THE USE OF RICE HUSK POWDER IN COMPOSITES CEMENT SYSTEM

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DECLARATION

I declare that the work in the thesis entitled the use of rice husk powder in composites cement system; has been performed by me in the Department of Industrial Design under the supervision of Mr. C.M. Gonah and Prof. A. M. Ahuwan. The information derived from literature has been duly acknowledged in the text and a list of references provided. No part of this thesis was previously presented for another degree or diploma at any university.

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DEDICATION

This is dedicated to my parents Hajiya Halima Bashir Ismail and Mal. Ibrahim Bello Ringim

CERTIFICATION

This thesis entitled THE USE OF RICE HUSK POWDER IN COMPOSITES CEMENT SYSTEM by Bello, Hadi Ibrahim meets the regulations governing the award of the degree of masters of Arts of Ahmadu Bello University, Zaria and is approved for its contribution to knowledge and literary presentation.

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ABSTRACT

High cost of cement has caused serious impact on housing delivery due to increase in energy costs and high demand which attracted many research interests in sourcing other alternatives cheaper materials. The use of rice husk powder in composites cement system was investigated. Paste of neat and blended Portland cement with 5%, 10%. 15%, 20%, 25%, 30%, 35%, 40%, 45%, 50%, and 55% rice husk powder were cured in air under the same temperature and humidity condition. Compressive strength of the various mix proportions at 28 days were determined using standard compressive strengths testing machine. The hydration products of the high percentage replacement composite cement paste*s* were identified by x-ray diffraction analysis and compared with those made from 100% ordinary Portland cement paste*s*. Results indicate that only lower percentage replacement level such as 5% and*10%* rice husk powder contained the required amount*s* of silica which aided the hydration process producing samples with compressive strength*s of* 19.72N/mm^2 and11.57N/mm^{2}when compared with control sample with 33.33N/mm^2. However, result of the x-ray diffraction analysis showed that the observed hydration products were mostly as expected due to the high replacement levels but the degree to which crystal phases were identified in one of the samples was unusual. The unusual behavior may be due to the fact that the calcium hydroxide initially formed was consumed during the curing period confirming the important pozzolanic reactions of the rice husk powder at such high replacement.

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ABBREVIATIONS

A.S.T.M = American Society for Testing Materials.

CE = Cured on exposure to weather condition.

CN = Cured normally.

OPC = Ordinary Portland Cement.

RCH = Rice Husk.

RHA= Rice Husk Ash.

XRD = X-Ray Diffraction.

J.C.P.D.S = Joint Committee on Powdered Diffraction Standards.

I.C.D.D = International Centre for Diffraction Standard.

OPERATIONAL TERMS

i. **Cement**: A binding agent powdery mixture of clay and limestone that hardens when wetted.

ii **Cementitious Materials**: Any material to which a liquid may be added to form a paste having adhesive and cohesive properties and that subsequently will harden into a solid mass.

iii. **Compressive Strength**: The maximum compressive stress that a material is capable of developing based on original area of cross section.

vi. **Cement Mortar**: A plastic mixture consisting of one part of Portland cement, four parts of sand, and a small amount of lime, all blended in water.

v. **Composite Material**: A combination of two or more materials (reinforcing elements, fillers and composite matrix binder) differing in form of composition on a macro scale.

vi. **Concrete**: A composite material that consists essentially of a building medium within which are embedded particles or fragments of aggregates (maximum aggregate size 75mm or 0.2 in).

vii. **Clinker**: A final product of Portland cement production which consist of hard granular masses from 3 to 20mm in size.

viii. **Microstructure:** The structure of an object, organism, or material as revealed by a microscope at magnifications greater than 25x.

ix. **Pozzolana;** siliceous and aluminous material, which in itself possess little or no cementitious value but will, in a finely divided form and in the presence of

moisture react with calcium hydroxide to form compound possessing cementitous properties.

x. **Pozzolan cement:** cement produced by grinding Portland cement with a pozzolanic material or a mixture of pozzolanic material with hydrated lime.

xi. **Crystal:** is a solid body bounded by natural planner surfaces, generally called crystal faces that are the external expression of a regular internal arrangement of constituent atom or ion

NOMENCLATURE

C = CaO, S = SiO_2, Ŝ = SO_3, A = Al_2O_3, F = Fe_2O_3,

M = MgO and H = H_2O. Ć = CO_2

Formula	Name	Abbreviation
2CaO.SiO	Dicalcium Silicate	C_2S
3CaO.SiO	Tricalicium Silicate	C_3S
$3CaO.Al_2O_3$	Tricalcium Aluminate	C_3A
$4CaO.Al_2O_3.Fe_2O_3$	Tetracalcium Alumino Ferrite	C_4AF
MgO	Magnesium Oxide in free state	M

CHAPTER 1

1.0 Introduction

Portland cement is the essential binding agent in cement composite systems, which in turn is the most commonly used construction material worldwide due to its many advantages including lower relative price, durability and other properties. (Escalante and Sharp,2004). Its cost is increasing due to the increase in energy cost and high demand of the product. However, to meet this demand other alternative cheaper materials are being sourced. (Escalante and Sharp,2004).

Materials of natural origin such as volcanic ash (VA) or industrial by product, like granulated blast furnace slag (GBFS) and pulverized fuel ash (PFA) have been widely used as partial replacement of Portland cement in concrete construction (Escalante and Sharp, 2004). Similarly, an agricultural waste rice husk has been described as a pozzolana with promising cementing properties when used with Portland cement. (Amjad and Abdul, 2004).

The advantages of these replacement materials are improved technical properties, lower cost and a reduction of waste accumulation. For instance, replacement of 5% of the aforementioned materials can provide a decrease of about $75x10^6$ tons of CO_2. (Escalante and Sharp, 2004).

The idea of adding pozzolana to Portland cement or Portland cement concrete is widely practiced because it does not only reduce cost and conserves energy, but decreases the heat of hydration, increases compressive strength and durability. (Amjad and Abdul, 2004).

The introduction of replacement materials in Portland cement produces additional complexity to the chemical reactions developed during hydration of the composite cements. (Escalante and Sharp, 2004). It has been observed that both latent hydraulic and pozzolanic materials participate in the overall hydration process and in the development of microstructure. The dominant product of these reactions is C - S - H gel, which is principally responsible for the mechanical properties of the hydrated cement. C – S – H gel is generated by the interaction of the replacement materials with Portlandite (CH), liberated during the hydration of the alite and belite present in the cement (Escalante and Sharp, 2004).It is evident from many studies that variation in curing condition affects pozzolanic reactivity of the composite cement. (Amjad and Abdul, 2004). However, little or no attention has been paid to investigate how composite cement systems react at fluctuating weather conditions of Zaria.

1.1 Statement of the Problem

Cement consumption is generally considered as an indicator of the growth of the Gross National product (GNP) of a country. In Nigeria, it is estimated that the demand for cement is about 18 million tones annually used mainly in the construction and building industries. (Dadu,2008). While local production for the past 10 years is between 2.5 to

6.5 million tones annually. However, to meet this demand an alternative and cheaper material is desirable.

Rice-husk, a pozzolanic materials and agricultural waste are land filled and burnt in open piles which is not environmentally friendly. An easy and efficient way of utilizing this waste is by using it with cement to serve the construction and building industries.

1.2 Aim of the Research

The study aims at investigating the compressive strength and identifying the crystal phases of the composites.

1.3 Objectives

1. Determining the compressive strength of the composite by using standard compressive strength testing machine.
2. Identifying the crystal phases of the hydration products by x-ray diffraction analysis.

1.4 Scope

The scope of the study embraces the crystal phase identification of the composite cement and the determination of its compressive strength.

1.5 Significance of the Research

The result obtained from this research will advance and possibly open new frontiers of knowledge in the production of cement composites by utilizing local raw materials. If implemented, it would amount to saving cost on procurement of construction materials like cement and also will help in controlling environmental problems that are associated with the

burning of waste in rice production.

1.6 Delimitation of the Study

The investigation is delimited to production of composites from mixture of ordinary Portland cement with rice husk powder.

1.7 Limitation of the Study

Some of the problems encountered in carrying out the study were lack of some research facilities like;

1. Differential Thermal Analyzer: For examining the heat change during hydration of the cement paste.
2. Scanning Electron Microscope: This is useful in the study and characterization of the composite.

1.8 Research Question/Hypothesis

1. What are the crystalline phases of the composites and their relative proportion?
2. What are the measures of the compressive strength of the composites?

1.9 Basic Assumptions

a. That the crystalline phases and their relative proportions would comply with the International Centre for Diffraction Data (ICDD) standards.

b. That the compressive strength achievable through mixing method would yield satisfactory results.

CHAPTER 2

2.0 Literature Review

2.1 Origin of Cement

Cement can be described as a material with adhesive and cohesive properties which make it capable of bonding mineral fragments into a compact whole. This definition embraces a large variety of cementing materials, but for constructional purpose, the meaning of the term "cement" is restricted to the bonding materials used with stones, sand, bricks, building blocks, etc. (Neville,1981).

The use of cement dates back to antiquity and one can only speculate as to its discovery. Cement was used by the Egyptians in constructing the Pyramids while the Greeks and Romans used volcanic tuff (paste) mixed with lime for cement. This was the first concrete history. (Austin,1984). The active silica and alumina in the ash combined with lime to produce what became known as pozzolanic cement from the name of the village Pozzuoli, near Vesuvius, where the Volcanic ash was first found. The name "Pozzolanic cement" is used to this day to describe cements obtained simply by grinding of natural materials at normal temperature. A number of the Roman structures in which masonry was bonded by mortar, such as the coliseum in Rome, the point du Gard near Nimes and concrete structure such as Pantheon in Rome have survived to this day, with the cementatious material still hard and firm. (Lawali, 2007) In the ruins at Pompeii, the mortar is often less weathered than the rather soft stone. The middle Ages brought a general decline in the quality and the use of cement, and it was only in the eighteenth century that an advance in the knowledge of cements occurred. John Smeaton,

commissioned in 1756 to rebuild the Eddystane Lighthouse, off the Cornish coast, found that the best mortar was produced when Pozzolana was mixed with limestone containing a considerable proportion of clay matter. By recognizing the role of the clay hitherto considered undesirable, Smeaton was the first to understand the chemical properties of hydraulic lime that is a material obtained by burning a mixture of lime and clay. (Lawali, 2007).

There followed a development of other hydraulic cements, such as the 'Roman Cement' obtained by James Parker through Calcining nodules of argillaceous limestone, culminating in the patent for "Portland Cement" taken out by Joseph Aspdin, a Leeds Bricklayer, stone mason and builder, in 1824. The cement was prepared by heating mixture of clay and hard limestone in a furnace until all the CO_2 had been driven off. This temperature was much lower than that necessary for clinkering.The prototype of modern cement was made in 1845 by Isaac Johnson, who burnt a mixture of clay and chalk until clinkering, so that the reactions necessary for the formation of strongly cementitious compounds took place. Portland cement recognizes addition of gypsum after burning but nowadays other materials may also be added or blended. (Lawali, 2007).

2.2 Ceramics and Glasses

The term "ceramics" is applied to a range of inorganic materials of wide uses and are non-metallic and in most cases have been produced at high temperature. The word "ceramic" is derived from the Greek "Keramos" or "potter clay" though the group of materials now so described includes glass products; cements and plastics: some abrasive

and cutting tools materials, building materials, refractory linings for furnaces, porcelain and other refectory coatings for metals. (Higgins,1998).From the point of view of composition and structure ceramics can be classified into four main groups namely: Amorphous ceramic, Crystalline ceramics, Bonded ceramics and Cements.

Amorphous ceramic are substances referred to generally as “glasses”. They include those as “Obsidian” which occur naturally and man-made glasses used for the manufacture of bottles, windows and lenses Crystalline ceramics may be single-phase material like magnesium oxide or aluminium oxide, or various mixtures of materials. In addition some carbides and nitrides belong to this group. Bonded ceramics are materials in which individual crystals are bonded together by a glassy matrix as in a large number of products derived from clay. Cements are crystalline but some may contain both crystalline and amorphous phases. (Higgins,1998).

