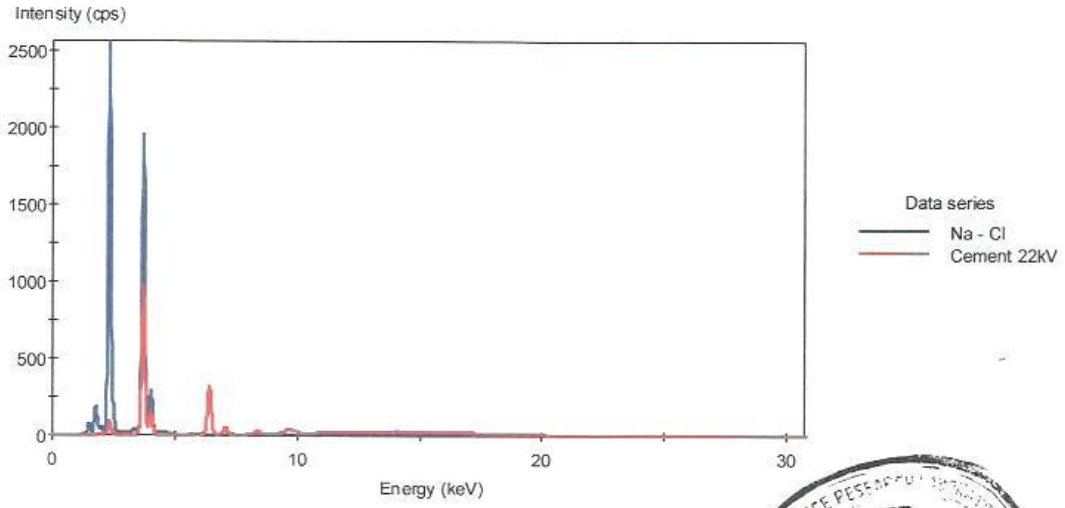
SAMPLE: WURNO SAMPLE 3

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	1.372 Wt %
MgO	0.823 Wt %
Al2O3	2.783 Wt %
SiO2	7.997 Wt %
P2O5	0.727 Wt %
SO3	41.241 Wt %
Cl	0.067 Wt %
K2O	0.138 Wt %
CaO	42.449 Wt %
TiO2	0.113 Wt %
Cr2O3	0.004 Wt %
Mn2O3	0.149 Wt %
Fe2O3	2.107 Wt %
ZnO	0.000 Wt %
SrO	0.031 Wt %

Spectra for all conditions





Printed on 17/02/2014 4:40:07 PM

Appendix 18

WURNO PLASTER XRF (Sample 1)

