

KWAME NKRUMAH UNIVERSITY OF SCIENCE AND TECHNOLOGY,
KUMASI, GHANA

**Investigation of Structural and Radiological Shielding Properties of Pozzolana-
Portland Cement**

by
KNUST

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DECLARATION

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

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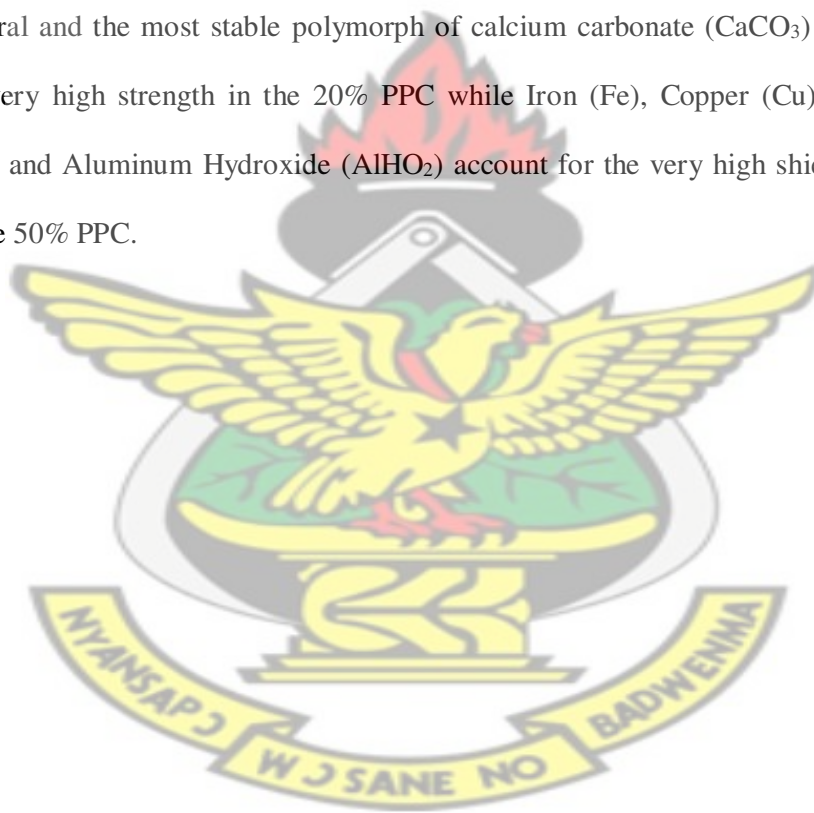
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ABSTRACT

The physical properties of the ordinary Portland cement (OPC) are enhanced when the matrix of the OPC is partially replaced by admixtures such as Pozzolana. This research investigated the radiological shielding properties and the structural strength of Pozzolana-Portland Concrete (PPC) at different ratios of 0% (S_0), 10% (S_1), 20% (S_2), 30% (S_3), 40% (S_4), 50% (S_5), 60% (S_6), 70% (S_7) and 80% (S_8) of Pozzolana. Radiological shielding property of a material is its ability to shield or absorb ionizing particles/rays; thus preventing it from being released into the environment when a source thereof is housed in containment. Ionizing radiations are found in nuclear reactors, γ -rays and x-rays equipment used in energy and health sectors. They are also found in sources used in quality assurance industries. Compression test was performed using CONTROL electronic type compression testing machine. Test samples were made of concrete (mixture of Portland and Pozzolana cement) cubes and cylinders. The experiments were conducted at a temperature of 27 ± 2 °C at the following days of aging; 2, 7, 14 and 28. The compression test results showed that concrete of Portland-Pozzolana cement (PPC) at 20% Pozzolana has more compressive strength than that of the ordinary Portland cement at 28 days of curing. Flexural and ultrasonic tests were conducted to confirm the results. Neutron activation analysis (NAA) was also carried out to determine the elemental compositions of Pozzolana and ordinary Portland cement. The neutron activation analysis results showed that Europium, Hafnium, Copper, Calcium, Iron, Cobalt and Aluminum, all of which have significant macroscopic cross sections (shielding properties) dominating more in Pozzolana cement than in the ordinary Portland cement. X-ray shielding analysis was carried out to ascertain the veracity of the NAA results. Linear attenuation coefficient (μ_1) of each sample at photon energy of 0.15

MeV was determined with the 50% PPC showing highest value. Further, X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analyses were performed to identify the phases contributing to the highest strength at 20% PPC (33 MPa) and highest shielding, μ_1 at 50% PPC (1.533 cm^{-1}). The results in all showed that Pozzolana positively affects structural and radiological shielding properties of the ordinary Portland cement (OPC), since the OPC (Control) concrete had a compressive strength of 28.44 MPa at day 28 and a linear attenuation coefficient (μ_1) of 0.140 cm^{-1} . The XRD results showed that Calcite (CaCO_3), which is a carbonate mineral and the most stable polymorph of calcium carbonate (CaCO_3) accounts for the very high strength in the 20% PPC while Iron (Fe), Copper (Cu), Manganese (Mn) and Aluminum Hydroxide ($\text{Al}(\text{OH})_3$) account for the very high shielding ability of the 50% PPC.



DEDICATION

This research work is dedicated to my Mother, Madam Kafui Abla Agbodotsi and my Siblings; Bertha, Alberta and Albert.

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TABLE OF CONTENTS

DECLARATION.....	ii
ABSTRACT	iii
DEDICATION	v
ACKNOWLEDGEMENT	vi
TABLE OF CONTENTS.....	viii
LIST OF FIGURES	xiv
LIST OF TABLES	xvii
LIST OF ABBREVIATIONS	xx
ELEMENTS/COMPOUNDS AND SYMBOLS	xxii
CHAPTER ONE.....	1
INTRODUCTION	1
1.1 Background.....	1
1.2 Radiation Shielding and Protection	1
1.2.1 Need for Radiation Shielding Materials at Low Cost in Ghana	2
1.3 Need for Low Cost High Performance Concrete from Local Materials	2
1.3.1 Global Cement Demand and Subsequent Environmental Implications	4
1.4 Research Problem Statement	5
1.4.1 Relevance and Justification	6
1.5 Objective	7
1.5.1 Specific objective	7
1.6 Scope.....	7
1.7 Thesis Organization	8
CHAPTER TWO.....	9

LITERATURE REVIEW	9
2.0 Introduction	9
2.1 Portland Cement	9
2.1.2 Types of Portland cement.....	12
2.1.3 Chemical Reactions of Cement	13
2.2 Pozzolana.....	18
2.3 Concrete.....	20
2.3.1 Process of Concrete Manufacture	20
2.3.2 Aggregates in Concrete	21
2.3.3 Curing of Concrete.....	21
2.3.4 Compaction.....	22
2.3.5 Setting	23
2.3.6 The Different Grades of Ordinary Portland Cement (OPC).....	23
2.4 Portland-Pozzolanic Cement (PPC).....	24
2.4.1 Effects of Pozzolana on OPC	24
2.5 Structural or Mechanical Property Analysis of PPC.....	25
2.5.1 Fineness of Cement and Its Effect on Strength	27
2.5.2 Factors Responsible for Governing Compressive Strength of	28
2.6.2 Radiological Shielding Property Analysis of Matter (Concrete).....	32
2.6.2.1 Attenuation Coefficient	32
2.6.2.2 Bremsstrahlung	33
2.6.2.3 Thermal and Fast Neutrons.....	37
2.6.2.4 Microscopic Cross Sections, σ [barns or cm^2].....	37
2.6.2.5 Macroscopic Cross Section, Σ [cm^{-1}].....	39
2.7 Compression Test.....	40

2.7.1	Compressive Strength	42
2.8	Flexural Test	42
2.8.1	Types of Flexural Test.....	42
2.8.2	Flexural Test Formulae	43
2.10	Neutron Activation Analysis (NAA)	49
2.11	X-Ray Shielding Analysis	53
2.11.1	Estimation of Material Thickness (x) for Shielding.....	54
2.12	X-ray Powder Diffraction (XRD)	54
2.12.1	X-ray Powder Diffraction (XRD) Instrumentation.....	55
2.12.2	Applications of XRD.....	56
2.12.3	Strengths and Limitations of X-ray Powder Diffraction.....	57
2.12.3.1	Strengths.....	57
2.12.3.2	Limitations.....	57
2.12.4	Sample Collection and Preparation for XRD	58
2.12.5	Data Collection, Results and Presentation of XRD	59
2.12.7	Crystal Geometry Equations for X-ray Diffraction Analysis	65
2.12.7.1	Lattice Model for D-spacings and Miller indices	66
2.12.7.4	Method of Calculation of Atomic Packing Factor for Calcite.....	69
CHAPTER THREE		73
MATERIALS AND METHODS		73
3.0	Introduction	73
3.1	Materials and Samples Preparation.....	73
3.2	Sample Moulding.....	73
3.3.4	Neutron Activation Analysis (NAA)	81

3.3.5	The Shielding Experiment.....	82
3.3.7	Methodology in Scanning Electron Microscopy (SEM).....	87
CHAPTER FOUR		88
RESULTS AND DISCUSSION		88
4.0	Introduction	88
4.1	Analysis of Compressive Strength Results.....	88
4.2	Flexural Strength Results for Confirmatory Test)	90
4.3	Verification of Compressive Test Results Using UT	91
4.4	Shielding Property Analysis of PPC from Elemental Composition	92
4.5	Shielding Property Analysis of PPC:Attenuation Coefficient Point of View	97
4.6	Crystal Structural Analysis of PPC.....	103
4.7	Microstructure of Some Characteristic PPC (S ₀ , S ₂ , S ₅ and S ₈).....	113
CHAPTER FIVE.....		115
CONCLUSION AND RECOMMENDATION.....		115
5.1	Mechanical behaviour	115
5.1.2	Validation of Mechanical Properties of PPC.....	116
5.2	Shielding Properties.....	116
5.2.2	Validation of Shielding Properties of PPC.....	116
5.3	Structural, Radiological and Economic Advantages of PPC over OPC	116
5.4.1	Observations	117
5.4.3	The Phases /Elements responsible for the Improvement in the Shielding	118
Properties of PPC		118
5.5	Recommendation	119

REFERENCES.....	119
APPENDICES.....	127
APPENDIX (A).....	127
Tables of Mix Ratios (1:2:4) and Compressive Strength: W/C = 0.5	127
APPENDIX (B).....	132
Tables of Compressive Strength: W/C = 0.6	132
APPENDIX (C).....	135
Tables of Compressive Strength: W/C = 0.7	135
APPENDIX (D).....	137
Table of Flexural Strength (Modulus of Rupture): W/C = 0.5	137
APPENDIX (E).....	138
Tables of Ultrasonic test	138
APPENDIX (F)	141
NAA Results	141
APPENDIX (G).....	143
X-Ray Shielding Experiment	143
APPENDIX (H).....	149
Derived Data for Calculating Linear attenuation Coefficients	149
APPENDIX (I)	152
XRD Data.....	152
APPENDIX (J).....	162
CALCULATIONS	162
J1 Calculation of Compressive Stress	162
J2 Flexural Strength (Modulus of Rupture) Calculation	163

J3	Shielding Calculation Analysis	164
J4	Calculation of Sample (Concrete) Mass Estimation.....	165

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LIST OF FIGURES

Figure 2.1: Cement Production Process.....	10
Figure 2.2: Ternary subsystem in Portland cement systems.....	12
Figure 2.3: Rate of heat evolution (Mindess and Yong, 1981).....	15
Figure 2.4: Linear Attenuation of gamma rays under conditions of good geometry	35
Figure 2.5: Fractured Test Specimen at Failure	41
Figure 2.6: Layout of a single loading axis. Left: 5000 kN axis, right: 2000 kN axis	41
Figure 2.7: Schematic diagram: Flexural Test machine	43
Figure 2.8: Flexural Test Machine Loaded (Centre-point Loading)	43
Figure 2.9: PUNDIT TESTER (General View Layout) (Halmshaw, 1987).....	49
Figure 2.10: Ultrasonic pulse velocity testing (Halmshaw, 1987).....	49
Figure 2.11: X-Ray Diffraction schematic diagram.....	61
Figure 2.12: Crude sketches of the (111) set of Miller planes.....	64
Figure 3.1: Mould Design and Preparation.....	74
Figure 3.2: Mould Being Assembled: (a) Metallic mould (b) Multi cubic mould	74
Figure 3.3: Thorough Mixing of concrete being carried out manually	75
Figure 3.4: Concrete Cubes Being Cast.....	75
Figure 3.5: Concrete Cubes Ready for Laboratory Analysis	76
Figure 3.6: Casting Cylindrical Concrete	76
Figure 3.7: Cylindrical Concrete ready for flexural test.....	77
Figure 3.8: Young's Machine Dial Gauge	78
Figure 3.9: Young's Machine Loaded with Concrete Cube	79
Figure 3.10: Flexural Testing Machine (Centre-point Loading) used.....	80
Figure 3.11: The PUNDIT plus set (Used in the experiment)	81
Figure 3.12: Block Diagram of X-Ray Equipment Arrangement	83

Figure 3.13: X-ray incident beam Sizing on Concrete Cube Surface via Radiography	83
Figure 3.14: A sample being placed in front of X-ray source.....	84
Figure 3.15: Sample undergoing x-ray irradiation (15cm thick)	84
Figure 3.16: Sample undergoing x-ray irradiation (30cm thick)	85
Figure 3.17: Sample undergoing x-ray irradiation (45cm thick)	85
Figure 3.18: $I_0, I_{15}, I_{30}, I_{45}$ for the various PPC being recorded at the console	86
Figure 4.1: Compressive Strength Development over Days.....	88
Figure 4.2: Various PPC Compressive Strength Evolution over Days	89
Figure 4.3: Compressive Strength vs. Age (Days), As W/C Varied for 20%PPC.....	89
Figure 4.4: Avg. Modulus of Rupture vs Pozzolana Percent.....	90
Figure 4.5: (a) Avg. Elastic Modulus of PPC	91
Figure 4.5: (b) Avg. Ultrasonic Pulse Velocity of %PPC	91
Figure 4.6: Percentage Composition of Some Significant Elements in Pozzolana.....	93
Figure 4.7: Density of Some Elements in Pozzolana or OPC.....	94
Figure 4.8: Elements in OPC and Pozzolana of Significant Shielding Characteristics.....	94
Figure 4.9: Linear Attenuation Coefficients of Some Elements in Pozzolana or OPC.....	95
Figure 4.10: Macroscopic Cross Section of Some Elements in Pozzolana or OPC.....	95
Figure 4.11: Linear attenuation Curve for S_0	98
Figure 4.12: Linear attenuation Curve for S_1	98
Figure 4.13: Linear attenuation Curve for S_2	99
Figure 4.14: Linear attenuation Curve for S_3	99
Figure 4.15: Linear attenuation Curve for S_4	100
Figure 4.16: Linear attenuation Curve for S_5	100
Figure 4.17: Linear attenuation Curve for S_6	101
Figure 4.18: Linear attenuation Curve for S_7	101

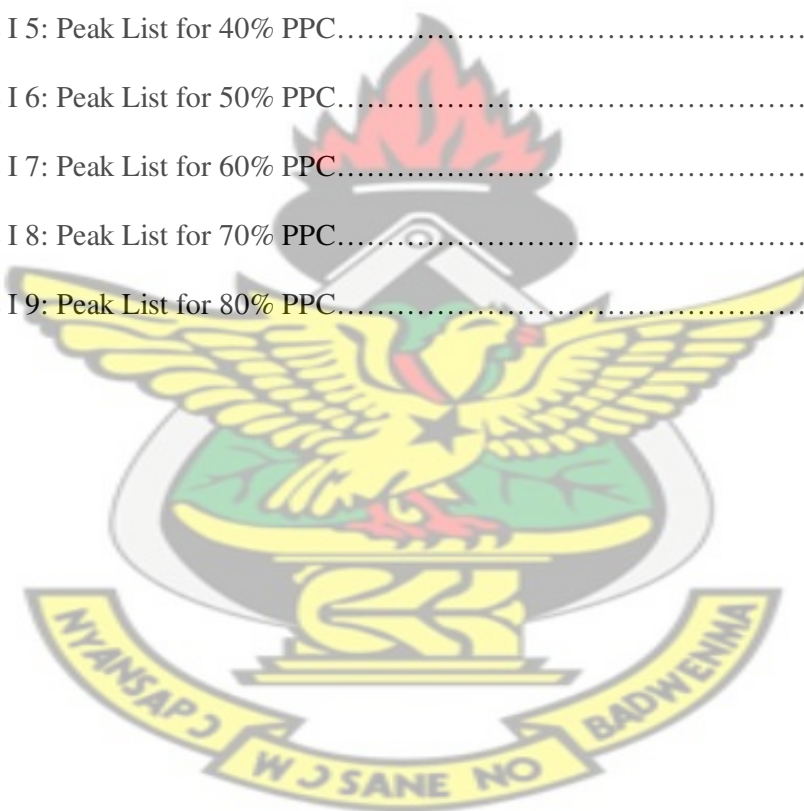
Figure 4.19: Linear attenuation Curve for S_8	102
Figure 4.20: Linear Attenuation Coefficients of Various PPC Compositions.....	102
Figure 4.21 (a): X-Ray diffraction graph of S_0 (Control):.....	104
Figure 4.21 (b): Percentage Abundance of Compounds in S_0 (Control):.....	104
Figure 4.22 (a): X-Ray diffraction graph of S_1	105
Figure 4.22 (b): Percentage Abundance of Compounds in S_1	105
Figure 4.23 (a): X-Ray diffraction graph of S_2	106
Figure 4.23 (b): Percentage Abundance of Compounds in S_2	106
Figure 4.24 (a): X-Ray diffraction graph of S_3	107
Figure 4.24 (b): Percentage Abundance of Compounds in S_3	107
Figure 4.25 (a): X-Ray diffraction graph of S_4	108
Figure 4.25 (b): Percentage Abundance of Compounds in S_4	108
Figure 4.26 (a): X-Ray diffraction graph of S_5	109
Figure 4.26 (b): Percentage Abundance of Compounds in S_5	109
Figure 4.27 (a): X-Ray diffraction graph of S_6	110
Figure 4.27 (b): Percentage Abundance of Compounds in S_6	110
Figure 4.28 (a): X-Ray diffraction graph of S_7	111
Figure 4.28 (b): Percentage Abundance of Compounds in S_7	111
Figure 4.29 (a): X-Ray diffraction graph of S_8	112
Figure 4.29 (b): Percentage Abundance of Compounds in S_8	112
Figure 4.30: Microstructure of 100% OPC; Control (S_0).....	113
Figure 4.31: Microstructure of 20% PPC (S_2)	113
Figure 4.32: Microstructure of 50% PPC (S_5)	114
Figure 4.33: Microstructure of 80% PPC (S_8)	114

LIST OF TABLES

Table 2.1: Typical compositions of phases in Portland cement clinkers.....	11
Table 2.2: Composition and properties of ASTM Types I to V Cements	13
Table 2.3: Chemical Composition of Portland and Pozzolana Cement (%wt).....	24
Table 2.4: Mechanical properties of pozzolana cement mortar	26
Table 2.5: Average values of compressive strength for mortar mixes	27
Table 2.6: Some standard specifications of Portland cement from different.....	28
Table 2.7 Typical Mix Proportions (Cement: Sand: Aggregate and w/c ratio)	29
Table 2.8: Comparison of mass attenuation coefficients for sampled soil and sand... 30	
Table 2.9: Measured values of linear attenuation coefficients (cm^{-1}) for different	31
Table 2.10: Total Mass and Linear Attenuation Coefficients for Some Materials	37
Table 2.11: Elements/Compounds & Their Macroscopic Cross Sections for neutron	40
Table 2.12: Quality of concrete as a function of the UPV.....	48
Table 2.13: Isotopic neutron sources:.....	52
Table 2.14: The seven crystal systems and the restrictions.	63
Table 4.1: Macroscopic Cross Section Interpretation for Pozzolana	96
Table 4.2: Macroscopic Cross Section Interpretation for OPC	96
Table A 1: Percentage Composition of Materials.....	127
Table A 2: Two (2) Days Compressive Strength at $W/C = 0.5$	128
Table A 3: Seven (7) Days Compressive Strength at $W/C = 0.5$	129
Table A 4: Fourteen Days (14) Compressive Strength at $W/C = 0.5$	130
Table A 5: Twenty-eight Days (28) Compressive Strength at $W/C = 0.5$	131
Table B 1: Seven (7) Days Compressive Strength at $W/C = 0.6$	132
Table B 2: Fourteen Days (14) Compressive Strength at $W/C = 0.6$	133
Table B 3: Twenty-eight Days (28) Compressive Strength at $W/C = 0.6$	134

Table C 1: Seven (7) Days Compressive Strength at W/C = 0.7.....	135
Table C 2: Fourteen Days (14) Compressive Strength at W/C = 0.7.....	136
Table C 3: Twenty-eight Days (28) Compressive Strength at W/C = 0.7.....	137
Table D 1: Modulus of Rupture (Flexural Strength) of Concrete at 28 Days.....	137
Table E 1: Seven (7) Days Ultrasonic test: W/C = 0.5.....	138
Table E 2: Fourteen Days (14) Ultrasonic test.....	139
Table E 3: Twenty-eight Days (28) Ultrasonic test.....	140
Table F 1: Neutron Shielding (Macroscopic Cross Section) Interpretation.....	141
Table F 2: X/Gamma Ray Shielding (Attenuation Coefficient) Interpretation.....	142
Table G 1: Percentage Composition of Materials.....	143
Table G 2: X-Ray data for 0% PPC.....	144
Table G 3: X-Ray data for 10% PPC.....	144
Table G 4: X-Ray data for 20% PPC.....	145
Table G 5: X-Ray data for 30% PPC.....	145
Table G 6: X-Ray data for 40% PPC.....	146
Table G 7: X-Ray data for 50% PPC.....	146
Table G 8: X-Ray data for 60% PPC.....	147
Table G 9: X-Ray data for 70% PPC.....	147
Table G 10: X-Ray data for 80% PPC.....	148
Table H 1: Fraction transmitted (FT) & Natural log of FT for 0% PPC.....	149
Table H 2: Fraction transmitted (FT) & Natural log of FT for 10% PPC.....	149
Table H 3: Fraction transmitted (FT) & Natural log of FT for 20% PPC.....	149
Table H 4: Fraction transmitted (FT) & Natural log of FT for 30% PPC.....	149
Table H 5: Fraction transmitted (FT) & Natural log of FT for 40% PPC.....	150
Table H 6: Fraction transmitted (FT) & Natural log of FT for 50% PPC.....	150

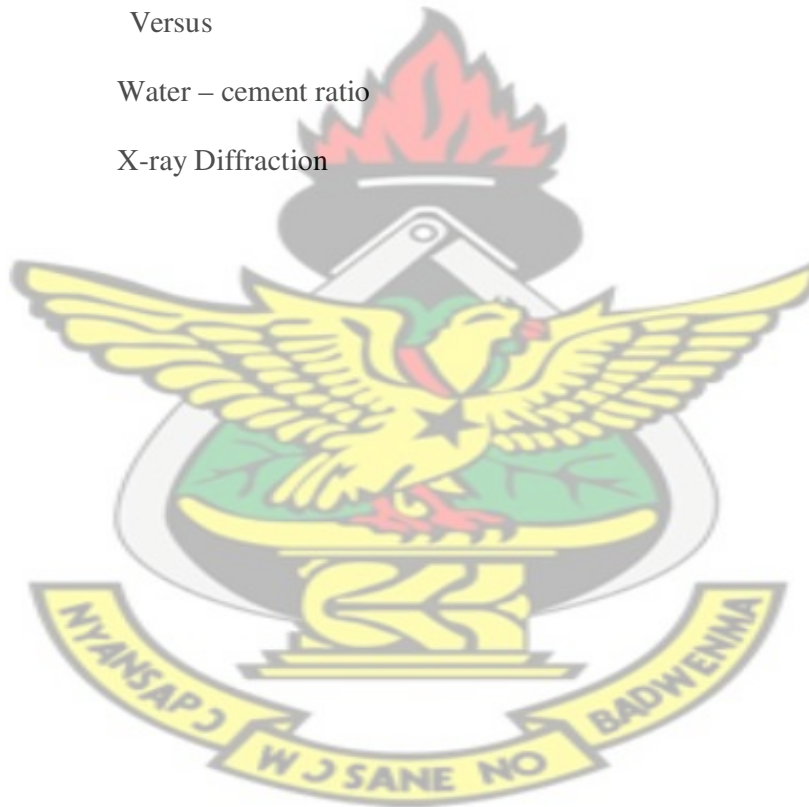
Table H 7: Fraction transmitted (FT) & Natural log of FT for 60% PPC.....	150
Table H 8: Fraction transmitted (FT) & Natural log of FT for 70% PPC.....	150
Table H 9: Fraction transmitted (FT) & Natural log of FT for 80% PPC.....	151
Table H 10: Linear Attenuation Coefficients Determined from the Data.....	151
Table I 1: Peak List for OPC (Control): 0% PPC.....	153
Table I 2: Peak List for 10% PPC.....	154
Table I 3: Peak List for 20% PPC.....	155
Table I 4: Peak List for 30% PPC.....	156
Table I 5: Peak List for 40% PPC.....	157
Table I 6: Peak List for 50% PPC.....	158
Table I 7: Peak List for 60% PPC.....	159
Table I 8: Peak List for 70% PPC.....	160
Table I 9: Peak List for 80% PPC.....	161



LIST OF ABBREVIATIONS

ACI	American Concrete Institute
APF	Atomic Packing Factor
ASTM	American Society for Testing of Materials
Avg.	Average
BCC	Body-centered cubic
BS	British Standard
CA	Coarse aggregate
C.S	Compressive strength
DT	Destructive Testing
FA	Fine aggregate
FCC	Face-centered cubic
Fig	Figure
Figs	Figures
GP	General Purpose Portland cement
GPR	Ground Penetrating Radar
HCP	Hexagonal Close – Packed
HVL	Half Value Layer
IAEA	International Atomic Energy Agency
ISO	International Standards Organization
LOI	Loss on ignition
ln	Natural log
NAA	Neutron Activation Analysis
NDE	Non Destructive Evaluation
NDT	Non Destructive Testing

OPC	Ordinary Portland cement
PPC	Portland Pozzolana cement/concrete
RC	Reinforced concrete
SBEIDCO	Sustainable Built Environment Infrastructures in Developing Countries
SEM	Scanning Electron Microscopy
UPV	Ultrasonic Pulse Velocity
UPVM	Ultrasonic Pulse Velocity Method
UT	Ultrasonic Test
vs.	Versus
W/C	Water – cement ratio
XRD	X-ray Diffraction



ELEMENTS/COMPOUNDS AND SYMBOLS

Al	Aluminum
Ca	Calcium
CaCO ₃	Calcite
Ca(CO ₃)	Calcium Carbonate
Ce	Cerium
Cl	Chlorine
Co	Cobalt
Cr	Chromium
Cs	Cesium
Cu	Copper
Eu	Europium
Fe	Iron
Hf	Hafnium
K	Potassium
La	Lanthanum
Mg	Magnesium
Mn	Manganese
Na	Sodium
Pb	Lead
Sc	Scandium
Si	Silicon
Ti	Titanium
V	Vanadium

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CHAPTER ONE

INTRODUCTION

1.1 Background

Structural integrity of concrete materials for use in construction must be of high quality standards to guarantee safety and reliability. Constant research and development culture ensures materials quality control and assurance. Materials play a major role in speedy transformation of any economy both developed and developing countries. Persistent research into materials for safety, reliability and availability for current and future utilization cannot be over emphasized.

Radiological shielding ability is expected of concrete materials used in construction of containment structures for nuclear facilities and/or that of nuclear waste storage facilities. This is to ensure safe use and storage of nuclear and/or radioactive materials or equipment made thereof. Ionizing radiation facilities such as X-ray and other scanning equipment in the health sector as well as other institutions must be properly housed in radiation proof buildings. Concrete of high shielding capability is thus needed to avoid the incidence of radiation leakage during operation or storage.

1.2 Radiation Shielding and Protection

Radiation protection, sometimes known as radiological protection, is the science and practice of protecting people and the environment from the harmful effects of ionizing radiation. Ionizing radiation is widely used in industry and medicine, and can present a significant health hazard. It causes microscopic damage to living tissue, which can result in skin burns and radiation sickness at high exposures (known as "tissue

effects"), and statistically elevated risks of cancer at low exposures ("stochastic effects"); (Glasstone and Sesonske, 1967).

1.2.1 Need for Radiation Shielding Materials at Low Cost in Ghana

Ghana being in the lower middle-income level status among the committee of nations must be prepared to use her local materials at relatively low cost in resolving the challenges in the energy and housing sector of the economy, as supports from donor partners keep dwindling.

With an expanding economy and a growing population, Ghana's peak demand for electricity is projected to exceed the generating capacity of 3,000 MW and 4,400 MW by 2015 and 2020 respectively. In order to meet the peak energy demand target by 2020, the existing electricity capacity of 2,200 MW needs to be doubled (Nyarko *et al* 2003). Nuclear energy has been identified as one of the energy options that could contribute substantially to meet our energy needs. The containment structures housing these plants must be of concrete of high compressive strength and of high radiological shielding capabilities to prevent any nuclear disaster during operation. Again due to the ever growing population with its concomitant health problems, the health sector has witnessed high demand in the use of ionizing radiation sources in medicine, hence the need for research into cheaper and better local shielding materials.

1.3 Need for Low Cost High Performance Concrete from Local Materials

Mehta and Aitcin (1990) suggested the term High-Performance-Concrete (HPC) for concrete mixtures that possess the following three properties: high-workability, high-strength, and high durability. Durability rather than high strength appears to be the principal characteristic for high-performance concrete mixtures being developed for use in hostile environments such as earthquake prone zones, seafloor tunnels, offshore

and coastal marine structures, and confinement for solid and liquid wastes containing hazardous materials. Strength, dimensional stability, impermeability, and high workability are usually the principal characteristics required of high-performance concrete. Seismic activities have been the major factors considered during design and construction of energy/nuclear power plants. Materials to be used in the construction of such plants must exhibit high level of strength and robustness to avoid any disaster in the event of earthquake.

Currently, it is possible to have an alternative building material to the ordinary Portland cement (OPC), which is cheaper in cost. This material (Pozzolana) when used in combination with Portland cement at a particular ratio can exhibit strength comparable to that of pure Portland cement, thereby reducing cost. Pozzolana was named after Pozzuoli (or Pozzoli), a place where variety of volcanic stuff was found near the Bay of Naples in Rome. Portland cement, on the other hand, was named after the Isle of Portland in England when Engineer Joseph Aspdin patented the product in 1824. Portland cement is a part of the paste in a concrete mixture that helps hold the aggregates together (Kingery *et al*, 1976; Lea, 1970).

A typical concrete mixture consists of Portland cement, water, aggregates, and possibly admixtures. The ratio of water to cement in a mix design helps determine the strength of the concrete. A concrete with a low water to cement ratio will have a higher strength than a mixture with a higher water to cement ratio. Admixtures may be added to the mix design for numerous reasons. Accelerators for the hydration process, retarders that slow the hydration process, air entrainers, plasticizers, superplasticizers, and pigments may be added to the mix design in order to achieve certain desired results or workability of the concrete. In all cases, curing the concrete

in the correct conditions is essential to obtain the desired results. An ideal environment for concrete curing is one in which the concrete is kept hydrated until the process of hydration is complete. Good hydration decreases the permeability of the concrete and ultimately increases the strength of the concrete (McLellan and Shand, 1984).

It is economically advantageous to use a combination of OPC and Pozzolana rather than using only OPC, as mineral admixture for attaining the same or higher strength level. Among the mixes, a combination of 80% OPC and 20% Pozzolana, showed the highest increase in strength (Sogbey, 2011 and Sogbey *et al*, 2012). Using between 20% and 30% PPC mix leads to cost saving and environmental sustainability.

1.3.1 Global Cement Demand and Subsequent Environmental Implications

Each year, the concrete industry produces about 12 billion tonnes of concrete and uses about 2.86 billion tonnes of Portland cement worldwide (Global Cement Report, 2008). In the manufacture of one tonne of cement, approximately 0.8 tonne of CO₂ are released into the atmosphere. About 50% of the CO₂ produced during cement manufacture is due to fossil fuel consumption and the rest due to the calcination of the limestone (Claus and Guimaraes, 2007). The cement industry accounts for 5 – 8% of global CO₂ emission. This makes the cement industry the second largest producer of this greenhouse gas (Scrivener and Kirk, 2007). Also the SO₃ and NO_x released as a result of Portland cement manufacture can cause serious environmental impact such as greenhouse effect and acid rain (Dongxu *et al*, 2000).

It is obvious that minimization of production of Portland cement clinker would greatly help to reduce the CO₂ emission produced by the cement industry. The obvious solution is to promote the use of pozzolanic materials in mortar and concrete works

thereby reducing the world's demand for Portland cement which eventually reduces the emission of CO₂ into the atmosphere.

