KWAME NKRUMAH UNIVERSITY OF SCIENCE AND TECHNOLOGY

KUMASI, GHANA.

Utilisation of Lithomargic Clay as a Fireclay Refractory Material



By

Joseph Alhassan Adam, BSc. (Hons)

A thesis submitted to

The Department of Materials Engineering,

Kwame Nkrumah University of Science and Technology

In Partial Fulfilment of the Requirements for the Degree of

Master of Science in Environmental Resources Management

Department of Materials Engineering

SANE

October 2012

DECLARATION

"I hereby declare that this submission is my own work towards the MSc. and that, to the best of my knowledge, it contains no material previously published by another person nor material which has been accepted for the award of any other degree of the University, except where due acknowledgement has been made in the text".

| Signature | | DATE | |
|--------------------|---------|------------------|--|
| ADAM ALHASSAN JOSE | 2PH | | |
| (STUDENT) | | | |
| Signature | | DATE 23-04-13 | |
| DR. | ANTHONY | 23/04/13 | |



i

material

ACKNOWLEDGEMENTS

I will like to begin by thanking the Almighty and Ever Living God for having brought me this far. Without Him, I certainly would not have done anything on this project work. I acknowledge the constructive advice, support and contribution of my supervisor, Dr. Anthony Andrews, without whom this work would not have been conceived and executed. To my parents and siblings, I say thank you for your support financially, spiritually and morally. You played a role I cannot forget.

I could not also have finished up without the love and encouragement from my special friend Abdulai Sheila and my roommate Samuel Mensah Opoku. You were always there to encourage me each time I was down and when things were not going on well for me. God bless you.

Last but certainly not the least, I like to acknowledge the support received from the following

BADW

for their immense contributions and advice:

Materials Engineering Department
 Miss Beautrice Acheampong and Mr. Atti

• Civil Engineering Department Mr. Gilbert and Mr. Micheal

• Technology Consultancy Center (TCC) Mr.

Micheal Konnney

- -

Thank you for all the support. I appreciate all you have done for me.

Uti/isation of lithomargic clay as a fireclay refractory materia/

Adam A. Joseph (MSc. Thesis 2012).

Page ii



CONTRIBUTION FROM THIS STUDY

A. Andrews, J. Adam, S.K.Y. Gawu, (2013) Development of fireclay aluminosilicate refractory from lithomargic clay deposits, Ceramic International volume 39, issue l, pages 779 - 783.



material

Uti/isation of lithomargic clay as a fireclay refractory materia/ ABSTRACT

Lithomargic clay until now has not been used to produce ceramic bodies due to its low plasticity. In this work, fireclay refractory bricks have been produced from lithomargic clay by adding Mfensi clay as a binder. Three different lithomargic — Mfensi clay formulations were prepared and fired at temperatures ranging from 1200 °c to 1400 °c. The effects of soaking time on the properties of the fired bricks were also investigated. The soaking times at 1350 °c and 1400 °c were varied at 30, 60, 90 and 120 minutes, respectively. The fired bricks were characterised based on their bulk density, apparent porosity, water absorption, linear firing shrinkage, and cold crushing strength values. The chemical and mineralogical compositions of the fired bricks were also determined.

The results show that the cold crushing strength increased with increasing firing temperature and binding content up to 1400 °c and 20 weight percent, respectively. The highest cold crushing strength recorded was 11.95 MPa at 1400 °c. The increase of highly refractory phases (cristobalite and mullite) and the densification of the bricks due to fluxing

agents were re>s1E-for the high cold crushing strength values. The linear firing shrinkage values were less than 11% which are within limits acceptable for refractory clays. The optimum properties were achieved at a firing temperature of 1400 °c for 1 hour. Effect of soaking time revealed that optimum properties could be obtained by firing either at 1350 °c for 120 minutes or 1400 °c for 30 minutes. The properties obtained in this work indicate that, fireclay aluminosilicate refractory bricks could be produced from lithomargic clay by addition of suitable clay binders. The fireclay produced could be used for low to high heat duty applications such as crucibles and kiln linings.

duty applications such as crucibles and kiln linings.

Adam A. Joseph (MSc. Thesis 2012).

Uti/isation of lithomargic clay as a fireclay refractory

Adam A. Joseph (MSc. Thesis 2012).

Page iv

LIBRARY

KWAME Nt<Rl.iMAH 'N'VERSWI or SCtENCE TECHNOtors

TABLE OF CONTENTS DECLARATION

| ••••• | i | |
|----------|---|-----------|
| ACKNO | WLEDGEMENTS | ii ::: |
| | ACT | |
| ABSIRA | ACT | |
| TABLE | E OF | CONTENTS |
| | | 1 |
| LIST OF | F TABLES | viii |
| LIST OF | F FIGURES | ix |
| CHAPT | ER ONE: INTRODUCTION | |
| 1.1 | Background | |
| 1.2 I | Problem Statement and Justification . | |
| 1.3 | Aim and Objectives | |
| 1.4 | Structure of Thesis | |
| CHAPTI | ER TWO: L <mark>ITERATURE REVIEW</mark> | |
| 2.1 | Introduction to Refractories | |
| 2.2 | Classification of Refractories | |
| | Alumina | 24 |
| 2.3 | - silicĐ_Brefractories | |
| 2.3.1 | l Fireclay refractories | |
| 2.32 | 2 Silica refractory | |
| 2.3.3 | 3 High alumina refractories | |
| 2.4 Fire | eclay Refractory-Materials | |
| 2.4.1 | l Clay | |
| 2.4.2 | 2 Lithomargic clay | |

| Uti/isation of lithomargic clay as a fireclay refractory materia/ |
|---|
| 2.5 Aluminosilicate Refractory Bricks ManufactureI |
| |

| 2.5.1 | Clay transformations | | 12 |
|-------|----------------------|--|----|
|-------|----------------------|--|----|





Uti/isation of lithomargic clay as a fireclay refractory

material

| 2.6 Cha | racterisation of Aluminosilicate Bricks13 | |
|--------------|--|--------|
| 2.6.1 | Apparent porosity 14 | • |
| 2.6.2 | Bulk density14 | • |
| 2.6.3 | Water of Absorption14 | - |
| 2.6.4 | Cold crushing strength 15 | |
| 2.6.5 | Pyrometric cone equivalent (PCE) |) |
| 2.6.6 | Thermal conductivity15 | ,) |
| 2.6.7 | Thermal stability |) |
| 2.7 Som | ne Properties of Fireclay Aluminosilicate Bricks |) |
| 2.8 Applicat | tion of Fireclay Aluminosilicate Refractory17 | |
| CHAPTER T | THREE: EXPERIMENTAL PROCEDURE | |
| 3.1 Intr | oduction19 |) |
| 3.2 Material | s and Material Preparation | |
| 3.3 C | haracterisation of Clay Samples |) |
| 3.3.1 Che | mical and mineralogical analysis 20 | |

- 3. 3.3—Determinati0TõfÃiGberg limits
 22

| 3.6.4 | Uti/isation of lithomargic clay as arefractory material4Determination of water of absorption |
|--------------|--|
| 3.6. | 5 Determination of cold crushing strength (CCS) |
| | Page vi fireclay |
| CHAPT 4.1 | ER FOUR: RESULTS AND DISCUSSION 28 Chemical Analysis28 |
| 4.2 | Mineralogical Analysis29 |
| 4.3 | Specific Gravity, Atterberg Limits and Moisture Content 30 |
| 4.4 | Surface Characteristics of Fired Bricks |
| 4.5 | Effect of Firing Temperature on Linear Shrinkage32 |
| 4.6 | Effect of Firing Temperature on Apparent Porosity and Water Absorption 34 |
| 4.7 | Effect of Firing Temperature on Bulk Density |
| 4.8 | Effect of Firing Temperature on Cold Crushing Strength37 |
| 4.9 | Effect of Firing Temperature on Mullitilisation38 |
| 4.10 | Effect of Soaking Time on Properties and Mineralogy of Fired Bricks |

| CHAPTER FIVE: CONCLUSION AND RECOMMENDATIONS |
|--|
| 5.1 Conclusion |
| 5.2 Recommendation |
| REFERENCES 49 |
| APPENDIXA: Si02 / A-tŒSÝSÎEM |
| TIMES |
| APPENDIX D : PLASTICITY TEST RESULTS OF CLAY SAMPLES INVESTIGATED |

Adam A. Joseph (MSc. Thesis 2012).

Page iX

material

LIST OF TABLES

| Table 2.1 Typical composition of aluminosilicate refractory (Calister, 2003) | . 6 |
|--|-----|
| Table 2.2: Typical composition and properties of Fireclay Bricks (Schacht, 2004) | 17 |
| Table 3.1: Formulation matrix from lithomargic (X) and Mfensi (X) clay samples | 22 |
| Table 4.1: Chemical composition of lithomargic and Mfensi clays | 29 |
| Table 4.2: Atterberg limits of Mfensi, Afari, Fosu and Lithomargic clays | 31 |
| Table 4.3: Average moisture content of moulding samples. | 31 |



fireclay

LIST OF FIGURES

| Figure 3.1: Flow diagram showing the processes involved in the production of refractory | |
|--|----|
| bricks | 19 |
| Figure 3.2: Temperature profile for firing bricks | 24 |
| Figure 4.1: XRD spectra of Mfensi (a) and Lithomargic (b) clay samples before firing 3 | 30 |
| Figure 4.2: Typical photograph of fired and unfired bricks | 32 |
| Figure 4.3: Effects of firing temperature on linear shrinkage. | 34 |
| Figure 4.4: Effect of firing temperature on apparent porosity. | 35 |
| Figure 4.5: Effect of firing temperature on water absorption | 6 |
| Figure 4.6: Effects of firing temperature on bulk density | 37 |
| 38 | |
| Figure 4.8: XRD patterns of fired bricks at various temperatures (a) Sample A (b) Sample | ; |
| B and (c) Sample C | 40 |
| Figure 4.9: Effect of soaking time on water of absorption | |
| Figure 4.10: Effect of soaking time on apparent porosity | 42 |
| Figure 4. I1: Effect of soaking time on bulk density | 43 |
| Figure 4.12: Effect of soaking time on cold crushing strength | 44 |
| Figure 4.13: Effect of soaking time on linear shrinkage | 45 |

Figure 4.14: XRD patterns of bricks fired at (a) 1350 °c and (b) 1400 °c at different





CHAPTER ONE

INTRODUCTION

1.1 Background

Refractories are the primary materials used by the metallurgical industry in the construction of all the internal linings of furnaces for melting, smelting in vessels, for holding and transporting molten metal and slag in furnaces. This is because refractories can withstand very high temperatures without physical and chemical deterioration (Lee, 2003). Aluminosilicate refractories are manufactured using refractory clays. They mainly are composed of kaolinite clay mineral (A120r2SiOr2H20). Refractory clays are usually contaminated with limited amounts of impurity oxides, including Ti02, Fe203, CaO, MgO and alkali oxides which act as fluxing agents at high temperatures (Schacht, 2004). According to the A1203—Si02 phase diagram (Aramaki and Roy, 1962), there exist two eutectic points at 5.5 weight percent A1203 and eutectic temperature of about 1595 °c and at 78 weight percent A1203 and eutectic temperature of about 1840 °c. The fluxing agents decrease theeutectic temperature-öf aluminosilicate refractories. However, the amount of fluxes is kept

at minimal to minimise their effect on the development of liquid phase at higher temperatures (Chesters, 1973).

