UTILIZATION OFKAOLIN (KANKARA CLAY) AND GREY CAST IRON FOR THE PRODUCTION OF COMPOSITE REFRACTORY

BY

Ruth Mataba GADZAMA

DEPARTMENT OF INDUSTRIAL DESIGN
FACULTY OF ENVIRONMENTAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA, NIGERIA

JANUARY, 2015

UTILIZATION OF KAOLIN (KANKARA CLAY) AND GREY CAST IRON FOR THE PRODUCTION OF COMPOSITE REFRACTORY

By

Ruth Mataba Gadzama,
Cert. (1978), Dip. (1981), B. A. (1995) (ABU), MBA(2000) Unimaid, M. Tech. (2005) ATBU.
Ph. D/Env. Design/11291/07-08

DISSERTATION SUBMITTED TO THE SCHOOL OF POST GRADUATE STUDIES, AHMADU BELLO UNIVERSITY ZARIA.
IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF Ph.D IN INDUSTRIAL DESIGN
FACULTY OF ENVIRONMENTAL DESIGN
AHMADU BELLO UNIVERSITY, ZARIA,
NIGERIA.

JANUARY, 2015.

DECLARATION

I hereby declare that the work in this dissertation titled 'The Utilization of Kaolin (Kankara Clay) and Grey Cast Iron for the Production of Composite Refractory' was conducted by me in the Department of Industrial Design, under the supervision of Prof. U.A.A. Sullayman, Prof. A.M. Ahuwanand Dr. D. S.Yawas.

All information from the literature have been fully acknowledged in the text and in the list of references provided. No part of this work has been presented for another degree or diploma in any institution.

Ruth Mataba GADZAMA _____ _____

(Signature) (Date)

CERTIFICATION

This dissertation titled Utilization of Kaolin(Kankara Clay) and Grey Cast Iron for the Production of Composite Refractory meets the regulations governing the award of degree of Doctor of Philosophy (Ph.D) of Ahmadu Bello University, and is hereby approved for its contribution to knowledge and literary presentation.

Prof. U.A.A. Sullayman _____ _____
Chairman Supervisory Committee (Signature) (Date)

Prof. A.M. Ahuwan _____ _____
Member Supervisory Committee (Signature) (Date)

Dr. D. S. Yawas _____ _____
Member Supervisory Committee (Signature) (Date)

Dr. Vershima Alkali _____
Head of Department (Signature) (Date)

Prof. A. Z. Hassan _____ _____
Dean, School of Post Graduate Studies (Signature) (Date)

DEDICATION

First of all, I dedicate this work to God Almighty,who took care of me during the course of this study. Secondly to my deceased husband Yerima and my daughterBaby Rebecca. It is also dedicated to our lovely children even in the time of sorrow Hyeladi, Patience,Godiya, Samson and again to Baby Rebecca (deceased), who is resting in the Lord at Jesus feet praying even right now for me to succeed. I pray to God, the father to make the deceased rest in perfect peace. May God, give them eternal rest.

ACKNOWLEDGEMENTS

I wish to thank God, the creator of heaven and earth, who created me and everything about my life my family. He that knows everything in secret, for giving me, this opportunity, knowledge and understanding to complete this work successfully. I wish to express my profound gratitude to my supervisors, Professor U.A.A. Sullayman and Professor A. M. Ahuwan. I hereby, profoundly thank them, for I grew up in their hands as my mentors. For Dr. D.S. Yawas I also thank him for opening my understanding and encouragement in the area of my study.

Once again my gratitude goes to my major supervisor Professor Sullayman, for admitting and supervising me. May God Almighty bless him for his encouragement and depth of concern about my work. To all members of staff of Industrial Design and Fine Art Departments, A.B.U, Zaria from 1977 to date, I say thank you. I came as a small child but I am going finally as an old woman full of knowledge and experience. I thank God almighty for His guidance, my brothers and sisters for their encouragement, Mrs Mariam, Ngilari, Kwasuku, Medugu, Jaffugurda, Ruth Nkwabyigu Mataba and finally Mamman Gadzama all of Mataba family. I thank them for their help, encouragement, inspiration and guidance throughout the course of this study, especially when I lost my husband and daughter within two days interval; that is 1st and4thof February, 2013. In the midst of this grief, my children Hyeladi, Patience, Godiya and Samson were my strong pillars encouraging me to continue with my study. I hereby thank them all for the support and encouragement. Also to be thanked are Mr and Mrs James Ewule and their family, Mr and Mrs Madu Gadzama and family plus my good friend, Sister Rachael Ochu. I also want to thank Mr Lawrence at IDC, Samaru, Zaria and Engr. Micheal Joseph of the Department of Mechanical Engineering, A. B. U. Zaria for their kind gesture, assistance and contributions to this study.

ABSTRACT

In this study, Kaolin (Kankaraclay) was used as a matrix material which was reinforced with Grey cast iron (GCI) to produce ceramic matrix composite (CMC) refractory. Characterization and fabrication of Kankara clay matrix with reinforced grey cast iron particles were developed in this study. The CMCs were prepared by varying the percentage weight of the grey cast iron from 5% to 45% at intervals of 5%wt. The Aim of the research is to identify, process, and formulate some selected materials such asKaolin (Kankara clay) and Grey cast iron to produce kiln shelves, shelf bars, props that can withstand high temperature and maintain its structural integrity. To get performance results, test sample specimens were made from the CMC material, dried for 14 days and then subjected to firing at a temperature of 1280^0C using kerosene burner kiln which was found to be adequate in yielding hard, strong, stiff and smooth glossy effects. The grey cast iron was obtained at Metallurgical Development Centre (MDC), Jos, Plateau State of Nigeria and the Kankara clay was obtained at Kankara, Kastina state of Nigeria. Physical property test(Apparent porosity, water absorption, linear shrinkage and bulk density) were conducted as well as mechanical property tests (Hardness, tensile strength, compressive strength, Cold Crushing Strength). Chemical analyses of the materials were conducted in Pretoria and Durban, South Africa for x-ray diffraction (XRD), x-ray fluorescence (XRF), scanning electron microscopy (SEM) and Thermo Gravimetric analyzer (TGA). Casting method was used as a shaping method to produce experimental samples and subsequently the prototype of the products. The physical and mechanical properties of the produced composites were determined and the results obtained showed that there wasincrease in the hardness value, cold crushing strength, ultimate tensile strength and compressive strength of the developed composite up to 25% wt of grey cast iron which thereafter drops with increasing grey cast iron content. The optimum value of these properties are 94shores of Hardness value at 45%wt Grey cast iron, $13.32N/mm^2$, $1.87N/mm^2$, $7.15N/mm^2$ at 25%wt Grey cast iron (GCI) respectively. Also, there was an increase in bulk density with increase in the grey cast iron content with optimum value of 1.26g/cm3 at 0%wt GCI however, linear shrinkage, water absorption and apparent porosity reduces with increasing GCI content with optimum values of 0%, 13.47% and 22.41% respectively. The thermal analysis curve shows that the samples are stable until 700^0CSummary of finding of the research shows that the utilization of Kankara clay and grey cast iron for the production of composite refractory was successfully carried out using 25%wt GCI developed into a good composite refractory.

TABLE OF CONTENTS

Cover page	i
Title page	ii
Declaration	iii
Certification	iv
Dedication	v
Acknowledgements	vi
Abstract	vii
Table of Contents	viii
List of Figures	xvii
List of Tables	xix
List of Plates	xxi
List of Appendices	xxiii
List of Abbreviations, Symbols and Terms	xxv
Definition of Special Terms	xxix
CHAPTER ONE	1
INTRODUCTION	1
1.1 Background of the Study	1
1.2 Statement of the Problem	3
1.3 Aim and Objectives of the Study	3

1.4 Significance of the Study 4

1.5 Justification of the Study 6

1.6 Limitation of the Study 7

1.7 Scope and Delimitation of the Study 7

1.8 Basic Assumption 8

CHAPTER TWO **9**

REVIEW OF LITERATURE **9**

2.1 Introduction 9

2.2 Clay Origin and Classification 9

2.3 Nature of clay 10

2.4 Classification of clay 11

2.4.1 Residual or Primary Clay 11

2.4.2 Sedimentary or Secondary Clay 16

2.4.3 Types of clay 23

2.4.3.1 Kaolin or China Clay 23

2.4.3.2 Ball Clay 24

2.4.3.3 Fire Clay 25

2.4.3.4 Saggar Clays 27

2.4.3.5 Bentonite 27

2.4.3.6 Earthenware Clay 28

2.4.3.7	Stone Ware Clay	29
2.5	Uses of clay	29
2.6	Composite Materials	30
2.6.1	Definition of composite	32
2.6.2	Merits of composites	33
2.6.3	Classification of composites	34
2.6.3.1	Metal Matrix Composites	34
2.6.3.2	Ceramic matrix Composites	35
2.6.3.3	Polymer Matrix Composites	35
2.6.4	Hybrid Composites	37
2.6.5	Phases of Composite	38
2.6.5.1	The fiber phase	38
2.6.5.2	The Matrix Phase	40
2.6.6	Reinforcement Mechanism of Composite	41
2.6.6.1	Large-particle Composites	41
2.6.6.2	Dispersion-strengthened Composites	42
2.6.6.3	Fiber Reinforced Composites	43
2.6.7	Characteristics of ceramic composites	47

2.7	Grey Cast Iron	50
2.7.1	Properties of Grey Cast Iron	50
2.7.2	Classification of Grey cast irons	52
2.7.3	Microstructure of Grey cast iron	52
2.7.4.	Metallurgy of Grey Cast Iron	55
2.7.5	Effect of major alloying elements in Grey Cast iron	61
2.7.5.1	Silicon	62
2.7.5.2	Chromium	63
2.7.5.3	Molybdenum	63
2.7.5.4	Copper	64
2.7.5.5	Vanadium	64
2.8	Refractory	64
2.8.1	Classification of refractories	67
2.8.2	Degree of Refractories	71
2.8.2.1	Kaolin	71
2.8.3	The Aluminosilicate Refractories Products and Uses	72
2.8.3.1	Fireclay refractories	73
2.8.3.2	High Alumina refractories	73
2.8.3.3	Mullite Refractories	74

2.8.4	Refractory materials	74
2.8.5	Consumers of refractories	81
2.8.6	Refractory metals	82
2.8.7	The super-alloys	83
2.8.8	Refractoriness versus the ceramic bond	83
2.8.9	Castable and fused refractories	85
2.8.10	Super refractories	86
2.9	Cermet	87
2.9.1	Production of Cermet	89
2.9.2	Applications of Cermets	90
2.9.2.1	Ceramic-to-metal joints and seals	90
2.9.2.2	Bio ceramics	91
2.9.2.3	Transportation	92
2.10	Some Previous Studies on Ceramic-Metal Composites	92
CHAPTER THREE		94
EXPERIMENTAL METHOD		94
3.1	Introduction	94
3.2	Materials Collection	94
3.33.3	Equipment Used for Research	95
3.4	Methods	96

3.4.1	Preliminary Work on the Kankara clay and Grey cast iron	96
3.4.1.1	Processing of the raw Materials	96
3.4.1.1.1	Screening and beneficiation	98
3.4.1.1.2	Kankara clay calcination	98
3.4.1.1.3	Crushing	99
3.4.1.1.4	Grinding	99
3.4.1.1.5	Particle size analysis	99
3.4.1.1.6	Compositional Analysis and Microstructure of the Kankara clay	100
3.4.1.1.7	Density	100
3.4.1.1.7	Refractoriness	101
3.5	Development of the Ceramic Matrix Composites materials	101
3.6	Methods/Standards for Testing Properties of Materials	105
3.7	Characterization of the Ceramic Composites	106
3.7.1	Physical properties	106
3.7.1.1	*Density*	106
3.7.1.2	*Water Absorption (W.A)*	107
3.7.1.3	Plasticity Test:	107
3.7.1.4	Bulk density and apparent porosity	108
3.7.1.5	Shrinkage Test	109

3.7.1.6 Absorption Test 110

3.7.1.7 Vetrification Test 110

3.7.1.8 Linear shrinkage 111

3.7.2 Mechanical properties Determination 111

3.7.2.1 *Cold crushing Strength (CCS)* 111

3.7.2.2 Hardness values determination 113

3.7.2.3 *Tensile Strength* 114

3.7.2.4 *Compressive Strength* 114

3.7.3 Microstructural and Interfacial analysis 114

3.7.3.1 Scanning Electron Microscope Analysis (SEM) 114

3.7.3.2 X-ray Diffraction Patterns of Composites 115

3.7.3.3 X-Ray Fluorescence Test *(XRF)* *116*

3.7.3.4 Thermal Analysis of Samples 117

3.8 Production of kiln materials using the developed Composite 118

CHAPTER FOUR 121

RESULTS AND DISCUSSION 121

4.1 Introduction 121

4.2	Physical Properties	121
4.2.1	Bulk density	121
4.2.2	Density	123
4.2.3	Apparent porosity	124
4.2.4	Percentage Water absorption	126
4.2.5	Linear shrinkages	127
4.2.6	Refractoriness	129
4.3	Mechanical Properties	131
4.3.1	Cold Crushing Strength (CCS)	131
4.3.2	Hardness Value	132
4.3.3	Tensile Strength	134
4.3.4	Compressive Strength	135
4.4	Microstructural analysis	137
4.4.1	Scanning Electron Microscope Analysis	137
4.4.2	X-ray Diffraction Patterns of Composites	143
4.4.3	Thermal Analysis of Samples	148
4.4.4	X-Ray Fluorescence Test *(XRF)*	152

CHAPTER FIVE 159

CONCLUSION AND RECOMMENDATION 159

5.1 Conclusion 159

5.2 Recommendation 160

REFERENCES 161

APPENDIX A 165

APPENDIX B 176

LIST OF FIGURES

Title		Page
Figure 2.1:	Schematic representations of *(a)* continuous and aligned, (b) discontinuous and aligned; and (c) discontinuous and randomly oriented fiber-re in forced composites.	45
Figure2.2:	Schematic diagram of the properties of grey cast iron	51
Figure 2.3:	Graphite flake types in Grey Cast irons	57
Figure 2.4:	Graphite flake size in Grey Cast irons.	58
Figure 2.5:	Figure 2.5: Microstructures of typical grey Irons.	59
Figure 2.6:	Composition limits for grey, mottled and white cast irons	60
Figure 2.7:	Common Shapes of Bricks	80
Figure 2.8	Cermet production process	90
Figure 3.1:	Material Processing Process Chat	97
Figure 3.2:	Plasticity Test samples	108
Figure 3.3:	Shrinkage bar	110
Figure 4.1:	Variation of Composite's Bulk Density with of group cast from	122
Figure 4.2	Variation of Composite's Density with % grey cast iron	124
Figure 4.3:	Variation of Composite's Apparent Porosity with % Grey cast iron	125
Figure 4.4:	Variation of Composite's Water Absorption with % Grey cast iron	126
Figure 4.5:	Variation of Composite's Linear Shrinkages with % grey cast iron	128
Figure 4.6:	Variation of Composite's Refractoriness with % grey cast iron	130
Figure 4.7:	Variation of Composite's Cold Crushing Strength with %	131

grey cast iron 103
Figure 4.8: Variation of Composite's Hardness Values with % grey cast iron 133
Figure 4.9: Variation of Composite's Ultimate Tensile Strength with % grey cast iron 134
Figure 4.10: Variation of Composite's Ultimate Compressive Strength with % grey cast Iron 136
Figure 4.11: XRD pattern for Kankara clay Sample with 0% Grey Cast Iron 144
Figure 4.12: XRD pattern for Sample with 10% Grey Cast Iron 144
Figure 4.13: XRD pattern for Sample with 25% Grey Cast Iron 145
Figure 4.14: XRD pattern for Sample with 35% Grey Cast Iron 145
Figure 4.15: XRD pattern for Sample with 40% Grey Cast Iron 146
Figure 4:16 XRD pattern for Sample with 45% Grey Cast Iron 146

LIST OF TABLES

Title	Page
Table 2.1: Mineral resources locations in Nigeria	13
Table 2.2: Specifications of Raw Materials for Fireclay Refractories	26
Table 2.3: Minimum tensile and transverse strength for the various classes of Grey Cast iron 54	
Table 2.4: Dimensional parameters for the test samples	54
Table 2.5: Cast iron types and their carbon equivalents	61
Table 2.6: Graphitizing and car biding effects of several elements used in Grey Cast iron compared with silicon	62
Table 2.7: Chemicomineralogical Classification of Refractories	68
Table 2.8: Classification of refractories with fussion temperature	69
Table 2.9: Compositions of silica and basic bricks	75
Table 2.10: Compositions of fireclays and high alumina bricks	76
Table 2.11: Properties of refractory bricks	76
Table 2.12: Selection/applications of different refractories	79
Table 2.13: Refractories used in various furnaces	81
Table 2.14: Physical properties of some super-refractory	87
Table 3.1: Composite blends formulation	102
Table 4.1: Analytical concentration table for Sample with 0% Grey Cast Iron	152
Table 4.2: Analytical concentration table for Sample with 5% Grey Cast Iron	153
Table 4.3: Analytical concentration table for Sample with 10% Grey Cast Iron	153
Table 4.4: Analytical concentration table for Sample with 15% Grey Cast Iron	154
Table 4.5: Analytical concentration table for Sample with 20% Grey	

Cast Iron 154

Table 4.6: Analytical concentration table for Sample with 25% Grey Cast Iron 155

Table 4.7: Analytical concentration table for Sample with 30% Grey Cast Iron 155

Table 4.8: Analytical concentration table for Sample with 35% Grey Cast Iron 156

Table 4.9: Analytical concentration table for Sample with 40% Grey Cast Iron 156

Table 4.10: Analytical concentration table for Sample with 45% Grey Cast Iron 157

LIST OF PLATES

Title		Page
Plate 3:1	Kankara clay particles	95
Plate 3:2	Grey cast iron particles	95
Plate 3:3	Beneficiation of kankara clay for the production of sample	98
Plate 3:4	Blunger machine for mixing the composite material	103
Plate 3:5	Pugging machine for blending and softening leather hard clay	103
Plate 3:6	Using digital kiln for firing test sample	104
Plate 3:7	Test samples parked inside manual kiln for firing	104
Plate 3:8	Soil Test Compression Machine	112
Plate 3:9	Hardness Testing Machine (Durometer)	113
Plate 3:10	Kiln Bars	119
Plate 3:11	Kiln Props	119
Plate 3:12	Kiln Shelves with reaction of grey cast iron that change the colour of the composite	120
Plate 4:1	Micrograph of Sample with 0% Grey Cast Iron	138
Plate 4:2	Micrograph of Sample with 5% Grey Cast Iron	138
Plate 4:3	Micrograph of Sample with 10% Grey Cast Iron	139
Plate 4:4	Micrograph of Sample with 15% Grey Cast Iron	139
Plate 4:5	Micrograph of Sample with 20% Grey Cast Iron3	140
Plate 4:6	Micrograph of Sample with 25% Grey Cast Iron	140
Plate 4:7	Micrograph of Sample with 30% Grey Cast Iron	141
Plate 4:8	Micrograph of Sample with 35% Grey Cast Iron	141
Plate 4:9	Micrograph of Sample with 40% Grey Cast Iron	142
Plate 4:10	Micrograph of Sample with 45% Grey Cast Iron	142
Plate 4:11	DTA/TGA curve for Sample with 0% Grey Cast Iron	148

Plate 4:12 DTA/TGA curve for Sample with 10% Grey Cast Iron 149
Plate 4:13 DTA/TGA curve for Sample with 25% Grey Cast Iron 149
Plate 4:14 DTA/TGA curve for Sample with 35% Grey Cast Iron 150
Plate 4:15 DTA/TGA curve for Sample with 45% Grey Cast Iron 150

LIST OF APPENDICES

Title	Page
APPENDIX A	165
Table A1: Composite's bulk density (B.D)	165
Table A2: Composite's density (D)	166
Table A3: Composite's Apparent Porosity (A.P)	167
Table A4: Composite's Water Absorption (W.A)	168
Table A5: Composite's Linear Shrinkages (LS)	169
Table A6: Composite's refractoriness	170
Table A7: Composite's Cold Crushing Strength (CCS)	171
Table A7: Composite's hardness values	172
Table A8: Composite's Hardness Values	173
Table A.9: Composite's Ultimate Tensile Strength (UTS)	174
Table A10: Composite's Ultimate Compressive Strength (UCS)	175
Table A11: Thermal degradation temperature of composites	175
APPENDIX B	176
Figure B1: X-ray Florescence test Spectrum	176
Figure B2: X-RD TEST SAMPLE 1,2,3	177
Figure B3: X-RD TEST SAMPLE 1	178
Figure B4: X-RD TEST SAMPLE 2	179
Figure B5: Test results from sieving analysis	180
Figure B6: Test results from sieving analysis	182
Figure B7: Determination of the Grain size distribution	183
Plate B1: COMPRESSING MACHINE	184
Plate B2: composite Test sample in different sizes	184
Plate B3:Jaw crushing machine	185

Plate B4: Collection of materials 185
Plate B5: The developed composite sample for the best material to be use for the production of shelves 186
Plate B6: Moisture content test in Agric laboratory A.B.U Zaria 186
Plate B7: Universal Testing Machine 187
Plate B8: Vernier Caliper for linear measurement 187
Plate B9: Pyrometer for measurement of temperature 188
Plate B10: ASTM Standard Sieves 188
Plate B11: Part of prop producing machine 189
Plate B12: Pugged clay and mould for shelve production 189
Plate B13: Collection of Fired Kiln shelve, Kiln bars and Props 190
Plate B14: Production Mould 190

LIST OF ABBREVIATIONS, SYMBOLS AND TERMS

S/N	SYMBOL	DEFINATION	UNIT
1	m_t	Mass of evaporating dish	G
2	m_h	Mass of evaporating dish + wet sample	g
3	m_{t1}	Mass of evaporating dish + dry sample	g
4	m_w	Mass of water in sample	g
5	m_d	Mass of dry grains	g
6	W%	Water content percentage	
7	D_m	Density of mercury	kg/cm^3
8	P_{app}	Apparent porosity	
9	W_D	Dry weight	N
10	W_{SD}	Suspended weight	N
11	W_S	Saturated weight	N
12	L_b	Dimension of green shelves	Mm
13	L_d	Dimension of fired shelves	Mm
14	CCS	Cold crushing strength	N/mm^2
15	W_d	Dry weight before soaking	N
16	W_w	Wet weight after soaking	N
17	W.A	Water absorption	
18	UTS	Ultimate tensile strength	N/mm^2
19	UCS	Ultimate compressive strength	N/mm^2
20	XRD	X-ray diffraction	
21	XRF	X-ray fluorescence	
22	NIS	Nigeria Industrial Standard	
23	ISO	International Standard Organization	
24	SEM	Scanning Electron Microscope	

25	TGA	Thermo Gravimetric Analysis
26	GCI	Grey cast iron
27	LS	Linear shrinkage
28	ASTM	American Society of Testing Machine
29	PVC	Poly-vinyl-chloride

DEFINITION OF SPECIAL TERMS

Fireclay: This is a clay mineral whose alumina content is either beyond 22% or up to 45% of' the total weight while the silica content is below 78%.

CMC: Ceramic Matrix Composite is a composite material whose continuous phase is a ceramic material.

Benefaciation: This is the systematic process of refining raw materials in order to optimize their performance or make them useful in the manufacturing process.

Aluminosilicates: A class of ceramic materials whose major constituents are alumina and silica. In addition to alumina and silica, all clay minerals belong to the aiuminosilicate ceramics. Examples include fireclay, kaolin, ball clay, kyanite, nontronite, montmorillonite. sillimanite, etc.

Densification: This is the process of manufacturing refractory bricks and other ceramic products by means of compaction through pressing.

Density: The ratio of the mass of a given material to its volume. It is a measure of how compact the grain structure of the matter is arranged.

Grog: This refers to pre-fired clay materials that are crushed into several particle sizes to be used for refractory production.

Refractoriness: These measure the ability of bricks to withstand high temperatures over a long period of use without melting or fusion.

SEM: Scanned Electron Microscope is an optical instrument used for viewing the surface morphology or surface structure of a material.

Stilts: Three legged refractory furniture for the mass packing of kiln in gloss firing of flat wares.

Thermal Shock Resistance: A condition whereby devices or refractory products are subjected alternately to extreme heat and extreme cold; used to screen out processing defects. It is a test to determine the ability of a refractory material to withstand heat and cold subjecting it to rapid and wide changes in temperature.

Saggars: Temperature resistant containers which serve as muffle for special materials or ceramic products. e.g. in wood fired kiln.

Slag: The by-product of iron and steel making which is brought about by the reaction in the blast furnace. Slag separates from the iron ore and stays on top, while the pig iron stays below because of its higher density.

Optical Microscopy: A process of magnification of crystals or aggregates by means of rnicrophotographv or photomicrography, in which case object and eye view lenses are jointly used.

Kiln: Kiln is a high temperature ceramic furnace used for firing and glazing of ceramic wares.

Kaolin: Kaolin clay is a very pure form of clay, which approached the ideal formula of Al_2O_3. $2SiO_2.2H_2O$. It is a primary clay formed by weathering on the site of feldspar.

Composite: Composite materials are produced by combining two or more dissimilar materials into a new material that may be better suited for a particular application than either of the original materials alone

Shelves: Shelves are structural materials used in kiln for the arrangement and supporting of fired materials.

CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

Ceramic matrix composites (CMC) as the name implies combine reinforcing ceramic or metal phases in a matrix to create materials with new and superior properties (Hsieh, 2005). CMCs have been developed to overcome the intensive brittleness and lack of reliability of monolithic ceramics, with a view to introduce ceramics in structural parts used in several environments such as rockets and engines, gas turbines for powder plants, heat shields for space vehicle fusion reactor face wall, aircraft brakes, heat treatment for nacres, etc.

It is generally admitted that the use of CMCs in advanced engines will allow an increase of the temperature at which the engine can be operated and eventually the elimination of the cooling fluids, both resulting in an increased of yield (Schysslev, 2005).

The ceramic matrix (CMCs) can be made with other materials like Carbon, Alumina, Silica, Zirconiumand so on. Ceramic composites mayalso be produced by traditional ceramic fabrication methods including mixing the powdered matrix material with the reinforcing phase followed by processing at elevated temperature. Hot pressing and sintering are some of such fabrication routes that are successfully employed for preparing composites, reinforced with a

discontinuous phase (particulate or short fibers). (However, the composites reinforced with continuous or long fibers are rarely fabricated by conventional sintering methods, due to mechanical damage of the fibers and their degradation caused by chemical reactions between the fiber and the matrix materials at high sintering temperature. Additionally sintering techniques result in high porosity of the fiber reinforced composites), (Agbodion 2007).

Ceramic matrix composites reinforced with long fibers are commonly fabricated by infiltration methods, whereby the fibers are pre-set in the mould and ceramics matrix is poured over the fibers. In this group of fabrication techniques the ceramic matrix is formed from a fluid (gaseous or liquid), infiltrated into the fiber structure (either woven or non-woven).

Prior to the infiltration of ceramic matrix composites, most kiln furniture were produced from refractory materials, which were able to withstand high temperatures but not as high as silicon nitrides and silicon carbide. According to (Hsieh and Tuan, 2005) the ceramic matrix composites possess additional qualities like high mechanical strength, at high temperatures, high toughness light stiffness and high corrosion resistance at high temperatures.

The use of ceramic matrix composites for kiln furniture is therefore based on the above qualities aforementioned.

1.2 Statement of the Problem

In Nigeria today, it is difficult to import kiln shelves, kilnbars, kiln stands and props because of high cost of purchasing them. Ewule, (1988)reported that most of the pottery establishments that were using kiln shelves in Nigeria were depending on imported kiln shelves and props manufactured in overseas countries, like U.K, U.S.A, Germany, etc.

With the rapid development of ceramic industry in Nigeria, high demand for props, shelves, and shelf bars manufactured from our abundant raw materials become imperative. The need to open ways for research in this novel area is what motivate this study.

The essence of this study is the utilization of locally available aluminosilicate materials in Nigeria in a matrix with grey iron particles for the formulation of ceramic matrix composite (CMC) for the production of kiln furniture needed for the firing of ceramic articles.

1.3 Aim and Objectives of the Study

The main aim of this study is to develop ceramic composites using Kankara clay (kaolin) as a matrix and grey iron powder as a reinforcement which can be used for the production of kiln furniture that can withstand high temperature.

The specific objectives of the study are to:

i. Conduct a characterization study of the Kankara clay (kaolin) and grey iron powder

ii. Produce ceramic composite refractory using powder metallurgy technique by varying the percentage of grey cast iron powder from (5-45%wt) at an interval of 5%wt in the Kankara clay (kaolin).

iii. Determine the physical and mechanical properties i.e bulk density, porosity, linear shrinkage and waterabsorption, cold crushing strength, Ultimate compressive strength, Tensile strength and hardness values of the composite refractory material.

iv. Determine the micro structural analysis, i.e scanning electron microscopy (SEM) /energy dispersive spectrometer (EDS), X-ray diffraction (XRD), X-ray Florescence (XRF) and Thermo-gravimetric analysis (TGA).

v. Produce kiln shelves, props and kiln shelf bars with the best composition out of the refractory bodies composed by this study

1.4 Significance of the Study

The studyintroduces the development of alternative materialfor the production of kiln equipment that is more efficient than the one that are used or produced in Nigeria currently. This study seeks to encourage ceramists and

students to try alternative materials and improved method of making their own kiln furniture to be used in the studio, cottage industries or small scale ceramic industries. These products are also expected to have better refractory properties and last longer in use when compared to similar products made of clay (kaolin) alone.

The techniques and methods used to arrive at the findings of the research serve as contribution to the technology of ceramics in Nigeria. It adds value to economic fortunes of Nigeria and reduces importation of this type of refractory. The results that were obtained in this research would form a basis for both industrial ceramic designers and researchers to design and develop ceramic composites refractory components using Kankara clay in an admixture with metal powder in the production of kiln accessories which is of great benefits to Nigerian ceramists. The following are some of the expected contributions to knowledge for carrying out this research.

The research contribute to knowledge by producing ceramic composite refractory in specific items like props, kiln shelves and resting bars, using locally available materials, which add more to the economic value of the abundant clay minerals in Nigeria.

The research brings about reduction in the importation of props, shelves, saggars and kiln shelf bars. It also encourages exportation of these products to other nations which bring more foreign exchange to the country, if the products

are developed at industrial scale. It leads to specialization thereby leading to an advanced ceramic knowledge that move the country forwards.

1.5 Justification of the Study

Ceramic matrix composites (CMC) have in recent years found large applications in the ceramic industry especially in applications involving high temperatures. Like kiln and kiln furniture. Other materials produced from such composites are silicon nitride and silicon carbide. (Chesti, 1986)

In most of these materials, the ceramic matrix composites provide increased strength which is a basic requirement of kiln furniture. Taking into cognizance this property, it is worth undertaking such a study that will replace the type of kiln furniture found in our studies.

According to Helferich and Scherick (1989) the market for ceramic matrix composites has been growing steadily and by 1999, it was valued at about $212.6 million worldwide. The development of ceramic metal composite matrix in Nigeria has been very minimal inspite of the fact that the raw materials are readily available.

The ever increasing demand for such high strength kiln furniture produced from CMC in place of the refractory onespresently used is a motivating factor for such a study.

1.6 Limitation of the Study

The main thrust of this study covered only the utilization of Kankara clay and grey iron through powder technology to produce ceramic-matrix composite refractory. The physical, mechanical and microstructural characterization of the composite refractory material was carried out. Other composites ceramics/refractories such as silicon nitride (SiN), silicon carbide (SiC), zircon ($ZrSiO_4$), silicon alumina nitride (SiALON), are not covered by this study.

1.7 Scope and Delimitation of the Study

The experiments were conducted under laboratory conditions to assess the physical microstructural and mechanical properties of the developed Kankara clay/metal particulate composites for kiln furniture application, with particular interest on production of kiln shelf bars, props and kiln shelves. Experimental samples were produced with the developed powder metallurgy technique in an admixture with kaolin. The samples were examined under SEM/EDS, XRD, XRF and TGA in order to study the effects of particulate reinforcement on the mechanical and physical properties of the composite kiln material.

1.8 Basic Assumption

i. It is assumed that good refractory kiln shelf bars, props and kiln shelves could be made from local materials and grey cast iron particles if the right materials and process techniques are followed.

ii. That the knowledge provided by this study will help to reduce the operational cost of running ceramic studios and industries.

CHAPTER TWO

REVIEW OF LITERATURE

2.1 Introduction

This chapter reviews some relevant literature with references to the following topics: -

i. Clay Origin and Classification

ii. Composites Materials

iii. Grey cast iron

iv. Refractory

2.2 Clay Origin and Classification

Clay is the product of geological weathering of the earth surface. It is the product of the geological disintegration of feldspartic, pegmatic and granite types of rock which agents of disintegration include rain, running water, ice or glacier, wind, roots of plants, animals, carbon-dioxide and extreme differences in temperature. Clay is composed of microscopically fine particles.Pure clay contains aluminium (Al_2O_3) silica (SiO_2) and water (H_2O) hence the pure day is chemically referred to as Hydrous Aluminium Silicate with the chemical formula of Al_2O_3, $2SiO_2$ $2H_2O$. China clay is a good example of pure clay.

In feldspartic clay the most important and abundant minerals are silica and alumina in combination with small amount of sodium, calcium and potash etc. However, the predominant oxide or element gives the feldspartic clay its names such as sodium feldspar or albite (Na_2O, $Al_2O_3.6SiO_2$), Potassium feldspar or orthoclase- (K_2O. Al_2O_3. SiO_2), Calcium feldspar or anorthite- ($CaO.Al_2O_3.4SiO_3$), Lithium feldspar namely Spondumen- (Li_2O. Al_2O_3. $4SiO_3$) and Petalite having a chemical formula of Li_2O. Al_2O_3. $8SiO_2$ (NeIson 1971).

In weathering or during the process of disintegration, soda sodium oxide (Na_2O), potash (K_2O) arid or lime (CaO) being soluble are carried away or off by water leaving alumina and silica which become hydrated after long exposure to water. The alumina and silica combine chemically with water as hydrated aluminium silicate with the chemical formula of ($Al_2O_3.6SiO_2$. $2H_2O$.)this constitutes the pure clay (Kaolinite clay). The above process of clay formation is known as kaolinization of clay from feldspar. Expressed below is a breakdown of feldspar in the kaolinization process.

K_2O. $Al_2O_3.6SiO_2$. + H_2O = $Al_2O_3.2SiO_2$. $2H_20$ +K_2O (SiO_7) + SiO_2……2.1

2.3 Nature of clay

Clay is the essential ingredient of all pottery or ceramics bodies. It is the hydrated silicate of aluminium ($A1_2O_3.2SiO_2$. $2H_2CO_2$. Clay is a common mineral, cheap and abundant in nature. It can be crumbled, mixed with water and made into different shapes and forms. As a material, clay is soft, plastic, and impressionable and has a cold feel. It can be pounded, moulded, flattened, rolled, pitched, coiled, pressed, pulled, pushed, squeezed and thrown on the potter's wheel. Large as well as small delicate objects which are either sculptural or architectural can be modeled from clay (Saibu, 2005).

Also in clay, different surface texture can be achieved (smooth or rough texture). The material also responds to blending, combining, shaping, drying and firing. When fired, clay becomes hard, dense, rock-like, durable and permanent. In shaping objects from this humble material, the need for great skill in handling and the avoidance of mal-handling is paramount importance.

2.4 Classification of clay

There are two major classes of clay namely:

i. Residual or Primary Clay

iiSedimentary Secondary Clay

2.4.1 *Residual or Primary Clay*

Residual or primary clay is the major source of China clay as well as kankara clay or the Kaolinite clay. In other words china clay or kankara clay can also be referred to as primary or residual clay, Residual clays are clays that have not been transported by natural agencies such as water, wind, frost or glacier. The clays are found side by side with unaltered igneous rocks from which they were formed. The rock beds of primary clay are broken down into clay largely by the action of ground water seeping through the rock and gradually leaching out the more soluble substances- Hot gases and steam within the earth interior also help in the disintegration of feldspartic rock leading to the formation of clay. Plants and animal action within and outside the earth crust also contribute to course disintegration of igneous rock to form residual clay.

Residual clays are found on irregular pockets. Since the clay is not water borne, there have been no opportunities for selective sorting out of the various particle sizes constituting the clay. This simply implies that large and small clay particles are found intermingling within the clay pockets.Due to absent of transportation, which usually leads to pulverization of the feldspartic rock, primary clay consist mainly of large particles. The clays are relatively pure and free from contamination by organic and or inorganic matter or minerals. The

value of primary clays lies in their purity, whiteness and freedom from impurity of whatever form (organic and inorganic impurities).

Most primary clays lack plasticity. To improve the clay plasticity or workability plastic clay materials such as ball clay and or bentonite is usually added to the clay. Primary clay has high fire strength and low fire shrinkage. It has high resistance to excessive temperature such that it can withstand temperature of about 1750°C. The clay requires less deflocculant than sedimentary clay. Primary clay can be found in many places in Nigeria. The table 2.1 shows some important locations of some of the major ceramics minerals in Nigeria

Table 2.1: Mineral resources locations in Nigeria

NO	MINERAL	SITE LOCATION	STATE	ESTIMATED RESERVE (TONNES)	REMARK
1	Kaolin	Kankara	Katsina	20,000,000	Residual
		Major porter, Jos	Plateau	190,00,00	“
		Oshide	Ogun		“
		Iseyin	Oyo		“
		Ifon	Ondo		“
		Ozubulu	Anambra	796,00	Sedimentary
		Illo	Sokoto		Residual
		Darazo	Bauchi	10,000,000	N.A.
		Mararaba-Rido	Kaduna	5,500,000	N.A.

		Kpaki, Pategi	Niger	N.A.	N.A.
		Igbande, (Ozonnogogo)	Edo	N.A	Sedimentary
2	Ball	Abeokuta	Ogun	N.A	Black
		Auchi	Edo	N.A.	Black
		Ujogba	Edo	N.A.	Cream
		Nsu	Imo	N.A.	“
		Giru	Kebbi	N.A.	**N.A**
3	Feldspar	Okuta	Ogun	N.A.	Potash
		Lanlate	“	N.A.	“
		Egbe	Kwara	N.A.	“
		Bari	Niger	N.A.	“
		Okene	Kogi	N.A.	“
		Gwoza	Borno	N.A.	“
		Ijero	Osun	N.A.	“
4	Quartz/silica	Pankshin	Plateau	27,962	White
		Biu	Borno	N.A.	“
		Ijero	Ekiti	4,000,000	Sand
		Lokoja	Kogi	N.A.	“
		Ugheli	Delta	N.A.	“
		Shabu	Plateau	2,540,000	“
5	Sand	Badagry	Lagos	N.A.	Sand
		Epe	“	N.A.	“
		Igbokoda	Ondo	N.A.	“
		P/Harcourt	Rivers	N.A.	“
6	Talc	Shagamu	Ogun	N.A.	Sand

		Kumunu	Niger	40,000,000	N.A.
		Ilesha	Oyo	N.A.	N.A.
		Okolom	Kogi	N.A.	N.A.
		Zonkwa	Kaduna	N.A.	N.A.
7	Bentonite	Geshua	Yobe	N.A.	N.A.
		M/Belwa	Adamawa	N.A.	N.A.
		Esan/Ishan	Edo	N.A.	N.A.
8	Limestone	Ukpila	Edo	10,161,00	White
		Jakura	Kogi	68,000,000	
		Igumale	Benue	30,161,000	N.A.
		Mfamoging	C/River	26,000,000	Grey
		Nkalagu	Enugu	720,000,000	Grey
		Ewekoro	Ogun	7.1 billion	Clayey
		Arochukwa	Imo	101,000,000	N.A.
		Shagamu	Ogun	N.A.	Grey
		Iseluku	Delta	N.A.	N.A.
		Sokoto	Sokoto	N.A.	N.A.
9	Dolomite	Osara	Kogi	2,000,000	White
		Itobe	Benue	1,000,000	“
		Igara	Edo	N.A.	“
		Mura	Plateau	N.A.	“
		Elebu	Kogi	N.A.	“
		Igbetti	Oyo	N.A.	“
		Burum	FCT	8,000,000	“
		Kwakuti	Niger	2,540,000	“

		B/Gwari	Kaduna	N.A.	N.A.