Work done with clay pozzolanas in Ghana indicates that by replacing approximately 30% by mass of ordinary Portland cement with burnt clay pozzolana with intimate mixing, results in Portland pozzolana cement (PPC) which exhibits compressive strength values good for both load-bearing and non-load-bearing structural applications (Atiemo, 1997, 1998). Pozzolan materials when used in concrete works improve durability which is the ability of concrete to resist weathering action, chemical attack and abrasion. Pozzolan materials also bring in other technical advantages such as low heat of hydration and high ultimate strength. The higher strength of concrete with pozzolanas at later ages is as a result of the pozzolanic reactions increasing the amount of calcium silicate hydrates (C-S-H) while diminishing Ca(OH)₂ (Helmuth, 1987).

1.4 Research Problem Statement

The intense radiation involved in running nuclear power plants, is one of the overriding factors that a country has to address in deciding whether to opt for the nuclear energy or not. One of the most expensive or capital intensive components of nuclear power plants, is construction of the containment structure which is of reinforced concrete design for the purpose of structural and radiation protection.

Again, X-rays, other scanning equipment and radioactive waste in Ghana must be housed or stored under the international law governing nuclear safety and security. Currently the materials used for radiological shielding in our health sector are lead (Pb) and concrete. However, the high cost of lead (Pb) has compelled the sector to often resort to the use of concrete made from OPC which has relatively low shielding

ability. The need for cheaper and better radiological and structural shielding materials should be of paramount interest to us, most especially a nuclear power emerging country such as Ghana, to avoid any possible nuclear disaster in the near future as well as prevent the public from being exposed to ionizing radiations from health centres in the country.

1.4.1 Relevance and Justification

This research project sought to investigate the structural and radiological shielding properties of concrete made from Portland-Pozzolan cement (PPC) at varied ratios of Pozzolana to determine if the properties in both strength and radiation shielding thereof can be used for nuclear power plant containment structure as well as housing other nuclear facilities and X-rays in Ghana and beyond. The industrial use of Pozzolan cement as structural material in both nuclear and construction industries has become economically important most especially, in the face of soaring price of the usual Portland cement used in the construction industry. This calls for a special research into its use as a physical protection material and for other civil engineering structures.

Searching for a binding and/or physical protection material of a cheap source of same qualities as that of the costly Portland cement in the construction industry is the way forward. Consideration, therefore, of Pozzolan cement in Ghana for the construction industry has become economically necessary due to abundance of deposits of Pozzolana in the country Momade and Atiemo (2004) as a source of cheap raw material for the cement industry, as an alternative to the Portland cement. Pozzolana, besides being cheap, is also environmentally friendly. The need to reduce the excessive dependence on imported materials and rather concentrate on the production

and use of local cementitious materials for the construction industry is, therefore, essential.

The research outcome will, therefore, be of paramount importance not only to nuclear industry or the health sector but also to the construction industry on the globe, since the condition for maximum strength of PPC would have been established.

1.5 Objective

The main objective of this research was to investigate the structural and radiological properties of concretes made of Pozzolana - Portland cement (PPC) at varied ratios.

1.5.1 Specific objective

The specific objectives would be to:

- (1) Assess some selected structural characteristics of Pozzolana Portland concrete (PPC) at varied ratios (Compressive & flexural strengths, Elastic modulus).
- (2) Determine the optimum condition for the precipitation of a crystal phase which yields maximum strength.
- (3) Study radiation shielding potential of OPC with Pozzolana by investigating their physical and chemical compositions (Shielding properties and elements/compounds respectively).

1.6 Scope

This research was to experimentally determine the structural and radiological shielding properties of the Portland-Pozzolan concrete (PPC), from materials characterization point of view.

1.7 Thesis Organization

Chapter One introduces the background of the research, the objectives, research problems, its relevance and justification, and scope. Chapter Two highlights literature review on ordinary Portland cement (OPC), Pozzolana cement, concrete, tests; as well as shielding properties of materials. In Chapter Three, the materials and various experimental methods adopted to determine and analyze the physical, chemical, mineralogical, mechanical and engineering properties of blended cements had been discussed. Chapter Four includes experimental results and discussions and Chapter Five, provides the concluding remarks and recommendations.



CHAPTER TWO

LITERATURE REVIEW

2.0 Introduction

This chapter gives some background information on the material characteristics of cement and/or concrete. Shielding characteristics of ordinary Portland cement (OPC), Pozzolana Portland cement (PPC) and that of Pozzolana were reviewed. Methods employed in the structural and radiological analyses of the samples were also reviewed.

2.1 Portland Cement

The ordinary Portland cement (OPC) is clinker, consisting mostly of calcium silicates, obtained by heating to incipient fusion, a predetermined and homogeneous mixture of materials principally containing lime (CaO) and silica (SiO_2) with a smaller proportion of Alumina (Al_2O_3) and iron oxide (Fe_2O_3), (Indian Standards: PPC Specifications, 1993). Cement is defined as finely ground inorganic hydraulic binder which, when mixed with water, forms a paste which sets and hardens by means of hydration reactions and chemical processes and which, after hardening, retains its strength and stability even under water (EN 197-1, 2000). The production of ordinary Portland cement (OPC) involves the calcination of limestone and clay in a rotary kiln to a temperature of about 1400°C . The clinker produced is then inter-ground with gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), which regulates the setting of cement. The flow diagram of the process is shown in Figure 2.1.

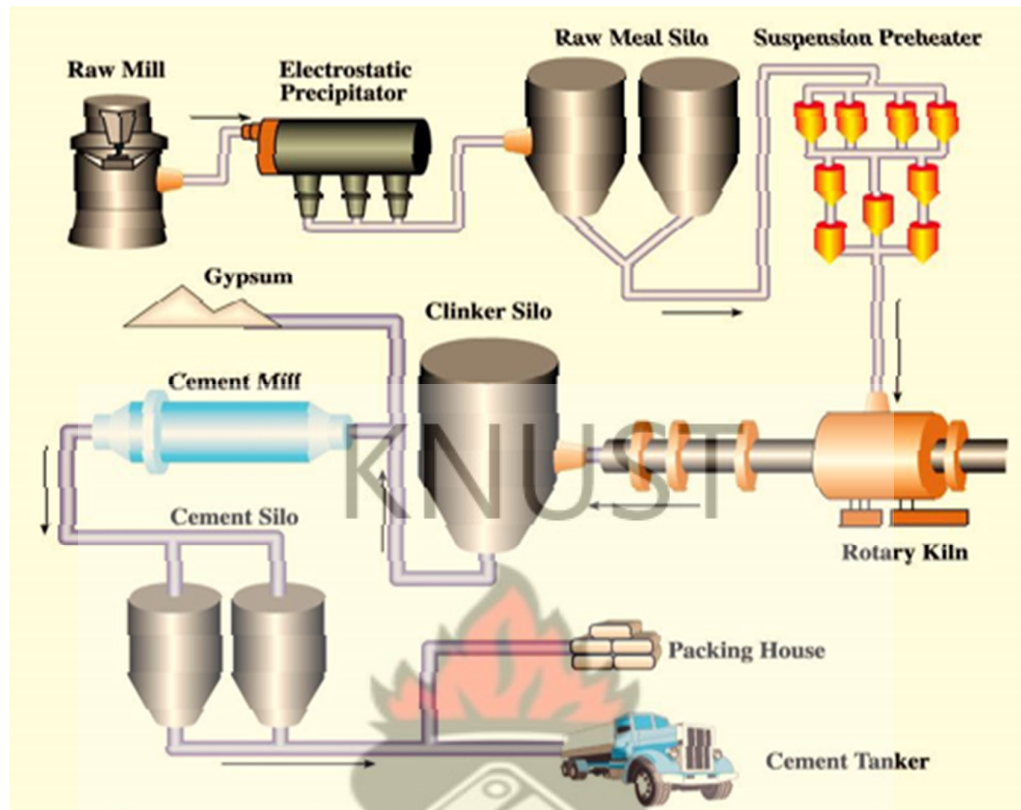


Figure 2. 1: Cement Production Process. (IAEA – AFRA; RAF/1/004, 2013)

2.1.1 Compounds in Portland cement

Portland cement is composed mainly of the four clinker minerals, namely, tricalcium silicate, $3\text{CaO} \cdot \text{SiO}_2$ (C_3S); dicalcium silicate, $2\text{CaO} \cdot \text{SiO}_2$ (C_2S); tricalcium aluminate $3\text{CaO} \cdot \text{Al}_2\text{O}_3$ (C_3A); and tetracalciumaluminoferrite (C_4AF) to which 3-5% gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) is added. Chemically, CaO , SiO_2 , Al_2O_3 and Fe_2O_3 constitute about 80% of Portland cement. The other significant minor oxides are MgO , SO_3 , K_2O , and Na_2O . Although the amounts of these oxides are relatively small, they affect the hydration process of Portland cement (OPC) and the composition of hydration products (Lea, 1970).

Alite (tricalcium silicate) forms the bulk of clinker, 40-70% by mass, with hexagonal crystal sizes up to about $150 \mu\text{m}$. It reacts rapidly with water and is responsible for

much of the early-age (≤ 28 days) strength development. Belite (dicalcium silicate) forms 15-45% of clinker with rounded crystal sizes ranging from 5 to 40 μm . It is less reactive than alite but does contribute to later-age strengths (> 28 days). Upon hydration, both alite and belite form poorly-crystallized calcium silicate hydrates (C-S-H) and well-crystallized calcium hydroxide (portlandite). Tricalcium aluminate constitutes 1-15% of clinker with a particle size of between 1-60 μm and occurs as either cubic or orthorhombic form. It is highly reactive with water. Tetracalciumaluminoferrite (Ferrite; C_4AF) constitutes between 1% and 18% of clinker content (Stutzman, 2004). Typical compositions of phases by mass percent, in Portland cement clinkers are shown in Table 2.1 below. Figure 2.2 outlines typical compositions of phases in cement (ternary diagram). The most essential phase needed for strength is calcium carbonate (CaCO_3). The ternary subsystem C_3A - CaSO_4 - CaCO_3 shown is expected to be applicable for Portland cement pastes. For convenience, CaCO_3 , CaSO_4 , and C_3A are chosen as components rather than CO_2 , SO_3 and Al_2O_3 , (Taylor, 1997).

Table 2.1: Typical compositions of phases in Portland cement clinkers

Phase	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	Mn_2O_3	Fe_2O_3
Alite	0.1	1.1	1	25.2	0.1	0.1	0.1	71.6	0	0	0.7
Belite	0.1	0.5	2.1	31.5	0.1	0.2	0.9	63.5	0.2	0	0.9
Aluminate (Cubic)	1	1.4	31.3	3.7	0	0	0.7	56.6	0.2	0	5.1
Ferrite	0.1	3	21.9	3.6	0	0	0.2	47.5	1.6	0.7	21.4
Aluminate (Orthorhombic)	0.6	1.2	28.9	4.3	0	0	4	53.9	0.5	0	6.6
Aluminate (Low Fe)	0.4	1	33.8	4.6	0	0	0.5	58.1	0.6	0	1
Ferrite (Low Al)	0.4	3.7	16.2	5	0	0.3	0.2	47.8	0.6	1	25.4

(Source: Taylor, 1997)

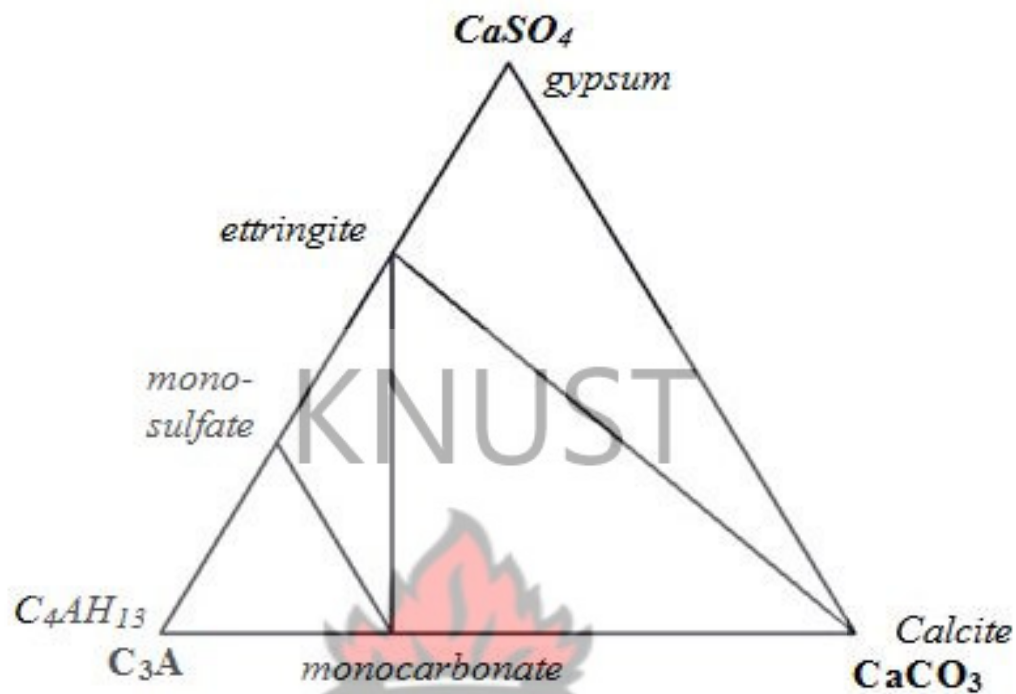


Figure 2.2: Ternary subsystem C_3A - $CaSO_4$ - $CaCO_3$, in Portland cement systems
(Source: Taylor, 1997)

2.1.2 Types of Portland cement

Standards provide some quality restrictions to cement compositions and performance. Different types of cement are used to meet different performance criteria. Properties can be estimated from compositions and fineness. For example, ASTM specifies 5 types of Portland cements as shown in Table 2.2 below, as follows:

Type I — Normal:- Ordinary Portland cement used in most structural applications.

Type II — Moderate Sulfate Resistant:-Used when concrete is exposed to sulfates in groundwater.

Type III — High Early Strength:- Commonly used in prestressed concrete and similar applications.

Type IV — Low Heat:- Typically used in massive constructions such as dams; reduces cracking due to thermal stresses.

Type V — Sulfate Resistant

Table 2.2: Composition and properties of ASTM Types I to V Cements

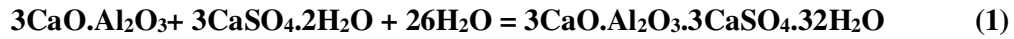
Description	ASTM type				
	I	II	III	IV	V
Type	Normal	Moderate sulfate	High early Strength	Low heat	Sulfate resistant
CS, %	50	45	60	25	40
C ₂ S, %	25	30	15	50	40
C ₃ A, %	12	7	10	5	4
C ₄ AF, %	8	12	8	12	10
Fineness, m ² /kg	350	350	450	300	350
Compressive strength at 1 day,	7	6	14	3	6
Heat of hydration at 7day, J/g	330	250	500	210	250

Source: (Lea, 1970)

2.1.3 Chemical Reactions of Cement

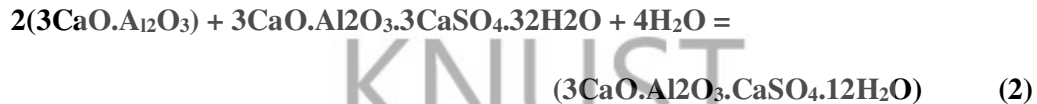
The various particles of cement components react chemically when they mix with water producing various phases. These reactions are by hydration and hydrolysis of these clinker minerals with gypsum leading to the setting and hardening of the cement. The main reactions of the individual cement compounds that take place sequentially are as follows (Lea, 1970; Zhou, 2006):

The C₃A reacts with both water and gypsum (CaSO₄.2H₂O) to produce unstable ettringite (AFt).

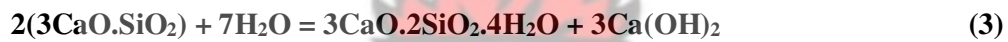


C_3A gypsum water ettringite (AFt)

This reaction is responsible for the setting of cement. Once all the gypsum is used up, the ettringite becomes unstable and reacts with the remaining C_3A to produce stable calcium monosulphate aluminate hydrate crystals (AFm): i.e.

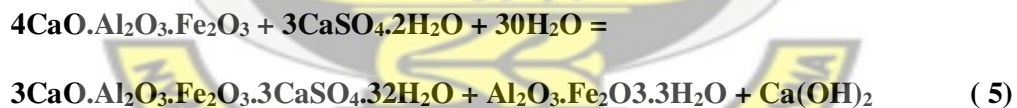


The C_3S (alite) and C_2S (belite) react with water in a complete hydration to produce calcium silicate hydrates and calcium hydroxide as follows:

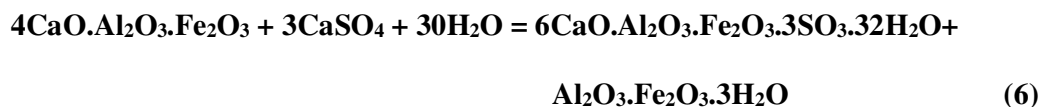


The products of these reactions contribute to the strength of cement products, with C_3S contributing about 75% of it.

Like C_3A , C_4AF (ferrite) reacts with gypsum and water to form ettringite, lime and alumina hydroxides:



The complete compound is called garnet, which is produced by the further reaction of C_4AF with ettringite. Thus;



The reactions above (Eqn.1 – 6) are accompanied by heat liberation. Measuring the liberated heat of hydration is one of the methods of determining the degree of hydration.

2.1.4 Hydration process of Portland cement

The hydration of Portland cement is exothermic. Variation in the liberated heat of hydration mirrors, in one way or another, the hydration mechanisms. The process of hydration can be followed by monitoring the heat evolution by means of conduction calorimetry. Figure 2.3 shows a typical heat evolution curve reported by (Mindess and Yong, 1981). It gives a descriptive narration on the heat evolution. This process is divided into 4 stages, including the pre-induction (i), dormant (ii), acceleration (iii) and post-acceleration (iv) stages.

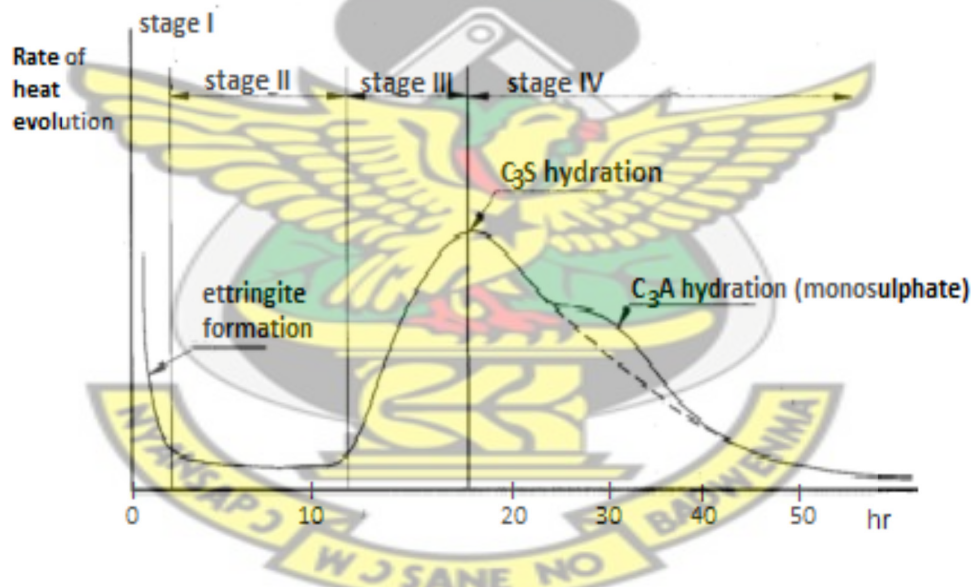


Figure 2.3: Rate of heat evolution (Mindess and Yong, 1981)

2.1.4.1 Pre-induction stage (Stage I)

Almost immediately upon adding water, some of the clinker sulphates and gypsum dissolve, producing an alkaline, sulfate-rich solution. Soon after mixing, the (C_3A) phase - the most reactive of the clinker minerals - reacts with the water to form an aluminate-rich gel (Stage I). The gel reacts with sulfate in solution to form small rod-

like crystals of ettringite (Eqn.1). Hydration of free lime and the wetting of the cement also occur at this stage (Lee, 1983). C_3A hydration is a strongly exothermic reaction but it does not last long, typically only a few minutes. The fast heat evolution at the pre-induction period is attributed to the hydration of C_3A , the hydration of free lime and the wetting of the cement (Eqn.1). C_3A is the most active at this stage and reacts with gypsum to produce ettringite. The main products are ettringite and calcium hydroxide (Lea, 1970; Zhou, 2006). The duration of this stage is less than 60 minutes. This reaction leads to the initial setting time of cement.

2.1.4.2 Dormant stage (Stage II)

The dormant or induction stage follows stage I, and is characterized by a period of about two hours of relatively low heat evolution. Here, cement exhibits low reactivity. The first part of the dormant period – up to perhaps half-way through - corresponds to when concrete can be placed. As the dormant period progresses, the paste becomes too stiff to be workable. The final setting time is achieved within this stage.

Two of the most often mentioned concepts in the dormant stage are the “membrane or protection layer concept” and the concept of “delayed nucleation and growth” (Breugel, 1997).

2.1.4.3 Acceleration stage (Stage III)

At the end of the dormant period, the alite (C_3S) and belite (C_2S) in the cement start to hydrate, with the formation of calcium silicate hydrate and calcium hydroxide (Eqns. 3 and 4). This corresponds to the main period of cement hydration known as the acceleration stage during which time cement product strength increases. The C_3S and belite C_2S cement grains react from the surface inwards, and the anhydrous particles become smaller through a gradually thickening shell (Breugel, 1997). C_3A hydration also continues, as fresh crystals become accessible to water (Eqn. 1). The

period of maximum heat evolution occurs typically between about 10 and 20 hours after mixing and then gradually tails off. In a mix containing Portland cement as the only cementitious material, most of the strength gain occurs within about a month. Ferrite hydration also starts quickly as water is added, but then slows down, probably because a layer of iron hydroxide gel forms, coating the ferrite and acting as a barrier, preventing further reaction (Eqn. 5).

2.1.4.4 Post-acceleration stage (Stage IV)

The rate of heat liberation slows down (stage IV) after peaking in Stage III. The hydration of C_2S becomes the main contribution to this stage (Richardson *et al*, 1989). The main products in this stage are $3CaO.2SiO_2.3H_2O$, $Ca(OH)_2$ and $3CaO.Al_2O_3.CaSO_4.12H_2O$ (AFm).

2.1.5 Thermal Analysis of Portland cement

Cement undergoes phase changes when it is subjected to heat treatment between 20 °C and 1100 °C. The reactions that occur with increased temperature are summarized as follows:

- From 30 °C to 105 °C; surface water begins to evaporate and a part of the bound water escapes. It is generally considered that the evaporable water is completely eliminated at 120 °C.
- Between 110 °C and 170 °C: the decomposition of gypsum (with a double endothermal reaction) and ettringite occurs and the loss of water from part of the carboaluminate hydrates takes place.
- Between 180 °C and 500 °C: the loss of bound water from the decomposition of the C-S-H and carboaluminate hydrates occurs and
- From 450 °C to 550 °C: dehydroxylation of the portlandite (calcium hydroxide) takes place.

- Between 700 °C & 900 °C: decomposition of calcium carbonate; (Lea, 1970, Sharma and Pandey 1999).

2.2 Pozzolana

The Pozzolana is an essentially siliceous material which while in itself possessing little or no cementitious properties will, in finely divided form and in the presence of water, react with calcium hydroxide at ambient temperature to form compounds possessing cementitious properties. The term includes natural volcanic material having pozzolanic properties as well as other natural and artificial materials, such as diatomaceous earth, calcined clay and fly ash (Indian Standards: PPC Specifications, 1993; Grim, 1962).

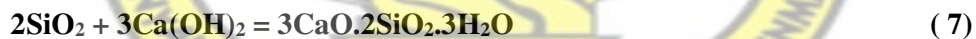
2.2.1 Calcination of Clays

Calcination of clays is an important process in the development of satisfactory pozzolanic properties. The pozzolanic reactivity is induced by the destruction of the clay minerals: kaolinite $[\text{Al}_2(\text{Si}_2\text{O}_5)(\text{OH})_4]$, montmorillonite $[\text{Al}_2(\text{OH})_2\text{Si}_4\text{O}_{10}]\cdot n\text{H}_2\text{O}$, and illite $[\text{K}_2\text{O}_3\text{Al}_2\text{O}_3\cdot 6\text{SiO}_2\cdot 3\text{H}_2\text{O}]$. On heating, clay first loses free water at 100 °C – 150 °C, and from 150 °C to 500 °C it further loses water that is adsorbed on or between lattice planes of the lattice structure. Disintegration of the lattice structure occurs between 550 °C and 650 °C with the liberation of H^+ and OH^- ions producing amorphous aluminous and siliceous compound called meta-kaolinite. All these reactions are endothermic. The rate of loss of water becomes high as the temperature rises to 650 °C. Dehydration is complete at 750 °C (Nutting, 1943; Millienz *et al*, 1949). The structure is completely disrupted between 800 °C – 980 °C. Between 940 °C and 980 °C, an exothermic reaction occurs. Mullite ($2\text{Al}_2\text{O}_3\cdot 3\text{SiO}_2$) appears at around 950 °C for kaolinite, 1050 °C for montmorillonite and 1100 °C for illites. The

optimum calcination temperature for kaolinitic and montmorillonitic clays for maximum pozzolanic reactivity is between 600 °C and 800 °C, and 900 °C for illites (Millienz *et al*, 1949). Kaolinite loses more than 14% of its mass on complete calcination whilst typical montmorillonites and illites lose 5% and 10% of their mass respectively. When the crystalline structure of the clay is ruptured it releases siliceous and aluminous compounds and become very unstable and chemically active.

2.2.2 Clay Pozzolan Action

During hydration of cement, hydrated silicates formed remain stable in contact with lime solution and in the presence of water undergo hydrolysis liberating more lime into the solution (Lea, 1970). The silicate compositions of cement minerals contribute mainly to the structural and strength properties of cement products. They form about 70% of the bulk of cement. The presence of Pozzolana active components reduce the amount of free Ca(OH)_2 in cement products considerably. The unstable but reactive siliceous and aluminous compounds of the calcined clay react chemically with the lime producing mostly hydrated silicates of low solubility as given in a simplified form in Equations below:



The products from the pozzolanic reaction add up to the strength of cement products and enhance water-tightness. Extensive studies as well as X-ray diffraction evidence show that pozzolanic reactions are by direct combination of lime and the pozzolana compounds and not by ion exchange (Lea, 1970). Studies on the effect of alumina in pozzolana proved that the presence of alumina in the pozzolana enhances the strength properties of pozzolana cement, particularly during brief curing (Murakami, 1952).

They react with lime to form compounds which are very complex. The effects of iron oxide in clay pozzolana were also studied by other researchers. Comparing two clay types which were montmorillonite, Gipps and Britton (1960) observed that the red and yellow clays which contained large amounts of free Fe_2O_3 were more pozzolanically reactive than the black cotton soils, which did not have free Fe_2O_3 , even though the black soils contain more clay minerals and have higher silica content.

2.3 Concrete

Concrete is an artificial building material made of the mixture of hydraulic binding material, aggregate and water. Often small amount of various chemicals called admixtures are also added that control such properties as setting time and plasticity (Neville, 1981). There are no special requirements for water. It is important that its quantity should be as small as possible. Aggregates are cheaper than cement and it is, therefore, economical to put into the mix as much of aggregate and as little of cement as possible.

Some applications use concrete alone (i.e. plain concrete), without steel reinforcement, but those applications are few. The combination of two materials, concrete; which is excellent in compression and steel; which is superb in tension, serves to make reinforced concrete an efficient structural material (Neville, 1981).

2.3.1 Process of Concrete Manufacture

The process of concrete manufacture is simply:

Aggregates + Cement + Water + Chemical Admixtures = Concrete

However, the place of manufacture can either be at a construction site as a small batch produced in a portable concrete mixer or at a large batching plant at the construction

site or transported by concrete mixing truck from a concrete plant some distance from the construction site. In the latter case the concrete is called ready mix concrete.

If ready mix concrete is being ordered from a concrete plant the manufacturer needs to know the following:

- Intended use of it (i.e. kerb, slab, etc.)
- Amount required in cubic meters
- Strength required (i.e. Mega Pascals, MPa)
- Maximum size aggregate (i.e. 14 mm, 20 mm, etc.)
- Method of placement (i.e. pump, off the chute, etc.) and any admixtures required.

2.3.2 Aggregates in Concrete

Aggregates are mainly natural, but sometimes artificial in origin. Since at least three quarters of the volume of concrete is occupied by aggregate, its quality is of considerable importance. According to their particle size, they are classified as fine aggregate (< 5 mm) like sand and coarse aggregate (> 5 mm) like gravel.

2.3.3 Curing of Concrete

The term 'curing' is a stage in concrete manufacturing process which includes maintenance of a favorable environment for the continuation of chemical reactions, i.e. retention of moisture within, or supplying moisture to the concrete from an external and protection against extremes of temperature. In order to obtain good concrete it has to be cured during the early stages of hardening. Curing is the procedure used for promoting hydration of cement, and consists of a control of temperature and of moisture movement from and into the concrete. Curing is

necessary because hydration of cement can take place only if the capillaries are filled with water. This is why a loss of water by evaporation from the capillaries must be prevented (Boundy and Hondros, 1964).

In the case of site concrete, active curing stops nearly always before the maximum possible hydration has taken place. This results in loss of strength. Curing may be aided by oiling and wetting the forms before casting; the forms may also be wetted during hardening and after stripping, the concrete should be sprayed and wrapped with polythene sheets or suitable covering. Once the concrete has set, wet curing can be provided by keeping the concrete in contact with a source of water. This may be achieved by spraying or flooding, or by covering the wet concrete with wet sand, earth, sawdust, or straw. The period of curing is usually specified to be minimum seven days (Kingery *et al*, 1976).

2.3.4 Compaction

Compaction means the elimination of entrapped air from the concrete until it has achieved as close a configuration as possible for a given mix. The strength of concrete of given mix proportions, is seriously affected by the degree of its compaction.

Green concrete has all the three phases – solids, water and air. In order to make the concrete impervious and attain its maximum strength, it is required to remove the entrapped air from the concrete mass when it is still in plastic state. If the air is not removed completely, the concrete loses strength considerably. It has been noted that 5% voids reduce the strength by about 30%, and 10% voids reduce the strength by over 50%. Compaction eliminates air bubbles and brings enough fine material both to the surface and against the forms to produce the desired finish. One can use such hand tools as steel rods, paddling sticks, or tampers, but mechanical vibrators are best. Any

compacting device must reach the bottom of the form and be small enough to pass between reinforcing bars. Since the strength of the concrete member depends on proper reinforcement location, one must be careful not to displace the reinforcing steel (Boundy and Hondros, 1964).

2.3.5 Setting

Initial set is when the cement paste loses its plasticity and stiffens considerably. Final set is the point when the paste hardens and can sustain some minor load. Both are arbitrary points and these are determined by Vicar Needle penetration resistance.

Slow or fast setting normally depends on the nature of cement. It could also be due to extraneous factors not related to the cement. The ambient conditions play an important role. In hot weather, the setting is faster, in cold weather, setting is delayed. Some types of salts, chemicals, clay, etc if inadvertently get mixed with the sand, aggregate and water could accelerate or delay the setting of concrete (Boundy and Hondros, 1964).

2.3.6 The Different Grades of Ordinary Portland Cement

The Bureau of Indian Standards (BIS) has classified OPC in three different grades. The classification is mainly based on the compressive strength of cement-sand mortar cubes of face area 50 cm^2 composed of 1 part of cement to 3 parts of standard sand by weight with a water-cement ratio arrived at by a specified procedure. The grades are grade 33, grade 43 and grade 53. The grade number indicates the minimum compressive strength of cement sand mortar in N/mm^2 at 28 days (Indian Standards: PPC Specifications, 1993).

2.4 Portland-Pozzolanic Cement

This composite material is an intimately inter-ground mixture of Portland clinker and Pozzolana with the possible addition of gypsum (natural or chemical) or an intimate and uniform blending of Portland cement and fine Pozzolana (Indian Standards: PPC Specifications, 1993). Table 2.3 below shows the chemical composition of Pozzolana and Portland cement.

Table 2.3: Chemical Composition of Portland and Pozzolana Cement (% wt)

Compound	Pozzolana	Portland
SiO ₂	46.25	27.43
Al ₂ O ₃	17.34	5.40
Fe ₂ O ₃	10.26	3.48
CaO	10.18	53.71
MgO	2.90	1.41
K ₂ O	1.64	0.92
Na ₂ O	3.64	0.16
SO ₃	0.80	2.59
Cl ⁻	0.01	0.004

Source: (SBEIDCO, 2009)

2.4.1 Effects of Pozzolana on OPC

Pozzolana combines with lime and alkali in cement when water is added and forms compounds Neville and Books (1990) which contribute to:

1. High strength.
2. Sulphate resistance (sulphate affects durability of concrete)

3. Good workability, reduced bleeding and control of destructive expansion from alkali-aggregate reaction.
4. Reduced heat of hydration thereby controlling temperature differentials, which causes thermal strain and resultant cracking in mass concrete structures like dams making an enviable good material for nuclear reactor containment structure.