2.3 Ordinary Portland Cements

The most important and most costly material in composite cement system is the cementing agent, Portland cement. It is not a trade name but the name of a type of cementing materials, in a similar way that asphalt, epoxy, lime, etc are cementing materials. It was called Portland cement because it resembled cement obtained by grinding natural rocks found on the Isle of Portland near England. (Bye, 1990).Gonah and Milestone (2005) explained that Cement is a binder that chemically binds solid materials together. The term “Cement” here connotes Portland type of cement. It sets under water thus, referred to as hydraulic. When it is employed in building construction, nuclear waste management and petroleum (Oil and Gas) industry is simply termed cement. Bye

(1990) asserted that the mineral used in cement manufacture cover the complete range of geological eras including all types of limestone and the metamorphosed form of marble. He further stated that the difference of most practical interest is crystal size, porosity and hardness. In cement making overall chemical composition is the main consideration. The clays used generally contain a mixture of two white and minerals of the Kaolinite group and three layer minerals of the illite montmorrilonoid (swelling clays) groups. Free silica (usually quartz) may also be a major constituent.

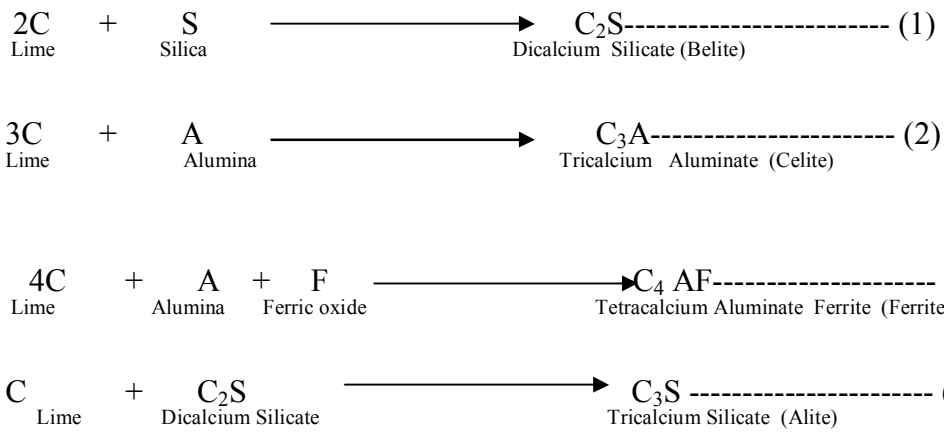
2.3.1 Difference Types of Portland Cements

Portland cement is classified into types I, II, III, IV and V. However Portland cement is recognized worldwide and generally designated as type I cement. Over 90% cement used in Nigeria, the United Kingdom, the United States, and other parts of the world fall into this category. Ordinary Portland cement (OPC) is the most ideal and preferred because of its multipurpose application. However, in spite of all the advantages of OPC, it still has numerous limitations in service on exposure to some environments while special kind of Portland cement can be manufactured, it is common to use additional supplementary cement materials (SCM) to make a composite cement. (Gonah and Milestone, 2005).

2.3.2 The Manufacture of Ordinary Portland Cements

Austins (1984) reported that two types of materials are necessary for the production of Portland cement: a source of Calcium (Calcareous) such as limestone and chalk and a source of Silica (argillaceous) such as clay. He further explained that these raw materials when finely ground, mixed and burnt in a rotary kiln formed cement clinker. Various

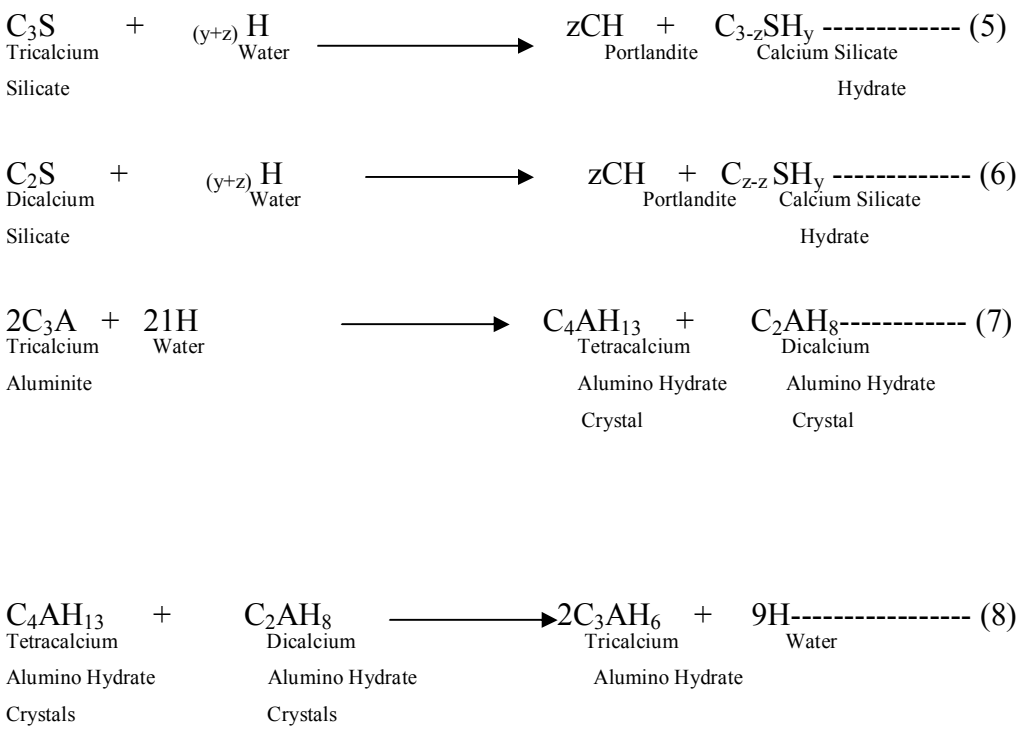
reactions, such as evaporation of water, evolution of carbon dioxide and reactions between lime and clay materials take place in the process. The chemical reactions in cement manufacture are summarized as follows: (Gonah and Milestone, 2005).



The manufacturing process involves the selection of an efficient engineering method for crushing, grinding, blending and conveying of solids with their attendant heat treatments. Energy usage in the process is large and constantly monitored. (Bye, 1990).Two distinct processes are used in the production of clinker, wet and dry processes. In the wet process slurry of the finely divided raw materials is made and pumped into a long rotary Kiln. According to Bye (1990) and Austin (1984), the wet process was at one time widespread but rapid increases in fuel costs have accelerated its replacement since it involves the evaporation of water which constitutes 35% of the slurry. In the dry process the raw materials are prepared for pyroprocessing as a blend of finely ground powders. The initial heating to about 800^0C is then carried out in a pre-heater using the CO_2 evolved from the limestone and the hot combustion gases from the fuel, which is burnt in a relatively short rotary kiln.

2.3.3 Hydration of Portland Cements

Portland cement is hydraulic material that sets or hardens when mixed with water. Although many theories have been proposed to explain the setting and hardening of cement, it is generally agreed that hydration and hydrolysis are involved. The hydration products have very low solubility in water, if this were not true concrete in contact with water would be rapidly attacked. Much attention has been given to the heat evolved during the hydration of cement. The various compounds contribute to the heat of hardening (base on equal weights i.e. gram for gram) after 28 days as follows $C_3A > C_3S > C_4AF > C_2S$ (Austin, 1984) and (Neville, 1987). A complex series of hydration reaction of cement is shown in the following equations (Gonah and Milestone, 2005).



C_3A + $3C\hat{S}H_2$ + 25 – 26 H ⟶ $C_6A\hat{S}H_{31,32}$ ---------- (9)

Tricalcium Aluminate — Gypsum — Water — Ettringite

C_4AF + 13 H ⟶ C_4AFH_{13} ---------- (10)

Tetracalcium Aluminoferrite — Water — Complex Hydrated Product of Tetracalcium Alumino Ferrite

2.4 Composite Cement System

A composite material can be defined as a multiphase material that exhibits a significant proportion of the properties of both constituent phases such that a combination of properties is realized. According to this principle, combined action, better property combinations are fashioned by the judicious combination of two or more distinct materials (Callister, 1997).

Composite can be laminar, fibre-reinforced or particulate-reinforced. The latter fits composite cement systems which have two fundamental different forms: Ideal composite hard material and ideal composite soft material (Neville, 1987).

$E = (1-g)\, E_m + g\, E_p$ ---------- (11)

Ideal composite hard material has a continuous matrix of an elastic phase with a high modulus of elasticity, and embedded materials of lower modulus (Neville, 1987).

Whereas a composite soft material, consists of elastic particles with high modulus of elasticity, embedded in a continuous matrix phase with a lower modulus (Neville, 1987).

$E = [(1-g) / E_m + g/E_p$ ---------- (12)

Where E = modulus of elasticity of the composite material, E_m = modulus of elasticity of the particle phase, E_p= modulus of elasticity of the particle phase and g = fractional volume of particles. OPC as previously stated has multipurpose characteristic, but it is

also evident that there are limitations. Some of these shortcomings can be tackled by blending the OPC with one or more siliceous materials (Escalante and Sharp, 2004).The blends are known as composite cement systems. The major advantages of these combinations are improved durability, environmental benefits and positive economic contribution (Bye, 1990).

2.5 Pozzolana

Pozzolana is a natural or an artificial material containing silica in a reactive form. Amjad and Abdul (2004) reported a more formal definition which describes pozzolana as a siliceous or siliceous and aluminous materials which in itself possess little or no cementitious value but will, in a finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperature to form a compound possessing cementitous properties. It is essential that pozzolana be in a finely divided form as it is only then that silica can combine with calcium hydroxide to produce calcium silicates.

Some natural pozzolana, for example,volcanic ash, pumilite, diatomaceous earth, etc are reactive by nature while others need to be heat treated. The effect on pozzolana is variable. Some pozzolana improve their reactivity by calcinations in the temperature range of 500-1100^{0}C, depending on the mineralogical composition. Rice-husk burnt at 450^{0}C has been found to produce good pozzolanic properties. (Amjad and Abdul, 2005). In considering pozzolanas in general it is recommended that the silica has to be amorphous, since crystalline silica has very low reactivity. The pozzolanic activity of any

materials containing reactive silica can be determined by adding calcium oxide to the pozzolana with water free from carbon dioxide and measuring the alkalinity and calcium oxide content after a specified reaction time. Cook *et al*. (1976) reported that investigation carried out by Damer indicated that Rice husk ash (RHA) can be classified as a pozzolana on the basis of its reactivity measured in the above manner and that it conforms to the requirements of an ASTM C618 class N pozzolana.

2.6 Rice Husk

Rice milling generates a by-product known as rice husk which is disposed as waste material. During milling of paddy rice about 78% weight is received as rice, broken rice and bran while the rest 22% is received as husk .It was observed that the rice husk constitutes about one-fifth of weight of the total rice products. (Lawali, 2007). This figure however, vary, with variety of paddy, it age and moisture content and with the degree of wetting and polishing. Further investigations as regard husks are that they are valueless as food, not only because of their low nutritive value, but because their high silicon content which renders them harmful to the digestive and respiratory organs of the body. (Lawali, 2007).

Nigeria is a rice producing country, and the husk generated during milling is mostly, used as fuel in the boiler for processing paddy; producing energy through direct combustion and by gasification. Houston (1972) observed that the mode of disposal has frequently proved difficult because of the tough, woody, abrasive nature of the husks, their low nutritive properties, resistance to weathering, great bulk, and ash content. About 700,000

tonnes of rice husk is produced annually causing damage to the land and the surrounding areas in which it is dumped. (FAO,2002).

2.7 Mixing Methods for Concrete

As for all materials, the performance of concrete is determined by the microstructure which is determined by its composition, curing conditions, the mixing method and mixer conditions used to process the concrete. The mixing procedure includes the type of mixer, the order of introduction of materials in the mixer and the energy of mixing (duration and power). To control the workability or rheology of the fresh concrete, for example, it is important to control the processing method. (Ferraris, 2001).

To determine the mixing method best suited for a specific application, factors to be considered include location of the construction site (distance from the batching plant), the amount of concrete needed, construction schedule (volume of concrete needed per hour) and the cost. However, the main consideration is the quality of the concrete produced. This quality is determined by the performance of the concrete and by the homogeneity of the material after mixing and placement. The efficiency parameters of a mixer are affected by the order in which the various constituents of the concrete are introduced into the mixer, the type of mixer, and the mixing energy (Ferraris, 2001).