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

ANALYSED: 12/03/2014 1:16:47 PM

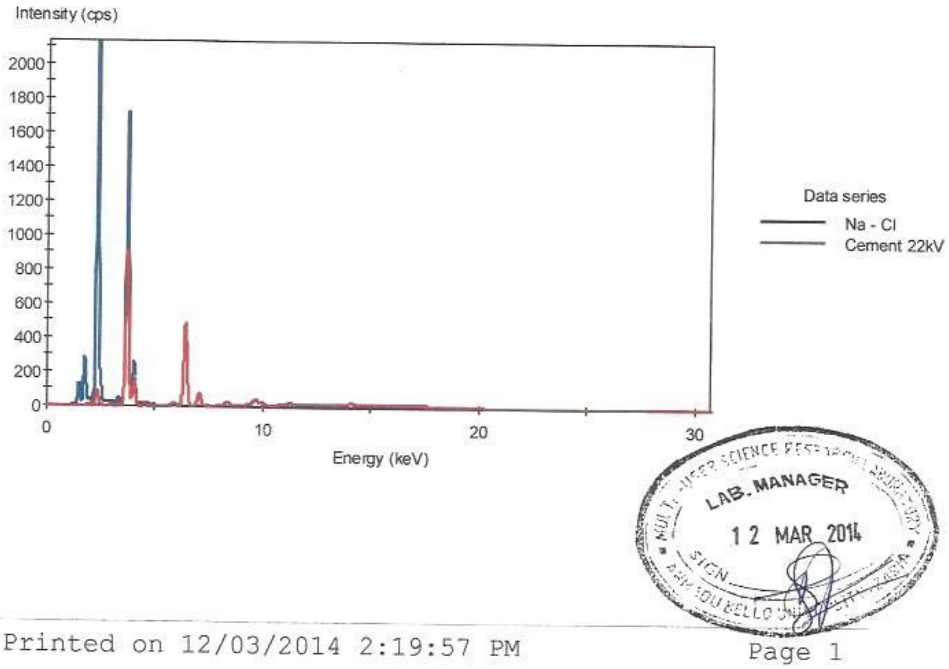
SAMPLE: WURNO 01

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	1.185 Wt %
MgO	1.307 Wt %
Al2O3	5.204 Wt %
SiO2	11.497 Wt %
P2O5	0.704 Wt %
SO3	36.229 Wt %
Cl	0.101 Wt %
K2O	0.222 Wt %
CaO	39.834 Wt %
TiO2	0.339 Wt %
Cr2O3	0.005 Wt %
Mn2O3	0.211 Wt %
Fe2O3	3.125 Wt %
ZnO	0.003 Wt %
SrO	0.035 Wt %

Spectra for all conditions



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12 MAR 2014
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Page 1

Appendix 19

WURNO PLASTER XRF (Sample 2)

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

ANALYSED: 12/03/2014 1:22:52 PM

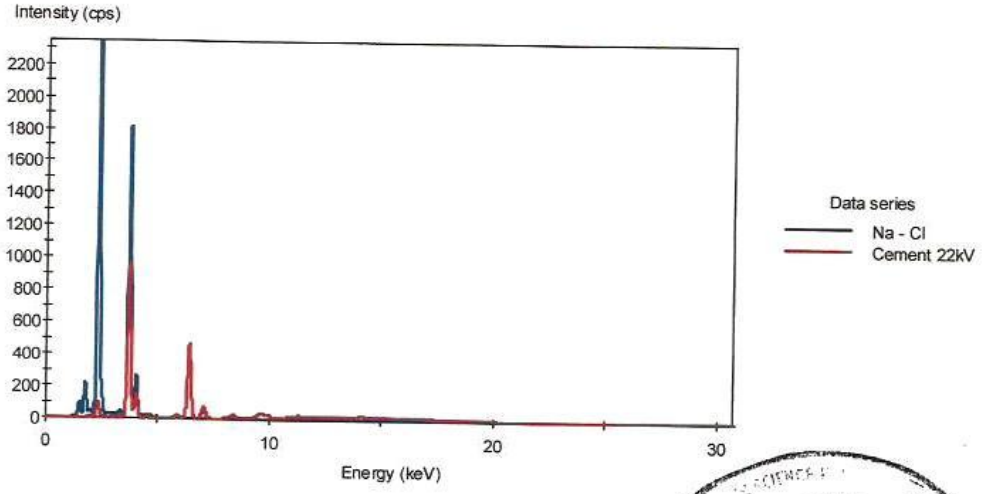
SAMPLE: WURNO 02

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	1.387 Wt %
MgO	1.176 Wt %
Al2O3	3.778 Wt %
SiO2	9.089 Wt %
P2O5	0.814 Wt %
SO3	38.632 Wt %
Cl	0.145 Wt %
K2O	0.182 Wt %
CaO	41.263 Wt %
TiO2	0.263 Wt %
Cr2O3	0.005 Wt %
Mn2O3	0.223 Wt %
Fe2O3	3.005 Wt %
ZnO	0.003 Wt %
SrO	0.035 Wt %

Spectra for all conditions



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12 MAR 2014
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Page 2

Appendix 20

WURNO PLASTER XRF (Sample 3)