2.5 Structural or Mechanical Property Analysis of PPC

Research on the pozzolanic activity of some clay samples in Ghana for housing construction has been carried out in Ghana (Atiemo, 2005; Momade and Atiemo, 2004). Some of the results are presented in Table 2.4. The studies showed that pozzolana cements absorb more water in the preparation of the paste as compared to ordinary Portland cement and the water demand increased in all cases as the replacement of cement was increased. However, the setting times (initial and final) of the pozzolana cements were lower than that of ordinary Portland cement and decrease with increasing amount of pozzolana.

The results also showed that the 28-day compressive strengths of the pozzolana cement mortars up to 30% pozzolana content satisfied the class 32.5 cement as recommended by EN 197-1 (2000) for concrete works and general construction. The Mfensi samples gave the highest strength values in all cases. Generally, the strengths reduce with increasing amount of pozzolana. The study concluded that the clay pozzolana cements are suitable for both concrete and general construction with 25 - 30% clay pozzolana content.

Wild *et al* (1997) also reported on pozzolanic properties of selected burnt brick samples produced from clays from four European countries. Clay bricks are burnt at

between 800 °C and 900 °C. The bricks were ground in a ball mill and the brick powder samples were used as partial cement replacement materials (at replacement levels of 10%, 20%, 30% by mass) to prepare mortars. The water/binder (w/b) ratio of 0.4 and the sand to binder (cement plus ground brick) ratio was kept constant at 2.5:1. The average values of compressive strength together with the standard deviations are reported in Table 2.5. The values obtained were comparable to that of OPC (control), (Momade and Atiemo, 2004; Wild *et al*, 1997).

Table 2.4: Mechanical properties of pozzolana cement mortar

Sample	Cement Content, %	Pozzolana Content, %	Water, Demand, %	Setting time, min		Water Absorption, %	Compressive strength, N/mm ²		
				Initial	Final		3 days	7 days	28 days
Control	100	0	36.9	108	275	2.1	24.9	36.8	40.2
Bibiani	80	20	37.5	98	265	2.3	21.8	35.2	38.8
	75	25	39.0	92	225	2.1	19.5	31.8	35.6
	70	30	-	-	-	2.1	16.9	25.3	33.5
	60	40	-	-	-	1.85	12.6	22.6	27.1
Mfensi	80	20	38.2	102	250	2.2	23.6	35.5	39.8
	75	25	39.6	90	210	2.2	22.6	33.3	36.8
	70	30	-	-	-	2.0	21.3	27.8	34.1
	60	40	-	-	-	1.8	16.8	23.5	28.8
Wenchi	80	20	38.7	86	241	2.5	22.8	34.1	44.3
	75	25	40.1	75	194	2.3	20.4	31.4	40.8
	70	30	-	-	-	2.2	18.9	28.4	36.8
	60	40	-	-	-	2.4	16.6	24.8	31.3

(Momade and Atiemo, 2004)

Table 2.5: Average values of compressive strength for mortar mixes

Sample	Pozzolana Content, %	Compressive strength, N/mm ²		
		7 days	28 days	90 days
Control	0	51.5 ± 2.5	60.5 ± 2.9	65.6 ± 1.6
Brick B	10	48.3 ± 2.02	61.6 ± 2.5	66.3 ± 3.8
	20	42.8 ± 4.2	58.3 ± 3.6	67.5 ± 2.1
	30	38.4 ± 2.5	56.0 ± 1.3	64.9 ± 1.9
Brick D	10	48.5 ± 1.2	58.0 ± 2.8	64.1 ± 2.5
	20	48.0 ± 1.3	54.6 ± 0.9	63.9 ± 2.9
	30	42.3 ± 0.6	51.8 ± 1.6	59.2 ± 1.9
Brick L	10	47.5 ± 3.4	56.9 ± 2.0	60.4 ± 1.8
	20	43.4 ± 3.5	51.8 ± 4.5	55.6 ± 2.9
	30	38.7 ± 1.6	50.0 ± 3.7	49.5 ± 2.0
Brick P	10	51.3 ± 1.7	64.7 ± 3.5	67.8 ± 3.0
	20	47.5 ± 1.1	57.8 ± 3.5	64.1 ± 1.2
	30	37.7 ± 2.9	56.1 ± 3.1	60.8 ± 1.2

(Wild *et al*, 1997)

2.5.1 Fineness of Cement and Its Effect on Strength

Fineness defines the surface area of cement particles present in per unit weight, which implies that more fineness means more particles in unit weight. This enhances the reaction rate which in turn will result in faster gain of strength at earlier stages as well as liberates higher heat, therefore proper curing in initial days is very essential. The fineness of cement has a major influence on the rate of strength development and heat evolution in concrete. The fineness of cement is specified in terms of its surface area. Traditionally, the Blaine apparatus is used to determine it. The Blaine apparatus measures the rate at which air can flow through a porous bed of cement, which is proportional to the specific surface area of cement. It does not give the true specific surface area, but it is a fast and cheap test for quality control and enables comparison of

different cements. There are advanced methods to determine the true specific surface area based on adsorption measurements with some gases. These tests require very expensive, sophisticated equipment (Kingery *et al*, 1976). Below are some specifications (Table 2.6).

Table 2.6: Some standard specifications of Portland cement from different countries

Standard	British	EU	India	New Zealand	ASTM
Type of Cement	OPC	CEM I		GP	Type I
Fineness, cm ² /g	2250				Min. 2600 Avg. 2800
Compressive strength, N/mm ² at 7 days at 28 days	29	≥ 16 32.5 – 52.5	22 —	25 40	55
Setting time Initial minute Final hour	Min. 45 Max. 10	>75		45 10	45 Max. 8
Chemical composition MgO, % SO ₄ ⁻² as SO ₃ , % LOI, %	Max. 4.0 Max. 3.0 Max. 3		Max. 6.0 Max. 2.75 Max. 4	Max. 4.5 Max. 3.5	Max. 5.0 Max. 3.0

(Kingery *et al*, 1976)

2.5.2 Factors Responsible for Governing Compressive Strength of Concrete

The compressive strength is governed by the following factors:

The typical mix proportions of cement, sand, aggregate and water-to-cement which produce concrete of 15 MPa, 20 MPa and 25 MPa strength is given in Table 2.7 (ACI, 1996). Typical strengths range from 15 MPa to 50 MPa. For instance, for simple driveways, footpaths and other domestic work, typical strengths are in the order of 15 MPa to 25 MPa. The structural strength of concrete required for bridge deck slabs should be about 32 MPa, columns and superstructures 40 or 50 MPa and concrete pavements about 30 MPa.

Table 2.7 Typical Mix Proportions (Cement: Sand: Aggregate and w/c ratio)

Mix Proportions				
Strength	Cement	Sand	Aggregate	Water/Cement Ratio
15 MPa	1	2.5	4	0.60
20 MPa	1	2	3	0.55
25 MPa	1	2	2.5	0.50

Source: (IAEA, Vienna, 2002)

2.6 Radiation Shielding Characteristics of OPC

Many man-made sources of ionizing radiation abound in our technological age in addition to natural sources. These ionizing radiations have found applications in many areas including medicine, industry, research, and agriculture (Mann et al, 1998). In all these areas of applications, the use of ionizing radiation sources has been found to produce benefits but not without some detriment associated with their use. Applications in medicine have been reported as the largest contributor to population dose (Miah *et al*, 1998; Osibote *et al*, 2007). In order to minimize the detriments, it has been recommended that the use of ionizing radiation should be such that radiation dose to workers, the public, and patients are as low as reasonably achievable (IAEA-IIS, 1996).

Three ways by which exposure to people are reduced include: locating the facilities away from areas where people can easily access them, minimizing the time people spend near the facilities, and shielding the facilities. The most-used and effective way is shielding, especially in hospitals where space is limited. Shielding of rooms housing radiation facilities in hospitals or diagnostic centers is usually done using concrete or gypsum board lined with lead. Concrete is a mixture of sand, cement, and gravel. The ability of concrete to shield ionizing radiations is determined by its mass

(or linear) attenuation coefficient. The higher the mass attenuation coefficients, the more efficient concrete will be in shielding ionizing radiations. The attenuation coefficient of a concrete material depends on the attenuation coefficients of its constituents. Previous reports have assessed the shielding effectiveness by measurement of mass attenuation coefficients (MACs) of soil, cement, gravel, and limestone (Salinas *et al*, 2006; Awadallah and Imran, 2007). Table 2.8 shows comparison of mass attenuation coefficients for sampled soil and sand in different countries and Table 2.9 shows, measured values of linear attenuation coefficients (cm^{-1}) for different concrete constituents.

Table 2.8: Comparison of mass attenuation coefficients for sampled soil and sand in different countries

MATERIAL	Attenuation 6.661 keV	Coefficient for 1173.2 keV	1332.5 keV	Country
Limestone	0.060±0.002	0.042±0.002	0.040±0.002	Bangladesh
	0.086±0.002	0.068±0.005	0.055±0.001	Jordan
	0.064±0.001	0.043±0.002	0.040±0.000	Nigeria
Beach Soil	0.076±0.002	0.056±0.002	0.052±0.002	Bangladesh
	0.080±0.002	—	0.058±0.001	Brasil
	0.078±0.009	0.058±0.001	0.054±0.001	Nigeria
Ordinary Sand	0.077±0.002	0.058±0.002	0.054±0.002	Bangladesh
	0.077±0.002	0.056±0.001	0.053±0.001	Nigeria
River Soil	0.070±0.002	0.046±0.002	0.049±0.002	Bangladesh
	0.067±0.004	0.049±0.0002	0.045±0.004	Nigeria
Hill Soil	0.075±0.002	0.055±0.001	0.050±0.001	Bangladesh
	0.070±0.001	0.056±0.003	0.052±0.003	Nigeria
Land Soil	0.072±0.002	0.053±0.001	0.048±0.001	Bangladesh
	0.069±0.005	0.049±0.001	0.044±0.003	Nigeria
Cement	0.071±0.002	0.050±0.002	0.047±0.002	Bangladesh
	0.071±0.002	0.051±0.003	0.047±0.003	Nigeria

(Salinas *et al* 2006)

In Table 2.8, it could be noticed that there are differences in values for different countries for same material. This is because the attenuation coefficient of a material depends on the attenuation coefficients of its constituent elements (mineral composition) which differ slightly from country to country.

Table 2.9: Measured values of linear attenuation coefficients (cm^{-1}) for different Concrete Constituents

Energy (keV)	Limestone $\rho=0.94\text{g/cm}^3$	Beach soil 1.44g/cm^3	Ordinary sand 1.35g/cm^3	River soil 1.18g/cm^3	Hill soil 1.35g/cm^3	Land soil 1.11g/cm^3	cement 1.27g/cm^3
60	0.103	0.253	0.201	0.157	0.186	0.138	0.170
661.6	0.060	0.112	0.104	0.079	0.102	0.074	0.089
1173.2	0.040	0.084	0.076	0.058	0.076	0.054	0.065
1274.5	0.039	0.081	0.074	0.056	0.073	0.050	0.064
1332.5	0.038	0.078	0.072	0.053	0.070	0.049	0.059

(Salinas *et al* 2006)

Thus, radiation shielding characteristics of Pozzolana cement, aside structural properties' study on the contribution of Pozzolana to strength improvement of the ordinary Portland cement (OPC), very little research on shielding ability of Pozzolana has been conducted comparable with that of OPC.

2.6.2 Radiological Shielding Property Analysis of Matter (Concrete)

Radiological Shielding property of matter/material is the material's ability to absorb/attenuate ionizing radiation when passing through it. And it is based on the material's chemical and/or elemental composition. In other words, elemental/chemical composition of a material forms the basis for its radiological shielding property characteristics. The interactions of alpha and beta radiation are governed by deterministic processes and therefore alphas and betas have a finite range and can be completely stopped. X-ray/Gamma (photon) interactions, on the other hand, are stochastic or probabilistic events.

2.6.2.1 Attenuation Coefficient

Since the interactions are governed by laws of probability, a neutron or x/gamma-ray (photon) beam does not have a finite range; it can only be reduced in intensity by increasingly thicker absorbers (shield). The fractional reduction in intensity per unit thickness of absorber is called the attenuation coefficient, while the fractional absorption of energy from the beam per unit thickness of absorber is called the absorption coefficient of the absorbing material. Both coefficients are functions of the photon energy and the absorber material (Cember, 2009).

Energy transfer from a radiation field to the absorbing medium is the basis for all types of radiation effects. Charged particles, including betas, positrons, protons, and alphas excite or ionize the atoms in the absorbing media by colliding with their extranuclear electrons. These primary ionizing particles lose a finite amount of energy in each collision. The average loss per ionization in air or in soft tissue is 34 eV for betas and 35.5 eV for alphas. The linear density of the ions thus produced is called the specific ionization and is a measure of the rate of energy loss by the primary ionizing

particle. Beta particles have a relatively low specific ionization of the order of 100 ip/cm in air. Alphas, because of their double charge and their relatively slow speed, have a high specific ionization, producing of the order of tens of thousands ion pairs per centimeter in air. Successive collisions ultimately lead to the expenditure of the entire kinetic energy of the primary ionizing particle. The distance traveled in the absorber until this point is reached is called the range of the ionizing particle, and it is determined by the energy of the primary ionizing particle and by the nature of the absorber. If one measures the distance in terms of the density thickness, one would find that the range is approximately independent of the nature of the absorber (Cember, 2009).

2.6.2.2 Bremsstrahlung

Primary ionizing particles can also interact with the nuclei of the absorbing media. This interaction is especially important in the case of high-energy betas and electrons. When this happens, some of the electron's kinetic energy is converted into electromagnetic energy that is radiated as X-ray photons called bremsstrahlung.

The production of bremsstrahlung is enhanced by high-atomic-numbered absorbing media. The interaction of X-rays and gamma radiation with matter differs qualitatively from that of alpha and beta particles. Although gamma rays are called ionizing radiations, they are indirectly ionizing. The photons of electromagnetic radiation interact with the extra nuclear electrons in absorbing media by knocking out electrons by one of two different mechanisms. The mechanisms are Compton Scattering, Photoelectric Absorption and Pair Production.

In Compton Scattering, an outer electron is ejected and the photon, now reduced in energy by the energy imparted to the electron, is scattered. The ejected electron is

called a Compton electron. In the other mechanism, called photoelectric absorption, the photon knocks out a tightly bound electron, all the photon's energy is transferred to the absorbing atom, and the photon disappears. Some of the photon's energy is used in freeing the tightly bound electron from the atom and the rest of the photon's energy is converted into kinetic energy of the ejected photoelectron. If the energy of the photon exceeds 1.02 MeV, another interaction mechanism, called pair production, can occur. In pair production, the photon interacts with the nucleus of the absorbing atom and all of its energy is converted into mass through the production of a positron and a negative electron.

The 1.02-MeV threshold energy required for pair production represents the energy equivalent of the mass of the two newly created particles. Any energy that the photon may have in excess of 1.02 MeV is transferred to the pair of particles as kinetic energy. The Compton electrons and photoelectrons as well as the electron and positron pair become primary ionizing particles and proceed to lose their energy by ionization and excitation in the media in which they were produced. They are the agents through which energy is transferred from the X-ray or gamma field to the absorbing medium. When the positron loses all its kinetic energy, it combines with a negative electron. The two particles are annihilated and two photons, called annihilation radiation, of 0.51 MeV are created (Cember, 2009). Below are typical linear attenuation curves for some materials (Figure 2.4).

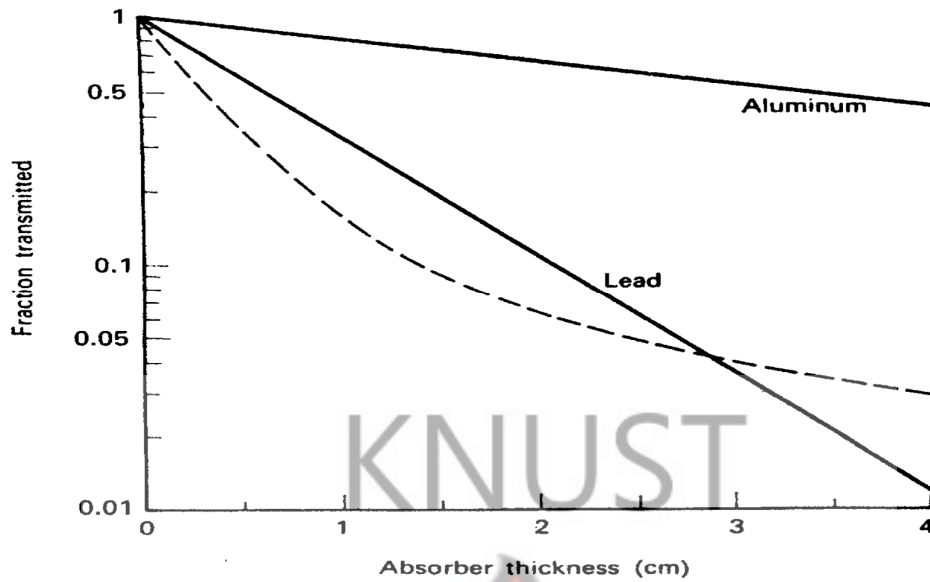


Figure 2.4: Linear Attenuation of gamma rays under conditions of good geometry

The solid lines are the attenuation curves for 0.662-MeV (monoenergetic) gamma rays. The dotted line is the attenuation curve for a heterochromatic beam (Cember, 2009).

The equation of the straight line in Figure 4 is

$$\ln I = -\mu t + \ln I_0 \quad (9)$$

Or

$$\ln\left(\frac{I}{I_0}\right) = -\mu t \quad (10)$$

Taking the inverse logs of both sides of Eq. (10), the following expression was obtained,

$$\frac{I}{I_0} = e^{-\mu t} \quad (11)$$

where

I_0 = gamma-ray intensity at zero absorber thickness,

t = absorber thickness,

I = gamma-ray intensity transmitted through an absorber of thickness t ,

e = base of the natural logarithm system, and

μ = slope of the absorption curve = the linear attenuation coefficient.

Since the exponent in an exponential equation must be dimensionless, μ and t must be in reciprocal dimensions, that is, if the absorber thickness is measured in centimeters, then the attenuation coefficient is called the linear attenuation coefficient, μ_l , and it must have dimensions of “per cm.” If t is in g/cm^2 , then the absorption coefficient is called the mass attenuation coefficient, μ_m , and it must have dimensions of per g/cm^2 or cm^2/g . The numerical relationship between μ_l and μ_m , for a material whose density is $\rho \text{ g}/\text{cm}^3$, is given by equation:

$$\frac{\mu_l}{\rho} \left[\frac{\text{cm}^{-1}}{\text{g cm}^{-3}} \right] = \mu_m \left[\frac{\text{cm}^2}{\text{g}} \right] \quad (12)$$

The attenuation coefficient is defined as the fractional decrease, or attenuation of the gamma-ray beam intensity per unit thickness of absorber, as defined by the equation

(13):

Limit

$$-t \rightarrow 0 \quad \frac{-I/I}{-t} = \mu, \quad (13)$$

where $-I/I$ is the fraction of the gamma-ray beam attenuated by an absorber of thickness $-t$. The attenuation coefficient thus defined is sometimes called the total attenuation coefficient. Values of the attenuation coefficients for some materials are given in Table 2.10.

Table 2.10: Total Mass and Linear Attenuation Coefficients for Some Materials at 0.1 MeV, gamma energy

	Al	Fe	Ca	K	Na	Pb	H ₂ O	Concrete
$\frac{\mu}{\rho}$ [cm ² /g]	0.161	0.344	0.238	0.215	0.151	2.29	0.167	0.169
μ [cm ⁻¹]	0.435	2.718	0.369	0.187	0.147	59.94	0.167	0.389
ρ [g/cm ³]	2.7	7.90	1.55	0.87	0.971	11.35	1	2.3

Source: (Cember, 2009)

2.6.2.3 Thermal and Fast Neutrons

In the context of interaction with matter, neutrons are classified according to their kinetic energy as thermal (slow) and fast. Neutrons are produced through nuclear reactions and by nuclear fission. All neutrons have kinetic energy when they are produced and hence may be considered to be fast. These fast neutrons lose energy by colliding elastically with atoms in their path, and then, after being slowed to thermal energy, they are captured by nuclei in the absorbing medium. Many nonradioactive isotopes become radioactive after capturing a neutron. When a hydrogen nucleus is struck by a fast neutron, the nucleus is knocked out of the atom and becomes a proton, which is a positively charged, high specific ionization primary ionizing particle. It loses its kinetic energy by ionization and excitation interactions with the absorber atoms.

2.6.2.4 Microscopic Cross Sections, σ [barns or cm²]

The microscopic cross section of a nucleus is used to characterize the probability that a nuclear reaction will occur. It is also known as nuclear cross section. The concept of a nuclear cross section can be quantified physically in terms of "characteristic area"

where a larger area means a larger probability of interaction. The standard unit for measuring a nuclear cross section (denoted as σ) is the barn, which is equal to 10^{-28} m² or 10^{-24} cm². Cross sections can be measured for all possible interaction processes together, in which case they are called total cross sections, or for specific processes, distinguishing elastic scattering and inelastic scattering; of the latter, amongst neutron cross sections the absorption cross sections are of particular interest. Types of reactions frequently encountered are s: scattering, γ : radiative capture, a: absorption (radiative capture belongs to this type), f: fission, the corresponding notation for cross-sections being: σ_s , σ_γ , σ_a , etc. A special case is the total cross-section σ_t , which gives the probability of a neutron to undergo any sort of reaction;

$$\sigma_t = \sigma_s + \sigma_\gamma + \sigma_f + \dots \quad (14)$$

In nuclear physics, it is conventional to consider the impinging particles as point particles having negligible diameter. Cross sections can be computed for any sort of process, such as capture scattering, production of neutrons, etc. In many cases, the number of particles emitted or scattered in nuclear processes is not measured directly; one merely measures the attenuation produced in a parallel beam of incident particles by the interposition of a known thickness of a particular material. The cross section obtained in this way is called the total cross section and is usually denoted by a σ_t or σ_T .

The typical nuclear radius is of the order of 10^{-12} cm. It might therefore, be expected that the cross sections for nuclear reactions are to be of the order of πr^2 or roughly 10^{-24} cm². Actually the observed cross sections vary enormously. Thus, for a slow neutron (n) absorbed by the (n, gamma) reaction the cross section in some cases is as much as 1,000 barns, while the cross sections for transmutations by gamma-ray absorption are in the neighborhood of 0.001 barns (Perkins, 1999).

2.6.2.5 Macroscopic Cross Section, Σ [cm^{-1}]

Macroscopic cross sections are used in determining the nuclear reaction rate, and are governed by the reaction rate equation for a particular set of particles. The interaction is usually viewed as a "beam and target" though we have experiments where one particle or nucleus can be the "target" [typically at rest] and the other is treated as a "beam" [projectile with a given energy]. For neutron interactions incident upon a thin sheet of material (ideally made of a single type of isotope), the nuclear reaction rate equation is written as:

$$r_x = \Phi \sigma_x \rho_A = \Phi \Sigma \quad (15)$$

(Eqn. (15) shows the relationship between σ/cm^2 and Σ/cm^{-1})

Or

$$\Phi(x) = \Phi_0 e^{-\Sigma_T X} \quad (16)$$

where:

- r_x : number of reactions of type x, units: [1/time/volume]
- Φ : neutron beam flux intensity through an absorber of thickness x, [1/area/time]
- Φ_0 = neutron beam flux intensity at zero absorber thickness
- σ_x : microscopic cross section for reaction x, units: [area] (usually barns or cm^2)
- ρ_A : density of atoms in the target in units of [1/volume]
- $\Sigma_T = \Sigma_s + \Sigma_a \equiv \sigma_x \rho_A$: total macroscopic cross-section [1/length]
- Σ_s = macroscopic cross-section for scattering
- Σ_a = macroscopic cross-section for absorption
- e = base of the natural logarithm system
- x = thickness of materials

Formally equation (15) defines the macroscopic neutron cross-section (for reaction x) as the proportionality constant between a neutron flux incident on a (thin) piece of material and the number of reactions that occur (per unit volume) in that material. The distinction between macroscopic and microscopic cross-section is that the former is a property of a specific lump of material (with its density), while the latter is an intrinsic property of a type of nuclei. Table 2.11 shows some elements with their total macroscopic cross sections.

Table 2.11: Elements or Compounds & Their Macroscopic Cross Sections for neutrons

Elements or Compound	Density [g/cm^3]	Macroscopic Cross Section, Σ [cm^{-1}]
H ₂ O	1	3.472
Co	8.9	4.097
Hf	13.3	5.069
Sc	2.5	1.608
Mn	7.2	1.221
Pb	11.35	0.369
Fe	7.9	1.155
Eu	5.22	89.166
Concrete	2.3	0.240

Source: (Glasstone and Sesonske, 1967).

2.7 Compression Test

Concrete mixtures can be designed to provide a wide range of mechanical and durability properties to meet the design requirements of a structure. The compressive strength of concrete is the most common performance measure used by the engineer in designing buildings and other structures (BS 1881, Part 116, 1983). The compressive strength is measured by breaking cylindrical or cubic concrete specimens in a compression testing machine as shown in Figures 2.5 and 2.6 (Bungey, 1996). In this work compressive strength was calculated from the failure load divided by the cross-sectional area resisting the load and reported in units of pound-force per square

inch (psi) in US Customary units or mega Pascal (MPa) in SI units. Concrete compressive strength requirements can vary from 2500 psi (17 MPa) for residential concrete to 4000 psi (28 MPa) and higher in commercial structures. Higher strengths up to and exceeding 10,000 psi (70 MPa) are specified for certain applications (ACI, 1996).



Figure 2.5: Fractured Test Specimen at Failure

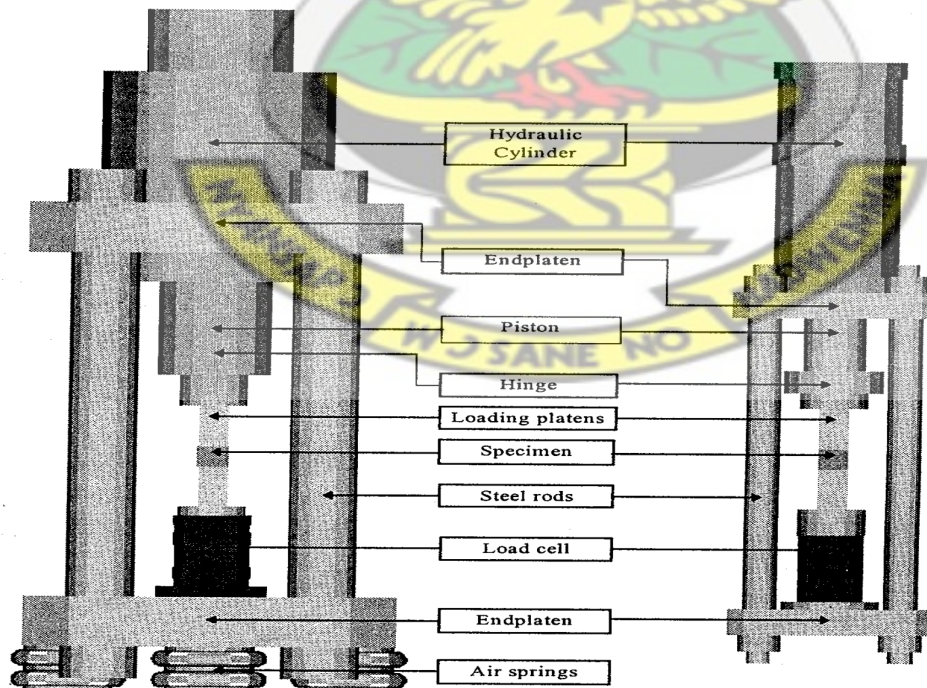


Figure 2.6: Layout of a single loading axis. Left: 5000 kN axis, right: 2000 kN axis (Bungey, 1996).

2.7.1 Compressive Strength

Compressive Strength is the most important parameter of cement. There are differences in the standards in the size of test pieces, the quality of sand, the cement: sand ratio and the amount of water (w/c ratio) used in the preparation of test pieces, the kind of compaction employed, and other mixing related activities. Compressive strength formula is given as follows:

$$\text{Compressive stress } (\delta) = \frac{\text{Force (F)}}{\text{Area (A)}} = \frac{N}{\text{mm}^2} \text{ [MPa]} \quad (17)$$

2.8 Flexural Test

Flexural testing machine was used to measure the mechanical behavior of the concrete subjected to simple beam or cylindrical loading. It is also called a transverse beam test. The flexure test produced tensile stress in the convex side of the specimen and compression stress in the concave side. This created an area of shear stress along the midline. To ensure the primary failure came from tensile or compression stress the shear stress was minimized. This was done by controlling the span to depth ratio; the length of the outer span divided by the height (depth) of the specimen. For most materials $S/d=16$ is acceptable. Some materials require $S/d=32$ to 64 to keep the shear stress low enough (Stanton and Bloem, 1957, Timoshenko, 1998).

2.8.1 Types of Flexural Test

There are two test types; 3-point flex and 4-point flex. In a 3-point test the area of uniform stress is quite small and concentrated under the center loading point. In a 4-point test, the area of uniform stress exists between the inner span loading points (typically half the outer span length), (Stanton and Bloem, 1957, Timoshenko, 1998). The 3-point flex type had been employed in this project. Figures 2.7 and 2.8 show typical 3-point flexure testing machines.

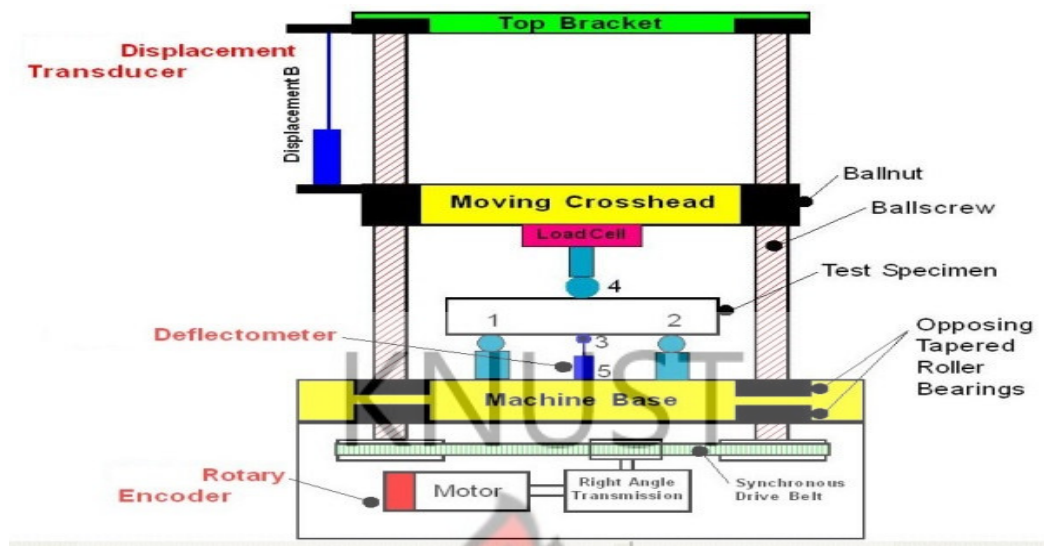


Figure 2.7: Schematic diagram: Flexural Test machine



Figure 2.8: Flexural Test Machine Loaded (Centre-point Loading)

2.8.2 Flexural Test Formulae

The following formulae were used in the result computation.

$$\text{Flexural strength } (\delta_f) = \frac{PL}{\pi R^3} ; \quad \text{for a circular cross section} \quad (18)$$

$$\text{Flexural strength } (\delta_f) = \frac{3PL}{2bd^2} \quad ; \quad \text{for rectangular cross section} \quad (19)$$

$$\text{Flexural strain } (\varepsilon_f) = \frac{6Dd}{L^2} \quad (20)$$

$$\text{Flexural modulus of elasticity } (E_f) = \frac{L^3m}{4bd^3} \quad (21)$$

In these formulae above, the following parameters are used:

- R = radius of cylindrical concrete (mm)
- σ_f = Stress in outer fibers at midpoint [Modulus of Rupture], (MPa)
- ε_f = Strain in the outer surface
- E_f = flexural Modulus of elasticity, (MPa)
- P = load at a given point on the load deflection curve, (N)
- L = Support span, (mm)
- b = Width of test beam, (mm)
- d = Depth of tested beam, (mm)
- D = maximum deflection of the center of the beam, (mm)
- m = The gradient (i.e., slope) of the initial straight-line portion of the load deflection curve, (P/D), (N/mm), (Callister, 2001, Stanton and Bloem, 1957, Timoshenko, 1998).