2.7.1 Types of Mixers

There are two main categories of mixer; batch mixers and continuous mixers. The first type of mixer produces concrete a batch at a time, while the second type produces

concrete at a continuous rate. The first type needs to be emptied completely after each mixing cycle and then reloaded with the materials for the next batch. In the second type the materials are continuously introduced at one end as the fresh concrete exits at the other end. Two main types of batch mixer can be distinguished by the orientation of the axis rotation; horizontal or vertical (pan mixers). The drum mixers have a drum with fixed blades, rotating around its axis, while the pan mixers may have either the blades or the pan rotating around the axis. Continuous mixers are usually non-tilting drums with screw type blades rotating in the middle of the drum. The drum is tilted down ward toward the discharge opening. The mixing time is determined by the slope of the drum (usually about 15^0). (Ferraris, 2001).

2.8 Curing Condition

Adequate curing is essential to bringing the concrete to the required strength and quality. The aim of curing is to promote the hydrations of the cementing material. This is accomplished by preventing moisture loss and, when necessary by controlling temperatures (Parker, 1997).The length of the curing time depends upon the properties desired and upon atmospheric conditions, such as temperature, humidity, and wind velocity. Short curing period are used in fabricating concrete products such as block or precast structural elements. Curing time is shortened by the use of elevated temperature. (Parker, 1997).

2.9 Microstructural Analysis

Ceramic materials are used today in nearly all areas of engineering. The properties of the materials are influenced significantly by their microstructures; therefore the quality of

these materials always must be carefully inspected. For this purpose, the microstructure is examined with different microscopic techniques such as transmitted or reflected light microscopy using polarized light, scanning election microscopy (S.E.M) or transmission Election Microscopy (TEM). (Schneider, 1991).

The study of microstructures is concerned essentially with the ability to distinguish between materials having different chemical composition, structure or orientation and to distinguish between details separated by only small distances. Techniques for revealing microstructural features are almost all based either on the response of their details to incident radiation or the quality and intensity of the radiation emitted by features when appropriately excited. (Schneider, 1991)

2.10 Compressive Strengths

The characteristic parameter for the load capacity of concrete is typically its compressive strength. In addition to that, knowledge of its tensile and flexural tensile strength is required. The compressive strength of concrete is taken as the maximum compressive load it can carry per unit area. Concrete strengths of up to 80N/mm^2 can be achieved by type of cement, mix proportions, method of compaction and curing conditions (Jackson and Dhir, 1999).

Concrete structures, except for road pavements are normally designed on the basis that concrete is capable of resisting only compressive, the tension being carried by steel reinforcement. In the United Kingdom a 150mm cube is commonly used for determining the compressive strength. The standard method described in BS 1881; part 116 requires that test specimens should be cured in water at 20$\pm$ 2^{0}C and crushed, by loading it at

constant rate of stress increase of between 12 and 24N/mm^2/Min immediately after it has been removed from the curing tank. However, the following factors influence the strength of concrete: Constituent materials, methods of preparation curing and test conditions. (Jackson and Dhir, 1999).

The determination of compressive strength assesses hydraulicity rather than the chemical reactivity. Nevertheless, there is usually correlation between an increase in the strength of a concrete or mortal and an increase in the degree of hydration of the cement (Bye, 1990).The tensile strength of concrete is important in the design of concrete roads and runaways. The tensile strength of concrete is usually taken to be about one-tenth of its compressive strength. (Jackson and Dhir,1999). This may vary, however, depending on the method used for measuring tensile strength and the type of concrete. In general the direct tensile strength and the split cylinder tensile strength vary from 5 to 13 percent and the flexural strength from 11 to 23 percent of the concrete cube compressive strength. (Jackson and Dhir, 1999).

2.11 X-Ray Diffraction Analysis

X-ray diffraction constitutes a powerful and readily available method for determining atomic arrangement in matter. This method depend upon the fact that x-ray wavelength of the order of 1 nanometer are readily available and that is the order of magnitude of atomic dimensions, when an x-ray beam fall on matter, scattered x-radiation is produced by all the atoms. These scattered waves spread out spherically from all the atoms in the sample, and the inference effect of the scattered radiation from the different atoms cause

the intensity of the scattered radiation to exhibit maxima and minima in various directions (Parker, 1997).

Here the beam of X-rays is made to impringe on the sample at an angle θ with a plane of atoms within it and the patial distribution of the scattered X-rays is measured because the wavelength (λ) of X-rays is of the order of interatomic plane distance d (tenth of nanometer) crystalline materials or least material with some order in its atomic arrangement diffracts the beam. The key condition for constructive interference of the reflected beam follows the Bragg's law:

$d \sin\theta = n\lambda, \quad n = 1, 2, \ldots\ldots\ldots\ldots\ldots (13)$ (Vijaya and Rangarajan, 2006).

Where

d = lattice spacing

θ = Braggs angle, the angle between the wave vector and the lattice plane.

n = is an integar, the order of the reflection

λ = wave length of the x

Some of the uses of X-ray diffractions are in crystallographic analysis of many materials such as metals, minerals, clay minerals and plastics, pharmaceuticals and semi conductors.

The X-ray diffraction can also be used to:

i. Differentiate between crystalline and amorphous materials.

ii. Determine the structure of crystalline materials (crystals axis, size and shape of the unit cell).

iii. Determine electron distribution within the atoms and throughout to unit cell.

iv. Determine the orientation of single crystals.

v. Determine the texture of polygrained materials

vi Identify crystalline phases and measure the relative proportions.

vii. Measure limits of solid solubility and to determine phase diagram;

viii .Measure strain and small grain size.

ix. Measure various kind of randomness disorder, and imperfection in crystals.

x. Determine radial distribution functions for amorphous solid and liquids. (Parker, 1997).

2.12 Crystal Structure and Chemical Bonds

According to the regularity with which the atoms or molecules are arranged in solids they may be broadly classified as crystalline and non-crystalline solids. There are other rare and special classes, viz liquid crystals and quasi crystals. The crystal structure of material is determined by the type of bonding between neighbouring atoms (Vijaya and Rangarajan, 2006). Crystals are classified according to the way in which the atoms or groups of atoms are arranged in three dimensional pattern, i.e. according to the three types of lattices. To describe a given type of lattice the term unit cell is used. A unit cell is defined as the fundamental block which, when repeated in the three directions, will generate the entire lattice. (Vijaya and Rangarajam, 2006).

2.13 Relevant Works on Rice Husk-Cement Composites

One of the earlier references of the potential of using RHA for making cement in United States of American was published by Hough and Barr (1956) and reported by Lawali (2007). The authors referred back to the successful making of building blocks using RHA mix with cement as early as 1923. Shah et al (1970) reported that a production plants was

set up in the Pakistan Punjab using a husk incinerator made from an oil drum. It was claimed that more than 4% carbon in the ash resulted in a loss of strength.

Rahman (2006) carried out an extensive study on the use of Rice husk ash in sandcrete blocks for masonary units. He worked on Different mix proportion of sand, cement and Rice husk ash. On comparing the compressive strength of the various mix proportions with British statutory minimum standard, it was found that the sandcrete blocks of 1:5 mortar mixes with 40% R.H.A (by weight of cement) could be used in both load and non-load bearing wall. Oyetola and Abdullahi (2009) investigated the compressive strength of some commercial sandcrete blocks in Minna, Nigeria. Preliminary analysis of the constituent materials of the ordinary Portland cement (OPC)rice husk hollow sandcrete blocks were conducted to confirm their suitability for block making. Test Results indicate that most commercial sandcrete blocks in Minna town were below standard and the compressive strength of the OPC/RHA sandcrete blocks increases with age at curing and decreases as the percentage of R.H.A content increases. Their study arrived at an optimum replacement level of 20%. Dashan and Kamang (1999) reported a study on some characteristic of Acha husk ash/ordinary Portland cement (AHA/OPC) concrete. Test results indicated that the compressive strength for all the mixes containing AHA increases with age up to 14 day hydration period but decreases to the 28 days hydration period while the conventional concrete increases steadily up to 28-day hydration period. Mbiminah (1991), worked on the effect of Rice husk on the compressive strength and durability of burnt clay bricks. Test results show that Rice husk has a decreasing effect on

the compressive strength of the brick and increasing effect on the water absorption of the bricks.

On the microstructure of composite cement system many research works were carried out. Hill and Sharp (2002) studied the mineralogy and microstructure of three composite cements with high replacement levels. The samples were cured in air under the same temperature and humidity condition and tested at various times for up to 6 month. The hydration products were identified by x-ray diffraction method and their microstructure determined by Scanning Election Microscopy (SEM). Results showed that although the observed hydration products were mostly as expected, due to the high replacement, the degree to which those phases was present was unusual. In particular the calcium hydroxide initially formed in the BFS – cement system was totally consumed within six month indicating the important pozzolanic behaviour of BFS at such high replacements.

Escalante –Garcia, and Sharp (2004) examined the chemical composition and microstructure of hydration products in blended cements. In their study paste of neat and blended Portland cement (incorporating either 60% ground granulated blast furnace slag or 30% pulverized fuel ash, or 22% volcanic ash) were cured for one year at temperature ranging from 10-60^{0}C. The hydration products were characterized by x-ray diffraction, (X.R.D) scanning electron microscopy and energy dispersion spectroscopy. Test results indicated that the apparent porosity of the pastes increased with increasing curing temperature. Chemical analysis data for the hydration products show that in the presence of the replacement material the composition of the C-S-H shifted toward higher SI and AI contents, where as that of Ca was lower.

CHAPTER 3

3.0 Materials and Methods

3.1 Materials and Methods

Rice husk was collected from Basawa rice mill, Basawa Village, Sabon Gari Local Government area, Kaduna State, Ordinary Portland Cement Used in the study was the Sokoto cement brand, a product of Cement Company of Northern Nigeria (CCNN). Laboratory rotary mill of Nigerian Institute of Leather and Science Technology was used. Other materials such as Tampling Rod, Sampling Equipment (Scoop and Shovel, trowel, Containers like measuring cylinder, moulds and sieves), Capping compound (a moist storage cabinet or room), Compressive Strength Testing Machine, Curing chamber, were provided by the Concrete Laboratory section of Building Department of the Ahmadu Bello University, Zaria. X-ray Diffractometre (software driven) and Mortar and Pestle were sourced from Engineering Materials Equipment Development Institute Akure, Ondo state.

3.1.1 Preparation of rice husk powder

Rice husk powder was prepared by grinding the rice husk in a rotary mill. The powder produced was sieved with British Standard sieves and were allowed to pass through 2µm sieve.

3.2 Production of Specimens

The cement was mixed with the rice-husk powder and the control mix without the powder. Both were measured and mixed with water after determining the water/cement ratio. The cube moulds were made of steel and wood in two halves and bolted together. The moulds were kept clean and lubricated with oil to prevent concrete from sticking to the mould. The sections were each time bolted tightly together and they were held down firmly on the base plate. The moulds used are of 70mm x 70mm in accordance with B.S 1881. The mixing was performed in accordance with B.S 1881. This required that mixing should be performed thoroughly to ensure all the constituent materials were uniformly distributed throughout the whole mix. The mixing was done manually with shovel on clean flour in the concrete flow of the concrete laboratory of the Department of Building Ahmadu Bello University, Zaria. The compressive strength of the sample was determined in accordance with the standard procedure for pre-cast concrete block. The weights of the samples were always taken before the compressive strength test was conducted. Three samples blocks were cured each at 28 days after casting at different replacement levels.

3.3 Casting and Compacting of the Concrete

The moulds were filled with the fresh concrete mix in three equal layers and tamped with steel rod 25 times at each layer. The concrete in the moulds after casting were left for 24 hours at room temperature and carefully detached. This is done by loosening the bolds on the mould. They were then taken and immerse in the water tank known as the curing chamber and other specimens were cured over rain outside the laboratory for 28 days.

They were removed from the curing medium on the 28th day and kept in an open space to allow water to dry from their surfaces. The samples were dried in an electric oven at 110 ^{0}C for an hour to ensure their complete drying.