Lithomargic clay underlying Awaso bauxite deposits in Ghana result from incomplete bauxitisation process. The chemical and mineralogical characteristics indicate that lithomargic clay consist mainly of kaolinite and gibbsite (Momade and Gawu, 2009). The Si02 (mainly from the kaolinite) and A1203 contents varies between 27-52% and 31-45%, respectively. The impurities include Fe203, Ti02, 1420, CaO, MgO and Na20 (Momade et.

Adam A. Joseph (MSc. Thesis 2012).

KWANE NKR(.iMAH 'NIVERS:TY or SCIENCE 8TECHNOL KUMAS I

LIBRARY

Page 1

al., 2004). The chemical composition suggests that lithomargic clays could be used as fireclay aluminosilicate refractories. Studies have also shown that this material is light in weight; has a soft texture, resistant to chemical and oxidation attack ((Momade and Gawu, 2009). However, production of refractory ceramic bodies from this clay has not been successful due to its low plasticity. To make this material usable, there is the need to add a binder to produce a mass which could be moulded into bricks to retain their shape during subsequent drying and firing. There are a number of possible local clays which have good plasticity and therefore could be used as a binder to improve the plasticity of lithomargic clay. One of such clays is Mfensi clay.

Mfensi clay sample has been investigated and found to possess good properties — about 41% clay and 47% free quartz; it has adequate plasticity (PI=33%) and good workability. It has been

used to produce pottery wares such as water coolers, grinding bowls and bricks (Nsiah, 2007). This clay therefore could be used as a binder to improve the plasticity of lithomargic cla to enable the use of this material. A mixture of Mfensi clay with ^{rgic clay} could resúlffi7aCrials of varying properties useful for the producton of __aluminosilicate refractory products. This thesis thus, concerns with the characterisation of lithomarge and Mfensi clays in the Western and Ashanti region of Ghana, respectively, with the aim of developing aluminosilicate refractory bricks from these minerals.

1.2 Problem Statement and Justification Adam A.Joseph (MSc. Thesis 2012). The amount of alumina and silica in a clay mineral determines its potential use as an aluminosilicate refractory raw material. Lithomargic clay has been found to possess high

content of alumina (up to 45.9%) as revealed by Momade and Gawu (2009). The high alumina content makes this clay suitable for use as fireclay aluminosilicate refractory. Nevertheless, this material is not usable at present due to its low plasticity.

Development of products which can be produced from a country's natural resources is very important as far as the industrialisation of a nation and saving foreign exchange is concerned. Presently, metallurgical industries in Ghana import most refractory-related consumables, as the demand cannot be met locally (Appiagyei, 1993). Thus the country expends a lot of foreign exchange importing refractories to meet the needs of local industries. A survey conducted by Appiagyei and Sraku (1993) and updated by Gawu and Momade (1996) on the use of clay and clay products in Ghana, revealed that a number of metallurgical industries import refractory materials from all over the world to line their furnaces. In Ghana the most commonly used refractories are aluminosilicate based. It is estimated that about 8,085,000 kg

of aluminosilicate refractories are consumed annually (Kisiedu et. al., 2008). The demand is expected to rise as a result of increased metallurgical industriesiwthe countrye-BúT6îúhe abundance of refractory clays in the country and the demand for refractories by industries, it is pertinent to develop and manufacture refractories locally using the locally available raw materials. One of such refractory clays is lithomargic clay.

1.3 Aim and Objectives

The main aim of this project is to produce dense fireclay aluminosilicate refractory bricksfrom

lithomargic clay using Mfensi clay as a binder. The specific objectives are as follows:

Adam A.Joseph (MSc. Thesis 2012).

A.

- Determination of the mineralogical and chemical composition of lithomargic and Mfensi clays.
- 2. Formulation and firing of lithomargic-Mfensi clay bricks at different temperatures . and soaking times.
- 3. Characterisation of fired bricks based on their bulk density, apparent porosity, water of absorption, linear firing shrinkage and cold crushing strength.

1.4 Structure of Thesis

The thesis is structured into five Chapters. Chapter one talks about the introduction and background of refractories in Ghana. This is followed by the problem statement, justification, aim and objectives of study. Chapter Two summarises the relevant literature in the area of study. Chapter Three describes the methodology used for the development of aluminosilicate refractory from lithomargic clay. The results and discussion of data are presented in Chapter

Four. Chapter Five summaries and provides conclusions to the study followed by sóme

BADY

recommendations as to how refractory bricks could be produced to meet

RAD RAD SANE

the needs of the local industries in the country.

Page 4

Uti/isation of lithomargic clay as a fireclay refractory material



Adam A.Joseph (MSc. Thesis 2012).

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction to Refractories

Refractories are a group of ceramic materials capable of withstanding high temperatures as well as abrasion and corrosive environments for prolonged periods of time. Thus, refractories are constructional materials with the ability of retaining their shape and strength against high temperatures under arduous conditions (Mohammed, 2009). Refractories are used for lining furnaces, ladles and reactors in any high temperature operation (Gupta, 2002). Their major function is to protect the wall of furnaces, reactors as well as confine or transfer heat without fusion.

2.2 Classification of Refractories

The range of refractories employed in industry is literally

enormous, and different classificatiorymethods exist.

Refractories have been variously classified by their properties,

chemical composition/characteristics, and physical forms (Nwajagu, 2005). On the basis of

-ehemical composition, the primary constituents of any

refractory may be a single or multiple compounds. Refractories

Adam Joseph (MSc. Thesis 2012). Page

Uti/isation of lithomargic clay as a refractory material in which the predominant constituents are alumina, silica or a combination thereof may be placed in the following categories: fireclay refractory, high alumina refractory and silica refractory.

Α.

2.3 Alumina - Silicate Rrefractories

On the basis of chemical composition, the primary constituents of any alumina - silicate refractory may be a single compound like alumina, silica or mullite, or a combination of these materials. Their melting points are as follows: silica (Si02) — 1723 °c; alumina (A1203) 2050 °c; and mullite (71.8 A1203, 28.2 Si02) 1996 °c. Aluminosilicate refractories are those which contain more than 25 percent A1203 and not more than 70 percent Si02. Depending on the content of Si02 and A1203, aluminosilicate refractories are subdivided

basically into semi-acid or semi-silica (15-25 wt% A1203), fireclay (25-45 wt% A1203),

and high alumina kinds (over 45 wt% A1203). Typical compositions for fireclay and high-

alumina fireclay refractories are listed in Table 2.1.

 Table 2.1 Typical composition of aluminosilicate refractory (Calister, 2003)

| Defus storm trues | | Composition (wt. 0/0) | | | | |
|-------------------|-------|-----------------------|-------|-------|-------|----------------|
| Refractory type | A120 | 3 Si02 Mgo | CaO | Fe203 | Ti02 | Porosity (0/0) |
| Fireclay | 25-45 | 50-70 0 - 1 | 0 - 1 | 0 - 1 | 1 - 2 | 10 -25 |

Adam A.Joseph (MSc. Thesis 2012).

Page 7

5

| 50-90 10-45 | 0 - 1 | 0 - 1 | 0 - 1 | 1-4 | 18 - 25 | |
|-------------|-------------|-------------------|---------------------|-------------------------|-----------------------------|-----------------------------------|
| | 50-90 10-45 | 50-90 10-45 0 - 1 | 50-90 10-45 0-1 0-1 | 50-90 10-45 0-1 0-1 0-1 | 50-90 10-45 0-1 0-1 0-1 1-4 | 50-90 10-45 0-1 0-1 0-1 1-4 18-25 |

The high alumina kinds are subdivided into sillimanite, mullite, mullite-corundum, and corundum (Krivandin and Markov, 1980). Metakaolin contains 46 wt% A1203, sillimanite 63% A1203 and mullite 71.8 wt% A1203. Fireclay refractories are the subject of this research work.

Α.

fireclay

2.3.1 Fireclay refractories

Fireclay refractories consist of alumina and silica with various amounts of silica ranging from less than 78% and containing more than 25% of A1203. Fireclay bricks or firebricks are made from refractory clays or fireclays which can withstand very high temperatures (above 1200

^oc) without becoming soft or melting. The preparation of clay, moulding, drying and burning of refractory are the same as the manufacture of ordinary building bricks (Rajput, 2004). However, more care and quality control is needed in the selection and processing of the raw materials for making firebricks. As the quantity of impurities increases the amount of A1203 decreases, the melting point of fireclay brick decreases. Fireclay bricks are extensively used in the iron and steel industry, nonferrous metallurgy, glass industry, pottery kilns, cement industry, and by many others, owing to its relative cheapness and widespread location of the raw materials used to manufacture firebricks.

Adam A.Joseph (MSc. Thesis 2012).

Uti/isation of lithomargic clay as a refractory material 2.3.2 Silica refractory

Silica refractoriés are well adapted to high temperature service because of their high refractoriness, high mechanical strength and rigidity at temperatures almost up to their – melting points (above, as well as their ability to resist the action of dust, fumes and acid slags. The American Society for Testing Materials (ASTM —C874) divides silica brick into Type A and Type B based on-the brick's flux factor. Flux factor is determined by adding the alumina content and twice the total alkali content. The Type A class includes silica brick with a flux factor of 0.50 or below; Type B includes all silica brick with a flux factor above 0.50. Both classes require that brick meet the following criteria: A1203 less than I .5%; Ti02 less than 0.2%; Fe203 , less than 2.5%; CaO less than 4%; and average

Α.

modulus of rupture strengths not less than 3.5 MPa. This system for classifying silica brick was preceded by a less exact system which still is referenced today. Under the earlier system, non- insulating silica brick were either of conventional or superduty quality. Insulating silica brick were classified only as superduty. Brick classified as superduty silica brick could not

contain more than a total of 0.5% alumina, titania and alkalies (Kirabira,

2005).

2.3.3 High alumina refractories

High alumina bricks serve as a multi-purpose refractory material for severe environments. They are used extensively in the steel industry for such applications as hot metal cars, electric furnace roofs, and muffles for a variety of furnaces. Most high alumina refractories are classified according to their alumina content, which could range from 50 to 99%. They are designated as 50%, 60%, 80%, and 90% alumina (ASTM 07-70). Two Adam A.Joseph (MSc. Thesis 2012). Page 9

Uti/isation of lithomargic clay as a fireclay refractory material classes of high alumina refractories are distinguished by a microstructure that is essentially a single, crystalline phase: mullite and corundum refractories.



Mullite is about 72% alumina with 28% silica. The manufacturing procedures are designed to maximise the formation of the mullite compound. A refractory with 71.80/0 alumina and 28.2% silica would be composed of only mullite if fired at equilibrium conditions. The 99% alumina class of refractories is called corundum. These refractories comprise single.phase, polycrystalline, alpha-alumina.