Sullayman, (2005)

2.4.2 *Sedimentary or Secondary Clay*

Sedimentary or secondary clay is clay which does not remained at its original site of formation. It is transported by action of running water or rain wind, glacier or frost from the site of the parent rock to new deposit Secondary clay is usually found arranged in strata or layers. The clay is much more common than the primary clay.

The transportation of sedimentary or secondary clay facilitates the grinding of the clay into smaller and fine particle sizes, which aid the plasticity of the clay. In the course of transportation, on reaching a plain or estuary, the force of transportation reduces with the coarse particle being unable to move or carried along. In this situation the coarse particles settle first leaving the fine clay particles suspended in water. When quiet water is reached such as in a lake or sea, the suspended fine clay particles sink to the bottom and settle along the course of transportation. During transportation, secondary clay usually comes in contact with organic and inorganic minerals. Minerals like iron, lignite, quartz, and mica

as well as decompose plants and animals remains are usually met on the way. Secondary clay has fine particles, which is present due to grinding in the course of transportation. The fineness of the clay brings about the high plastic nature of the clay. The clay is said to be impure, due to the presence of iron and other impurities met in the course of transportation. It has varieties of colour either in its raw or fired state. It has high drying strength and low fire strength when compared with primary clay. The presence of alkaline substances in the clay also helps to lower the maturing temperature of the clay as such the clay is usually low-fired.

The slaking property of secondary clay is however, higher than that of the primary clay. The clay requires more deflocculant than the primary clay. Secondary clay is found in large quantity in most of the towns and villages in Nigeria.

The following are some of the physical properties of clay, which need to be tested in order to determine the workability of the clay.

i. Plasticity
ii. Shrinkage
iii. Porosity
iv. Slaking property
v. Strength

vi. Colour

vii. Residue Test etc.

i. *Plasticity*

Clay plasticity is simply the ability of clay to stick or adhere together during shaping. Plasticity facilitates the formation of clays into different shapes and forms. Clays vary in plasticity depending on the geological history of the clay. Clay plasticity is mainly attributable to the fineness of the clay particle size. Clay particles are plate like in shape, each of which clings to one another thereby enhancing clay plasticity. Also enhancing clay plasticity is the chemical attraction, which exist between the clay particles. Clay plasticity is dependent also on the availability of organic and carbonaceous matter present in the clay. These organic matters act as gum or glue, binding the particles together. Temperature and aging of the clay also improve its plasticity.

Test for Clay Plasticity

A simple test for clay plasticity can be carried out by looping a pencil or snake like roll of clay around one's finger. If the coil of clay cracks excessively, this simply imply that the clayisnot plastic enough. This clay may not throw well on a potter's wheel. A test to determine clay plasticity can also be carried out by throwing several ceramics pieces on a wheel. If the clayshows excessive cracks and or deforms easily under forming pressure, this means that the clay is not plastic enough.

ii. ***Shrinkage***Green ceramic product diminishes or reduces in size as they get dried up or drier. This diminishing tendency of the green ceramics articles is known as shrinkage. There are two types of shrinkage:

a) Drying shrinkage
b) Firing shrinkage

a) **Drying shrinkage: -** This takes place before-a ware is fired and or after firing has taken place. Drying shrinkage is caused by the loss of water of plasticity or pure water from the green ware when the ware is exposed to draft or heat.

b) **Firing shrinkage: -** This usually takes place during biscuit or bisque firing and glaze firing of ceramics ware. Firing shrinkage involves the elimination of the chemically combined water from the ware and the subsequent vitrification of the article. Clay containing fine particles shrinks more than those with large particles. This is usually so because fine particle clays contain more water of plasticity. Similarly, clay with fine particles also dries more slowly. The shrinkage rate of clay during drying is between 5%-12% while in firing it is between 8%-12% (Nelson 1971).

Test for Shrinkage

To test for the rate of clay shrinkage, the first thing is to roll out a plastic clay slab. The clay slab is either cut or marked and measure at the green ware stage. When the slab is totally dried, a second measurement is taken. A final measurement is made after firing. At the various stages of measurement, a

reduction in the size of the ware will be observed. Where excessive shrinkage is noticed in the clay slab, non-plastic clay materials could be added to reduce the plasticity as well as the rate of shrinkage of the clay and improve the rate of drying. Such non plastic materials include grog, silica sand, kaolin etc.

iii. *Porosity*

This is simply the tendency of clay to allow water to pass through as a result of the particle sizes of the clay. The porosity of clay varies, depending on the state of the clay. For instance the porosity of fired clay depends on the hardness and the vitrification of the clay. While clay porosity at the green ware stage depends on the particle size of the clay. Clay with coarse particle size will be more porous than clay with fine particle sizes.

Test for Porosity

To carry out porosity test a biscuit or bisque test slat is weighed. It is soaked in water overnight and wiped clean of the surface water and weighed the second time. The percentage gain in weight stands for the porosity of the clay. On the other hand, if test for porosity is to be made before firing, the plastic clay (fine particle clay) and the non-plastic clay (coarse clay) are placed in two different perforated containers underneath which are calibrated bakers to collect water. Water is poured into the containers of clays and the rate of water flow is recorded. It will be noticed that the clay with' large particle sizes will be more permeable

than the one with fine particles. This process will help to determine the porosity of the wet clay.

iv. *Slaking Property of Clay*

Slaking is the ability of clay to dissolve or break into smaller fragment when soaked in water. All clay has the tendency to slake when soaked in water. The rate at which clay slakes depends on the clay particle sizes. Clay with fine particles slakes slower than those with large particles. This is due to the compressed nature of the clay. Clays that slake easily are more preferable than those that slake after a long period of soaking in water.

v. *Clay Strength*

This property of clay provides the clay the capability to withstand handling during forming and the subsequent drying/firing of the wares. Clay has what could be regarded as green, drying and fired strength. Clay strength is an important property of clay. In an industry for instance, clay with high green strength will be able to withstand mal-handling during forming and dressing with a reduced breakage, Similarly clay high in drying strength, will hold firm during loading of the dried ceramics wares in the kiln while clay high in firing strength,

will be able to hold its shape when glaze suspension (glaze slip) is applied during glazing.

Test for Clay Strength

Clay strength is determined by breaking a bar supported on two knife-edges by means of a load applied to the center.

vi. Clay Colour

This property of clay helps in the classification of clay either as primary or secondary clay. Clay colour varies and this could be determined by the mode of formation, the degree of dispersion or transportation as well as the type of impurity or oxides met in the course of transportation from the parent rock. Clay has varieties of colour right from the green state to the fired state. Most primary clay appears white at the green state and in firing it also maintains its whiteness. Secondary clay at the green state has varieties of colour which range from brown, red, tan, Grey, black to deep red, off white and on firing.

vii. Residue/ Residue Test

Residues are fragments of plants and animals, which intermingle with the clay in the course of formation/transportation from the parent rock. To determine clay residue, the clay is soaked in water for at least one day or twenty-four hours. The long time exposure to water is to enable the clay dissolve or slake

completely. The dissolved clay is thereafter passed through 120-sieve mesh and washed with running water until the clay particles have passes through thesieve. The materials that could not pass through the sieve aredried in an oven over-night and analyzed. These materials constitute the residue.

2.4.3 Types of clay

2.4.3.1 Kaolin or China Clay

Kaolin or china clay is a very pure form of clay, which approached the ideal formula of Al_2O_3. $2SiO_2.2H_2O$. It is primary clay formed by weathering on the site of feldspar. It is white in colour before and after firing and can be fired to an extremely high temperature. Kaolin provides a source of alumina and silica for glazes and is an important ingredient in high fire white ware and porcelain bodies. Kaolin is usually coarse in particle sizes and is therefore non-plastic compared to most secondary clay. It is mostly found in pockets rather than stratified beds and is mingled with unaltered rock fragments of feldspar and quartz. Before the clay can be used these rock fragments are got rid of by some methods of purification.

Kaolin is highly refractory and has a melting point of about 1,800°C. When use alone kaolin is very difficult to shape because of its poor plasticity and also because of its refractoriness. It is difficult to measure into hard dense object.

For practical work it is advisable that other material be added to kaolin to increase it workability and lower the kiln temperature needed to produce a hard dense object. It shrinkage is quite low because of its coarse grain structure. It also has little drying •strength. However, there is sedimentary or secondary kaolin, which are plastic, smooth but lack whiteness possess by the primary kaolin.

2.4.3.2 Ball Clay

Ball clay is chemically similar to kaolin after firing. It usually has Grey colourin its unfired state, which is attributable to the presence of organic matter. Like kaolin the clay is derived from granite type of rock. The particles are mostly deposited-in swampy areas where the organic acids and gaseous compound released from decaying vegetative matters help to break down the clay particles into finer sizes.

Unlike kaolin, ball clay is high in iron content, more fusible, much more plastic, and fine in particle sizes and imparts a great drying strength when used as a body component. Ball clay is nevertheless complementary in character with kaolin. These two clays are often combined in clay body to obtain a practical workable mass. It can be used as a source of alumina and silica for glazes, as well as a binding agent.

Due to its excessive plasticity, ball clay cannot be used alone in pottery; about 20% addition of the clay to a pottery body would greatly improve the plasticity of the body. However, excessive application of ball clay on a clay body would make it to be sticky, shrink excessively and gives it a high drying strength. In a porcelain body, the addition of ball clay will decrease the translucency of the formed ware.

2.4.3.3 Fire Clay

The term fire clay refers to refractoriness or resistance to heat. The physical characteristic of clays may vary widely but if they are highly refractory, such clay may be termed fire clay. Some fire clays have a fine plastic quality, while others are coarse and granular and are unsuitable for throwing. Fire clay generally contains some iron as impurity but seldom has calcium or feldspar. The fire colour also varies. In fact any clay which resists fusion or deformation up to 1,500°C may be termeda fire clay.

Refractoriness in fire clay or the heat resistance in the clay presupposed the absent of iron in the clay. In fire clay, the more plastic varieties are sometime found adjacent to coal veins or seams. This plastic fire clay fire to brownish colour.

Fire clay is commonly used for the manufacturing of insulating bricks, hard firebricks, kiln furniture, furnaces, boilers and melting pots. In metallurgical industries high temperature melting is carried out in firebricks furnaces. Its industrial uses are due to the clay high content in alumina or flint.

TABLE 2.2: Specifications of Raw Materials for Fireclay Refractories

Specifications	Non-Plastic Fireclay		Plactic fireclay	
	Grade 1	Grade II	Grade 1	Grade II
Al2O3 (Raw) Max %	35	25-35	_	_
Fe2O2 (Raw) Max %	3	3	5	5
L. O. I Max %	15	15	_	_
P.C.E. (Orthon) Min.	32-34	30-32	30	23
General Appearance and other features	Hard, dence, non-flake, less crumbly		Soft, fine texture, long vitrification range, no grit	
Appearance on firing for 2hrs at	(1400°C)	(1400°C)	(1400°C)	(1400°C)

temperature show in brackets	Should not give iron spots, blister melt holes, etc.

Source: Chesti, 1986

The above table affirms that the maximum $A1_20_3$ contents, maximum Fe_20_2 content, the maximum loss on ignition, general appearance and features of a fireclay refractory minimum firing range of 1300°C-1400°C (P.C.E) are important factors in judging the qualities of fireclay materials. This is especially so if refractory production is the object of use of a fireclay.

2.4.3.4 Saggar Clays

Saggar clays are clay boxes in which ceramics wares are fired to protect them from direct heat and flame in the kiln. This therefore implies that saggar clay is that clay which has been found suitable for the manufacture of saggers. The clay must be quite refractory to resist thermal shock and fatigue due to continuous and or repeated firing in the kiln. The ability of saggar clay to withstand high heat and its refractory quality could imply that the clay is derivable from fire clay.

2.4.3.5 Bentonite

Bentonite is a type of clay formed in the pre-historic ages from the airborne dust of volcanic eruptions. It is composed largely of silica. An addition of three

percent ofbentonite to a glaze would help in the adhesion of the glaze to the ware without a noticeable change in the glaze.When used in non-plastic (short) clays, one part bentonite is usually equal to five parts ball clay. It is advisable that the clay be mixed dry since it becomes quite gummy when mixed alone with water. Bentonite is used as a plasticizer in small quantity with non-plastic clay. Rhodes (1959) observed that improving the plasticity and workability of clay would require the inclusion of plasticizers such as bentonite.

2.4.3.6 Earthenware Clay

Earthenware clays are made up of a group of low firing clays that mature at temperature ranging from cone 08 to cone 02 (1940-1060 °C). The clays contain a relatively high percentage of iron oxide and other mineral impurities, which serve as, flux (a substance that lowers the maturing temperature of the clay).These minerals make the clay on firing to be rather too fragile and quite porous. Earthenware clay unlike stoneware clay, which is almost completely vitreous after firing, is quite porous and its porosity is usually between five and fifteen percent. The clay, cannot be made vitreous for it deform and often bloat, blister at temperature above 1150^{o}C

In green state, earthenware clay has varieties of colour. These colour ranges from red to brown, greenish, Greyish, bluish and pink. This difference in colour is due to the presence of iron oxide, other mineral impurities and the effect of light. On firing earthenware clay, also gives different colours. These colours include pink, red, brown and/or black. Earthenware also known as common red clay may be highly plastic even too sticky to be used alone. The clay could lack plasticity with excessive sand content. While the bricks maker would require the non-plastic earthenware clay, which he can press and fire without showing any form of deformation for brick, the potter needs smooth plastic earthenware clay to shape his product either on the potter's wheel or manually. Apart from its use in fashioning out aesthetic and or utilitarian vases earthenware clay commercially can be used for the making of building bricks, tiles, drain-tiles, roofing tiles and other products.

2.4.3.7 Stone Ware Clay

Stoneware clays are commonly of the sedimentary type, and are found in seams or layer often associated with fire clay or coal. Stoneware clays are generally plastic and fire in a range from cone 6 to cone 10. Any clay that can be fired to a temperature of 1200^{o}C or about cone 7 and which as a result of the fire treatment is of serviceable hardness and density may be termed as stoneware clay. Stoneware clays may burn to a buff, tan, Grey , light brown or dark brown or

brown black colour depending on the amount of iron or other organic and inorganic impurities present as well as the exact temperature and atmospheric condition of the firing. The fact that it is difficult to get a clay which possesses all the desire qualities of plasticity, colour, temperature, texture, shrinkage, porosity etc. a stoneware body or other forms of clay bodies have to be compounded which will meet up with the desired bodies characteristics by the potter.

2.5 Uses of clay

Clay has many uses. In the ancient time, it was used as a writing medium. Presently clay is being used in pottery and ceramic production (dinnerware, sewage, bricks, sewage pipes and tiles) and children toy. It is also used in modeling for industrial product design and prototyping.

a. **Kaolin** is used for the pottery and porcelain, also as a writing medium even in modern time's forms part of the materials for paper production.
b. **Ball clay** is mainly used in making pottery; it is sometimes called potter's clay.
c. **Fire clay** is mainly used for making firebrick, which is used to line furnaces, kilns, and for crucibles. Other forms of fireclay included kyanite, sillimanite, andalusite, and corundum.
d. **Common clay** is used for brick, clay pipe, clay floor and wall tiles, and other building materials.

e. **Bentonite** is used in molds for casting metals and in materials called drilling muds that are used in drilling for petroleum. Bentonite that expands when mixed with water and it is used in engineering work to seal leaks and in ceramic to improve on clay that is not plastic to make the clay plastic.

2.6 Composite Materials

Composite materials are produced by combining two or more dissimilar materials into a new material that may be better suited for a particular application than either of the original materials alone.The most common exampleof a composite material is the fiber-glass-reinforced plastic commonly used in household goods and inmany industrial applications.

The plastic alone is relatively weak and has a low elastic modulus,that is, it bends and stretches easily. However, it is very stable chemically and constitutes an excellent matrix for the composite.The glass fibers provide the strength and stiffness; their modulus of elasticity may be 50 times greater than that of the plastic. Since the glass fiber can withstand a much higher tensile stress before strain or yielding occurs, they take most of the load when the composite is stressed.

Many of our modern technologies require materials with unusual combinations of properties that cannot be met by the conventional metal alloys, ceramics and polymeric materials. This is especially true for materials that are needed for aerospace, underwater and transportation application.Metals, ceramics,

glasses, polymers and cement can be combined in composite materials to produce unique characteristics such as stiffness, toughness and high temperature strength.Portland cement concrete, asphalt concrete and similar materials are considered as aggregate composites,whereas reinforced and pre-stressed concrete can be regarded as a first prototype of modern composite materials.

Many composite materials are composed of just two phases; one is termed the matrix, which is continuous and surrounds the other phase, often called the dispersed phase.Composites are materials that comprise strong load carrying material (known as reinforcement) imbedded in weaker material (known as matrix). Reinforcement provides strength and rigidity, helping to support structural load, while the matrix or binder (organic or inorganic) maintains the position and orientation of the reinforcement. Significantly, constituents of the composites retain their individual, physical and chemical properties: yet together they produce a combination of qualities which individual constituents would be incapable of producing alone. (Hull,*et al.*, 1996).

2.6.1 Definition of composite

The most common definition is the one given by (Jartiz**,** 1965)**,** who defined composite as“amultifunctional material systems that provide characteristics, not obtainable from any discrete material. They are cohesive structures made by physical combination of two or more compatible materials, different in composition and characteristics and sometimes in form.

(Kelly, 1967) very clearly stresses that; the composites should not be regarded simply as a combination of two materials. In the broader significance: the combination has its own distinctive properties. In terms of strength, resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

(Berghezan, 1966), define composite as "Compound materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their shortcomings", in order to obtain improved materials.

(Van Suchetclan, 1972) explains composite materials as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. They can also be considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical properties.

2.6.2 Merits of composites

Advantages of composites over their conventional counterparts are their ability to meet diverse design requirements with significant weight savings as well as strength-to-weight ratio. Some advantages of composite materials over conventional ones are as follows (Todd, 2009):

a. Tensile strength of composites is two to three times greater than that of steel or aluminium (depending on the reinforcements).
b. Improved torsional, stiffness and impact properties.
c. Higher fatigue endurance limit (up to60% of ultimate tensile strength).
d. 30%-40% lighter than other material structures designed to the same functional requirements.
e. Lower residual stress as compared to other structural metallic materials like steel, aluminium and so on.
f. Composites are less noisy while in operation and provide lower vibration transmission than metals.
g. Composites are more versatile than metals and can be tailored to meet performance needs and complex design requirements.
h. Long life, offering excellent fatigue, impact, environmental resistance and reduced maintenance.
i. Composites enjoy reduced lifecycle cost compared to metals.
j. Composites exhibit excellent corrosion resistance and fire retardation.
k. Improved appearance with smooth surfaces and readily incorporable integral decorative melamine are other characteristics of composites.
l. Composite parts can eliminate joints/fasteners, providing part simplification and integrated design compared to conventional metallic parts.

2.6.3 Classification of composites

Broadly, composite materials can be classified into three groups based on matrix material used (Kopeliovich, 2010). These include:

a. Metal Matrix Composites (MMC)
b. Ceramic Matrix Composites (CMC)
c. Polymer Matrix Composites (PMC)

2.6.3.1 *Metal Matrix Composites*

Metal Matrix Composites have many advantages over pure metals like higher specific modulus, higher specific strength, better properties at elevated temperatures and lower coefficient to thermal expansion. Because of these attributes, metal matrix composites are now considered for wide range of applications viz, combustion chamber nozzle (in rocket and space shuttle), housings, tubing, cables, heat exchangers, structural members etcetera. (Kopeliovich, 2010)

2.6.3.2 *Ceramic matrix Composites*

One of the main objectives in producing ceramic matrix composites is for applications with high toughness requirement. Naturally, it is often found that there is an improvement in strength and stiffness of ceramic matrix composites. (Kopeliovich, 2010)

Ceramic fibers, such as alumina and SiC (Silicon Carbide) are advantageous in very high temperature applications, and also where environment

attack is an issue. Since ceramics have poor properties in tension and shear, most applications as reinforcement are in the particulate form for example zinc and calcium phosphate. Ceramic Matrix Composites (CMCs) are used in very high temperature environments, these materials use a ceramic as the matrix and reinforce it with short fibers, or whiskers such as those made from silicon carbide and boron nitride.(Kopeliovich, 2010)

2.6.3.3 *Polymer Matrix Composites*

Most commonly used matrix materials are polymeric. In general, the mechanical properties of polymers are inadequate for many structural purposes, particularly: their strength and stiffness are low as compared to that of metals and ceramics. These difficulties are overcome by reinforcing them with other materials. The reason for their common use is that the processing of polymer matrix composites does not involve high pressure and temperature. In addition, equipment required for manufacturing polymer matrix composites are simpler in operation and structure. For these reasons, polymer matrix composites develop rapidly and became popular for structural applications. (Kopeliovich, 2010)

There are two types of polymer composite: these are: (Kopeliovich, 2010)

a. Fibre reinforced polymer (FRP)
b. Particle reinforced polymer (PRP)

a. Fibre Reinforced Polymer

Fibre reinforced composite contain fibres and a polymer matrix. Fibres are the reinforcement and the main source of strength while matrix glues all the fibres together in shape and transfer stresses between the reinforcing fibres. The fibres carry the loads along their longitudinal directions. Sometimes, fillers might be added to smooth the manufacturing process, impact special properties to the composites, and /or reduce the product cost.

Common fibre reinforcements include asbestos, carbon /graphite fibres, beryllium, berylliumcarbide, berylliumoxide, molybdenum, aluminiumoxide, glass fibres, polyamide and natural fibres. Similarly, common matrix materials include polythene, epoxy, phenolic, polyester, polyurethane, polyetheretherketone (PEEK), vinylester. Among these resin materials, PEEK is most widely used. Epoxy, which has higher adhesion and less shrinkage than PEEK, comes in second for its high cost. (Kopeliovich, 2010)

b. Particle Reinforced Polymer

Particles used for reinforcement include ceramics and glasses such as small mineral particles, metal particles such as aluminium and amorphous materials and carbon black. Particles are used to increase the modulus of the matrix and to decrease the ductility of the matrix. Particles are also used in order to reduce the cost of the composites. Reinforcements and matrices can be common in

expensive materials and are easily processed. An example of particle-reinforcedcomposites is anautomobiletyre, which has carbon black particles in a matrix of poly-isobutylenesastomeric polymer. (Kopeliovich, 2010)

2.6.4Hybrid Composites

Hybrid composites are relatively new and obtained by using two or more different kinds of fibers in a single matrix; hybrids havea better all-around combination of properties than composites containing only a single fiber type. A variety of fiber combinations and matrix materials are used, but in the most common system, both *carbon and glass fibers* are incorporated into a *polymeric resin.*

The carbon fibersare strong and relatively stiff and provide a low-density reinforcement; however, they are expensive. *Glass fibers* are inexpensive but lack the stiffness of carbon. The glass *carbon* hybrid is stronger and tougher, has a higher impact resistance and maybe produced at lower cost than either of comparable all-carbon or all-glass reinforced plastics.

Two different fibers may be combined in a number of ways for example, the fibers may all be aligned and intimately mixed with one another; or laminations may be constructed consisting; layers, each of which consists of a single fiber type, alternating one with another.

The failure does not occur suddenly when hybrid composites stressed in tension. The carbon fibers are first to fail, at time the load is transferred to the

glass fibers. Upon failure of glass fibers, the matrix phase must sustain the applied load.Hybrid composites find *applications* such as lightweight water and air transport structural components, sporting and light weight orthopaedic components.

2.6.5Phases of Composite

2.6.5.1The fiber phase

This refers to any polymer, metal or ceramic that has been drawn into long and thin filament. An important characteristic of most materials is that a small diameter fiber is much stronger than the bulk material. The probability of the presence of a critical surface flaw that lead to fracture diminishes with decreasing specimen volume; this feature is used to advantage in the fiber-reinforced, the materials used for reinforcing fibers have tensile strengths. On the basis of *diameter* and *character, fibers* are grouped into three different *classifications: (i) whiskers*, (ii) *fibres* and (iii) *wires*.

1 **Whiskers:**These are very thin single crystals that have extremely large length-to-diameter ratios. As a consequence of their small size, they have a high degree of crystalline perfection and are virtually flaw free, which accounts for their exceptionally high strengths; they are the strongest known materials. In spite of these high strengths, whiskers are not utilized extensively as a reinforcement medium because they are extremely

expensive. Moreover, it is difficult and often impractical to incorporate whiskers into a matrix. Whisker materials include graphite, silicon carbide.

2 **Fibre:** Materials that are classified as fibers are either polycrystalline r or amorphous and have small diameters; fibrous materials have small diameters; fibrous materials are generally either polymers or ceramics for example, the polymer aramids, glass, carbon, boron, aluminium oxide and silicon carbide.

3 **Wires**: These are fibres that have relatively large diameters; typical materials include steel, molybdenum and tungsten. Wires are utilized as a radial steel reinforcement in automobile tires, in filament-wound rocket casings and in wire wound high pressure hoses.

2.6.5.2 ***The Matrix Phase***

The functions of matrix phase include

(i) The matrix phase binds the fibers together and acts as the medium by which an externally applied stress is transmitted and distributed to the fibers; only a very small proportion of an applied load is sustained by the matrix phase. The matrix material should be ductile. In addition, the elastic modulus of the fiber should be much higher than that of the matrix.

(ii) The matrix protects the individual fibers from surface damage as a result of mechanical abrasion or chemical reactions with the environment.

(iii) The matrix separates the fibers and by virtue of its relative softness and plasticity, prevents the propagation of brittle cracks from fiber to fiber, which could result in catastrophic failure.

It is essential that adhesive bonding forces between fiber and matrix be high. Adequate bonding is essential to maximize the stress transmittance from the weak matrix to the strong fibers. In general, *only metals* (such as aluminium and copper) and commercial thermoplastic and thermosetting *polymers* are used as matrix materials, because some ductility is desirable.

2.6.6Reinforcement Mechanism of Composite

2.6.6.1 *Large-particle Composites*

The term, large is used to indicate that particle-matrix interactions cannot be treated on the atomic or molecular level; rather continuum mechanics is used. For most of these composites, the particulate phase is harder and stiller than the matrix. These reinforcing particles tend to restrain movement of the matrix phase in the vicinity of each particle. In essence, the matrix transfers some of the applied stress to the particles, which bear a fraction of load. The degree of

reinforcement or improvement of mechanical behavior depends on strong bonding at the matrix-particle interface(Calister and Reithwisch, 2010; Khanna, 2010).

Some polymeric materials to which fillers have been added arc really large-particle composites. Again the fillers modify or improve the properties of the material and/or replace some of the polymer volume with a less expensive material i.e., the filler. Another familiar large-particle composite is concrete, being composed of cement (the matrix) and sand and gravel (the particulars) (Calister and Reithwisch, 2010; Khanna, 2010).

The particles should be of approximately the same dimension in all directions (equated). For effective reinforcement, the particles should be small and evenly distributed throughout the matrix. The mechanical properties are enhanced with increasing particulate content. Large particle composites are utilized with all three material types (metals, polymers and ceramics). Cermets are examples of ceramic-metal composites. The most common cermet is the cemented carbide,*w*hich is composed of extremely hard particles of a refractory carbide ceramic such as tungsten carbide (WC) or titanium carbide (TIC), embedded in a matrix of a metal such as cobalt or nickel. These composites are utilized extensively as cutting tools for hardened steels.

Both elastomers and plastics are frequently, reinforced with various particulate materials. Use of many of the modern rubbers would be severely

restricted without reinforcing particulate materials such as carbon black. When added to vulcanize rubber, this extremely inexpensive material enhances tensile strength, toughness and tear and abrasion resistance. Automobile tires contain on the order of 15 to 30%volof carbon black (Calister and Reithwisch, 2010, Khanna, 2010).

2.6.6.2 *Dispersion-strengthened Composites*

Metals and metal alloys may be strengthened and hardened by the uniform dispersion of several volume per cent of fine particles of a very hard and inert material. The dispersed phase may be metallic or non-metallic; oxide materials are often used. Again, the strengthening mechanism involves interactions between the particles and dislocations within the matrix, as with precipitation hardening. The dispersion strengthening effect is not as pronounced as with precipitation hardening; however, the strengthening is retained at elevated temperatures and for extended time periods because the dispensed particles are chosen to be unreactive with the matrix phase (Calister and Reithwisch, 2010;2012; Khanna, 2010)

The high-temperature strength of nickel alloys may be enhanced significantly by the addition of about 3 vol % of thoria (ThO_2) as finely dispersed particles; this material is known as thoria: dispersed (or TD) nickel. The same effect is produced in the aluminium-aluminium oxide system. A very thin and adherent - alumina coating is caused to form on the surface of extremely small

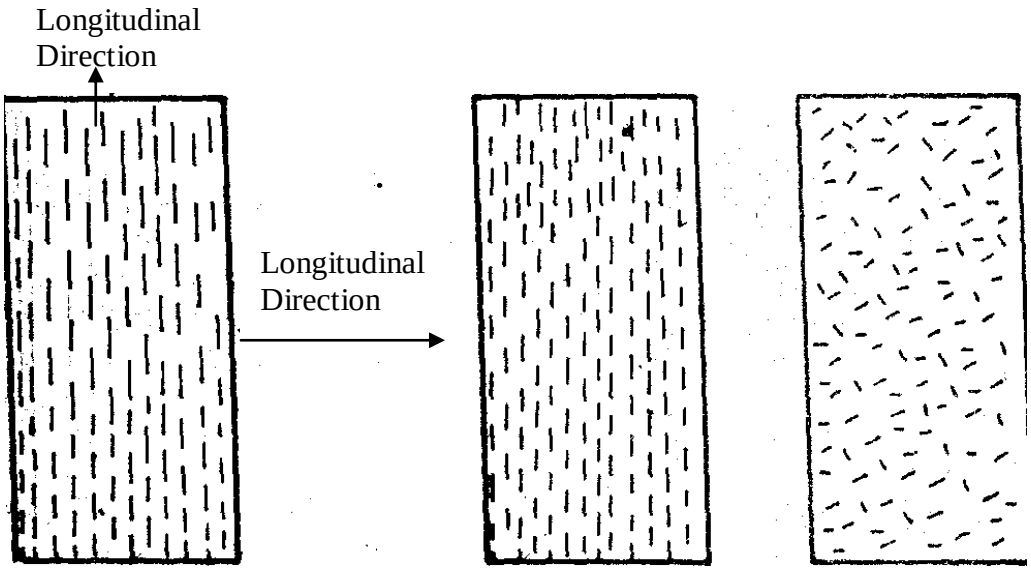
(0.1 to 0.2μm thick) flakes of aluminium, which are dispersed within an aluminium metal matrix; this material is termed sintered aluminium powder (SAP)(Calister and Reithwisch, 2010;2012; Khanna, 2010)

2.6.6.3 *Fiber Reinforced Composites*

Technologically, the most important composites are those which the dispersed phase is in the form of a fiber. Design goals of fiber-reinforced composites often include high-strength and/or stiffness on a weight basis. Those characteristics are expressed in terms of specific strength correspond, respectively, to the ratios of tensile strength to specific gravity and modulus of elasticity to specific gravity. Fiber-reinforced composites with exceptionally high specific strengths and moduli have been produced that utilize low-density fiber and matrix materials.

The arrangement or orientation of the fibers relative to another, the fiber concentration and the distribution all have a significant influence on the strength and other properties of reinforced composites. Somecritical fiber length is necessary for effective strengthening and stiffening of the composite material. The critical length is independent on the fiber diameter d and its ultimate (or tensile) strength $б_f$ and on the fiber-matrix bond strength (or the shear yield strength of the matrix) $T_C$according to the formula below: (Calister and Reithwisch, 2010; Khanna, 2010)

$$l_c = \frac{\sigma_f . d}{\tau_c} - - - - - - - - - - - - - - - - - - 2.2$$



(a) (b) (c)

Figure2.1: Schematic representations of *(a)* continuous and aligned, (b) discontinuous and aligned; and (c) discontinuous and randomly oriented fiber reinforced composites. Source: (Khanna, 2010)

For a number of glass and carbon fiber-matrix combinations, this critical length is on the order of 1 mm, which ranges between 20 and 150 times the fiber diameter. Fibers for which $l >> l_c$(normally $l > 15l_c$) are termed continuous; discontinuous or short fibers have lengths shorter than this (Calister and Reithwisch, 2012, Khanna, 2010).

The properties of a composite having its fibers *aligned* are high anisotropic, that is, dependent on the direction in which they are measured. For continuous fiber reinforcement the strain in the matrix and the strain in the fiber under load are initially the same. At low stresses, both fiber and matrix deform elastically

but, with increasing stress, the matrix may deform plastically while the fiber will still be elastic.

Tensile failure at 90° to the fiber direction occurs at very low stresses. This appears to be the effect of stress concentrations at the fiber-resin interfaces. To counteract this, so called cross-plied laminates are made having alternate orientations of fiber rotated by 90°.By way of summary, then, aligned fibrous composites are inherently anisotropic in that the maximum strength and re-inforcement are achieved along the alignment *(longitudinal)* direction. In the *transverse* direction, fiber reinforcement is virtually non-existent; fracture usually occurs at relatively low tensile stresses. For other stress orientations, composite strength lies between these extremes (Khanna, 2010).

Most practical composites that are being developed for engineering applications contain *discontinuous fibers*. Since fibers do not span the whole length of the specimen, the bond between the matrix and the fiber is broken at the fiber's end, which is carrying less stress than the middle part of the fiber. The stress in a discontinuous fiber varies along its length. The strength of the composite with discontinuous fibers is always lower than that prepared with continuous fibers. Even though reinforcement efficiency is lower for discontinuous than for continuous fibers, *discontinuous and aligned fiber compo-sites* arc becoming increasingly more important in the commercial market (Khanna, 2010).

Chopped glass fibers are used most extensively; however, carbon and aramid discontinuous fibers are also employed. These short fiber composites can be produced having moduli of elasticity and tensile strengths that approach 90% and 50% respectively, of their continuous fiber counterparts.

Normally, when the fiber orientation is *random, short and discontinuous fibers* are used; reinforcement of this type is schematically shown in Figure2.1 (c). Applications involving totally multidirectional applied stresses normally use discontinuous fibers, which are randomly oriented in the matrix material. The reinforcement efficiency is only one fifth that of an aligned composite in the longitudinal direction; however the mechanical characteristics are isotropic (Khanna, 2010).

2.6.7 Characteristics of ceramic composites

Composite materials are produced by combining two dissimilar materials into a new material that may be better suited for a particular application than either of a original material alone. The characteristics of ceramic composites are a remarkable combination of the properties of refractories and the traditional insulation materials.

a) Lower thermal conductivity

Because of the low thermal conductivity (0.1 kCal/m per hour per ^{o}C at 600 ^{o}C for a blanket with 128 kg/m^{3} density) it is possible to construct thinner linings

with the same thermal efficiency as conventional refractories. As a result of thinner lining, the furnace volume is higher (Kawaguchi et al, 2007).

b) Light weight

The average density of ceramic fiber is 96 kg/m^3. It is one tenth of the weight of insulating brick and one third of the weight of asbestos/calcium silicate boards. For new furnaces structural supports can be reduced by 40 percent (Beerkens, 2002).

c) Lower heat storage

Ceramic composites linings absorb less heat because of their lower density. Furnaces can therefore be heated and cooled at faster rates. Typically the heat stored in a ceramic composites refractories system is in the range of 2700 – 4050 kCal/m^2 as compared to 54200-493900 kCal/m^2 (20000 – 250000 Btu/Ft^2) for conventionally lined systems(Beerkens, 2002).

d) Thermal shock resistant

Ceramic composites refractories resist thermal shock due to their resilient matrix. This also allows for faster heat up and cools down cycles, thereby improving furnace availability and productivity.

e) Chemical resistance

Composite refractories resist most of the chemical attack and is unaffected by hydrocarbons, water and steam present (Limpt Van, 2007).

f) Mechanical resilience

The high mechanical resilience in ceramic composites refractories makes it possible to manufacture fiber-lined furnaces off-site, transport them to the site in assembled form without the risk of damage (Muschick, 2005).

g) Ease of maintenance

In case of physical damage, the section of damaged ceramic composites can be quickly removed and replaced with a new piece. Entire panel sections can be prefabricated for fast installation with minimal down time (Magalhaes et al, 2005).

h) Thermal efficiency

Thermal efficiency of a furnace with ceramic composites is improved in two ways. First, the low thermal conductivity of ceramic composites allow the lining to be thinner and therefore the furnace can be smaller. Secondly, the fast response of ceramic fiber to temperature changes also allows for more accurate control and uniform temperature distribution within the furnace. Other advantages offered by ceramic composites refractory (Magalhaes et al, 2005, Nìmec and Jebavá, 2006) are: lightweight furnace or kiln furniture, simple steel fabrication work, low down time, increased productivity, additional capacity, low maintenance cost, longer service life, higher thermal efficiency and faster response.

2.7 Grey Cast Iron

Grey Castiron is the most widely used of all cast metals. It is employed in greater tonnages in industry than all other castmetals combined. Typical applications, for Grey Cast iron castings are for automotive engine, blocks, machine tool castings, agricultural implements, cast iron pipe arid fittings, bathtubs, household appliances, electric motor frames and the like. The good strength wear resistance, cast ability low notch sensitivity, and high damping capacity, together with manufacturing economy, explain the extensive use of cast iron. In the production of a casting, molten metal is taken from the furnace (usually a cupola or electric furnace) and poured into a prepared sand mold.

After the metal has solidified the sand mold is broken up to allow removal of the casting. The gates and risers are cut from the casting after which it is then cleaned and inspected. Heat treating, when required, is performed after cleaning.

2.7.1 Properties of Grey Cast Iron

Grey Cast iron, or grey iron, is a type of cast iron that has a graphitic microstructure. It is named after the Grey color of the fracture it forms, which is due to the presence of graphite. It is the most common cast iron and the most widely used cast material based on weight (Degarmo et a!, 2003).

It is used for housings where tensile strength is non-critical, such as internal combustion engine cylinder blocks, pump housings, valve bodies, electrical

boxes, and decorative castings. Grey cast iron's high thermal conductivity and specific heat capacity are often exploited to make cast iron cookware and disc brake rotors (Smith et al, 2006).

Grey Cast iron is a common engineering alloy because of its relatively low cost and good machinability, which results into the graphite flakes when breaking up the chips. The graphite also gives Grey Cast iron an excellent damping capacity because it absorbs the energy. It also experiences less solidification shrinkage than other cast irons that do not form a graphitemicrostructure (Smith et al, 2006).

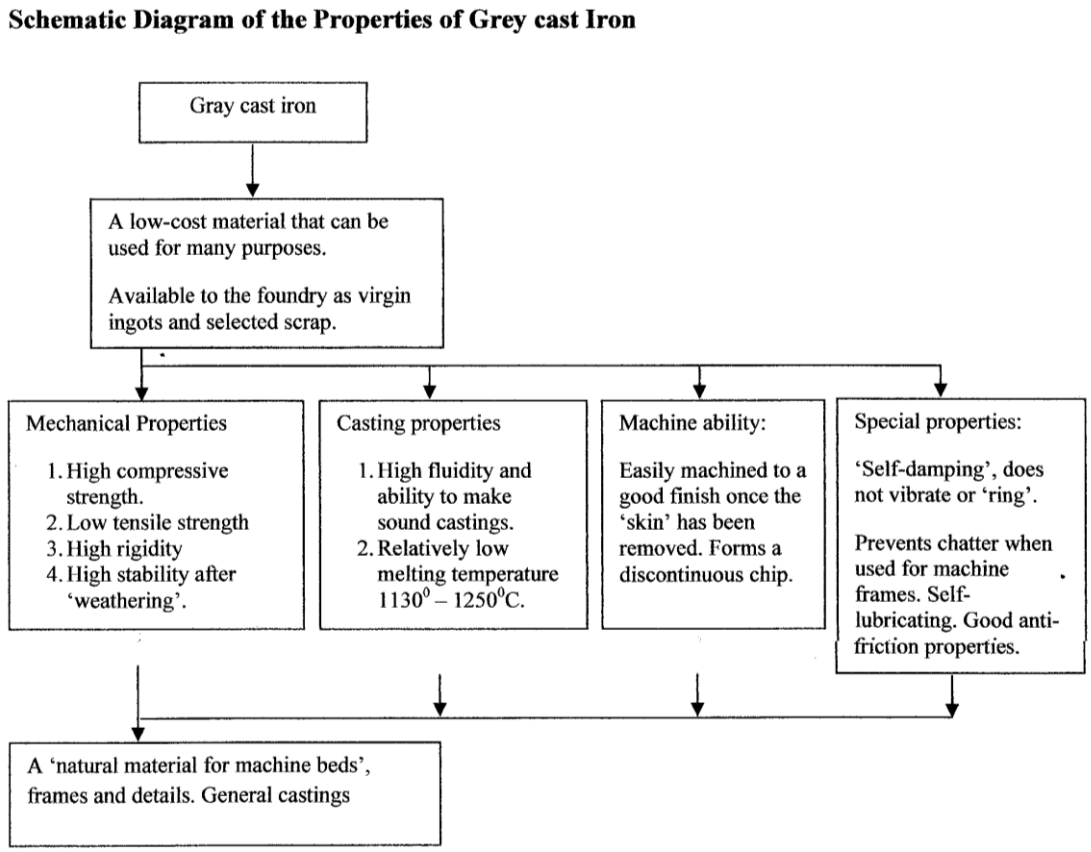


Figure (2.2): Schematic diagram of the properties of grey cast iron

2.7.2Classification of Grey cast irons

In general, Grey cast irons are classified on the basis of tensile strength. Thus a class 20 cast iron has a tensile strength of 20,000psi, a class 30 iron 30,000 psi, and so on. See Table 2-1. Tensile strength is a property that is easily understood and can be related to many other mechanical properties. A simple test for cast iron without elaborate equipment and with a test bar in the un-machined condition is known as the transverse bend test.