Table 3.1 Design

S/N	Composition of Composite Cement System in %		Curing Period in Days	Curing Condition
	OPC	RICE HUSK POWDER	28	
1	100	0	✓ ✓	CN CE
2	95	5	✓ ✓	CN CE
3	90	10	✓ ✓	CN CE
4	85	15	✓ ✓	CN CE
5	80	20	✓ ✓	CN CE
6	75	25	✓ ✓	CN CE
7	70	30	✓ ✓	CN CE
8	65	35	✓ ✓	CN CE
9	60	40	✓ ✓	CN CE
10	55	45	✓ ✓	CN CE
11	50	50	✓ ✓	CN CE
12	45	55	✓ ✓	CN CE

Key: CN= Cured normally,
CE= Cured on exposure to weather condition



Plate I: One of the specimens

3.5 Compressive Strength Test

This was carried out by determining the failure loads of the cube specimens using the crushing/compressive strength testing Machine. The applied load axially on the cubes specimens ultimate compressive load the cube can withstand was reached after which the cube experience failure by crushing, the meter reading was taken immediately and later adjusted to zero reading for the next crushing.



Plate II: Tested specimens

3.6 X-Ray Diffraction Analysis

The X-Ray diffraction analysis of higher replacement samples was carried out for phase identification and quantification. This was done by using software driven X-ray diffraction instrument which is capable of given accurate spectra of powdered or polished substrates using its database. The Instrument consists of three basic elements: An x-ray tube, a sample holder and x-ray detector. The composite samples were grounded into particles size of about 0.002 mm to 0.005 mm cross section using agate Mortar and Pestle. They were pressed into a sample holder to obtain a smooth flat surface. The mechanical assembly that makes up the sample holder, detector arm and associated gearing is referred to as goniometer which is used to maintain the angle and rotate the

sample. All the samples were kept at an angle θ and the detector at angle of 2θ with respect to the incident beam in order to maintain a constant focusing distance.

X-rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target by applying a voltage and bombarding the target material with electrons. When the electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristics x-ray spectra are produced. The diffraction spectrum produced consists of a plot of reflected intensities versus the detector angle 2θ. The 2θ values were calculated based on the wave length of the anode material of the x-ray tube. A reduced peak position to the inter planar spacing d that corresponds to the hkl planes caused the reflections. The values of the d-spacing depend on the shape of the unit cell. The d-spacing was gotten as a function of 2θ from Bragg's law.

$d = \lambda / 2 \sin \theta$

The crystalline structure was automatically marched using a software driving data base of the International Centre for Diffraction Data (ICDD) or formally known as Joint Committee on Powdered Diffraction Standard (JCPDS) an organization that maintains the data base of inorganic and organic spectra. The interpretations of the results are shown in tables 4.2 and 4.3 while the diffraction spectra graphs are attached in appendixes A though E.

CHAPTER 4

4.0 Results and Discussion

4.1 Results Of Compressive Strength Test

Table 4.1 Compressive Strength of Rice-husk Cement Samples

Mix Ratio (%)	Age (days)	Average Weight (gm)	Failure Load (kN)			Average Failure Load (kN)	Compressive Strength (N/mm^2)
100%OPC 0% RHP	28	845.7	120	190	180	163.33	33.33
95%OPC 5%RHP	28	711.7	110	80	100	96.67	19.72
0%OPC 10%RHP	28	682.0	50	70	50	56.67	11.57
85%OPC 15%RHP	28	609.3	10	10	10	10.00	02.04
80%OPC 20%RHP	28	505.7	08	08	08	08.00	01.63
75%OPC 25%RHP	28	505.0	08	05	08	07.00	01.43
70%OPC 30%RHP	28	498	08	05	08	07.00	01.36
65%OPC 35%RHP	28	478	08	05	04	06.67	01.36
60%OPC 40%RHP	28	466	04	05	02	03.67	0.75
55%OPC 45%RHP	28	375	03.3	03.33	03.33	03.33	0.68
50%OPC 50%RHP	28	350	02.67	02.67	02.67	02.67	0.54
45%O5555% RHP	28	300	02	02	02	02.00	0.41

Key: RHP = Rice husk Powder

4.2 Results Of X-Ray Diffraction Analysis

Table 4.2: Observed Crystals (Phases) in the X-Ray Diffraction Spectrum Peaks

Samples	Predominant Peaks, 2θ Intensities and hkl Values				Mineral Name	Chemical Formula	Crystal Phase	Space Group (SG)	Crystal System
		2θ	Int	hkl					
A	1	31	58	137	Julgoldite	$Ca_2(Fe,Al)_3$ (SiO_4) (Si_20_7) $(OH)_2$! H_20	Calcium Aluminium Iron Silicate Hydroxide Hydrate	C2/m	Monoclinic
	2	35	25	313					
	3	34.3	21	-					
	4	33	19	-					
	5	47.5	15	-					
A8	1	35	27.5	-	Amstallite	Ca Al (Si_3Al) O_8 $(OH)_4$!H_2O	Calcium Aluminium Silicate Hydroxide Hydrate	12/a	Monoclinic
	2	52.5	19.5	-					
	3	36	19	-					
	4	35.5	16.5	-					
	5	74	16	-					
A9	1	33	29.5	-	Crossite	(Na, Ca)$_2$ Fe, Mg,Al)$_5$ $(Si,Al)_8O_{22}$ $(OH)_2$	Sodium Calcium Iron Magnesium Aluminium Silicate Hydroxide	C2/m	Monoclinic
	2	23	26.5	-					
	3	31	23	-					
	4	34.4	22	-					
	5	25	19	-					
A10	1	24	24	-	Perlialite	$K_9NaCaAI_{12}$ Si_{24} O_{72}! 15 H_2O	Potassium Sodium Calcium Aluminium Silicate Hydrate	C2/m	Hexagonal
	2	23.5	22.5	-					
	3	24.5	22	-					
	4	31	20.5	-					
	5	25	19.5	-					
A11	1	24.5	30	-	Franzinite	$(Na,Ca)_7$ $(Si,Al)_{12}$ O_{24} $(SO_4,$ $OH,CO_3)_3$! H_2O	Sodium Calcium Aluminium Carbonate Sulfate Silicate Hydroxide	P3*1	Hexagonal
	2	23	28	-					
	3	25	27	-					
	4	23.5	25.5	-					
	5	24.4	23.5	-					

							Hydrate.		

Table 4.3 Corresponding Lattice Parametres

SAMPLES	Lattice parameters					
	a(Å)	b(Å)	c(Å)	α^0	β^0 -	$\square^0$
A	8.92	6.09	19.37	-	97.5	-
A8	18.837+0.005	11.515+0.003	5.188+0.001	-	100.88+0.02	-
A9	9.647	17.905	5.316	-	-	-
A10	18.49+0.03	-	7.5+0.01	-	-	-
A11	2.884	-	26.58	-	-	-

4.3 Discussions

4.3.1 Compressive strength

From table 4.1, it can be seen that at 28 days sample A10 has the highest 28 days compressive strength (33.33N/ mm^2 water cement ratio (w/c) = 0.32 and Samples A2 and A3 have 19.72N/mm^2 and 11.57N respectively. The British Standard BS 12 minimum strengths at 28 days is 29 N/mm^2 and water/cement ratio (w/c) is 0. 4. (Bye 1990). Sample A2 gave about 65% of the minimum standard value this is due to effect of surface area and water/cement ratio and could only have potential for non-load bearing application.

4.3.2 X-ray diffraction analysis

Results of the x-ray diffraction analysis of the some of the samples, representing the composites with higher percentage replacement are presented in table 4.2. The results compared the observed crystals phases in the XRD spectrum peaks of the control sample with that of the higher replacement samples (Samples A8, A9, A10 and A11).

Table 4.2 shows crystal structures matched in accordance with the ICCD data. Samples A, A8 and A9 shared similarities in crystal phases and crystal system and this means similar formula (Ca_3SiO_2) or C3S (Bye, 1990). The observed Overlapping of crystal Systems with indication for monoclinic structures (ICDD cards no; 24-0198,41-1420 and 20-0376) verify present of C_3S the main strength-producing constituent of cement (Austin, 1984). Mg^{2+}, $Al^{3+}Fe^{3+}$ with smaller amount of K^+ and Na^+ are the most

frequently found foreign ions in C_3S phase (Bye, 1990) and they were found in across the samples. Samples A_{10} and A_{11} have the same crystal systems which are hexagonal structures (ICDD cards no; 38-0395 and 30-170). They also overlapped to each other. It is of interest to note that among the samples only sample A_9 has the same space group with the control sample and is the only sample without water of crystallization. Other lattice parameters observed were recorded and presented in table 4.3. It can be said generally that the observed crystals of the hydration products were mostly as expected due to the high replacement, In particular the calcium hydroxide crystals were found in all the samples except in A_{10} this indicates why the pozzalanic reaction did not take place at such high replacement.

However, with exception of sample A_{10} it explicate that at 28 days not all the calcium hydroxides were consumed in all the samples Indicating that at high percentage replacement more curing period is required for important pozzolanic reaction to take place and this is in line with the findings of Hill and Sharp. (2002).

CHAPTER 5

5.0 Summary, Conclusion and Recommendation

5.1 Summary

The study was able to explore some of the rice husk in Basawa, Sabongari Local Government Area Zaria. Of much interest in the study are the results of the compressive strength and x-ray diffraction analyses. The 28 days compressive strength of the cubes made reveals that Basawa Rice husk has potential as partial replacement of ordinary Portland cement (Rice-husk Portland cement composite). However, the x-ray diffraction analysis pattern indicates that higher percentage replacement can be achieved only at higher curing period.

5.2 Conclusion

The important findings of the study focused more on the compressive strength and x-ray diffraction analyses. However, it can be said that rice husk powder can be used in the production of composite cement particularly for low load bearing concrete and the percentage of the rice husk needed should be between 5-10% and for higher percentage (that is between 10%-50%) no pozzalanic reactions were observed.

5.3 Recommendation

The significance of cements in national development need not to be over-emphasized, in view of this, the researcher wishes to make the following recommendations:

i. Long term strength development of the composite cement beyond 28 days should be investigated in future studies, e.g. 90 days and 1 year.

ii. Since curing conditions affect pozzolanic reactivity, the composite cements concrete should be studied in details under various curing conditions in future research work.

iii. X-ray diffraction is of limited value in examining the hydration products because variation in their quantity significantly influence diffracted intensity, so more research should be carried out by using scanning electron microscopy.

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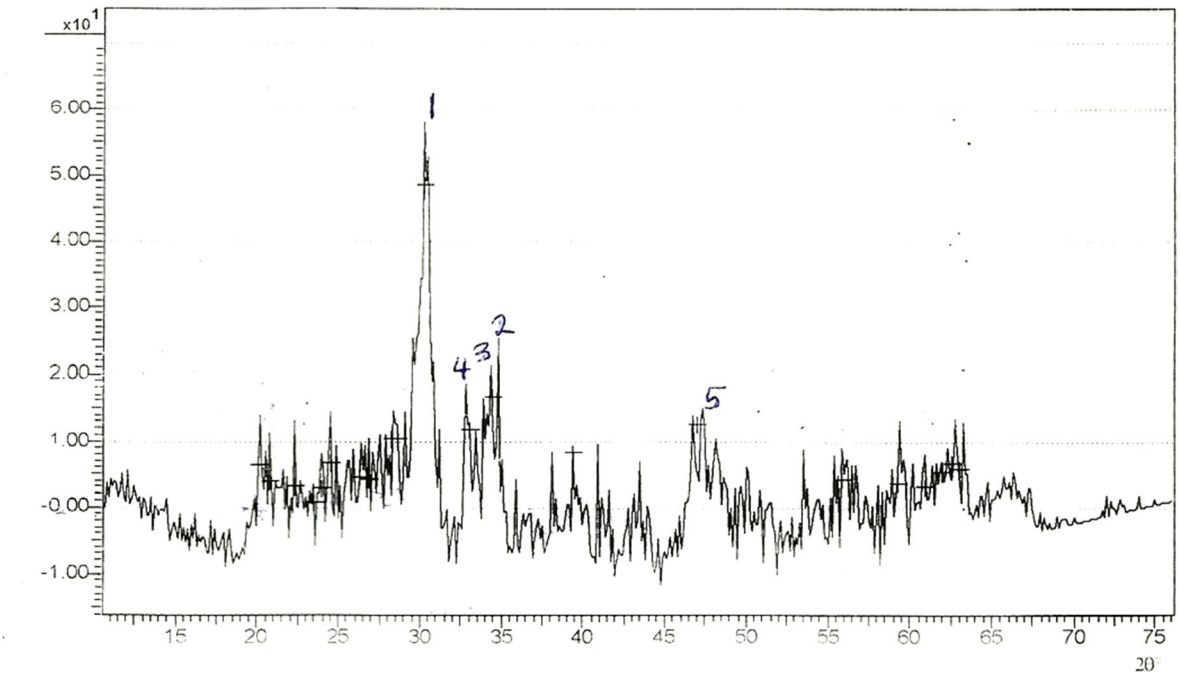
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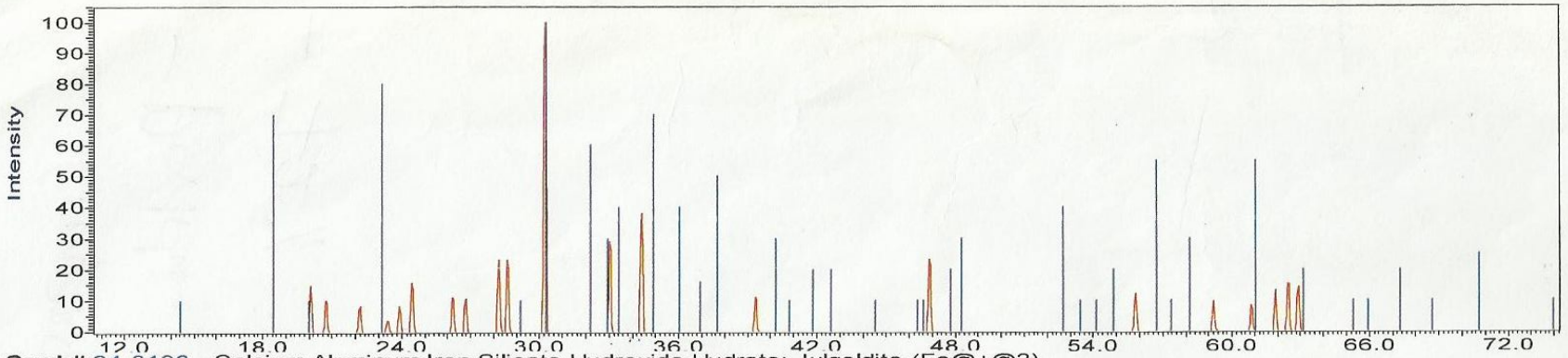
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APPENDIX 'A'