LIBRARY KWAME NKRUMAH INCVERSIIV OF KUMAS ISC'ENCE 8

fireclay

2.4 Fireclay Refractory Materials

2.4.1 Clay

Α.

The most common raw material for the manufacture of fireclay bricks (chamotte refractory bricks) is clay. Clay is a sedimentary material consisting of fine particles (under 2 microns in diameter) of crystallised aluminium hydrosilicates with a layered structure. The most important clay mineral for use in refractory is kaolinite (Appiagyei and Sraku-Lartey, 1993). Thus, aluminosilicate refractories are prepared from refractory clays whose principal constituent is kaolinite group (A120r2SiOr2H20). This group has three members (kaolinite, dickite and nacrite) having a formula of A12Si205 (01-1)4. They are formed under acidic condition of low temperature and pressure, by destruction of certain minerals such as feldspar in aluminosilicate rock by weathering as a consequence of the removal of Potassium (K), Adam A.Joseph (MSc. Thesis 2012).

Uti/isation of lithomargic clay as a refractory material sodium (Na), calcium (Ca), Magnesium (Mg) and ferrous by leaching and subsequent addition of Fe and H (Whitten and Brooks, 1972). The different minerals are polymorphs, meaning that they have the same chemistry but different structures. The general structure of the kaolinite group is composed of silicate sheets (Si205) bonded to

aluminium xide/hydroxide layer (Al₂(OH)₄) called gibbsite layers. Clays of this group are used as filler for paint, rubber and plastics and the largest use is in the paper industry that uses kaolinite to produce a glossy paper such as in used in most magazines.

2.4.2 Lithomargic clay

The formation of most lateritic bauxite deposit in the tropical and semi-tropical regions of . the world has been attributed to two major processes;





Adam A.Joseph (MSc. Thesis 2012).

- a) A direct transformation of aluminosilicate source rocks through interplay of physical, chemical and biological weathering processes resulting in a residual deposit rich in alumina.
- b) An indirect process characterized by weathering of source rocks through a clay mineral stage and after that a transformation to bauxite by a further removal of alkali, alkaline earth and silica.

The latter process normally results in accumulation of clays of various colours (lithomargic), below the bauxite separating it from underlying source rock (Gawu and Momade, 1996). This clay is composed of mainly kaolinite. The Si02 and A1203 contents of the lithomargic clay, according to the data presented by Momade et al (2004), varied between 27.0-38.3% and 31.3-45.9%, respectively. The impurities include 0.4-18.1% iron

(as Fe203), 1.0-2.2% Ti02, 0.1-1.8% potassium (as 1<20) and 0.01-0.19% sodium (as Na20). For clays high in alumina and silica, the most common application is in the refractory industry, where their desired properties come as a result of the presence of the mullite phase

which clevet6p"Ñ7ãn 1200 and 1450 °c. Further studies are being made

Do-other areas of application of this clay.

2.5 Aluminosilicate Refractory Bricks Manufacture

There are four basic forms in which refractories are manufactured: shaped products (bricks), unshaped products (monolithics), functional products and heat insulating products (Kirabira, 2005). The bricks are used to form the wall, arches, and floors of various high temperature equipment while the unformed compositions which include mortars, gunning Uti/isation of lithomargic clay as a fireclay refractory material mixes, castable (refractory concretes), and ramming mixes are cured in place to form functional products which are used mainly as tap and gas purging systems in steel manufacturing while heat-insulating are products for the refractory lining of thermal plants.

(Kirabira, 2005).

KNUST

Refractory manufacturing like any other conventional ceramic product goes through several stages. The major technical goals manufacturing a given refractory are embodied in its of properties, performance of the component intended application as well as shape and size requirements. The main aspects of manufacturing consist of choices materials, among raw processing methods and design parameters. The major insights manufacture have to do with the features of phase of composition and microstructure, technically known as material These are developed through processing and are character. themselves responsible for product properties and behaviour (Kirabira, 2005).

The fabrication rocess for refractories also depends on the particular combination of chemical compounds and minerals used to produce specified levels of thermal stability, corrosion resistance, and thermal expansion, among other property requirement. Refractory fabrication of shaped products involves five major processes: raw material Adam Joseph (MSc. Thesis 2012). Page

Uti/isation of lithomargic clay as a refractory preparing/processing, forming/shaping, drying, firing/sorting and packaging (Chester, 1973). Raw material processing involves crushing or grinding raw materials, classifying and/or grading by size, calcining, and drying. The materials are then mixed materials and formed into shapes other (for shaped with refractories) under moist or wet conditions.

Bricks are formed by mixing raw materials with water and/or other binders and pressing

| Α. | | | 11 |
|----|----------|----------|----|
| | fireclay | material | |

the mixture into a desired shape. After forming and drying, refractories are fired. Firing sometimes referred to as sintering or thermal treatment, involves heating the dry-formed material to high temperatures in order to achieve a ceramic bond. Firing of refractory materials results into a densified thermally stable structure, and bond development through partial vitrification, sintering and/or crystallization. This final process of firing gives raw materials their refractory properties and hence the properties of the final product (Kirabira,

2005).

2.5.1 Clay transformations

According to Velde (1992), there are four ranges of temperature which produce characteristic transformations in clay materials: the free-water dehydration range (or drying range) (50-120 °c); the clay stability range (120-600 °c); the anhydrous clay range (600 - 900 °C); and the re-crystallization range (above 900 °c)". In the manufacture of ceramics (bricks and tiles), the 600 — 1000 °c zone is of greatest importance in transforming the dried clay into a new, more

Adam A. Joseph (MSc. Thesis 2012).

Uti/isation of lithomargic clay as a fireclay refractory material rigid substance. In this range the interaction of the clay and non-clay additives occursúTÕiããminerals or physical states (glass or new crystalline

phases) In clays, an important volume change (shrinkage) takes place as they re-crystallize into other phases, losing their crystalline water above 1000 °c (Grim, 1968). This loss is important to the ceramic process. It brings the material into a stable thermal region. Shrinkage effect can be modified by the addition of sufficient non-clay materials called temper or grits. Sands of various types, pure quartz, alkali feldspar, grog (ground-up burned refractory materials), or chamotte (calcined and ground-up kaolinite or fireclay) are used depending on the quality and use of the ceramic product (Idenyi and Nwajagu, 2003).





All clay minerals, when heated to temperatures in excess of 1200 °c, are capable of being recrystallized to form the minerals mullite (3A120r2Si02), corundum (A1203), and, where Mg is present, olivine (Mg.Fe)2.Si04, while tridymite or cristobalite (Si02) results both from changes in the mullite composition, and from the incorporation of free quartz (Prentice, 1990).

The silica-alumina system, illustrates the relationship between these high temperature ceramic phases with temperature and composition as shown in Appendix A. However, the temperatures and times used in industrial brick production are inadequate for some of these changes to go to completion and fusion usually ceases at a stage of vitrification in which some of these minerals are beginning to form (Appiagyei, 1993). The development of the felted crystals of mullite is believed to be important in the production of strength in refractory bricks (Schacht, 2004).

2.6 Characterisation of-ÃGûG4ílicate Bricks

•be-quality of refractory material and its suitability for a particular application primarily depends on its physical chemical and mineralogical properties (Didier-Werke, 1982). It may be possible to assess the quality of a refractory on the basis of a single property or a group of properties (Dondi et. al., 2004). The most common properties that are considered in selecting the optimum refractory lining configuration are briefly discussed below.

LIBRARY KWAME NKRUMAH

TECHNO

Adam Joseph (MSc. Thesis 2012).

Uti/isation of lithomargic clay as a fireclay refractory material INPt'ERSt1V OF SCtENCE R KUMAS Ž

| А. | | | 13 |
|----|----------|----------|----|
| | fireclay | material | |
| | | | |

2.6.1 Apparent porosity

The apparent porosity is a measure of the volume of the open pores, into which a liquid can penetrate, as a percentage of the total volume. This is an important property in cases where the refractory is in contact with molten charge and slags. A low apparent porosity is desirable since it would prevent easy penetration of the refractory size and continuity of pores will have important influences on refractory behaviour. Large numbers of small pores are generally preferable to an equivalent number of large pores (Mazen, 2006).

2.6.2 Bulk density

A useful property of refractories is bulk density, which defines the material present in a given volume. An increase in bulk density of a given refractory increases its volume stability, its heat capacity, as well as resistance to slag penetration. For many refractories the bulk density

provides a general induction of the product quality (Nwobodo and Davies,

2000).

2.6.3 WatõÄbsorption

Water of absorption of a brick is defined as the weight of water in a brick expressed as a percentage of the brick's dry weight. It varies roughly from 4.5 to 21 % and the variation is mainly due to the variable raw material and the manufacturing process (Dondi et. al., 2004). The extrusion process in the manufacturing produces denser brick in comparison to the moulded bricks and denser bricks in turn would exhibit less absorption.

Adam A.Joseph (MSc. Thesis 2012).

2.6.4 Cold crushing strength

This is the products ability to resist failure under compressive load at room temperature. This property often serves as a guide for estimating other properties such as the degree of sintering, firing temperature and the purity of materials. The strength of refractories is also of value in determining the ability to withstand handling and transporting (Mohammed, 2009).

2.6.5 Pyrometric cone equivalent (PCE)

Temperature at which a refractory will deform under its own weight is known as its softening temperature which is indicated by PCE (Schacht, 2004). Refractories, due to their chemical complexity, melt progressively over a range of temperature. Hence refractoriness or fusion point is ideally assessed by the cone fusion method. The equivalent standard cone which melts to the same extent as the test cone is known as the pyrometric cone equivalent (PCE). The pyrometric cone equivalent indicates only the softening temperature. But, in service the refractory is subjected to loads which would deform the refractory aúmuch lowefGFRure than

that indicated by PCE. With change in the environmental conditions, such as reducing

atmosphere, the PCE value changes drastically

(Schacht, 2004).

2.6.6 Thermal conductivity

Thermal conductivity is defined as the quantity of heat that will flow through a unit area in a direction normal to the surface area in a defined time with a known temperature gradient — under steady state conditions across the area (Routschka, 2004). It indicates the general

SANE

Adam A. Joseph (MSc. Thesis 2012).

Uti/isation of lithomargic clay as a fireclay refractory material



Α.

Adam A.Joseph (MSc. Thesis 2012).

heat flow characteristics of refractories. Thermal conductivity depends upon the chemical and mineralogical compositions as well as the glassy phase contained in the refractory and the application temperature (Dondi et. al., 2004). The conductivity usually changes with rise in temperature. In cases where heat transfer is required though the brick work, for example in recuperators, regenerators, muffles, etc. the refractory should have high conductivity. Low thermal conductivity is desirable for conservation of heat by providing adequate insulation (www.Refractories/4.13.7.8 Classification ofRefractories.pdf).