2.9 Ultrasonic Model Used for the Concrete Structural Analysis

The Ultrasonic method used for assessing comparative strength of concrete is detailed in the following model. Ultrasonic techniques involved the propagation and detection of mechanical vibrations that have interacted in some way with the structure under test. When the surface of a semi-infinite solid was excited by a time varying mechanical force, energy was radiated from the source as three distinct types of elastic wave propagation. The fastest of these waves has particle displacements in the

direction of travel of the disturbance and was called the longitudinal, compression or P-wave. The compression wave velocity V_p is a function of the dynamic Young's modulus E , the Poisson's ratio ν , and the mass density ρ , and is given by:

$$V_p = \sqrt{\frac{E(1-\nu)}{\rho(1+\nu)(1-2\nu)}} \quad (22)$$

Or

Longitudinal pulse velocity (in km/s or m/s) is given by;

$$V_p = \frac{L}{T} \quad (23)$$

where,

V_p : is the longitudinal pulse velocity,

L : is the path length,

T : is the time taken by the pulse to traverse that length.

The second fastest is the shear, transverse or S-wave, which has particle displacements perpendicular to the propagation direction. The shear wave velocity V_s is a function of the dynamic shear modulus G and ρ ,

$$V_s = \sqrt{\frac{G}{\rho}} \quad (24)$$

Young's and the shear moduli are related by:

$$E = 2G(1+\nu) \quad (25)$$

Compression and shear wave velocities are theoretically interrelated by Poisson's ratio ν , which can be expressed as:

$$\nu = \frac{0.5 - (V_s/V_p)^2}{1 - (V_s/V_p)^2} \quad (26)$$

the compression and shear waves propagate throughout the material in all directions.

The third type of wave-motion produced travels along the surface and has elliptical particle motion, where the component of displacement normal to the surface is greater than the component in the direction of wave propagation. The velocity, V_R , of this surface wave, known as the Rayleigh wave, in simplified form is given by:

$$V_R = AV_S \quad (27a)$$

Where A is a function of ν and V_S . The ratio of V_R/V_S increases as Poisson's ratio increases. For values of ν from zero to 0.5, the ratio of V_R/V_S changes from approximately 0.87 to 0.96.

Ultrasonic inspection of concrete is basically the evaluation of one or more of these wave velocities. Since wave velocity is a direct indication of stiffness of the material, a higher wave velocity is associated with higher stiffness. When an ultrasonic wave is incident on a plane boundary between two media, some of the ultrasonic energy is transmitted through the boundary and some is reflected. The percentages of energy transmitted and reflected depend on the specific acoustic impedance, Z,

$$Z = \rho V \quad (27b)$$

Where ρ is the density of the material and V is the velocity of the wave. For two materials of different acoustic impedances Z_1 and Z_2 , the percentage energy transmitted E_T is given by (Halmshaw, 1987) as follows:

$$E_T = \frac{4Z_1Z_2}{(Z_1+Z_2)^2} \times 100 \quad (28)$$

and the percentage of reflected energy E_R , by:

$$E_R = \left(\frac{Z_1 - Z_2}{Z_1 + Z_2} \right)^2 \times 100 \quad (29)$$

The equations are valid for both compression and transverse waves, but as a transverse wave cannot be sustained in a liquid, a transverse wave at normal incidence is always completely reflected at a solid/liquid or solid/gas interface.

The resolution of an interrogating signal is indirectly proportional to signal wavelength λ , given by the relationship:

$$c = \lambda f, \quad (30)$$

Where c is the phase velocity and f the frequency of excitation. Additionally, an electro-acoustic transducer has directional properties, where the main energy falls to zero at an angle of divergence θ , given approximately by:

$$\sin \theta = \frac{1.22\lambda}{D} \quad (31)$$

Where D is the diameter of the transducer (Krautkramer, 1969).

Thus, high frequencies in the MHz range are preferred for ultrasonic inspection. In practice an upper limit is imposed on the frequency by very high attenuation of vibrations whose wavelengths are comparable with the grain size of the material to be inspected. For fine grained materials such as steel or aluminum, frequencies of tens of MHz will propagate without undue attenuation, and thus it is possible to produce a pulse in which most of the energy is contained within a beam of about 5° for a 16 mm 5 MHz transducer. In structural concrete however, the coarsest aggregate is of the order of 20 mm, which imposes a practical upper limit of several hundred kHz. Frequencies of the order of 50 kHz to 100 kHz are popular for long range inspection of concrete Bungey and Millard (1996) and 10 m for 54 kHz to 3 m for 82 kHz,

however these frequencies imply wavelengths around 50 mm, which for a standard 50 mm diameter transducer, offers no directional properties at all and low resolution. Nevertheless, the relation of wave velocities to mechanical properties, low relative cost and speed of operation make ultrasonic a popular choice, with much research being conducted worldwide to improve the techniques.

2.9.1 Quality of Concrete as a Function of the Ultrasonic Pulse Velocity

An ultrasonic pulse velocity (UPV) measurement in concrete is a well-established non-destructive technique. Low frequency narrow band transducers are greased and coupled to the structure (Figures 2.9 & 2.10). The time taken for a pulse to propagate through the material gives the velocity of sound, which is related to concrete quality. Once the velocity is determined, an idea about quality, uniformity, condition and strength of the concrete tested can be attained. The principle of assessing quality of concrete is that comparatively higher velocities are obtained when the quality of concrete in terms of density, homogeneity and uniformity is good. In case of poorer quality, lower velocities are obtained. Table 2.12 shows the use of velocity obtained to classify the quality of concrete (Qasrawi, 2000).

Table 2.12: Quality of concrete as a function of the UPV

UPV(m/s)	Concrete Quality
Above 4500	Excellent
3500 to 4500	Good
3000 to 3500	Doubtful
2000 to 3500	Poor
Below 2000	Very Poor

Source: (Qasrawi, 2000)

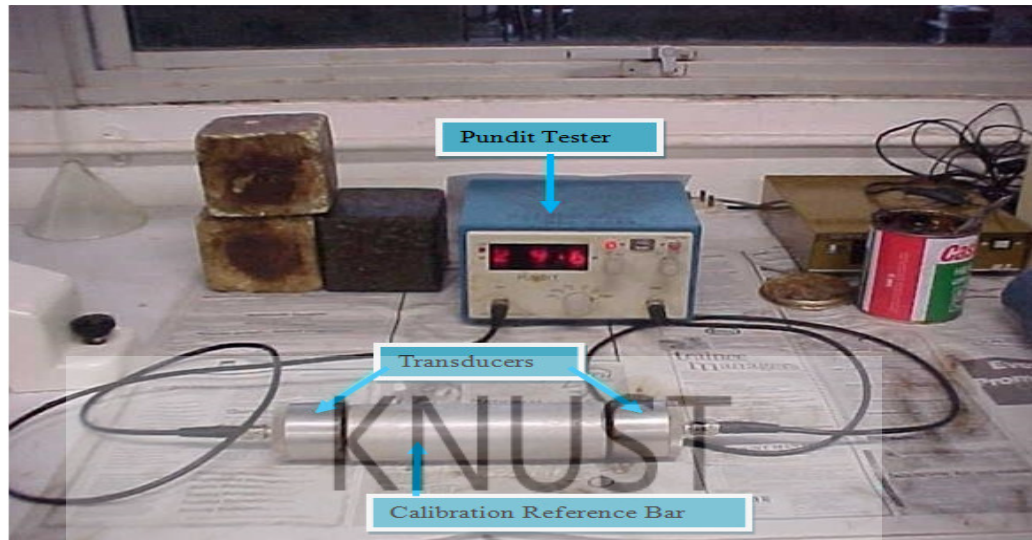


Figure 2.9: Pundit tester (General view layout) (Halmshaw, 1987)

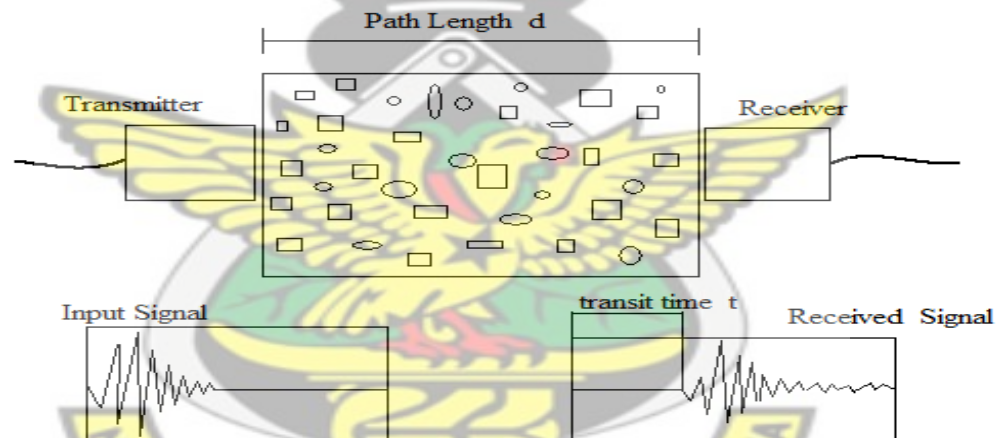


Figure 2.10: Ultrasonic pulse velocity testing (Halmshaw, 1987)

2.10 Neutron Activation Analysis (NAA)

Neutron activation analysis (NAA) is a physical method of analysis of material for elemental composition. The comparative activation analysis method involves irradiation and counting of a sample and a standard(s) containing a known amount of the element(s) of interest under identical conditions (same neutron flux and same

counting geometry). Under such conditions the following equations have been employed. (Quarshie, 2008).

$$A_S = N_S \sigma \Phi (1 - e^{-\lambda t}) \quad (32)$$

$$A_R = N_R \sigma \Phi (1 - e^{-\lambda t}) \quad (33)$$

Where:

A_S = activity (disintegrations/sec) of the nuclide in the sample at the end of irradiation.

A_R = activity (disintegrations/sec) of the nuclide in the standard at the end of irradiation.

Hence;

$$\frac{A_S}{A_R} = \frac{N_S}{N_R} = \frac{W_S}{W_R} = \frac{C_S}{C_R} \quad (34)$$

Where;

W_S = weight of the element in the sample in μg

W_R = weight of the element in the standard in μg

C_S = measured activity of nuclide in the sample

C_R = measured activity of the nuclide in the standard

Therefore,

$$\text{Concentration of element } (\mu\text{g/g}) = \frac{C_S W_R}{C_R W} \quad (35)$$

where W = weight of sample in grams

In NAA, samples are activated by neutrons. During irradiation the naturally occurring stable isotopes of most elements that constitute the rock or mineral samples, biological materials are transformed into radioactive isotopes by neutron capture. Then the activated nucleus decays according to a characteristic half-life; some

nuclides emit particles only, but most nuclides emit gamma-quanta, too, with specific energies. The quantity of radioactive nuclides is determined by measuring the intensity of the characteristic gamma-ray lines in the spectra. For these measurements a gamma-ray detector and special electronic equipment are necessary. As the irradiated samples contain radionuclides of different half-lives different isotopes can be determined at various time intervals.

2.10.1 Principles of the method

In the process of NAA the neutrons interact with the stable isotopes of the target element converting them to radioactive ones. The so-called compound nucleus emits gamma rays promptly with extremely short half lives in the order of millisecond (ms) and these can be measured during irradiation through a technique called prompt gamma activation analysis (PGAA). In most cases, the radioactive isotopes decay and emit beta particles accompanied by gamma quanta of characteristic energies, and the radiation can be used both to identify and accurately quantify the elements of the sample.

Subsequent to irradiation, the samples can be measured instrumentally by a high resolution semiconductor detector, or for better sensitivity, chemical separations can also be applied to reduce interferences. The qualitative characteristics are: the energy of the emitted gamma quanta (E) and the half-life of the nuclide ($T_{1/2}$). The quantitative characteristic is: the intensity (I), which is the number of gamma quanta of energy E measured per unit time.

First step of the analysis: sample preparation means in most cases only pulverising, homogenising, mass determination, packing, as well as the selection of the best analytical process and the preparation of the standards, if any.

Second step of the analysis: for irradiation one can choose from the various types of neutron sources according to need and availability.

Third step of the analysis: after the irradiation the analysts face the dilemma whether or not a chemical separation should be carried out for better sensitivity. If it is needed, the measurement shall be made after the separation. If there is no chemical treatment, the measurement is performed after a suitable cooling time (t_c).

Fourth step of the analysis: measurement, evaluation and calculation involve taking the gamma spectra and the calculating trace element concentrations of the sample. The most widely used gamma spectrometers consist of germanium based semiconductor detectors connected to a computer used as a multichannel analyzer for spectra evaluation and calculation.

2.10.2 Irradiation facilities

In the case of the most frequently used isotopic neutron sources an alpha emitting radioactive material is mixed with beryllium and an (α, n) reaction generates the neutrons (Table 2.13).

Table 2.13 Isotopic neutron sources:

-emitter	Half life	Neutrons s ⁻¹ Ci ⁻¹ emitted	Average neutron energy [MeV]
²²⁷ Ac	22 y	1.5×10^7	4
²²⁶ Ra	1620 y	1.3×10^7	3.6
²³⁹ Pu	2.4×10^4 y	1.4×10^7	4.5
²¹⁰ Po	138 d	2.5×10^6	4.3

2.10.3 Procedure for NAA

1. Before starting the irradiation the following have to be answered:

How can we determine Ca, Na, Cl in the samples in the presence of interfering components (e.g. Mg, Al, Si):

Choose the proper:

- Nuclear reaction
- Analytical gamma line
- Irradiation, decay and measuring times,
- Calculate the quantity of the elements to be used for standardization.

2. Sample preparation:

- weigh the samples into polyethylene bags using analytical balance
- prepare standards

3. Irradiation of the samples using pneumatic system of the reactor

4. Measure the gamma-spectra, evaluate the spectra (determine the peak areas at the given gamma-lines)

5. Identify the isotopes in the spectra using gamma library. Determine the elemental concentrations and their uncertainties using standard method.

2.11 X-Ray Shielding Analysis

Broad beam transmission (B_T) is the ratio of radiation output of a source (in $\text{mSv mA}^{-1} \text{min}^{-1}$ at 1m) with barrier in place (I) to that without the barrier in place (I_0). Thus:

$$I = I_0 e^{-\mu x} \quad (36a)$$

$$\frac{I}{I_0} = e^{-\mu x} = B_T \quad (36b)$$

$$\frac{I}{I_0} = e^{-\mu x} \quad (\text{for Good geometry/Narrow beam, } B = 1) \quad (36c)$$

$$\ln\left(\frac{I}{I_0}\right) = -\mu x \quad (\text{in the form of } y = mx) \quad (36d)$$

Where B_T = Broad beam transmission, and B = Build-up factor

In **X-ray**, for useful beam, and primary protective barrier design; equation typical of X-ray is given by:

$$\frac{I}{I_0} = e^{-\mu x} = B_T = K = \frac{Pd^2}{WUT} \quad (36e)$$

Where P = exposure rate (Rwk^{-1}), d = distance (m), K = fraction of beam transmitted

W = work load (mA min wk^{-1}), U = Use factor and T = occupancy (Turner, 2007)

2.11.1 Estimation of Material Thickness (x) for Shielding

The thickness of material for shielding was estimated from the following equations:

Let N be number of half-value layer (HVL); then:

$$\text{Total material's thickness (x)} = NHVL \quad (37a)$$

$$\text{But } N = -\frac{\ln B_T}{\ln 2} \quad \text{and} \quad HVL = \frac{\ln 2}{\mu} \quad (\text{Turner, 2007}) \quad (37b)$$

$$\Rightarrow X = NHVL = \left(-\frac{\ln B_T}{\ln 2}\right) \left(\frac{\ln 2}{\mu}\right) = -\frac{\ln B_T}{\mu} \quad (37c)$$

Where; μ (cm^{-1}) is linear attenuation coefficient.

2.12 X-ray Powder Diffraction (XRD)

X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The discovery of X-rays in 1895 enabled scientists to probe crystalline structure at the atomic level.

X-ray diffraction has been in use in two main areas;

- For the fingerprint characterization of crystalline materials and
- The determination of their structure.

Every crystalline solid has its unique characteristic X-ray powder pattern which may be used as a "fingerprint" for its identification. Once the material has been identified, X-ray crystallography may be used to determine its structure; that is how the atoms pack together in the crystalline state and what the interatomic distance and angle are etc. X-ray diffraction is one of the most important characterization tools used in solid state chemistry and materials science. The analyzed material is finely ground, homogenized, and average bulk composition is determined.

2.12.1 X-ray Powder Diffraction (XRD) Instrumentation

X-ray diffractometers consist of three basic components:

- X-ray tube,
- Sample holder, and
- X-ray detector.

X-rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced.

The incident beam will ionize electrons from the K-shell (1s) of the target atom and X-rays are emitted as the resultant vacancies are filled by electrons dropping down from the L (2p) or M (3p) levels.

These spectra consist of several components, the most common being K_{α} and K_{β} . K_{α} consists, in part, of $K_{\alpha 1}$ and $K_{\alpha 2}$. $K_{\alpha 1}$ has a slightly shorter wavelength and twice the intensity as $K_{\alpha 2}$. The specific wavelengths are characteristic of the target material (Cu, Fe, Mo, Cr). Filtering, by foils or crystal monochrometers, is required to produce monochromatic X-rays needed for diffraction. $K_{\alpha 1}$ and $K_{\alpha 2}$ are sufficiently close in

wavelength such that a weighted average of the two is used. Copper is the most common target material for single-crystal diffraction, with Cu K_{α} radiation = 1.5418 Å. These X-rays are collimated and directed onto the sample. As the sample and detector are rotated, the intensity of the reflected X-rays is recorded. When the geometry of the incident X-rays impinging the sample satisfies the Bragg Equation, constructive interference occurs and a peak in intensity occurs. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor.

The geometry of an X-ray diffractometer is such that the sample rotates in the path of the collimated X-ray beam at an angle θ while the X-ray detector is mounted on an arm to collect the diffracted X-rays and rotates at an angle of 2θ . The instrument used to maintain the angle and rotate the sample is termed a goniometer. For typical powder patterns, data is collected at 2θ from $\sim 5^{\circ}$ to 70° , angles that are preset in the X-ray scan.

2.12.2 Applications of XRD

X-ray powder diffraction is most widely used for the identification of unknown crystalline materials example; minerals, inorganic compounds. Other applications include:

- characterization of crystalline materials
- identification of fine-grained minerals such as clays and mixed layer clays that are difficult to determine optically
- determination of unit cell dimensions
- measurement of sample purity

With specialized techniques, XRD can be used to:

- determine crystal structures using Rietveld refinement
- determine modal amounts of minerals (quantitative analysis)
- characterize thin film samples by:
 - determining lattice mismatch between film and substrate and to inferring stress and strain
 - determining dislocation density and quality of the film by rocking curve measurements
 - measuring super lattices in multilayered epitaxial structures
 - determining the thickness, roughness and density of the film using glancing incidence X-ray reflectivity measurements
- make textural measurements, such as the orientation of grains, in a polycrystalline sample

2.12.3 Strengths and Limitations of X-ray Powder Diffraction

2.12.3.1 Strengths

- Powerful and rapid (< 20 min) technique for identification of an unknown mineral
- In most cases, it provides an unambiguous mineral determination
- Minimal sample preparation is required
- XRD units are widely available
- Data interpretation is relatively straight forward

2.12.3.2 Limitations

- Homogeneous and single phase material is best for identification of an unknown

- Must have access to a standard reference file of inorganic compounds (d-spacing, *hkl*s)
- Requires tenths of a gram of material which must be ground into a powder
- For mixed materials, detection limit is ~ 2% of sample
- For unit cell determinations, indexing of patterns for non-isometric crystal systems is complicated
- Peak overlay may occur and worsens for high angle 'reflections'

2.12.4 Sample Collection and Preparation for XRD

Determination of an unknown requires: the material, an instrument for grinding, and a sample holder.

- Obtain a few tenths of a gram (or more) of the material, as pure as possible
- Grind the sample to a fine powder, typically in a fluid to minimize inducing extra strain (surface energy) that can offset peak positions, and to randomize orientation. Powder less than ~10 μm in size is preferred
- Place into a sample holder or onto the sample surface:
 - smear uniformly onto a glass slide, assuring a flat upper surface
 - pack into a sample container
 - sprinkle on double sticky tape

Typically the substrate is amorphous to avoid interference

- Care must be taken to create a flat upper surface and to achieve a random distribution of lattice orientations unless creating an oriented smear.
- For analysis of clays which require a single orientation, specialized techniques for preparation of clay samples are given.

For unit cell determinations, a small amount of a standard with known peak positions (that do not interfere with the sample) can be added and used to correct peak positions.

2.12.5 Data Collection, Results and Presentation of XRD

2.12.5.1 Data Collection

The intensity of diffracted X-rays is continuously recorded as the sample and detector rotate through their respective angles. A peak in intensity occurs when the mineral contains lattice planes with d-spacing appropriate to diffract X-rays at that value of θ . Although each peak consists of two separate reflections ($K\alpha_1$ and $K\alpha_2$), at small values of 2θ the peak locations overlap with $K\alpha_2$ appearing as a hump on the side of $K\alpha_1$. Greater separation occurs at higher values of θ . Typically these combined peaks are treated as one. The 2λ position of the diffraction peak is typically measured as the center of the peak at 80% peak height.

2.12.5.2 Data Reduction

Results are commonly presented as peak positions at 2θ and X-ray counts (intensity) in the form of a table or an x-y. Intensity (I) is either reported as peak height intensity, that intensity above background, or as integrated intensity, the area under the peak. The relative intensity is recorded as the ratio of the peak intensity to that of the most intense peak (relative intensity = $I/I_1 \times 100$).

2.12.5.3 Determination of an Unknown

The d-spacing of each peak is then obtained by solution of the Bragg equation for the appropriate value of λ . Once all d-spacing have been determined, automated search/match routines compare the ds of the unknown to those of known materials. Because each mineral has a unique set of d-spacing, matching these d-spacing

provides an identification of the unknown sample. A systematic procedure is used by ordering the d-spacing in terms of their intensity beginning with the most intense peak. Files of d-spacing for hundreds of thousands of inorganic compounds are available from the International Centre for Diffraction Data as the Powder Diffraction File. Commonly this information is an integral portion of the software that comes with the instrumentation.

Single crystal X-ray diffraction is a powerful technique that is commonly used to determine the structures of new materials. However, the technique is limited by the ability to grow nearly perfect crystals that are suitable for diffraction. Due to this limitation and the time and cost-intensive nature of the technique, single crystal diffraction is not used for routine structural characterization of known materials.

For routine structural characterization of materials, X-ray powder diffraction is far more common. The samples for powder diffraction may be large crystals, or they may be in the form of a powder composed of microcrystals that are too small to be seen by the human eye. The underlying principles of the experiment are the same in both powder diffraction and single crystal diffraction, although the data analysis is much simpler in powder diffraction (Warren, 1990; York, 1997). Figure 2.11, is a typical schematic diagram for XRD set up.

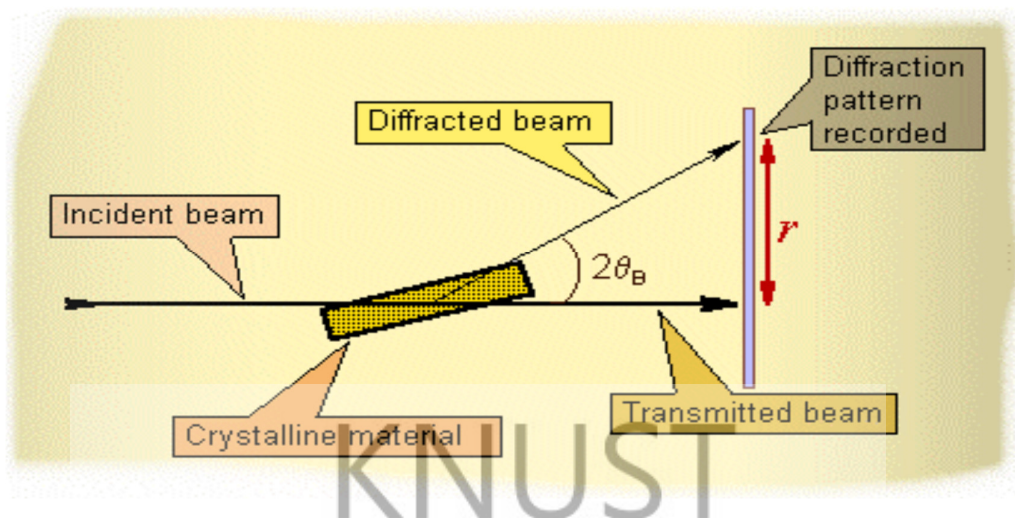


Figure 2.11: X-Ray Diffraction schematic diagram

2.12.6 Unit Cells, Miller Planes, and Diffraction Data

It can be recalled that the conditions for diffraction are governed by the conditions for constructive interference. An expanded view of the diffraction of X-rays from the repeating planes of atoms in a crystalline structure is shown in Figure 2.11. Powder diffraction patterns are typically plotted as the intensity of the diffracted X-rays vs. the angle 2θ . Peaks will appear in the diffraction pattern at 2θ , θ values when constructive interference is at a maximum, that is, when Bragg's Law (Eqn. 38) is satisfied.

$$n\lambda = 2d \sin\theta \quad (38)$$

In this experiment, the first order ($n = 1$) diffraction of X-rays with a wavelength of $1.54056 \text{ \AA}$ was observed. By measuring the 2θ values for each diffraction peak, the d -spacing (the distance between the diffracting planes) for each diffraction peak was calculated using data analysis software. The data analysis software has a program for automatically calculating the d -spacings for all of the peaks in the diffraction pattern.

Each crystalline substance has a unique X-ray diffraction pattern. The number of observed peaks was related to the symmetry of the unit cell (higher symmetry generally means fewer peaks). The d -spacings of the observed peaks were related to the repeating distances between planes of atoms in the structure. And finally, the intensities of the peaks are related to what kinds of atoms are in the repeating planes. The scattering intensities for X-rays are directly related to the number of electrons in the atom. Hence, light atoms scatter X-rays weakly, while heavy atoms scatter X-rays more effectively. These three features of a diffraction pattern: the number of peaks, the positions of the peaks, and the intensities of the peaks, define a unique, fingerprint X-ray powder pattern for every crystalline material.

Unit cells in three-dimensional repeating structures have different shapes based upon the symmetry of the structure. In all cases, the unit cells are parallelepipeds, but the different shapes arise depending on restrictions placed on the lengths of the three edges (a , b , and c) and the values of the three angles (α , β , and γ). The seven different unit cell shapes or the so-called seven crystal systems that result from these restrictions are listed in Table 2.14 (Askeland, 1985, Callister, 2001).

Table 2.14: The seven crystal systems and the restrictions placed on the lattice parameters of the unit cell.

Crystal System	Lattice Parameter Restrictions
Cubic	$a = b = c$ $\alpha = \beta = \gamma = 90^\circ$
Tetragonal	$a = b \neq c$ $\alpha = \beta = \gamma = 90^\circ$
Orthorhombic	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90^\circ$
Monoclinic	$a \neq b \neq c$ $\alpha = \gamma = 90^\circ; \beta \neq 90^\circ$
Triclinic	$a \neq b \neq c$ $\alpha \neq \beta \neq \gamma \neq 90^\circ$
Hexagonal	$a = b \neq c$ $\alpha = \beta = 90^\circ; \gamma = 120^\circ$
Trigonal*	$a = b \neq c$ $\alpha = \beta = 90^\circ; \gamma = 120^\circ$

The difference between trigonal and hexagonal systems is the symmetry. A hexagonal unit cell has C_6 symmetry, whereas a trigonal unit cell only has C_3 symmetry. The restrictions shown above are for the so-called hexagonal setting of a trigonal setting. There is an alternative way to define the unit cell for trigonal systems, known as the rhombohedral setting.

Each peak in a diffraction pattern arises from a unique set of repeating planes in the structure. These sets of planes are oriented in all different directions in three-dimensional space. Diffraction from a particular set of planes may not be observed or the peak intensity may be low due to symmetry (patterns of systematic absences) or other factors that contribute to low intensity.

How does one specify these different sets of planes in a particular structure? This is done by assigning a set of *Miller indices* ($h\ k\ l$), three integers that denote the orientation of the planes with respect to the unit cell. For a given set of planes, one plane in the set will intercept the unit cell at the following points along the three axes relative to the origin:

$$\left(\frac{a}{h}, 0, 0\right) \text{ and } \left(0, \frac{b}{k}, 0\right) \text{ and } \left(0, 0, \frac{c}{l}\right)$$

The other planes in the set are related to this first set by the translational symmetry. For example as shown in Figure 2.12, if the front bottom left corner of the box is taken as the origin of the unit cell, the (111) Miller plane intercepts the unit cell at $(a, 0, 0)$, $(0, b, 0)$, and $(0, 0, c)$. The next plane in the set is also shown, intercepting the unit cell behind the first one at the same relative locations. These are but two in a family of planes extending through the whole structure separated by an interplanar spacing designated d_{111} . Now consider the (222) Miller planes. The first one in the set shown on the right intercepts the unit cell at $(a/2, 0, 0)$, $(0, b/2, 0)$, and $(0, 0, c/2)$. There are twice as many of the (222) planes in the repeating structure, and their interplanar spacing, d_{222} , is half as large as the interplanar spacing of the (111) planes. If a Miller index is zero, then the plane does not intercept the axis at all along that axis.

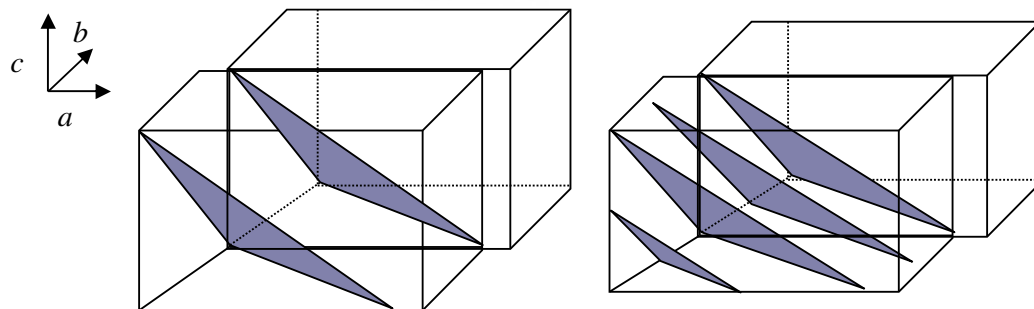


Figure 2.12: Crude sketches of Miller planes

Refining the lattice parameters of the unit cell from X-ray powder diffraction data requires knowing how to assign the Miller indices ($h\ k\ l$) to each diffraction peak, a process known as indexing the pattern. Normally for a known material, this is done by comparing the data to that reported in the literature or in a database of diffraction patterns known as the JCPDS. The Miller indices relate the peak positions or d -spacings to the lattice parameters by an equation specific to the crystal system. For example, in a structure with an orthorhombic unit cell the relationship is expressed in equation (39) below:

$$\frac{1}{d_{hkl}^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \quad (39)$$

where a , b , and c are the lattice parameters of the unit cell and hkl are the Miller indices identifying the repeating planes causing the diffraction peak with spacing d_{hkl} . If the symmetry of the unit cell is higher, for example in a cubic system where $a = b = c$, then equation 39 simplifies to equation (40a).

$$\frac{1}{d_{hkl}^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad (40a)$$

In crystal systems with angles not equal to 90° , the equation includes terms involving the angles as well. In a cubic system, one peak position, d , can be used to determine the lattice parameter provided the Miller indices can be assigned. However, a better way to obtain the lattice parameter is to input all of the peak positions that can be indexed and refine the lattice parameters using a number of data points and regression analysis (Shoemaker *et al*, 1996).

2.12.7 Crystal Geometry Equations for X-ray Diffraction Analysis

Perfect crystals are comprised of a repetition of smaller structures called unit cells. The repeating unit cells form lattice structures. It is the planes of these lattice

structures that diffract the x-rays. Crystals are most often described in terms of three crystal axes, **a**, **b**, and **c**. These axes which may be any length and do not have to be at right angles, form the basis for the coordinate system.

All crystals can be described using equation: **ha + kb + lc = positive constant** (40b) where h, k and l are called Miller indices and are the reciprocals of intersection distances of the lattice planes. Each type of the crystal system is defined by a Miller indices series and more specifically by the gaps (D-spacing) that are present. The Miller indices are used to determine the interplanar distance (Shoemaker *et al*, 1996).

2.12.7.1 Lattice Model for D-spacings and Miller indices

The following are the models for analyzing D-spacing, Unit cell volume and Angle between planes.