MD-10. 12/22/09
Exposure Time:1200/1200sec. Radiation:CuKa, avg Sample:A
Operator:EMDI Akure
File:00743.smd



2θ	d	I int	I max	I rel	I corr	FWHM
20.12	4.41238	384	7.3	14.8	18.0	0.90531
20.79	4.27171	258	4.9	9.9	11.8	0.90531
22.24	3.99669	215	4.1	8.3	9.5	0.90531
23.44	3.79554	94	1.8	3.6	4.0	0.90531
23.96	3.71354	210	4.0	8.1	8.9	0.90531
24.51	3.63245	407	7.8	15.7	17.1	0.90531
26.28	3.39130	286	5.5	11.0	11.6	0.95265
26.83	3.32272	276	5.3	10.6	11.1	0.95225
28.28	3.15591	601	11.5	23.2	23.8	0.95166
28.66	3.11485	596	11.4	23.0	23.4	0.95162
30.28	2.95198	2592	49.6	100.0	100.0	0.95199
33.08	2.70818	746	12.9	28.8	28.0	1.0604
34.45	2.60346	1004	17.6	38.7	37.2	1.0427
39.41	2.28625	285	9.3	11.0	10.2	0.56478
46.93	1.93611	595	13.6	23.0	20.5	0.8072
55.88	1.64522	319	5.4	12.3	10.7	1.0085
59.25	1.55945	251	4.8	9.7	8.3	0.81435
60.92	1.52069	213	4.1	8.2	7.1	0.77571
61.96	1.49763	337	6.5	13.0	11.1	0.7556
62.52	1.48565	398	7.6	15.4	13.2	0.74629
62.96	1.47620	366	7.0	14.1	12.1	0.73958



Card # 24-0198 - Calcium Aluminum Iron Silicate Hydroxide Hydrate; Julgoldite-(Fe@+@2).

Formula: Ca2 (Fe , Al)3 (Si O4) (Si2 O7) (O H)2 ! H2 O

24-0198 Quality: Normal

Ca2 (Fe , Al)3 (Si O4) (Si2 O7) (O H)2 ! H2 O
Calcium Aluminum Iron Silicate Hydroxide Hydrate
Julgoldite-(fe@+@2)

Rad:FeKa Lambda:1.9373 Filter:Beta filter used d sp:Debye-Scherrer
Cutoff: Int:Debye-Scherrer I/Icor:
Ref:Moore, P., 4 93, (1971)

Sys:Monoclinic S.G.:C2/m
a:8.92 b:6.09 c:19.37
α: β:97.5 γ: Z:4 mp.
Ref2
Dx:3.605 Dm:3.565 SS/FOM: F30=1.9(0.097,164) Volume[CD]:1043.23

εα:1.776(4) ηοβ:1.814(4) εγ:1.836(4) Sign:- 2V:50-70$DE
Ref3

Color:Black

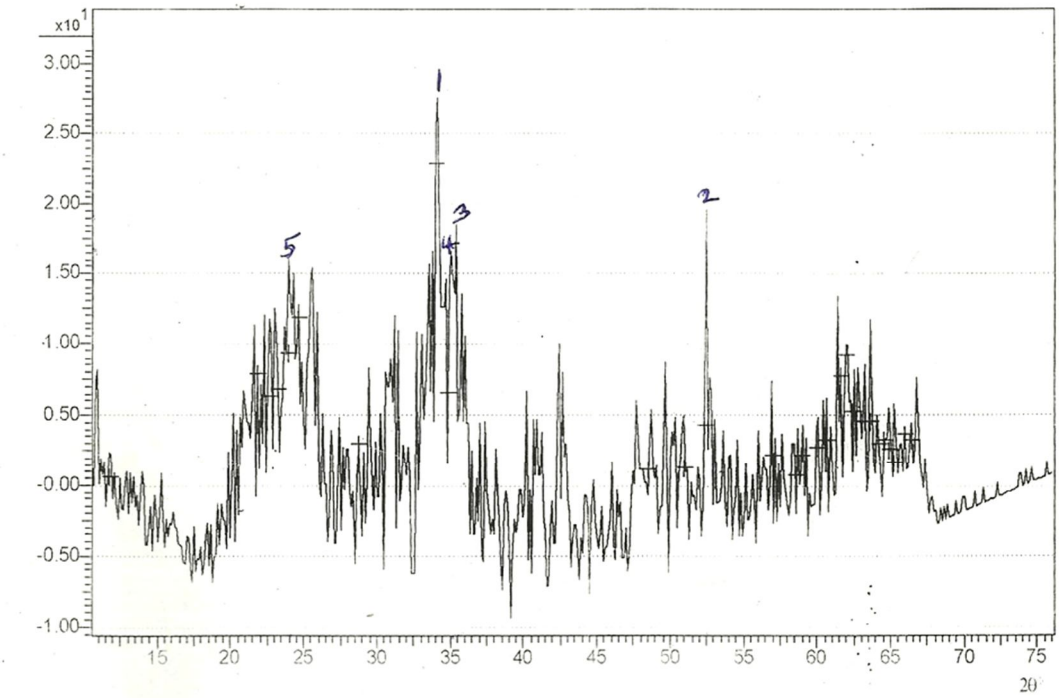
Optical data specimen from Amerika stope Langban, Sweden. Specimen from Langban, Sweden. Related to pumpellyite, contains minor Mn, Mg. Analysis (wt.%): \Si O2\ 34.0, \Al2 O3\ 1.3, \Fe2 O3\ 29.6, Ti O2 0.1, CaO 22.0, MgO 0.2, FeO 8.7, MnO 0.2, BaO 0.01, \H2 O\ 4.69. Plus 3 additional reflections to 1.103.

53 reflections in pattern. Wavelength 1.5406 was used to calculate the diffraction angles.

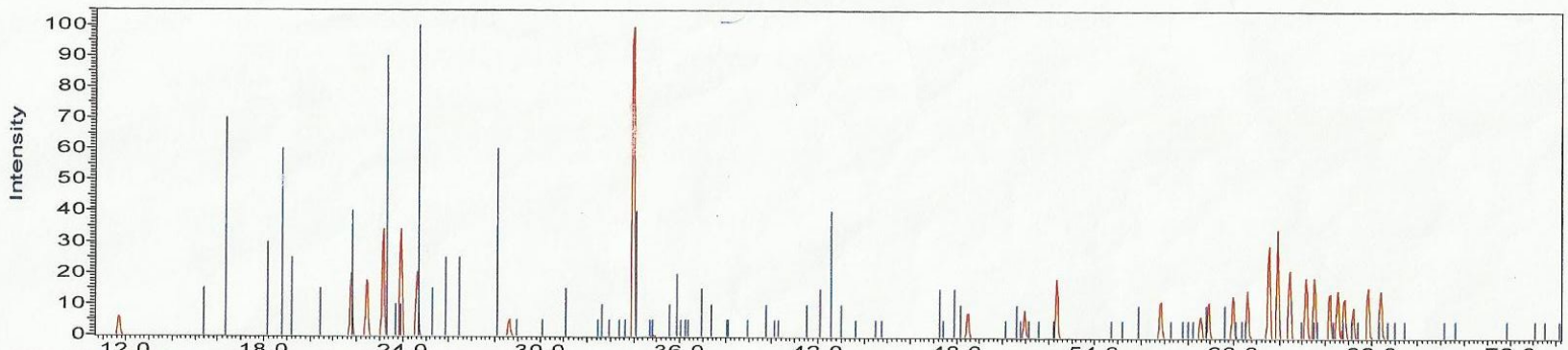
2θ	Int.	h k l	2θ	Int.	h k l	2θ	Int.	h k l	2θ	Int.	h k l
9.2336	20	0 0 2	32.9398	30	3 0 2	40.8168	10	0 2 6	56.7067	55	4 2 4
10.0207	20	1 0 0	33.4078	40	3 0 -4	41.8261	20	2 2 4	57.3627	10	0 0 12
14.4142	10	1 0 2	33.4078	40	3 1 -1	42.5910	20	3 2 0	58.1552	30	1 3 7
18.4694	70	0 0 4	34.9105	70	0 2 4	42.5910	20	4 0 -4	61.0319	55	3 2 8
18.4694	70	1 1 1	34.9105	70	3 1 -3	44.5078	10	3 0 6	63.1080	20	-1 3 9
20.0273	10	2 0 0	36.0261	40	2 1 5	44.5078	10	3 1 -7	65.2377	10	6 0 2
23.1440	80	2 0 2	36.0261	40	1 1 -7	46.3089	10	1 3 1	65.9123	10	0 2 12
29.1698	10	2 0 4	36.9155	16	2 0 6	46.5857	10	-4 0 6	67.3075	20	1 1 13
29.1698	10	0 2 0	36.9155	16	1 2 4	47.7816	20	-1 3 3	68.6532	10	-3 3 9
30.2729	100	3 0 0	37.6861	50	1 0 -8	48.2398	30	0 2 8	70.6626	25	-5 3 3
30.2729	100	1 1 5	37.6861	50	2 2 2	52.6504	40	-4 2 4	73.9300	10	5 3 3
32.1966	60	1 2 -2	40.2274	30	1 0 8	53.3789	10	-3 0 10			
32.1966	60	2 0 -6	40.2274	30	2 0 -8	54.0942	10	3 2 6			
32.9398	30	1 2 2	40.8168	10	4 0 0	54.8298	20	2 2 8			

APPENDIX 'B'

MD-10. 12/22/09
Exposure Time:1200/1200sec. Radiation:CuKa, avg Sample:A8
Operator:EMDI Akure
File:00744.smd