2.6.' Thermal stability

The life of a refractory lining also depends on thermal stability especially in batch processes. An example is when the product is to be cooled suddenly or in heat exchanger. The resistances to change in temperature are determined by the coefficient of expansion, the elastic modulus and the dispersity of the particles of the materials. Materials with small coefficient of expansion, high values of elastic modulus and of heterogeneous dispersity are

more resistant to changes in temperature. Bigger particles accommodate more easily temperature changes whãæ; maller ones are more elastic (Budnikov, 1964).

2.7 Some Properties of Fireclay Aluminosilicate Bricks

The physical and chemical properties of a refractory product are characteristics which give

an indication of the products performance under specific operating conditions. Fireclay brick

are classed in standards based on their refractoriness or ability to withstand high temperatures

without shrinking and spalling (Schacht, 2004). The general composition and t>pical properties are given in Table 2.2.

Adam

| Uti | /isation of lithomar | gic clay as a fireclay | y refractory mate | erial |
|---------------|--------------------------|--------------------------|--------------------------|--------------------------|
| A Joseph (N | ISc 2012). | | | Page |
| Table 2.2: Ty | pical composition a | and properties of Fire | eclay Bricks (Sch | acht, 2004) |
| | Low Duty | Medium Duty | High Duty | Superduty |
| | 1000 ^o c Max. | 1200 ^o c Max. | 1400 ^o c Max. | 1600 ^o c Max. |
| | E.2 | N. T. T. | | |
| | / | emical Analysis | | |
| A1203 | 25.4 | 29.3 | 37.0 | 41.9 |
| Si02 | 68.1 | 62.9 | 57.8 | 53.2 |
| Fe203 | 1.5 | 2.3 | 1.3 | 1.0 |
| Ti02 | 1.5 | 2.9 | 2.3 | 2.2 |
| N O+K O | 1.5 | 1.5 | 1.3 | 1.2 |
| | Ph | ysical Properties | 10. A | |
| Bulk Density, | 2.00 | 2.10 | 2.11 | 2.35 |
| g/cm3 | | | | |
| Apparent | 19.0 | 20.5 | 18.0 | 12.5 |
| Porosity, % | | | | |
| Modulus of | 5.2 | 3.8 | 9.7 | 8.5 |
| Rupture, MPa | | 200 | 100 | |
| Crushing | 28 | 32 | 35 | 22 |
| Strength, MPa | | EN | 0.2 to 10.2 | |
| PLC, 5 hr. @ | | 2 1 5 | -0.2 10 +0.2 | |
| 14000C | | | | 0.0 to -1.2 |
| PIC 5 hr @ | | | | |



2.8 Application of Fireclay Aluminosilicate Refractory

Fireclay bricks are used principally in furnace construction, to confine hot atmospheres,

and to thermally insulate structural members from excessive temperatures (Callister, 2003).

They are used for the lining of many types of metallurgical furnace: blast furnaces, openhearth

furnaces, soaking pits, heat-treatment furnaces, and also for lining of steel-teeming-o ladles,

Uti/isation of lithomargic clay as a refractory chimneys, gas flues, steam boilers, regenerator checkers of coke ovens, among others

(Krivandin and Markov, 1980; Ruh, 1986). For fireclay brick, strength is not


ordinarily an important consideration, because support of structural loads is usually not required. Some control is normally maintained over the dimensional accuracy and stability of the finished product (Callister, 2003).



CHAPTER THREE

EXPERIMENTAL PROCEDURE

3.1 Introduction

This chapter describes the experimental techniques used in the development of fireclay aluminosilicate refractory bricks. The characterisation methods employed before and after the production of the refractory bricks have also been described. Figure 3.1 summarizes the various processing routes used.



Figure 3.1 : Flow diagram showing the processes involved in the production of refractory

bricks.

3.2 Material And Material Preparation

Lithomargic clay was taken from Awaso district near Sefwi-Bekwai in the Western region of Ghana. The clay which was used as a binder was taken from Mfensi village about 20km from Kumasi in the Ashanti region. Before selecting Mfensi clay as a binder, other clay deposits such as Afari clay and Fosu clay were tested for Plasticity Index. From the results

Adam A. Thesis 2012).

Joseph (MSc.

obtained, Mfensi clay was selected as a suitable binder material for lithomargic clay. The results for the plasticity index are presented in Chapter Four.

Both lithomargic and Mfensi clay samples were allowed to dry in an oven at 105 oc for 24 hours before processing. The dried clay samples were crushed to smaller particle sizes in a mortar to liberate the mineral constituents. Principal alkalis and dead organic matters were removed by washing i.e. soaking the crushed clay in water for 48 hours and then filtered off the water. This treatment was necessary since the presence of alkalis (Na and K) and organics are known to retard mullite formation and lower refractoriness and strength. They also increase porosity and shrinkage as they burn-off during firing; they also affect the colour of fired bricks (Udochukwu, 2007). The lithomargic clay was denoted as sample X, and that of Mfensi, Y.

- 3.3 Characterisation of Clay Samples
- 3.3.1 Chemical and mineralogical analysis

Lithomargic and Mfensi clay samples were analysed to determine their mineralogical composition and chemical constituents. The chemical composition analysis was carried out using the X-ray fluorescence spectrometer (XRF-Spectro X-LAB 2000). Samples were dried in an oven at a temperature of 110 °c for 1 hour, milled and sieved to allow the particles to a 106 µm seve before XRF analysis. Four grams (4 g) of each sample was pass through weighed with an electronic mass balance, homogenised and then pressed into pellets under 5,000 kg of load using a hydraulic press. The pellets were then analysed in the XRF spectrometer for 8 hours. Loss of ignition was determined by weighing two grams

Thesis 2012). Adam A.

(2 g) of each sample in a crucible and then heated in a furnace at 1050 °c for 3 hours. The samples were removed and cooled in a desiccator after which it was reweighed. Difference in weight was recorded as loss of ignition. The results of the chemical composition of the clays are presented in Chapter Four.

The mineralogical composition of lithomargic, Mfensi clays and fired bricks were determined using X-ray diffractometer (Siemens 1)5000) operating at 40 kV and 40 mA and using Cu Ka radiation. Samples were scanned at $10 - 50^{\circ}$ (20) and step size of 0.01 $^{\circ}$

The results of the mineralogical compositions are presented in Chapter Four.

3.3.2 Specific gravity tests

The specific gravity of the clay samples was determined as specified by the British Standard BS 1377:90. A 50 ml density bottle with the stopper on was dried in an oven and weighed as Ml. 25 g of the clay sample was oven-dried at 105 °c for 24 hours. The sample was transferred into the density bottle and the bottle with its contents and the stopper weighed again as M2.

The sample in the bottle was then covered with water of known specific gravity. With the stopper removed, the container was placed in a vacuum desiccator, and the air gradually evacuated. When no air was seen to be released, the bottle was removed from the desiccator and filled with water. The stopper was again inserted and the bottle immersed in a waEÿh-until a constant temperature of 25 °c was attained. The bottle was then wiped dry and weighed as M3. The bottle was afterwards emptied of its contents, cleaned and completely filled with water, stoppered and weighed as M4.

The specific gravity (Gs) was calculated as:

$$G_s = \frac{(m_2 - m_1)}{(m_4 - m_1) - (m_3 - m_2)}$$
[3.11]

3.3.3 Determination of Atterberg limits

Mfensi, Fosu and Afari clays were obtained and Atterberg limits determined for each to select a suitable binder for lithomargic clay. Liquid and plastic limit tests were determined from which the plasticity index (PI) were calculated. Clay samples were sieved using 425 pm sieve size and the undersize used for the tests. Tests were conducted according to ASTM D 4318. The results of this test are presented in Chapter Four.

3.4 Formulation, Moulding and Drying of Bricks

The lithomargic (X) and Mfensi (Y) clays were oven dried for 24 hours at 105 °c. The dried clay samples were ground into powder using a pestle and mortar. For each of the dried clays, three lithomargic-Mfensi clay formulations were prepared by mechanical agitation at varying proportions by weight as shown in Table 3.1



oulding the moisture content of the moulding mass was determined by quickly

obtaining a test sample and weighing it before and after drying at 105 °c for 24 hours. The

moisture content required to achieve a desired plastic state for the clays was calculated as follows:

Moisture content =
$$\frac{Wws-Wds}{wws} \times 100$$
 [3.2]

where Wws is the average weight of clay sample before drying and Wds is the average weight of clay sample after drying.

Samples for each batch (Table 3.1) were prepared by considering a bulk mass of 500 g. Batch samples were mixed with water, tempered and thoroughly worked into a plastic state. A total of 30 bricks of dimensions 5.2 cm x 5.2 cm x 5.2 cm were moulded.

Drying was done in open air for 24 hours followed by oven drying at a temperature of 105 °c for 48 hours. Gauge marks were made on samples of the moulded bricks to determine the drying and firing shrinkages. The shrinkage was determined by the relation

Where DS is percent drying shrinkage, AL is change in length after drying and L is the

original length of the bricks.

3.5 Firing

Moulded bricks were fired in a muffle furnace (HF2) after drying. The dried moulded bricks

were fired to 600 °c and soaked for 60 minutes at a heating rate of 2 °c per minute. This was

followed by firing to set temperature (1200 °c) at a slower heating rate of 1.8 °c per minute.

gird and a firectay refractory marerial

The bricks were sOáked at this temperature for I hour followed by cooling to room temperature. The temperature profile for firing is shown in Figure 3.2. Similar temperature profile was used to fire at set temperatures 1250, 1300, 1350 and 1400 °c, respectively.

To optimize the soaking time, bricks of composition C (i.e. 80% lithomargic — 20% Mfensi) were fired at 1350 and 1400 °c for 30, 60, 90 and 120 minutes, respectively and the physical properties measured. The mineralogical compositions were also determined.





Figure 3.2: Temperature profile for firing bricks.

SANE

Adam A. Joseph (MSc. Thesis 2012).

Page 29

3.6 Characterisation of Fired Bricks

The fired bricks were characterised based on surface appearance, mineralogical composition, linear firing shrinkage, apparent porosity, water of absorption, bulk density and cold crushing strength.

3.6.1 Measurement of linear firing shrinkage

The shrinkage properties of the fired bricks were determined by measuring both the green and fired dimensions of the 5.2 cm x 5.2 cm x 5.2 cm bricks, using a vernier calliper. The green dimensions were determined after oven drying for 48 hours. All three sides were measured and the average linear shrinkage was computed. Linear shrinkage for each side

was calculated as a percentage of the original green dimension using the relation:

LS = -X 100 [3.41 where LS is the linear firing shrinkage (0/0), Lg is the green length of the brick (mm) and Lf is the length of brick after firing (mm).