Symmetry

Cubic:
$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad (41)$$

Tetragonal:
$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \quad (42)$$

Hexagonal:
$$\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \quad (43)$$

Rhombohedral:
$$\frac{1}{d^2} = \frac{(h^2 + k^2 + l^2)\sin^2\alpha + 2(hk + kl + hl)(\cos^2\alpha - \cos\alpha)}{a^2(1 - 3\cos^2\alpha + 2\cos^3\alpha)} \quad (44)$$

Orthorhombic:
$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \quad (45)$$

Monoclinic:
$$\frac{1}{d^2} = \frac{1}{\sin^2\beta} \left(\frac{h^2}{a^2} + \frac{k^2 \sin^2\beta}{b^2} + \frac{l^2}{c^2} - \frac{2h^2 l \cos\beta}{ac} \right) \quad (46)$$

Triclinic:
$$\frac{1}{d^2} = \frac{1}{V^2} (S_{11}h^2 + S_{22}k^2 + S_{33}l^2 + 2S_{12}hk + 2S_{23}kl + 2S_{13}hl) \quad (47)$$

Where

$$S_{11} = b^2 c^2 \sin^2 \alpha \quad (48)$$

$$S_{22} = a^2 c^2 \sin^2 \beta \quad (49)$$

$$S_{33} = a^2 b^2 \sin^2 \gamma \quad (50)$$

$$S_{12} = abc^2 (\cos \alpha \cos \beta - \cos \gamma) \quad (51)$$

$$S_{23} = a^2 bc (\cos \beta \cos \gamma - \cos \alpha) \quad (52)$$

$$S_{13} = ab^2 c (\cos \gamma \cos \alpha - \cos \beta) \quad (53)$$

2.12.7.2 Volumetric Model for Unit Cell

Symmetry

Cubic: $V = a^3 \quad (54)$

Tetragonal: $V = a^2 c \quad (55)$

Hexagonal: $V = \frac{\sqrt{3} a^2 c}{2} \quad (56)$

Rhombohedral: $V = a^3 \sqrt{1 - 3 \cos^2 \alpha + 2 \cos^3 \alpha} \quad (57)$

Orthorhombic: $V = abc \quad (58)$

Monoclinic: $V = abc \sin \beta \quad (59)$

Triclinic:

$$V = abc \sqrt{1 - \cos^2 \alpha - \cos^2 \beta - \cos^2 \gamma + 2 \cos \alpha \cos \beta \cos \gamma} \quad (60)$$

2.12.7.3 Planar Model for Angle ϕ between planes $(h_1k_1l_1)$ and $(h_2k_2l_2)$

Symmetry

Cubic:
$$\cos \Phi = \frac{h_1h_2 + k_1k_2 + l_1l_2}{\sqrt{(h_1^2+k_1^2+l_1^2)(h_2^2+k_2^2+l_2^2)}} \quad (61)$$

Tetragonal:
$$\cos \Phi = \frac{\frac{h_1h_2 + k_1k_2}{a^2} + \frac{l_1l_2}{c^2}}{\sqrt{(\frac{h_1^2+k_1^2}{a^2} + \frac{l_1^2}{c^2})(\frac{h_2^2+k_2^2}{a^2} + \frac{l_2^2}{c^2})}} \quad (62)$$

Hexagonal:
$$\cos \Phi = \frac{h_1h_2 + k_1k_2 + \frac{1}{2}(h_1k_2 + h_2k_1) + \frac{3a^2}{4c^2}l_1l_2}{\sqrt{(h_1^2 + k_1^2 + h_1k_1 + \frac{3a^2}{4c^2}l_1^2)(h_2^2 + k_2^2 + h_2k_2 + \frac{3a^2}{4c^2}l_2^2)}} \quad (63)$$

Rhombohedral:

$$\cos \phi = \frac{a^4 d_1 d_2}{V^2} [\sin^2 \alpha (h_1h_2 + k_1k_2 + l_1l_2) + (\cos^2 \alpha + \cos \alpha)(k_1l_2 + k_2l_1 + l_1h_2 + l_2h_1 + h_1k_2 + h_2k_1)] \quad (64)$$

Orthorhombic:
$$\cos \phi = \frac{\frac{h_1h_2}{a^2} + \frac{k_1k_2}{b^2} + \frac{l_1l_2}{c^2}}{\sqrt{(\frac{h_1^2}{a^2} + \frac{k_1^2}{b^2} + \frac{l_1^2}{c^2})(\frac{h_2^2}{a^2} + \frac{k_2^2}{b^2} + \frac{l_2^2}{c^2})}} \quad (65)$$

Monoclinic:
$$\cos \phi = \frac{d_1 d_2}{\sin^2 \beta} \left[\frac{h_1h_2}{a^2} + \frac{k_1k_2 \sin^2 \beta}{b^2} + \frac{l_1l_2}{c^2} - \frac{(l_1h_2 + l_2h_1 \cos \beta)}{ac} \right] \quad (66)$$

Triclinic:

$$\cos \phi = \frac{d_1 d_2}{V^2} [S_{11}h_1h_2 + S_{22}k_1k_2 + S_{33}l_1l_2 + S_{23}(k_1l_2 + k_2l_1) + S_{13}(l_1h_2 + l_2h_1) + S_{12}(h_1k_2 + h_2k_1)] \quad (67)$$

2.12.7.4 Method of Calculation of Atomic Packing Factor for Calcite

V_c = Total unit cell volume

V_s = Volume of two (2) atoms in a unit cell

Lattice Parameters of Calcite (CaCO_3)

Hexagonal Closed Packed Crystal Structure (HCP)

Axes: $a = b \neq c$

In ideal HCP; $c = 1.633a$ (Askeland, 1985). (68a)

$$V_c = abc \sin 60^\circ \quad (68b)$$

but $a = b$, and $\sin 60^\circ = \frac{\sqrt{3}}{2}$,

$$\Rightarrow V_c = a^2 c \frac{\sqrt{3}}{2} \quad (68c)$$

$$V_s = \frac{4}{3} \pi R^3 \times 2$$

$$\text{but } R = \frac{a}{2}$$

$$\begin{aligned} \Rightarrow V_s &= \frac{4}{3} \pi \left(\frac{a}{2}\right)^3 \times 2 \\ &= \frac{4}{3} \pi \frac{a^3}{8} \times 2 = \frac{\pi a^3}{3} \end{aligned}$$

HCP has three (3) unit cells (Kakani, 2004)

$$\Rightarrow V_s \text{ per Unit cell} = \frac{\pi a^3}{3} \quad (69)$$

$$\begin{aligned} \Rightarrow \text{APF} &= \frac{V_s}{V_c} = \frac{\pi a^3}{3a^2 c \frac{\sqrt{3}}{2}} = \frac{2a\pi}{3\sqrt{3}c} \\ &= \frac{2(a)3.142}{3\sqrt{3}(1.633a)} \times 100 = 74\% \end{aligned}$$

$$\Rightarrow \text{APF (Calcite)} = 74\% \quad (70)$$

This implies that Calcite has 26% void comparable to FCC materials. This is stronger than the BCC materials of APF of 68%.

2.13 Scanning Electron Microscope

The electrons produced and emitted by the SEM is made to interact with the atoms in the sample under investigation. This produces signals which contains information or knowledge basically about the surface topography and composition of the sample. The electron beam is generally scanned in a raster scan pattern and the position of the beam is combined with the detected signal to produce an image. The SEM can achieve a powerful and much better resolution than 1 nanometer. Moreover, with the SEM, specimens or samples can be observed in either high or low vacuum conditions, in wet conditions, at a broad range of cryogenic temperatures and at wider range of elevated temperatures too.

The most common method of operation is the detection by secondary electrons, which are emitted by the atoms excited by the electron beam. The number of secondary electrons is a function of the angle between the surface and the beam of electron. On a flat surface, the plume of secondary electrons is mostly contained by the sample, but on a tilted surface, the plume is partially exposed and more electrons are emitted. By scanning the sample and detecting the secondary electrons, an image displaying the tilt of the surface is created.

2.13.1 Principles and Capacities of SEM

Signals produced by the SEM include Secondary electrons (SE), Back-scattered electrons (BSE), Characteristic X-rays, Light (Cathodoluminescence) (CL), specimen current and transmitted electrons. Secondary electron detectors are basically the standard equipment used in SEMs, but it is rare that a single machine would have detectors for all possible signals. In the most common standard detection mode, the SEM can produce a very high resolution of images of a sample, and it is capable of

revealing details less than 1nm in size. Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three-dimensional appearance of a sample useful for understanding its surface structure.

Back-scattered electrons (BSE) are beam electrons that are reflected from the sample by elastic scattering. BSE are often used in analytical SEM along with the spectra from the characteristic x-rays, because the intensity of the BSE signal is strongly related to the atomic number (Z) of the specimen. BSE images can provide information about the distribution of different elements in the sample. For this reason BSE imaging can be used to image substances of 5 or 10 nm diameter which would otherwise be difficult or impossible to detect in secondary electron images in biological specimens.

Characteristic X-ray are emitted when the electron beam removes an inner shell electron from the sample, causing a higher energy electron to fill the shell and release energy. These characteristic X-rays are used to identify the composition and abundance of elements in the sample. For materials, back scattered electron imaging quantitative X-ray analysis, and X-ray mapping of specimens often requires that the surfaces be ground and polished to an ultra-smooth surface. Specimens that undergo WDS or EDS analysis are often carbon coated. In general, metals are not coated prior to imaging in the SEM because they are conductive and provide their own pathway to ground.

Fractography is the study of fractured surfaces that can be done on a light microscope or commonly, on an SEM. The fractured surface is cut to a suitable size, cleaned of any organic residues, and mounted on a specimen holder for viewing in the SEM. Integrated circuits may be cut with a focused ion beam (FIB) or other ion beam

milling instrument for viewing in the SEM. The SEM in the first case may be incorporated into the FIB.

Metals, geological specimens, and integrated circuits all may also be chemically polished for viewing in the SEM. Special high-resolution coating techniques are required for high- magnification imaging of inorganic thin films.

2.13.2 A Standard Application of SEM

Scanning electron microscope (SEM) is a scientific instrument that uses a beam of highly energetic electrons to examine the surface and phases distribution of specimens on a micro scale through the live imaging of secondary electrons (SE) and back-scattered electrons (BSE). One of the main activities of SEM Laboratory at MINT is for failure analysis on metal part and components. The capability of SEM is excellent for determining the root cause of metal failures such as ductility or brittleness, stress corrosion, fatigue and other types of failures. Many of the requests for failure analysis are from local petrochemical plants, manufacturers of automotive components, pipeline maintenance personnel and engineers who involved in the development of metal parts and component.

CHAPTER THREE

MATERIALS AND METHODS

3.0 Introduction

This chapter presents the materials used and methodology employed in sample preparation and testing. The tests carried out are compressive strength test, flexural strength test, ultrasonic test and neutron activation analysis (NAA), and the determination of shielding or radiological properties (linear attenuation coefficients, μ) of Pozzolana and Portland cement, using X-ray. XRD and SEM analyses are carried out for crystal structure and microstructure examinations of PPC respectively.

3.1 Materials and Samples Preparation

The Pozzolana was obtained from Building and Road Research Institute (BRRI) of the Council for Scientific and Industrial Research (CSIR), Kumasi. The OPC used as reference is from the cement factory (GHACEM) Tema.

Crushed gravel with a maximum nominal size of 10 mm was used as coarse aggregate and natural sand conforming to Zone II with a fineness of 2.52 mm was used as fine aggregate.

3.2 Sample Moulding

A concrete mix of cement, sand and stone in the ratio 1:2:4 was used for all concrete cubes and cylinders cast in this work. The OPC was partially replaced by Pozzolana in varied percentages of its volume (0%, 10%, 20%, 30%, 40%, 50%, 60%, 70%, and 80%). Figures 3.1 – 3.18 showed mould preparation and concrete casting. These involved (1) assembly of polished wood board to size (15 cm³), (2) cutting to size cylindrical moulds (plastic, PV pipe) of diameter 10 cm and 50 cm long, (3) the pastes

were poured in cubic moulds of $15 \times 15 \times 15 \text{ cm}^3$ (collapsible, multiple wooden moulds) capable of casting 32 cubes of concrete at a time and as well as the cylindrical moulds. The pastes were allowed to undergo setting.



Figure 3.1: Mould design and preparation

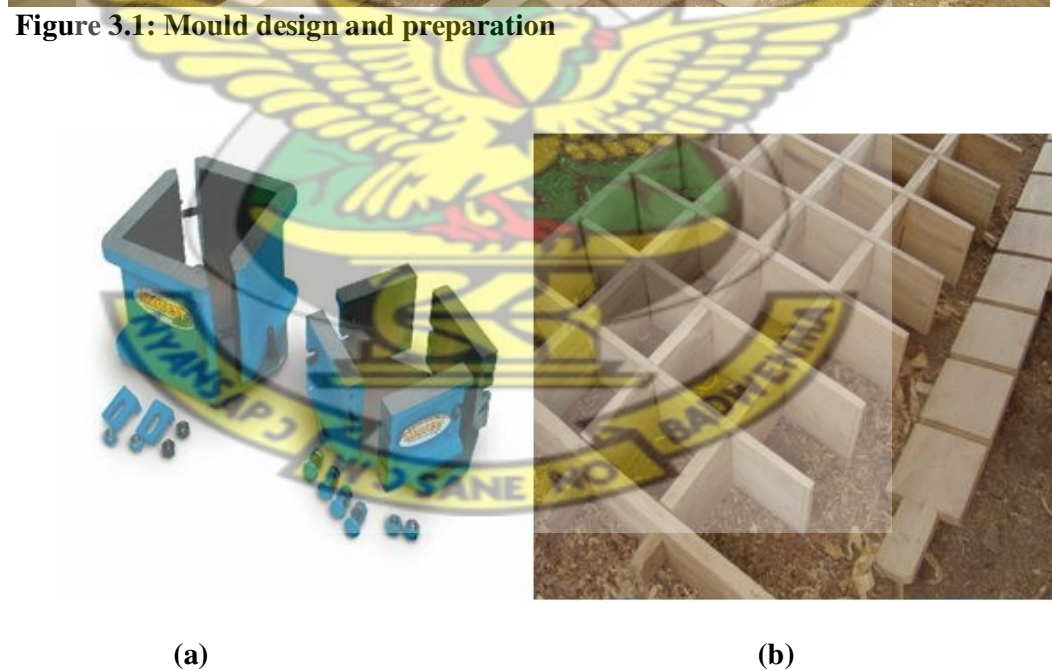


Figure 3.2: Mould being assembled: (a) Metallic mould (b) Wooden multi cubic mould



Figure 3.3: Thorough mixing of concrete being carried out manually



Figure 3.4: Concrete cubes being cast



Figure 3.5: Concrete cubes ready for laboratory analysis



Figure 3.6: Casting cylindrical concrete



Figure 3.7: Cylindrical concrete ready for flexural test

3.3 Methods

The tests employed for this project are as follows:

1. Compression test
2. Flexural test (Tensile test)
3. Ultrasonic test (Nondestructive test)
4. Neutron Activation Analysis (NAA)
5. X-ray Shielding Analysis
6. X-Ray Diffraction (XRD) Analysis
7. Scanning Electron Microscopy (SEM) Analysis

Equipment used were compression testing machine, flexural test machine, electronic vibrator, shovels and other mixing apparatus all from the Ghana Standards Board while the ultrasonic testing machine, x-ray source for shielding analysis, research reactor for neutron activation analysis were from the Ghana Atomic Energy

Commission. The x-ray diffraction machine and the scanning electron microscope were from the University of Ghana.

Plastic sheets were used to cover the specimens to prevent water from evaporating. After 24 hours, the specimens were stripped from their respective moulds and taken to curing location (water bath). The strength tests were carried out at 2, 7, 14, and 28 days taking the average of three specimens of each percentage of Pozzolana for each test at a curing temperature of $25 \pm 1^\circ\text{C}$.

3.3.1 Compression Test

The compressive tests were carried out using compression testing machine with range 0-2000kN crushing force. This Young's machine was used for compressive tests on the second (2) day, seven (7) day, fourteen (14) day and 28 day cured concrete specimens. For each test day, the concrete cubes were placed in the loading apparatus (Figure 3.9), and the load was actuated at a controlled loading rate. Once the specimen reached its critical load, one of the load indication needles on the dial gauge of the machine (Figure 3.8) recorded the exact failure point. The measurements were recorded, and an average value was taken for use in later calculations.



Figure 3.8: Young's machine dial Gauge



Figure 3.9: Young's machine loaded with Concrete Cube

3.3.2 Flexural test

Flexural test was conducted only on 28 days' curing of the cylindrical specimen with w/c 0.5. The concrete cylinders were collected from their curing location (water bath) for testing. Each of the cylinder diameter and length, as well as the beam dimensions, was measured with a dial caliper and tape measure. Flexural testing machine (Figure 3.10) was used to measure the mechanical behavior of the concrete subjected to cylindrical loading. The flexure test produced tensile stress in the convex side of the specimen and compression stress in the concave side. For this test, the concrete cylinders were placed in the loading apparatus and the load was actuated at a controlled loading rate. Once the specimen reached its critical load, one of the load indication needles recorded the exact failure point. Equation (18) was used to compute flexural strength or modulus of rupture.

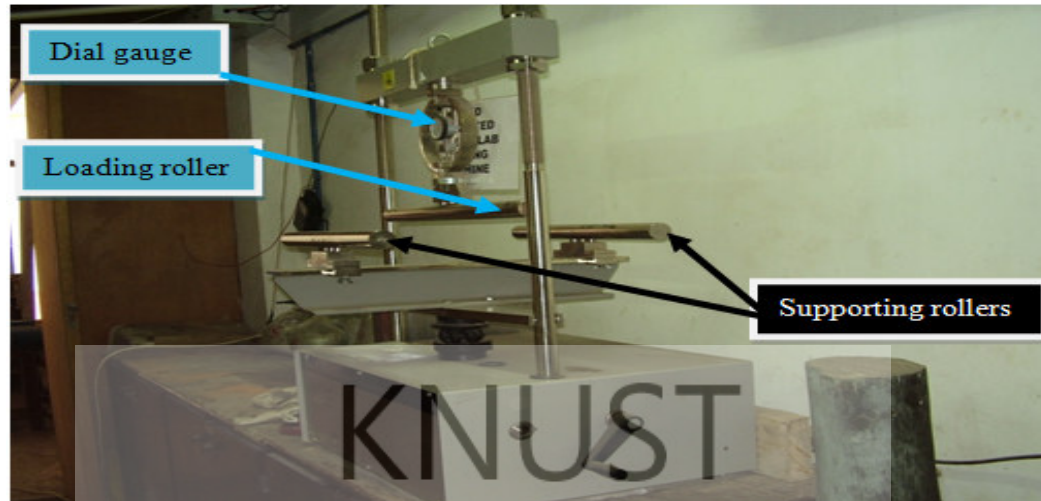


Figure 3.10: Flexural testing machine (Centre-point loading) used

3.3.3 Ultrasonic Test

Ultrasonic test machine of frequency 10MHz (Figure 3.11) was used. A pulse of longitudinal vibrations is produced by an electro-acoustical transducer, which was held in contact with one surface of the concrete under test. When the pulse generated was transmitted into the concrete from the transducer using a liquid coupling material such as grease or cellulose paste, it underwent multiple reflections at the boundaries of the different material phases within the concrete. A complex system of stress waves developed, which included both longitudinal and shear waves, and propagated through the concrete. The first waves to reach the receiving transducer were the longitudinal waves, which were converted into an electrical signal by a second transducer. Electronic timing circuits enable the transit time T of the pulse to be measured. Equations (22) and (23) were used to compare direct readings. Once the velocity is determined, an idea about quality, uniformity, condition and strength of the concrete tested was attained. The principle of assessing quality of concrete was that comparatively higher velocities were obtained when the quality of concrete in terms

of density, homogeneity and uniformity was good. In case of poorer quality, lower velocities were obtained.



Figure 3.11: The PUNDIT plus set (Used in the experiment)

3.3.4 Neutron Activation Analysis (NAA)

For NAA, the samples were counted on a computer-based gamma-ray spectroscopy system which consists of an N-type HPGe detector model GR 2518-7500SL mounted in a cylindrical lead shield (100 mm thick) and cooled in liquid nitrogen. The detector is coupled to a computer-based PCA-MR 18192 Multi-Channel Analyzer (MCA). The elements were identified using gamma ray spectrum analysis software, ORTEC MAESTRO-32. Each sample weighing about 0.1g was placed on a polyethylene of 1mm wall thickness, wrapped and then heat sealed. The wrapped samples were then concealed in rabbit capsules of height 5 mm and external diameter of 1.4 mm, this was then stuffed with cotton wool and then heat sealed. For long and medium-lived irradiation, all the samples were concealed in one capsule, stuffed with cotton wool and then heat sealed. For short-lived irradiation, each sample was placed in one individual capsule.

The samples were sent into the inner irradiation sites of the reactor for thermal irradiation by means of pneumatic transfer system operating at 65 psi. Three types of irradiations schemes were used: one to determine short-lived radionuclides (10sec) and other medium (1hr) or long-lived radionuclides (above 1hr). The samples for medium-lived were sent into the reactor for irradiation time of one hour and left overnight to decay before counting . The samples for long-lived were sent into the reactor for irradiation time of more than one hour and left for two weeks to decay before counting .

In the same manner, the samples for short-lived radionuclides were irradiated one after the other in their individual capsules for ten seconds and the counting done immediately. SL-7 (IAEA Standard Comparator) was used for the Pozzolana composition while GBW-7 (Chinese Standard Comparator) was used for the OPC compositional analysis.

3.3.5 The Shielding Experiment

X-ray at 0.5m with 150kVp energy and three cubes of concrete of thickness 15cm each, of Pozzolana Portland cement (PPC) in varied ratios of Pozzolana. Viz S_0 (0%), S_1 (10%), S_2 (20%), S_3 (30%), S_4 (40%), S_5 (50%), S_6 (60%), S_7 (70%) & S_8 (80%).

With I_0 established, I_x at 15cm, 30cm and 45cm respectively, of materials thickness were determined and tabulated. Plots of $\ln(I/I_0)$ against Materials thickness (x) produced gradients equal to the linear attenuation coefficients (μ) of the various compositions of the PPC. Radiological results interpretations then followed. Figures 3.12 to 3.18 showed the block diagram of X-ray set up, x-ray beam sizing at 0.5m from the source and laboratory work on x-ray shield analysis respectively.

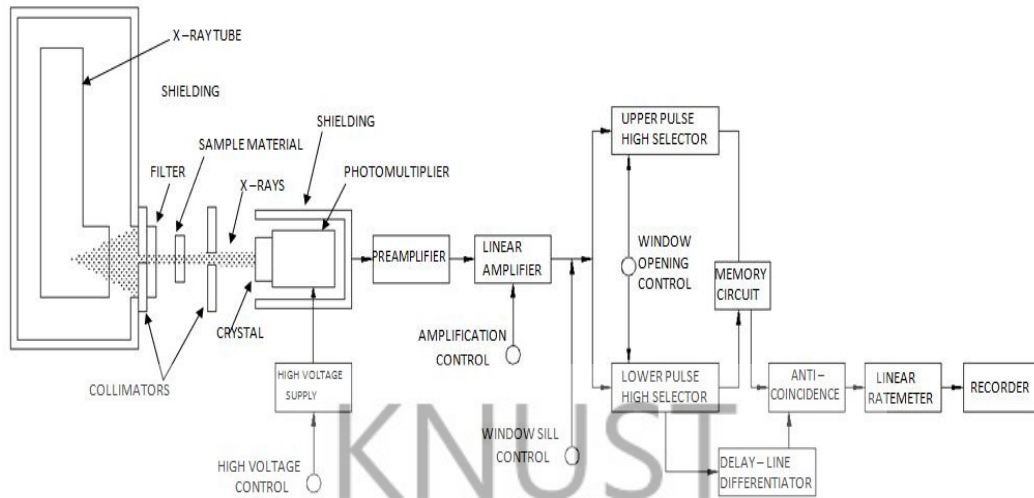


Figure 3.12: Block diagram of X-Ray equipment arrangement



Figure 3.13: X-ray incident beam sizing on concrete cube via Radiography



Figure 3.14: A sample being placed in front of X-ray source



Figure 3.15: Sample undergoing x-ray irradiation (15cm thick)



Figure 3.16: Sample undergoing x-ray irradiation (30cm thick)



Figure 3.17: Sample undergoing x-ray irradiation (45cm thick)



Figure 3.18: I_0 , I_{15} , I_{30} , I_{45} for the various PPC being recorded at the console

3.3.6 X-Ray Diffraction (XRD) Analysis

The optical transform experiment was performed with the lasers and the 35-mm slides to simulate single crystal X-ray diffraction. To study the structure of a molecule or an extended solid, a single crystal of the material was substituted for the 35-mm slide and use X-rays instead of visible light. The resulting diffraction patterns, captured as spots on photographic film or counts on an electronic detector, would provide information about the size and symmetry of the molecular unit cell. X-ray powder diffraction was a tool used for characterizing the products of a solid state synthesis reaction. At the simplest level, diffraction patterns was analyzed for phase identification, which is determining what crystalline substances are present in a given sample. More quantitatively, the peak positions was used to refine the lattice parameters for a given unit cell.

3.3.7 Methodology in Scanning Electron Microscopy

The Scanning Electron Microscopy was a scientific application method which was employed to produce images of samples of microscopic nature by scanning it with a focused beam of electrons. The instrument used in this exercise was known as the Scanning Electron Microscope (SEM).

Accelerated electrons in an SEM carry significant amounts of kinetic energy, and this energy was dissipated as a variety of signals produced by electron-sample interactions when the incident electrons are decelerated in the solid sample. These signals include secondary electrons (that produce SEM images), backscattered electrons (BSE), diffracted backscattered electrons (EBSD that were used to determine microstructures and orientations of the samples.

Essential components include the following:

- Electron Source ("Gun")
- Electron Lenses
- Sample Stage
- Detectors for all signals of interest
- Display / Data output devices
- Infrastructure Requirements: Power Supply, Vacuum System, Cooling system, Vibration-free floor, Room free of ambient magnetic and electric fields.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.0 Introduction

This chapter discusses the results of the compressive test, flexure test, ultrasonic test, neutron activation analysis (NAA) test, X-ray Shielding analysis, X-Ray Diffraction (XRD) analysis and Scanning Electron Microscopy (SEM) analysis.

The compressive strength of concrete is analysed with variation in age and mixture proportion. Subsequently, the influence of age and mixture proportion on the UPV – strength relationship of concrete is also investigated to ascertain results. The shielding properties of Pozzolana, OPC and PPC are analysed.

4.1 Analysis of Compressive Strength Results

Figures 4.1-4.3, showed the results of the compressive tests and the following deductions were made.

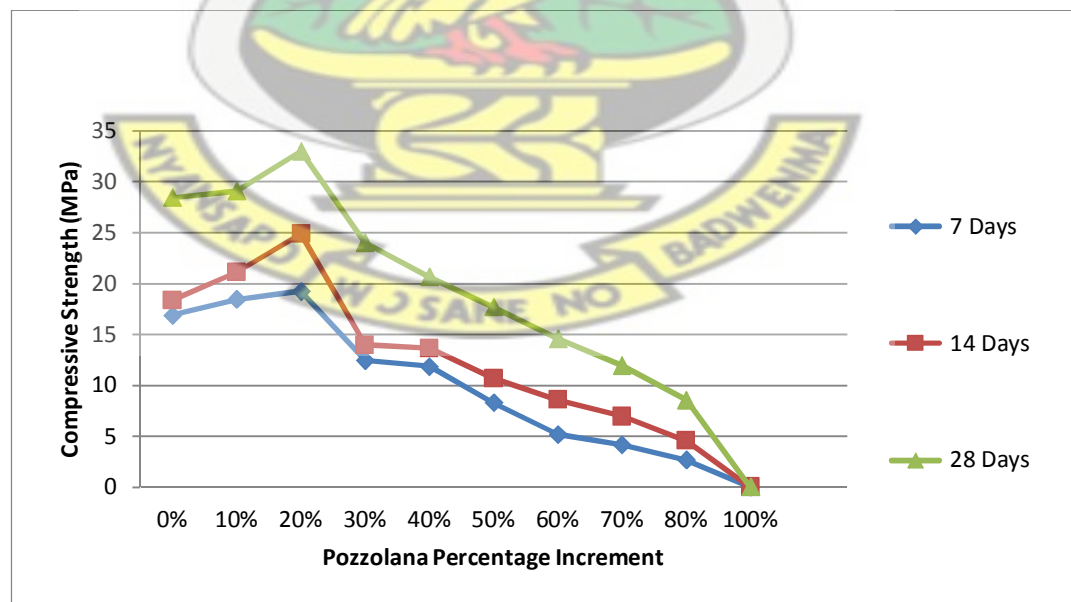


Figure 4.13: Compressive strength development over days: w/c 0.5

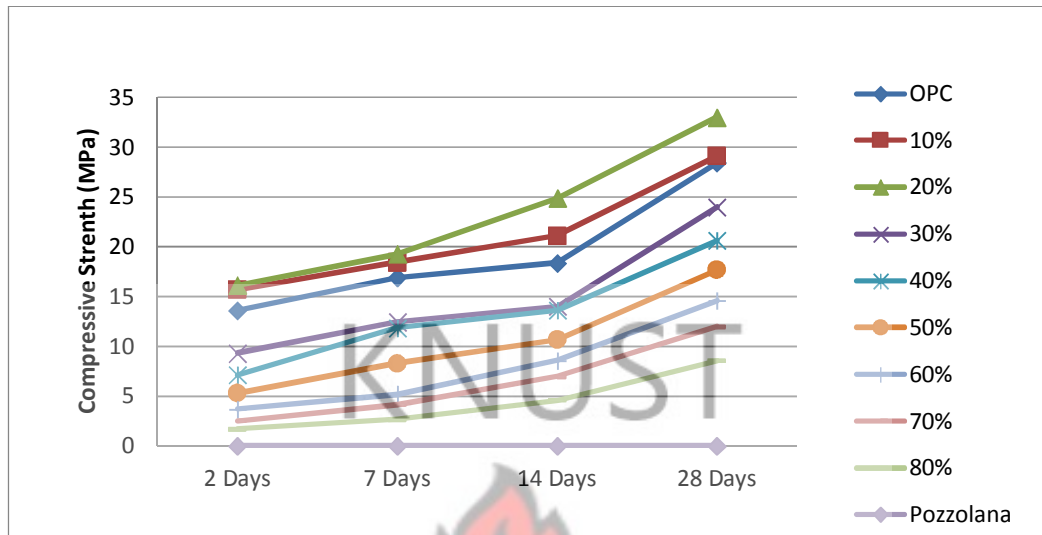


Figure 14.2 : Various PPC compressive strength evolution over days w/c 0.5

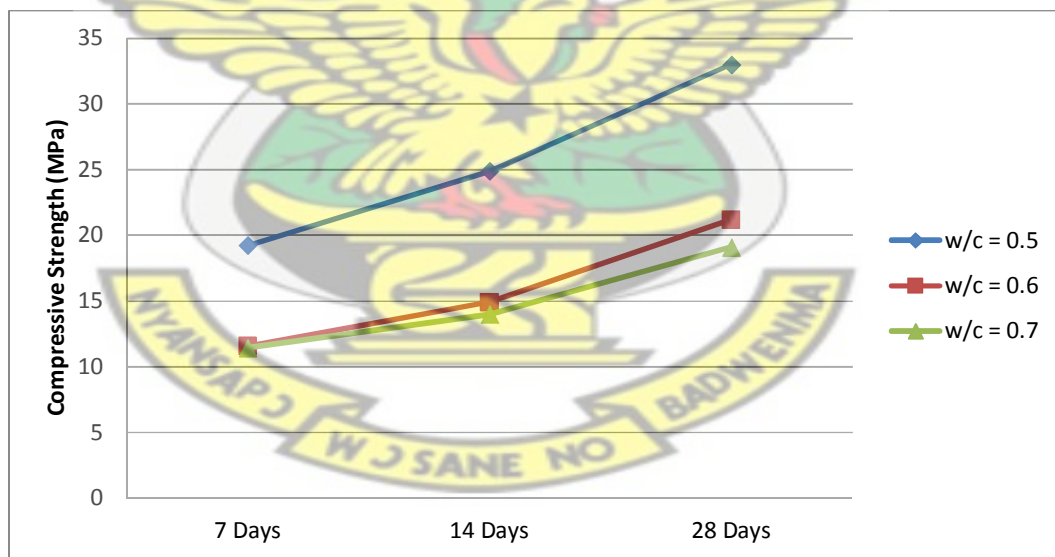


Figure 15: Compressive strength vs. age (days), as w/c varied for 20% Pozzolana

Figure 4.1 showed the evolution or continued increment of compressive strength with aging. In other words, the strength of concrete increase over days (7, 14 and 28 days), as twenty-eight (28) days strength has the highest value of compressive strength.

Figure 4.2 showed a summary of development of compressive strength of concrete over days of aging: 2, 7, 14 and 28 putting 20% PPC at the top. It is the best ratio (20% PPC) that exhibits highest compressive strength compared to the control (OPC).

The results in Figure 4.3 showed that concrete strength is inversely proportional to water/cement ratio. Thus as w/c increases, concrete strength decreases.

4.2 Flexural Strength Results for Confirmatory Test

In order to confirm the revealing results from the compressive test, the results from Figure 4.4 showed that 20% PPC had the highest modulus of rupture, an indication in conformity with the results from Figures 4.1 to 4.3.