2θ	d	I int	I max	I rel	I corr	FWHM
11.74	7.53736	93	1.8	5.9	14.0	0.90531
21.81	4.07501	319	9.1	20.2	24.2	0.6082
22.50	3.95232	280	7.5	17.7	20.9	0.64787
23.19	3.83510	538	8.0	34.0	39.5	1.165
23.95	3.71534	550	10.5	34.7	39.7	0.9075
24.66	3.60968	321	13.1	20.3	22.9	0.42634
28.68	3.11236	81	4.3	5.1	5.4	0.34638
34.00	2.63638	1584	24.1	100.0	100.0	1.2018
34.86	2.57358	2	7.8	0.1	0.1	0.0053692
48.53	1.87580	121	2.3	7.7	7.1	0.95741
51.01	1.79046	134	2.6	8.5	7.7	0.94433
52.39	1.74621	291	5.6	18.4	16.7	0.9328
56.89	1.61854	181	3.5	11.4	10.3	0.87258
58.61	1.57498	103	2.0	6.5	5.8	0.83106
58.96	1.56653	176	3.4	11.1	9.9	0.82191
60.01	1.54159	205	3.9	12.9	11.5	0.79591
60.64	1.52705	234	4.5	14.8	13.2	0.78168
61.58	1.50594	470	9.0	29.7	26.4	0.76254
61.96	1.49763	543	10.4	34.3	30.5	0.7556
62.49	1.48629	339	6.5	21.4	19.0	0.74676
63.20	1.47125	305	5.8	19.3	17.1	0.73631
63.56	1.46364	304	5.8	19.2	17.0	0.73163



Card # 41-1420 **-** Calcium Aluminum Silicate Hydroxide Hydrate; Amstallite. **Formula:** Ca Al (Si3 Al) O8 (O H)4 ! H2 O

41-1420 Quality: Indexed

Ca Al (Si3 Al) O8 (O H)4 ! H2 O
Calcium Aluminum Silicate Hydroxide Hydrate
Amstallite

Rad:CuKa1 Lambda:1.54056 Filter: d sp:Guinier
Cutoff: Int:Guinier I/Icor:
Ref:Quint, R., 1987 253, (1987)

Sys:Monoclinic S.G.:I2/a
a:18.837±0.005 b:11.515±0.003 c:5.188±0.001
α: β:100.88±0.02 γ: Z:4 mp
Ref2
Dx:2.38 Dm:2.358 SS/FOM: F30=60(0.0135,37) Volume[CD]:1105.09

εα:1.5328(4) ηoβ:1.5340(4) εγ:1.5378(4) Sign:- 2V:57(2)$DE
Ref3

Color:Colorless

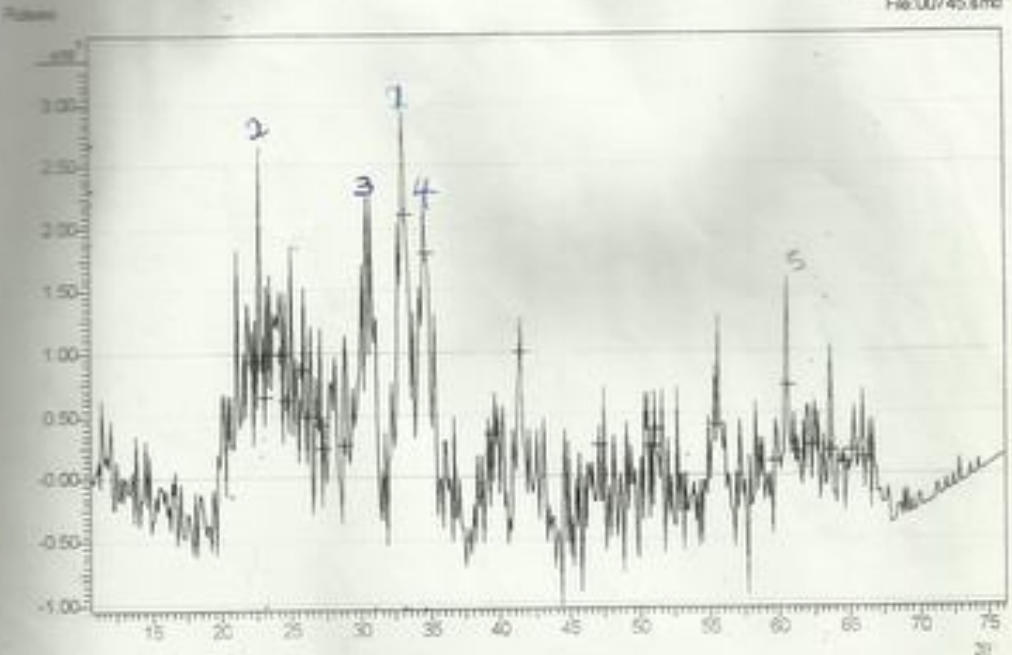
Specimen from Amstall, Lower Austria, Austria. Microprobe analysis (wt.%): CaO 13.80, Al2 O3 22.84, Si O2 49.41, Cl 1.75, H2 O (elemental analyzer) 12.40; Ca0.98 Al1.78 Si3.26 O12.80 Cl0.20 H5.46 .

117 reflections in pattern. Wavelength 1.5406 was used to calculate the diffraction angles.

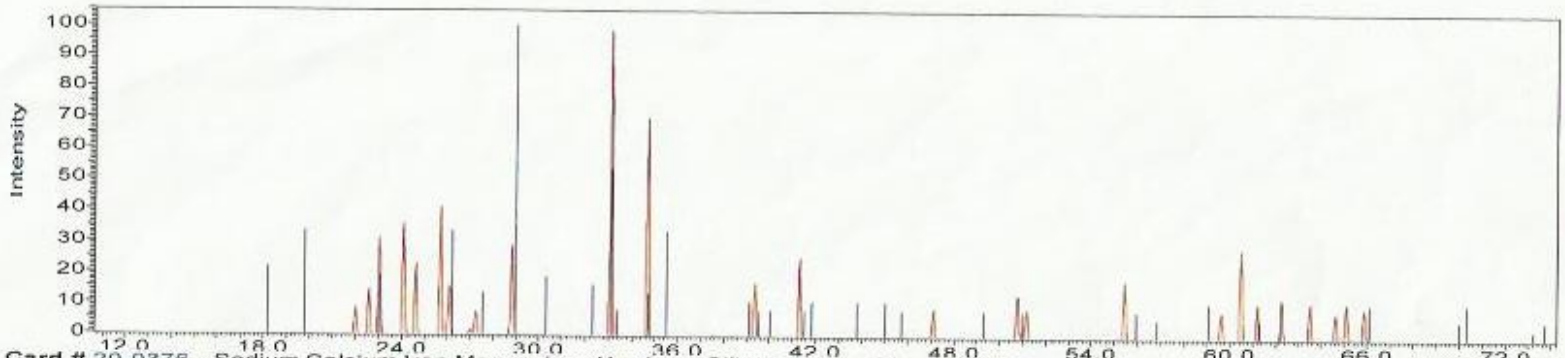
2θ	Int.	h	k	l	2θ	Int.	h	k	l	2θ	Int.	h	k	l	2θ	Int.	h	k	l
9.0628	100	1	1	0	35.5101	10	1	1	-2	48.1311	10	5	5	-1	63.5899	5	8	2	-3
9.5641	5	2	0	0	35.8327	20	0	4	1	50.1083	5	2	4	2	64.3278	5	3	3	3
15.3976	15	0	2	0	35.8327	20	5	3	-1	50.5841	10	6	0	2	64.7288	5	0	8	0
16.3110	70	3	1	0	36.0261	5	7	1	-1	50.5841	10	8	4	0	64.7288	5	9	1	-3
18.1341	30	2	2	0	36.2065	5	2	4	-1	50.7347	5	7	3	-2	65.0842	5	10	4	-2
18.8094	60	1	1	-1	36.3278	5	3	1	-2	50.7347	5	0	6	1	65.0842	5	13	1	-1
19.1831	25	4	0	0	36.8998	15	4	0	-2	51.1307	5	2	6	-1	65.3921	5	10	4	1
20.4375	15	1	1	1	37.3289	10	1	1	2	51.1307	5	8	4	-1	66.2818	5	2	4	3
21.8250	40	3	1	-1	38.0337	5	2	4	1	51.5332	5	4	6	0	66.2818	5	11	3	1
23.2916	90	0	2	1	38.1006	5	2	2	-2	52.1976	5	9	1	-2	66.6558	5	12	2	-2
23.6690	10	1	3	0	38.9232	5	8	0	0	54.6880	5	11	1	-1	66.9799	5	7	7	0
23.9144	10	2	2	-1	39.7462	10	5	1	-2	55.1515	5	11	1	0	67.3624	5	1	7	-2
24.6896	100	4	2	0	40.0967	5	6	2	1	55.8438	10	1	1	3	69.1147	5	11	5	0
25.2594	15	5	1	0	40.2461	5	4	2	-2	57.2457	5	9	3	-2	69.1147	5	9	3	-3
25.8640	25	3	1	1	40.2461	5	5	3	1	57.7561	5	3	5	2	69.5831	5	8	6	-2
26.4584	25	2	2	1	41.4841	10	7	3	0	57.9949	5	3	7	0	71.7792	5	6	6	2
28.0818	60	5	1	-1	41.4841	10	6	0	-2	57.9949	5	10	2	-2	73.0005	5	2	0	-4
28.0818	60	4	2	-1	42.0711	15	1	3	-2	58.1954	5	7	5	1	73.4620	5	6	8	-1
28.9473	5	6	0	0	42.0711	15	8	2	0	58.7649	10	8	0	2	73.4620	5	11	1	2
30.0642	5	1	3	1	42.5699	40	4	4	1	58.7649	10	1	3	-3	74.0649	5	1	1	-4
31.0491	15	0	4	0	42.9950	10	1	5	-1	59.5992	10	11	3	-1	79.3933	5	3	9	1
31.0491	15	3	3	-1	43.6271	5	4	0	2	59.5992	10	3	1	3	79.3933	5	9	7	-2
32.4365	5	4	2	1	43.6271	5	1	3	2	60.0688	5	3	7	-1	80.1910	5	3	1	4
32.5943	10	2	4	0	44.4618	5	3	5	-1	60.3283	5	9	5	0	80.1910	5	9	1	3
32.9398	5	6	2	0	44.7629	5	9	1	0	60.3283	5	0	6	2	81.8419	5	7	7	2
33.3821	5	5	1	1	47.2279	15	2	4	-2	60.5026	5	11	1	-2	88.6939	5	4	10	-1
33.6143	5	5	3	0	47.3847	5	0	6	0	60.5026	5	5	3	-3	88.6939	5	15	3	1

APPENDIX 'C'

MD-10, 12/22/09
Exposure Time:1200/1200sec Radiation:CuKa, avg Sample:A9
Operator:EMDI Akure
File:00745.smd



2θ	d	Int	I max	I rel	I corr	FWHM
22.04	4.03266	129	10.6	8.4	9.9	0.21117
22.62	3.93079	225	10.7	14.5	17.0	0.36471
23.07	3.85488	470	7.8	31.0	35.8	1.0711
24.12	3.68895	548	11.2	35.5	40.1	0.85275
24.65	3.61074	349	7.5	22.5	25.2	0.8057
25.72	3.46348	635	9.9	41.1	45.1	1.1453
26.11	3.41318	238	6.3	15.4	16.8	0.6869
27.03	3.29822	26	5.6	1.7	1.8	0.084576
27.26	3.27098	112	3.8	7.3	7.8	0.54266
28.64	3.09515	448	4.1	29.0	30.3	2.0084
33.13	2.70376	1547	22.4	100.0	100.0	1.262
34.71	2.58399	1099	19.4	71.0	70.0	1.0408
39.40	2.28713	259	4.9	16.8	16.0	0.98271
41.36	2.18331	393	11.3	25.4	24.0	0.63676
47.14	1.92785	133	3.8	8.6	7.9	0.64091
50.81	1.79687	203	3.7	13.1	11.9	0.99955
51.22	1.78360	137	5.1	8.8	8.0	0.48488
55.47	1.65636	278	6.5	17.9	16.1	0.85942
59.67	1.54946	128	2.5	8.3	7.3	0.80392
60.53	1.52956	440	8.4	28.4	25.2	0.78407
61.26	1.51307	170	3.3	11.0	9.7	0.76878
62.31	1.49014	193	3.7	12.5	11.0	0.74968
63.55	1.46394	173	3.3	11.2	9.9	0.73181
64.67	1.44132	127	2.4	8.2	7.2	0.72061



Card # 20-0376 - Sodium Calcium Iron Magnesium Aluminum Silicate Hydroxide; Crossite.
Formula: (Na , Ca)2 (Fe , Mg , Al)5 (Si , Al)8 O22 (O H)2