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

ANALYSED: 12/03/2014 1:28:59 PM

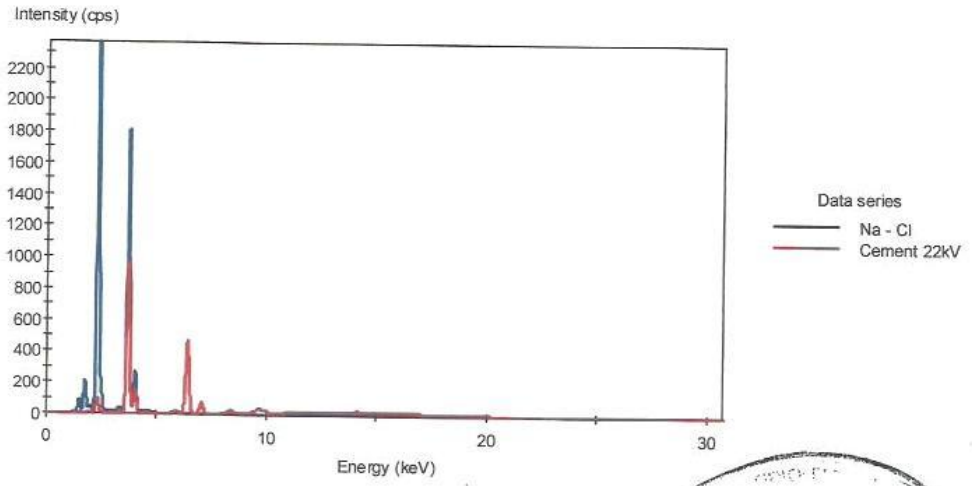
SAMPLE: WURNO 03

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	1.279 Wt %
MgO	0.959 Wt %
Al2O3	3.589 Wt %
SiO2	8.207 Wt %
P2O5	0.632 Wt %
SO3	39.062 Wt %
Cl	0.054 Wt %
K2O	0.182 Wt %
CaO	42.315 Wt %
TiO2	0.275 Wt %
Cr2O3	0.006 Wt %
Mn2O3	0.241 Wt %
Fe2O3	3.164 Wt %
ZnO	0.002 Wt %
SrO	0.033 Wt %

Spectra for all conditions



LAB. MANAGER
12 MAR 2014

Printed on 12/03/2014 2:19:58 PM

Page 3

Appendix 21

DANGE GYPSUM XRF (Sample 1)

GYPSUM XRF

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

ANALYSED: 17/02/2014 4:02:36 PM

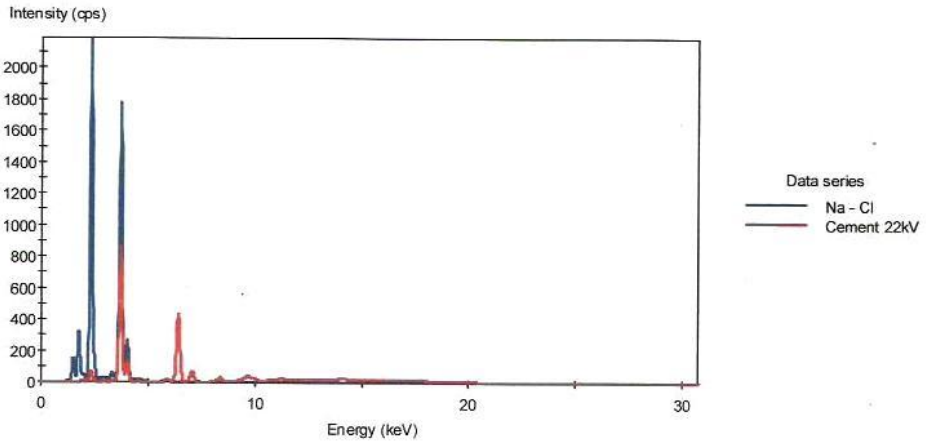
SAMPLE: DANGE SAMPLE 1

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	1.051 Wt %
MgO	1.286 Wt %
Al2O3	6.021 Wt %
SiO2	13.146 Wt %
P2O5	0.578 Wt %
SO3	36.900 Wt %
Cl	0.052 Wt %
K2O	0.251 Wt %
CaO	37.548 Wt %
TiO2	0.245 Wt %
Cr2O3	0.006 Wt %
Mn2O3	0.193 Wt %
Fe2O3	2.687 Wt %
ZnO	0.002 Wt %
SrO	0.034 Wt %

Spectra for all conditions





Appendix 22

DANGE GYPSUM XRF (Sample 2)