A test bar of standard dimensions is cast and then placed on supports located 12, 18, or 24 inches apart. The load required to rupture the bar is measured and the value obtained can be reasonably correlated with the tensile strength of the iron. The maximum deflection at the center of the bar indicates ductility of the iron. Transverse rupture loads for the various classifications are also shown in Table 2.1.Chemical analysis alone is not sufficient to designate a cast iron be-cause of the wide range of mechanical properties that can be obtained with a given analysis.

2.7.3 Microstructure of Grey cast iron

The microstructure is as important as the chemical analysis in determining final properties of a casting. Properties such as machine-ability and wear resistance are almost entirely dependent upon the microstructure. The micros tincture consists of two principal parts; the graphite flakes, and the matrix metal surrounding the flakes. The matrix structure of Grey Cast iron can be readily

changed by heat treatment but the graphite, once formed, is little influenced by heat treatment. Examination of the microstructure of cast iron is done in two steps: First, a representative specimen is polished with a fine abrasive, followed by viewing under the microscope at 25X and later at 100X to establish the size and distribution of graphite that is present. This is done by comparing the microstructure with standard charts (Figure2.2 and 2.3).

The charts are made for comparison with unetched specimens at 100X. Secondly, the specimen is etched with a recommended reagent to make the matrix structure visible. Microscopic examination of the etched specimen and identification of the matrix structure completes the examination. Three typical matrix structures for Grey Cast iron are shown in Figure2.4.

Table 2.3: Minimum tensile and transverse strength for the various classes of Grey Cast iron

Astm class	Min. Tensile strength, psi	Min. Transverse strength breaking load, lbs.		
		Test bar a 7/8 dia 18" span	Test bar b 1.2 dia 18" span	Test bar c 2.0 dia 24" span
20	20,000	900	1800	6000
25	25, 000	1025	2000	6800
30	30, 000	1150	2200	7600
35	35, 000	1275	2400	8300
40	40,000	1400	2600	9100
45	45, 000	1540	280C	970C
50	50, 000	1675	3000	10300
60	60, OO	1925	3400	12500

Table 2.4: Dimensional parameters for the test samples

	Diameter	Length	Controlling Sections
Test bar (A)	7/8”	15"	0.5”
Test Bar (B)	1.2”	21”	150.5”
Test bar (C)	2.0”	27”	1.0” – 2.0”

2.7.4. Metallurgy of Grey Cast Iron

The metallurgy of cast irons is very complex and cannot be represented on the binary iron-carbon diagram which is used in discussing steels. However, the basic composition of Grey Cast irons is often described in terms of a carbon equivalent factor (C.E.) This factor gives the relationship of the percentage of carbon and silicon in Grey Cast iron to its capacity to produce graphite. It is calculated as follows:

$$\text{C. E.} = \text{T. C.} + {}^{1}/_{3}\,(\%\text{Si}) \quad \ldots\ldots \quad (2.3)$$

Where:

T.C. = Total Carbon

(Phosphorus should be included in the formula but is here omitted because of the low phosphorus content of domestic pig irons.) Thus if an iron contained 3.4 percent carbon and 2.4 percent silicon, its carbon equivalent would be: C.E. = 3.4% + 1/3 (2.4%) = 4.2%

The carbon equivalent has metal-surgical significance by indicating the mode of solidification of the iron and thus its basic microstructure (See Table 2.2 on page 30). Irons with a carbon equivalent of over 4.3 are called hypereutectic. Those with less than 4.3 carbon equivalent are hypoeutectic cast irons. The average effect of carbon equivalent on tensile strength is shown in Figure 2.5 on

page 39. Another method of representing the type of structure which will be formed for various carbon-silicon combinations is shown in Figure 2.6 on page 39. Silicon is by, far the strongest graphitize of all the elements that may be added to cast iron. Consequently, it is the predominant element in determining the relative properties of combined graphitic carbon that will be present in the final casting. Thus, the foundry metallurgist may control the properties of the cast iron largely by adjusting silicon content relative to the amount of carbon and section size of the casting.

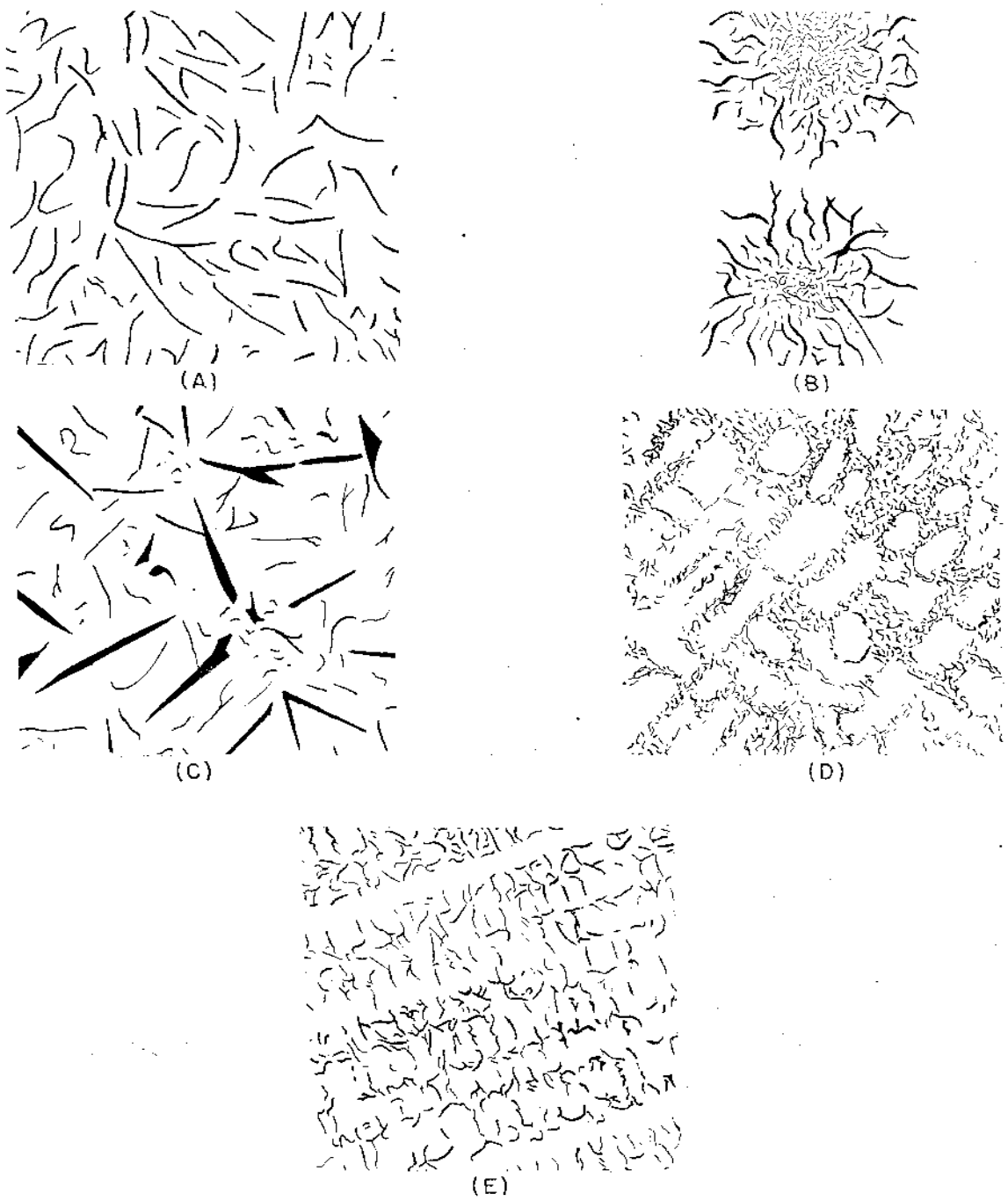


Figure2.3:Graphite flake types in grey irons. ASTM and AFS standards.100X.

Type a, Uniform distribution, random orientation. Type B, Rostette groupings, random orientation. Type C, Superimposed flake sizes, random orientation.Type D, Inter-dendritic segregation, random orientation. Type E, Inter-dendritic segregation, preferred orientation.

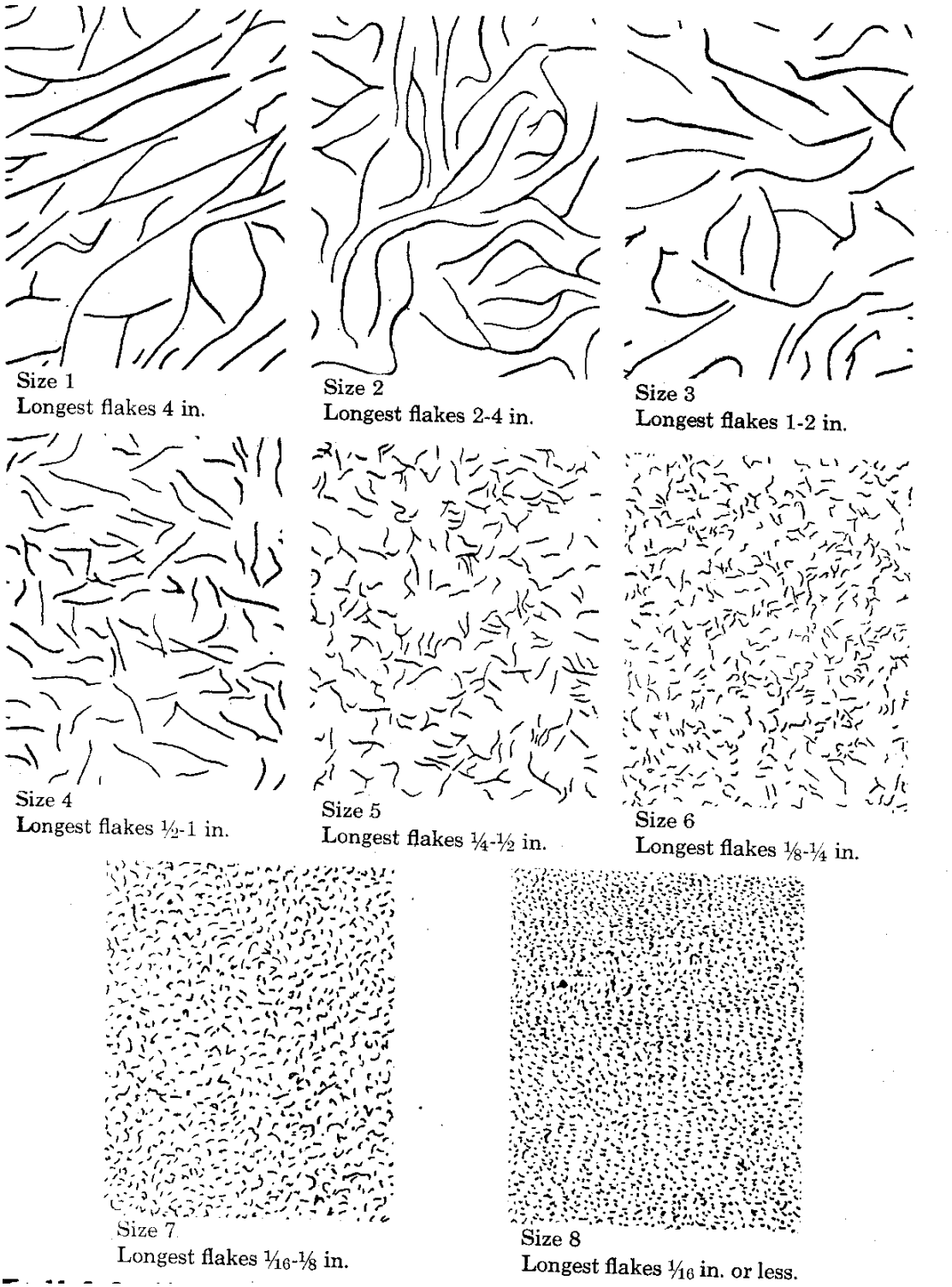


Figure 2.4:Graphite flake size in Grey Cast irons. 100X. (American Society for Testing and Materials and American Foundryman's Society.)

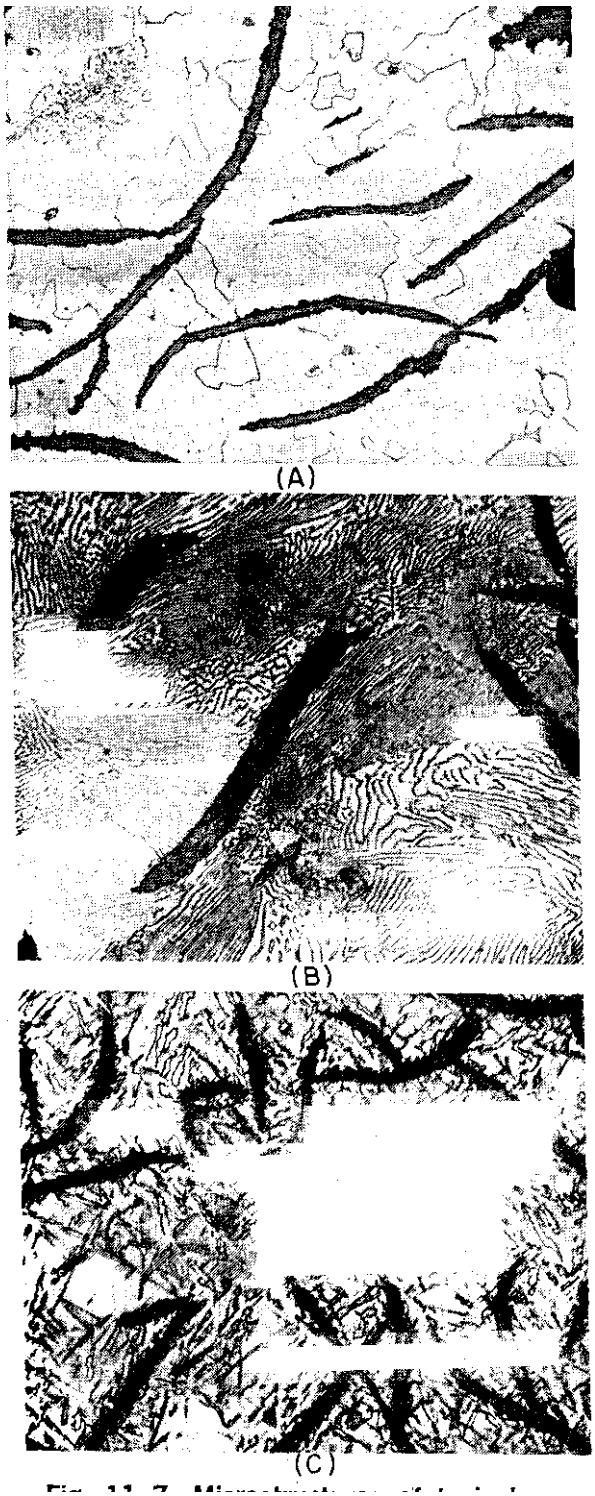


Figure 2.5: Microstructures of typical grey Irons.

Table 2.5: Cast iron types and their carbon equivalents

Type	Description	Range of section thickness, in.	Total range Carbon, percent	Silicon range, percent	Carbon equi-valent(c.e.)
S	Soft	0.10 - 0.40	3.4 -3.8	2.4- 2.8	4.2-4.5
A	Automotive	0.15 - 1.0	3.25- 3.35	2.2 - 2.4	4.0-4.2
P	Pressure	0.50 - 2.5	3.0 -3.2	1.8- 2.2	3.6 -4.0
H	Heavy section	1.5 Minimum	3.9 -3.1	1,0-1.8	3.3 - 3.6

by permission from international nickel co., inc.

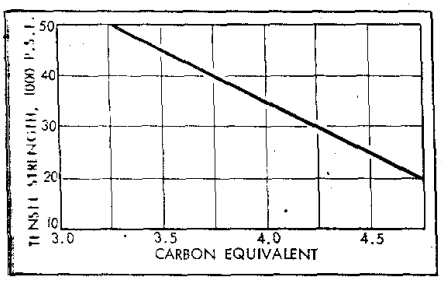


Figure 2.5: Relationship between carbon equivalent and tensile strength of Grey iron test specimens. (Grey and Ductile Iron Founders' Society) 2010

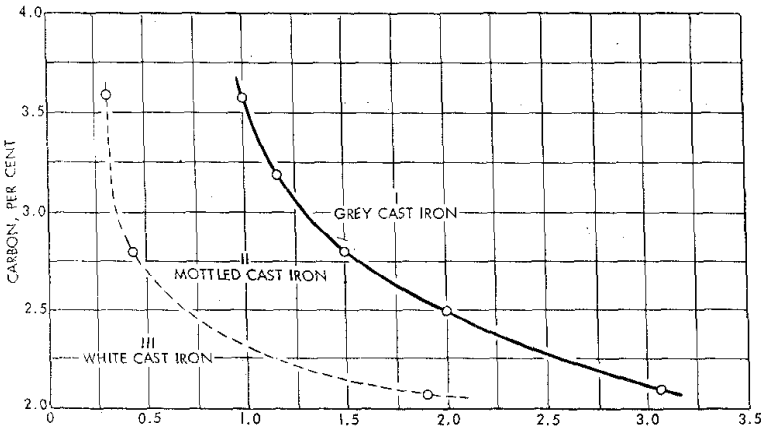


Figure 2.6: Composition limits for grey, mottled and white cast irons(Grey and Ductile Iron Founders' Society) 2010

Under commercial casting conditions Grey Cast irons solidify under non-equilibrium conditions. The cooling rate (section size) and chemical analysis determine the microstructure of Grey Cast iron and consequently its properties. Very rapid cooling (also low silicon and (low carbon contents) results in the attention of the metastable structures consisting of free carbide and pearlite.Martensite or even sonic retained austenite may also be present. Very slow cooling promotes the stable system, which consists of ferrite and graphite. The rate of cooling also influences grain size and size of the graphite flakes. Slow cooling produces soft coarse structures, and rapid cooling, fine grained, hard structure.

2.7.5 Effect of major alloying elements in Grey Cast iron

The elements commonly present in or added to Grey Cast iron may be divided into two groups according to their effect on the iron during solidification and cooling. Elements which promote graphite formation are called “graphitizes” and those which tend to retain the carbon in the form of iron carbides are termed “carbide stabilizers”. The graphitizing effect of several elements as compared with silicon as shown in Table 2.6.

Table 2.6: Graphitizing and car biding effects of several elements used in Grey Cast iron compared with silicon.

Effect	**Element**		(A) This figure holds up to about *2%* aluminum from 2 to 4% the graphitization value gradually decreases to zero (B) Mean value, 0.1 to 0.2% titanium has a stronger action than silicon on the graphite formation, while larger quantities have a lesser effect than silicon (C) The value falls to 0.05 for carbon content of over about 3% (D) For manganese contents between 0.8and1.5%. Below 0.8% manganese has a weaker effect on carbide formation, and may even have a strong graphite-forming influence below 0.6% in the presence of sulfur. (E) For molybdenum contents between 0.8 and 1.5%. Below this range molybdenum has a weaker action of graphite-formation; above it has a stronger action. (F) Mean value
Graphitizers	silicon aluminum titanium nickel copper	+ 1.00 + 0.50 (a) + 0.40 (b) + 0.35 + 0.20 (c)	
CARBIDE STABILIZERS	manganese molybdenumo hromium vanadium	-0.25 (d) - 0,30 (a) - 1.00 - 2.50 (f)	

Adapted from grey cast iron castings handbook, 1958

2.7.5.1*Silicon*

This is the strongest graphitize all alloying elements. It probably forms Fe_3Si leaving free graphite upper limit of effectiveness is at about 3.0 percent silicon. Nickel assists in graphitization but only about half as effective as silicon;

promotes density and free-from porosity by allowing lower silicon content; progressively and uniformly hardens and strengthens the matrix by of coarse pearlite to fine pearlite and finally to martensite; helps in refining the grain and promotes dispersion of graphite in a finely divided state, thus improving strength and toughness; is widely used, especially in combinations with chromium or molybdenum to obtain uniformity of castings of varying section thickness.

2.7.5.2 Chromium

This helps in the formation of stable carbides, intensifying the chilling properties of cast iron; promotes the formation of more finely laminated and harder pearlite, thus increasing the strength, hardness, and wear resistance of the iron matrix; in amounts of 1 to 1.5 percent adds resistance to softening and scaling at temperatures of up to 1400^0C tends to prevent excessive graphitization in thick sections; is frequently used in combination with nickel, copper, molybdenum, and vanadium.

2.7.5.3*Molybdenum*

This decreases rate of austenitic transformation to pearlite during cooling, resulting in finer pearlite with a consequent increase in strength, hardness, and fatigue life; is the most effective alloying element for increasing strength; promotes structural uniformity in heavy sections and improves machinability; contributes to retention of strength at elevated temperatures.

2.7.5.4*Copper*

This promotes graphite formation, but has only about one-fifth the graphitizing ability of silicon; is a mild strengthener of the matrix and tends to break up massive cementite spots; reduces hardness of iron having a tendency to chill but in normal Grey iron increases hardness, resistance to wear and certain types of corrosion.

2.7.5.5 *Vanadium*

This is a powerful grain refiner, an intense hardener and carbide former; result in greater depth of chill hardness, toughness, and resistance to wear; improves machine ability in highly alloyed irons by promoting fine pearlite structure in compositions that might otherwise be acicular.

2.8 Refractory

A refractory material is one which has the ability to withstand high temperature without breaking or deforming (Chesti, 1986). Refractory products are used wherever high temperatures are required and include refractory bricks for furnace linings, tubes for electric furnaces, crucibles, thermocouple sheaths, refractory cements, among others. The classifications of refractory materials according to their chemical nature are basic, neutral and acid refractories. The more important characteristics which are required of a refractory according stochastic (1986) are:

i. High melting point or high refractoriness, which is closely related to thermochemical stability.

ii. Mechanical strength at high temperature in terms of high refractoriness under load, high thermal shock resistance, low thermal shrinkage, low porosity and permeability.

iii. Resistance to chemical attack in the particular situation in which it is used, for instance, high resistance to corrosion by slag.

Refractories are considered as inorganic materials, mainly of mixtures of oxides, obtained for naturally occurring minerals, which are capable of withstanding very high temperature condition, without any undue deformation, softening, change in composition, they include silica, magnetite, chrome, carbon, dolomite, alumina-silicates. Most industries dealing with the treatment of ores and other materials for the manufacture of metallurgical, chemical and ceramic products operates at a very high temperature condition. So, the equipment used for the treatment of these materials must sustain the operating temperatures and other working conditions such as erosive, corrosive properties of the materials, and local conditions. In the metallurgical industry, the most commonly used refractory include those made of chromite, dolomite, magnetite, chrome magnetite, silica and alumina silicate minerals $(Al_2Si_2O_5)\ (OH)_4$. Others include fosterage, zircon refractories, zirconia refractories (Aigbodion and Asuke, 2007).

Furthermore refractories are ceramic materials that can withstand unusually high heat as well as abrasion and the corrosive effects of acids and alkalis. They are used in furnaces, stills for cracking of petroleum, ceramic kilns, boilers, incinerators, electrolytic cells for aluminium production. They are an important constituent of nuclear reactors. Jet engines would not last very long without refractory parts. Fireclay formed into firebrick and hundreds of specialized shapes, is an important raw material for refractories.

In recent years, non-clay refractories have been developed to meet increasingly severe demands. They are being made of alumina, zirconia, silicon carbide, chromic, magnetite, graphite and other less common materials. The cost of these refractories is much higher than that of fireclay. However, their use in critical parts of a furnace will keep it in operating condition for longer periods, with less time taken out for repairs.

In brief

i. Refractories are heat resistant materials.

ii. They can withstand high temperatures without being fused, (550 to 2150^{o}C)

iii. Crucibles and furnace sides and bottoms containing molten metal are made up of refractories.

iv. Refractories are used as ladles for pouring metal into the mould.

v. Refractories constitute furnace walls and roof and thus minimize heat losses.

vi. The main constituents of foundry refractories are MgO, Al_2O_3 and their mixtures.

Properties

i. Refractories should be heat, corrosion and abrasion resistant.

ii. They should possess high fusion temperature.

iii. They should possess low thermal coefficient of expansion.

iv. They should not have chemical affinity with the molten metal they hold.

v. They should be able to withstand high temperatures and pressures (due to the weight of the molten charge)

vi. They should possess heat and electric insulating properties.

2.8.1 Classification of refractories

Refractories may be grouped into the following three categories:

1. Acid Refractories.
2. Basic Refractories.
3. Neutral Refractories.

Table 2.7: Chemicomineralogical Classification of Refractories

Acid Refractories	Basic Refractories	Neutral Refractories
Silica	Agnesite	Alumina
Aluminosilicates	Magnesite-chrome	Carbon
Zircon	Dolomite	Chrome
Zircon	Foresterite	Chrome-agnesite

SOURCE: Ahmed, 1986

Hubble (1999) reaffirms that refractories may be classified in a number of ways. From the chemical stand point he said, refractory substances, in common with matter in general, are three classes, namely; acid, basic and neutral. Theoretically, he said, acid refractories should not be used in contact with slags, gases or fumes; whereas basic refractories can best be used in contact with a basic chemical environment. Actually, for various reasons, he said these rules are often violated. Hence, the time honoured chemical classification is largely academic, and has a little value as a guide to actual application.

(Hubble, 1999) further stated, "the existence of a truly neutral refractory may be doubted". Classification by use, such as blast furnace refractories is generally broad". He opined that refractories would better be classified with reference to the raw materials used in their preparation and minerals

predominating after processing for use. He believes that this classification offers the best possibility for a clear understanding of the origin and nature of steel plant refractories.

Table 2.8: Classification of refractories with fussion temperature

Type	Approx. Fusion Temperature (°C)
Acid Refractories. They are:-	
i. Silica	1700
ii. Aluminium silica (46% $A1_2O_3$ + 54% SiO_2)	1780
iii. Alumina ($A1_2O_3$)	2050
iv. Silimanite (63% $A1_2O_3$ + 37% SiO_2)	1900
Basic Refractories. They are	
i. Magnesia	2800
Bauxite and dolomite are also basic refractories.	
Neutral Refractories. They are	
i. Chromite	2180
ii. Graphite	3000

Source: Hassan, 2000

The prime ingredient for Silica refractories or acid refractories is Silica. These materials, well known for their high-temperature load-bearing capacity, are commonly used in the arched roofs of steel and glass-making furnaces; for these applications, temperatures as high as 1650°C may be realized. Under these

conditions some small portion of the brick will actually exist as a liquid. The presence of even small concentrations of alumina has an adverse influence on the performance of these refractories. Therefore, the alumina content should be held to a minimum, normally to between 0.2 and 1.0 wt. %. These refractory materials are also resistant to slags that are rich in silica (called acid slags) and are often used as containment vessels for them. On the other hand, they are readily attacked by slags composed of a high proportion of CaO and/or MgO (basic slags), and contact with these oxide materials should be avoided.

Basic refractories are rich in percales or magnesia (MgO); they may also contain calcium, chromium and iron compounds. The presence of silica is deleterious to their high-temperature performance. Basic refractories are especially resistant to attack by slags containing high concentrations of MgO and CaO, and find extensive use in some steel making open hearth furnaces. Special refractories are relatively high-purity oxide materials, many which may be produced with very little porosity. Included in this group are alumina, silica, magnesia, beryllia (BeO), zirconia (ZrO_2) and mullite (Al_2O_3-2SiO2).

Others include carbide compounds, in addition carbon and graphite. Silicon carbide (SiC) has been used for electrical resistance heating elements, as a crucible material, and in internal furnace components. Carbon and graphite are refractory, but find limited application because they are susceptible to oxidation

at temperatures in excess of about 800°C. The special refractories are relatively expensive.

2.8.2 Degree of Refractories

Refractoriness is a function of the ability of materials in use to withstand high temperature. It specifically measures the ability of materials to withstand the effect of high temperature without melting or giving way to distortion.

Hubble stated that the melting point of refractory materials in the pure state varies from 1815^0F to 3315^0F as shown in Figure-1 below

2.8.2.1 Kaolin

Kaolin derives its name from kaolinite, which is named for its type locality, i.e., Kao-Ling, Jinxi, China. Kaolinite mineral group lends its name from kaolin, which also belongs to the large general group known as the clays.

Clay minerals are defined as fine particle-sized hydrous alumina-silicates, which develop plasticity when water is introduced, and becomes hard when dried or fried. Individual slay particles are always smaller than 0.004mm. They are argillaceous matters formed from the decomposition of granite and feldspathic rocks under the action of weather. The theoretical pure clay is usually formed insitu and it has the chemical formula $A1_20_3.2Si0_2H_20$ (or $A1_2Si0_25(0H)_4$ as aluminium silicate hydroxide). Kaolinites's structure according to Amethyst galleries, Inc. (2002) is composed of silicate sheet (Si205) bonded to

alluminiumoxide/hydroxide layers (A12(0H4) called gibbsite layers. Gibbsite, according to Amethst (2002) is an aluminium oxide mineral that has the same structure as these aluminium layers in kaolinite. The silicate and gibbsite layers are tightly bonded together with only weak bonding existing within these silicate/gibbsite paired layers (called s-g layers).

Kaolinite shares the same chemistry as the minerals halloysite, dickite and nacrite. The four minerals are polymorphs meaning they have the same chemistry, but different structures. Kaolinite is by far the most common. And most clay deposits contain at least some amounts of kaolinite. In many cases, clay deposits abound that may be 100% kaolinite pure.

Kaolinite is of importance in the production of ceramics, porcelain, paper, paint and plastics. Kaolinite is useful in making refractories because of its thermal characteristics, especially when passed through Tyler mesh 200, to screen out part of its inherent sand/silica.

2.8.3 The Aluminosilicate Refractories ProductsAnd Uses

This section expounds the available information on the aluminosilicate refractory products and their uses, especially as it relates to blast furnace application. Aluminosilicate refractories refer to refractories or refractory products made of high alumina clays. Sunch clay includes kaolin, fireclays, kyanite, sillimanite,

andalusite, millite, bauxite and corundum. Refractories made from these minerals are often called clay-based refractories.

2.8.3.1 Fireclay refractories

The cheapest and common of the day based refractories n the fireclay materials. These according to the encyclopedia Britannica ultimate reference CD-ROM (2003), “are made from clay containing the aluminosilicate mineral kaolinite (A12 [SiO2][OH]4) plus impurities such as alkalis and iron oxides. The alumina contents range from 25%-45%”. Fireclay bricks, or firebricks, exhibit relatively low expansion upon heating, therefore, moderately resistant against thermal shock, they are fairly inert in acidic environments. Fireclay bricks are used in lining portions of the interiors of blast furnaces, blast furnace stoves and coke ovens.

2.8.3.2 High Alumina refractories

High alumina refractories are products of bauxite, which is a naturally occurring material containing aluminium hydroxide (A1[OH3] and kaolinitic clays. These raw materials are often roasted to produce a mixture of synthetis alumina and mullite (an aluminosilicate mineral with the chemical formular of 3A1203.2SiO2. it is said by definition, that high-alumina refractories usually have between 50 and 87.5% alumina. They are much more resistant than fireclay refractories at high temperatures and in basic environments. They also exhibit better volume stability

and abrasion resistance. High alumina clays include sillimanite, kyanite, andalusite, mullite and bauxite. High alumina bricks are used in lining blast fuenace stoves, and in liquid steel ladles. Extra high alumina refractories have been 87.5 – 100% AI2O3 content. The alumia grains are fused or densely sintered together to obtain high density. Extra high alumina refractories have volume stability to over 1800°C.

2.8.3.3 Mullite Refractories

Mullite is an aluminosilicate mineral with a chemical formular of 3A12O3.2SiO2 and an alumina content of about 70%. It melts at a temperature of 1,850°C. It is obtained by mixing various clays with bauxite.

Mullite refractories are solidified by sintering in Electric Arc Furnaces (EAF) at high temperatures. Among the aluminosilicate refractories, they are the most stable. They also have excellent resistance to high temperature grading. Mullite bricks are used in blast furnace stoves and in the fore hearth roofs of glass-melting furnaces.

2.8.4 Refractory materials

i. Refractories are obtained from ores of Silica or those of Silica and Alumina.
ii. After mining or chemical production and calcining, refractory materials are crushed, ground and prepared to size. They are then mixed with other

materials and shaped as bricks (Figure 2.7 on page 43). Bricks are used lining-melting and other furnaces. Bricks of acid, basic and neutral factories and of different shapes are available for furnace construction

iii. Bricks during construction work are bonded and cushioned with thehelp of a mortar consisting of chrome, silicon carbide, silica and alumina. Mortar may be specified by the brick manufacturer.

iv. Brick work may be coated with thin mortar for further protect the same during furnace operation.

Table 2.9: Compositions of silica and basic bricks

Type of Brick		Silica (SiO_2)	Alumina (Al_2O_3)	Lime (CaO)	Magnesia (MgO)	Iron oxide (Fe_2O_3)	Chromic oxide ($Cr20_3$)	Other oxides
2	Silica	95-97	0.2-1.2	1.8-3.5	-	0.3-0.9	-	0.05-0.3
3	Basic							
i.	Chrome	3-6	15-33	-	14-19	11-17	30-45	1-2
ii.	Magnesite	3-6	0.4-2.0	1-5	85-95	05-4.0	-	0.5-1.0
iii.	Forsterite	33-39	-	-	47-55	9-11	-	3-4

Table 2.10: Compositions of fireclays and high alumina bricks

Type of Brick		Silica (SiO_2)	Alumina (Al_20_3)	Titania TiO_2	Other oxides
(A)	Fireclay				
i.	Super duty	49-53	40-44	2.0-2.5	3-4
ii.	Semi-Silica	72-80	18-24	1.0-15	1.5-2.5
iii.	Medium duty	57-70	25-36	1.3-2.1	4-7
iv.	Low duty	60-70	21-32	1.0-2.0	5-8
(B)	High Alumina				
i.	60% Alumina	31-37	57.5-62.5	2-3.3	3-4
ii.	80% Alumina	11-15	77.5-82.5	3-4	3-4
iii.	90% Alumina	8-9	89-91	0.4-0.8	1-2
iv.	99% Alumina	0.5-1.0	98-99	Trace	0.6

Hassan (2000)

Table 2.11: Properties of refractory bricks

Type	Class	Properties
i. High Alumina	50% Alumina to 90% Alumina	1. High refractoriness increasing Alumina content 2. High mechanical strength at high temperatures 3. Excellent to fair resistance to spalling 4. Greater resistance to corrosion than fireclay bricks
ii. Fireclay	Low, medium and High duty	1. Good spalling resistance and thermal insulation value 2. Fair resistance to fluxes and acid slags 3. Lower resistance to basic slags and fluxes
iii. Fireclay	Semi-Silica	1. Rigidity under load at high temperatures

		2. Resistance to structural spalling 3. Volume stability 4. Resistance to penetration and attack by volatile alkalies or fumes
iv. Silica	Superduty Conventional	1. High refractoriness and resistance to abrasion 2. High mechanical strength at high temperatures 3. Greater thermal conductivity as compared to that of high duty fireclay brick, at high temperatures. 4. High resistance to corrosion by acid slags 5. Fair resistance to attack by oxides of lime, magnesia and iron 6. Readily attacked by basic slags and fluorine 7. Poor resistance to spalling at low temperatures
v. Chrome-Fired		i. High resistance to corrosion by basic and moderately acid slags and fluxes ii. Basic slags do not adhere to Chrome bricks iii. Iron oxide, if absorbed, causes damaging expansion. iv. Chrome bricks possess thermal conductivity lower than that of magnesite brick but higher than that of fireclay brick
vi. Magnesite-Fired ➢ Magnesia 88-90% ➢ Above 90% ➢ Chemically bonded metal encased		1. Extremely high refractoriness and high thermal conductivity 2. Poor resistance to slags containing high percentage of silica 3. Great resistance to corrosion by basic slags

		4. Chemically bonded and metal encased bricks have marked resistance to spalling
vii.	Chrome-Fired magnesite Chemically bonded	1. Excellent to good resistance to spalling 2. High resistance to corrosion by basic slags 3. Mechanical strength and stability of volume at high temperatures
viii.	Forsterite-Fired	1. Excellent strength at high temperature 2. High refractoriness 3. Attacked by acid slags 4. Fair resistance to most basic slags 5. Marked resistance to corrosion by alkali compounds

Source:Hassan, (2000)

Table 2.12: Selection/applications of different refractories

Type of Refractory	Name of the furnace used for melting ferrous metals/alloys
i. Fire Brick	1. Bottom of Induction furnace 2. Sub-hearth of O.H. furnace 3. Doors of O.H. furnace 4. Spout of O.H. furnace 5. Spout of Direct arc furnace (acid)
ii. Silica Brick	1. Side walls and roof of direct arc furnace 2. Roof of O.H. furnace
iii. Magnesite chrome brick	1. Melting zone of basic Cupola 2. Direct arc furnace roof for (Ni and Ca melting.)
iv. Dolomite	1. Backing of O.H. furnace 2. Side walls of direct arc furnace (basic)
v. Chrome magnesite brick	1. Side walls of direct arc furnace (basic) 2. Side walls of O.H. furnace. 3. Blocks and Ends of O.H. furnace
vi. High Alumina fireclay	i. Ladle refractories as lining nozzle stopper, etc.
vii. Magnesite	1. Hearth and side walls of direct as furnaces for melting nickel and copper 2. Sub-hearth of direct are furnace for melting steel (basic)

Source: Hassan,(2005)

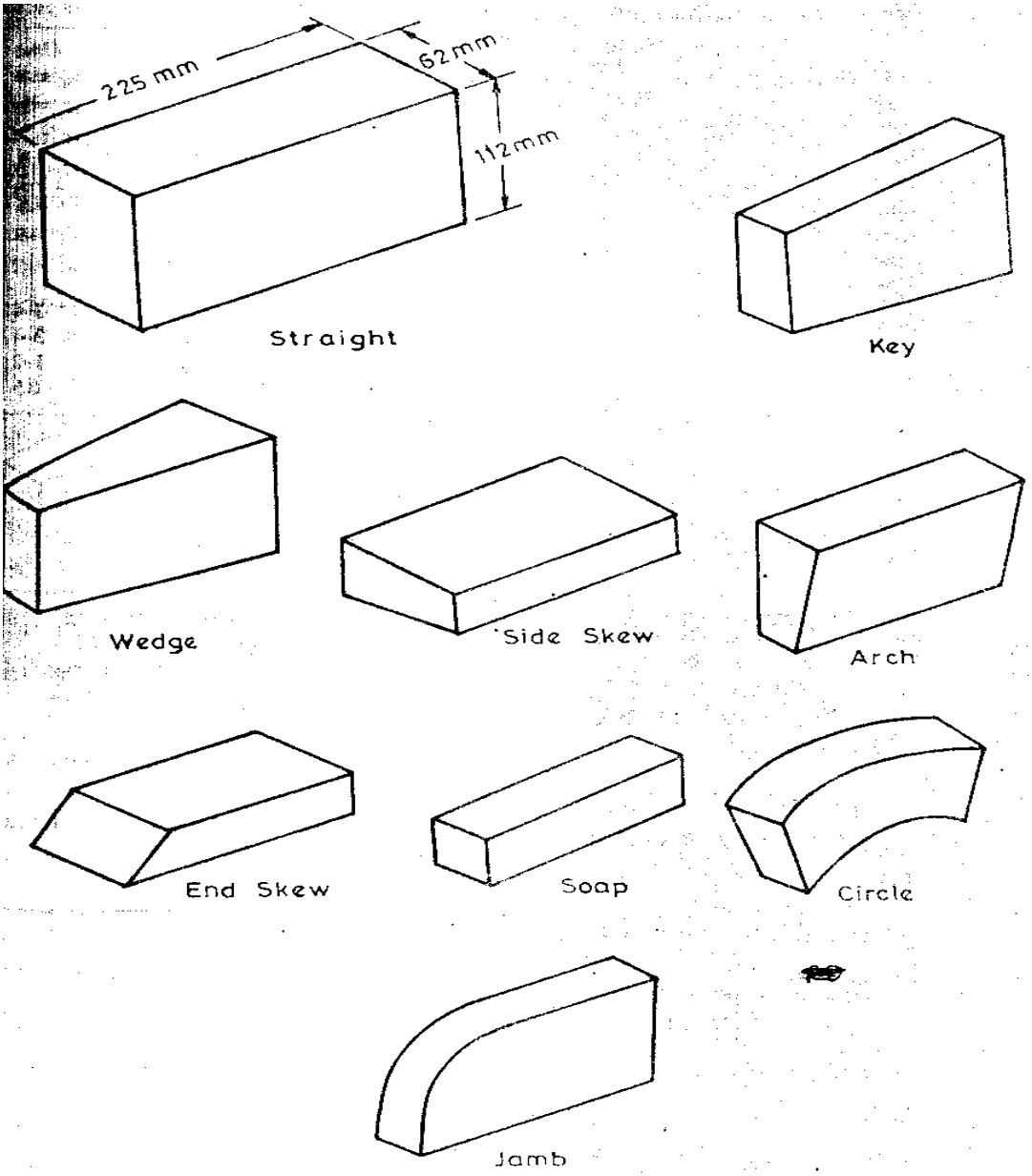


Figure 2.7: Common Shapes of Bricks (Source: Aigbodion, 2007)

Table 2.13: Refractories used in various furnaces

S.No Furnace	Refractories material
1. Iron blast furnace	Entire lining consists of fireclay but different qualities
(a) Hearth and Bosh	Coarsely ground firedclay
(b) Shaft or stock	Medium ground fireclay
(c) Top	Finely ground fireclay
(d) Hot blast stove	Porous fireclay bricks
(e) Hot blast main	Fireclay
(f) Bustle pipe	Fireclay
2. Acid Open-hearth Furnace	
(a) Portions above the working floor	Silica bricks
(b) Regenerative walls	
3. Basic Open-hearth Furnace	Fireclay bricks
(a) Roof	
(b) Side walls	Silica bricks
(c) Hearth	Silica bricks
4. Acid Bessemer Converter	Dolomite or Magnesite
(a) Body	
(b) Tuyers	Ganister
5. Basic Bessemer Converter	Fireclay
6. Basic Electric Furnace	Calcined Dolomite or Magnesite
(a) Roof	
(b) Bottom and Sides	Silica bricks
7. Cupola	A layer of fireclay bricks next to shell and upon this a layer of Magnesite
8. Reheating Furnace	
(a) Roof	Fireclay lining
(b) Hearth	Silica bricks
	Chromite or Magnesite bricks

Hassan, (2000)

2.8.5 Consumers of refractories

The chief consumers of refractories are such industries as:

i. Iron and steel

ii. Nonferrous metals

iii. Steam and power

iv. Oil refining

v. Glass and pottery

vi. Cement, lime and other minerals, and

vii. Chemicals,

2.8.6 Refractory metals

These are metals that have extremely high melting temperatures and classified as the refractory metals. Included is this group are niobium (Nb), molybdenum (Mo), tungsten (W), and tantalum (Ta). Melting temperatures range between 2468°C for niobium to 3410°C, the highest melting point of any metal, for tungsten.Interatomic bonding in these metals is extremely strong, which accounts for the melting temperatures, and, in addition, large elastic moduli and high strengths and hardness at ambient as well as elevated temperatures.