20-0376 Quality: Indexed

(Na , Ca)2 (Fe , Mg , Al)5 (Si , Al)8 O22 (O H)2

Sodium Calcium Iron Magnesium Aluminum Silicate Hydroxide

Crossite

Rad:FeKa	Lambda:1.9373	Filter:Beta filter used	d sp:
Cutoff:	Int:	I Icor:	

Ref:Borg., 15 67, (1967)

Sys:Monoclinic		S.G.:C2 m		
a:9.647	b:17.905	c:5.316		
α:	β:103.6	γ:	Z:2	mp

Ref2

Dx:3.223	Dm:3.483	SS/FOM: F30=11.9(0.0145,174)	Volume[CD]:892.48

εα:1.659	nβ:1.670	εγ:1.674	Sign:-	2V:60SDE

Ref3

Color:Blue

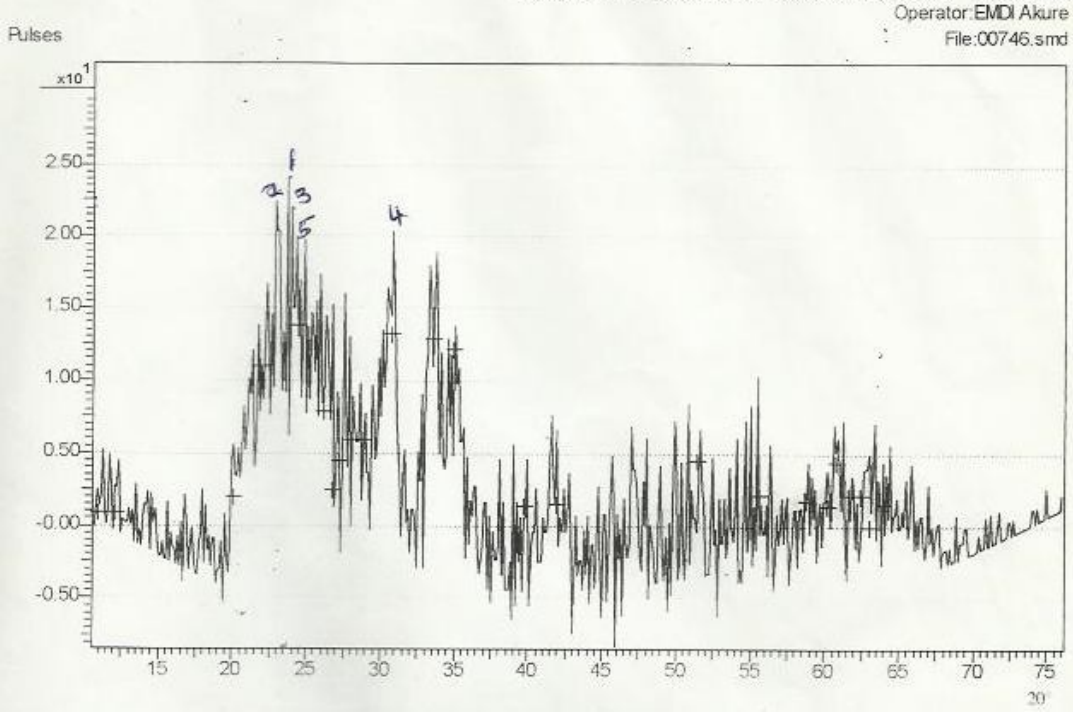
Fe +2 = 1.58, Mg = 1.36, Mn +2 = 0.02, Fe +3 = 1.06, Al +3 = 0.90, Ti = 0.04 formula units. Specimen from Healdsburg, California, USA (University of California 331-M-56). (Na , Ca , K)2.0 (Fe +2 , Mg , Mn)5.0 (Fe +3 , Al +3 , Ti)2.0 (Si , Al)8.0 O22.0 (O H)1.9 . To replace 20-470 and 31-1312.

34 reflections in pattern. Wavelength 1.5406 was used to calculate the diffraction angles.

2θ	Int.	h k l	2θ	Int.	h k l	2θ	Int.	h k l	2θ	Int.	h k l
9.8748	14	0 2 0	33.0775	40	1 5 1	45.0210	8	3 5 1	69.9389	4	-5 5 3
10.6374	100	1 1 0	33.3565	6	-3 3 1	45.6921	6	-4 0 2	70.2385	8	1 11 2
18.2017	16	-1 1 1	34.6875	10	0 6 1	49.2407	6	-4 6 1	73.1975	2	0 12 2
19.8015	25	0 4 0	35.4811	25	-2 0 2	50.9472	6	0 10 0	73.6618	4	-2 12 2
23.0224	14	-1 3 1	39.0991	8	-3 5 1	55.9177	6	4 6 1			
26.1891	25	1 3 1	39.5100	6	-4 2 1	56.8213	4	4 8 0			
27.5072	10	2 4 0	39.9853	6	3 3 1	59.0541	8	6 0 0			
28.9666	75	3 1 0	41.4841	6	1 7 1	61.2106	4	-2 6 3			
30.2624	14	2 2 1	41.8261	8	2 6 1	62.1670	4	0 12 0			
32.3041	12	3 3 0	43.7602	8	2 0 2	66.0701	8	-6 6 1			

APPENDIX 'D'

MD-10. 12/22/09
Exposure Time:1200/1200sec. Radiation:CuKa, avg Sample:A10
Operator:EMDI Akure
File:00746.smd



2θ	d	Iint	Imax	Irel	Icorr	FWHM
10.92	8.10329	146	1.7	6.9	17.2	1.4992
12.10	7.31675	265	1.7	12.5	24.7	2.6897
20.07	4.42473	46	2.8	2.1	2.4	0.28185
21.80	4.07709	1402	11.8	65.9	69.9	2.0647
24.44	3.64161	2128	14.6	100.0	100.0	2.5395
26.17	3.40476	742	8.7	34.9	33.8	1.5523
26.76	3.33175	15	3.3	0.7	0.7	0.085648
27.17	3.28220	54	5.3	2.5	2.4	0.18542
28.07	3.17926	328	6.7	15.4	14.5	0.89343
28.88	3.09131	849	6.7	39.9	37.2	2.3012
30.66	2.91575	812	14.0	38.1	34.8	1.0562
33.49	2.67593	893	13.6	42.0	37.2	1.2004
34.97	2.56559	810	12.9	38.1	33.4	1.1491
39.68	2.27148	131	2.2	6.2	5.2	1.1044
41.98	2.15185	173	2.3	8.1	6.8	1.3688
51.41	1.77738	197	5.2	9.2	7.5	0.67572
55.70	1.65023	191	2.9	9.0	7.1	1.1299
58.84	1.56938	188	2.6	8.8	7.0	1.1624
60.16	1.53804	54	2.2	2.6	2.0	0.38082
60.82	1.52293	291	5.2	13.7	10.8	0.83188
61.66	1.50416	99	2.9	4.7	3.7	0.4956
62.40	1.48812	151	2.9	7.1	5.6	0.73688
63.00	1.47540	50	0.7	2.3	1.8	0.94959
63.95	1.45583	151	2.3	7.1	5.5	0.92315

38-0395 Quality: Normal

K9 Na Ca Al12 Si24 O72 !15 H2 O
Potassium Sodium Calcium Aluminum Silicate Hydrate
Perlialite

Rad:FeKa	Lambda:1.9373	Filter:	d sp:Debye-Scherrer
Cutoff:	Int:Debye-Scherrer	I/Icor:	
Ref:Men'shikov, Yu., 113 607, (1984)			

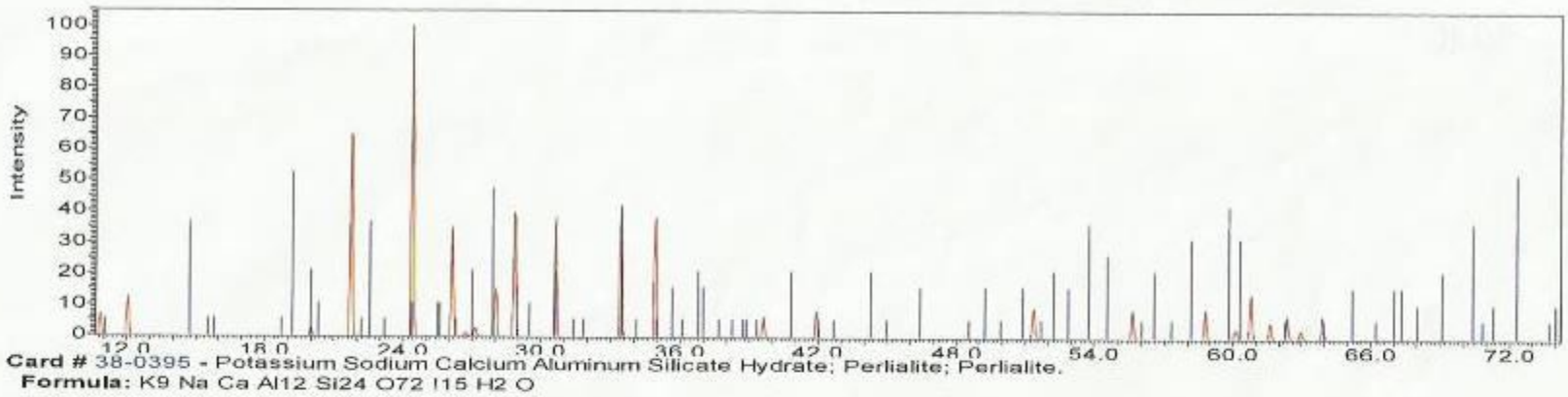
Sys:Hexagonal		S.G.:P6/mmm		
a:18.49±0.03	b:	c:7.51±0.01		
α:	β:	γ:	Z:1	mp
Ref2				
Dx:2.15	Dm:2.147	SS/FOM: F30=8.4(0.074,48)		Volume[CD]:2223.54
εα:	nβ:1.483(2)	εγ:1.488(2)	Sign:-	2V:
Ref3				

Color:White

Specimens from pegmatites of Mount Eveslogchorr and Mount Yukspor, Khibina massif, Kola Peninsula, USSR. Chemical analysis (wt.%): \K2 O\ 14.86, \Na2 O\ 0.98, CaO 0.96, SrO 1.65, FeO 0.21, MgO 0.07, \Ti O2\ 0.01, \Fe2 O3\ 0.92, \Al2 O3\ 20.13, \Si O2\ 50.72, \H2 O\ 9.52; K9.07 Na0.91 (Ca0.49 Sr0.46 Fe0.08) (Al11.35 Fe0.33 Mg0.05) Si24.27 O72.12 !15.2 H2 O. Pattern resembles those of synthetic zeolite L, K and Ba-G.

99 reflections in pattern. Wavelength 1.5406 was used to calculate the diffraction angles.