SAMPLE: DANGE SAMPLE 2

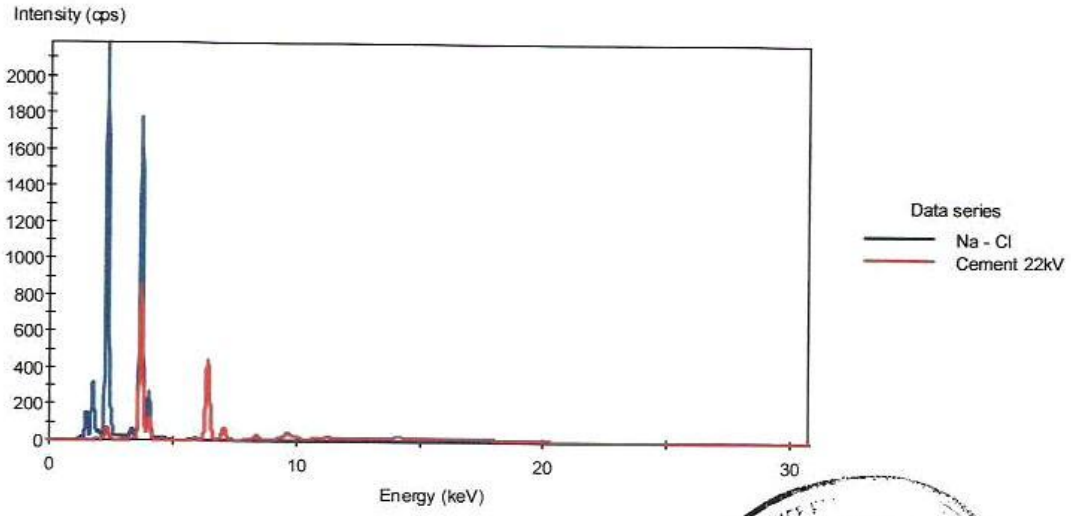
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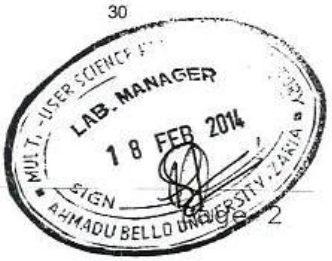
Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	1.354 Wt %
MgO	1.882 Wt %
Al2O3	5.932 Wt %
SiO2	12.921 Wt %
P2O5	0.649 Wt %
SO3	36.576 Wt %
Cl	0.077 Wt %
K2O	0.267 Wt %
CaO	37.211 Wt %
TiO2	0.252 Wt %
Cr2O3	0.004 Wt %
Mn2O3	0.145 Wt %
Fe2O3	2.694 Wt %
ZnO	0.002 Wt %
SrO	0.033 Wt %

Spectra for all conditions





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Page 2

Appendix 23

DANGE GYPSUM XRF (Sample 3)

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

ANALYSED: 17/02/2014 4:14:44 PM

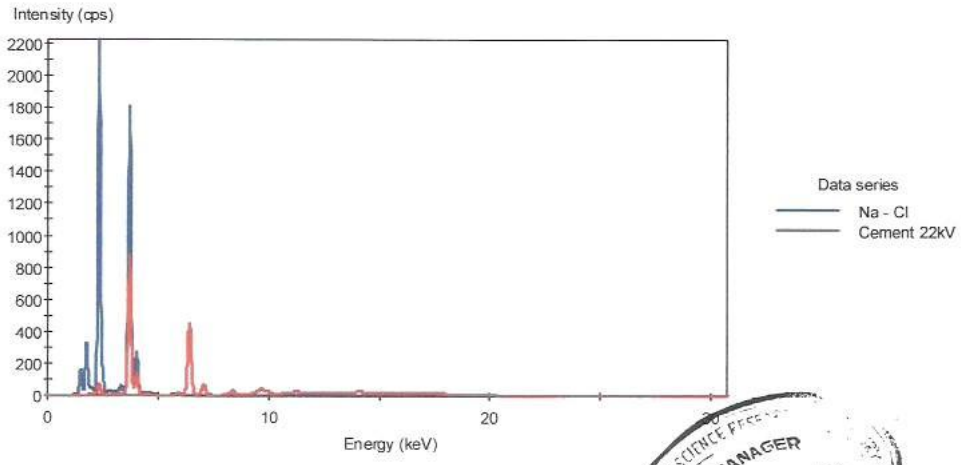
SAMPLE: DANGE SAMPLE3

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	0.985 Wt %
MgO	1.220 Wt %
Al2O3	6.061 Wt %
SiO2	13.075 Wt %
P2O5	0.571 Wt %
SO3	36.930 Wt %
Cl	0.051 Wt %
K2O	0.261 Wt %
CaO	37.593 Wt %
TiO2	0.273 Wt %
Cr2O3	0.005 Wt %
Mn2O3	0.171 Wt %
Fe2O3	2.770 Wt %
ZnO	0.002 Wt %
SrO	0.033 Wt %

Spectra for all conditions





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Page 3

Appendix 24

WEPPA GYPSUM XRF

LABORATORY REPORT NO 2706
RESULT OF ANALSYSIS

SAMPLE ID : ***Gypsum from WEPPA, Agenebode, Edo State***

OXIDE	%
SiO2	0.06
Al2O3	0.42
SO3	46.43
CaO	32.4
TiO2	0.013
MnO	0.023
Fe2O3	0.12
MgO	0.04
Na2O	0.033
LOI	20.45

Analyst: ***P. B. Otiono***
Assistant Director (Lab)