The applications of these metals are varied/ For example, tantalum and molybdenum are alloyed with stainless steel to improve its corrosion resistance. Molybdenum alloys are utilized for extrusion dies and structural parts in space vehicles; incandescent Hg filaments, X-ray tubes and welding electrodes employ tungsten alloys.Tantalum is immune to chemical attack by virtually all environments at temperatures below 150°C and is frequently used for applications requiring such a corrosion-resistant material.

2.8.7 The super-alloys

The super-alloys have superlative combinations of properties Most are used in aircraft turbine components, which must withstand exposure to severely oxidizing environments and high temperatures for reasonable time periods. Mechanical integrity under these conditions is critical; in this regard, density is an important consideration because centrifugal stresses are diminished in rotating members when the density is reduced.

The super-alloys are *classified* according to the predominant metal in the alloy, which may be cobalt, nickel or iron. Other alloying, elements include the refractory metals (Mb, Mo, W, and Ta), chromium and titanium. In addition to turbine applications, these alloys are utilized in nuclear reactors and petrochemical equipment.

2.8.8 Refractoriness versus the ceramic bond

Refractoriness is the ability of a material to withstand the action of heat without appreciable deformation or softening under particular service conditions.The *refractoriness* of refractory materials depends on chemical and mineralogical composition, on their dimensional stability on heating, and to some extent on their texture.

To obtain high refractoriness, manufacturing methods differed from those for porcelain or stoneware need to be used. The main ingredient of the body is a highly refractory, non-plastic material that should have a sufficient dimensional

stability at temperatures. For this reason ingredients that tend to shrink considerably during firing, such as fireclay, diaspore clay, silicamanite and magnesium oxide, must be well prefired to reduce their subsequent shrinkage on firing of the brick. The material is crushed into fractions of three different sizes that are mixed in suitable proportions to produce a mix of maximum density.

The loose refractory aggregate is mixed with a suitable bonding agent to provide a mass with adequate workability for shaping and forming operations and to develop a ceramic bond on firing. Refractories are generally fired at much higher temperatures than ordinary ceramic wares. Firing produces a ceramic bond and insures the necessary dimensional stability of the product when it is used for high-temperature applications.

Bond (binding*)* can be defined as a glassy matrix formed on cooling the liquid produced from the more fusible constituents of the mixture at firing temperatures. The presence of ceramic bond greatly increases the cold strength of a refractory, but it lowers its refractoriness at high temperatures. For high refractoriness the amount of glassy matrix should be as low as is compatible with the strength requirements of the refractory at room temperature.The effect of the ceramic bondon refractoriness can be illustrated by the following example:

Brick is made of a non-plastic, refractory material, which is well-fired clay, or old fireclay brick crushed to suitable size fractions, called grog. The grog is mixed with plastic fireclay as a bonding agent, which makes up as much as 50%

of the total mixture. Such a mix gives a considerable amount of the ceramic bond on firing, accounting for gradual softening and low refractoriness under-load of fireclay refractories. When temperature during firing is sufficiently high and the time is long enough, the glassy matrix may be gradually replaced by crystals. This is due to the dissolution of certain compounds and the crystallization of "elongated crystals of mullite. These tend to interlock with each 'other, giving rise to a strongly bonded mass, considerably increasing the refractoriness under load.

2.8.9 Castable and fused refractories

Refractoriesaremade by mixing refractory aggregate of suitable grading, such as alumina-silicates or high alumina, with a refractory high-alumina cement and water to a desired consistency. The mix is then cast, rammed, gunned or sprayed into shape and permitted to set until it becomes hydraulically bonded. On subsequent heating to high temperature this cementitious bond is dehydrated and replaced by a refractory bond that is developed between the matrix and aggregate particles. Castable refractories have been usedin applications where abrasion resistance at elevated temperatures is required and as a protective barrier against corrosive attack by hot gases and liquids that are highly detrimental to other structural materials.

Refractoriesare produced by mixing suitable refractory ingredients and melting them in an electric-arc furnace at temperatures of 1760-2480°C. The resultant liquid is then poured into a mold made of graphite plates burned in

refractory powder where the material solidifies and cools slowly in the mold to room temperature. The refractory ingots are then withdrawn from the mold and sawed into desired shapes and sizes. The fusion-cast process produces a unique refractory having at high density with little porosity due to large isolated voids, high hot strength, improved abrasion resistance and better resistance to corrosive attack by molten liquids and hot gases.Fusion castings of ceramics has been limited mainly to alumina, mullite, zirconia, silica, chromiumand the like.Hassan, (2000)

2.8.10 Super refractories

Higher refractoriness can be attained by using pure oxides of high melting points. The presence of even small amounts of impurities considerably lowers the melting point and reduces refractoriness under-load to a much greater extent than could be expected.Furthermore, the presence of the ceramic bond in a refractory represents an inherent weakness because it reduces their load bearing characteristics, decreases their chemical resistance to slag and fluxes, and may adversely affect their other properties. Consequently, the development of high refractory materials has been carried out along two lines - *first*, the use as very pure ingredients of high refractoriness and second, the elimination the ceramic bond by adoption of special method and *technique* in manufacturing procedures.

These techniques may involve compacting of fine powderfollowed by sintering at a suitable temperature at which gradual crystalization at the grain

boundaries occurs, binding the particles into a coherent, strong body, Since the bond is composed of crystals of the same material as the particles, self-bonded refractories exhibit High refractoriness, approaching that of the pure material itself.

Table 2.14: Physical properties of some super-refractory

Super-refractory Compound		Specific gravity	Melting point (°C)
Alumina,	Al_2O_3	3.97	2050
Beryllia,	BeO	3.03	2550
Magnesia,	MgO	3.58	2800
Silica,	SiO_2	2.65	1710
Thoria,	ThO_2	9.69	3300
Silicon carbide,	SiC	3.17	2540

Hassan, (2000)

2.9 Cermets

Cermet is an acronym derived from the words ceramic and metal, which are two major phases of this material. The ceramic phase includes the carbides, nitrides and carbonitrides of titanium, molybdenum, tungsten, tantalum, niobium, vanadium, aluminum and their solid solutions with titanium nitride as the major

constituent. The metallic binder phase consists of nickel alloyed with cobalt or molybdenum and constituents of the ceramic phase, depending on their solubility (Gruss and Friederich, 1995; Dobrzanski, (2002); Jaworska et. al, (2005)). The wear and corrosion-resistance properties of the carbide or nitride matrix are combined with the enhanced toughness of the metallic phase. The wear behaviour of the cermet materials is a function of both hardness and toughness (Hussainova, 2003; Brookes, 1989; Stricker, 1993).

Ceramics particles react with the metal bonding phase, which is in liquid solution resulting in densification of the composition through particle rearrangement by capillary forces (Gruss and Friederich, 1995; Umansky et. al,). The most popular method of the cermets obtaining is powder Metallurgy processing techniques with free sintering or using Sintering under pressure, for example HIP method (Roso, 2003). Metal bonding phase guarantees the material good wettability and adhesion to the ceramic phase. Good adhesion forces between ceramics and metal bonding phase results the higher toughness due to crack propagation through the ductile binder phase. However presence of the metal phase has influence on the cermets hardness decreasing. During the cermets sintering process there is almost not grain growth process. The first cermet TiC-Mo2C-(Ni,Mo,Cr), Titanit S was obtained by Plansee in 1930 but this material was not introduced because of their brittleness and problems with the materials

brazing to the tools body. The TiC – (Ni,Mo) cermets were introduced by Ford Motor Co. in 1960 (Ettamayer et al., 1995).

The success of cermets is based on their superiorwear resistance for machining ferrous and non-ferrous metals over a wide range of cutting speeds. At higher temperatures, cermets are oxidation resistances and they have better cutting properties, very good adhesion and diffusion wear resistance comparing to the cemented carbides. Therefore, they could work without lubricants. Cermets guarantee better surface quality after machining. Cermets have some defects in compare to the cemented carbides tool materials such: lower fracture toughness (but higher than other ceramic tool materials), lower resistance for the plastic deformation, higher thermal expansion (Ettamayer et al., 1995).

2.9.1 Production of Cermet

Cermet production is not notably different from the production of hard metal. The selected raw materials based on carbide, carbonitride and nitride are milled and spray dried in order to produce a granulate that can be pressed. The following pressing and sintering operations result in a compact and hard form. Further finishing operations such as grinding, honing and coating then produce a ready for use indexable insert (Plansee, 2013).

Compared to hard metal, powder preparation, pressing, sintering and grinding are more complex operations. State-of-the-art processes and production plants represent the basis for high-quality cermets manufactured by (Plansee, 2013)

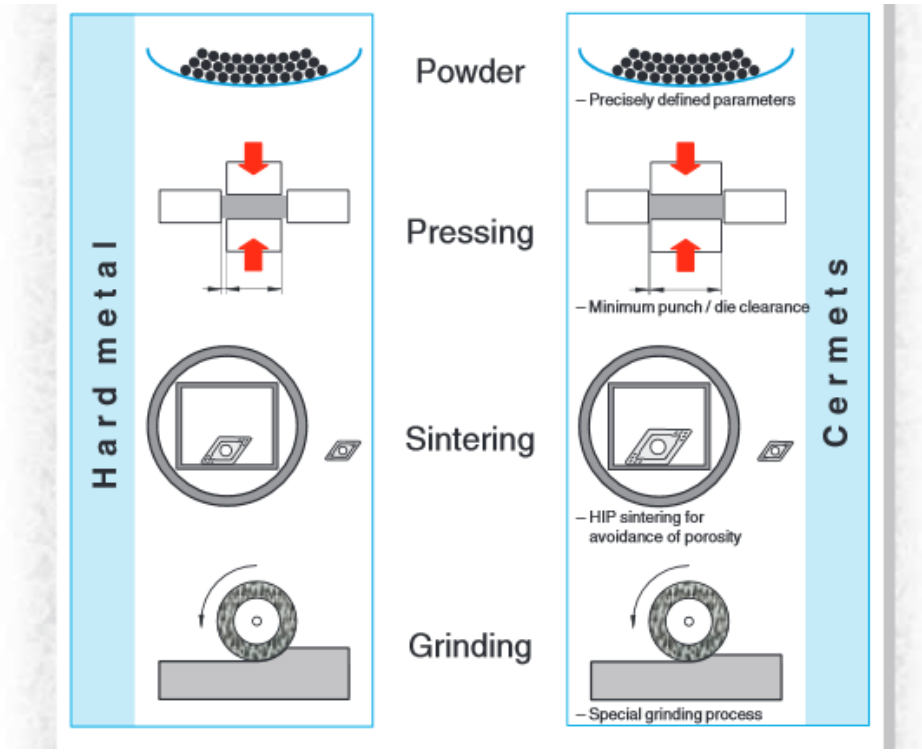


Figure (2.8): Cermet production process

2.9.2 Applications of Cermet

2.9.2.1 Ceramic-to-metal joints and seals

Cermets were first used extensively in ceramic-to-metal joint applications. Construction of vacuum tubes was one of the first critical systems, with the electronics industry employing and developing such seals. German scientists recognized that vacuum tubes with improved performance and reliability could be produced by substituting ceramics for glass. Ceramic tubes can be outgassed at higher temperatures. Because of the high-temperature seal, ceramic tubes withstand higher temperatures than glass tubes. Ceramic tubes are also

mechanically stronger and less sensitive to thermal shock than glass tubes (Pattee, 1968).

Today, cermet vacuum tube coatings have proved to be key to solar hot water systems.Ceramic-to-metal mechanical seals have also been used. Traditionally they have been used in fuel cells and other devices that convert chemical, nuclear, or thermionic energy to electricity. The ceramic-to-metal seal is required to isolate the electrical sections of turbine-driven generators designed to operate in corrosive liquid-metal vapors (Pattee ,1968).

2.9.2.2 Bio ceramics

Bioceramics play an extensive role in biomedical materials. The development of these materials and diversity of manufacturing techniques has broadened the applications that can be used in the human body. They can be in the form of thin layers on metallic implants, composites with a polymer component, or even just porous networks. These materials work well within the human body for several reasons. They are inert, and because they are reabsorb-able and active, the materials can remain in the body unchanged. They can also dissolve and actively take part in physiological processes, for example, when hydroxylapatite, a material chemically similar to bone structure, can integrate and help bone grow into it. Common materials used for bioceramics include alumina, zirconia, calcium phosphate, glass ceramics, and pyrolytic carbons.

One important use of bioceramics is in hip replacement surgery. A hip joint essentially is a multiaxial ball and socket. The materials used for the replacement hip joints were usually metals such as titanium with the hip socket usually lined with plastic. The multiaxial ball was tough metal ball but was eventually replaced with a longer lasting ceramic ball. This reduced the roughening associated with the metal wall against the plastic lining of the artificial hip socket. The use of

ceramic implants extended the life of the hip replacement parts (http://www.manufacturingcenter.com)

Cermets are also used in dentistry as a material for fillings and prostheses.

2.9.2.3 Transportation

Ceramic parts have been used in conjunction with metal parts as friction materials for brakes and clutches(Pattee, 1968)

2.10 Some Previous Studies on Ceramic-Metal Composites

Chakladerand Linger**,** (1976) developed ceramic/metal composites based on nickel/alumina and copper/alumina particulate systems. They observed that uniform distribution of metal phase along the alumina grain boundary was accomplished by coating of the alumina powders. Up to 68 weights % of nickel and 32 weight % of copper was deposited on the random shaped alumina particles. The coated particles were hot-pressed and sintered for densification.

Konopkaet al (2003) developed Ceramic–metal composites with an interpenetrating network; their preliminary results of studies of ceramic–metal composites Al_2O_3–Fe obtained via infiltration of a porous ceramic matrix are reported. Composites with a thin $FeAl_2O_4$ spinel layer, as observed in their work, on the ceramic/metal interface, will give an opportunity to develop an interpenetrating network with a strong ceramic–metal bonding.

Abed et al (1995) studies interfacial behaviour in ceramic-metal composites. A series of experiments was carried out to test the validity of a chemical thermodynamic approach to predicting the effect of nitrogen alloying of stainless steel on its interaction with silicon nitride during sintering of si-alon ceramic matrix composites. Their approach has been shown to be valid and several compositions of si-alon-steel composites have been fabricated. The steel particle size and the amount of steel in the composite are also shown to have an influence on the extent of densification. Conditions are described for optimum sintering of this type of material which has been shown to have application in several areas including ceramic-metal joining.

Hassan and Aigbodion, (2014) aimed at improving the refractory properties of Kankara clay (alumino-silicate) found in Kankara village , kastina state, Nigeria by blending with coal ash for the production of refractory bricks. Coal ash additions were varied from 5 to 25% wt in the blend. The addition of coal ash to Kankara clay enhanced the refractory properties of the clay.

CHAPTER THREE

EXPERIMENTAL METHOD

3.1 Introduction

The chapter describes and discusses the materials,equipment, preliminary studies on the Kankara clay and all the general techniques used in this research which includes: production of the composites, microstructural analysis, thermal ageing, X-ray Diffraction analysis, X-ray fluorescence analysis kinetic study of the ageing process, physical and mechanical properties of the developed ceramic metal composite.The processing of the ceramic composite powder mixtures, forming procedures are also described in this section. The characterization methods applied on the Kaolin (Kankara) and ceramic kiln shelves, bars and prop composites products by water adsorption and density measurement are explained. The mechanical properties measurement of the composite refractory made is also discussed in this section. The following procedures were used in the course of this research.

3.2 Materials Collection

The following under listed materials are the items used in the course of this study to produce test pieces using the Compression Testing Machine (CTM) by pressure sintering.

i. Kaolin (Kankara clay), which was used as the aluminosilicate is the matrix.

ii. Grey cast iron powder was also used as reinforcement.

iii. Water was used to wet mix the materials.

The Kankara clay (Kaolin) used in this work were obtained from Kankara area of Kastina State, Nigeria. The Grey cast iron particles used were obtained from National Metallurgical Development Centre, Jos, Nigeria. The photographs of the Kankara clay and Grey cast iron particles are shown in plate (3.1-3.2).



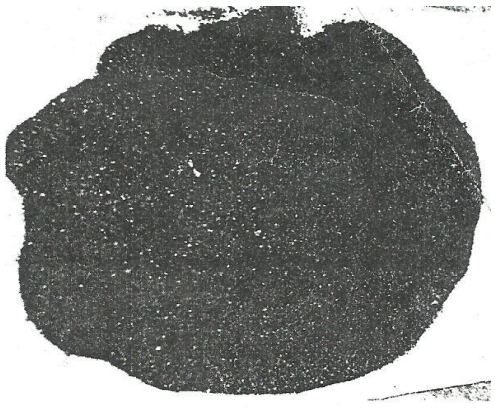


Plate 3.1: Kankara clay particles **Plate 3.2: Grey cast iron particles**

3.3 Equipment Used for Research

The equipment that were used in this research work include:

i. Soil Test Compression Machine.
ii. Universal Testing Machine.
iii. Density Bottles.
iv. Furnace.
v. Charpy Impact Machine.
vi. Hardness Testing Machine (Durometer).
vii. Vernier Caliper.
viii. Digital Balance.
ix. Thermometer.
x. Scanning Electron Microscopy(SEM)/Energy dispersive system(EDS)
xi. X-ray diffraction (XRD)
xii. X-ray fluorescence (XRF)
xiii. Crucible
xiv. Metallic mould
xv. Blunger Machine
xvi. Pugging machine

3.4 METHODS

3.4.1 Preliminary Work on the Kankara clay and Grey cast iron

3.4.1.1 Processing of the raw Materials

After collecting the kaolin from its deposit in Kankara, Katsina State of Nigeria and grey iron powder from National Metallurgical Development Centre, Jos, the kaolin was beneficiated by wet screening it through a 75µm mesh as deposited by (Schussler and ZumGahr, 1993). Other processing routes that followed include calcining, crushing, grinding, sieving and mixing of the composite refractory batch as shown in the processing chart in fig (3.1). The grey cast iron was also grated, grindered, burr milled and sieved into the required grain sizes of 100µm.

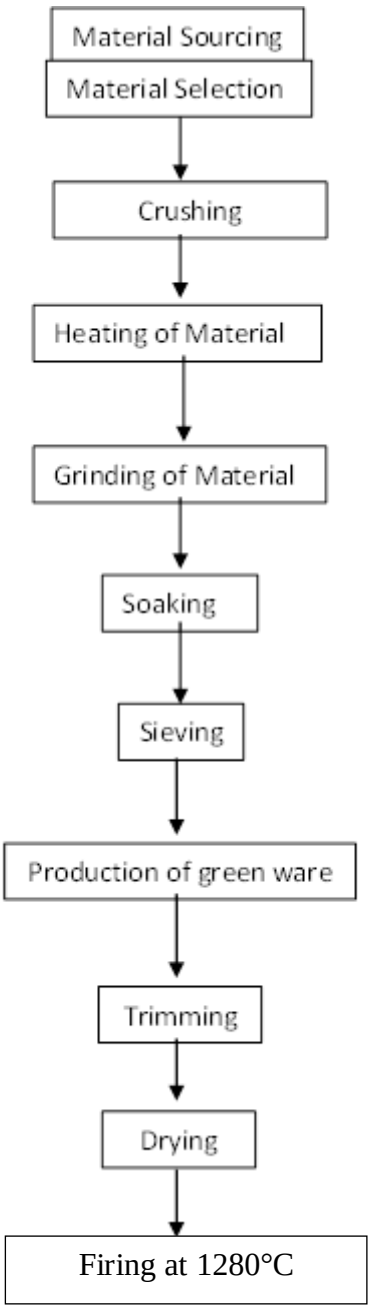


Figure 3.1: Material Processing ProcessChat

3.4.1.1.1 Screening and beneficiation

The Kankara clay as collected from its deposit in Kankara Kastina state was screened to remove unwanted particles and materials.The screened clay was beneficiated by soaking in water for a day (24 hours) see plate3.3. It was stirred and sieved manually washing with water and wet sieving through a 75µm mesh aperture size. The water was decanted while the material was placed on plaster bats for hardening into plastic state. The resulting plastic clay was prepared into rough block form and dried for 14days under the room temperature after which it was fired in a kiln.



Plate 3.3Beneficiation of kankara clay for the production of sample

3.4.1.1.2 Kankara clay calcination

The Kankara clay was packed in an air tight graphite crucible and placed inside a manual kiln and fired at gloss temperature of 1200°C for 5 hours to obtain the calcined Kankara clay which was used in this research.

3.4.1.1.3 Crushing

The calcined kankara clay was removed from the kiln and allowed to cool to room temperature which was then crushed to smaller particle size by means of pestle and mortar.

3.4.1.1.4Grinding

The crushed Kankara clay was grinded to fine particle sizes (powder) by means of a Burr mill.

3.4.1.1.5 Particle size analysis

The particle size distributions of the Kankara clay were determined using the (AFS) specifications. 100g each of the dried Kankara clay was taken and introduced in a set of sieves arranged in descending order of fineness and shaken for 15 minutes which is the recommended shaking time to achieve complete classification. The weight retained on each sieve was taken and expressed as percentages of the total sample weight. From the weight retained and the grain fineness number (AFS) were computed. The particles retained on the 75μm mesh aperture were used for the composite development.The sieving analysis which was carried out to determine the proportions of the different size fractions of the clay samples was as presented in fig. B5- B7 (Appendix B). The analysis showed that about 50% of the clay was made up of large clay

particles (coarse sand) not suitable for pottery. For the clay to be suitably used for ceramic composite production, a lot of additives would have to be included. In addition the clay would have to be mixed in a suitable proportion with another body like kaolin to increase its suitability or charge to different if possible.

3.4.1.1.6 Compositional Analysis and Microstructure of the Kankara clay

The X-ray diffraction patterns of the calcined sample derived from the Kankara clay was determine by X-ray diffraction analysis which was carried out with a Siemens D-500 diffractometer using Co-Kc radiation (Kc = 1.79026 A). The microscopic study of the ash were determined by JEOL JSM840A scanning electron microscope (SEM) complemented by EDS. The X-ray diffractogramand microstructure of the Kankara clay is presented in plate4.11 and plate4.1 respectively.

3.4.1.1.7 Density

The density of the respective Kankara clay was determined basically by measuring the mass and the volume by using the beam balance and the measuring cylinder respectively. It is then estimated from the formula given in(equation 2.30).Density of the bagasse ash is 1.72g/cm^3 which means that Kankara clay is a very light material. The value obtained fall within the range of density of carbon and silica which is 1.8 and 2.2 g/cm^3 respectively (Hassan, 2005)

3.4.1.1.7 Refractoriness

The Pyrometric Cone Equivalent (PCE) as recommended by ASTM Test C-24 was used in the determination of the refractoriness of the kankara clay (Aigbodion and Asuke, 2007, Hassan, 2005). The kankara clay was observed to have Seger Cone No. 23, with equivalent temperature of $>1500^{o}C$ as shown in table 4.5. This means kankara claycan withstand operating temperature of $>1500^{o}C$ without load. From these preliminary studies one can observe that kankara clayhave some of the properties of CMCs matrixdiscussed in the literature.

3.5 Development of the Ceramic Matrix Composites materials

The synthesis of the Ceramic Matrix Compositesthat was used in this study was produced using compression moulding method. The samples were produced by varying the %wt of grey cast ironwith particle size of 100µm in the range 5-45wt% at an interval of 5% addition.

The sieved particles of the Kankara clay and grey cast iron were wet blended in the right proportions with overall weight of 400g at 5%Wt interval according to the formulation in table 3.1. A homogeneous mixture was obtained by blending the filler (Grey cast iron) and binder

(Kankara clay) in a Blungermachine(see plate 3.4) for 30min and then transferred to a ponging machine (see plate 3.5) for5minutes at a speed of 1000rpm.the homogeneous mixture of constant mass of 400g were then allowed to age for two days under room temperature which was then compressed into a wood/metal mould of various shapes and capacity for accommodating the production of the composite samples. The moulded samples are allowed to dry for fourteen days under room temperature, sun dried for two days and transferred into the kiln for firing at a temperature of 1280°C for 5hours (see plate 3.6 and 3.7).

Table 3.1: Composite blends formulation

S/n	**Kaolin Powder (%)**	**Grey cast iron powder (%)**
1	100	0
2	95	5
3	90	10
4	85	15
5	80	20
6	75	25
7	70	30
8	65	35

9	60	40
10	55	45



Plate3.4: Blunger machine for mixing the composite material



Plate 3.5: Pugging machine for blending and softening leather hard clay



Plate 3.6: Using digital kiln for firing test sample



Plate 3.7: Test samples parked inside manual kiln for firing

3.6 Methods/Standards for Testing Properties of Materials

Natural materials are usually made up of different physical, chemical, mineralogical and other characteristics. These diverse natural differences in materials and their behaviours are generally brought about by their geologic origins and formation.

"Amethyst Gallery Inc'. (2000) stated that minerals can only be absolutely identified by x-ray analysis and chemical tests. The x-ray analyses the structure of the mineral, and the chemical tests determine the composition of the mineral. Structure and composition are the defining marks of a mineral, according to Amethyst, (2000).

Unfortunately for the average collector or researcher, these tests require expensive equipment, expert knowledge and lack of these often destroy the specimen. Fortunately, both structure and composition affect certain physical properties of minerals. It is through the idea and appropriate use of these properties that minerals can reliably be identified.

An idealized physical property is one that will give a unique result for each mineral and will always give the same result for each mineral over and over again for any and every specimen of that mineral. This is an ideal situation. Mineralogists are usually delighted to have a property that is simply consistent in providing the same result for every specimen of a certain mineral. At times a property may have a good range of possible result, so that two similar minerals might have a good chance of having different results.

3.7 Characterization of the Ceramic Composites

A series of characterization and analysis of the refractory made were subjected to apparent porosity determination, Bulk density and density measurement, firing shrinkage determination, leaching test, plasticity test and mechanical property determination and microstructural analysis were also carried out. These methods are explained briefly in this section.

3.7.1 Physical properties

The Physical properties that were determined for the different compositions developed include;

3.7.1.1 *Density*

The densities of the developed samples were determined using the ratio of their weight to their volume. The volumes of the samples were determined by the products of their length, width and height. Then their masses were obtained using a digital scale. The densities were then calculated using equation below. (Clyne, 2000)

$$\text{density} = \frac{\text{mass}}{\text{volume}} - - - - - - - - - - - - - - - - - - 3.5$$

3.7.1.2 *Water Absorption (W.A)*

Samples after firing were soaked in water at room temperature for a period of 24 hours; their weights were taken before and after soaking in water. The water absorbtion was determined as the percentage of water absorbed by the samples using the equation below.

$$\%W.A = \frac{Ww - Wd}{Wd}\ x\ 100 - - - - - - - - - - - - - - - - 3.6$$

Where;

Wd – dry weight before soaking

Ww – wet weight after socking

3.7.1.3 Plasticity Test:

The clay sample was soaked, and washed (sieved) through a fine mesh and allowed to settle overnight (24 hrs). The unwanted water that accumulated on top of the settled clay was siphoned and the slip poured into moulds to further dewater the clay. It was allowed to dry and wrapped in a polythene bag for two days to aged. A portion was then rolled and twisted around the finger. If there were no series of cracks or breaks on the twisted clay, it meant, the clay was quite plastic in nature and was good clay Otherwise, the clay was bad if it cracks and not suitable for pottery on its own.



A: plastic clay no cracks B: Non plastic clay with series of cracks

Figure 3.2: Plasticity Test samples

3.7.1.4 Bulk density and apparent porosity

The type of density most commonly determined for a ceramic object is the bulk density, which is the weight divided by the bulk volume. The density and porosity of the samples were determined using Archimedes'

technique (ASTM C 20-87). This method involves the determination of the following properties of sintered products: apparent porosity, water absorption, and bulk density. The dry weights of sintered specimens were recorded as the initial step of this method. Each test specimen was boiled for 2 hours in water. After boiling, each specimen was cooled to room temperature while still completely covered with water, and immersed in water for a minimum of 12 hours before weighing. The suspended weight in water of each specimen was measured on the balance with Archimedes' apparatus (Precisa-XP220A). After determining the suspended weight, the specimen was wiped with a wet sponge to remove drops of water from the surface. The damp specimen was then weighed to determine its saturated weight. The density and other properties were determined based on these three weights. In this case dry weight was noted as (D). Suspended weight was noted as (S) while saturated weight was (W).Volume of specimen = W_2/ D_m where D_m = density of mercury

$$\text{Bulkdensity} = \frac{W_1}{W_2} D_m \quad ----------3.1$$

The apparent porosity is then calculated as:

$$P_{(app)} = \frac{W - W_D}{W - W_{SD}} \times 100\ (\%) \quad ---------- \ 3.2$$

Where;

$W - W_D$ = actual volume of open pores of the specimen.

$W - W_{SD}$ = External volume of the specimen.

3.7.1.5 Shrinkage Test

A slab of clay measuring 15cm long and 1.5cm wide was made. Measurements were impressed on it as shown in figure 2. It was then allowed to dry and the impressed measurement in the centre re-measured. (The Venier caliper measurement was used to measure shrinkage), Bisque fire was then introduced to 980°C and the bar was re-measured. The bar was again fired to gloss temperature of 1280°C and above and measured again to determine the total clay shrinkage from dry to gloss.

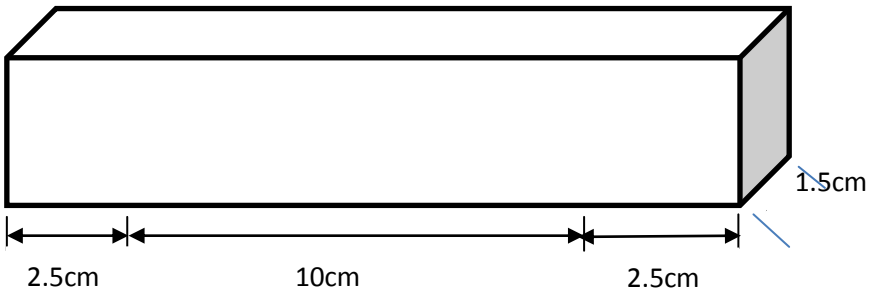


Figure3.3: Shrinkage bar

If the total shrinkage was more than 1cm the clay had high shrinkage rate and therefore not suitable for pottery work on its own.

3.7.1.6 Absorption Test

This was carried out as follows. Soft clay was moulded into small balls, allowed to dry and the weight measured. They were then fired to gloss firing temperature of 1280°C and soaked in water for 24hrs. A piece of dry cloth was used to clean off dripping water on removal from the water and the balls re-weighed. The weight difference between the gloss fired weight of the 'balls and the weight after they were soaked for 24 hrs in water was a measure of the absorption capacity of the clay.

3.7.1.7 Vetrification Test

This was carried out by firing clay composite continuously to high temperatures (1280°C and above). If the clay composite fused at a low temperature it was considered poor clay and not suitable for stone-wares or pottery work; since it did not withstand very high temperatures.

3.7.1.8 Linear shrinkage

The ceramic composites were made by mould pressing, at their green state; the samples were allowed to dry until they could be removed conveniently from the dry mold then their dimensions were taken. They were then fired in the furnace to 1600^{o}C then the new dimensions were measured, using the vernier caliper, and recorded. The linear shrinkages were then calculated as a percentage of the original wet length as shown below:

$$\%\ \text{of fire shrinkage} = \frac{L_B - L_D}{L_B} \times 100\ (\%) \quad ---------- 3.3$$

Where, L_B = Length of green shelves, L_D =Length of fired shelves.

3.7.2 Mechanical properties Determination

Mechanical properties that were determined for different compositions developed include;

3.7.2.1 *Cold crushing Strength (CCS)*

The cold crushing strength of the ceramic matrix composite was conducted using the soil compression hydraulic crushing machine (see plate 3.8).Test samples that were used to determine the cold crushing strength (CCS) were soaked in water at room temperature for a period of 24hours, wet samples were wiped with damped towel to remove dropping water then they were placed between two platens of the soil test compression machine model 4839 S/No 1482. An asbestos board of about 5mm thickness was placed between the platens of the press and bearing faces of the test pieces which were placed centrally on the platen. Load was applied at a rate of 20kN/minute using the hydraulic strength testing machine. The maximum load at failure was recorded as the crushing load. The cold crushing strength was calculated using the relationship

$$C.C.S = \frac{\text{Max Load (N)}}{\text{Area } (m^2)} \quad ---------- 3.4$$



Plate 3.8: Soil Test Compression Machine

3.7.2.2 Hardness values determination

The hardness test of composites is based on the relative resistance of its surface to indentation by an indenter of specified dimension under a specified load. Hardness of the composites were determined by using a direct reading Durometer, manufactured by Francisco Munoz Irles, C.B. model: 5019 and Serial No: 01554 (see plate 3.9). The Durometer measures the hardness of material according to ASTM D2240 ISO 7619. The Durometer measures in Shores.This test was carried out in the Nigerian Institute for Leather Science and Technology (NILEST), Samaru, Zaria.



Plate 3.9: Hardness Testing Machine (Durometer)

3.7.2.3*Tensile Strength*

The tensile test was also carried out on soil test compression machine model 4839 S/No 1482. For the test, the specimen was placed diagonally between the platens of the machine. The maximum loads reached before failure by the samples were measured and the tensile strengths were calculated using equation below;

$$\text{UTS} = \frac{\text{Maximum Load (N)}}{\text{Area (m}^2\text{)}} \quad - - - - - - - - - - - - - - - - 3.7$$

3.7.2.4*Compressive Strength*

The compressive test was also carried out on soil test compression machine model 4839 S/No 1482. The test was similar to the cold crushing strength testing but the samples were not soaked before testing. The maximum compressive loads reached by the samples before failures were measured and the compressive strengths were calculated using equation below;

$$\text{UCS} = \frac{\text{Maximum Compressive Load (N)}}{\text{Area (m}^2\text{)}} \quad \text{---------- 3.8}$$

3.7.3 Microstructural and Interfacial analysis

3.7.3.1 Scanning Electron Microscope Analysis (SEM)

Micrographs of the developed composite material samples were taken using Scanning Electron Microscope (SEM) EVO MA-10 manufactured by Carl.Samples of the composite material were cut to dimension 10mm X 10mm X 5mm. The material being ceramicsare not good conductor so they were coated with gold to allow the conduction through them. Samples after preparation were attached to multi-stub sample holder with the use of double-sided conductive carbon tape, after which, they were mounted onto the specimen chamber, while the column was put at vacuum. After reaching the vacuum target, the electron gun was switched on which passed accelerating voltage of 20kV and probe current of 227pA through the samples at a working distance of 7.0mm and/or 6.0mm. This was done atpictorial, Johannesburg, South Africa.

3.7.3.2 X-ray Diffraction Patterns of Composites

The developed compositeswereinvestigatedtoidentifyandquantifythereaction products in themusingX-raydiffraction (XRD) technique. The X-ray diffraction patterns of composites were determined by X-ray analysis which was carried out to determine the various element and phases distribution in the samples. The test wascarried out on a Philips X-ray diffractometer. The samples were prepared in diffraction data and collected with a BrukerD80-θX-ray diffractometer equipped with CoKα monochromating multilayered mirrors on the incident and beam sides at scan speed of 3m/min. A Rietveld refinement software, TOPASTM, was used for quantitative analysis.The particle size of the samples was calculated employing the Scherres equation:

$$D = \frac{K\lambda}{\beta Cos\theta} \quad \text{---------- 4.1}$$

Where θ is the angle between the incident and diffracted beams (degree), β the full with half maximum (rad.), D the particle size of the sample (nm) and λ that is wavelength of the X-ray.

3.7.3.3 X-Ray Fluorescence Test *(XRF)*

XRF test was carried out on X-ray spectrometer, designed for elemental analysis of wide range of samples. The machine makes use of PW 4030 X-ray Spectrometer, which is an energy dispersion, microprocessor controlled, analytical instrument, designed for detection and measurement of elements in sample ranging from sodium to uranium.

The test was carried out on the developed composite sample. The samples were grinded into powder form then weighed. A binder (PVC dissolved in Toluene) was added to the samples, which were carefully mixed and pressed in hydraulic press into pellets.The pellets were loaded in the sample chamber of the spectrometer then, voltage (300KV maximum) and a current (1mA maximum) was applied to produce X-ray, which excited the samples separately for 15mins. The spectrum from each sample was analyzed to determine the concentration of the elements in the sample. This test was carried out in the Chemistry Department, Ahmadu Bello University, Zaria.