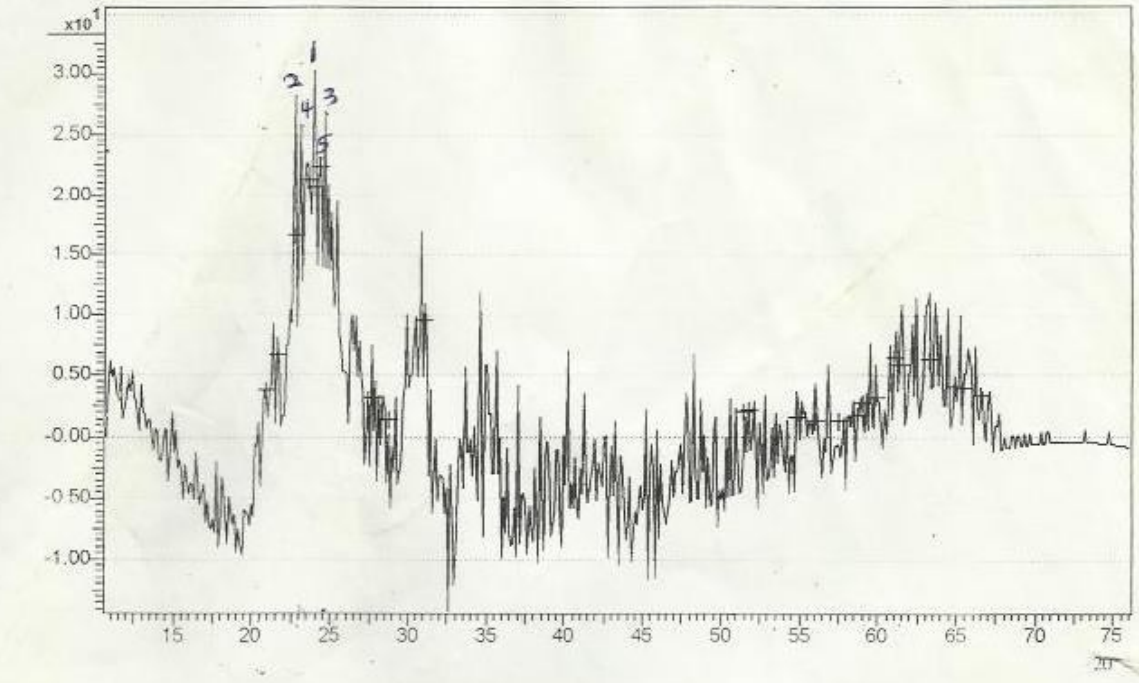
2θ	Int.	h	k	l	2θ	Int.	h	k	l	2θ	Int.	h	k	l	2θ	Int.	h	k	l
5.5190	100	1	0	0	34.1022	10	3	2	2	54.5120	50	6	5	0	78.6905	60	9	4	3
9.6058	20	1	1	0	34.9667	10	5	2	0	56.0290	10	6	5	1	80.4338	40	4	0	6
11.0509	10	2	0	0	35.6267	30	6	0	1	56.5167	40	8	3	0	81.5894	10	8	4	4
14.7524	70	2	1	0	36.0860	10	4	3	1	57.2847	10	9	0	2	82.0962	10	8	1	5
15.5335	10	1	1	1	36.7434	40	6	1	0	58.1150	60	5	4	3	82.6966	30	12	3	0
15.8126	10	2	0	1	36.9627	30	5	2	1	59.8117	80	9	2	1	83.6606	10	4	2	6
18.7053	10	2	1	1	37.6697	10	2	0	3	60.2415	60	7	5	0	84.5595	10	10	6	0
19.1957	100	2	2	0	38.2352	10	4	2	2	62.2134	10	8	3	2	85.2948	40	13	2	0
19.9817	40	3	1	0	38.7488	10	6	1	1	63.9320	10	9	2	2	87.4935	30	7	4	5
20.3051	20	3	0	1	38.8882	10	4	4	0	65.1353	30	11	0	1	88.1891	10	8	7	3
22.2062	10	4	0	0	39.3124	10	5	3	0	66.1758	10	8	5	0	90.0388	30	6	2	6
22.5488	70	2	2	1	40.7783	40	4	4	1	66.9799	30	7	6	1	91.1065	20	8	6	4
23.2052	10	3	1	1	41.9482	10	4	3	2	67.3075	30	11	1	0	92.9925	10	2	0	7
24.3667	20	1	0	2	42.6542	10	5	2	2	67.9731	20	9	3	2	93.3351	10	12	5	0
25.5022	20	4	1	0	44.2330	40	6	1	2	69.0567	40	10	3	0	94.1464	10	3	0	7
25.5767	20	1	1	2	44.9268	10	6	3	0	70.3591	70	10	3	1	94.8554	10	11	0	5
26.2678	10	2	0	2	46.4092	30	7	0	2	70.7848	10	5	1	5	95.4563	10	10	6	3
26.9975	40	3	2	1	48.4860	10	8	1	0	71.2782	20	7	7	0	96.8114	10	8	7	4
27.8579	90	5	0	0	49.1840	30	5	5	0	72.2879	100	6	0	5	97.8270	30	15	1	1
28.9666	30	3	3	0	49.8738	10	2	0	4	73.7286	10	9	5	1	99.1342	60	3	3	7
29.4553	20	4	2	0	50.8559	30	5	5	1	73.9974	20	12	1	0	99.4009	40	12	6	0
30.5915	40	2	2	2	51.6271	10	3	0	4	74.8168	30	10	4	1	102.3140	10	10	8	2
31.3619	10	3	3	1	52.1655	40	7	3	1	75.5151	10	12	0	2	102.7480	10	10	0	6
31.8085	10	4	2	1	52.8142	30	7	2	2	76.1560	20	0	0	6	104.2294	30	14	2	3
33.4849	80	6	0	0	53.7170	70	8	2	1	77.2514	40	2	0	6					



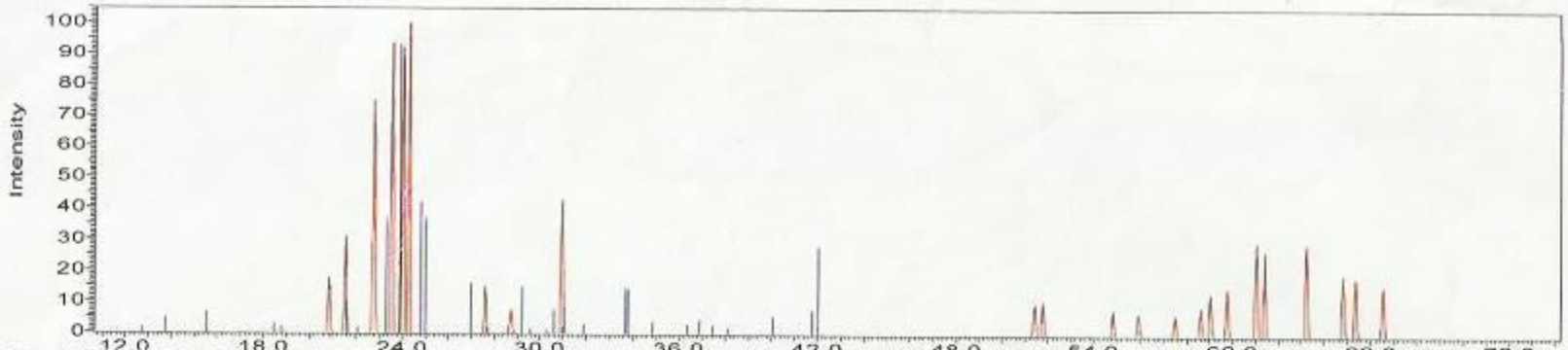
Card # 38-0395 - Potassium Sodium Calcium Aluminum Silicate Hydrate; Perlialite; Perlialite.
Formula: K9 Na Ca Al12 Si24 O72 I15 H2 O

APPENDIX 'E'

MD-10. 12/22/09
Exposure Time:1200/1200sec. Radiation:CuKa, avg Sample:A11
Operator:EMDI Akure
File:00747.smd



2θ	d	Iint	Imax	Irel	Icorr	FWHM
20.88	4.25339	209	4.0	17.9	19.4	0.90531
21.59	4.11585	362	6.9	30.9	32.9	0.90531
22.80	3.89956	882	16.9	75.4	77.9	0.90531
23.58	3.77255	1115	21.4	95.2	96.7	0.90531
24.05	3.69977	1088	20.8	92.9	93.4	0.90531
24.32	3.65907	1171	22.4	100.0	100.0	0.90531
27.65	3.22616	178	3.4	15.2	14.4	0.95183
28.77	3.10275	85	1.6	7.2	6.7	0.95162
30.98	2.88643	507	9.7	43.3	39.3	0.95242
51.46	1.77578	112	2.1	9.6	7.7	0.94091
51.83	1.76378	122	2.3	10.4	8.4	0.93783
54.86	1.67295	89	1.7	7.6	6.0	0.90414
55.97	1.64283	78	1.5	6.7	5.3	0.88796
57.54	1.60158	73	1.4	6.2	4.9	0.85953
58.66	1.57376	103	2.0	8.8	7.0	0.82973
59.07	1.56376	152	2.9	13.0	10.3	0.81894
59.80	1.54644	177	3.4	15.1	11.9	0.80083
61.06	1.51756	343	6.6	29.3	23.0	0.77282
61.43	1.50932	313	6.0	26.8	21.0	0.76547
63.24	1.47033	338	6.5	28.9	22.6	0.73572
64.84	1.43790	226	4.3	19.3	15.0	0.71932
65.39	1.42722	216	4.1	18.4	14.3	0.716
66.57	1.40466	181	3.5	15.5	12.0	0.80103



Card # 30-1170 - Sodium Calcium Aluminum Carbonate Sulfate Silicate Hydroxide Hydrate; Franzinite; Franzinite,
Formula: (Na , Ca)7 (Si , Al)12 O24 (S O4 , O H , C O3)3 ! H2 O

30-1170 Quality: Indexed

(Na , Ca)7 (Si , Al)12 O24 (SO4 , O H , C O3)3 ' H2 O

Sodium Calcium Aluminum Carbonate Sulfate Silicate Hydroxide Hydrate

Franzinite

Rad:CuKa1 Lambda:1.54056 Filter:Beta filter used d sp:

Cutoff: Int: I Ieor:

Ref:Merlino, S., Orlandi., 163. (1977)

Sys:Hexagonal S.G:P3*1

a:12.884 b: c:26.58

α: β: γ: Z:4 mp

Ref2

Dx:2.52 Dm:2.065 SS/FOM: F30=13.9(0.021,104) Volume[CD]:3821.09

εα: nωβ:1.510 εγ:1.512 Sign:- 2V:

Ref3

Color:White

Indexing checked with aid of single-crystal data. Specimen from pumice deposit, Pitigliano, Tuscany, Italy. Chemical analysis (wt.%): Si O2 32.44, Al2 O3 25.21, CaO 12.08, Na2 O 11.50, K2 O 4.24, S O3 10.65, C O2 1.54, H2 O 1.88, minor Fe2 O3 , MgO, Cl 0.36. (Na21.53 Ca12.50 K5.22 Mg0.20 Fe0.03 +3) (Si31.32 Al28.68) O120 (S O4)7.72 (C O3)2.03 (O H)3.48 Cl0.59 !4.31 H2 O .

32 reflections in pattern. Wavelength 1.5406 was used to calculate the diffraction angles.

2θ	Int.	h k l	2θ	Int.	h k l	2θ	Int.	h k l	2θ	Int.	h k l
7.9586	3	1 0 0	23.3288	40	2 1 3	30.9719	2	2 2 4	41.7653	8	3 0 10
8.5779	2	1 0 1	23.9014	100	3 0 0	31.8784	3	3 1 4	42.0300	30	3 3 0
10.3380	2	1 0 2	24.7804	45	1 0 7	33.6664	16	4 0 3			
12.7269	2	1 0 3	24.9926	40	2 1 4	33.7710	15	3 0 7			
13.7394	5	1 1 0	26.9808	17	2 1 5	34.8265	4	4 0 4			
15.5335	7	1 0 4	27.6814	2	2 2 0	36.3278	3	4 0 5			
18.4694	3	1 0 5	29.2186	16	2 1 6	36.8684	5	4 1 0			
18.7853	2	2 0 3	29.5751	1	3 1 2	37.4742	3	3 1 7			
21.6046	11	1 0 6	30.2729	1	1 1 8	38.0671	2	1 0 11			
22.0943	2	2 1 2	30.5700	8	3 1 3	40.0781	6	4 0 7			

APPENDIX F

Table 1 Compressive Strength of OPC and Lime/RHA Mixes

OPC/Lime + RHA	Cement: Sand	Water: cement	Cube strength (N/mm^2			
			3 days	7 days	14 days	28 days
OCP	1:3	0.49	12.35	24.20	25.40	28.54
50:50	1:3	0.82	2.19	5.13	5.19	5.41
40:60	1:3	0.85	3.10	7.88	7.90	8.16
30:70	1:3	0.87	2.45	6.90	6.95	7.10
20:80	1:3	0.89	2.27	6.05	6.15	6.50
10:90	1:3	0.90	1.98	4.47	4.58	4.70
100:00	1:3	0.70	0.20	0.60	0.89	1.15

SOURCE:LAWALI (2007).

Table 2 Similar Studies on Rice husk/OPC Composite

Work	Mixture	Ratio	28days compressive Strength in N/mm^2
Mehta (1978)	RHA/OPC	-	55
CBRI (1979)	Rice husk powder/Waste lime Sludge	-	5
CBRI (1979)	RHA/Lime (fine grounded)	-	20
CBRI (1979)	Rice husk powder/Lime (not fine grounded)	-	0.4
CBRI and Rehsi (1979)	Rice husk powder/Lime	-	4-7
Shrestha(1979)	Rice husk powder/Lime	-	9
Mohammed(1979)	RHA/Lime	-	9

SOURCE:LAWALI (2007).