3.6.2 Determination of apparent porosity

The apparent porosity was determined in accordance with ASTM C 20-80. The fired bricks

were dried in an oven at a temperature of 105 °c for 12 hours. The dry weight in air (Wda)

of-each specimen was measured. The specimen was suspended in a vessel of boiling distilled

water for 20 minutes and allowed to cool to room temperature. The weight soaked in water

(wsw) was measured. •he specimen was then removed from the water and the



as

surfaces gently cleaned with a damp cloth and weighed again in air to determine the saturated weight (Wsa). The apparent porosity, AP, of each brick was calculated using the following relation:

$$AP(\%) = \frac{W_{s}}{W} \frac{W_{sa}-W_{da}x}{100}$$
sa sw
[3.5]

3.6.3 Determination of bulk density

The bulk density (Db) was calculated for each test specimen using the results of the

apparent porosity test. The following equation was used:

Db(g/Cm3) = Wda

[3.61

wsa-wsw

3.6.4 Determination of water of absorption

The water of absorption (Wa) was calculated for each test specimen using the result of the

WJSANE

apparent porosity as follows:

 $\frac{Wsa}{W_a(\%)} = Wda \times 100$

[3.7]



Uti/isation of lithomargic clay a fireclay refractory material 3.6.5 Determination of cold crushing strength (CCS)

TÉTIVed bricks were positioned between the stationary plates of the universal testing machine which applied an axial compressive load on the brick. The load to failure was

registered on the gauge. The average load for each batch was taken from 5 samples and



Adam A. Joseph (MSc. Thesis 2012).

Page 32

Uti/isation of lithomargic clay as a refractory material

noted as Lav (N). The cold crushing strength (CCS) was computed by taking the ratio of the

2

average load to the cross sectional area, A (mm) of each sample as follows:



CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Chemical Analysis

The result of the chemical analysis by the X-ray fluorescence is shown in Table 4.1 The results show that the chemical compositions of both clays have A1203 and Si02 as major constituents forming over 70% of the total chemical composition. The Mfensi clay had higher Si02 content than lithomargic clay. The minor constituents were Fe203, CaO, MgO, K20, Na2() and Ti02. The chemical composition suggests that lithomargic clay could be used to produce fireclay aluminosilicate refractories. The organic matter content of Mfensi clay was higher (10.9%) than lithomargic clay (3.1%). The higher organic content of the binder could improve densification of the bricks at firing temperatures as the organic matter burn-out.



Adam Joseph (MSc.

2012).

| | Constituent | Lithomargic clay (X) | Mfensi clay (Y) | |
|---|-------------|----------------------|-----------------|--|
| _ | Si02 | 50.3 | 65.6 | |
| | A1203 | 42.2 | 19.5 | |
| | Feo | 0.4 | 0.8 | |
| | CaO | 0.2 | 0.2 | |
| | Mgo | 0.4 | 0.4 | |
| | K20 | 0.5 | 0.9 | |
| | MnO | 0.3 | 0.2 | |
| | | 0.4 | 0.9 | |
| | Ti02 | 1.4 | 0.6 | |
| | LOI | 3.9 | 10.9 | |
| 0 | | | | |

Table 4.1: Chemical composition of lithomargic and Mfensi clays

4.2 Mineralogical Analysis

A.

Figure 4.1 shows the XRD diffractogram of Awaso lithomargic and Mfensi clay samples.

An analysis of the lithomargic clay peaks revealed the presence of two major crystalline

phases which were kaolinite and gibbsite (minor fractions). The mineralogical composition

of the Mfensi clay consisted mainly of quartz with minor fractions of kaolinite.





Figure 4.1 : XRD spectra of Mfensi (a) and Lithomargic (b) clay samples before firing

4.3 Specific Gravity, Atterberg Limits and Moisture Content

The specific gravities of Mfensi and lithomargic clays determined using British Standard

BS 1377:90 were found to be 2.56 and 2.38 g/cm³, respectively. The higher density of

Mfensi clay recorded is as a result of its higher quartz content which was evident in the XRD

results shown in Figure 4.1. The relatively low density of lithomargic clay makes it suitable

SANE

for the production of lightweight fireclay refractories.

Table 4.2 shows the results of the Atterberg limits determined for Mfensi, Afari, and Ofosu

clays. An Atterberg limit of pgmargic clay has been added for comparison. The detailed

Adam Joseph

2012).

Uti/isation of lithomargic clay as a fireclay refractory material calculations and graphs are presented in Appendix B. The plasticity index (PI), except for

lithomargic clay, falls within the range 10-60% recommended for ceramic clays (Nnuka

and Enejor, 2001). Severe shrinkage during drying and firing of refractory bricks are not



desirable and must be reduced to the barest minimum. According to British Standard, BS 1377-7, for low shrinkage property, the PI must be within 10 to 20%. Hence Mfensi clay was selected as the suitable binder for lithomargic clay.

| Table 4.2: Atterberg limits of Mfensi, Afari, Fosu and Lithomargic clays | | | | | | |
|--|-------------------|--------------------|-----------------------|--|--|--|
| Clay type | Liquid Limit (LL) | Plastic Limit (PL) | Plasticity Index (PI) | | | |
| Mfensi | 43.62 | 21.72 | 21.90 | | | |
| Afari | 60.17 | 27.55 | 32.62 | | | |

| Fosu | 51.76 | 27.79 | 23.98 |
|------------------|-------|-------|-------|
| Lithomargic clay | 32.60 | 24.60 | 8.00 |

Table 4.3 shows the moisture content required to give a moulding mass of suitable plasticity for each of the clay mix. Samples A, B, and C required 6.20, 6.44, and 6.48% of moisture content, respectively. Thus the higher the binder content the higher the moisture content required. This is due to the high PI of Mfensi clay.

Table 4.3: Average moisture content of moulding samples.

refractory material Uti/isation of lithomargic clay as a Moisture weight (g) drying Sample ID Initial Weight after Moisture (g) con tent (g) content (0/0)A 6.20 \$0:00 46.90 3.10 _50.00 46.78 3.22 6.44 46.76 50.00 6.48 3.24 C Page



Adam Joseph

4.4 Surface Characteristics of Fired Bricks

After firing at set temperatures of 1200, 1250, 1300, 1350, and 1400 °c, respectively, the colour of the green bricks turned from white to gray-white as shown in Figure 4.2. The grayish white is an indication of low Fe203 content in the starting clay which is evident from the XRF analysis in Table 4.1. There were no observed cracks after firing for the different compositions at all temperatures. The absence of surface cracks after firing could be due to prolonged drying of the bricks (Wen and Nan, 2010). At lower temperatures (1200 and 1250 °c) the surfaces of the fired bricks were powdery for all compositions. Fireclays are usually associated with light colours such as gray, buffs, cream and white

(Chester, 1973).



Unfired

Fired

Figure 4.2: Typical photograph of fired and unfired bricks.

4.5 Effect of Firing Temperature on Linear Shrinkage

Figure 4.3 shows the effect of firing temperature on the linear firing shrinkage for the—

various formulated bricks. The linear firing shrinkage could be used as direct measure of

extent of densification. The linear firing shrinkage increased gradually from



Page

Adam A. Joseph (MSc. Thesis 2012).

39

Utilisation of lithamargia alou 28 2 firaday refractory material

1200 °c to 1250 °c, increased sharply from 1250 °c to 1300 °C and then increased again gradually from 1300 to 1400 °c for all formulations. That is, three stages of densification could be observed. The first stage (1200 °c to 1250 °c) was due to pore removal by vitrification and conversion of meta-kaolinite to mullite as confirmed by the XRD analysis (Figure 4.8). The second stage (1250 °c to 1300 °c) was due to the volume expansion that accompanied quartz to cristobalite phase transformation which resulted in pore closure (Carniglia and Barna, 1992). At the final stage of densification .(1300 °c to 1400 °c) grain grows as full density was reached (German, 1985). The final vitrified body was densed and contained primary mullite that was formed during vitrification, secondary mullite that was formed by precipitation of the liquid during cooling, solidified cristobalite and filler quartz from the binder material (Figure 4.8). It can be observed that there were no marked difference in the linear firing shrinkage values at 1350 °c and 1400 °C. The linear firing shrinkage values were slightly higher for the sample C followed by B and A, respectively. This is mainly attributed to the higher binder content; the higher the binder content the higher the shrinkage value. The linear firing shrinkage values were less than 11% for all formulations and therefore make it suitable



Page



Figure 4.3: Effects of firing temperature on linear shrinkage.

4.6 Effect of Firing Temperature on Apparent Porosity and Water Absorption Figure 4.4 shows the effect of firing temperature on the apparent porosity of investigated bricks. Apparent porosity depends on firing temperature and the formation of channels and pores within the body of the brick due to liberation of gases. These two factors have counter

effects because the reaction products resulted from the firing of the clay form a glass phase which fills the pores inside the body of the bricks. This results in reduction of pore ratio (Junse, 1994). It is observed that apparent porosity decreased with increased temperature. Sample C had relatively lowest apparent porosity values at all firing temperatures followed by sample B and A, respectively. The low apparent porosity of sample C is attributable to its high densification due to its high clay binder content.



LIBRARY

KWAME NKRUMAH IN'VERSITY OF SCIENCE & TECHNOLOGs

Page

Uti/isation of lithomargic clay as a fireclay refractory material



KUMAS E



42Adam A. Joseph (MSc. Thesis 2012).

Page



Figure 4.4: Effect of firing temperature on apparent porosity.

The water absorption depends on the apparent porosity and therefore similar behaviour was observed as shown in Figure 4.5. The water of absorption decreased with increased firing temperature. This is due to the formation of glassy or refractory phases resulting in shrinkage and closure of pores (Karman, 2006). The formation of these glassy and refractory phases

could support the reasons for an increase in cold crushing strength of the bricks as discussed under section 4.8. Thus, bricks fired at higher temperature are resistant to moisture penetration. The relatively lower water of absorption values obtained in this study at 1400 °c

meets the requirement of fireclay bricks according to the Turkish standard

(Karman, 2006).







Uti/isation of lithomargic clay as a fireclay refractory materia/

Figure 4.5: Effect of firing temperature on water absorption.

4.7 Effect of Firing Temperature on Bulk Density

Figure 4.6 shows the effect of firing temperature on the bulk density for the various formulated bricks. The bulk density increased as firing temperature increased from 1200 to 1400 °c for all formulations. The bulk densities increased sharply from 1200 to 1300 °c due to the formation of high viscosity siliceous phase which facilitates precipitation of primary mullite (German, 1985). The fraction of this liquid phase depends on batch composition and

firing temperature. Within the temperature range investigated, sample C with the highest binder content recorded the highest bulk density followed by sample B and sample A, respectively. Above 1300 °c most of the A1203/Si02 have transformed—into-nîüllite therefore slight change in densities were observed. The samples attained highest densities at 14000C where complete mullitisation has occurred. The sample C recorded the highest bulk density of 3.69 g/cm³ followed by

as



Uti/isation of lithomargic clay a fireclay refractory material sample B (3.65 g/cm3) and sample A (3.53g/cm3), respectively. The formation of a glassy phase caused by the presence of fluxing agents and alkali oxides (CaO, MgO, Na20, K20) helped improved densification of the fired bricks. The liquid glassy phase increased gradually by increasing sintering temperature. The glassy phase then fills the void spaces of the bricks increasing the density of bricks.



Figure 4.6: Effects of firing temperature on bulk density.