3.7.3.4 Thermal Analysis of Samples

Thermo gravimetric analysis (TGA/DTA) studies of samples were carried in a nitrogen atmosphere on a thermal analyzer (Perkin Elmer) at a heating rate of 10^{o}C/min. The instrument detects the mass loss with a resolution of 0.1 as function of temperature. The samples were evenly and loosely distributed in an open sample pan of 6.4mm diameter and 3.2mm deep with initial sample weight 0f 8-10mg. the temperature change was controlled from room temperature (25 ± 3°C) to 900°C with a heating rate of 10°C/min. High purity argon was continuously passed into the furnace at a flow rate of 60ml/min at room temperature and atmospheric pressure. Before starting each run, the argon was

used to purge the furnace for 30mins to establish an inert environment in order to prevent any unwanted oxidative decomposition. The TGA/ DTA curve were obtained from TGA runs using universal analysis 2000software from TA instruments. In this method a change in thermal stability was examined in terms of percentage weight loss as a function of temperature. At the same time DTA involves comparing the precise temperature difference between a sample and an inert reference material.

3.8 Production of kiln materials using the developed Composite

After the analyses werecarried out on the produced composite, an optimum composition was obtained at 25% grey cast iron addition. This optimum compositionof 25% grey cast iron was used to produce Kiln shelves, bars and props (see plates 3.10, 3.11 and 3.12). The produced kiln components were expected to resist more stress at high temperature than those made with only kaolin. The mixed blend was packed into a metal mould box and pressed using hydraulic press,a pressure of 10 kg/cm^2 was applied to enhance compaction, densification and surface smoothness of the samples. The mould shelves were dried in open air for 14 days. Followed by drying in the kiln for twelve (12) hours at 110^0C to expel any moisture left in the shelves and to avoid crack during firing

which was carried out in electric heat furnace. Preset at heating rate of 7^0C/min. the firing procedure used involved heating the samples at various temperatures as shown below:

a) 1100^0C for 8 hours

b) 1280^0C for 8 hours

After firing, the shelves were allowed to cool in the furnace at a cooling rate of 1^0C/min



Plate 3.10: Kiln Bars



Plate 3.11: Kiln Props



Plate 3.12: Kiln Shelves with reaction of grey cast iron that change the colour of the composite

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Introduction

The results obtained from the analyses carried out on the developed composite materials are presented in this section. Samples were referenced according to the percentage of theGrey cast iron compositions.

4.2 Physical Properties

The physical properties of the developed composite materials conducted at the National Metallurgical Development Centre, Jos, are presented as follows:

4.2.1 Bulk density

The results of bulk density (B.D) of the developed composite materials are presented in figure 4.1 and Table A1 (Appendix A).

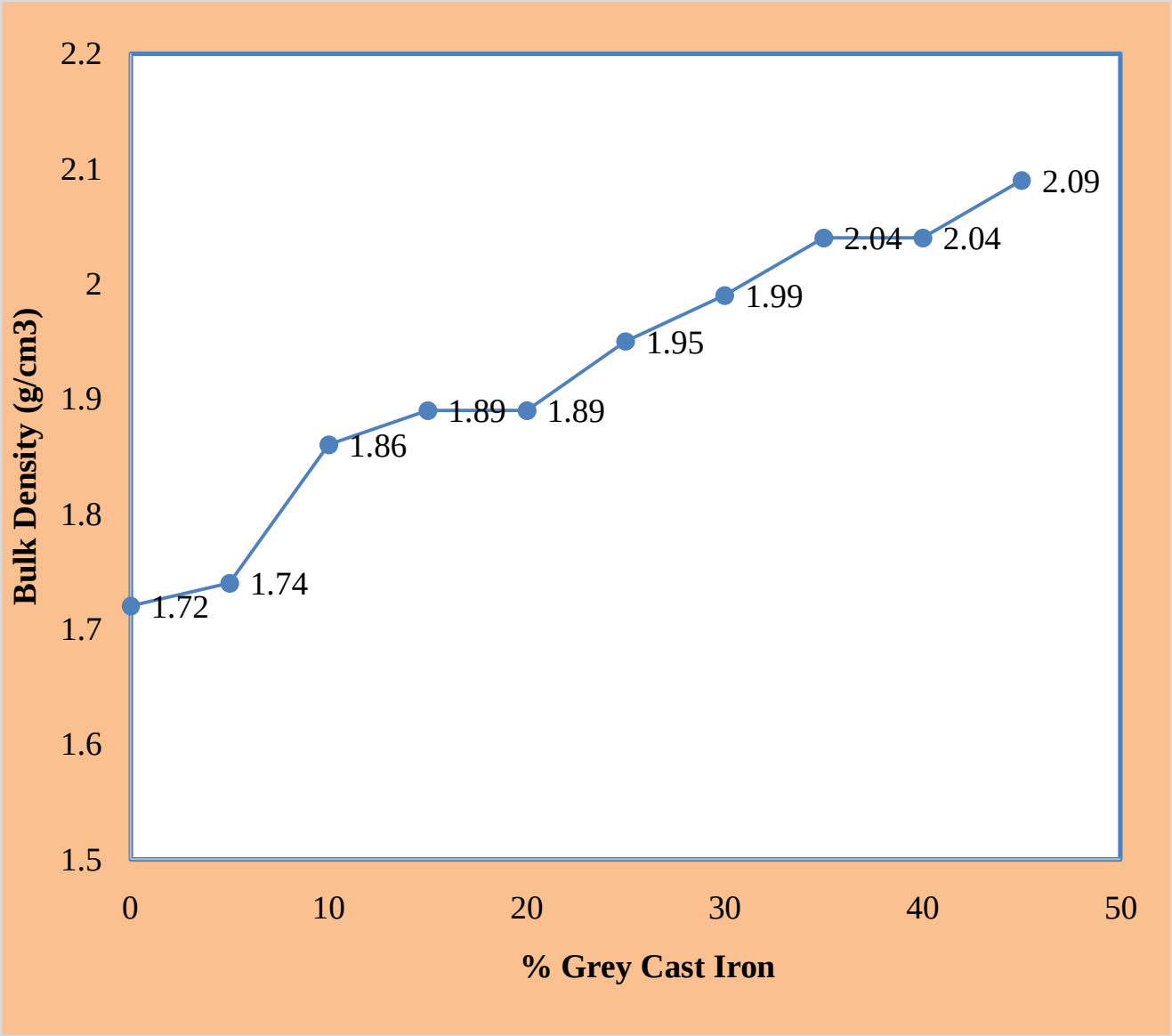


Fig. 4.1: Variationof Composite's Bulk Density with % grey cast iron

The results of the bulk density of the developed composite material as presented in the table A1 (Appendix A)and fig. 4.1 showsthat the bulk density of the developed material increases with increase in the %wt Grey cast iron addition. The bulk density increases from 1.72 to 2.10g/cm^{3}which is about 22.09% increase. This is associated to the high density of the Grey cast iron particle as compared to that of the clay. This implies that, using materials with higher

percentage of cast iron will result to kiln furniture of much weight than those produced with lower percentage. The variations in densities of the composite material agreewith earlier work reported by Ramachandra and Radhakrisha, (2006).

4.2.2 Density

The densities of the developed composite materials were calculated using the ratio of the masses of the developed composites to their respective volumes. The results obtained are presented in figure 4.2 and table A2 (Appendix A)

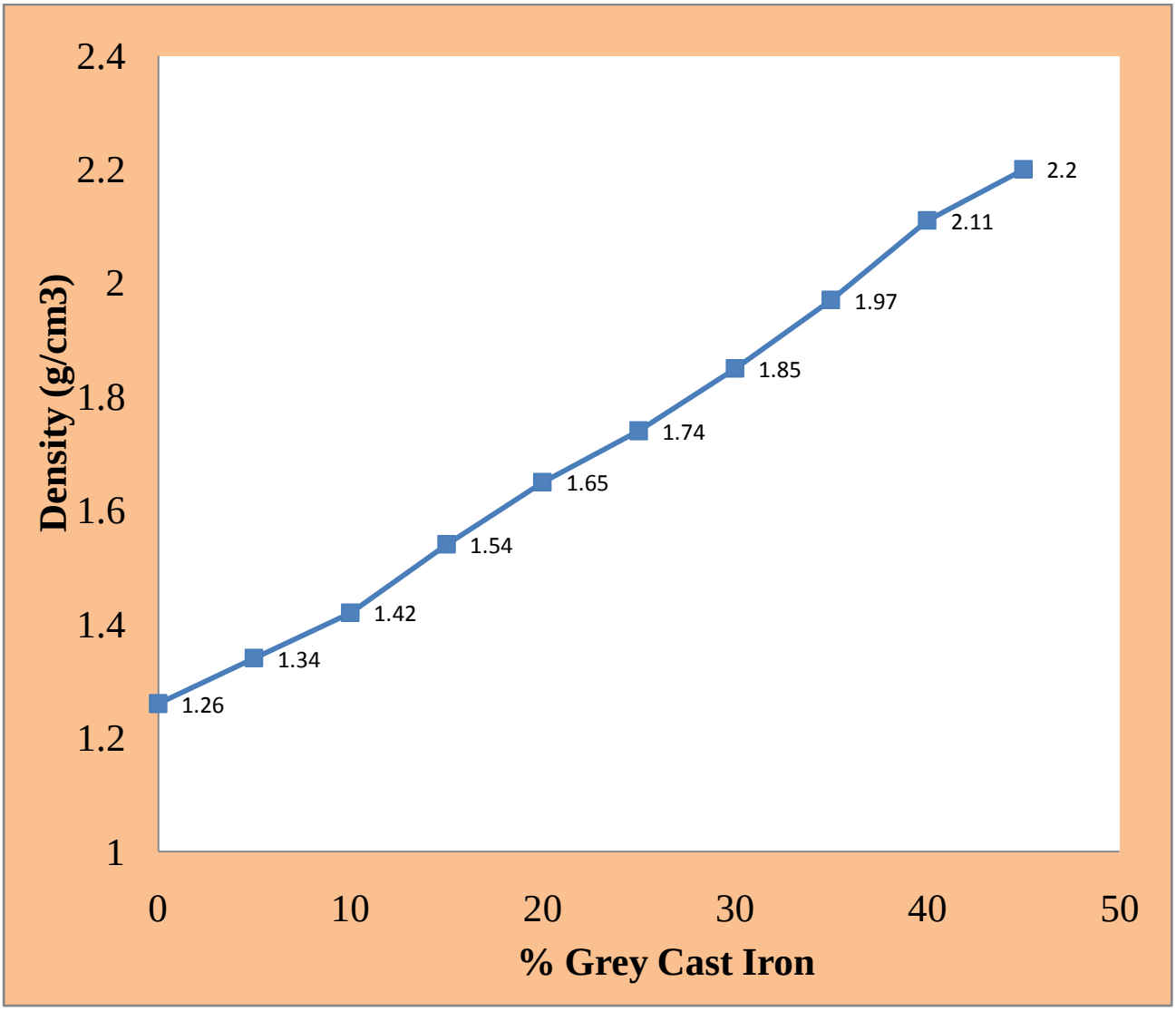


Fig. 4.2: Variation of Composite's Density with % grey cast iron

4.2.3 Apparent porosity

The results of the percentage apparent porosity of the developed composite materials are presented in figure 4.3 and tableA3 (Appendix A).

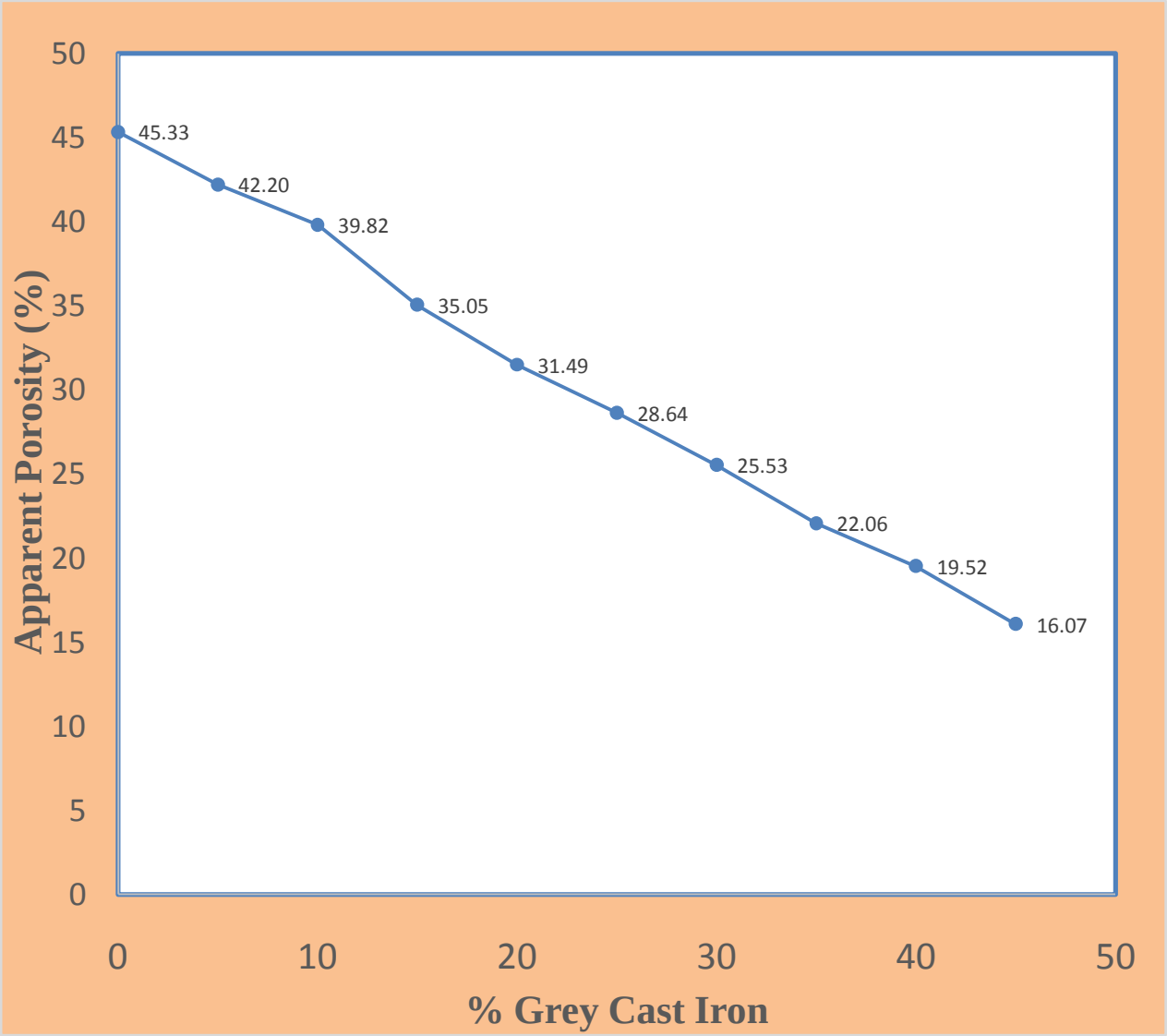


Fig. 4.3: Variation of Apparent Porosity with % grey cast iron addition

The results of the apparent porosity of the developed composite material as presented in table A3 (Appendix A) and figure 4.3, reveals that apparent porosity of the developed material decreases with increase in the %wt of Grey cast iron addition to the composite material. The apparent porosity decreases from 36.75 to 22.41% which is 39.02% decrease. This can be associated with the role played by

the melting of the cast iron powder into solids within the composite matrix; this reduces the number of pores present in the composite material

4.2.4 Percentage Waterabsorption

The rate at which the developed composite samples absorbs water when immersed in it for 24hrs was calculated using equation 3.2and the results are presented in figure 4.4 and Table A4 (Appendix A).

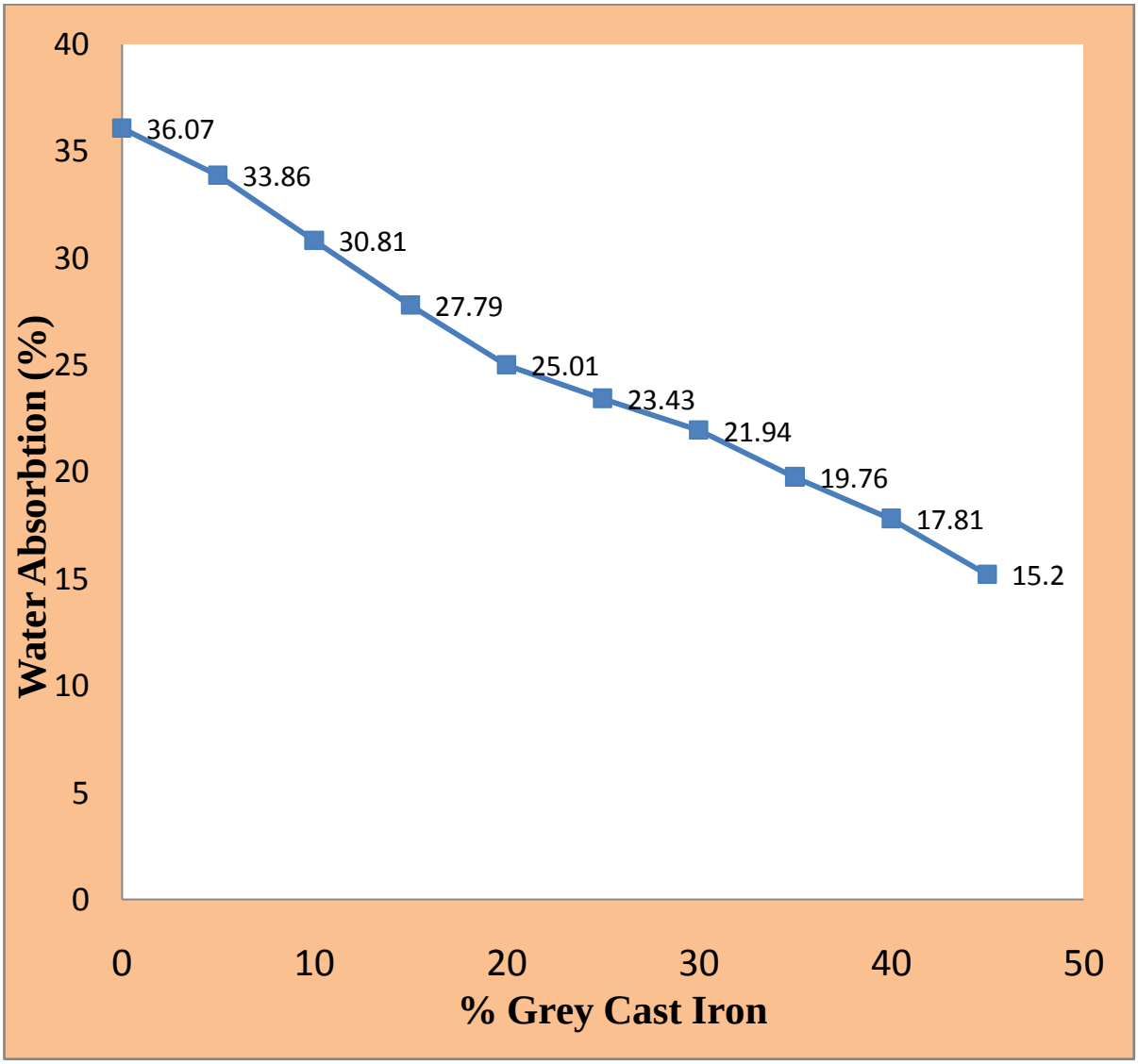


Fig. 4.4: Variation of Composite's Water Absorption with % grey cast iron

The results show that the rate at which the developed composite material absorbs water decreases with increase in percentage of grey cast iron addition. The water absorption decreases from 22.64% to 13.47 %, which is 40.50% decrease.

This implies that the introduction of the grey cast iron into the clay matrix lowers the rate at which it absorbs water. This is expected to make the shelves, props and bars produced from it to be stronger and more durable than those produced with clay only during operational usage especially in a moist area.

4.2.5 Linear shrinkages

The linear shrinkages of the developed composite materials are calculated using equation 3.3,The results for linear shrinkages are presented in figure 4.5 and Table A5 (Appendix A).

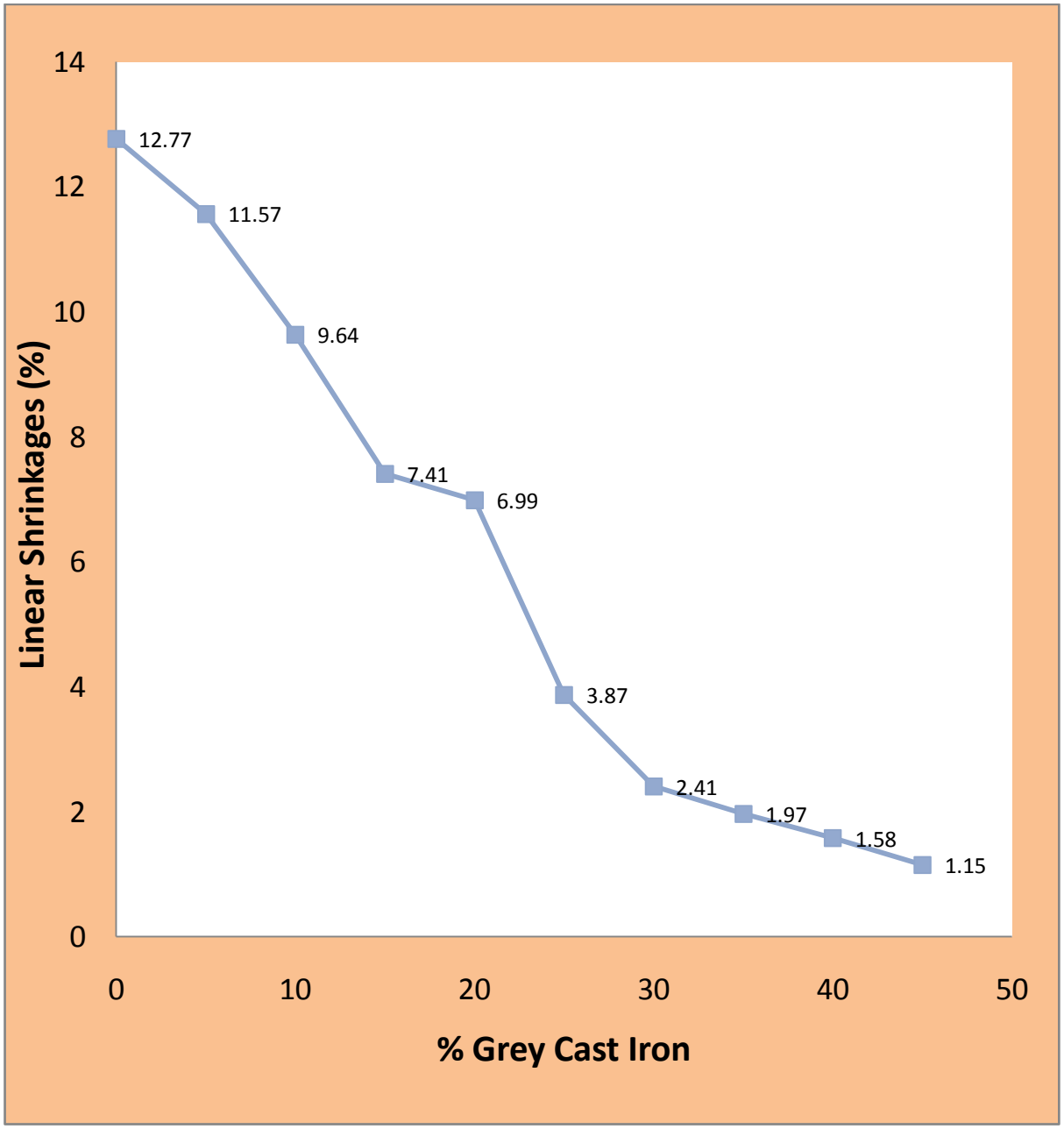


Fig. 4.5: Variation of Composite's Linear Shrinkages with % grey cast iron

The results of the change in length of the developed composite material after firing as presented in table A5 (Appendix A) and fig. 4.5 shows that the reduction in length of the developed composite material decreases with increase in the percentage of Grey cast iron addition up to 10% wt , the length then begins

to increase with further addition of Grey cast iron to the composite material. This can be explained by principle of saturation of mixture. The mixture of kankara clay and Grey cast iron reaches saturation level which lead to the outpour of excess cast iron melt thereby increasing the samples length. The amount of outpour increases with increase in the percentage of cast iron added to the composite material which accounts for increase in length of the composite materials after firing.

4.2.6 Refractoriness

The refractoriness of the developed composite materials was measured by detecting their cone numbers. The cone numbers were then traced from book of standard to detect the temperature equivalence. These results are presented in figure 4.6 and table A6 (Appendix A).

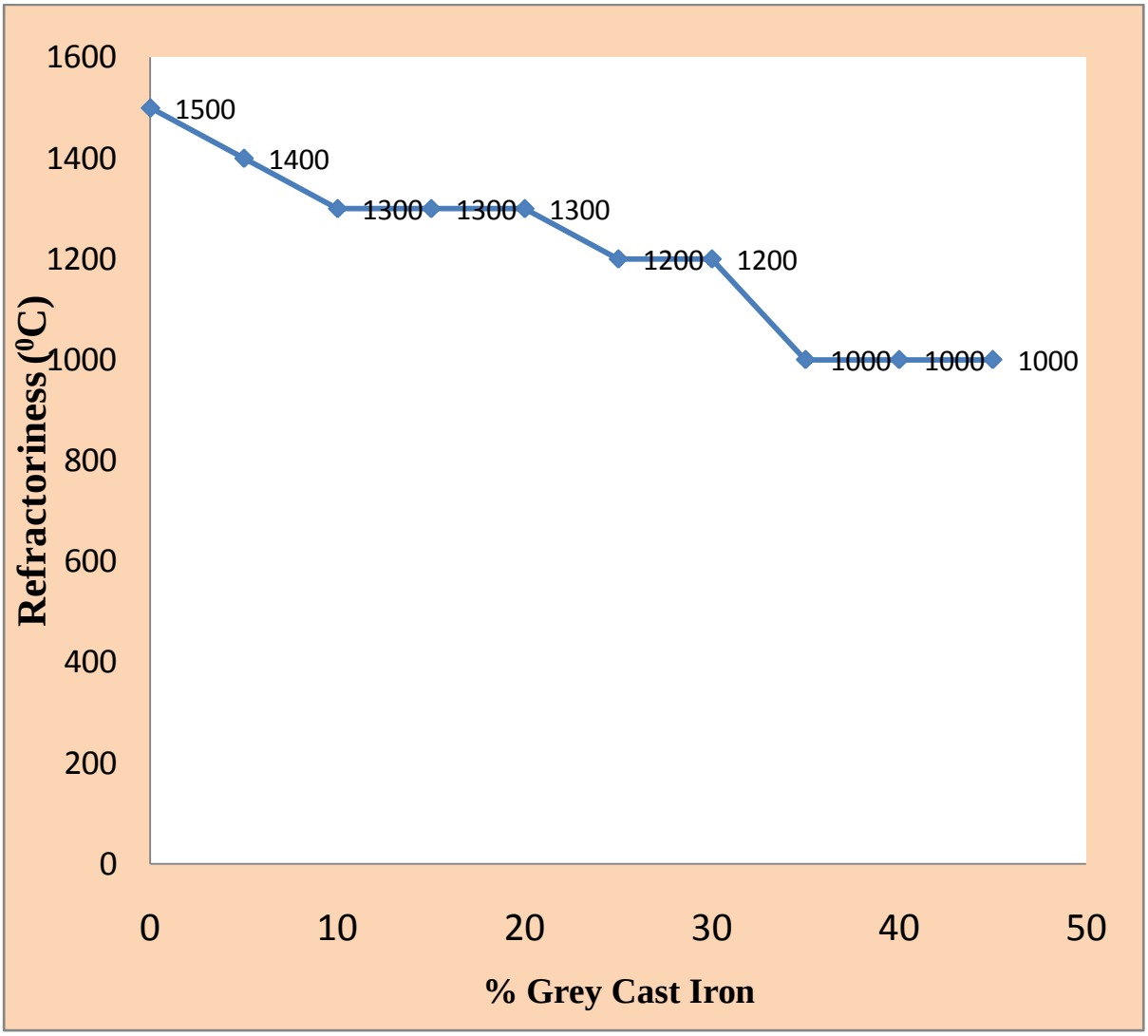


Fig. 4.6: Variation of Composite's Refractoriness with % grey cast iron

The results as presented in fig. 4.6 show that the refractoriness decreases with increase in the percentage of Grey cast iron addition. Although the melting point of grey cast iron is high but the refractoriness of pure Kankara clay is higher, the resulting mixture of the two materials produced a composites material with lower refractoriness. This can be associated to thermal instability resulting from the mixture of the two materials.

4.3 Mechanical Properties

TheResults obtained for the mechanical properties of the developed composite materials are presented in (fig. 4.7- 4.10):

4.3.1 Cold Crushing Strength (CCS)

The cold crushing strengths of the developed composite materials were calculated using equation 3.4 and the results were presented in figure 4.7 and table A7 (Appendix A).

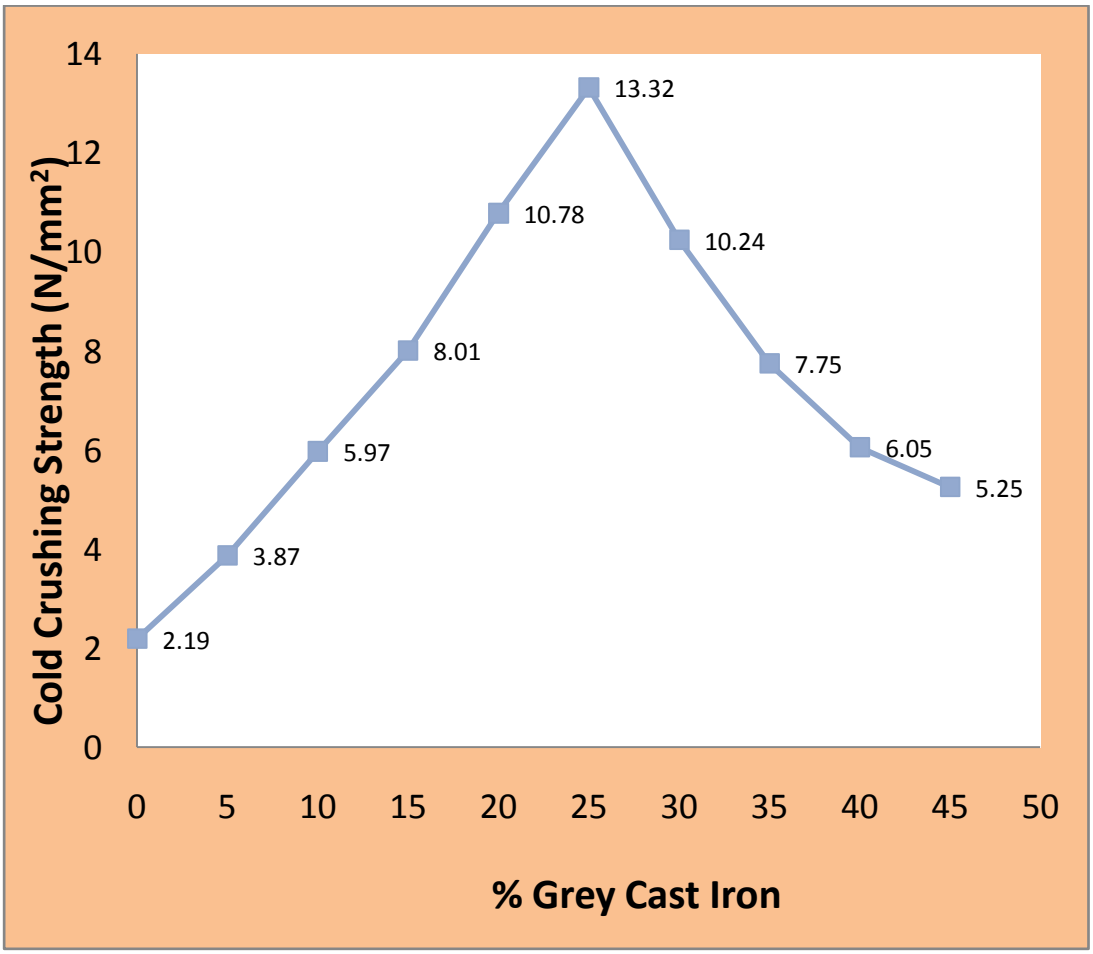


Fig. 4.7: Variation of Composite's Cold Crushing Strength with % grey cast iron

The results of cold crushing strength of the developed composite material as presented in fig. 4.7 shows that the cold crushing strength of the material increases withincrease in %wtaddition of Grey cast iron particle to an optimum value of 13.32N/mm^2 at 25% composition. The cold crushing strength falls on further addition of Grey cast iron to the developed material. This percentage composition of Grey cast iron that gave optimum strength coincides with the composition where saturation was obtained for the composites mixture. This implies that the introduction of Grey cast iron into the Kankara clay strengthens the clay until the matrix becomes saturated.

4.3.2 Hardness Value

The results of hardness values of the developed composite materials were measured and presented in figure 4.8 and table A8 (Appendix A).

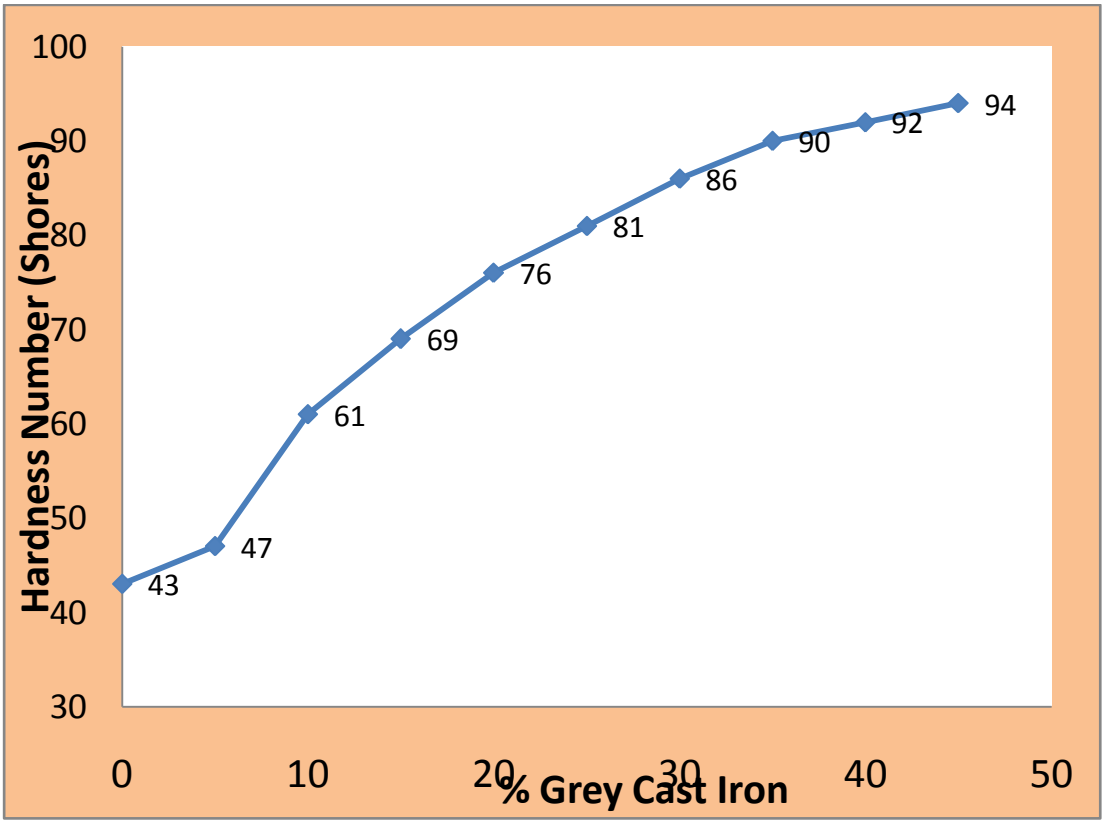


Fig. 4.8: Variation of Composite's Hardness Values with % grey cast iron

The hardness value results of the composite sample as presented in fig. 4.8show that the hardness value measure from the developed composite increases from 43 shores to 94 shores obtained from samples with 0% and 45% grey cast iron, this is about 118% increment. The results obtained confirms that the introduction of grey cast iron increases the hardness of the composite material developed which is associated with formation of bond between the iron molecules and clay particles at elevated temperature during firing which resists dislocation of adjacent atom in the structure of the developed material.

4.3.3 Tensile Strength

The ultimate tensile strengths of the developed composite materials were calculated using equation 3.6 and were presented in figure4.9 and tableA9 (Appendix A)

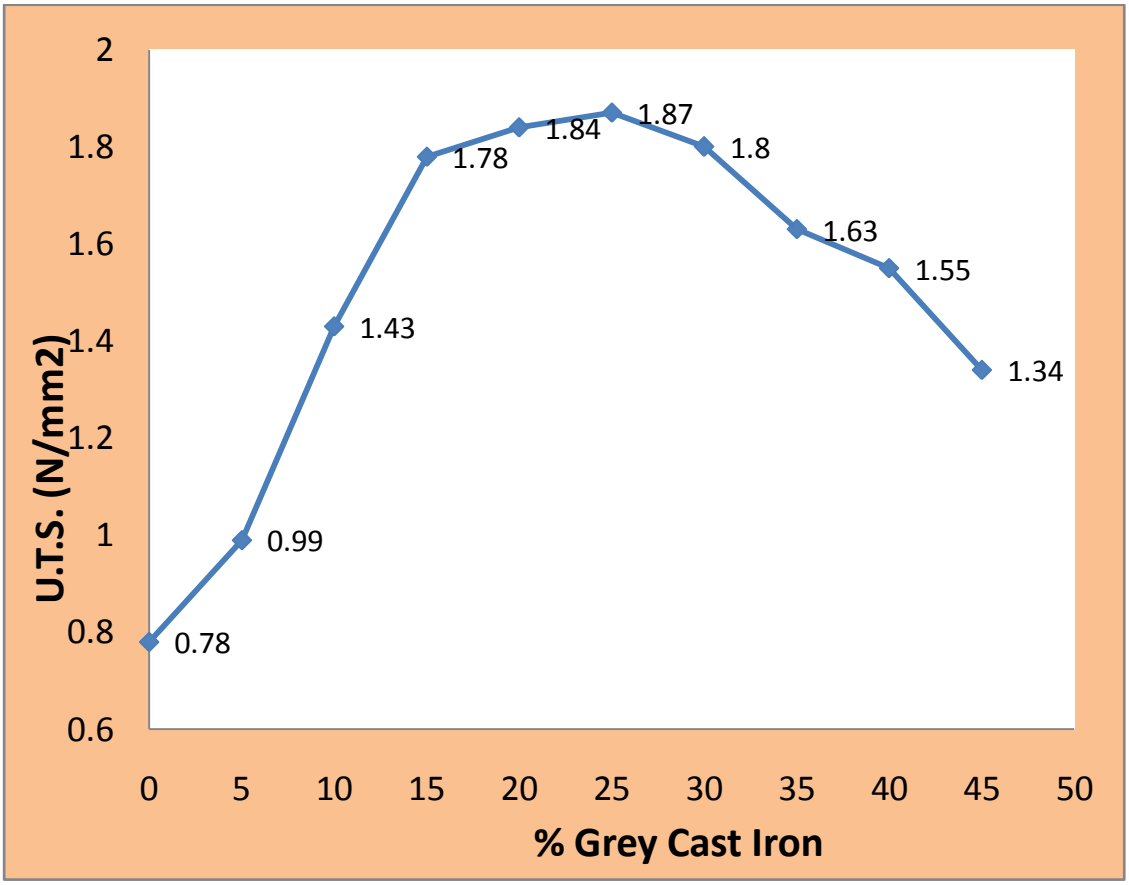


Fig. 4.9: Variation of Composite's Ultimate Tensile Strength with % grey cast iron

The results of the confirmatory study of ultimate tensile strength of the developed composite material as presented in the table A9 (Appendix A)and fig. 4.9 shows that the ultimate tensile strength of the developed composite material

increases with increase in percentage composition of grey cast iron from 0.78 N/mm^2obtained for pure kankara clay to an optimum value of 1.87 N/mm^2 at 25% composition of grey cast iron. Further increase in the %wt of Grey cast Iron beyond 25% lead to a gradual decrease in the Ultimate tensile strength of the composite.The composition with optimum tensile strength also has the optimum cold crushing strength.