Appendix 25

WEPPA PLASTER XRF

LABORATORY REPORT NO 2723

RESULT OF ANALYSIS

OXIDE %

WEPPA PLASTER

COMPOSTION	MEASURED VALUE	DETECTION LIMIT
SiO_2	1.60	0.01
CaO	40.59	0.006
SO_3	46.7	0.02
Al_2O_3	0.96	0.12
TiO_2	0.05	0.005
MnO	0.061	0.002
Fe_2O_3	0.258	0.004
Combined (H_2O)	**9.78**	

Analyst: ***P.B.Otiono***

Assistant Director (Lab)

Appendix 26

WURNO GYPSUM XRD

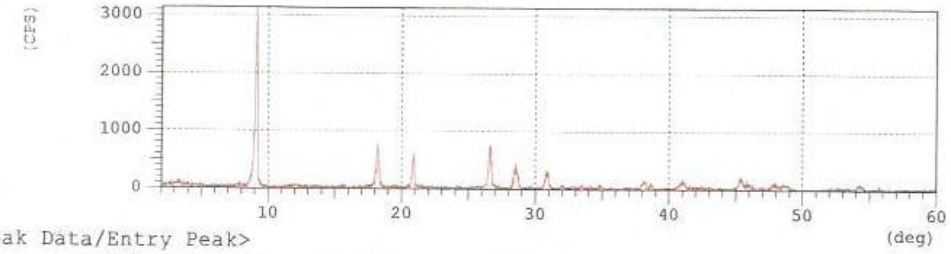
XRD

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

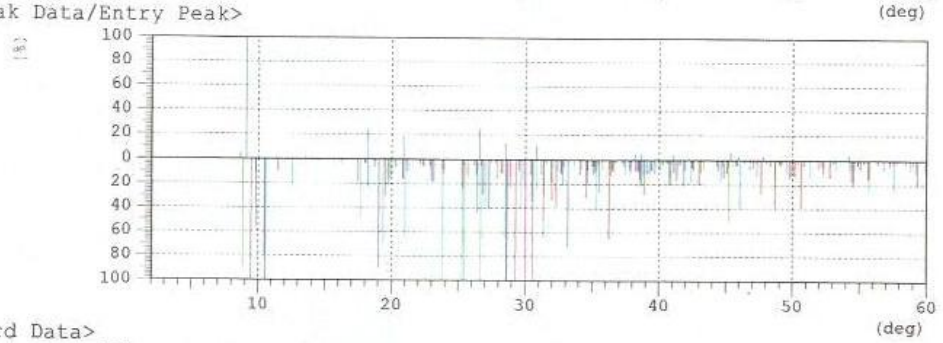
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File Name : WURNO-GYPSUM.PSE
Sample Name :
Comment : WHITISH
Date & Time : 03-21-14 06:38:33

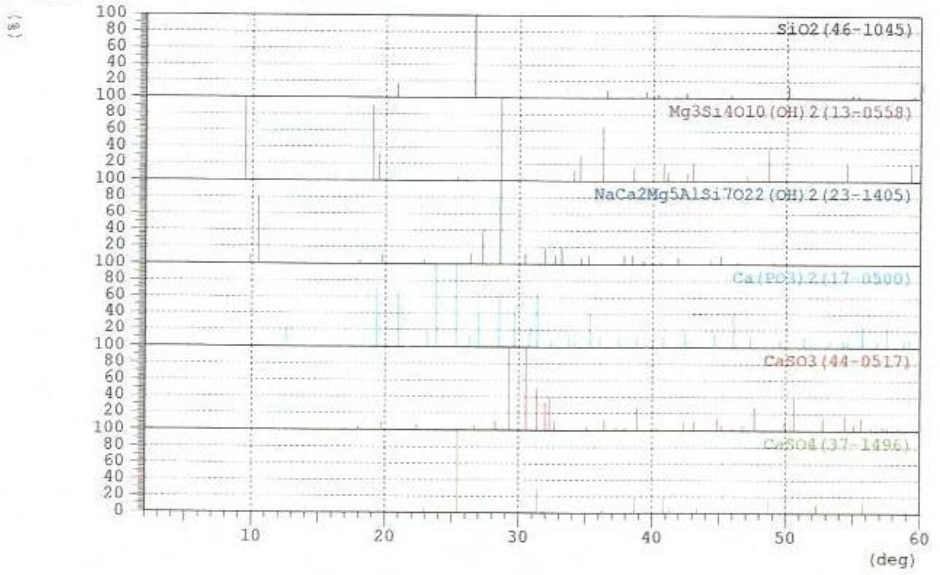
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Appendix 27