Uti/isation of lithomargic clay as a fireclay refractory materia/ 4.8 Effect of Firing Temperature on Cold Crushing Strength

Figure 4.7 shows the effect of firing temperature on the cold crushing strength of the various formulated bricks. The results indicate that absolute cold crushing strength values increased strongly with increasing firing temperatures. The cold crushing strengths

of the fired bricks are influenced by their phase composition. The fired bricks densified on firing within the temperature range of 1300 to 1400 °c. The increase of the highly refractory phases (cristobalite and mullite) and the densification of the bricks due to

the presence of flux agents improved the cold crushing strength of the bricks. Additionally, the presence of pores in the fired bricks affects the strength by reducing the cross-sectional area exposed to an applied load (Chukuogo, 1984). Porosity was less at high firing temperature and at high binder content; hence the high strength values.





46Adam A. Joseph (MSc. Thesis 2012).

Page



Figure 4.7: Effects of firing temperature on compressive strength.

4.9 Effect of Firing Temperature on Mullitilisation

Figure 4.7 shows the XRD patterns of samples fired at different temperatures for 1 hour. In the samples fired at 1200 °c, no mullite was formed. The crystalline phase present was mainly quartz. At 1250 °c, mullite starts to form with cristobalite as associated phases. Quartz was still evident at this temperature. However, for samples fired at 1400 °c, the

quartz phase strongly decreased with associated increase in cristobalite and mullite peak

intensities. The presence of mullite in fired bricks is as a result of the transformation of

kaolinite at high temperatures (Hamidouche et. al., 2002). Djangang et. al. (2008) showed

WJ SANE NO



that excess silica in the raw material mixture partially transforms to cristobalite beginning at 1300 °c and above. Mullite peaks are well developed at firing temperatures of 1350 and 1400 °c. This is very satisfying since the presence of mullite is an important factor due to its high temperature stability and excellent mechanical properties (Chen and Tuan, 2001).





(C) 1400°C M



Figure 4.8: XRD patterns of fired bricks at various temperatures (a) Sample A (b) Sample

B and (c) Sample C.

| | TRARY | |
|------------------------------------|---|---------|
| Adam A. Joseph (MSc. Thesis 2012). | KWAME NKRUMAH INIVERSITY OF SCIENCE & TECHNOLOG KUMAS I | Page 40 |

and a firecity refractory materia

4.10 Effect of Soaking Time on Properties and Mineralogy of Fired Bricks Based on the good combination of properties of bricks fired at 1350 and 1400 °c for I hour, further studies was carried out to determine the effect of soaking time on the properties measured at these set temperatures. Properties measured included water absorption, bulk density, linear firing shrinkage and cold crushing strength. Figures 4.9 to

4.13 show the results obtained.

The water absorption of fired bricks decreased as the soaking time increased (Figure 4.9). The decrease was higher for bricks fired at 1350 °c than 1400 °c. The bricks fired at 1350 °c decreased from 8.5% to 6.5% whereas bricks fired at 1400 °c decreased from 6.56% to 5.82%. Prolonged soaking time aided the formation of glassy phases which acts as cement to bind the mass together thereby decreasing the water of absorption.



Figure 4.9: Effect of soaking time on water of absorption.

Sec. 1

Similar behaviour was observed for the apparent porosity values (figure 4.10). At 1350 °c, the average apparent porosity decreased from 3.12 to 2.34 % as the soaking time increased. There was a sharp decrease from 60 to 90 minutes. This is mainly attributed to the expansion that accompanied quartz to cristobalite polymorphic transformation that occurs at such high temperatures, and for longer soaking times leading to closure of pores (Chester, 1973; Carniglia and Barna,1992, Routschka,1997). The decrease in porosity reduces the volume of the bricks whiles improving the mechanical properties (Kingery et. al., 1976). At 1400 °c, the average apparent porosity decreased from 2.4% to 2.2% after 60 minutes soaking time thereafter remained constant. At 120 minutes soaking time, the average apparent porosity was lower (2.19%) than bricks fired at 1350 °c (3.34%).



Figure 4.10: Effect of soaking time on apparent porosity



Uti/isation of lithomargic clay as a fireclay refractory materia/

Figure 4.11 shows the effect of soaking time on the bulk density. There was a marginal increase in bulk density from 3.62 g/cm^3 to 3.66 g/cm^3 when the soaking time increased from 30 to 60 minutes at 1350 °c; thereafter, the bulk density remained almost constant.



On the other hand, the bulk density of bricks fired at 1400 °c remained almost constant after 30 minutes soaking time. Thus slight density variation could be observed for bricks fired at 1350 °c and 1400 °c. This is because above 1300 °c most of the A1203 / Si02 have transformed into mullite therefore densification is minimised. The transformation depends on both temperature and soaking time. Hence, after 60 minutes of soaking time, the average bulk density remain almost constant indicating little or no further of glassy phase (German, 1985).



Time (minutes)

Figure 4.11 : Effect of soaking time on bulk density.

SANE

Figure 4.12 shows the effect of soaking time on cold crushing strength (CCS). The cold crushing strengthpfbricks fired at 1350 °c showed an increase from 11.02 MPa to 11.81 MPa after soaking for 30 and 60 minutes, respectively. Thereafter, there was a gradual

instrength at 90 (11.90 MPa) and 120 minutes (11.96 MPa). The strength of

Adam A. Joseph (MSc. Thesis 2012).

90

120

Uti/isation of lithomargic clay **as** a fireclay refractory materia/ bricks fired at 1400 °c remained almost the same (11.97 MPa) at the various soaking times. This could be attributed to the complete formation of refractory phases leading to a

constant strength. It could be explained that the cold crushing strength values at such high temperatures is influenced more by enhanced densification rather than phase composition. This is evident from Figures 4.13 and 4.14.



Figure 4.12: Effect of soaking time on cold crushing strength

Adam A. Joseph (MSc. Thesis 2012).

Page 54

Uti/isation of lithomargic clay a fireclay refractory material Figure 4.13 shows the effect of soaking time on the linear shrinkage of investigated bricks. Whereas the linear firing shrinkage values remained almost the same as the soaking time increased for bricks fired at 1400 °c, there was a gradual increase in shrinkage values for bricks fired at 1350 °c. However, the shrinkage value was about the same at 90 and 120 minutes soaking times. The shrinkage values observed at higher firing temperature is due to volume expansion that accompanied uart to cristobalite phase transformation. At such higher temperatures, nearly full densification would have been achieved at shorter soaking time; hence slight change in shrinkage values obtained.



Figure 4.14 compares the XRD patterns of bricks fired at 1350 °c and 1400 °c at different soaking times. It is interesting to note that there were no significant differences in crystalline phases present as the soaking time increased for the different firing temperatures. For both

Adam A. Joseph (MSc. Thesis 2012).

Page 55

Uti/isation of lithomargic clay as a fireclay refractory materia/ temperatures mullite, quartz and cristobalite were present. It is observed that as the soaking time increased, the crystallinity of the quartz phase increased, especially from 30 to 60 minutes. Even though the crystalline phases were the same as soaking time increased, the measured strength varied. This is particularly the case for bricks fired at 1350 °c. Thus the increase in strength as soaking time increases is mainly due to improved densification and not formation of crystalline phases at such high

temperatures.

Adam A Jacob (MCc Thesis 2012).



Page 45



Page 56



Uti/isation of lithomargic clay a fireclay refractory material

Figure 4.14: XRD patterns of bricks fired at (a) 1350 °c and (b) 1400 °c at different

soaking times.





Page 57

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The suitability of the lithomargic clay as a raw material for the production of fireclay refractory bricks was tested. Three lithomargic — Mfensi clay formulations were prepared, moulded, dried and fired at different temperatures ranging from 1200 °c to 1400 °c for 1 hour. The addition of a clay binder improved the plasticity of the lithomargic clay allowing ceramic bodies to be formed. Good combination of properties was obtained by addition of Mfensi clay. The linear firing shrinkages observed were less than and therefore make it suitable for refractory material. The apparent porosities were less than 4% for bricks fired at 1400 °c. The bricks fired at 1400 °c show accepted chemical and phase composition as well as densification, and cold crushing strength properties. The optimization of the soaking time at 1350 °c and 1400 °c for a soaking time of 30 minutes or at 1350 °c for a soaking time of 120 minutes to achieve optimum strength. The second option is desirable in situation where access

to high temperature furnaces become a constraint. The fireclay bricks developed in this work

could be used as low to high heat duty applicationy such as crucibles and kiln linings.

5.2 Recommendations

Mfensi clay was used in this investigation as a binding material to determine the optimum condition for the fireclay bricks, other clays such as the Fosu, Afari, and Mankranso should

SANE NO
be used in varied proportions, their physical properties determined and the results compared to the results obtained in this work to determine the best binding clay for the production of fireclay bricks.

Research into other physical properties such as the Thermal Shock Resistance, Thermal Conductivity, Pyrometric Cone Equivalence (PCE) and Modulus of Rapture should be carried out to determine possible areas of application of the firebricks produced in this work. Also to determine some properties under us.

Business men and government stakeholders should consider investing into the production of refractory bricks to service the local metallurgical industries in the country. However cost benefit analysis should be looked at.



REFERENCES

Appiagyei, A. K. Sraku-Lartey, K. (1993). Characterisation of some Ghanaian clay to

determine their suitability for the production of aluminosilicate refractories.

Proceedings for national seminar on current development in the mineral industries of

Ghana, Kumasi.

AdamA. Jossep. (MSc. Thesis 2012).

ireciay retractory material

Appiagyei, A. K. (1993). Development of aluminosilicate refractories from local raw materials, MPhil Thesis, Kwame Nkrumah University of Science and Technology,

Kumasi, pp. 57-71

Aramaki, S., Roy. R. (1962). Revised phase diagram for A1203-Si02, Journal of the American Ceramic Society, 45, pp. 229.

American Society for Testing and Materials, (1982). Annual Book of ASTM Standards, Part

17: Refractories, Glass, Ceramic Materials, Carbon and Graphite Products.

Blanchart P., Njopwouo D, Elimbi A., Melo U. C., Lecomte G. L, Nkoumbou C., Soro J., Bonnet J. P., (2008). Sintering of clay — chamotte ceramic composites for refractory bricks, Ceramic International, 34, pp. 1207–1213.

British Standard Testing of Engineering Ceramics, (1989). BS 1377 Section 1.2.





Tireclay refractory material

Budnikov, P.P. (1964). The technology of ceramics and refractories MIT press Cambridge

Callister, JR. W.D. (2003). Materials Science and Engineering: An Introduction, Sixth Edition. New York: John Wiley. pp. 384-410, 425-443

Carniglia, S.C., Barna, G.L. (1992). Handbook of Industrial Refractories Technology,

Noyes, Park Ridge, N.J

Chen, C.Y., Tuan, W. H. (2001). Processing of kaolin powder compact, Ceramic International, 27, pp. 795 — 800

Chester, J. H. (1973). Refractories, Production and Properties the Iron and Steel Institute, London, pp. 295-315

Chukuogo, C.E.B. (1984). Physical and chemical properties of some Nigerian clays, Research

and quality control. DSC, wam Nigeria.