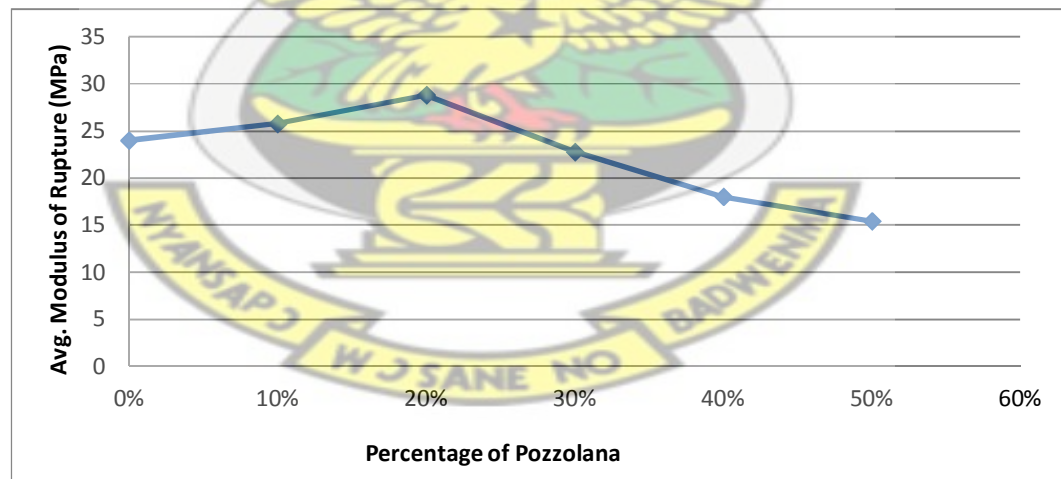


Figure 16 : Modulus of rupture vs Pozzolana percent

4.3 Verification of Compressive Test Results Using UT

Figures 4.5 a&b showed the results of the ultrasonic test analysis and the following inferences were made.

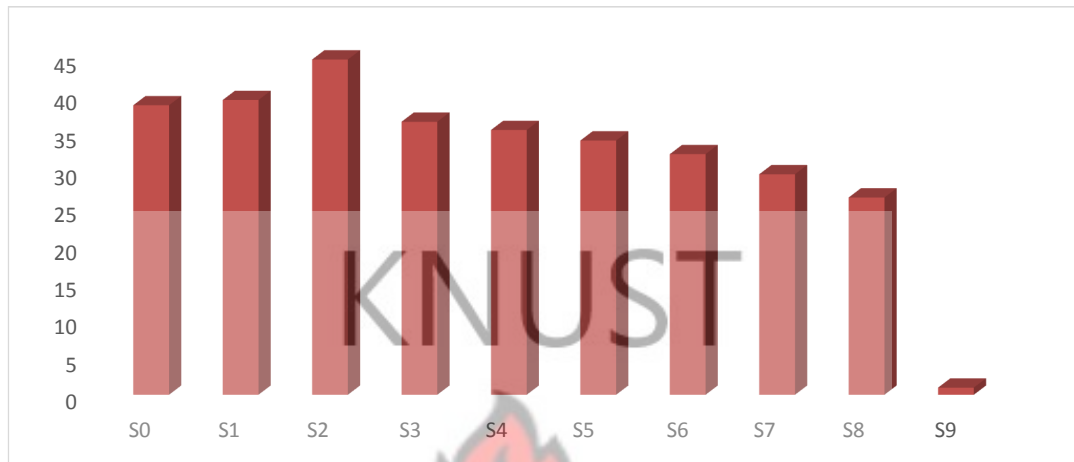


Figure 4.5 (a) Elastic modulus of PPC at day 28

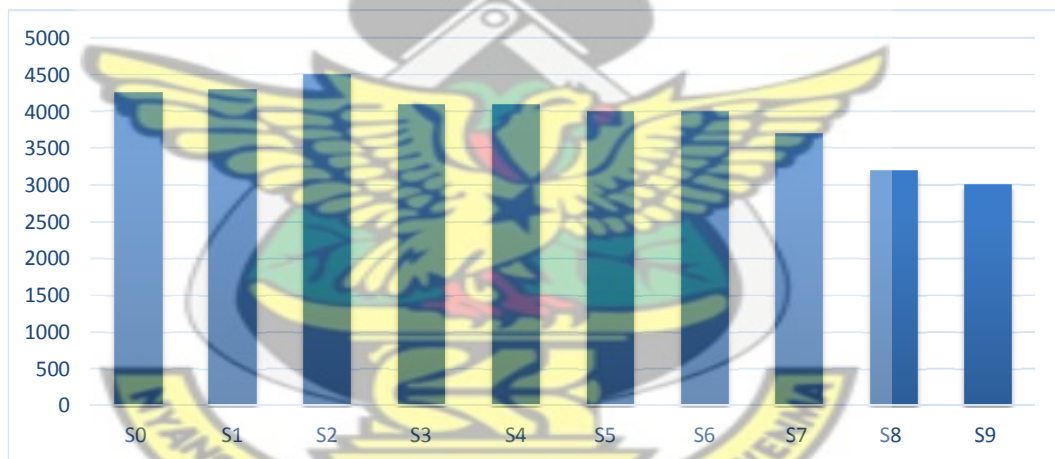


Figure 4.5 (b) Ultrasonic pulse velocity of PPC at day 28

In order to verify the validity of the variation in strength over the varied ratios of Portland Pozzolana concrete (PPC) mixes, additional specimens were cast for comparative or confirmatory tests using Ultrasonic test, where modulus of elasticity (E) was determined. The relationship between elastic modulus (E) and compressive strength is given by:

$$\text{Compressive stress } (\delta) = E\epsilon \quad (68)$$

where ϵ is strain according to Hooke's law (Callister, 2001 and Timoshenko and Gere, 1998). Equation (68) shows that, stress is directly proportional to elastic modulus. Figure 4.5(a), therefore, showed elastic modulus of 20% PPC to be of the highest value.

Equation (22) showed that UPV or V_p is directly proportional to elastic modulus (E). As such, in Figure 4.5(b), pulse velocity was highest for 20% PPC, the higher the pulse velocity the better the material strength (excellence) as shown in Table 2.12 (Quality of concrete as a function of the UPV).

It is evident from Figures 4.5(a & b) that, when no Pozzolana was introduced in the concrete formulation (OPC), the values of elastic modulus as well as UPV were low. These values however increased as the content of Pozzolana increased in the formulation until the point when 20 % Pozzolana introduction was reached. Figure 4.5(b), therefore, indicated that 20% PPC was of excellent quality.

4.4 Shielding Property Analysis of PPC (Elemental Composition)

Figure 4.6 showed the elemental composition in Pozzolana cement, with Fe, followed by Al, being in significant quantity. Figure 4.7 then showed the respective densities of these elements in Pozzolana and OPC. In Figure 4.8, common elements found in both Pozzolana and ordinary Portland cements were shown with Fe being the highest with significant shielding properties in Pozzolana.

In Figure 4.9, linear attenuation coefficients of these elements were shown, namely, Fe, Cu, Al, Ca, K and Na with Cu being the highest followed by Fe. Referenced to the cumulative shielding effect of elements, Pozzolana showed to have a better shielding

potential when compared to OPC. Figure 4.10 showed elements of significant macroscopic cross sections found in Pozzolana cement. These elements were Eu, Hf and Co with Eu having the highest macroscopic cross section as shown in Tables 4.1 and 4.2, putting Pozzolana cement as a material of higher shielding potential, compared to OPC. Figures 4.11 to 4.19, of various linear attenuation coefficients/curves confirmed this assertion.

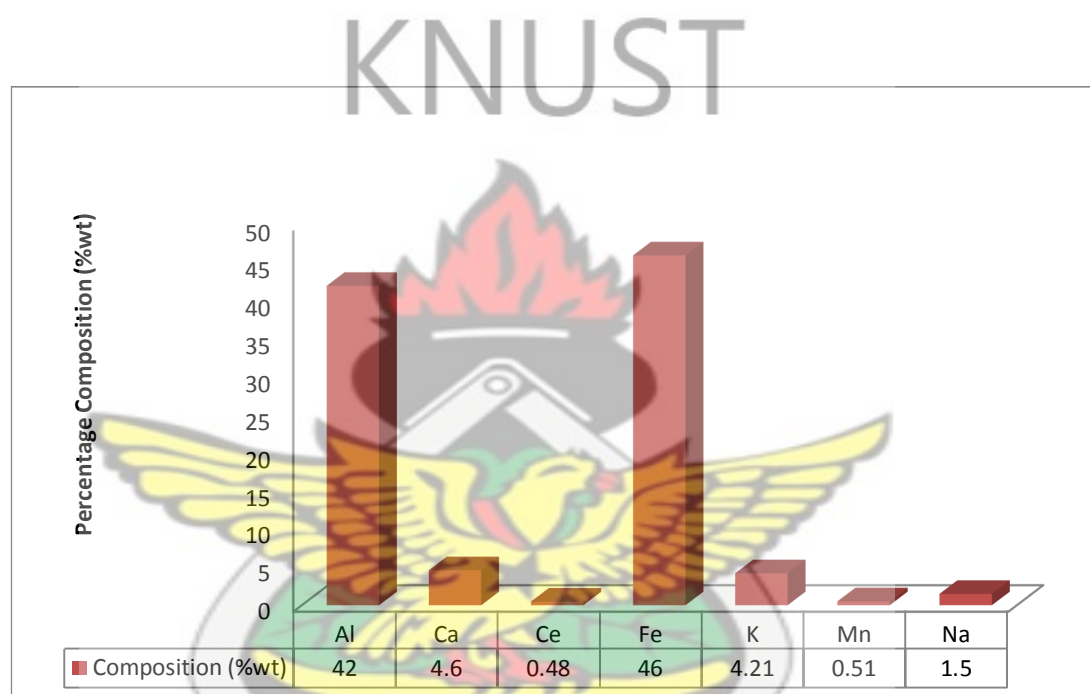


Figure 4.6: Percentage composition (% wt) of some significant elements in Pozzolana

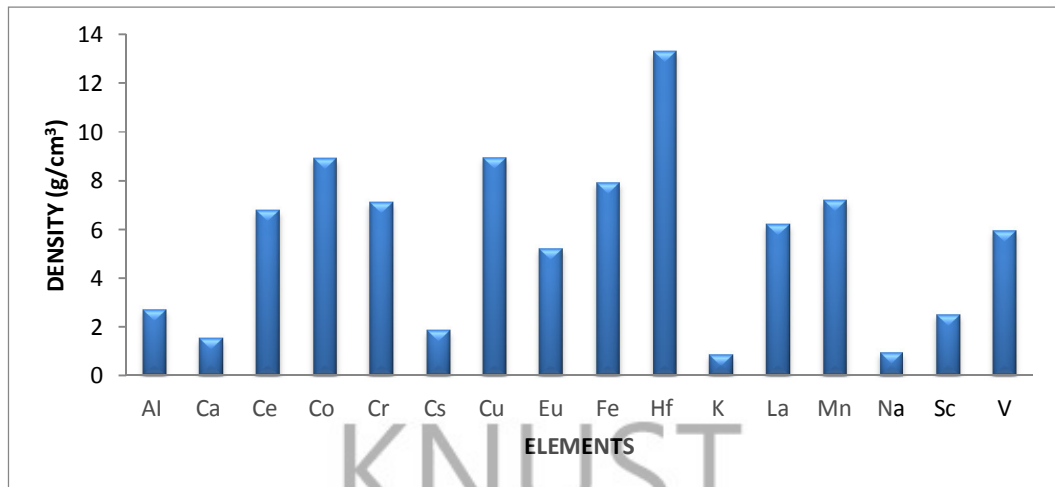


Figure 4.7: Density of some elements in Pozzolana or OPC

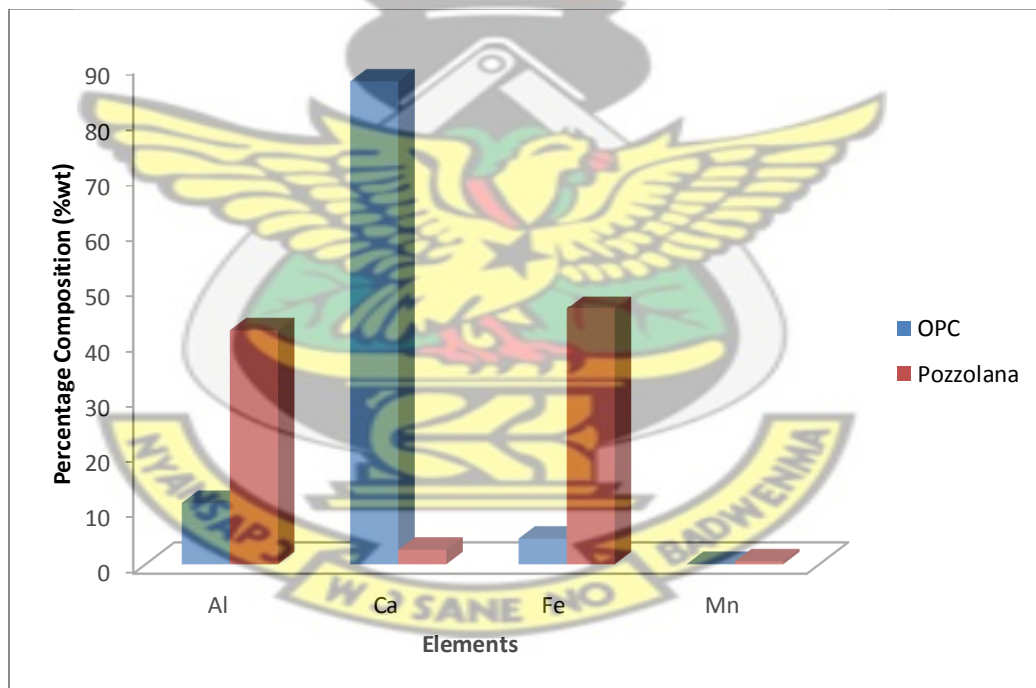


Figure 4.8: Elements in OPC and Pozzolana of significant shielding characteristics

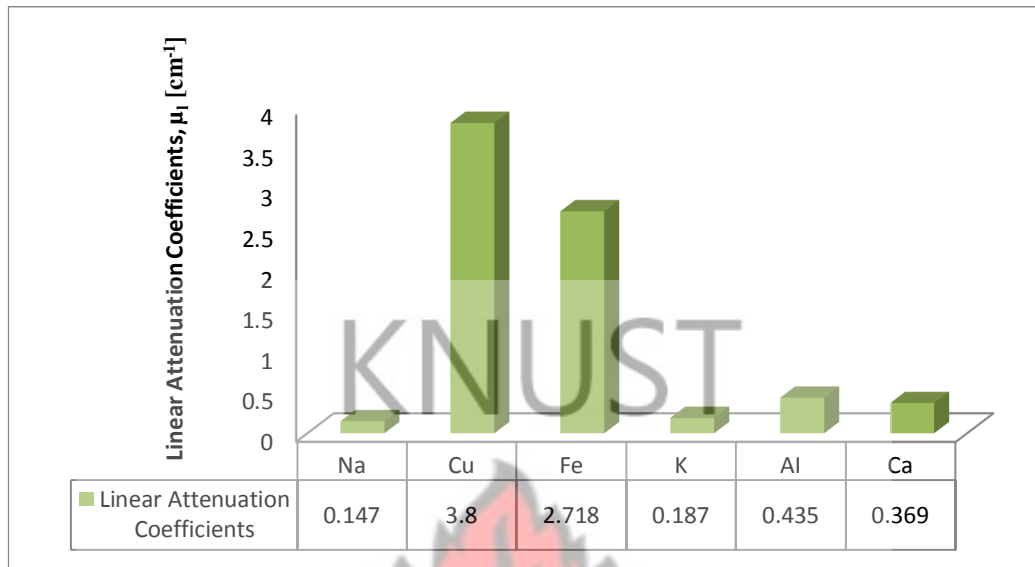


Figure 4.9: Linear attenuation coefficients of some elements in Pozzolana / OPC

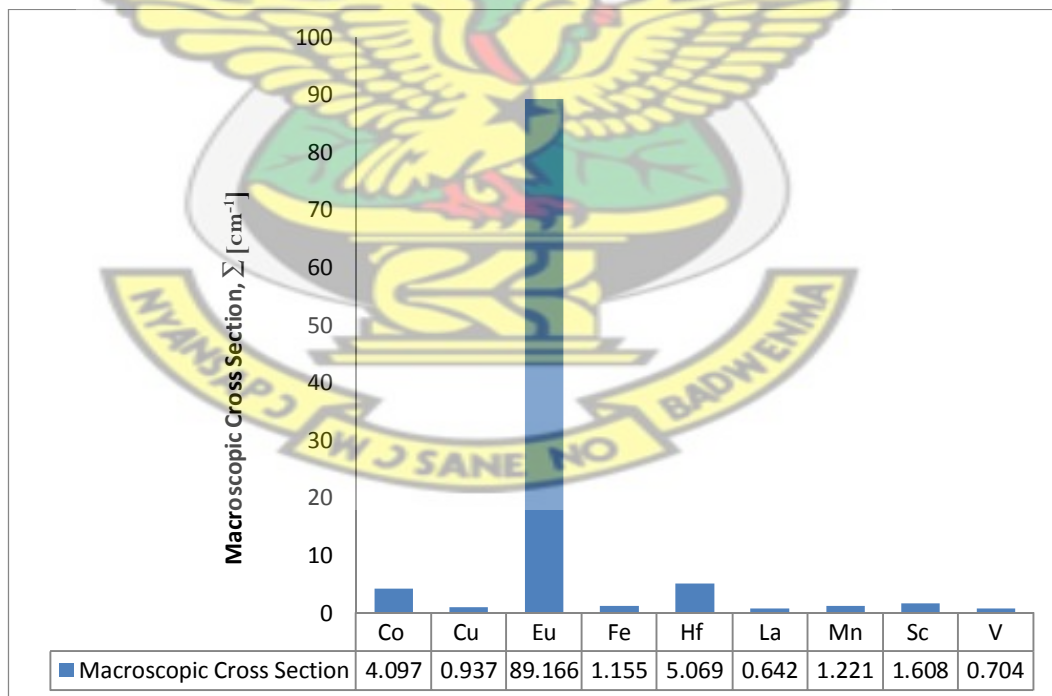


Figure 4.10: Macroscopic cross section of some elements in Pozzolana / OPC

Table 4.1: Macroscopic Cross Section Interpretation for Pozzolana

Elements	Composition (% wt)	Macroscopic Cross Section, Σ [cm ⁻¹]	(%wt) x (Σ)
Al	42	0.099	0.042
Ca	4.60	0.080	0.004
Ce	0.48	0.284	0.0014
Co	0.05	4.097	0.002
Cr	0.17	0.502	0.0009
Cs	0.15	0.408	0.0006
Cu	0.04	0.937	0.0004
Eu	0.008	89.166	0.00713
Fe	46	1.155	0.5313
Hf	0.08	5.069	0.0041
K	4.21	0.048	0.002
La	0.009	0.642	0.00006
Mn	0.51	1.221	0.006
Na	1.50	0.115	0.0017
Sc	0.03	1.608	0.00048
V	0.04	0.704	0.0003
Total Σ_T [cm⁻¹]			0.6044 cm⁻¹

Table 4.2: Macroscopic Cross Section Interpretation for OPC

Elements	Composition (% wt)	Macroscopic Cross Section, Σ [cm ⁻¹]	(%wt) x (Σ)
Al	11	0.099	0.011
Ca	87	0.080	0.07
Cu	0.006	0.937	0.00006
Fe	4.6	1.155	0.053
Mg	6.5	0.158	0.0103
Mn	0.06	1.221	0.0007
Ti	0.4	0.608	0.0024
Total Σ_T [cm⁻¹]			0.15 cm⁻¹

In calculations, it was determined whether or not Pozzolanic materials have a better shielding property than the ordinary Portland cement (OPC).

For gamma-ray and X-ray shielding analysis, linear attenuation coefficients μ_1 , [cm^{-1}] was considered. The higher the μ_1 , the better the material for shielding. This relation is given by:

$$\frac{I}{I_0} = B e^{-\mu x} = B_T = K = \frac{Pd^2}{WUT} \quad (\text{Referenced to equation 36e})$$

Whereas, for neutron shielding, total macroscopic cross section Σ_T , [cm^{-1}] was considered, the higher the Σ_T , the better the material for neutron shielding. The relation is given by:

$$\Phi(x) = \Phi_0 e^{-\Sigma_T x} \quad (\text{Referenced to equation 16})$$

It was therefore, shown in Tables 15 and 16 above, that total macroscopic cross section (Σ_T) of **Pozzolana** is greater than that of **OPC** hence Pozzolana is a better shielding material than OPC.

4.5 Shielding Property Analysis of PPC (Attenuation Coefficient)

While Figure 4.11 showed the linear attenuation curve for OPC as Control sample, Figures 4.12 to 4.19 showed the linear attenuation curves for the various PPC compositions drawn from the X-ray shielding experiment. In summary, Figure 4.20 showed linear attenuation coefficients for the various PPC test samples deduced from the shielding experiment, meanwhile 50% PPC (**S₅**) exhibited highest shielding property (1.53 cm^{-1}).