4.3.4 Compressive Strength

The results of ultimate compressive strengths of the developed composite materials were calculated using equation 3.7 and were presented in figure 4.10 and table A10 (Appendix A)

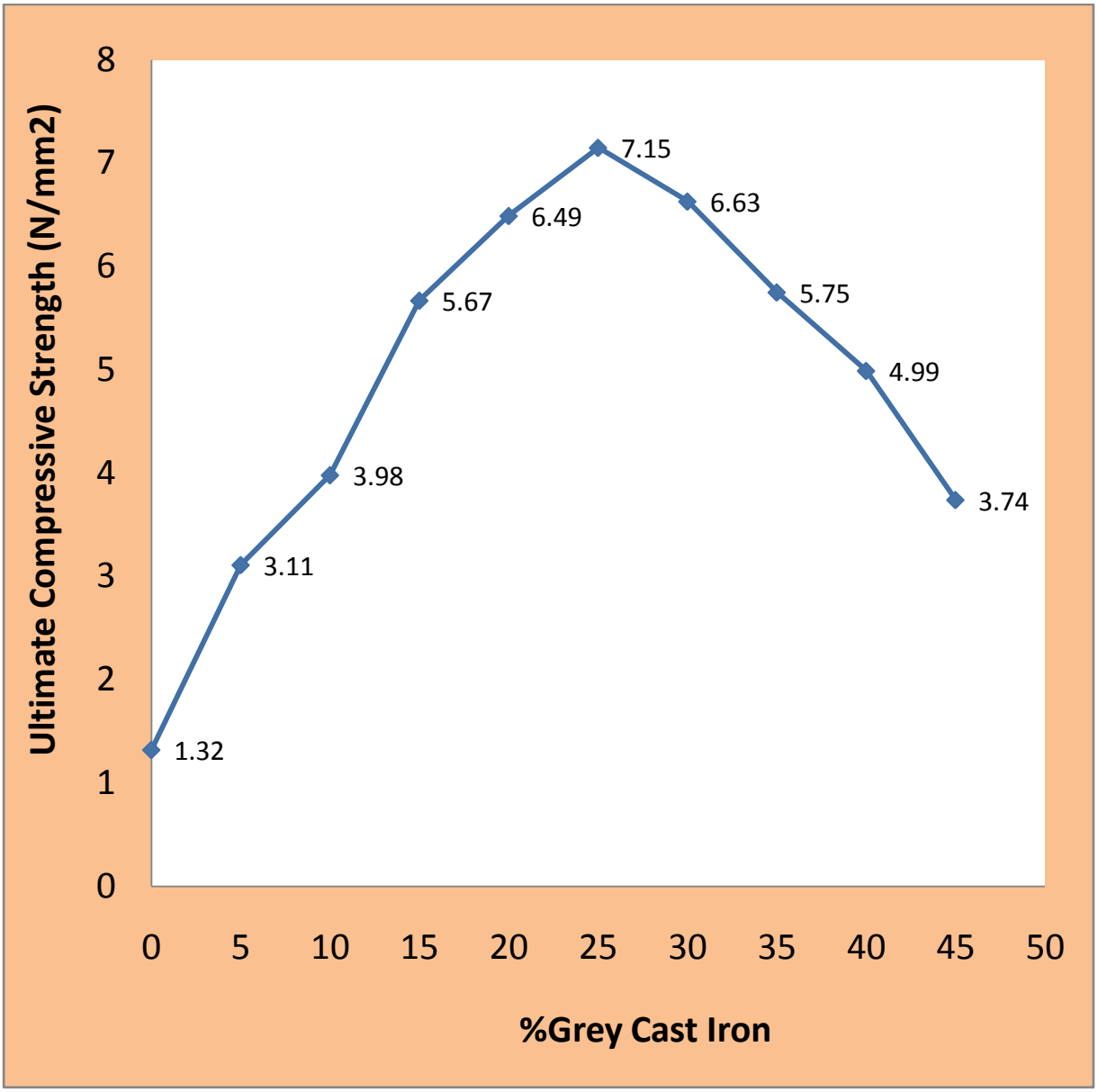


Fig. 4.10: Variation of Composite's Ultimate Compressive Strength with % grey cast Iron

The results of the confirmatory study of ultimate compressive strength of the developed composite material as presented in fig. 4.10 follows the same trend observed in cold crushing and tensile strength test results of the developed composite material. The ultimate compressive strength increases with increase in

percentage composition of grey cast iron from 1.32 N/mm^{2}obtained for pure clay to an optimum value of 7.15 N/mm^{2}which was also obtained at 25% composition of grey cast iron. This shows that 25% composition of grey cast iron,further increase in the %wt of Grey cast Iron beyond 25% lead to a gradual decrease in the Ultimate compressive strength of the composite.

4.4 Microstructural analysis

The Results obtained for the Microstructural analysisof the developed composite materials are presented below:

4.4.1 Scanning Electron Microscope Analysis

The micro structural features of as-received particle of Kankara clay and the develop composites from Kankara clay and Grey cast iron particles were investigated using a scanning electron microscope (model SEM-Philips XL- 30 SFEG). Elemental analysis of as-received and the ceramic composite samples were conducted by using SEM-Energy Disperse Spectrometry (SEM-EDS). The morphological characterization of the grains and pores were evaluated on polished surfaces as shown in plates(4.1 –4.10).The micrographs obtained from the scanning electron microscopic analysis carried out on the composite samples are presented in plates(4.1 –4.10).

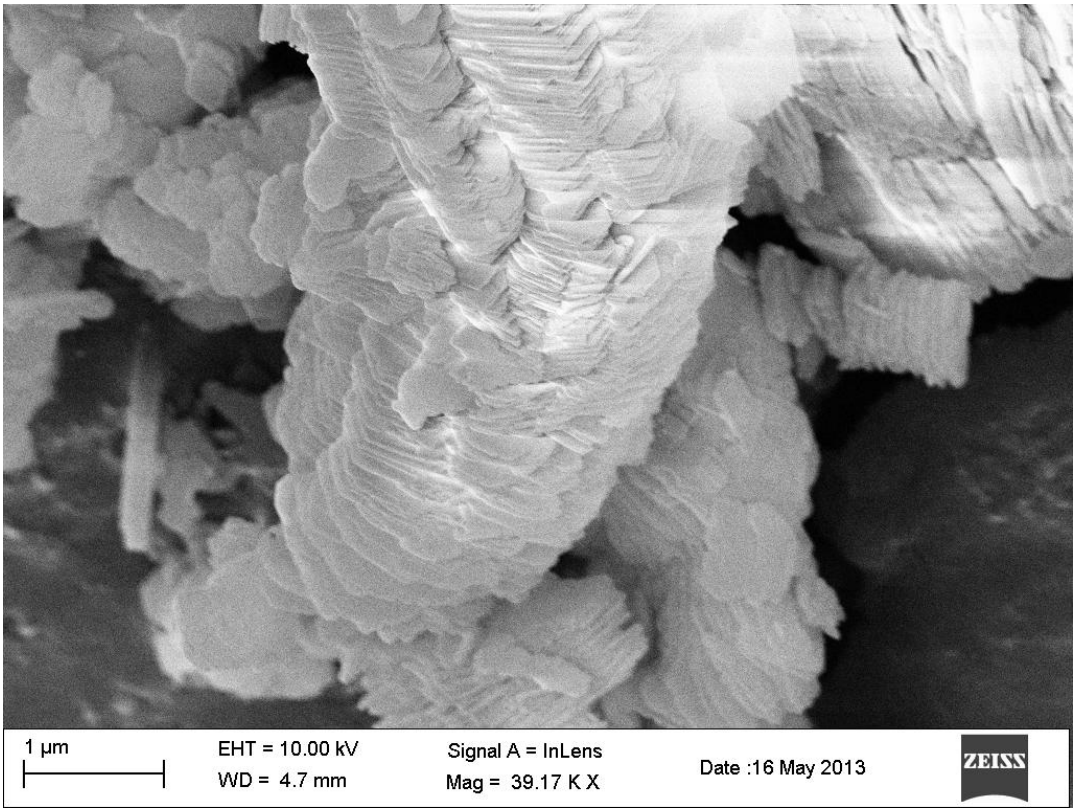


Plate 4:1 Micrograph of Sample with0% Grey Cast Iron

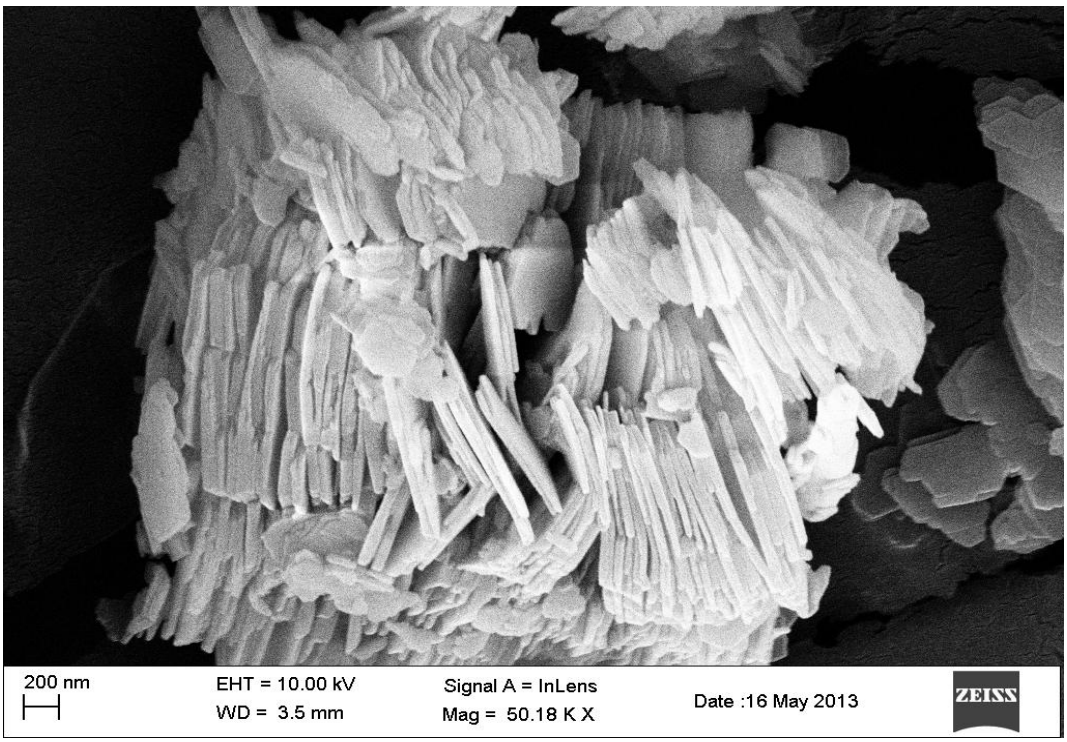


Plate 4:2 Micrograph of Sample with 5% Grey Cast Iron

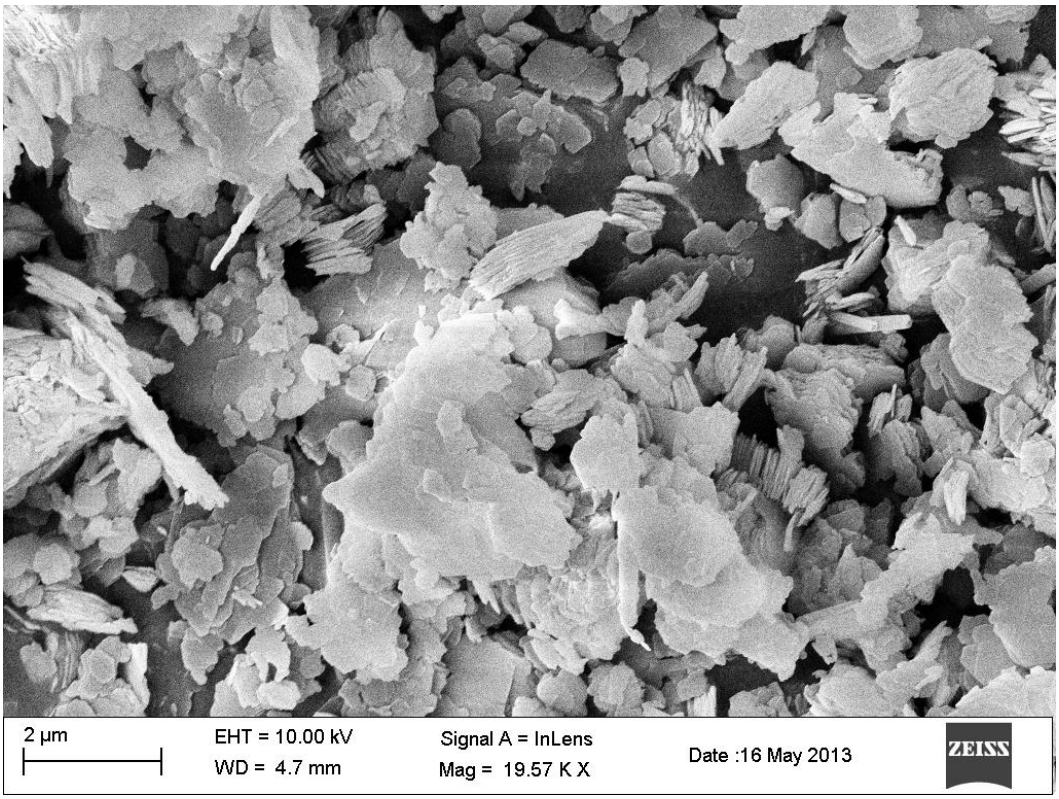


Plate 4:3 Micrograph of Sample with 10% Grey Cast Iron

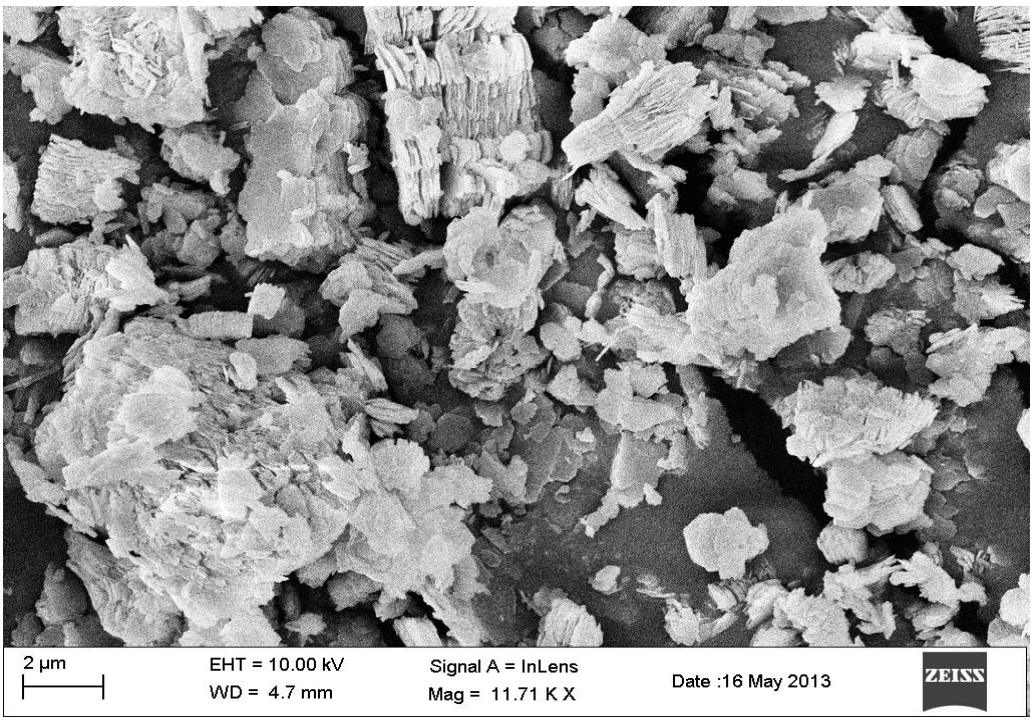


Plate 4:4 Micrograph of Sample with 15% Grey Cast Iron

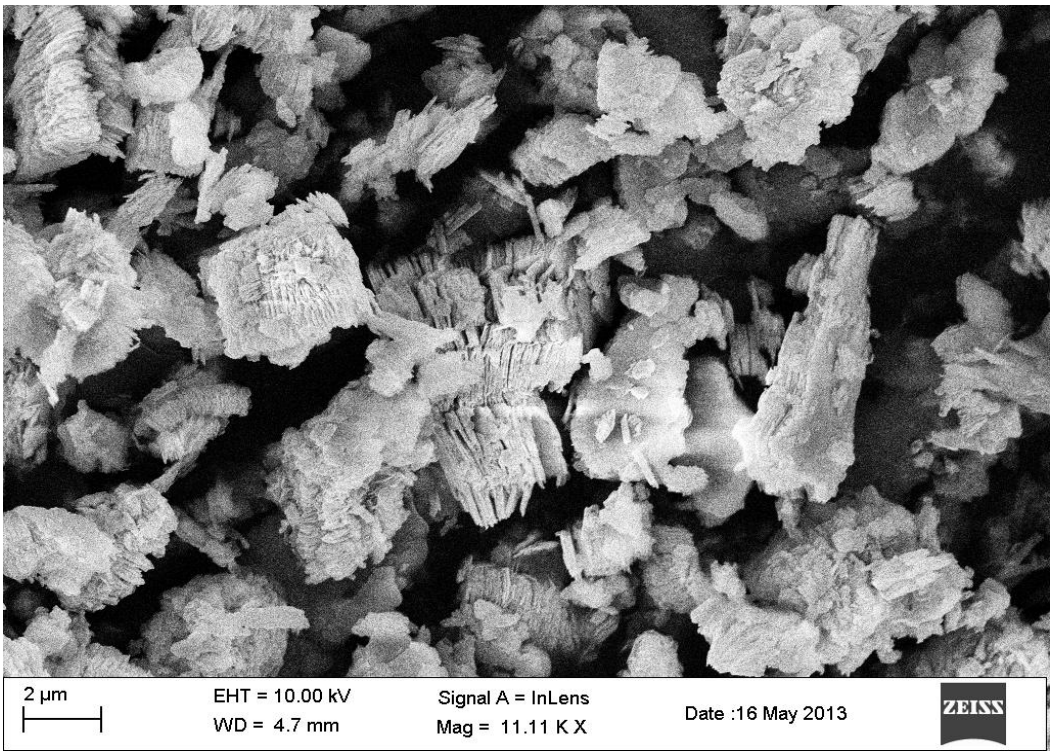


Plate 4:5 Micrograph of Sample with 20% Grey Cast Iron

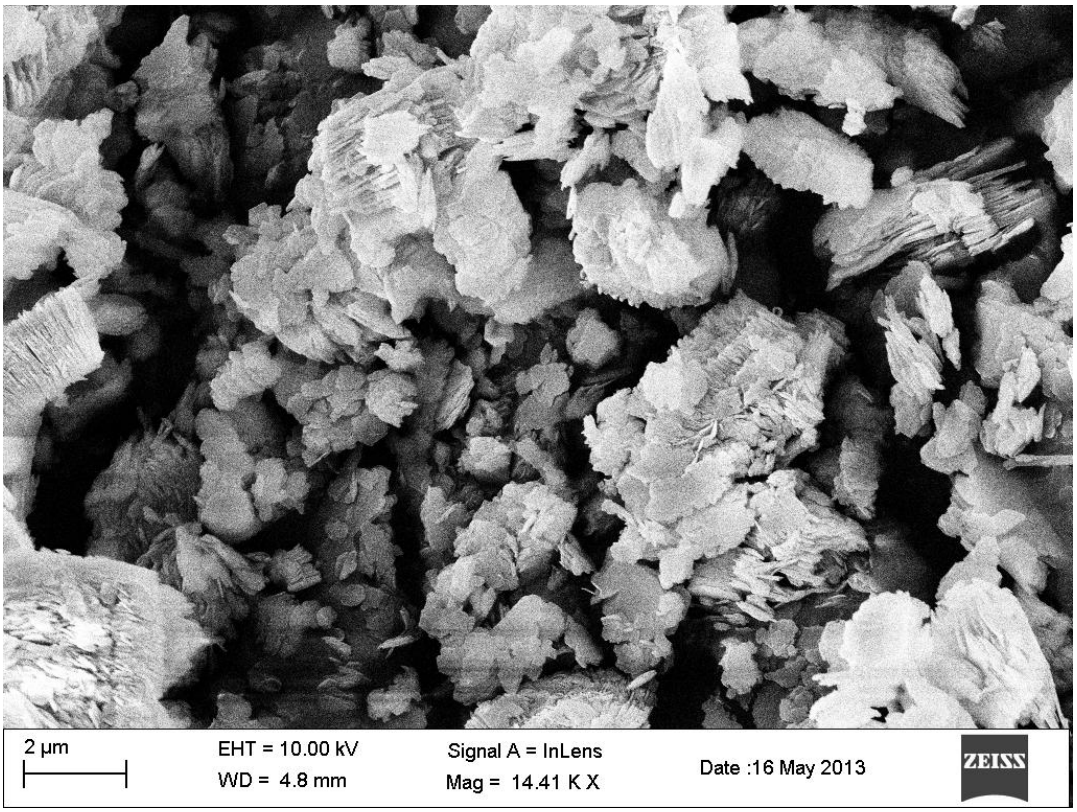


Plate 4:6 Micrograph of Sample with 25% Grey Cast Iron

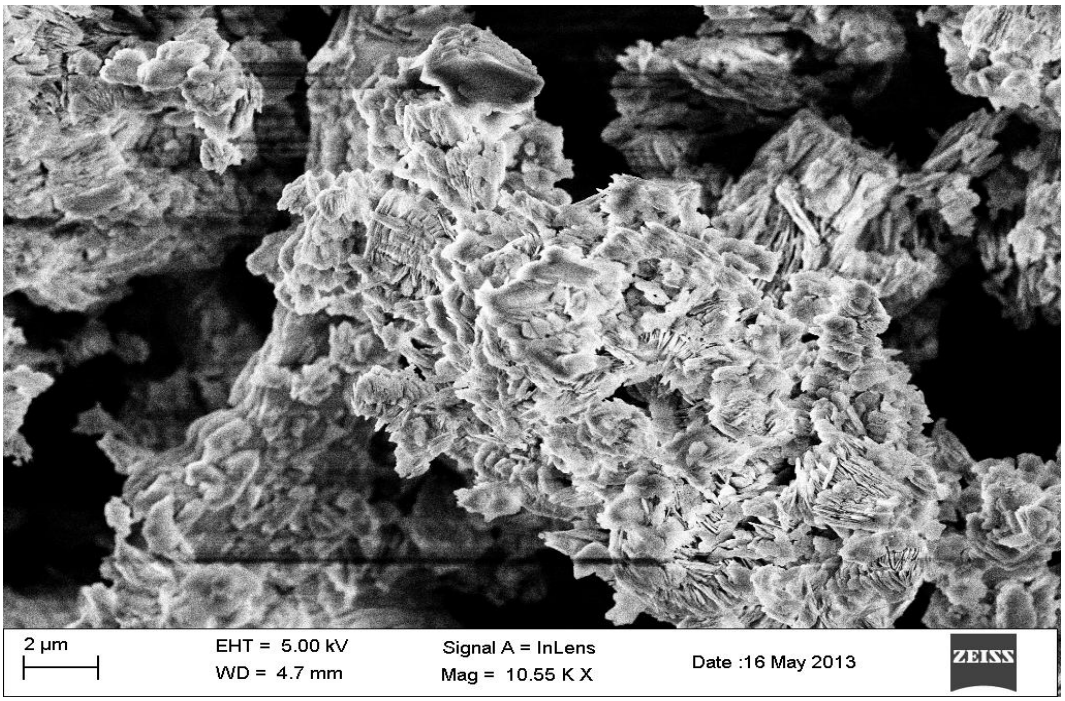


Plate 4:7 Micrograph of Sample with 30% Grey Cast Iron

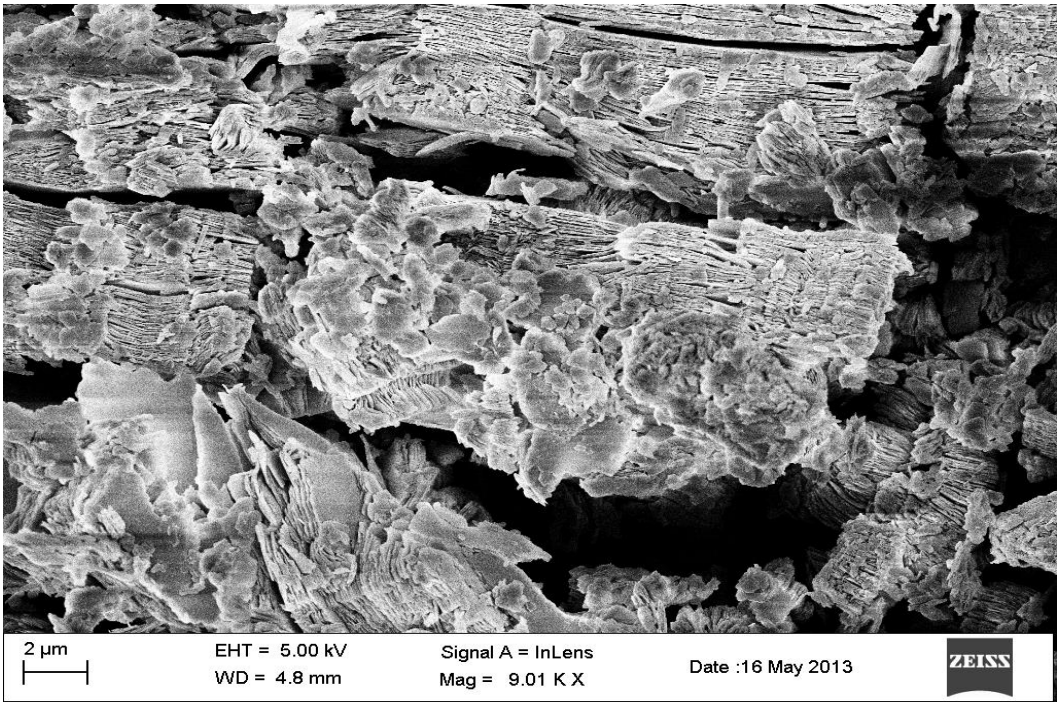


Plate 4:8 Micrograph of Sample with 35% Grey Cast Iron

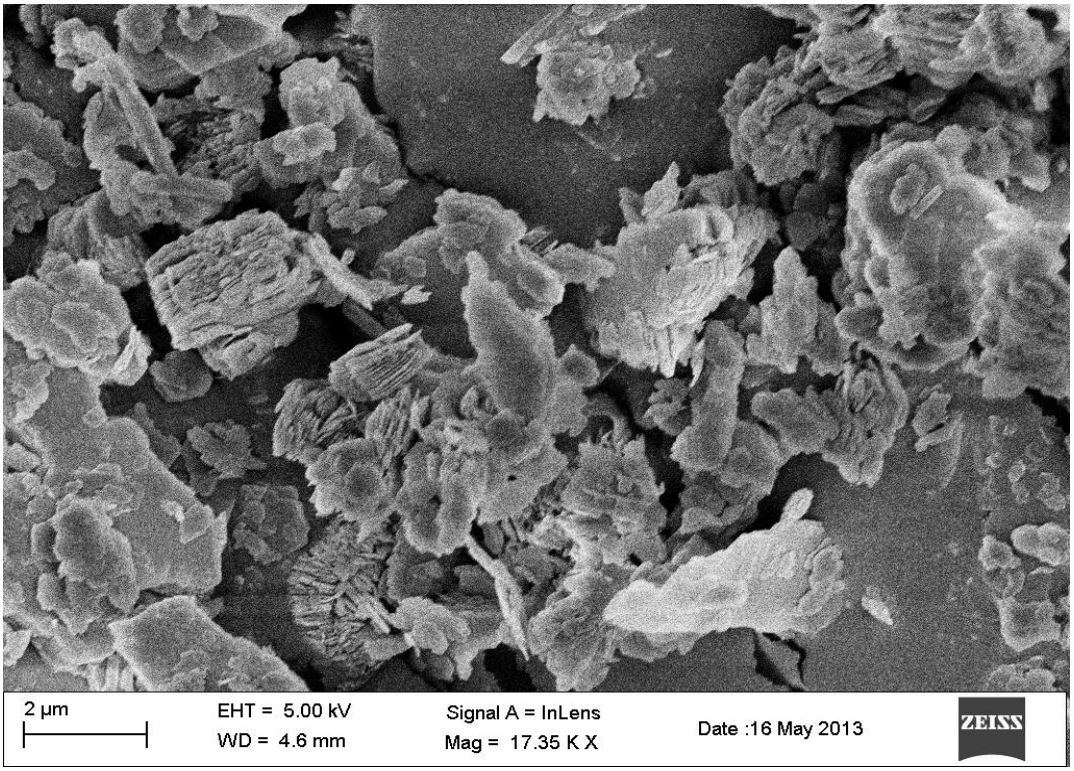


Plate 4:9 Micrograph of Sample with 40% Grey Cast Iron

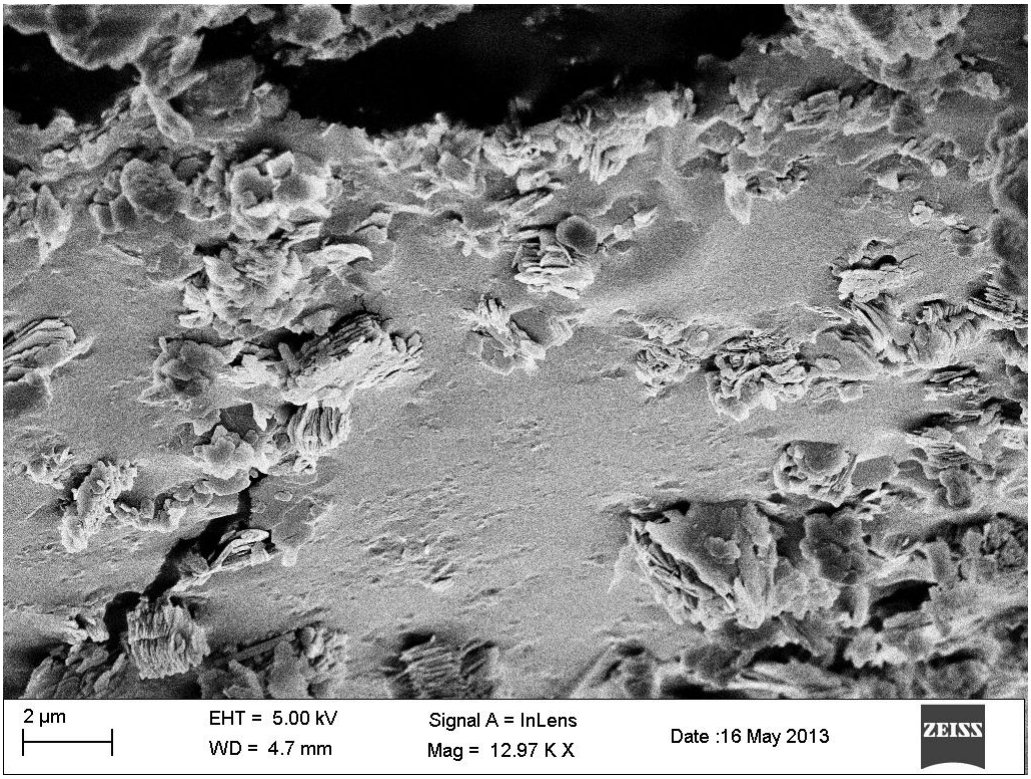


Plate 4:10 Micrograph of Sample with 45% Grey Cast Iron

The microstructure of the unreinforced Kankara clay is shown in Micrograph 4.1. The structure reveals the rich kaolin clay with an appreciable quantity of crystalline quartz flakes, which is mainly composed of alumina (41.87%) and Silica (55.937%). The composite material morphology reveals the dark amorphous iron oxide (Fe2O3) within the clay crystals which reaches saturation at 25% Grey cast iron addition. The morphology also reveals homogeneity at 25% Grey cast iron.

4.4.2 X-ray Diffraction Patterns of Composites

The X-ray diffraction patterns of the kankara clay and compositesrecorded from the X-ray diffractometry analysis carried out on the samples are presented in figure 4.11-4.16