Didier-Werke A.G., (1982). "Refractory materials and their properties", Refractory

Techniques, D-6200 Wiesbaden, Germany.

Djangang, G.N., Elimb, A., Melo, U.C., Lecomte, G.L., Nkoumbou, C., Soro, J., Bonnet,

J.P., Blanchart P., Njopwouo D, (2008). Sintering of clay — chamotte ceramic composites for refractory bricks, Ceramic International, 34, pp. 1207–1213.

BA

Adam A. Joseph (MSc. Thesis 2012).



K ('MASt

Dondi M. Marsigli M., Ventury 1., (2004). Microstructure and mechanical properties of clay

bricks: comparison between fast firing and traditional firing, Journal of

Materials in Civil Engineering, 16 (l), pp. 8-14

Gawu s. k.y and Momade 1996

German R.M., (1985). Liquid Phase Sintering, Plenum, New York

Grim, R. E., (1968). Clay mineralogy, second edition New York: McGraw-Hill. pp. 34-39

Gupta, R.B. (2002). Foundry Engineering, Smt. Sumitra Handa, New Delhi, India, pp. 534.

Hamidouche, M., Madjoubi, M.A., Loucif, K., Osmani, Fl., Kolli, M., Gonon, M., (2002).

Processing and characterization of refractory made of Algerian kaolin, in:

Proceedings of the Eighth Conference on Ceramics, INSA de Lyon, France, pp. 3—5.

Introduction to Refractories, www.cosmile.org/Manual/pdf/chapter12.pdf

[Accessed I1 Feb., 2012].

Junse K., (1994).^{4). Production of high-grade,} fine-porous brick material. Ziegel Industries

International 3: pp 152-153

Adam A. Joseph (MSc. Thesis 2012).

Idenyi, N.E. Nwajagu, C. O. (2003). Non-metallic Materials Technology, Enugu: Olicon Publications, pp. 1-47.

Karman, S., (2006). Firing temperature, firing time influence on mechanical and physical properties of clay, Journal of Scientific and Industrial Research, 65, pp. 153-159

Kingery W.D., Bowen H.K., Uhlmann DR., (1976). Introduction to Ceramics, 2nd edn., Wiley, New York.

Kesse, G.O., (1985). The Mineral and Rock Resources of Ghana, Balkema Publ., Rotterdam,

pp. 95-103.

Kirabira, J. B., (2005). Properties of Ugandan mineral and fireclay refractories Doctoral Thesis, KTH, Materials science and Engineering, Stockholm, Sweden.

Kisiedu, A.K, Tetteh, D.M.B. Obiri, H.A. Brenya, E.F., Ayensu, A. (2008). Development of

aluminosilicate refractories in Ghana, Journal of Applied Science and Technology, 13,

pp. 72**-75**.

Krivandin, V. A., Markov, B. L., (1980). Metallurgical Furnaces (Translated from the

Russian by V. V. Afanasyev) Moscow: Mir Publishers, pp. 229-261

Lee, W. E. (2003). Refractories, University of Sheffield, UK.

Adam A. Joseph (MSc. Thesis 2012).

Uti/isation of lithomargic clay as a fireclay refractory materia/



Adam A. Joseph (MSc. Thesis 2012).

Page 64

Mazen Al-Amaireh, (2006). Improving the physical and thermal properties of the fireclay

refractory bricks produced from bauxite, Journal of Applied Science, 6 (12), pp.

2605 - 2610

Mohammed A. A. (2009). Mechanical and Physical Properties of Silica Bricks Produced from Local Materials: Australian Journal of Basic and Applied Sciences, 3(2) pp. 418-423

Momade, F.W.Y. Gawu, S.K.Y. Affam, M. (2004). Characterization of Lithomargic Clay from the Awaso Bauxite Deposit. Paper presented at the 90th Anniversary International Conference and Exhibition of the Geological Survey Department of Ghana, International Conference Centre, and Accra.

of

Momade F.W.Y. Gawu S.K.Y., (2009). Geochemical and mineralogical characteristics of

lithomargic clay types from Awaso bauxite deposit, Ghana: implications for possible

industrial utilization, Journal of Science and Technology, 29, pp. 386—392.

SANE

Nnuka E.E., and Enejor C., (2001). Characterization of Nahuta Clay for Industrial and

Adam A. Joseph (MSc. Thesis 2012).

Uti/isation of lithomargic clay as a fireclay refractory materia/ Commercia ApplicationPEMA3) pp. 9-13

Nsiah, J.K. (2007). The study of Mfensi clay in the Ashanti region of Ghana, Ghana

Journal of Science, 47, pp. 123 - 129

Nwajagu, C. O. (2005). Foundry Theory and Practice (Abridged Edition). Enugu: Olicon Publications, pp. 56 — 76.

Nwobodo, C. S. Davies, T. J. (2000). The Effect of Apparent Porosity on the Modulus of

Rupture of Alumina-Chromia Refractory Matrix A Paper Presented at FUTO 2000,

Annual Conference of NAMMES, pp. 8.

Prentice, J. E. (1990). Geology of Construction Materials; Tropics in the Earth Sciences London: Chapman and Hall, pp. 139-170.

Rajput, R. K. (2004). Heat and Mass Transfer in S.I. Units, 2ndEdition New Delhi: S.

Chand and Co. Ltd. pp. 1-21

Routschka, G. (Ed.). (2004). Pocket manual—Refractory Materials: Basics, Structures and

Properties. 2nd Edition Vulkan-Verlag Essen.

Ruh, E. (1986). Metallurgical Refractories: Manufacture and Properties. In BEVER, M.B.

(ed.), Encyclopedia of Materials Science and Engineering, vol. 4. Oxford: Pergamon

Uti/isation of lithomargic clay as a fireclay refractory materia/ Press.

Schacht A. Charles, (2004). Refractories Handbook, Marcel Dekker, Inc.

Udochukwu M. (2007). Investigation of some refractory properties of kaolinite clay from four different locations in Abia state, Nigeria, Masters Thesis, Federal University of Technology, Owerri.

Velde, B. (1992). Introduction to Clay Minerals-Chemistry, Origins, Uses and

Environmental Significance London: Chapman and Hall, pp. 1-3

Wen, Y., Nan, L, (2010). Effects of sintering temperature on pore characterisation and strength of porous corundum-mullite ceramics. Journal of ceramics processing research Science Publishers Ltd. pp. 55.

Whitten D.G.A and Brooks J.R.V., (1972). A dictionary of Geology, Penguin Books Ltd. Harmondsworth, Middlesex England, pp. 84-85



Adam A. Joseph (MSc. Thesis 2012).

APPENDIX A

Si02 / A1203 PHASE DIAGRAM





20 40 60 80

Adam A. Joseph (MSC. Thesis 2012).

Uti/isation of lithomargic clay as a fireclay refractory materia/ SiO2 Mol. ⁰/0 A1203

Figure Al: Phase diagram of Si02 / A1203 system.



Table B2: Bulk densities (g/cm³) at various temperatures

| | A(90/10) | 13(85/15) | C(80/20) |
|---------------------|---------------|---------------|---------------|
| 1200 ^o c | 3.33 ± 0.09 | 3.41 ± 0.01 | 3.42 ± 0.06 |

Adam A. Joseph (MSc. Thesis 2012).



Table B3: Water of absorption (0/0) at various temperatures

| | A 90/10 | B 85/15 | c 80/20 |
|---------------------|---------------|---------------|-------------|
| 1200 ^o c | 32 ± 0.198 | 29 ± 0.10 | 27 ± 0.16 |
| 1250 ⁰ c | 31 ± 0.28 | 25 ± 0.34 | 22±0.13 |
| 1300 ^o c | 29 ± 0.07 | 23 ± 0.19 | 21 ± 0.03 |
| 1350 ^o c | 14 0.04 | 9±0.01 | 8 ± 0.006 |
| 1400 ^o c | 11 ± 0.02 | 8 ± 0.086 | 6 ± 0.056 |



Table 134: Apparent porosity (0/0) at various temperatures

| | A(90/10) | B(85/15) | C(80/20) |
|---------------------|---------------------|---------------|--------------------|
| 1200 ^o c | 10.81 ± 0.15 | 9.76 ± 0.05 | 9.40 ± 0.02 |
| | | | |
| Adam A. Joseph | (MSc. Thesis 2012). | | Page 58 Page 70 |



Page 71

| Uti/isation of | clay as a | refractory | |
|----------------|---------------------|-------------------------|--|
| | git they us a firec | lay retractory material | |

Table 135: Linear firing shrinkage (0/0) at various temperatures

| | | | A(90/10) |
|---------------------|---------------------|-----------------|--|
| | | 13(85/15) | C(80/20) |
| 1200 ^o c | 4.01 ± 0.21 | 4.36 ± 0.38 | 4.41 0.77 |
| 1250 ^o c | 4.35 ± 068 | 4.65 ± 0.72 | |
| | | | 5.44 ± 0.71 |
| 1300 ^o c | 8.90 ± 0.44 | 9.74 ± 0.09 | 9.90 ± 0.03 |
| 1350 ^o c | 10.10 ± 0.20 | 10.31 ±0.21 | 10.41 ± 0.71 |
| 1400 C | 10.41 <u>± 0.09</u> | 10.31 ± 0.28 | 10.55 ± 0.47 |
| 1400 ^o c | | | |
| | | 1 | |
| | | | The second secon |
| | | | |
| | | | |
| | | | |



Adam A. Joseph (MSc. Thesis 2012).