WURNO PLASTER XRD

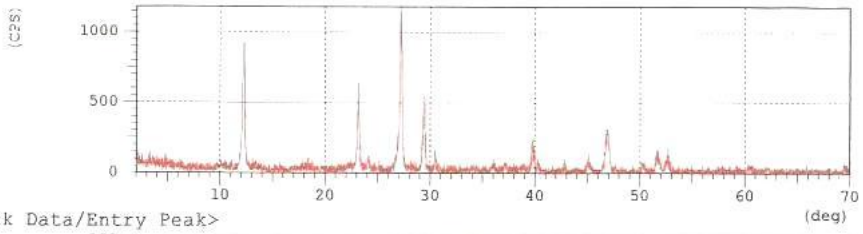
XRD

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

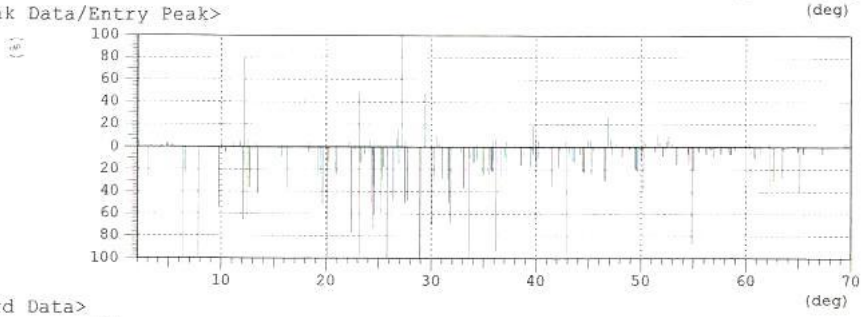
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File Name : WURNO-PLASTER.PSE
Sample Name :
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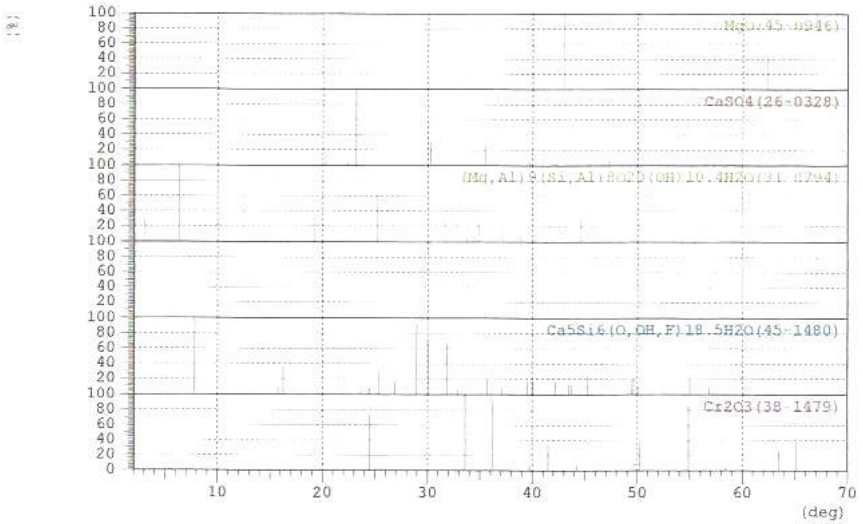
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Appendix 28

DANGE GYPSUM XRD

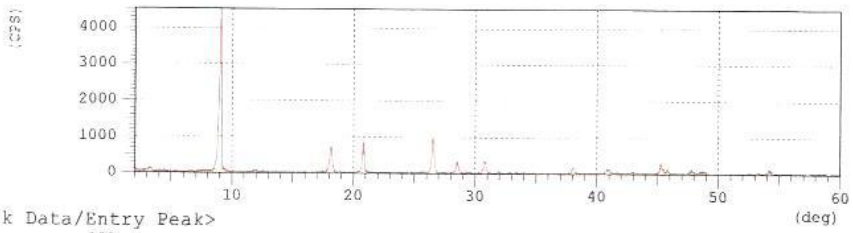
XRD

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

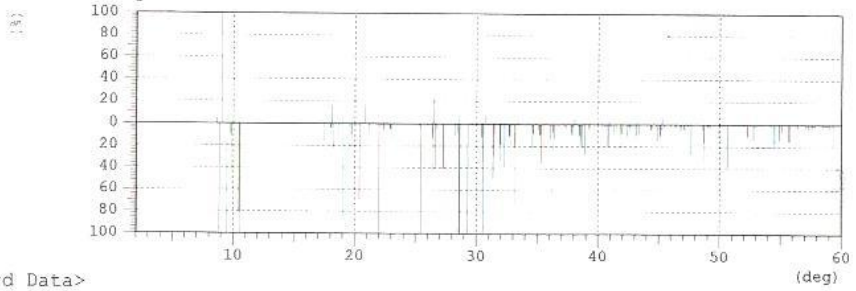
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File Name : DANGE-GYPSUM.PKR
Sample Name :
Comment : WHITISH
Date & Time : 03-21-14 06:49:53