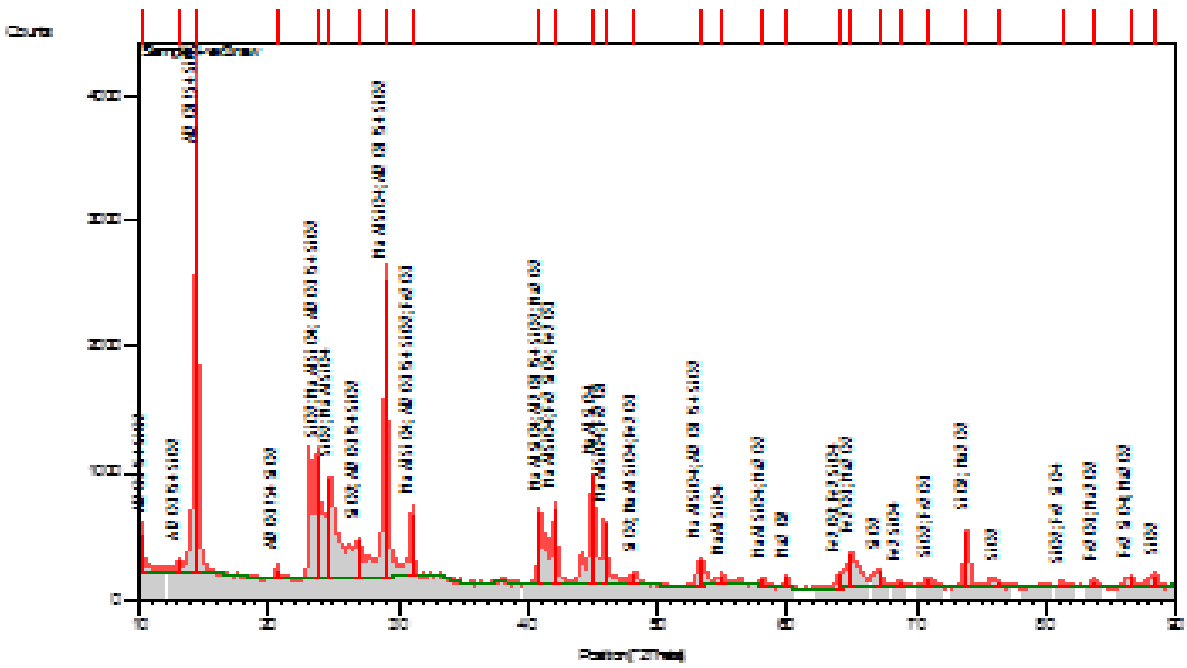


Figure 4.11: XRD pattern for Kankara clay Sample with0% Grey Cast Iron

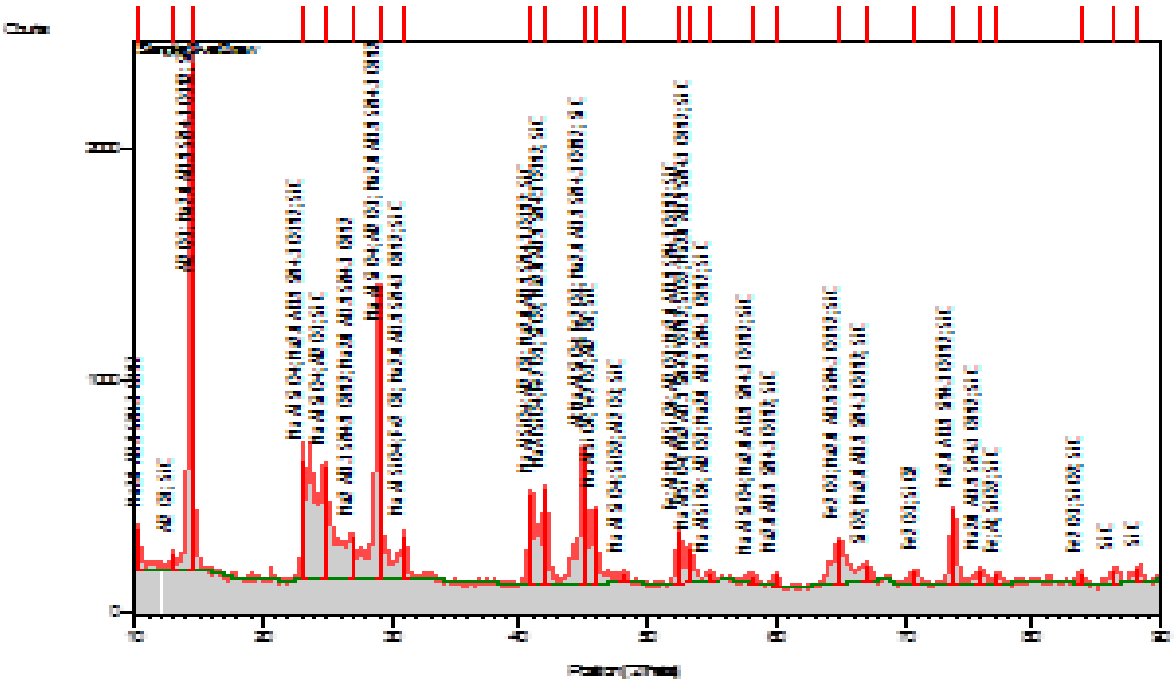


Figure 4:12 XRD pattern for Sample with 10% Grey Cast Iron

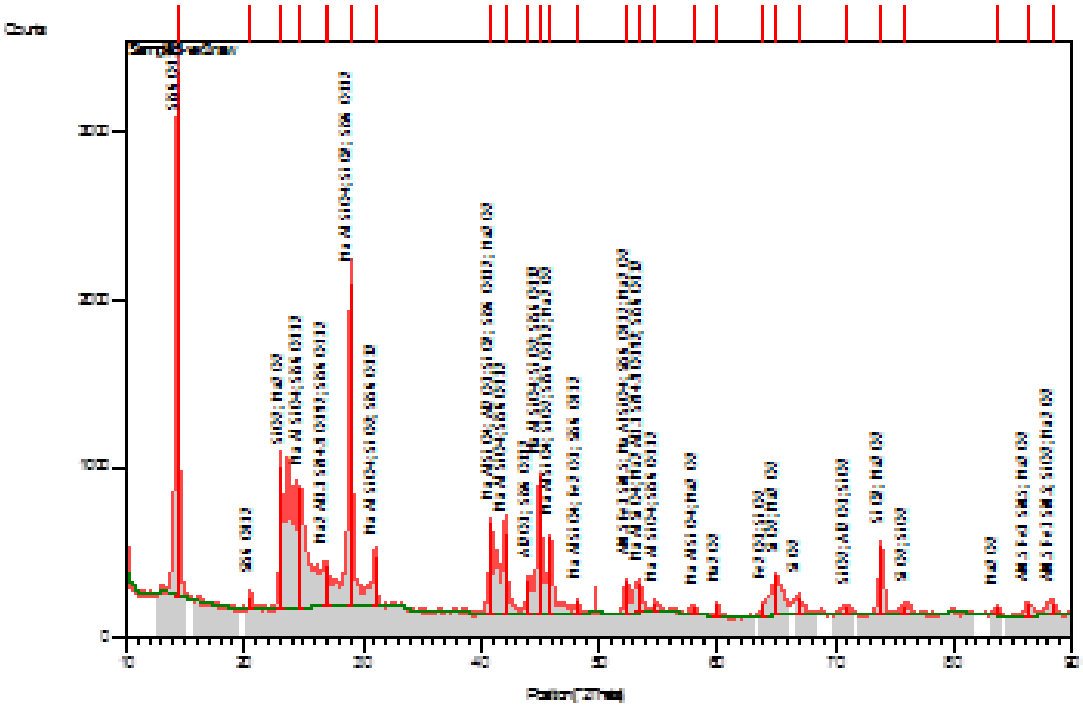


Figure 4:13 XRD pattern for Sample with 25% Grey Cast Iron

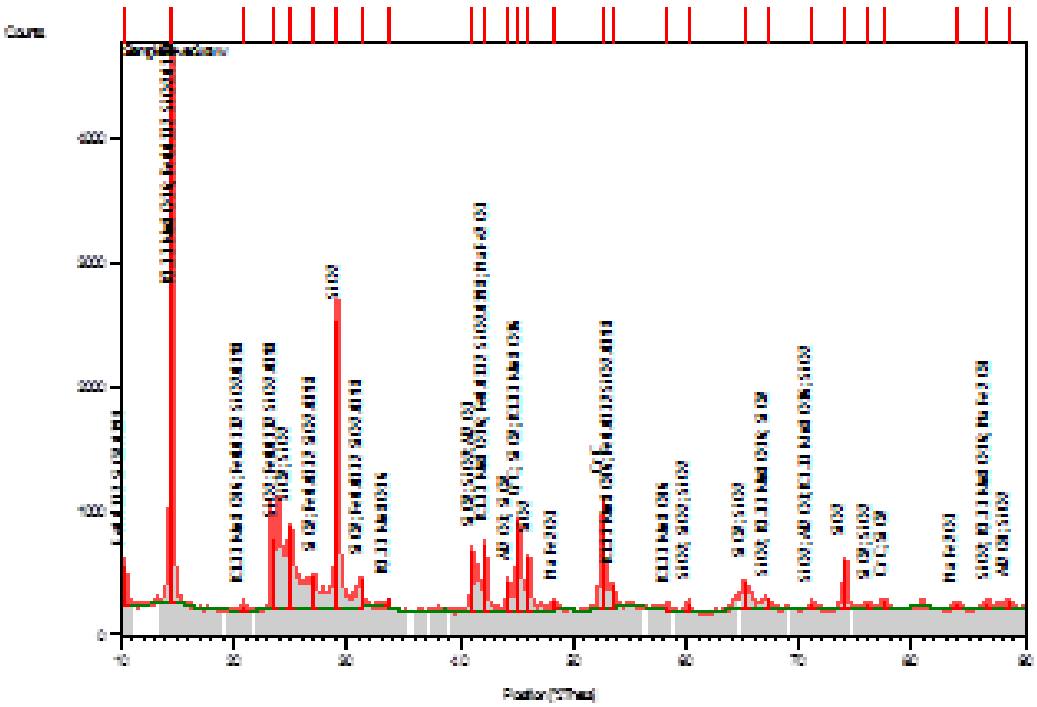


Figure 4:14 XRD pattern for Sample with 35% Grey Cast Iron

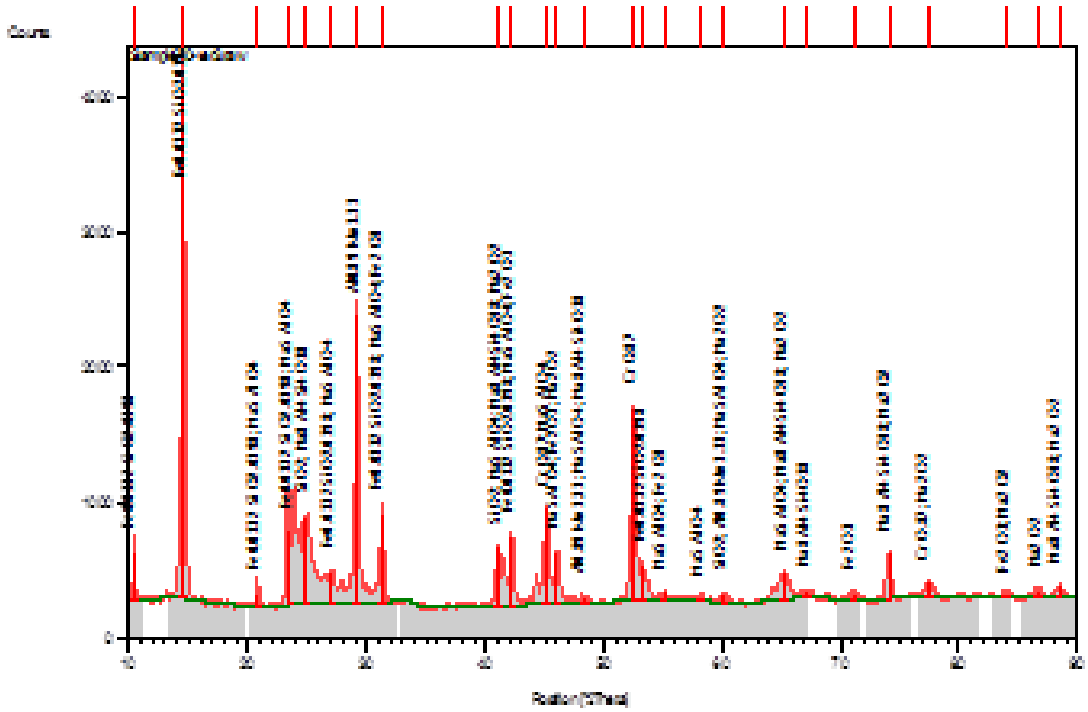


Figure 4:15 XRD pattern for Sample with 40% Grey Cast Iron

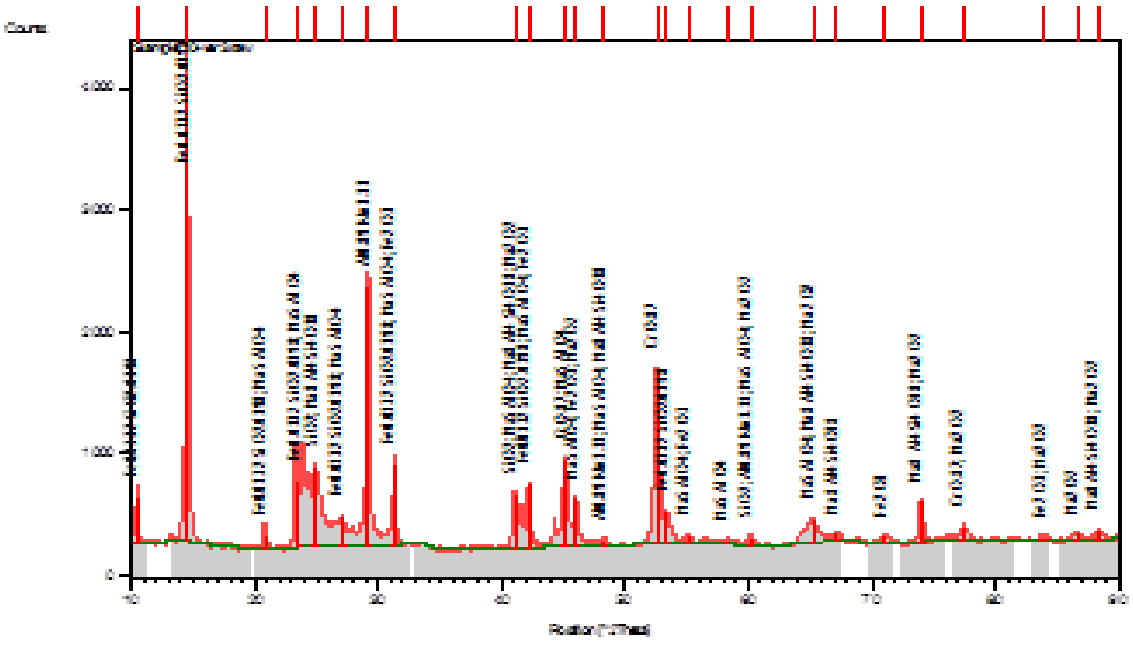


Figure **4:16 XRD pattern for Sample with 45% Grey Cast Iron**

Figure 4.12-4.16 showed the XRD patterns of the composites samples. The X-ray diffraction patterns of the oriented Kankara clay, scanned from 0 to 90°(2Ɵ) showed several peaks due to the different minerals present (see fig.4.11). the major diffraction peaks were 10.43, 23.58, 29.02, 31.01 and 41.06° and their interplanar distance are: 9.98, 3.67, 3.36, 3.24 and 2.11Å, and their relative intensity of X-ray scattering were: 1.44, 9.35, 102.01, 0.55 and 4.38, phases at this peaks were: Magnesium aluminium silicate (MgOAl2O3SiO2), Sillimanite (Al2 (SiO4) O, quartz low (SiO2), Mayenite, Syn ((CaO)12(Al2O3)7), Silicon Oxide (SiO2).

The X-ray diffraction patterns of the oriented composite kiln material scanned from 0 to 90°(2Ɵ) showed several peaks due to the different minerals (Fig. 4.12- 4.16). From fig. 4.12 to 4.16 it was observed that the phases in the composite kiln material are quite different from that of the clay. The major phase difference are SiC, Fe3C,Fe_2O_3 and Iron Silicate which resulted from the interfacial reaction of silica from the clay and iron, carbon in the Grey cast iron.

The results of XRD confirmed the formation SiO_2, Al_2O_3, Na Al Si O_4 and Fe2 Si O_4 of Kankara clay (see Fig. 4.13). But that of the composites with grey cast iron particles showed smaller interspacing distance and evidence of SiC, CrC, Fe_2O_3, Fe, Al0.5Fe_3Si0.$_5$ and K1.$_{33}$$Mn_8O_{16}$ (see fig. 4.14-4.16). The presence of all these elements in the clay help to strengthened and toughened the composites. It can be clearly observed that the diffraction peaks appear in the pattern corresponding to

phase with good crystalline nature. The grain size of the composites are found to be about 105-55nm.

4.4.3 Thermal Analysis of Samples

The degradation curves and thermal degradation temperature of compositesobtained from the DTA/TGA carried out on the samples are presented in (figure 4.11-4.15) and Table A16 (Appendix A) respectively.

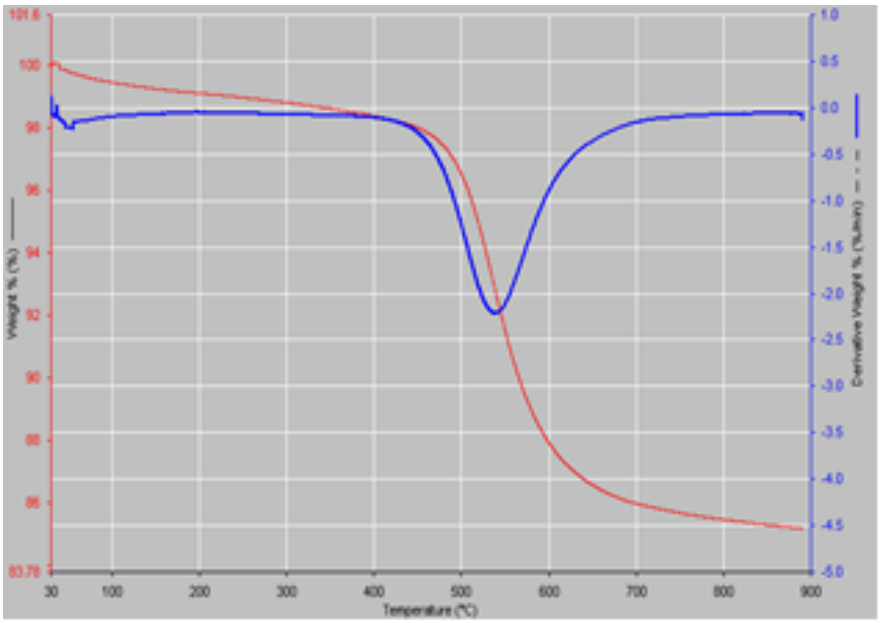


Plate 4:11 DTA/TGA curve for Sample with0% Grey Cast Iron

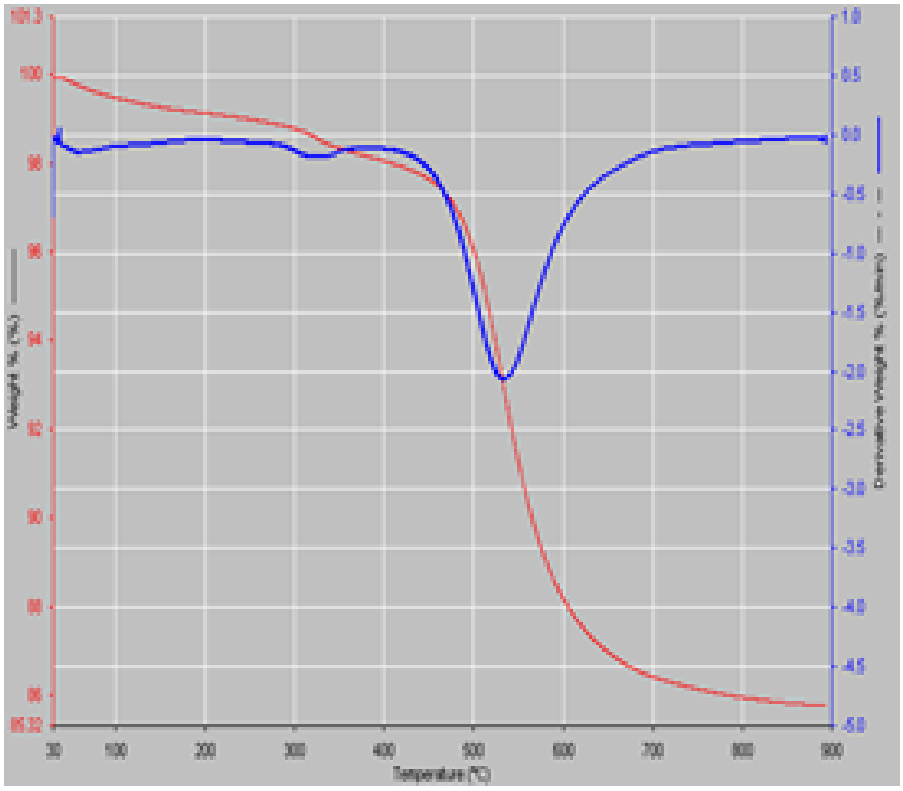


Plate 4:12 DTA/TGA curve for Sample with 10% Grey Cast Iron

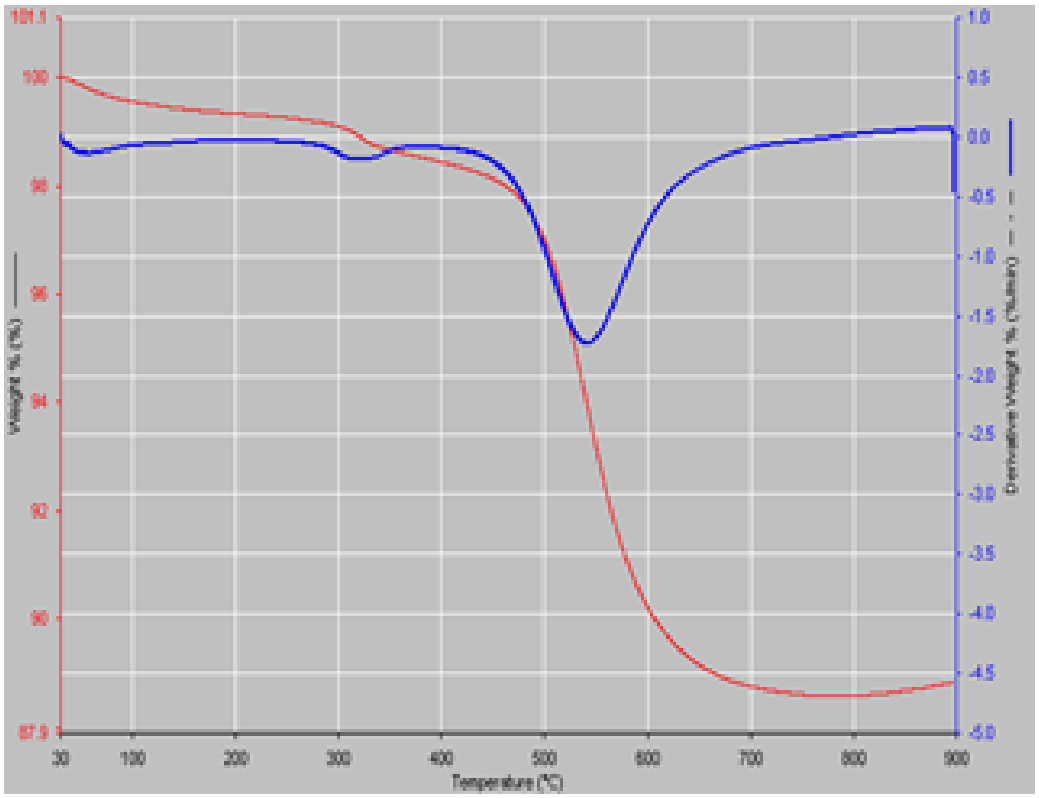


Plate 4:13 DTA/TGA curve for Sample with 25% Grey Cast Iron

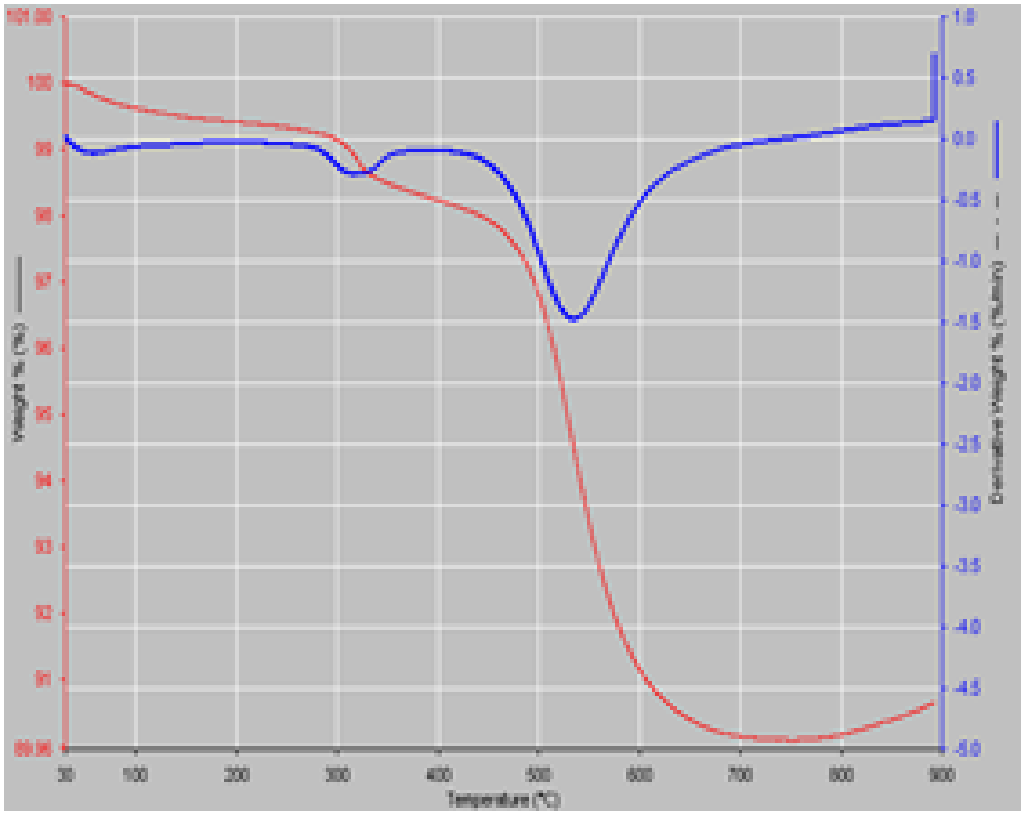


Plate 4:14 DTA/TGA curve for Sample with 35% Grey Cast Iron

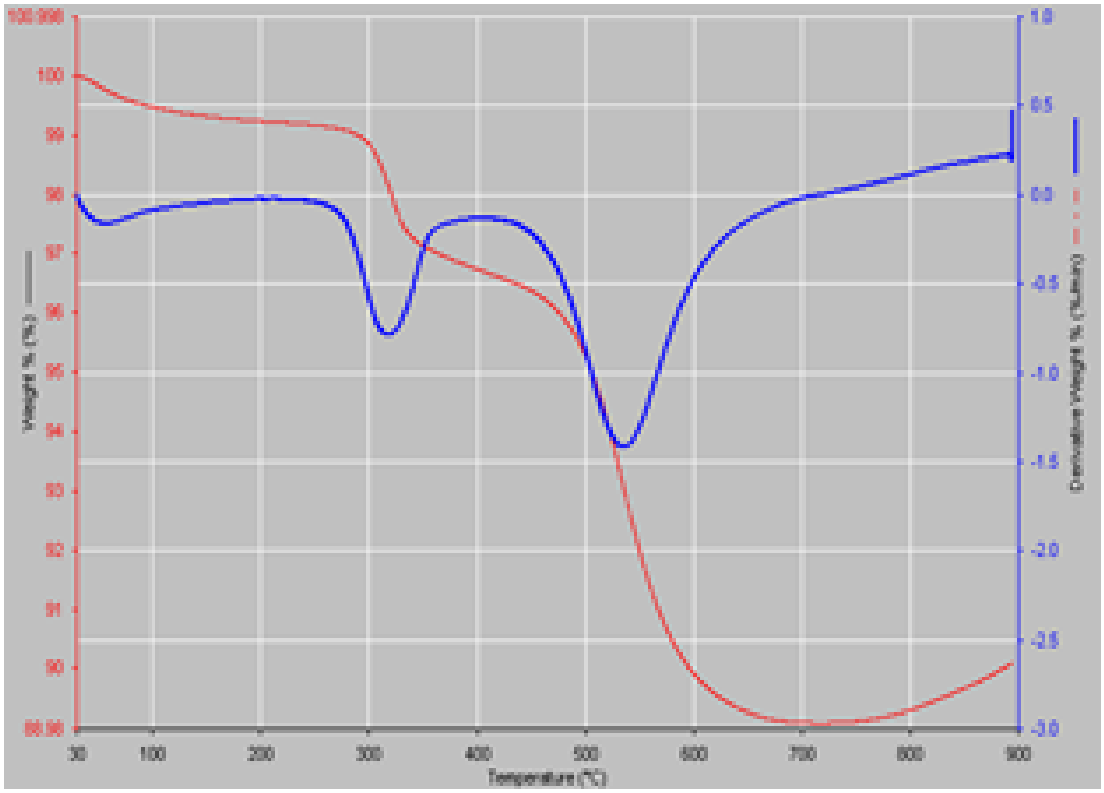


Plate 4:15 DTA/TGA curve for Sample with 45% Grey Cast Iron

The DTG/TGA analysis (plates 4.17-4.21) revealed that initial weight loss (~ 10%) observed between 500 and 600^{0}C is attributed to the vaporization of the water from the samples, while degradation of the composites started at higher temperature, precisely after 550^{0}C. Above this temperature, the thermal stability of samples gradually decreased and degradation of the samples occurred.

DTA curve shows that the temperature of maximal decomposition/ destruction was 520-600 ^{0}C. The presence of endothermic effects in samples is results of two processes – dehydrogenation and evaporation of some non-cellulosic materials. This conclusion was confirmed by the decreased mass of the sample. DTA curve also confirmed these results.

The endothermic effects observed in the temperature range indicated above were probably as a result of the bonds formed in the clay backbone. Finally, is worthy of note that the thermal analysis curves reveal that samples are stable until around 700^{0}C. This is in agreement with values of some other clay material reported in literature.

All the composites showed a small 10 % weight loss around 500^{0}C, which can be attributed to the evaporation of water. The weight loss rate gradually increased above 600^{0}C and distinct weight loss appearing within 500 to 600^{0}C. The results showed that addition of grey iron particles to kankara does not alter the thermal decomposition process since at temperatures greater than 900^{0}C the samples retained 80%weight. See table 4.17,

$T_{10\%}{}^{0}C$ = temperature at which 10% of the original weight is lost

$T_{90\%}{}^{0}C$ = temperature at which 90% of the original weight is lost

$T_{max}{}^{0}C$ = temperature at maximum decomposition

4.4.4 X-Ray Fluorescence Test *(XRF)*

The spectrum obtained from the X-ray florescence test carried out on reinforced and unreinforced clay samples are analyzed and the results obtained are presented in the tables below.

Table 4.1: Analytical concentration table for Sample with 0% Grey Cast Iron

Element	**Concentration** wt%
Na_2O	0.000
MgO	0.394
Al_2O_3	41.872
SiO_2	55.937
P_2O_5	0.250
SO_3	0.292
Cl	0.003
K_2O	0.342
CaO	0.126
TiO_2	0.120
Cr_2O_3	0.000
Mn_2O_3	0.008
Fe_2O_3	0.655
ZnO	0.002
SrO	0.000

Table 4.2: Analytical concentration table for Sample with 5% Grey Cast Iron

Element	Concentration wt%
Na_2O	0.051
MgO	0.443
Al_2O_3	41.671
SiO_2	55.017
P_2O_5	0.280
SO_3	0.274
Cl	0.000
K_2O	0.329
CaO	0.092
TiO_2	0.102
Cr_2O_3	0.002
Mn_2O_3	0.028
Fe_2O_3	1.709
ZnO	0.002
SrO	0.000

Table 4.3: Analytical concentration table for Sample with 10% Grey Cast Iron

Element	Concentration wt%
Na_2O	0.000
MgO	0.481
Al_2O_3	41.360
SiO_2	55.104
P_2O_5	0.238
SO_3	0.286
Cl	0.000
K_2O	0.339
CaO	0.095
TiO_2	0.109
Cr_2O_3	0.003
Mn_2O_3	0.026
Fe_2O_3	1.954
ZnO	0.002
SrO	0.002

Table 4.4: Analytical concentration table for Sample with 15% Grey Cast Iron

Element	Concentration wt%
Na_2O	0.049
MgO	0.406
Al_2O_3	40.560
SiO_2	51.604
P_2O_5	0.236
SO_3	0.242
Cl	0.007
K_2O	0.288
CaO	0.083
TiO_2	0.095
Cr_2O_3	0.008
Mn_2O_3	0.077
Fe_2O_3	6.342
ZnO	0.002
SrO	0.001

Table 4.5: Analytical concentration table for Sample with 20% Grey Cast Iron

Element	Concentration wt%
Na_2O	0.000
MgO	0.433
Al_2O_3	39.950
SiO_2	52.065
P_2O_5	0.235
SO_3	0.279
Cl	0.002
K_2O	0.305
CaO	0.072
TiO_2	0.097
Cr_2O_3	0.009
Mn_2O_3	0.077
Fe_2O_3	6.473
ZnO	0.001
SrO	0.001

Table 4.6: Analytical concentration table for Sample with 25% Grey Cast Iron

Element	Concentration wt%
Na_2O	0.049
MgO	0.401
Al_2O_3	39. 509
SiO_2	50.522
P_2O_5	0.267
SO_3	0.299
Cl	0.000
K_2O	0.287
CaO	0.080
TiO_2	0.107
Cr_2O_3	0.010
Mn_2O_3	0.097
Fe_2O_3	8.371
ZnO	0.000
SrO	0.000

Table 4.7: Analytical concentration table for Sample with 30% Grey Cast Iron

Element	Concentration wt%
Na_2O	0.000
MgO	0.432
Al_2O_3	38.265
SiO_2	48.128
P_2O_5	0.193
SO_3	0.279
Cl	0.000
K_2O	0.252
CaO	0.072
TiO_2	0.089
Cr_2O_3	0.015
Mn_2O_3	0.139
Fe_2O_3	12.133
ZnO	0.000
SrO	0.002

Table 4.8: Analytical concentration table for Sample with 35% Grey Cast Iron

Element	Concentration wt%
Na_2O	0.000
MgO	0.416
Al_2O_3	37.881
SiO_2	47.621
P_2O_5	0.185
SO_3	0.295
Cl	0.002
K_2O	0.253
CaO	0.078
TiO_2	0.097
Cr_2O_3	0.019
Mn_2O_3	0.163
Fe_2O_3	12.989
ZnO	0.000
SrO	0.000

Table 4.9: Analytical concentration table for Sample with 40% Grey Cast Iron

Element	Concentration wt%
Na_2O	0.000
MgO	0.396
Al_2O_3	36.727
SiO_2	45.431
P_2O_5	0.195
SO_3	0.290
Cl	0.000
K_2O	0.213
CaO	0.061
TiO_2	0.095
Cr_2O_3	0.021
Mn_2O_3	0.191
Fe_2O_3	16.377
ZnO	0.000
SrO	0.003

Table 4.10: Analytical concentration table for Sample with 45% Grey Cast Iron

Element	Concentration wt%
Na_2O	0.000
MgO	0.413
Al_2O_3	36.647
SiO_2	45.057
P_2O_5	0.211
SO_3	0.262
Cl	0.003
K_2O	0.201
CaO	0.075
TiO_2	0.082
Cr_2O_3	0.026
Mn_2O_3	0.199
Fe_2O_3	16.818
ZnO	0.000
SrO	0.004

The XRF chemical composition of the Kaolin (Kankara clay) and composite kiln samples are presented in (Table 4.17-4.26). The XRF analysis confirmed that SiO2 and Al_2O_3 were found tobe the major constituents of the clay and composite kiln material. Silicon dioxide (SiO2) and Alumina (Al_2O_3) were known to be among the hardest substances.

The analysis of the spectrum obtained from the test revealed that kankara clay has up to 41.872%, 55.937% and 0.655% of Al_2O_3, SiO_2 and Fe_2O_3 respectively (see Table 1) some other oxides traces presents are MgO, K2O, Na2O, Ca2O, TiO2 etc. The percentage of the Fe_2O_3 increases with increase in percentage of grey cast iron added. This means that introduction of the grey cast

led to the formation of a new phase constituted majorly of Fe3C and Fe_2O_3but XRF cannot detect carbon. The presence of these oxides accounts for the refractoriness of the kankara clay as well as the composite kiln material.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From the above analyses and discussion, the following conclusion were drawn

1. Characterization study of the Kankara clay and grey cast iron particle was successfully conducted.
2. The production of ceramic composite refractory using powder metallurgy technique, blending by varying the percentage of grey cast iron powder from 5-45wt% in the Kankaraclay is attainable, with good refractory properties.
3. The mechanical properties test results of hardness value, cold crushing strength, ultimate tensile strength and ultimate compressive strength on the developed composite refractories showed an increase in the mechanical properties with increasing grey cast iron content to maximum weight of 25%. After this wt%, it was observed that there was a decrease in mechanical properties.
4. The physical properties test results of bulk density, porosity, linear shrinkage and water absorption on developed composite refractory showed an increase in bulk density with increasing grey cast iron content. However porosity,

linear shrinkage and water absorption decrease with increasing grey cast iron content.

5. Micro structural analysis(i.e. scanning electron microscopy (SEM) /energy dispersive spectrometer (EDS), X-ray diffraction (X-RD) and X-ray fluorescence(X-RF) have shown an even spread of the elements of the composite.
6. The utilization of Kankara clay and Grey cast Iron for the production of composite refractory kiln forniture was successfully carried out using 25wt% grey cast iron which was the best composition of the composite refractory.
7. The developed composite was observed to be plastic up until 25 wt % grey cast iron content.

5.2 Recommendation

The following recommendations can be given following the findings of this research.

1. Ceramists, both students and industrialist should have the confidence to use locally produced kiln furniture as developed from kaolin and grey iron composite substitution for the imported ones.

2. Necessary equipment for material beneficiation and testing should be made available for research within the university and in the country.

Adequate support should be given by professional bodies to aid research and development inthe country.

REFERENCES

Abed, A.,Smith, A.C. and Hendry, A. (1995) *Interfacial behaviour in ceramic-metal composites.* Silicates Industries, 60 (11-12). pp. 313-319. ISSN 0037-5225 publishers: United State of America.

Aigbodion, V.S and Asuke,F. (2007); Effect of Kankara clay on some Refractory Properties of IgbokodaSilica Sand "Inter-Research journal on Engr. Sci and Tech(IREJEST) vol.4.No.2.

Aigbodion, V.S, J. O. Agunsoye, V. Kalu, F. Asuke, S. Ola(2010): Microstructure and Mechanical Properties of Ceramic Composites, *Journal of Minerals & Materials Characterization & Engineering*, Vol. 9, No.6, pp.527-538.

Beerkens R. G. C(2002): *Contribution in Mathematical Simulation in Glass Technology*, Ed. D. Krause, H.Loch, Schott Series on Glass and Ceramics, Science, Technologyand Applications, Springer Verlag, pp. 17-73.

Beerkens R. G. C. (2004): Ceramic Transactions*141*, 3, 78-90

Beerkens R. G. C(2006): *Analysis of Energy Consumption and Energy Saving Measures for Glass Furnaces*, 80th DeutscheGlastechnischeTagung der DGG (Deutsche GlastechnischeGesellschaft), Dresden Germany, 12.-14. June 12-14,

Brookes, K.J.A., (1989): Update on cermets, ceramics and superhards, Mettal working Production 8; 63-74.

Callister D. William, JR. and David G. Reithwisch, (2012): Fundamental of material science and Engineering; An intergrated Approach. 4th Ed, John Willey and Sons.

Callister D. William, JR. and David G. Reithwisch, (2010): Material science and Engineering: An introduction. 8th Ed. Willey & Sons. London.

Chaklader .A.C.Dand Linger .K.R. (1976): Ceramic/Metal Composites using Metal-coated Alumina Powder, **Composites**Volume 7, Issue 4, Pp. 239-243

Chawla .K.K(1993), Ceramic Matrix Composites, Chapman & Hill, New York, 1993.

Chesti, A.R (1986). Refractories Manufacture, Properties and Application. Prentice Hall of private limited, London Pp 1-140, P55-63.

Clyne T. W. (2000): Comprehensive composite material, vol 3 metal matrix composites (ser, Eds)

Crowe .C.R., Grey, R.A. et. al.(1985):, Microstructure Controlled Fracture Toughness, in ICCM-V, The Metallurgical Society, Inc., p. 843.

Dell K. Allen, (1997): Metallurgy theory and practice. American technical society. Chicago.

DobrzaĔski, L.A., (2002): Material science principles and metallography, WNT, Warsaw, (in Polish).

Eerkens, J. W., H. Neff and M. D. Glascock (2002a): Ceramic production among small scale and mobile hunters and gatherers: A case study from the south western Great Britain Journal of Anthropological Archeology. 21:2 p 275-285.

Ettamayer, P., Kolaska, H., Lengauer, W., (1995): Metallurgy and Properties, International Journal Refractory Metals and Hard Materials 13; 343-351.

Ewule .J.O. (1988): Development of Model Gas Kiln for Ceramic Production from Local Resources, M.AThesis Ahmadu Bello University, Zaria, Nigeria

Gruss, W.W., Friederich K.M., (1995): Ceramic Cutting Tools, First Edition, Noyes Publications, New Jersey.

Hassan, S.B (2000); Refractory Properties of Bauchi and OnibodeClay of Nigeria for Furnace Lining, "African Journal of sciences and technology.vol.1No.1.

Hsieh C.L, Tuan .W.H (2005): InvestigatedElastic Properties of Ceramic–Metal particulate composites,Materials Science and Engineering: AVolume 393, Issues 1-2, Pages 133-139

Hussainova, I., (2003): Effect of microstructure on the erosive wear of titanium carbide-based cermets, Wear 255; 121-128.

Jaworska, L., Smuk, B., Królicka, D., Wszoáek, J., (2005): Cermets for cutting edges, Kompozyty 3 21-25, (in Polish).

Jones, J. T. and Berard, M. F.(1993). "Ceramics: Industrial Processing and Testing", (Iowa State University Press, Iowa), pp. 75-141.

Jordan, M.M. Almendro-Candel, M.B. Romero, M. and Rincon, J.M. (2005). "Application of Sewage Sludge in the Manufacturing of Ceramic Tile Bodies", *Applied Clay Science. Vol.* 30, pp. 219– 224.

Kawaguchi M., Aiuchi K., Nishimura Y., Aoki.S(2007): *Helium gas Transfer into an E-glass melt with bubbling Technique*.Proceedings of the XXIst International Congress on Glass,Strasbourg, July 1-6.

Khanna, O. P., (2010): A text book of material science and metallurgy.Dhanpat Publication, New Delhi.

KonopkaK., Olszówka, Myalska,A. andSzafran. M.,(2003): Ceramic–metal composites with an interpenetrating network, Materials Chemistry and PhysicsVolume 81, Issues 2-3, Pages 329-332

Kopeliovich, D.(2010). Classification of Composite Material.Retrieved 13th September, 2011, from www.substech.com.

Limpt Van, J. A. C. (2007): *Modeling of Evaporation Processes in Glass Melting Furnaces*, PhD dissertation Eindhoven Universityof Technology.

Mataba R.N. G (2005): Decorative element of design and on festival pot among the Margi people. Unpublished master thesis.

Menezes, R. R. Ferreira, H. S. Neves, G. A. Lira, H. L and Ferreira, H. C. (2005)."Use of Granite Sawing Wastes in the Production of Ceramic Bricks and Tiles", *Journal of the European Ceramic Society.*Vol.25, pp.1149–1158.

Morrell.R.(1987): Handbook of Properties of Technical and Engineering Ceramics, HMSO, London.

Muschick W. (2005): *The Validation of Glass Furnace Simulations in a Multilevel Process*, Proceedings XXthA.T.I.V. Conferenceon Modern Technologies and Techniques for GlassManufacturing, pp. 53-55, Parma, September 14-16.

Nelson, C. S. and Cochrane R. H. A., (1970): A rapid X-ray method for the quantitative determination of selected minerals in fine grained and altered rocks. Tane 16: 151-162

Nìmec L., Jebavá M. (2006): Glass Technology: Eur. J. Glass Sci. Technol. A *47*, 68.

Khanna O.P (2010) scEng (mech. MEC. HONS) PHD Prof head (retd) mechanical Eng department YMCA institute of engfaibadharpra.

Pattee, H.E., (1968): Joining Ceramics and Graphite to Other Materials, A Report." Office of Technology Utilization National Aeronautics and Space Administration, Washington D.C.

Rhodes, D., (1960): Stoneware and Porcela mart of high fined Pottery Pitman publishing Ltd. London.

Rongen M., Beerkens R. G. C., Faber A., Breeuwer R. (2006): *Physical Fining Techniques of Glass Melts*, Proc. 8th European Society of Glass

Rosso, M., (2003): Ceramic and metal matrix composite, route and properties, Proceedings of the 12th Scientific International Conference "Achievements in Mechanical and Materials Engineering" AMME', Gliwice-Zakopane, XXXV-I.

Science & Technology Conference session B2-Refining and melting II, paper B2-3, Sunderland, September 10-14.

Schussler.A. ZumGahr.K.-H(1993).Ceramic-Metal Composite Surface Layers by Laser Processing, *International Journal of Materials and Product Technology - Vol. 8, No.2/3/4 pp. 213 - 219*

Smith, William F.,HashemiJavad (2006): Foundations of Material science and Engineering (4th Ed) Mac Graw Hill.

Stricker, R., (1993): Das potential der cermets, TechnischeRundschau 85

SullaymanU.A.A(2005): Utilization of Local Raw Materials for the Production of Dense Alumino-silicate Refractory Bricks for Blast Furnaces using Semi Dry PressingTechnique, Ph.D., Dissertation , Department of Industrial Design, A.B.U, Zaria.

SullaymanU.A.A (2011): LAP LAMBERT Academic Publishing GmbH & Co. KG Heinrich Bocking Str. 6-8, Saarbrucken, Germany.

Ramachandra and Radhakrishna, K. (2006): Sliding wear, Slurry erosive wear and corrosive wear of aluminium/SiC composite. Vol 24. J. Material Sci Poland Pp 334-349

Umansky,A., A Panasyuk, L.Beloborodov, V.Smirnov, N.Sereda, (1999): Study of structure and phase-formation in (TiC-Mo2C)-(Ni-Mo) composite materials, Proceedings of the 9th- World Ceramic Congress and Forum on New Materials CIMTEC, Florence, Part C, 723-728.

Design Fax Online, "Hybrid Hip Joint"; Retrieved fromhttp://www.manufacturingcenter.com/dfx/archives/0503/0503app_ideas.asp) by 2015.