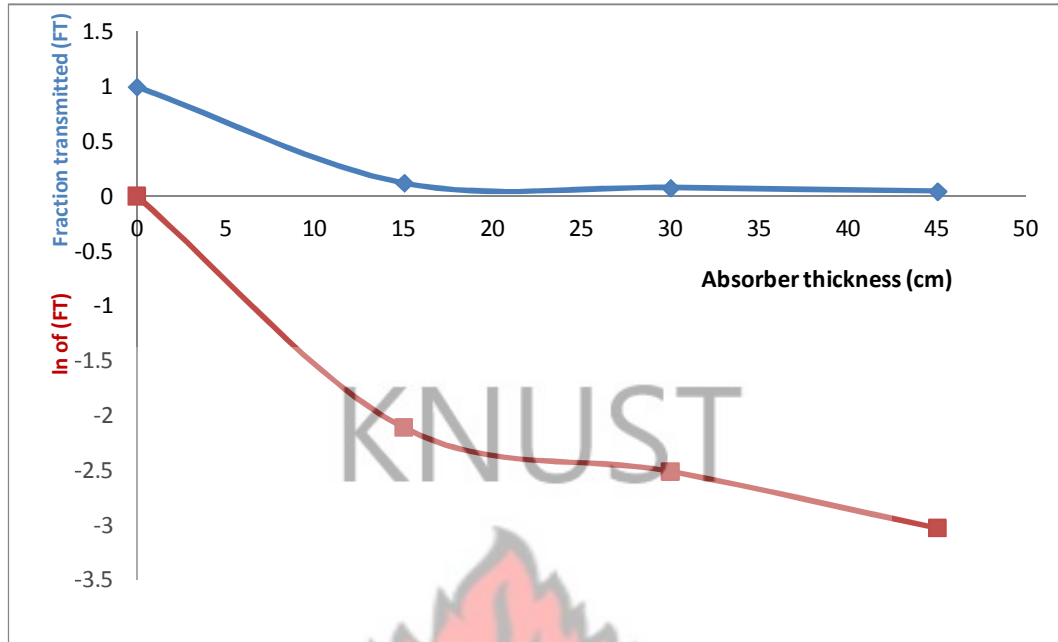


Figure 4.11: Linear attenuation curve for S_0

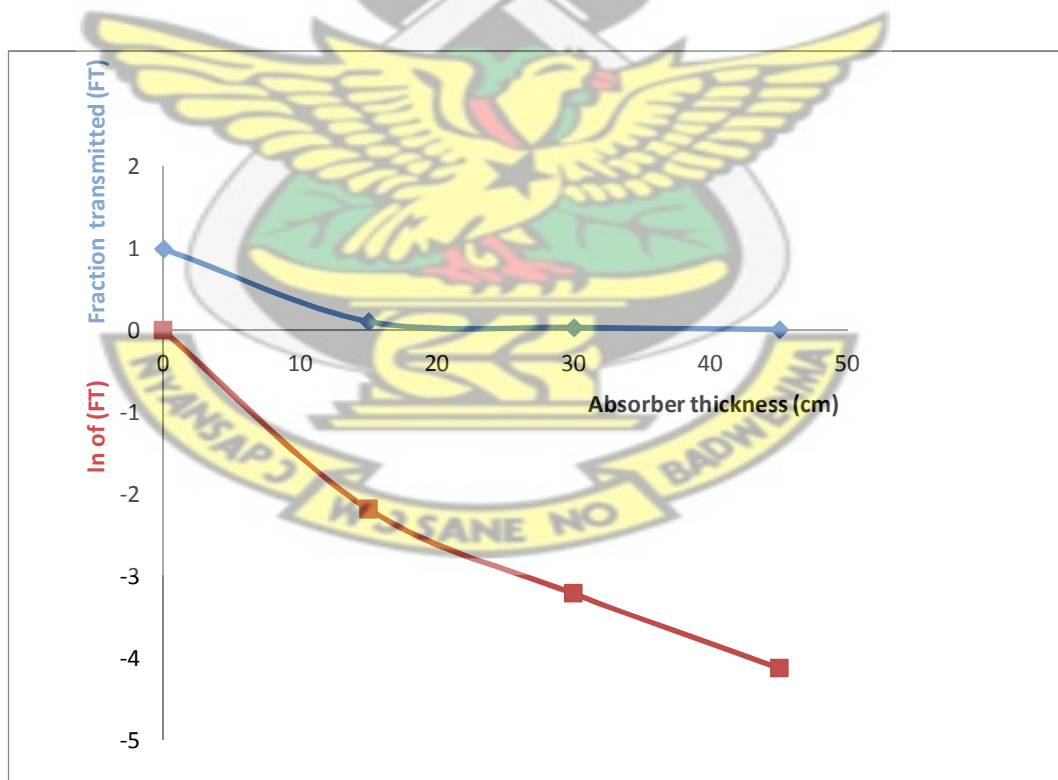


Figure 4.12: Linear attenuation curve for S_1

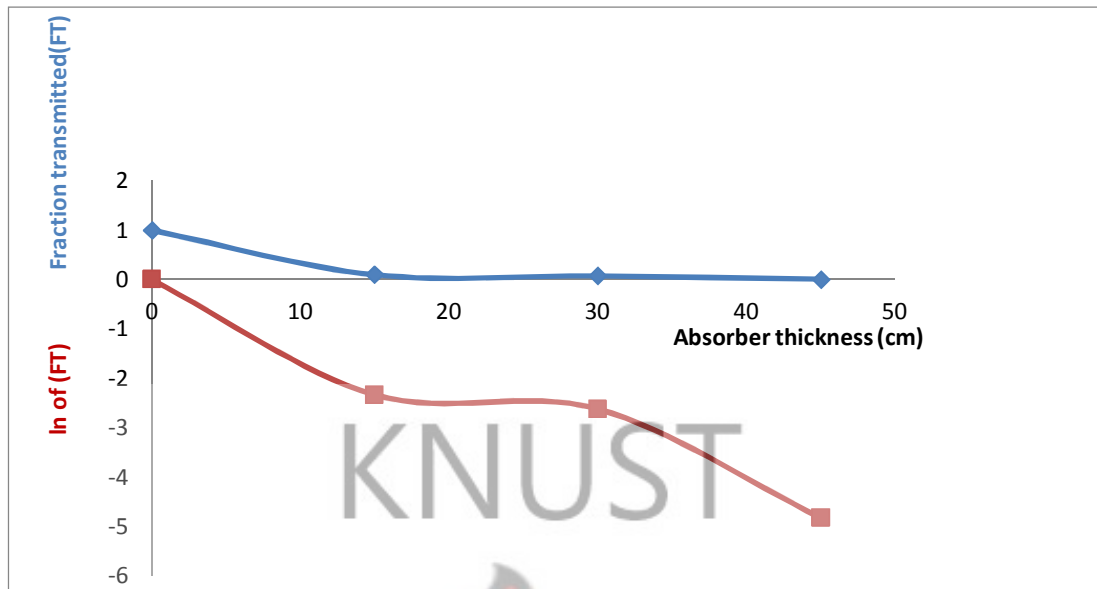


Figure 4.13: Linear attenuation curve for S_2

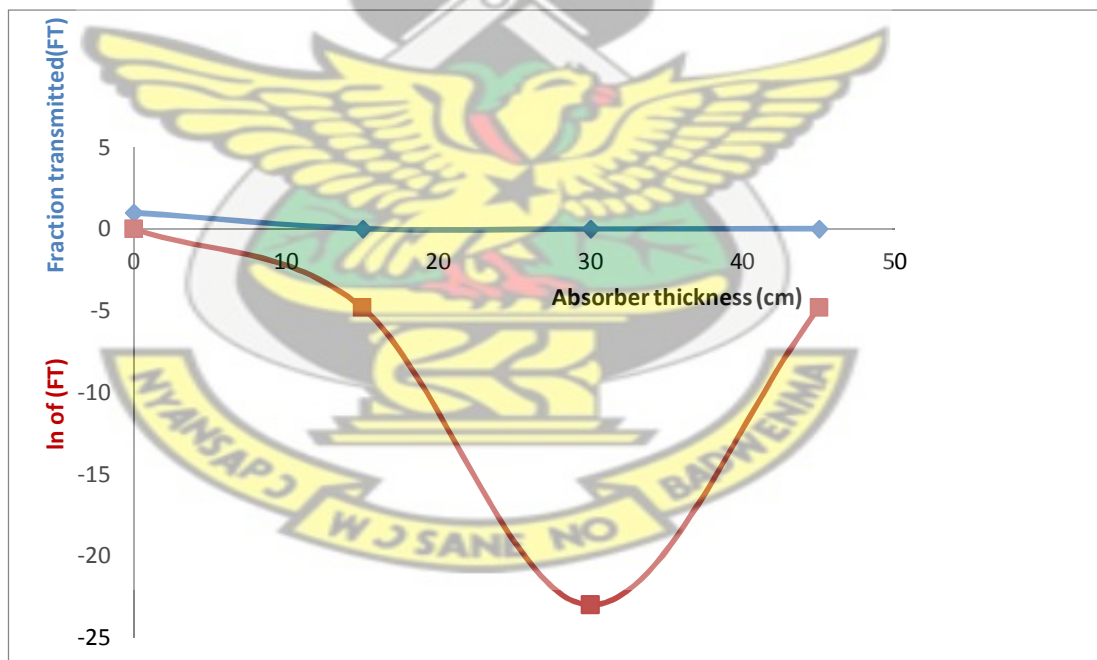


Figure 4.14: Linear attenuation curve for S_3

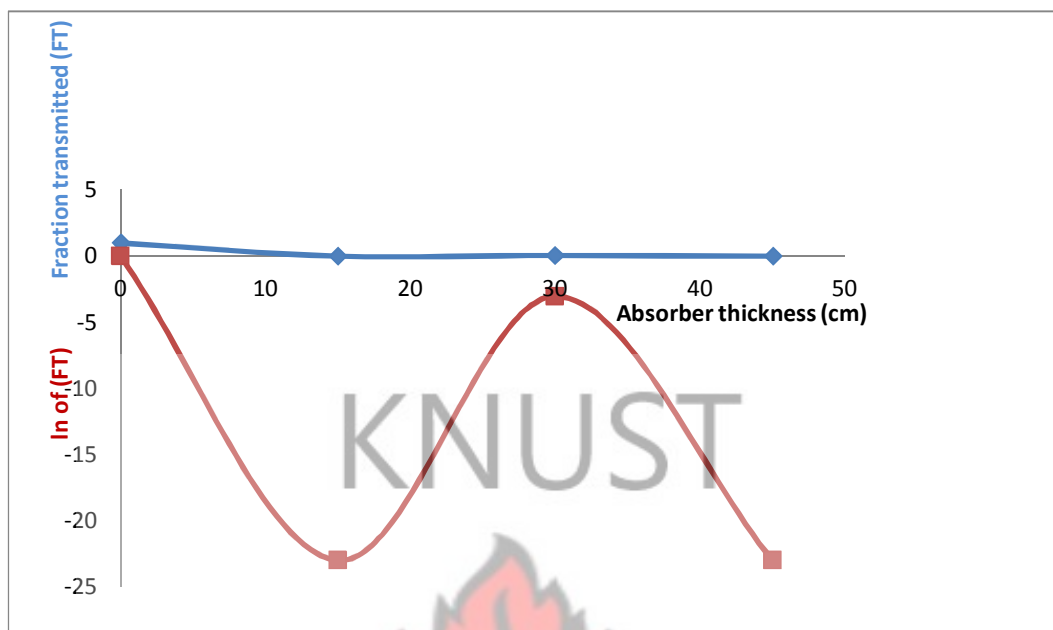


Figure 4.15: Linear attenuation curve for S₄

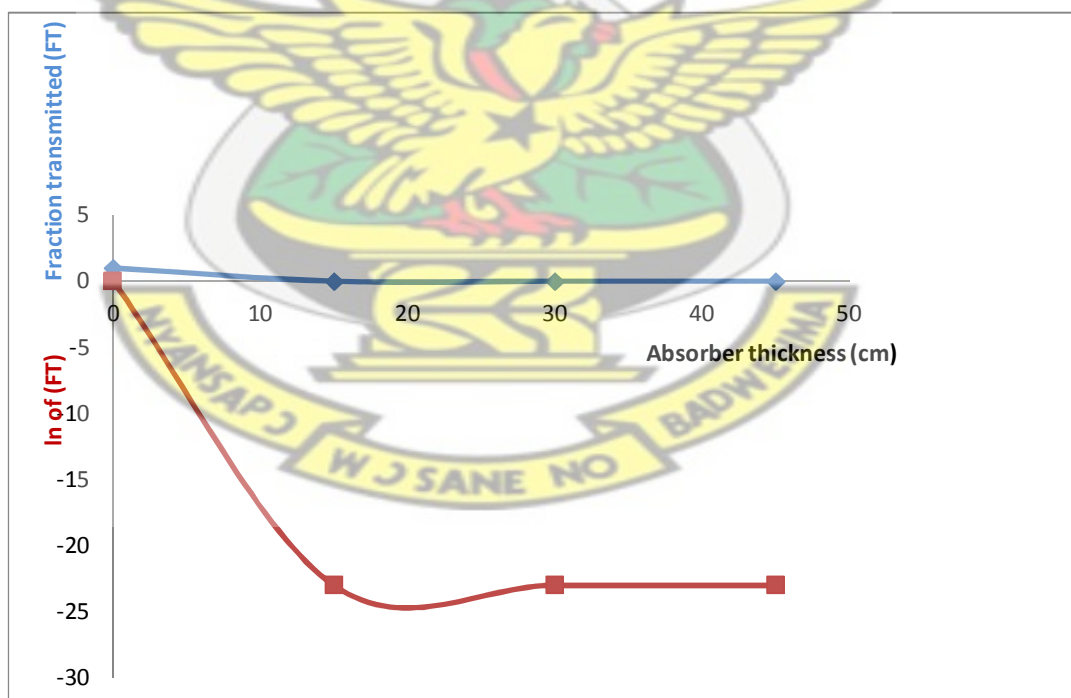


Figure 4.16: Linear attenuation curve for S₅

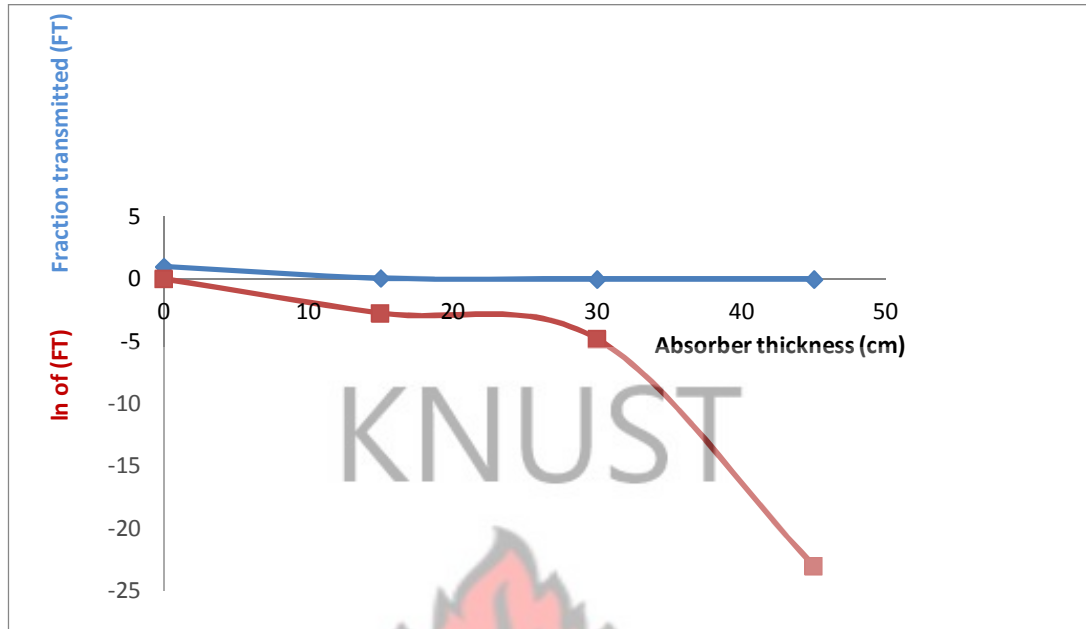


Figure 4.17: Linear attenuation curve for S_6

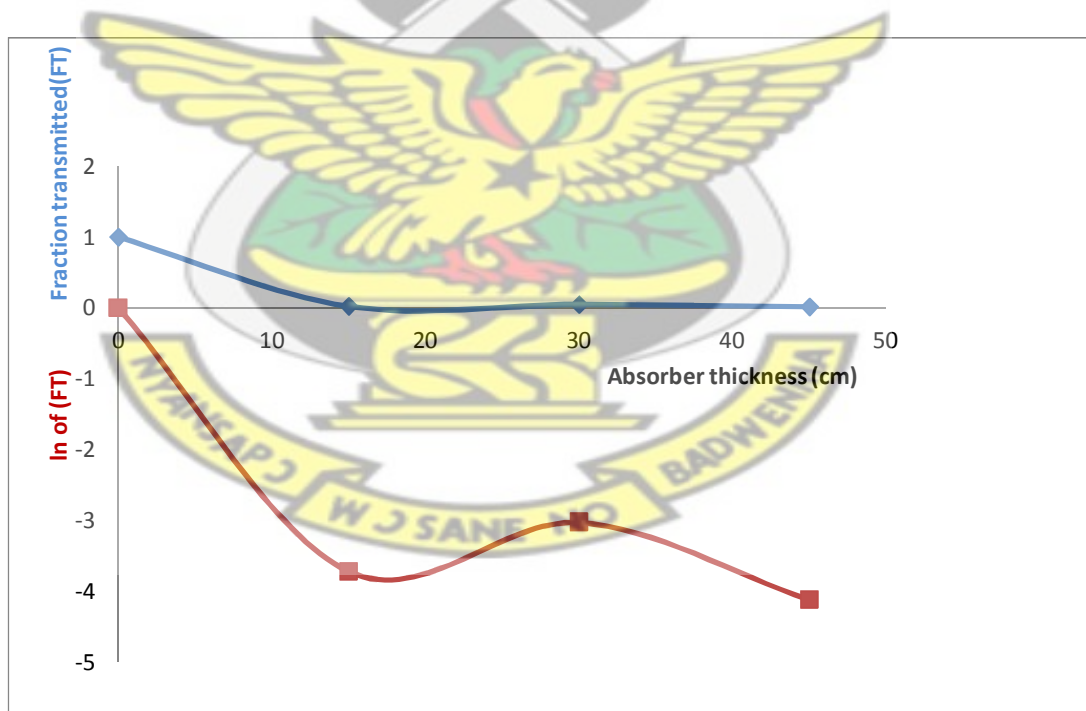


Figure 4.18: Linear attenuation curve for S_7

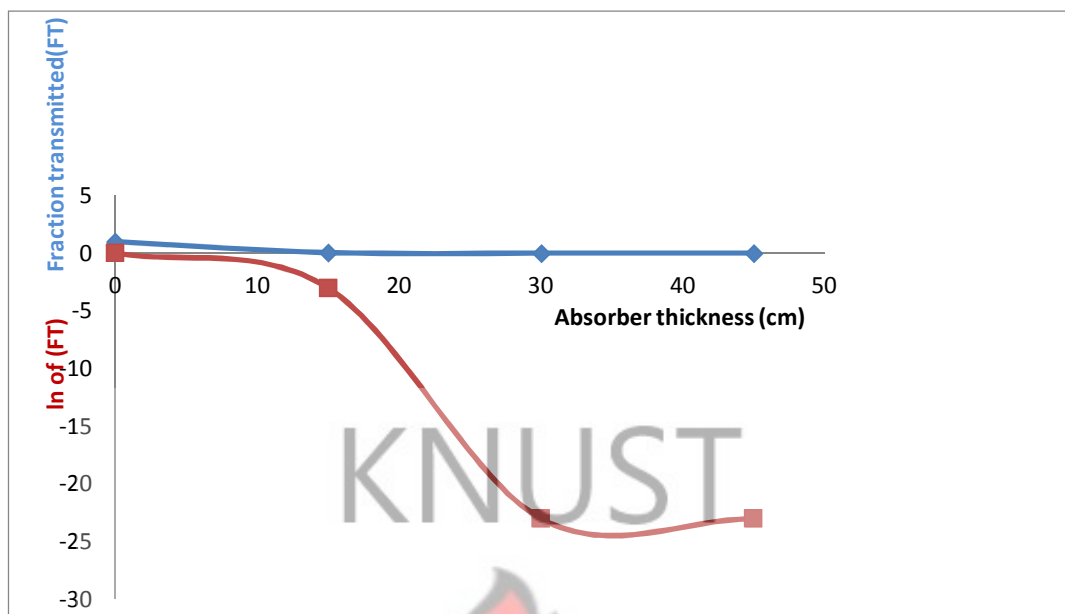


Figure 4.19: Linear attenuation curve for S₈

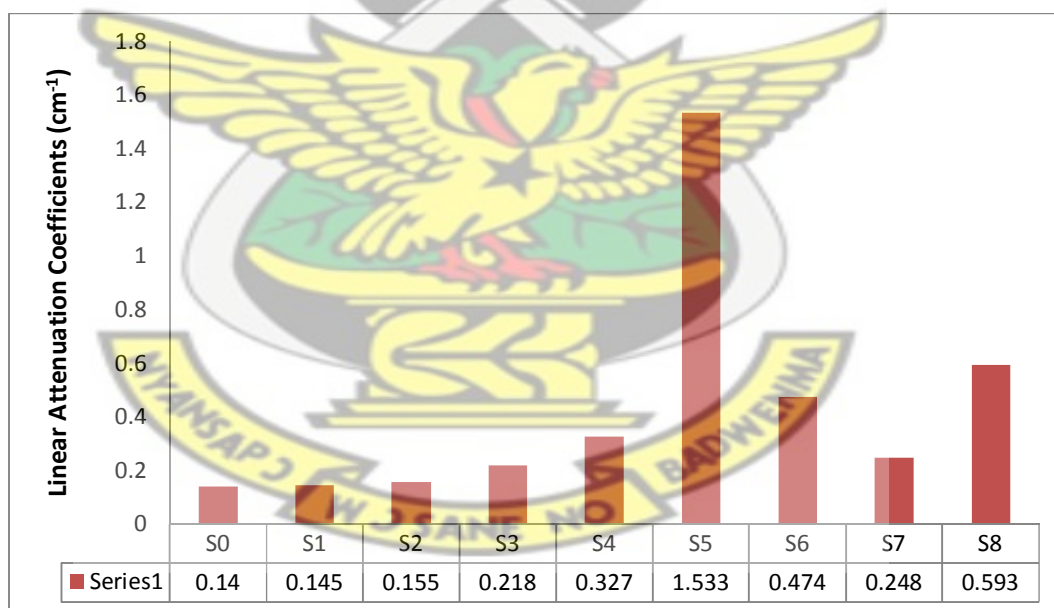


Figure 4.20: Linear attenuation coefficients of various PPC compositions

4.6 Crystal Structural Analysis of PPC (Phases Contributing to Strength and Shielding in Pozzolana Portland Concrete)

In Figures 4.21(a&b) to 4.30 (a&b), the X-Ray Diffraction analysis showed the following:

1. Figures 4.21(a&b) showed that in S_0 , calcium carbonate, $\text{Ca}(\text{CO}_3)$ with Rhombohedral crystal structure; and Calcite, CaCO_3 with Hexagonal crystal structure; were present in the matrix of silicon oxide. The percentages abundance of Calcite and calcium carbonate in this matrix were 14% and 12% respectively.
2. Figures 4.22 (a&b) indicated that S_1 had the same amount of Calcite and calcium carbonate. Each had percentage abundance of 8% in the matrix.
3. Figures 4.23 (a&b) showed that, in S_2 ; only Calcite was present in the matrix of silicon oxide. The Hexagonal crystallographic nature of Calcite with APF of 74%, therefore, contributed to the maximum strength exhibited in the S_2 . This was an indication that, the higher the percentage abundance of Calcite in the matrix, the stronger the concrete.
4. In S_3 , Figures 4.24 (a&b), beside Calcite, calcium carbonate phase (metastable) re-appeared in the matrix, therefore, rendering the strength in S_3 lower than that of (S_2).
5. Figures 4.25 (a&b), showed that, from 40% PPC (S_4) and above, CaCO_3 phase was overshadowed in the matrix. Phases such as Copper (Cu), Iron (Fe), Aluminum Hydroxide ($\text{Al}(\text{OH})_3$) and Manganese (Mn) mostly in their oxide forms, which are shielding-property enhancing elements/compounds, then built up contributing to improvement in the radiological shielding properties of the matrix.

6. In Figures 4.26 (a&b), this radiological shielding property improvement characteristic became maximum at 50% PPC replacement (S_5) due to the cumulative effect of the characteristic elements/compounds mentioned above.
7. Figures 4.27 – 4.29 (a&b) showed that the radiological shielding property improvement characteristic elements/compounds became minimal in the matrix of silicon oxide. This had enhanced the shielding potential of S_6 , S_7 and S_8 (Figure 4.20).

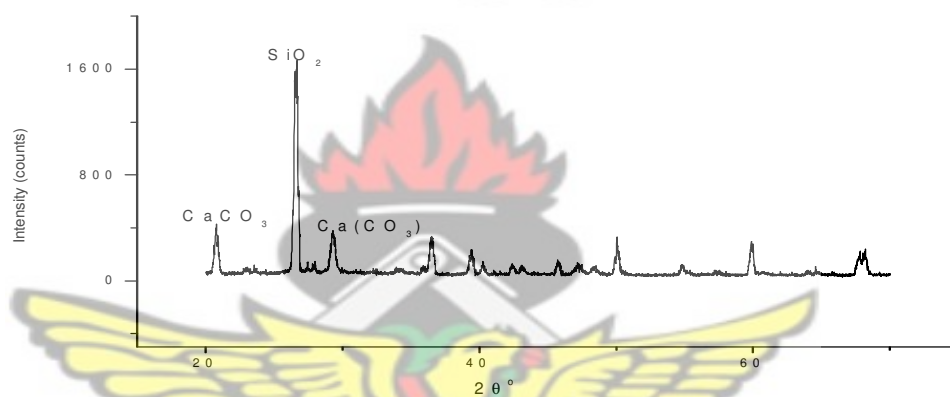


Figure 4.21 (a): X-Ray diffraction graph of S_0 (Control):



Figure 4.21 (b): Percentage Abundance of Compounds in S_0

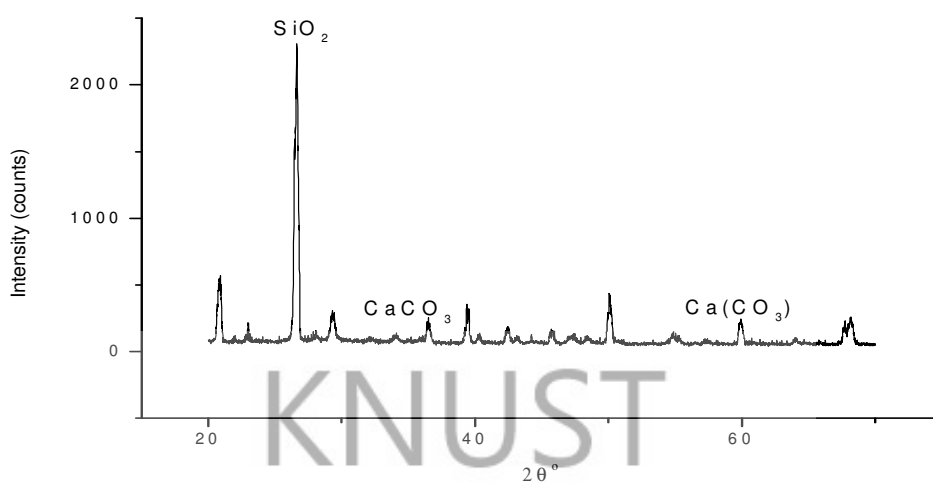


Figure 4.22 (a): X-Ray diffraction graph of S₁

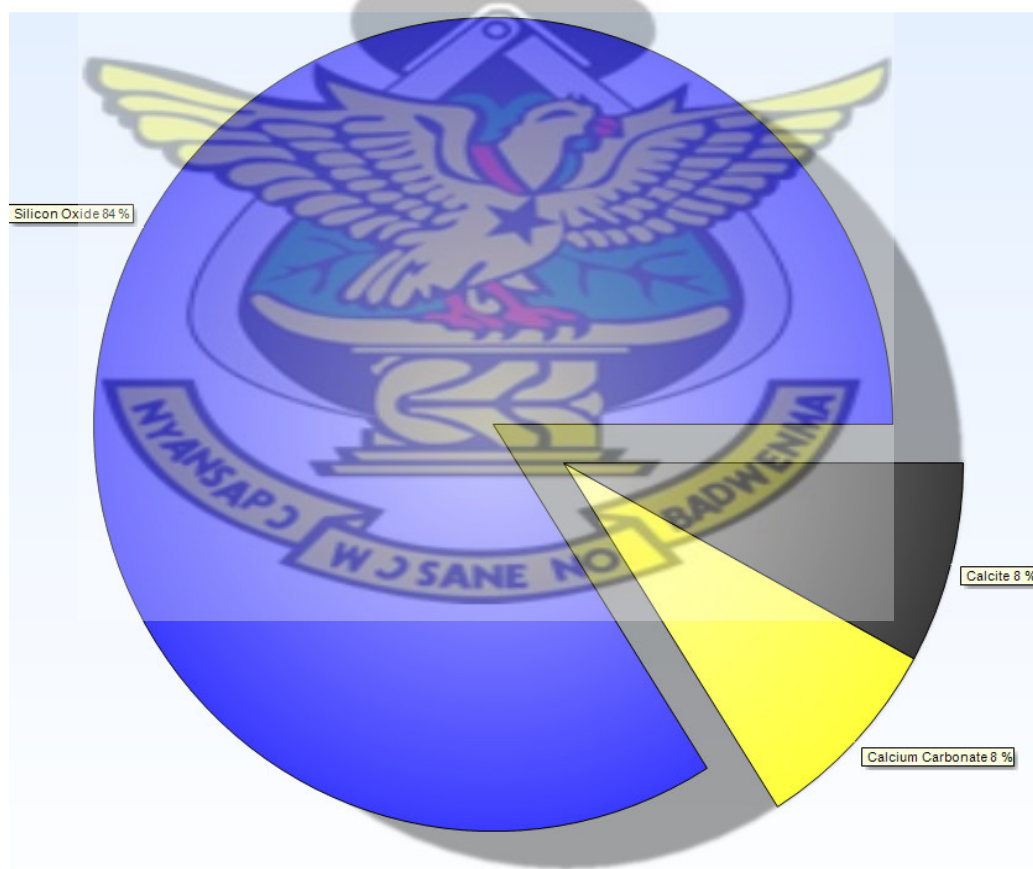


Figure 4.22 (b): Percentage Abundance of Compounds in S₁

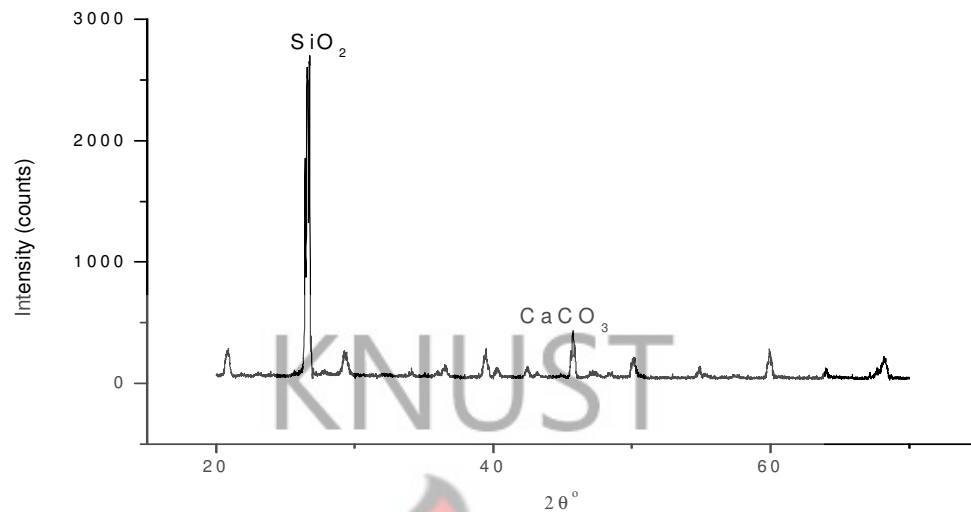


Figure 4.23 (a): X-Ray diffraction graph S_2



Figure 4.23 (b): Percentage Abundance of Compounds in S_2

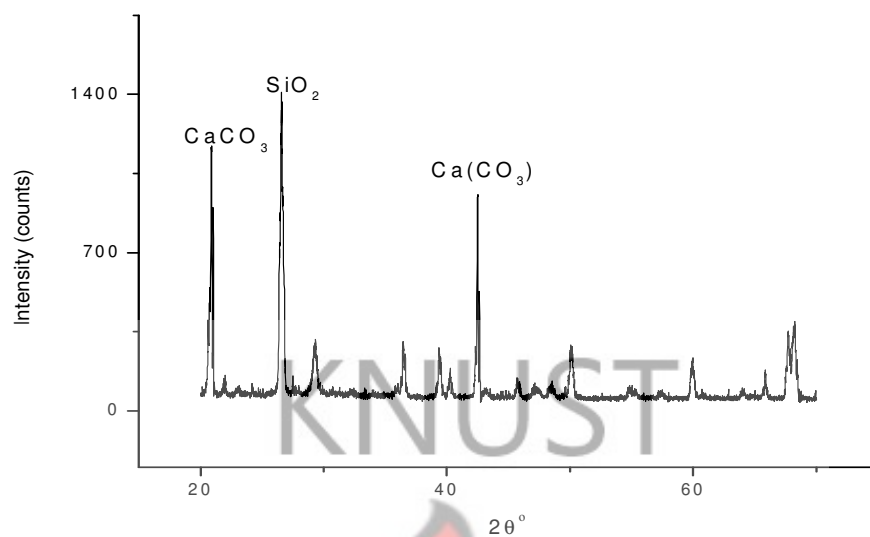


Figure 4.24 (a): X-Ray diffraction graph of S_3



Figure 4.24 (b): Percentage Abundance of Compounds in S_3

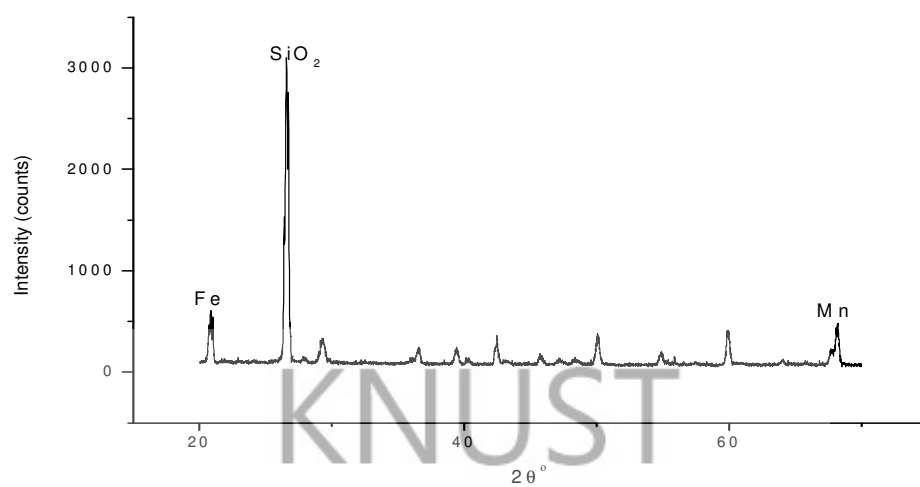


Figure 4.25 (a): X-Ray diffraction graph of S₄



Figure 4.25 (b): Percentage Abundance of Compounds in S₄

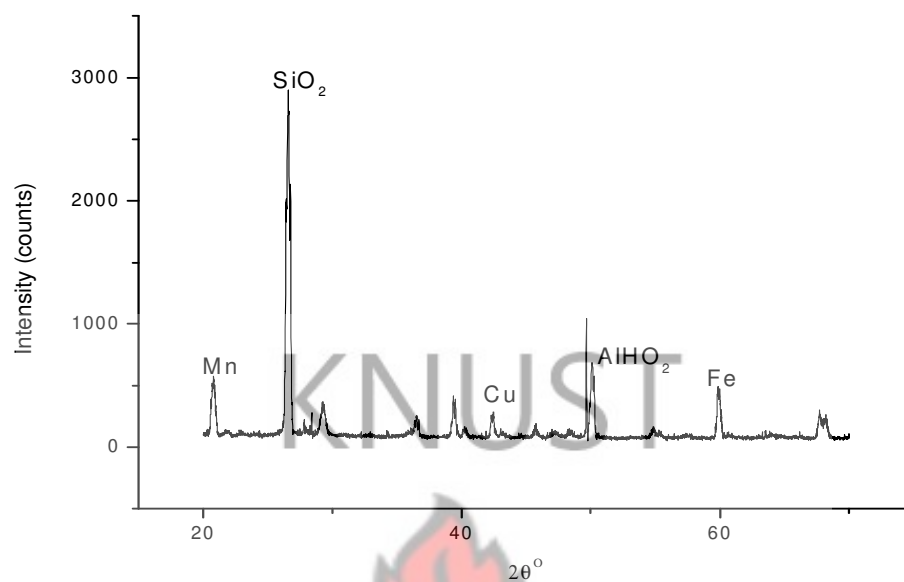


Figure 4.26 (a): X-Ray diffraction graph of S₅

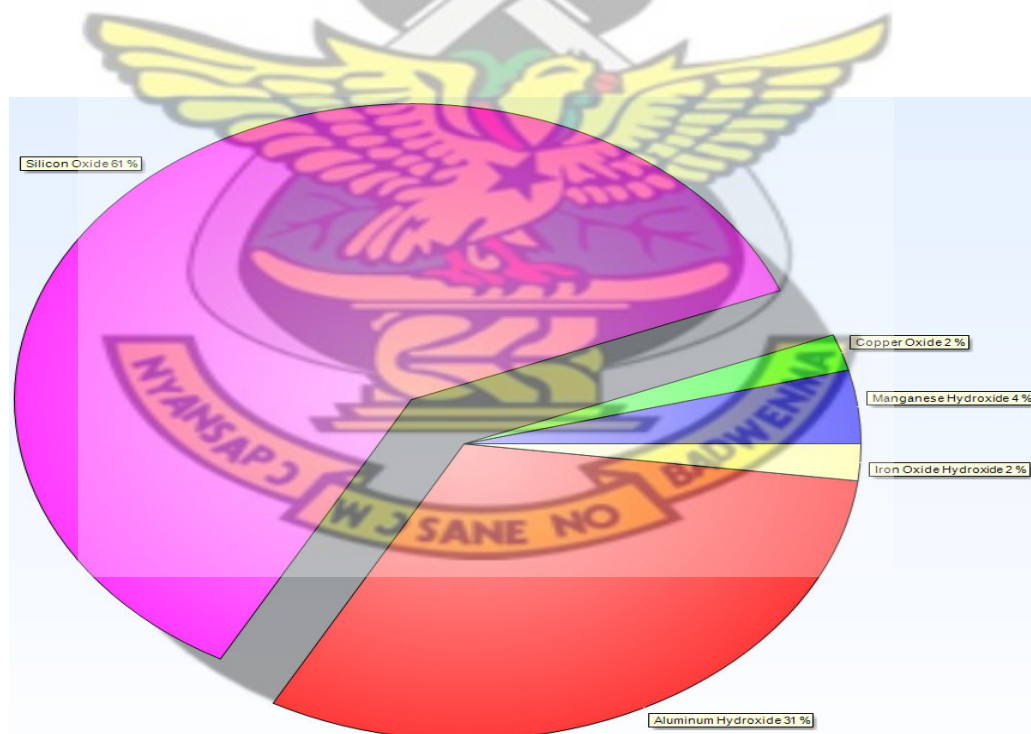


Figure 4.26 (b): Percentage Abundance of Compounds in S₅

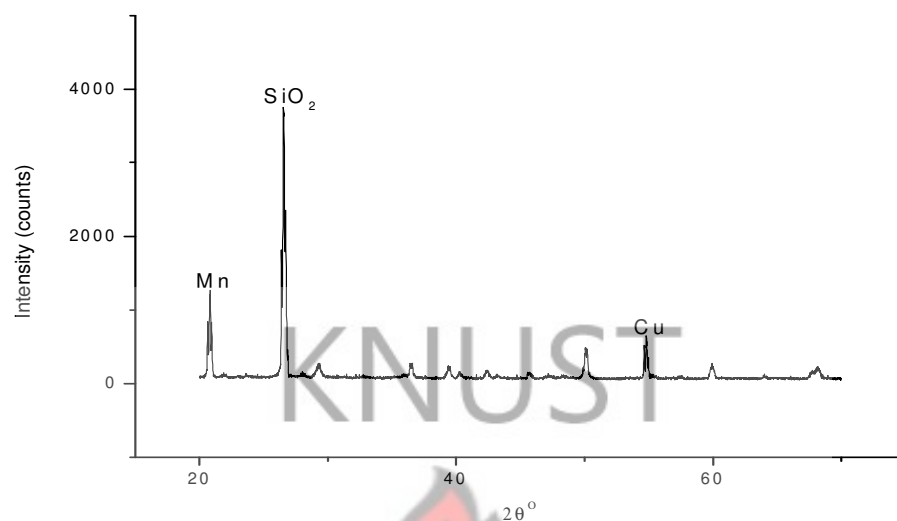


Figure 4.27 (a): X-Ray diffraction graph of S_6



Figure 4.27 (b): Percentage Abundance of Compounds in S_6

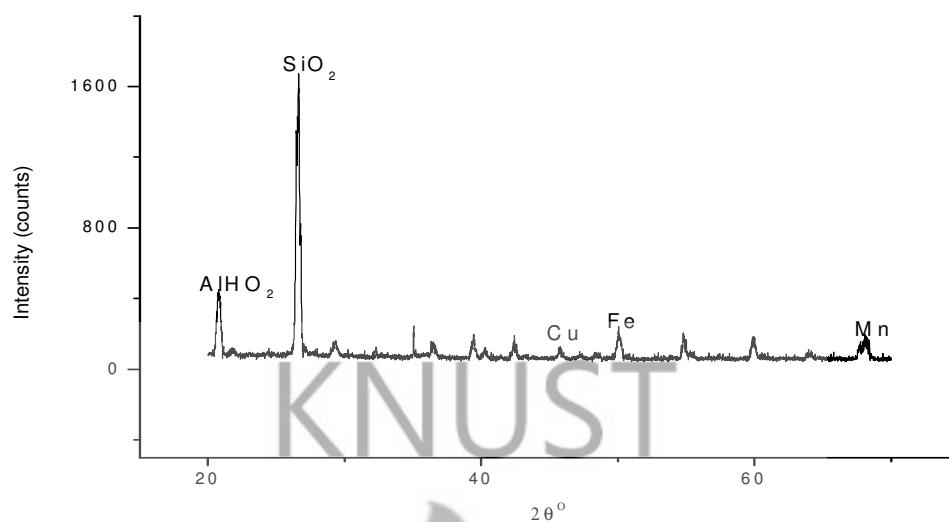


Figure 4.28 (a): X-Ray diffraction graph of S7

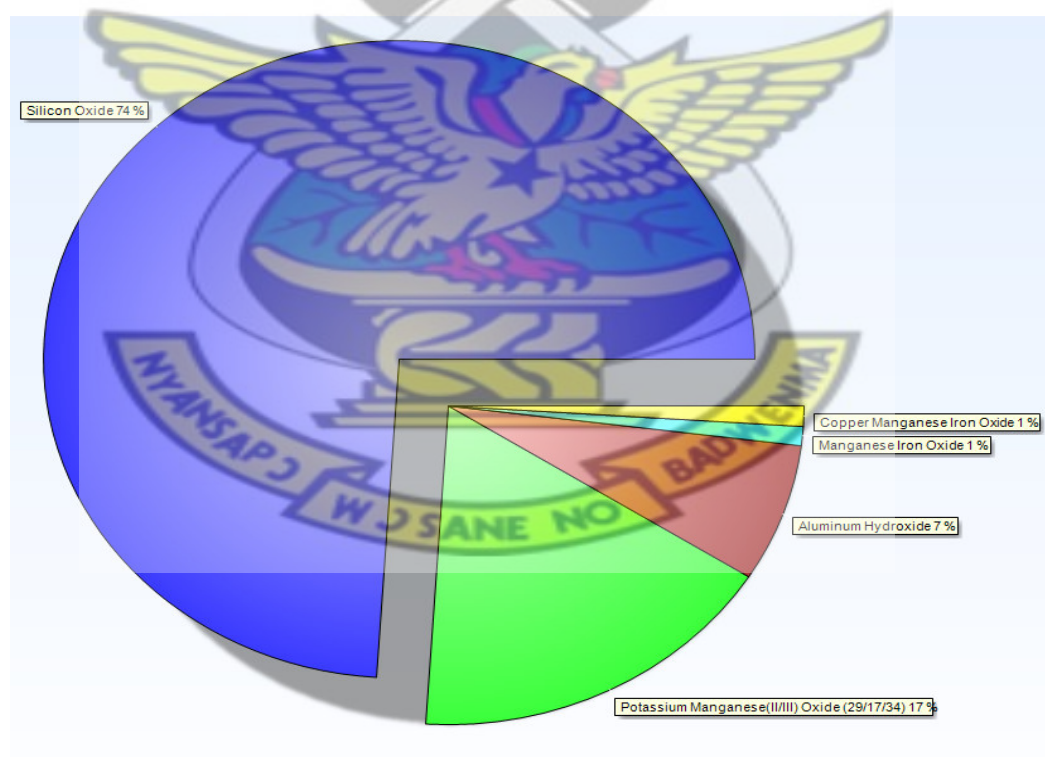


Figure 4.28 (b): Percentage Abundance of Compounds in S7

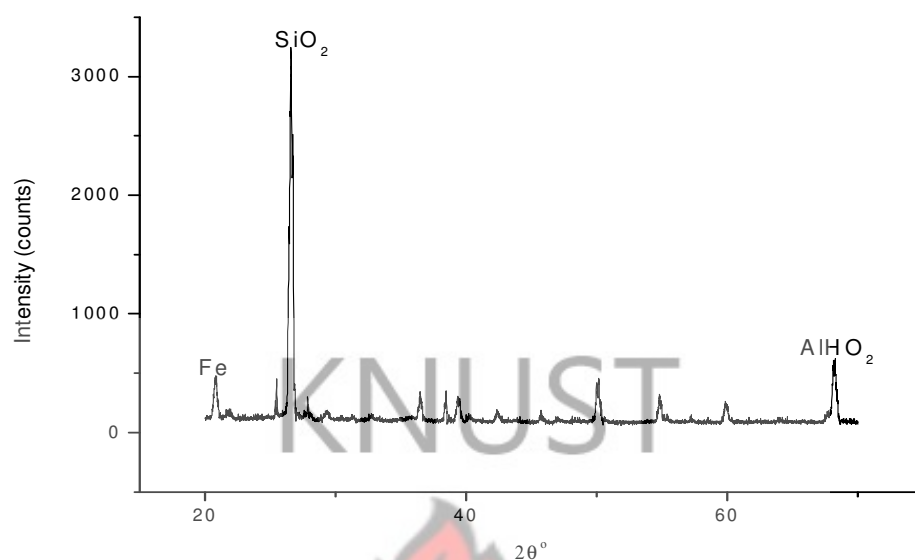


Figure 4.29 (a): X-Ray diffraction graph of S₈



Figure 4.29 (b): Percentage Abundance of Compounds in S₈

4.7 Microstructure of Some Characteristic PPC (S_0 , S_2 , S_5 and S_8)

The micrographs of the SEM analysis performed on some characteristic test specimens (S_1 , S_2 , S_5 and S_8) at 28 days were shown in Figures 4.30 – 4.33. Their various visual surfaces suggested the following phases (gray [calcite], dark [calcium carbonate] and white [compounds contributing to shielding rather than strength]). This confirmed the results from the tests already performed. S_2 which looked very dense (less dark spots) compared to S_0 , exhibited highest strength value while S_5 which had more of the white spots compared to S_8 , exhibited the highest shielding value.