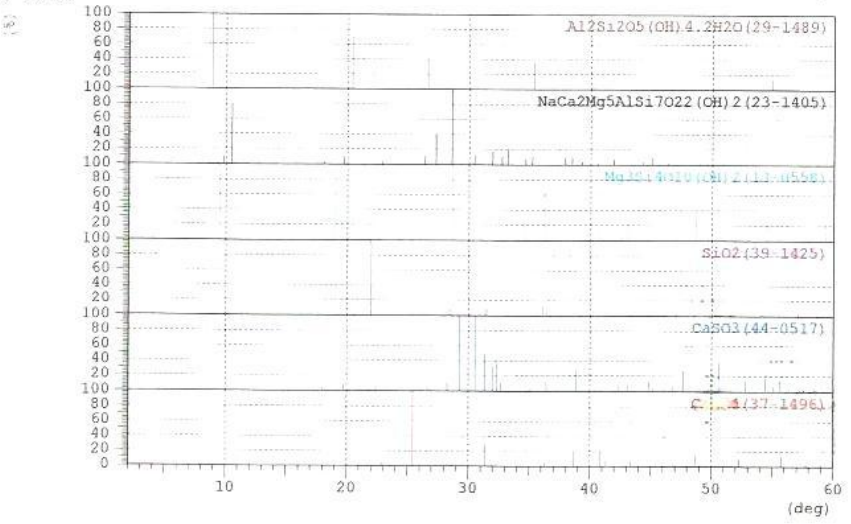
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Appendix 29