APPENDIX A

Table A1: Composite's bulk density (B.D)

S/No	Grey Cast Iron(%)	W_1(g)	W_2(g)	B.D(g/cm^3)
1.	0	14.72	115.59	1.72
2.	5	15.39	120.01	1.74
3.	10	16.67	121	1.86
4.	15	13.87	99.25	1.89
5.	20	18.90	135.03	1.89
6.	25	14.90	103.25	1.95
7.	30	15.44	104.79	1.99
8.	35	13.79	91.25	2.04
9.	40	13.81	91.24	2.04
10.	45	22.00	142.52	2.09

Table A2: Composite's density (D)

S/No	Grey Cast Iron (%)	Weight(g)	Volume(cm^3)	Density(g/cm^3)
1.	0	371.40	295.75	1.26
2.	5	394.90	295.75	1.34
3.	10	420.70	295.75	1.42
4.	15	454.40	295.75	1.54
5.	20	487.50	295.75	1.65
6.	25	517.80	295.75	1.74
7.	30	547.20	295.75	1.85
8.	35	582.70	295.75	1.97
9.	40	624.10	295.75	2.11
10.	45	650.70	295.75	2.20

Table A3: Composite's Apparent Porosity (A.P)

S/No	Grey Cast Iron (%)	Air Dried Weight (D) (g)	Oven Dried Weight (S) (g)	Wet Weight (W) (g)	A. P (%)
1.	0	449.6	382.3	505.4	45.33
2.	5	468.8	386.9	528.6	42.20
3.	10	484.5	391.1	546.3	39.82
4.	15	506.7	395.5	566.7	35.05
5.	20	533.6	399.8	595.1	31.49
6.	25	564.2	404.2	628.4	28.64
7.	30	593.5	407.7	657.2	25.53
8.	35	632.3	410.1	695.2	22.06
9.	40	658.5	412.8	718.1	19.52
10.	45	679.7	414.4	730.5	16.07

Table A4: Composite's Water Absorption (W.A)

S/No	Grey Cast Iron(%)	Dried Weight (D) (g)	Wet Weight (W) (g)	W.A (%)
1.	0	371.4	561.5	36.07
2.	5	394.9	570.8	33.86
3.	10	420.7	582.6	30.81
4.	15	454.4	605.9	27.79
5.	20	487.5	637.1	25.01
6.	25	517.8	663.3	23.43
7.	30	547.2	681.9	21.94
8.	35	582.7	708.9	19.76
9.	40	624.1	736.2	17.81
10.	45	371.4	561.5	15.20

Table A5: Composite's Linear Shrinkages (LS)

S/No	Grey Cast Iron (%)	Green Shelve Length (L_B) (mm)	Fired Shelve Length (L_F) (mm)	LS (%)
1.	0	83.00	72.40	12.77
2.	5	83.00	73.40	11.57
3.	10	83.00	75.00	9.64
4.	15	83.00	76.85	7.41
5.	20	83.00	77.20	6.99
6.	25	83.00	79.80	3.87
7.	30	83.00	81.00	2.41
8.	35	83.00	81.36	1.97
9.	40	83.00	81.69	1.58
10.	45	83.00	82.05	1.15

Table A6: Composite's refractoriness

S/No	Grey Cast Iron (%)	Refractoriness (^{0}C)
1.	0	> 1500
2.	5	1400
3.	10	1300
4.	15	1300
5.	20	1300
6.	25	1200
7.	30	1200
8.	35	1000
9.	40	1000
10.	45	1000

Table A7: Composite's Cold Crushing Strength (CCS)

S/No	Grey Cast Iron (%)	Load (KN)	CCS (N/mm^2)
1.	0	13.7	2.19
2.	5	24.1	3.87
3.	10	37.2	5.97
4.	15	50	8.01
5.	20	67.3	10.78
6.	25	83.2	13.32
7.	30	64.4	10.24
8.	35	48.5	7.75
9.	40	37.8	6.05
10.	45	32.7	5.25

Table A7: Composite's hardness values

S/No	Grey Cast Iron (%)	Hardness Number (Shores)
1.	0	38
2.	5	42
3.	10	57
4.	15	61
5.	20	70
6.	25	76
7.	30	81
8.	35	87
9.	40	90
10.	45	92

Table A8: Composite's Hardness Values

S/No	Grey Cast Iron(%)	Hardness Number(Shores)
1.	0	43
2.	5	47
3.	10	61
4.	15	69
5.	20	76
6.	25	81
7.	30	86
8.	35	90
9.	40	92
10.	45	94

Table A.9: Composite's Ultimate Tensile Strength (UTS)

S/No	Grey Cast Iron(%)	Load(KN)	UTS(N/mm^2)
1.	0	4.9	0.78
2.	5	6.1	0.99
3.	10	9	1.43
4.	15	11.1	1.78
5.	20	11.5	1.84
6.	25	11.7	1.87
7.	30	11.2	1.80
8.	35	10.4	1.63
9.	40	9.8	1.55
10.	45	8.5	1.34

Table A10: Composite's Ultimate Compressive Strength (UCS)

S/No	Grey Cast Iron(%)	Load(KN)	UCS(N/mm^2)
1.	0	8.3	1.32
2.	5	19.4	3.11
3.	10	24.8	3.98
4.	15	35.4	5.67
5.	20	40.5	6.49
6.	25	44.6	7.15
7.	30	41.4	6.63
8.	35	35.9	5.75
9.	40	31.1	4.99
10.	45	23.4	3.74

Table A11: Thermal degradation temperature of composites

Weight of Grey iron particles (%)	**$T_{10\%}$ (^oC)**	**$T_{20\%}$(^oC)**	**T_{max}(^oC) composition**
0	545	>900	523
10	602	>900	528
25	600	>900	530
35	601	>900	521
45	580	>900	520

APPENDIX B

X-RF TEST FORM DEPARTMENT OF CHEMISTRY ABU ZARIA

Oxford Instruments

OPERATOR: INSTRUMENT MANAGER

SAMPLE: 1

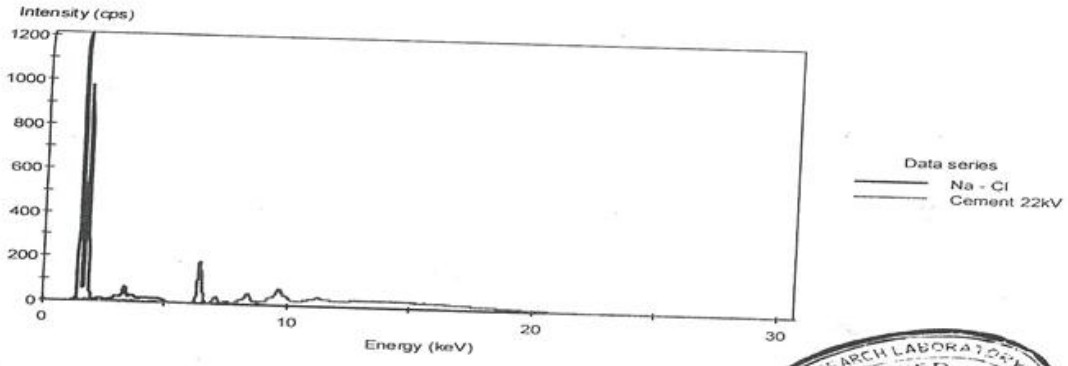
ANALYSED: 19/06/2013 1:53:29 PM

Analysis Method: Minerals

Analyte Concentration Table

Element	Concentration
Na2O	0.000 Wt %
MgO	0.394 Wt %
Al2O3	41.872 Wt %
SiO2	55.937 Wt %
P2O5	0.250 Wt %
SO3	0.292 Wt %
Cl	0.003 Wt %
K2O	0.342 Wt %
CaO	0.126 Wt %
TiO2	0.120 Wt %
Cr2O3	0.000 Wt %
Mn2O3	0.008 Wt %
Fe2O3	0.655 Wt %
ZnO	0.002 Wt %
SrO	0.000 Wt %

Spectra for all conditions





Printed on 19/06/2013 2:56:13 PM

Page 1

Figure B1: X-ray Florescence test Spectrum

Samples 1, 2,3.

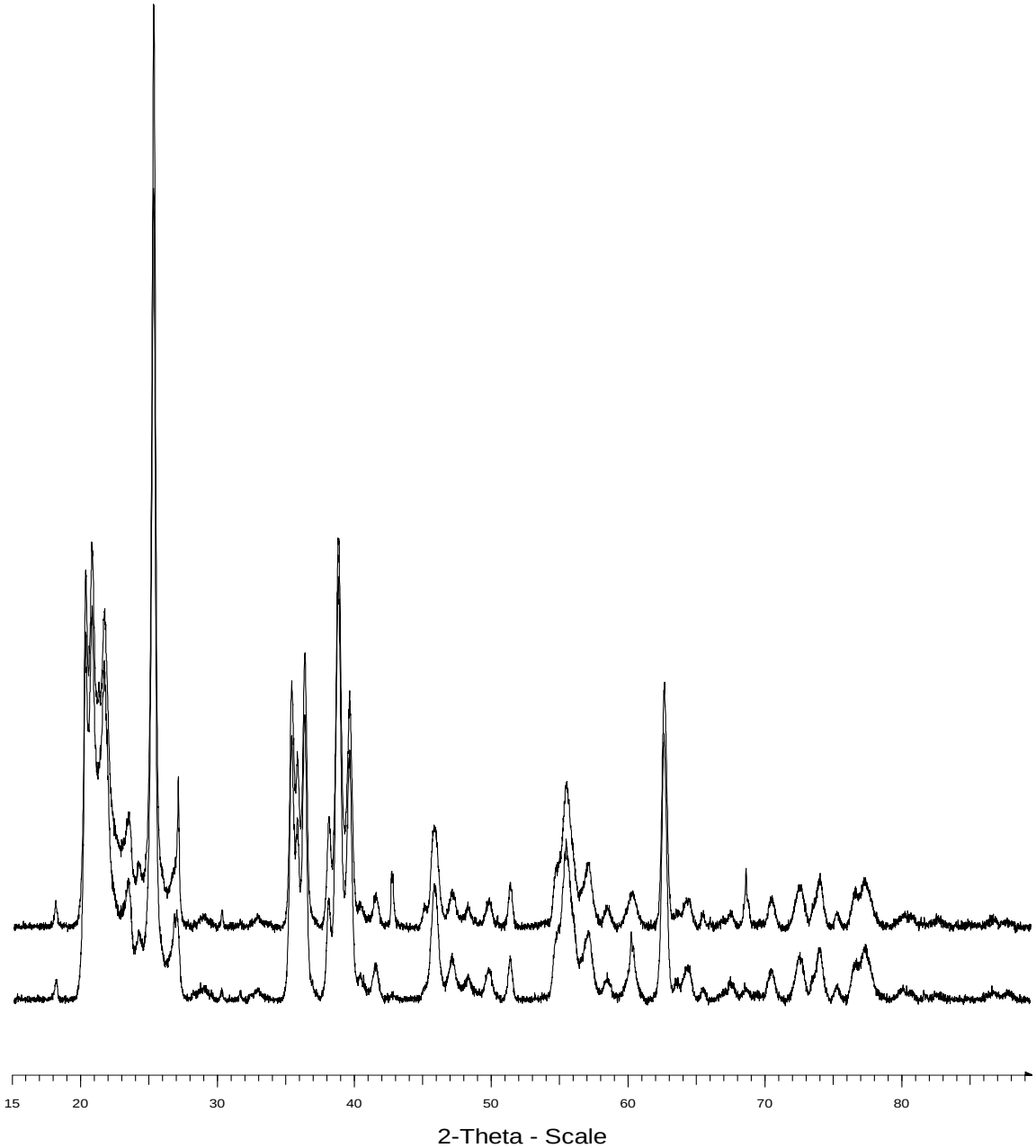


Figure B2:X-RD TEST SAMPLE 1,2,3

sample1

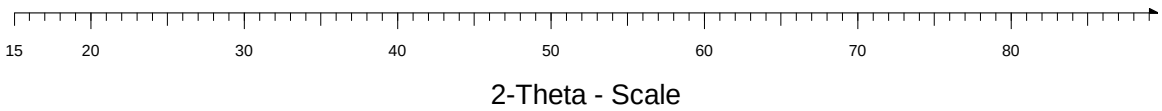


Figure B3:X-RD TEST SAMPLE 1

sample2

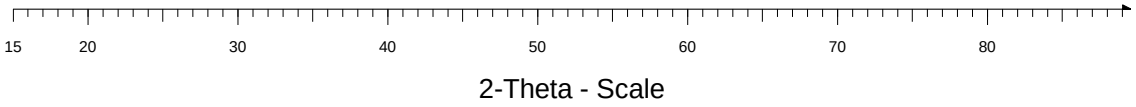


Figure B4:X-RD TEST SAMPLE 2

TEST RESULT FORM: SIEVING ANALYSIS

SAMPLE A (Lassa clay) OPERATOR:
Date: 6/8/03 LABORATORY: BIdp

FINAL SIEVE SELECTED: 0.1 MM Or 0.08 mm

Determination of the mass of the test sample

- size of largest elements contained in the soil D = 5
- the dry mass of test sample (m_d) has to he: $200D < m_0 < 600\ D$ $1\ 000 < m < 2000$

WET MASS OF THE SAMPLE M_h (g)	1585
DRY MASS OF THE SAMPLE - cither drying then $m_{f]tp)}$ - or using the measurement of the water content of the sample $md = m_{ul} \times m_{mh}$ m_{1b}	1504.17

Sieving the sample

SIEVE IN MM	MASS OF RETAINED FRACTIONS in grams	MASS OF ' CUMULATED RETAINED FRACTIONS (Rj)	% CUMULATED RETAINED FRACTIONS Rjx 100 m_d	% CUMULATED PASING FRACTIONS 100 -(Rjx $_{10}$o)
20				%
10				%
5				%
2	85.66	85.66	5.69	94.31 %
1	212,05	297.71	19.79	80.21 %
0.4	262.25	560.46	37.26	62.74 %
0,2	129.80	670.26	45.89	54.11 %
0.1 or 0.08	71.44	761.17	50.6	49.4 %
FRACTION PASSING FINAL SIEVE (Tn) (g)				741.7
CUMULATED RETAINED FRAC'FION OF FINAL SIEVE (Rn)				761.7
TOTAL MASS AFTER SIEVING: Rn + Tn				1502.87
EVALUATION OF LOSSES: 100 x - ($R_n + T_n$) =< 2 %? YES O No 0 M_d				

EARTH CONSTRUCTION TECHNOLOGIES BUILDING DEPARTMENT ABU, ZARIA.

Figure 6(a): Test Results Form: Sieving Analysis

Figure B5: Test results from sieving analysis

TEST RESULTS FORM: SIEVING ANALYSIS

SAMPLE Hole A (clay/kaolin) OPERATOR;

Date: 6/8/2014 LABORATORY: Bldp

MAXIMUM PARTICLE DIMENSION

MASS OF THE SAMPLE [0.1 mm]

MEASURING CYLINDER USED I litre , 2 litre [50]

DISPERSING AGENT =

METHOD FOR DETERMINING THE CORRECTION FACTORS: - CALIBRATION

-CONTROL 0

- MEASURING CYLINDER 0

CUMULATED RETAINED FRACTION OF TOTAL SAMPLE ON 0.1 mm OR 0.08mm SIEVE

R = 50.6 %

PASSING FRACTION OF TOTAL SAMPLE ON 0.1 mm SIEVE

T-49.4

START TIME OF THE FINAL MIX: .....
START TIME OF SEDIMENTATION ANALYSIS ... 0.8^{15} Am.....

Equivalent diameter in mm	TIME	TIME min	TEMP. °C	R (reading)	C (corrected readings]	R_I (R+C)	P-4rl C)	T Xp (2)
0,07	$08^{15.30}$	0.5	27.3	19.75	2..75	22.5	90 - -	44.46%
0.05	08^{17}	1	27.3	19.25	2.5	21.75	87	43. o/ /o
0.035	08^{18}	2	27.3	18.5	2.5	21	84	41.5 %
0.025	08^{21}	5	27.3	17.75	2.5, :	20.35	81.4	40.21 %
0.018	08^{25}	10	27.3	16.80	2.5	19.3	77.2	38. 14 %
0.012	08^{36}	20	27 "3	16.0	2.5	J8.5	74	36.56 %
0.009	08^{56}	40	27.3	15.5	2.5	18.	72	35.57.%
0.006	09^{36}	80	27.5	14.0	2.5	16.5	66	32.6 %
0.0035	12^{16}	240	27.8	13.5	2.5	15.6	64	31.6 %
0.0015	08^{16}	1440	26.6	13.0	2.25	15.75	63	31.12 %

(1) % passing fraction of particles of the sample smaller than 0.1 or 0.08 mm
(2) % passing fraction of particles of the total sample

- EARTH CONSTRUCTION TECHNOLOGIES BUILDING DEPARTMENT ABU, ZARIA

Figure 6(b): Test Results Form: Sieving Analysis

Figure B6: Test results from sieving analysis

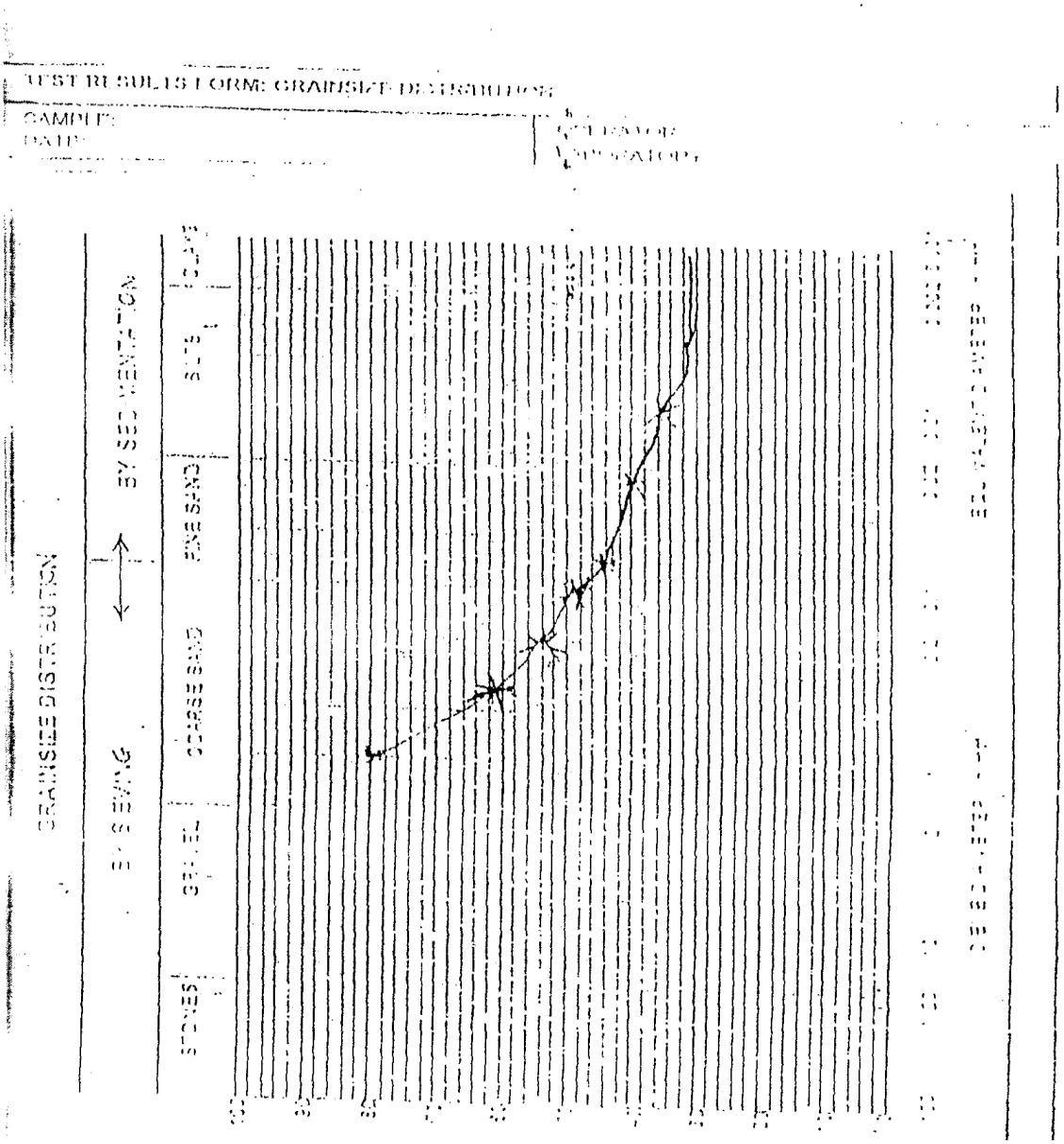


Figure B7: Determination of the Grain size distribution



Plate B1: COMPRESSING MACHINE



Plate B2: composite Test sample in different sizes.

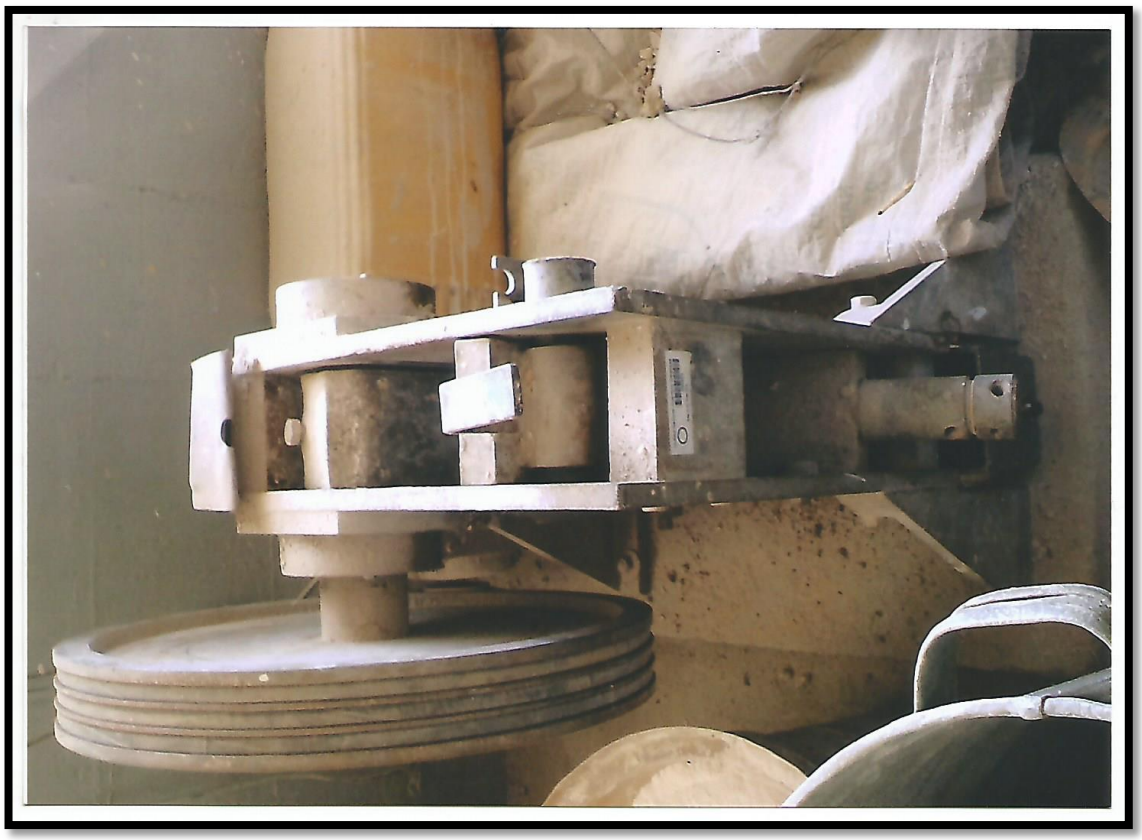


Plate B3: Jaw crushing machine



Plate B4: Collection of materials



Plate B5: The developed composite sample for the best material to be used for the production of shelves

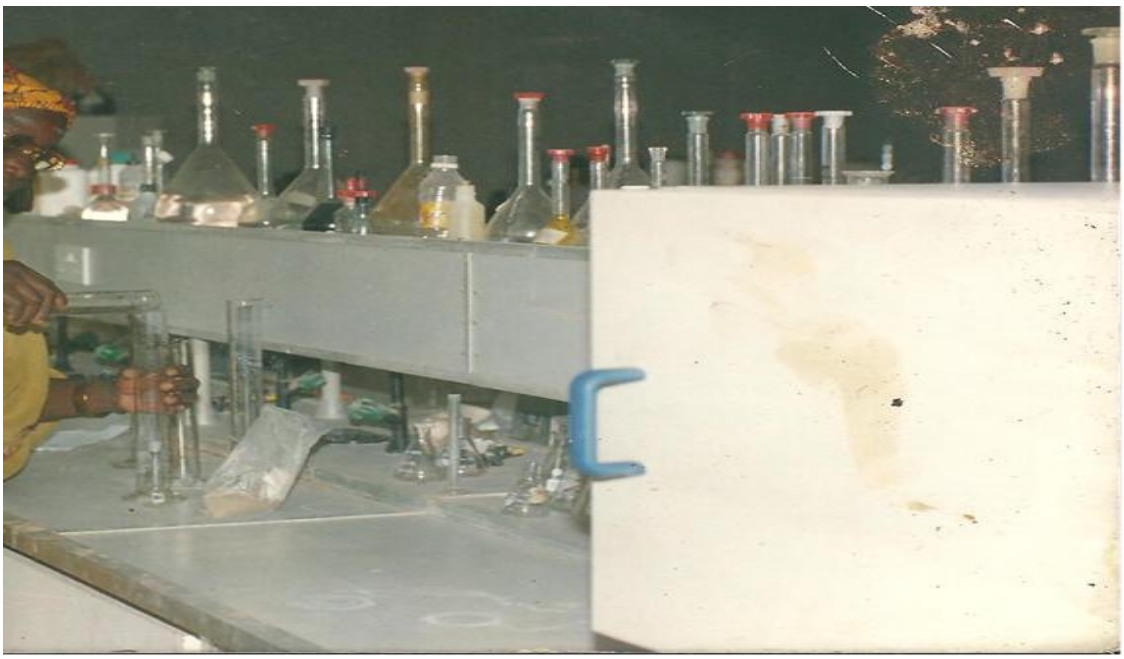


Plate B6: Moisture content test in Agric laboratory A.B.U Zaria

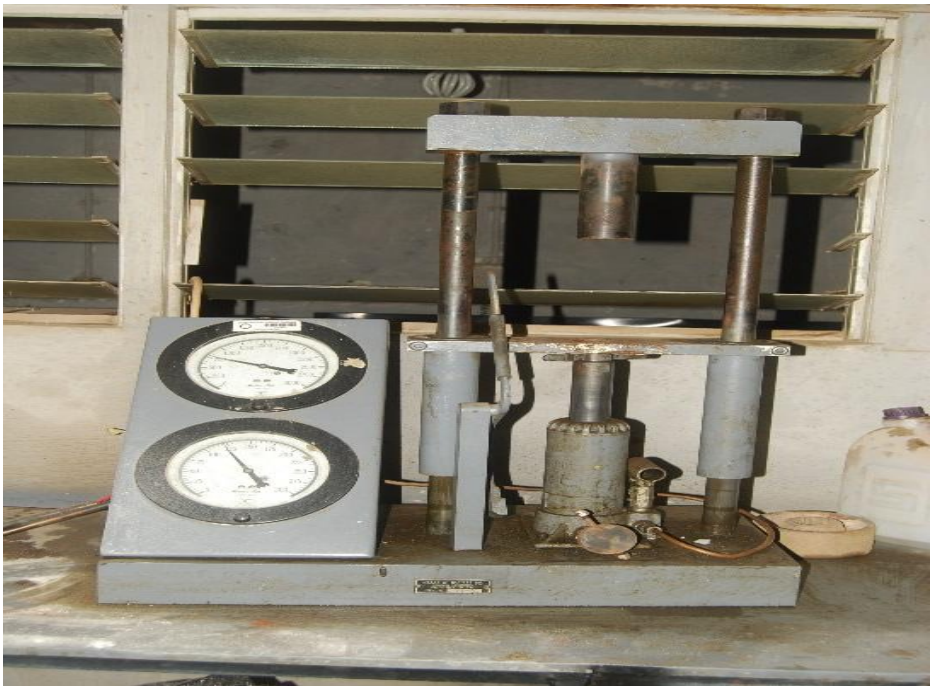


Plate B7: Universal Testing Machine

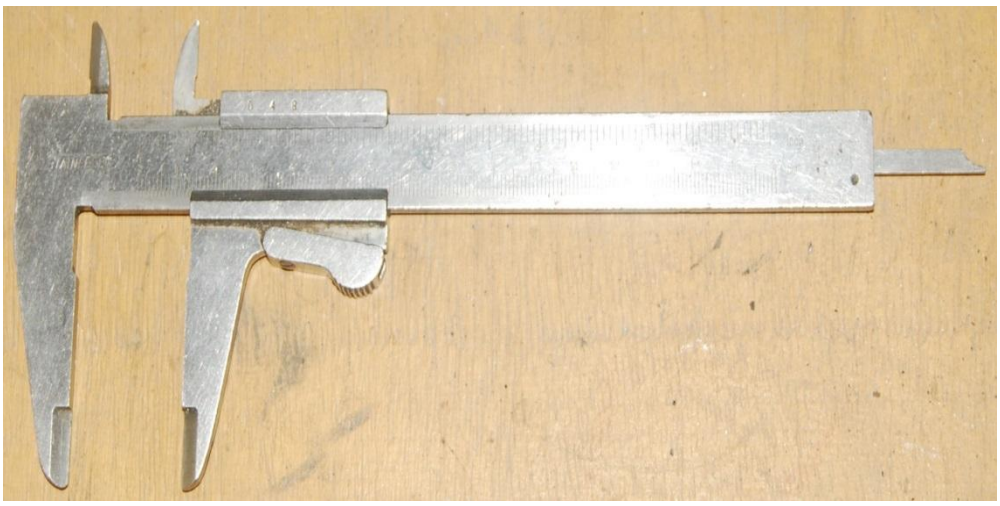


Plate B8: Vernier Caliper for linear measurement



Plate B9: Pyrometer for measurement of temperature



Plate B10: ASTM Standard Sieves

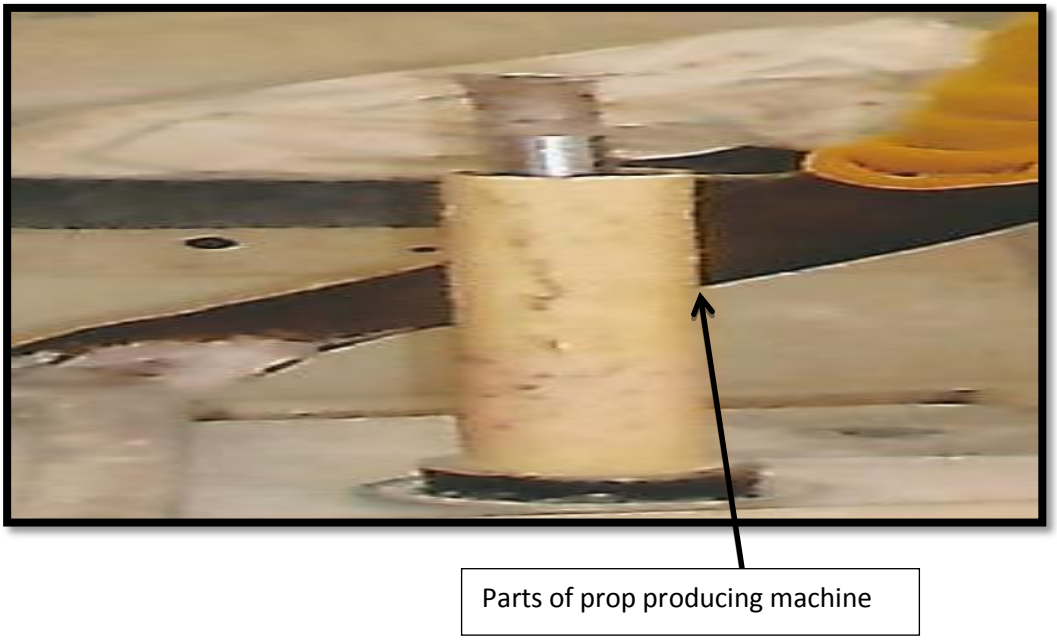


Plate B11: Part of prop producing machine

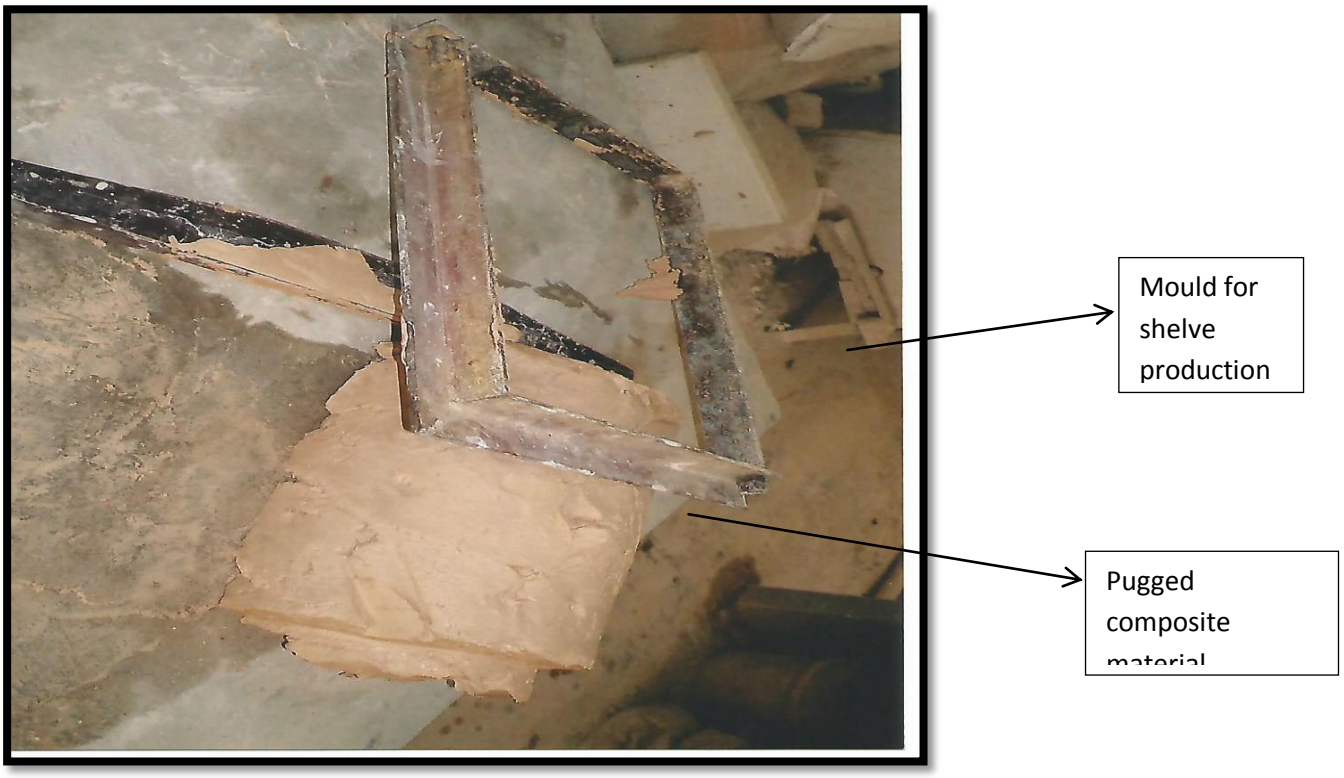


Plate B12: Pugged clay and mould for shelve production

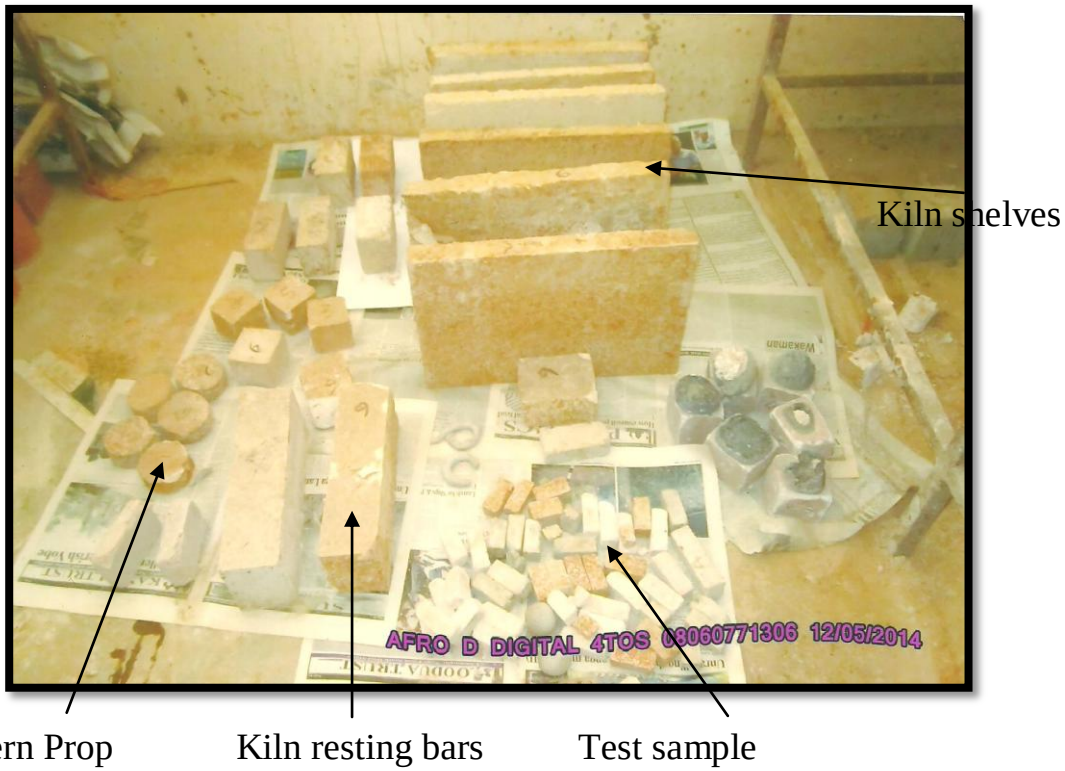


Plate B13: Collection of Fired Kiln shelve, Kiln bars and Props

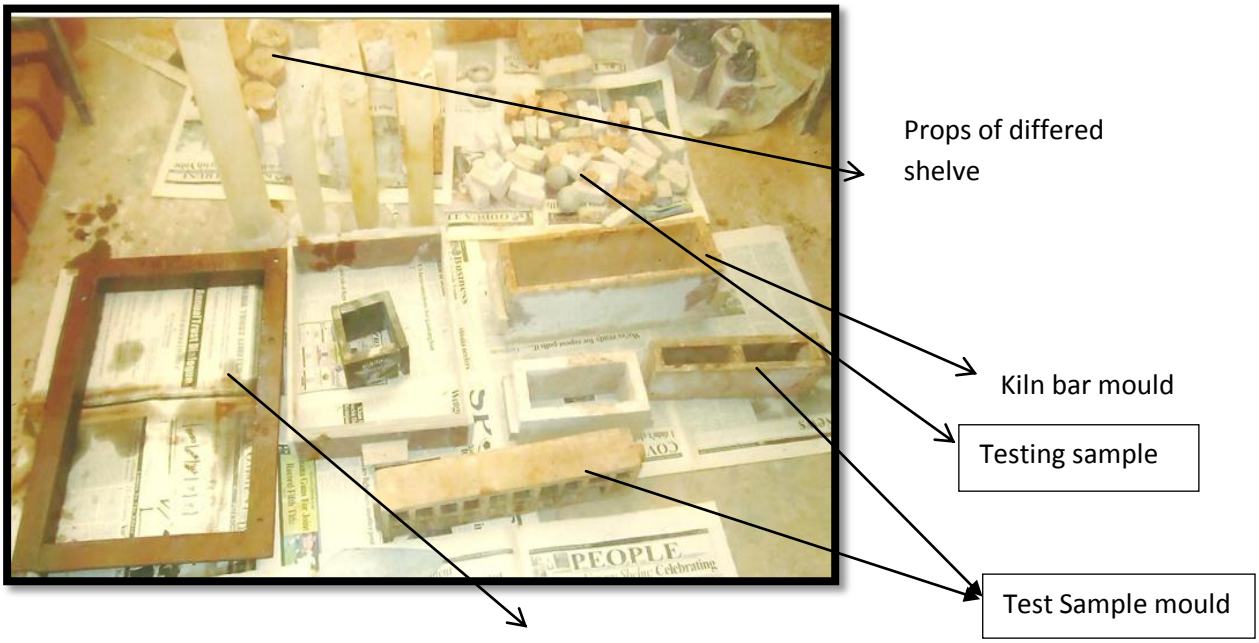


Plate B14: Production Mould for shelves