Page

Uti/isation of lithomargic clay as a fireclay refractory materia/

lithonntyic fireæy as a rirectay retractory material

APPENDIX C

VARIATION OF PROPERTIES AT DIFFERENT SOAKING TIMES

| | | | 90 Mins | 120 Mina |
|---------------------|----------------------|-----------------------|---------------------------------------|-------------|
| TEMP | 30 Mins | 60 Mins | | 120111115 |
| 1350 ^o c | 10.4 ± 1.63 | 10.45 ± 0.467 | 10.51 0.497 | 10.52 0.549 |
| 1400 ^o c | 10.52 ± 0.211 | 10.55 ± 0.496 | 10.55 0.710 | 10.55 0.69 |
| | | | 2 10 | |
| Т | able C2: Variation o | f Apparent porosity (| (<mark>0/0) at different soal</mark> | king times |

Table Cl : Variation of linear shrinkage (0/0) at different soaking times

TEMP

30 Mins

60 Mins

90 Mins

120 Mins



Table C3: Variation of water of absorption (0/0) at different soaking time

| TEMP | 30 Mins | -6ðMins | 90 Mins | 120 Mins |
|---------------------|--------------------|----------|-----------|-----------|
| 1350 ^o c | 8.5 ± 0.34 | 8 ± 0.63 | 6.94 0.53 | 6.49 0.45 |
| Adam A. Jos | eph (MSc. Thesis 2 | 2012). | | Page 73 |

| | Uti/isation of | clay as a | refractory | |
|--|--|--|---|---|
| 1400 °C | 6.56 ± 0.56 | 6£0.75 | 5.85 ± 0.93 | 5.82 0.36 |
| | | | | |
| | | | | 60 |
| | | | | |
| | | indigic city as a fire | eclay retractory mate | erial |
| | | | | |
| | - E | ZNIE | ICT | |
| | | | | |
| | | | | |
| | | | | |
| Т | able C4: Variation of | of Bulk density (g/c | m ³) at different soal | king times |
| Т | able C4: Variation of | of Bulk density (g/c — | m ³) at different soal | king times |
| T | able C4: Variation of | of Bulk density (g/c | m ³) at different soal | king times |
| T | able C4: Variation of | of Bulk density (g/c | m ³) at different soal | king times |
| T | able C4: Variation of | of Bulk density (g/c | m ³) at different soal | king times |
| ТЕМД | able C4: Variation of | of Bulk density (g/c | m ³) at different soal | king times |
| TEMP | able C4: Variation of able C4: Variatio of able C4: Variation of able C4: Variation of a | of Bulk density (g/c | m ³) at different soal | king times |
| TEMP | able C4: Variation of able C4: Variatio of able C4: Variation of able C4: Variation of a | of Bulk density (g/c | m ³) at different soal | king times |
| TEMP | able C4: Variation of 30 Mins | of Bulk density (g/c | m ³) at different soal | king times |
| T TEMP .350 ^o c | Table C4: Variation of 30 Mins 3.62 ± 0.06 | of Bulk density (g/c | m^3) at different soal 90 Mins 3.66 ± 0.04 | king times <u>120 Mins</u> 3.67 0.11 |
| T TEMP 350 ^o c | Table C4: Variation of 30 Mins 3.62 ± 0.06 | of Bulk density (g/c | m^3) at different soal 90 Mins 3.66 ± 0.04 | king times <u>120 Mins</u> 3.67 0.11 |
| T TEMP 1350 ^o c | Table C4: Variation of 30 Mins 3.62 ± 0.06 | of Bulk density (g/c | m^3) at different soal 90 Mins 3.66 ± 0.04 | king times <u>120 Mins</u> 3.67 0.11 |
| T TEMP .350 °c 400 °c | Table C4: Variation of 30 Mins 3.62 ± 0.06 3.67 ± 0.02 | of Bulk density (g/c | m ³) at different soal 90 Millis 3.66 ± 0.04 3.67 ± 0.08 | king times <u>120 Mins</u> 3.67 0.11 <u>3.69 \pm 0.07</u> |
| T TEMP .350 ^o c | Table C4: Variation of 30 Mins 3.62 ± 0.06 3.67 ± 0.02 | of Bulk density (g/c | m ³) at different soal 90 Mins 3.66 ± 0.04 3.67 ± 0.08 | ting times <u>120 Mins</u> 3.67 0.11 <u>3.69 \pm 0.07</u> |
| T TEMP 1350 ^o c 400 ^o c | able C4: Variation of 30 Mins 3.62 ± 0.06 3.67 ± 0.02 | of Bulk density (g/c | m ³) at different soal 90 Mins 3.66 ± 0.04 3.67 ± 0.08 | king times 120 Mins 3.67 0.11 3.69 ± 0.07 |

Table C5: Variation of cold crushing strength (MPa) at different soaking times

22 27

| TEMP | 30 Mins | 60 Mins | 90 Mins | 120 Mins |
|---------------------|-------------------|-------------------|------------|-------------|
| 1350 ^o c | 11.02 ± 0.062 | 11.81 ± 0.190 | 11.90±0.11 | 11.96±0.213 |

Adam A. Joseph (MSc. Thesis 2012).

Uti/isation of lithomargic clay as a fireclay refractory materia/ $1400 ^{\circ}$ c 11.95 ± 0.164 11.95 ± 0.102 11.97 ± 0.179 $12.01 \ 0.206$





Adam A. Joseph (MSc. Thesis 2012).

APPENDIX D

PLASTICITY TEST RESULTS OF CLAY SAMPLES INVESTIGATED

| Table DI: Lic | quid Limit | (Ll.) of Afar | ri clay | | [] |
|--------------------------------------|------------|-----------------------|-------------|-------------------|---------|
| Container No | B31 | AA42 | SI | X18 | |
| Mass of Container(| 3.64 | 3.72 | 3.8 | 3.69 | 3.7 |
| Number of blows | 40 | 33 | 28 | 18 | 6 |
| Mass of C + W | 16.67 | 16.66 | 18.08 | 17.9 | 18.39 |
| Mass of C+D | 11.95 | 11.64 | 12.96 | 12.75 | 12.03 |
| Mass of D soil | 8.31 | 7.92 | 9.16 | 9.06 | 8.33 |
| Mass of Water) | 4.72 | 5.02 | 5.12 | 5.15 | 6.36 |
| Moisture Content % Table m: Plast | 56.80 | 63.38 PL) of A fai | 55.90 | 56.84 | 76.35 |
| Container No | | | | R | |
| | ~ / | AJA | | 2.50 | |
| Mass of Container | AND B | 3.71 | | 3.58 | |
| Mass of C+W() | | 7.26 | _ | 7.16 | |
| Mass of C+D | | 6.49 | | 6.39 | 7 |
| Mass of D soil | | 2.78 | | 2.81 | |
| Mass of Water (| | 0.77 | 2/5 | <mark>0.77</mark> | |
| Moisture Content (| | 27.70 | 200 | 27.40 | |
| Avera e Moisture Content (% | SAN | ENO | 27.55 | | |
| | | 'LI | BRARY | | |
| | INIVER | KWAME | IENCE & TEL | CHNOLO. | Page 62 |
| dam A. Joseph (MSc. Thesis 2012). | | KHN | MAS ! | motorial | rage 02 |
| | | as u meciay | retractory | material | |

Table D3: liquid limit (LL) of Fosu clay

| | | | 1 | | |
|---------------------|---------------------|-----------|---------|-------|-------|
| Container No | A25 | | | | |
| | | | | X17 | |
| Mass of Container (| 3.72 | 3.72 | 3.71 | 3.53 | |
| | | | | | 3.65 |
| Number of blows | 37 | 26 | 20 | 16 | 11 |
| | $/N \square$ | 10 | | | |
| Mass of C + W | 15.59 | 13.28 | 13.95 | 13.5 | 11.94 |
| Mass of C+D | 11.66 | 10.06 | 10.4 | 9 94 | 8 93 |
| | 11.00 | 10.00 | 10.4 | 7.77 | 0.75 |
| Mass of D soil | 7.94 | 6.34 | 6.69 | 6.41 | 5.28 |
| Mass of Water | 3.93 | 3.22 | 3.55 | 3.56 | 3.01 |
| Moisture Content % | 49.50 | 50.79 | 53.06 | 55.54 | 57.01 |
| Table | D4: Plastic limit (| PL) of Fo | su clay | | |

Uti/isation of lithomargic clay a fireclay refractory

| Container No | 1 | A19 |
|----------------------|------|------|
| Mass of Container () | 3.74 | 3.66 |
| Mass of $C + W()$ | 7.58 | 8.25 |
| Mass of C+D (| 6.73 | 7.27 |

| Mass ofD soil () | 2.99 | 3.61 |
|--------------------------|-------|-------|
| Mass of Water (g) | 0.85 | 0.98 |
| Moisture Content (0/0) | 28.43 | 27.15 |
| Avera e Moisture Content | 27. | .79 |

Table 1)5: liquid limit (Ll.) of Mfensi clay.

| Container No | | A7 | | C18 |
|-------------------|------|------|------|------|
| Mass of Container | 3.67 | 3.71 | 3.65 | 3.71 |
| Number of blows | 32 | 22 | 15 | 11 |

Adam A. Joseph (MSc. Thesis 2012).

| | | ~ | 2 | | |
|------------------------|---------------------|---------------|--------|-------|--|
| Mass of C + W | 12.78 | 16.05 | 16.55 | 18.84 | |
| Mass of C+D | 10.06 | 12.27 | 12.5 | 13.96 | |
| Mass of Dr soil | 6.39 | 8.56 | 8.85 | 10.25 | |
| Mass of Water | 2.72 | 3.78 | 4.05 | 4.88 | |
| Moisture Content | 42.57 | 44.16 | 45.76 | 47.61 | |
| Tat | ole D6: Plastic lim | nt (PL) Mtens | 1 Clay | | |
| Container No | | Bi l |] | B22 | |
| Mass of Container | | 3.76 | | 3.72 | |
| Mass of C + W | N. | 8.07 | | 8.04 | |
| Mass of C+D | 26 | 7.31 | ~ | 7.26 | |
| Mass of D soil | 2 | 3.55 | | 3.54 | |
| Mass of Water | | 0.76 | 0.78 | | |
| Moisture Content | 2 UT | 21.41 | 22.03 | | |
| Avera e Moisture Conte | ent | - ASS | 21.72 | | |

Uti/isation of lithomargic clay as a fireclay refractory materia/

Page 64 Page 64



Uti/isation of lithomargic clav as a fireclav refractory

| materia/ |
|--|
| Table D7: liquid limit (LL) of lithomargic clay. |

| Container No | | | | C18 |
|--------------------------|-----------------|----------------|-------|-------|
| Mass of Container | 3.67 | 3.71 | 3.65 | 3.71 |
| Number of blows | 33 | 24 | 16 | 11 |
| Mass of $C + W$ | 15.35 | 16.05 | 16.55 | 17.21 |
| Mass of C+D | 12 58 | 13.01 | 13 21 | 13 45 |
| Magg of D goil | 9.01 | 0.2 | 0.56 | 0.74 |
| Mass of D soll | 8.91 | 9.3 | 9.56 | 9.74 |
| Mass of Water | 2.77 | 3.04 | 3.34 | 3.76 |
| Moisture Content | 31.09 | 32.69 | 34.94 | 38.60 |
| Table 1)8: Plastic 1 | limit (PL) of I | Lithomargic of | clay. | |
| Container No | | Bl i | B22 | |
| Mass of Container | 2 | 3.76 | 3.72 | |
| | | 1 | | |
| Mass ofC + W | 1-1 | 8.07 | 8.04 | |
| Mass of C+D | | 7.31 | 7.26 | |
| MassofDr soil | XB | 3.12 | 3.14 | |
| | 12 | 0.76 | 0.70 | |
| iviass of water | | 0.76 | 0.78 | |
| Moisture Content | 20 | 24.36 | 24.84 | |
| Avera e Moisture Content | 5 | 24.60 | | |



Adam A. Joseph (MSc. Thesis 2012).



Uti/isation of lithomargic clay as a fireclay refractory



Page Adam Joseph (MSc. Thesis 2012).



Figure m: Graph of Casangrade test for Mfensi clay



Figure 1)3: Graph of Casangrade test for Fosu clay

81Adam Joseph (MSc. Thesis 2012).



Uti/isation of lithomargic clay as a fireclay refractory materia/

Figure 1)4: Graph of Casangrade test for Afari clay





Page Adam Joseph (MSc. Thesis 2012).