DANGE PLASTER XRD

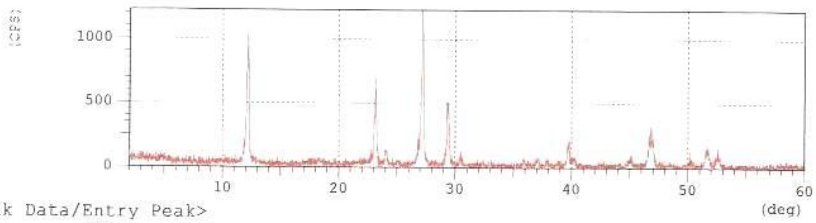
XRD

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

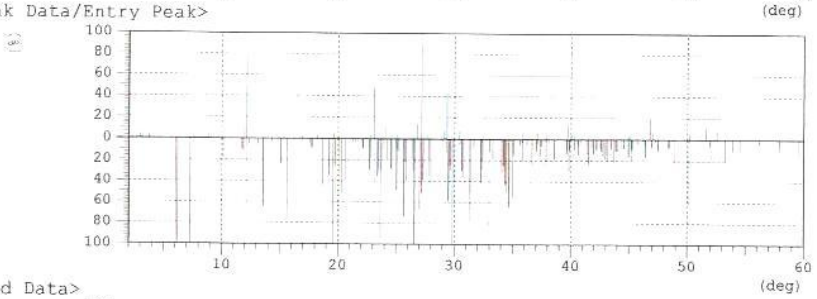
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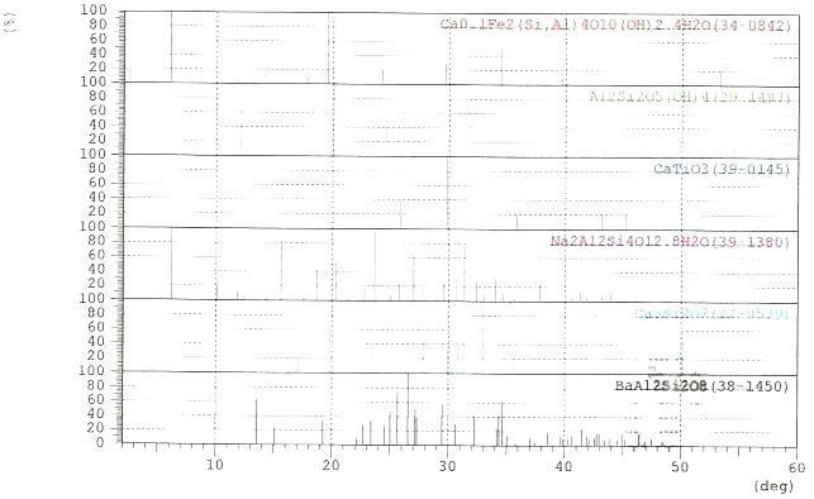
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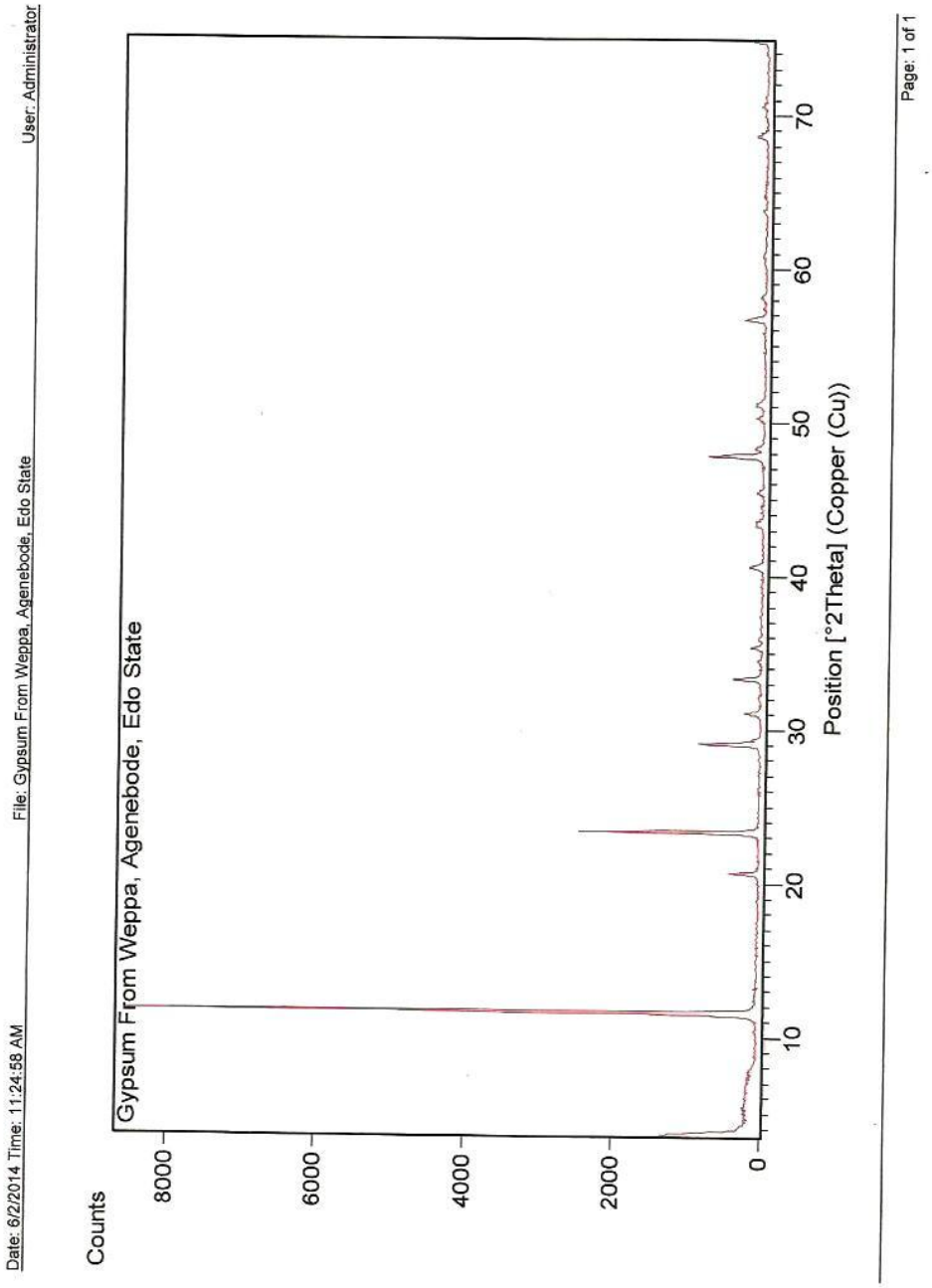


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Appendix 30

WEPPA GYPSUM XRD



Appendix 31

WEPPA PLASTER XRD