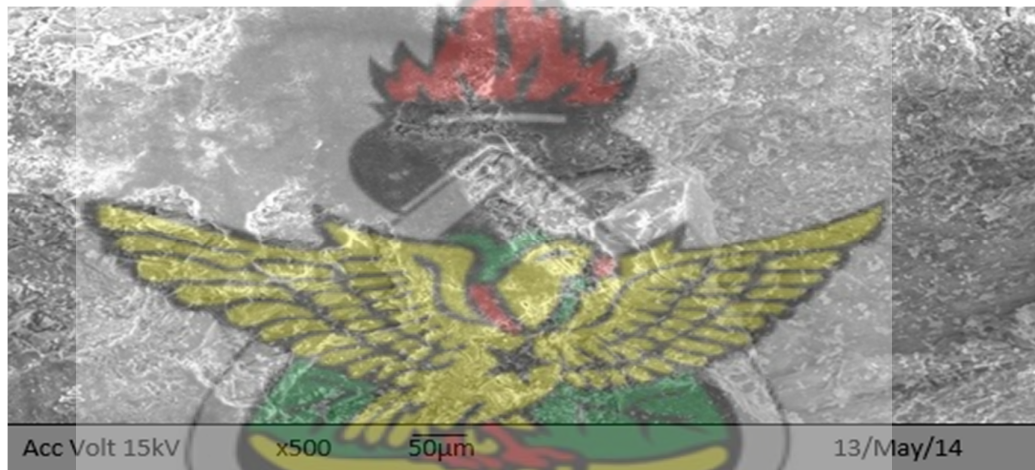


Figure 4.30: Microstructure of Control (S_0)

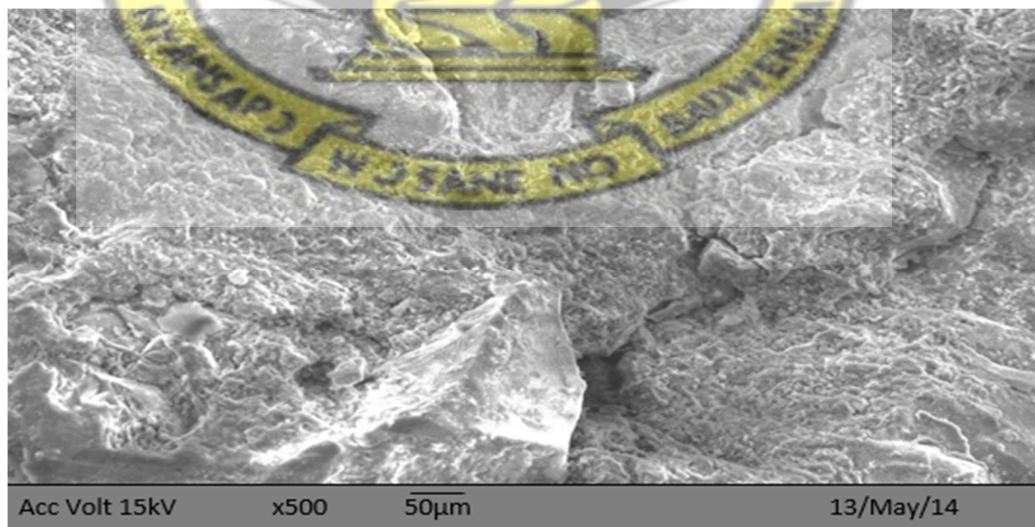


Figure 4.31: Microstructure of S_2

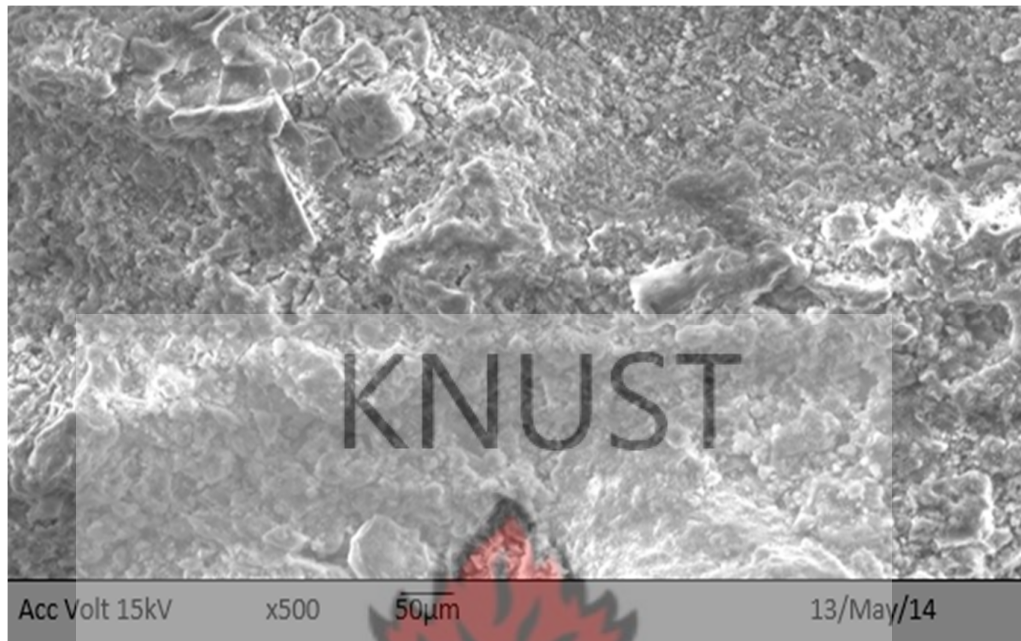


Figure 4.32: Microstructure of S₅



Figure 17.33: Microstructure of S₈

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 Mechanical Behaviour

The structural and radiological analyses of Pozzolana Portland Cement (PPC) and Ordinary Portland Cement (OPC) have been investigated using standard methods with various ratios of Pozzolana to ordinary Portland cement.

The results have shown that Pozzolana reacts more in the mixtures with proportions $\leq 20\%$ of addition, and contributes in a heavy manner to the development of the compressive strength. The mixture with 20 % of Pozzolana gave what was considered to be the best results. Thus, following the abundant nature of clay Pozzolana in Ghana, and its influence on strength of OPC, Pozzolana can be used for nuclear power plant containment structural construction with concrete mixture of Pozzolana and ordinary Portland cement at a ratio of 1:4 respectively, reducing total cost of construction and also increasing structural strength.

From the experiment, 100% Pozzolana cement is clay, therefore, had little or no strength (Figure 31). This implied that it could not be used all alone for the purpose of loads of any kind, unless used in combination with ordinary Portland cement (OPC).

The analysis of the mechanical property (Modulus) evolution of the studied cement pastes indicates that since the early age of 7 days, pastes with 20% of Pozzolana, gives resulting strengths higher than the paste of OPC (control). Whereas, the strengths developed by the paste with 30% PPC or more are still lower than those of the paste of OPC (control). The trend continues from 14 until 28 days. This signifies that the Pozzolana has accelerated the kinetics of the cement paste hydration. The

strength of the concrete increased constantly from 7 days through 14 days to 28 days proving the evolution of strength over periods of ageing.

5.1.2 Validation of Mechanical Properties of PPC

The flexural strength result indicated that 20% PPC peaked the modulus of rupture, and the ultrasonic test result showed that in parallel comparison, concrete of higher quality (high pulse velocity) corresponds to high elastic modulus, with 20% PPC leading.

5.2 Shielding Properties

The elemental composition analysis has shown that, the presence of elements of significant macroscopic cross sections dominate more in Pozzolana than in OPC. It can be concluded that Portland Pozzolanic concrete (PPC) is a better shielding material for neutrons and gamma/X rays, compared to concrete made from purely ordinary Portland cement (OPC) as indicated in Tables 4.1 and 4.2. Noticeable among them in Pozzolana, are Eu, Hf, Co, Fe, Mn and Sc the rest are Cu, Al and Ca.

5.2.2 Validation of Shielding Properties of PPC

X-ray shielding analysis to ascertain the conclusion drawn from the composition analysis showed that linear attenuation coefficient (μ_1) of PPC was improved tremendously due to the Pozzolana admixture.

5.3 Structural, Radiological and Economic Advantages of PPC over OPC

Following the abundant nature of clay Pozzolana in Ghana, it is economically advantageous to use a combination of OPC and Pozzolana rather than using only OPC, as mineral admixture for attaining the same strength level or better. Among the mixes, a combination of 80% OPC and 20% Pozzolana, showed the highest increase in strength for the entire range of water cement ratios. Using 20% PPC mix leads not

only to cost saving but also higher strength. Again 50% PPC also showed a better radiological shielding property, a unique discovery for radiation safety and protection, since the formula can be used in the nuclear and allied industries.

5.4 Findings

5.4.1 Observations

The following observations were made during the research:

- More volume of water was added as PPC % increased due to higher surface area of Pozzolana (finer in particle size than OPC). This phenomena restricted the mixes to be 0.5 w/c and above. Meanwhile OPC could make use of as low as 0.35 w/c ratio.
- Setting time for PPC is shorter than OPC. Thus PPC solidified faster than OPC.

5.4.2 The Phases or Crystallographic Structure responsible for the 20% PPC Mechanical Behaviour

Phases from the XRD results showed that, at 20% PPC, all metastable compounds of the calcium carbonate; $\text{Ca}(\text{CO}_3)$ react to form the most stable CaCO_3 (**Calcite**) which is Hexagonal Close- Packed (HCP) crystals. Calcite is of a crystal structure with Atomic Packing factor (APF) to be 74%. HCP is normally of APF which is equal to that of face centered crystal (FCC) of 74%, thus 26% void compared to APF of body centered crystal (BCC) of 68% or a simple cubic of APF of 52%.

Also, comparing the densities of Calcite (2.71g/cm^3), OPC (2.31g/cm^3) and Pozzolana (1.4g/cm^3) one can conclude that at equilibrium where calcite is dominant in the matrix, the end product thereof is expected to be strongest comparable to the OPC. Hence, the highest strength exhibited at 20% PPC. In other words, at 20%PPC; Calcite, CaCO_3 , crystal structure (Hexagonal Closed Packed) which is a carbonate

mineral and the most stable polymorph of calcium carbonate, $\text{Ca}(\text{CO}_3)$, crystal system (Rhombohedral) is dominant in the matrix of silicon oxide (SiO_2) and maximum strength is achieved.

5.4.3 The Phases or Elements responsible for the Improvement in the Shielding Properties of PPC

Beyond 30%PPC, Calcite (CaCO_3) is relatively consumed in the hydration process as more Pozzolana is added rendering the composite material less strong. But Copper (Cu), Iron (Fe), Aluminum Hydroxide ($\text{Al}(\text{OH})_3$) and Manganese (Mn) phases now build up contributing to improvement in its radiological shielding property. But the intensity of the composition of the above phases reduces after 50%PPC. Therefore, maximum shielding characteristic of PPC is achieved at 50%PPC.

The macroscopic cross sections (Σ) were computed from NAA result as 0.15 cm^{-1} for OPC as against 0.6044 cm^{-1} for Pozzolana (Tables 4.1 & 4.2 respectively). Meanwhile the x-ray shielding experimental results for linear attenuation coefficients (μ) for OPC and PPC at 150 keV were 0.14 cm^{-1} and 1.53 cm^{-1} respectively. This is an indication that Pozzolana improves significantly on the shielding property of OPC via Pozzolana partial replacement. This shielding property becomes highest at 50% Pozzolana partial replacement as shown in Figure 4.20.

In conclusion, effective and comprehensive shielding of radiation sources may be achieved by using **20% PPC** for structural shielding purposes while the **50% PPC** is used to line inside for radiological shielding purposes.

5.5 Recommendation

It is recommended from the results and conclusions drawn from the preceding analysis, that Pozzolana as an admixture, improves significantly the radiological and structural properties of the ordinary Portland cement (OPC). Composite materials, therefore, developed from these findings should be commercially exploited, leading to:

- Pilot production of pre-bagged PPC (30 to 50 %) cement with the ultimate aim of using the composite materials in the construction of facilities meant for housing radioactive materials in hospitals, and/or nuclear and its allied industries in the country as well as confinement for solid and liquid wastes containing hazardous materials.
- Pilot production of pre-bagged PPC (20%) cement with the ultimate aim of using the composite materials in the construction of facilities meant for hostile environments such as earthquake prone zones, seafloor tunnels, offshore and coastal marine structures. Seismic activities have been the major factors considered during design and construction of energy/nuclear power plants. Materials to be used in the construction of such plants must exhibit high level of strength and robustness to avoid any disaster in the event of earthquake.
- Comparative research on the shielding property of concrete made of PPC with 30 to 50 % Pozzolana replacement should be carried out: (a); firstly on x-ray with well interlocked grooved-concrete blocks (to reduce scattering effect) and (b); the latter repeated using gamma and neutron rays respectively.
- Establishment of test site to carry out strength and shielding tests on walls built of Pozzolana – Portland cement (PPC).

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APPENDICES

APPENDIX (A)

Tables of Mix Ratios (1:2:4) and Compressive Strength: W/C = 0.5

Cement: Sand: Stone Ratio = 1:2:4; Bulk Density of PPC = 1.4 g/cm³, Density of OPC= 2.3 g/cm³

Table A 1: Percentage Composition of Materials

Sample	Weight of cement	Weight of sand	Weight of stone	Weight of Pozzolana	Water W/C = 0.5	Water W/C = 0.6	Water W/C = 0.7
Control (S ₀)	4 kg	8kg	16 kg	0	2000 ml	2400ml	2800ml
S ₁ 10% Pozzo.	3.6kg	8kg	16kg	0.4kg	2000 ml	2400ml	2800ml
S ₂ 20% Pozzo.	3.2 kg	8 kg	16 kg	0.8 kg	2000 ml	2400ml	2800ml
S ₃ 30% Pozzo.	2.8 kg	8 kg	16 kg	1.2 kg	2000 ml	2400ml	2800ml
S ₄ 40% Pozzo.	2.4 kg	8 kg	16 kg	1.6 kg	2000 ml	2400ml	2800ml
S ₅ 50% Pozzo.	2 kg	8 kg	16 kg	2 kg	2000 ml	2400ml	2800ml
S ₆ 60% Pozzo.	1.6 kg	8 kg	16 kg	2.4 kg	2000 ml	2400ml	2800ml
S ₇ 70% Pozzo.	1.2 kg	8 kg	16 kg	2.8 kg	2000 ml	2400ml	2800ml
S ₈ 80% Pozzo.	0.8 kg	8 kg	16 kg	3.2 kg	2000 ml	2400ml	2800ml
S ₉ Control	0	8 kg	16 kg	4 kg	2000 ml	2400ml	2800ml

Table A 2: Two (2) Days Compressive Strength at W/C = 0.5

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Compressive Strength (MPa)
S ₀ (Control) Portland	22,500	360.00	13.6
S ₁ (10% Pozzolana)	22,500	353.25	15.7
S ₂ (20% Pozzolana)	22,500	362.25	16.1
S ₃ (30% Pozzolana)	22,500	208.25	9.3
S ₄ (40% Pozzolana)	22,500	162.00	7.2
S ₅ (50% Pozzolana)	22,500	119.25	5.3
S ₆ (60% Pozzolana)	22,500	83.25	3.7
S ₇ (70% Pozzolana)	22,500	58.50	2.6
S ₈ (80% Pozzolana)	22,500	38.25	1.7
S ₉ (Control) Pozzolana	22,500	0	0

Table A 3: Seven (7) Days Compressive Strength at W/C = 0.5

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Compressive Strength (MPa)
S ₀ (Control) Portland	22,500	380.03	16.89
S ₁ (10% Pozzolana)	22,500	414.90	18.44
S ₂ (20% Pozzolana)	22,500	433.35	19.26
S ₃ (30% Pozzolana)	22,500	280.13	12.45
S ₄ (40% Pozzolana)	22,500	266.63	11.85
S ₅ (50% Pozzolana)	22,500	186.08	8.27
S ₆ (60% Pozzolana)	22,500	116.55	5.18
S ₇ (70% Pozzolana)	22,500	93.38	4.15
S ₈ (80% Pozzolana)	22,500	60.08	2.67
S ₉ (Control) Pozzolana	22,500	0	0

Table A 4: Fourteen Days (14) Compressive Strength at W/C = 0.5

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Average Compressive Strength (MPa)
S ₀ (Control) Portland	22,500	413.33	18.37
S ₁ (10% Pozzolana)	22,500	474.98	21.11
S ₂ (20% Pozzolana)	22,500	560.00	24.89
S ₃ (30% Pozzolana)	22,500	315.00	14.00
S ₄ (40% Pozzolana)	22,500	306.68	13.63
S ₅ (50% Pozzolana)	22,500	240.08	10.67
S ₆ (60% Pozzolana)	22,500	193.28	8.59
S ₇ (70% Pozzolana)	22,500	156.60	6.96
S ₈ (80% Pozzolana)	22,500	102.60	4.56
S ₉ (Control) Pozzolana	22,500	0	0

Table A 5: Twenty-eight Days (28) Compressive Strength at W/C = 0.5

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Compressive Strength (MPa)
S ₀ (Control) Portland	22,500	639.90	28.44
S ₁ (10% Pozzolana)	22,500	654.75	29.10
S ₂ (20% Pozzolana)	22,500	742.50	33.00
S ₃ (30% Pozzolana)	22,500	540.00	24.00
S ₄ (40% Pozzolana)	22,500	464.18	20.63
S ₅ (50% Pozzolana)	22,500	397.58	17.67
S ₆ (60% Pozzolana)	22,500	328.28	14.59
S ₇ (70% Pozzolana)	22,500	269.10	11.96
S ₈ (80% Pozzolana)	22,500	192.60	8.56
S ₉ (Control)Pozzolana	22,500	0	0

APPENDIX (B)

Tables of Compressive Strength: W/C = 0.6

Table B 1: Seven (7) Days Compressive Strength at W/C = 0.6

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Compressive Strength (MPa)
S₀ (Control) Portland	22,500	228.29	10.15
S₁ (10% Pozzolana)	22,500	248.94	11.06
S₂ (20% Pozzolana)	22,500	260.01	11.56
S₃ (30% Pozzolana)	22,500	168.08	7.47
S₄ (40% Pozzolana)	22,500	159.98	7.11
S₅ (50% Pozzolana)	22,500	111.65	4.96
S₆ (60% Pozzolana)	22,500	69.93	3.11
S₇ (70% Pozzolana)	22,500	56.03	2.49
S₈ (80% Pozzolana)	22,500	36.05	1.60
S₉ (Control) Pozzolana	22,500	0	0

Table B 2: Fourteen Days (14) Compressive Strength at W/C = 0.6

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Average Compressive Strength (MPa)
S ₀ (Control) Portland	22,500	248.00	11.02
S ₁ (10% Pozzolana)	22,500	284.99	12.67
S ₂ (20% Pozzolana)	22,500	336.00	14.93
S ₃ (30% Pozzolana)	22,500	189.00	8.4
S ₄ (40% Pozzolana)	22,500	184.01	8.18
S ₅ (50% Pozzolana)	22,500	128.05	5.69
S ₆ (60% Pozzolana)	22,500	115.97	5.15
S ₇ (70% Pozzolana)	22,500	93.96	4.18
S ₈ (80% Pozzolana)	22,500	61.56	2.74
S ₉ (Control) Pozzolana	22,500	0	0

Table B 3: Twenty-eight Days (28) Compressive Strength at W/C = 0.6

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Compressive Strength (MPa)
S ₀ (Control) Portland	22,500	383.94	17.06
S ₁ (10% Pozzolana)	22,500	392.85	17.46
S ₂ (20% Pozzolana)	22,500	432.00	21.20
S ₃ (30% Pozzolana)	22,500	278.51	12.38
S ₄ (40% Pozzolana)	22,500	238.55	10.60
S ₅ (50% Pozzolana)	22,500	234.00	10.40
S ₆ (60% Pozzolana)	22,500	196.97	8.75
S ₇ (70% Pozzolana)	22,500	161.46	7.18
S ₈ (80% Pozzolana)	22,500	115.56	5.14
S ₉ (Control) Pozzolana	22,500	0	0

APPENDIX (C)

Tables of Compressive Strength: W/C = 0.7

Table C 1: Seven (7) Days Compressive Strength at W/C = 0.7

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Compressive Strength (MPa)
S₀ (Control) Portland	22,500	226.13	10.05
S₁ (10% Pozzolana)	22,500	247.50	11.00
S₂ (20% Pozzolana)	22,500	257.85	11.46
S₃ (30% Pozzolana)	22,500	167.85	7.46
S₄ (40% Pozzolana)	22,500	165.38	7.35
S₅ (50% Pozzolana)	22,500	101.70	4.52
S₆ (60% Pozzolana)	22,500	68.85	3.06
S₇ (70% Pozzolana)	22,500	49.95	2.22
S₈ (80% Pozzolana)	22,500	30.38	1.35
S₉ (Control) Pozzolana	22,500	0	0

Table C 2: Fourteen Days (14) Compressive Strength at W/C = 0.7

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Average Compressive Strength (MPa)
S ₀ (Control) Portland	22,500	247.50	11.00
S ₁ (10% Pozzolana)	22,500	272.25	12.10
S ₂ (20% Pozzolana)	22,500	315.00	14.00
S ₃ (30% Pozzolana)	22,500	186.75	8.30
S ₄ (40% Pozzolana)	22,500	182.03	8.09
S ₅ (50% Pozzolana)	22,500	121.50	5.40
S ₆ (60% Pozzolana)	22,500	114.08	5.07
S ₇ (70% Pozzolana)	22,500	92.25	4.10
S ₈ (80% Pozzolana)	22,500	60.75	2.70
S ₉ (Control) Pozzolana	22,500	0	0

Table C 3: Twenty-eight Days (28) Compressive Strength at W/C = 0.7

Pozzolana Percentage (%)	X- sectional area (mm ²)	Average Crushing Force (kN)	Compressive Strength (MPa)
S₀ (Control) Portland	22,500	362.48	16.11
S₁ (10% Pozzolana)	22,500	369.68	16.43
S₂ (20% Pozzolana)	22,500	429.75	19.10
S₃ (30% Pozzolana)	22,500	256.28	11.39
S₄ (40% Pozzolana)	22,500	232.43	10.33
S₅ (50% Pozzolana)	22,500	227.48	10.11
S₆ (60% Pozzolana)	22,500	186.98	8.31
S₇ (70% Pozzolana)	22,500	157.73	7.01
S₈ (80% Pozzolana)	22,500	112.50	5.00
S₉ (Control) Pozzolana	22,500	0	0

APPENDIX (D)**Table of Flexural Strength (Modulus of Rupture): W/C = 0.5****Table D 1: Modulus of Rupture (Flexural Strength) of Concrete at 28 Days**

Pozzolana Percentage (%)	Average Breaking Force (N)	Avg. Modulus of Rupture (MPa)
S₀ (Control) Portland	6,533.30	8.00
S₁ (10% Pozzolana)	7,001.20	8.60
S₂ (20% Pozzolana)	7,862.20	9.61
S₃ (30% Pozzolana)	6,216.00	7.60
S₄ (40% Pozzolana)	4,920.00	6.01
S₅ (50% Pozzolana)	4,200.00	5.13

APPENDIX (E)

Tables of Ultrasonic test

Table E 1: Seven (7) Days Ultrasonic test: W/C = 0.5

Pozzolana Percentage (%)	Avg. Velocity (ms^{-1})	Avg. Elastic Modulus (GN/m^2)
S₀ (Control) Portland	3540	16.20
S₁ (10% Pozzolana)	3740	17.10
S₂ (20% Pozzolana)	3850	19.30
S₃ (30% Pozzolana)	3440	15.45
S₄ (40% Pozzolana)	3400	14.85
S₅ (50% Pozzolana)	3200	12.27
S₆ (60% Pozzolana)	3000	9.18
S₇ (70% Pozzolana)	2500	8.15
S₈ (80% Pozzolana)	2000	7.67
S₉ (Control) Pozzolana	1800	0.95

Table E 2: Fourteen Days (14) Ultrasonic test

Pozzolana Percentage (%)	Avg. Velocity (ms ⁻¹)	Avg. Elastic Modulus (GN/m ²)
S ₀ (Control) Portland	4200	27.20
S ₁ (10% Pozzolana)	4250	28.70
S ₂ (20% Pozzolana)	4400	31.50
S ₃ (30% Pozzolana)	4000	22.30
S ₄ (40% Pozzolana)	3900	20.85
S ₅ (50% Pozzolana)	3700	18.50
S ₆ (60% Pozzolana)	3300	17.18
S ₇ (70% Pozzolana)	3200	15.44
S ₈ (80% Pozzolana)	3000	14.40
S ₉ (Control) Pozzolana	2000	0.95

Table E 3: Twenty-eight Days (28) Ultrasonic test

Pozzolana Percentage (%)	Avg. Velocity (ms ⁻¹)	Avg. Elastic Modulus (GN/m ²)
S ₀ (Control) Portland	4250	38.60
S ₁ (10% Pozzolana)	4300	39.30
S ₂ (20% Pozzolana)	4500	44.70
S ₃ (30% Pozzolana)	4100	36.40
S ₄ (40% Pozzolana)	4090	35.30
S ₅ (50% Pozzolana)	4010	33.90
S ₆ (60% Pozzolana)	4000	32.08
S ₇ (70% Pozzolana)	3700	29.40
S ₈ (80% Pozzolana)	3200	26.30
S ₉ (Control) Pozzolana	3000	0.95

APPENDIX (F)

NAA Results

Table F 1: Neutron Shielding (Macroscopic Cross Section) Interpretation

Elements	Density (ρ), g/cm ³	Total Macroscopic Cross Section, Σ_T [cm ⁻¹]	Composition (% wt)
Al	2.7	0.099	42
Ca	1.55	0.080	4.60
Ce	6.78	0.284	0.48
Co	8.90	4.097	0.05
Cr	7.10	0.502	0.17
Cs	1.87	0.408	0.15
Cu	8.94	0.937	0.04
Eu	5.22	89.166	0.008
Fe	7.90	1.155	46
Hf	13.30	5.069	0.08
K	0.87	0.048	4.21
La	6.19	0.642	0.009
Mn	7.20	1.221	0.51
Na	0.971	0.115	1.50
Sc	2.50	1.608	0.03
V	5.96	0.704	0.04

Table F 2: X/Gamma Ray Shielding (Attenuation Coefficient) Interpretation

ELEMENTS	Total Mass Attenuation Coefficients [$\frac{\mu}{\rho}$], (cm ² /g)	Linear Attenuation Coefficients μ , [cm ⁻¹]
Na	0.151	0.147
Cu	0.427	3.800
Fe	0.344	2.718
K	0.215	0.187
Al	0.161	0.435
Ca	0.238	0.369
Mg	0.160	0.278
Ti	5.16	23.22

APPENDIX (G)

X-Ray Shielding Experiment

Cement: Sand: Ratio = 1:2; Bulk Density of PPC = 1.4 g/cm³, Density of OPC= 2.3 g/cm³

W/C ratio = 0.6

Table G 1: Percentage Composition of Materials

Sample	Weight of cement	Weight of sand	Weight of Pozzolana	Water W/C = 0.6
Control (S ₀)	8 kg	16kg	0	4800ml
S ₁ 10%Pozzo.	7.2kg	16kg	0.8kg	4800ml
S ₂ 20%Pozzo.	6.4 kg	16 kg	1.6 kg	4800ml
S ₃ 30% Pozzo.	5.6 kg	16 kg	2.4 kg	4800ml
S ₄ 40% Pozzo.	4.8 kg	16 kg	3.2 kg	4800ml
S ₅ 50% Pozzo.	4 kg	16 kg	4 kg	4800ml
S ₆ 60%Pozzo.	3.2 kg	16kg	4.8 kg	4800ml
S ₇ 70%Pozzo.	2.4 kg	16 kg	5.6 kg	4800ml
S ₈ 80% Pozzo.	1.6 kg	16 kg	6.4 kg	4800ml
S ₉ Control	0	16 kg	8 kg	4800ml

Table G 2: X-Ray data for 0% PPC

I₀/150kVp/PC	I₁₅/150kVp/PC	I₃₀/150kVp/PC	I₄₅/150kVp/PC
52.0	7.0	8.5	3.0
57.0	4.5	1.0	7.5
65.0	6.0	5.0	8.0
63.0	7.5	8.0	8.0
60.0	9.0	9.0	8.0
68.0	9.0	9.0	8.0
63.5	9.0	9.0	8.0
63.5	9.0	9.0	8.0
63.0	9.0	9.0	8.0
63.0	9.0	9.0	8.0
Average: 61.8	Average: 7.9	Average: 7.7	Average: 7.5

Table G 3: X-Ray data for 10% PPC

I₀/150kVp/PC	I₁₅/150kVp/PC	I₃₀/150kVp/PC	I₄₅/150kVp/PC
52.0	7.0	2.5	15.0
57.0	8.5	7.0	5.5
65.0	7.0	9.0	5.0
63.0	7.0	9.0	3.0
60.0	7.0	7.5	3.0
68.0	7.0	6.0	3.0
63.5	7.5	6.0	3.0
63.5	7.0	6.0	3.0
63.0	7.0	6.0	2.5
63.0	7.0	6.5	1.0
Average: 61.8	Average: 7.2	Average: 6.45	Average: 4.4

Table G 4: X-Ray data for 20% PPC

I₀/150kVp/PC	I₁₅/150kVp/PC	I₃₀/150kVp/PC	I₄₅/150kVp/PC
52.0	4.5	6.0	0.5
57.0	3.0	4.5	6.5
65.0	3.0	6.0	9.0
63.0	5.5	4.5	7.0
60.0	6.0	5.5	6.0
68.0	6.0	6.0	6.0
63.5	6.0	4.5	5.5
63.5	6.0	5.0	3.5
63.0	7.0	5.0	3.0
63.0	9.0	5.0	3.0
Average: 61.8	Average: 5.5	Average: 5.2	Average: 5.0

Table G 5: X-Ray data for 30% PPC

I₀/150kVp/PC	I₁₅/150kVp/PC	I₃₀/150kVp/PC	I₄₅/150kVp/PC
52.0	12.0	6.0	1.0
57.0	0.5	0.0	1.0
65.0	3.5	0.0	1.0
63.0	3.0	0.0	3.0
60.0	3.0	0.0	1.0
68.0	3.0	0.5	1.0
63.5	2.0	2.5	1.0
63.5	0.5	3.0	1.0
63.0	0.0	3.0	1.0
63.0	0.0	3.0	0.5
Average: 61.8	Average: 2.75	Average: 1.8	Average: 1.15

Table G 6: X-Ray data for 40% PPC

I₀/150kVp/PC	I₁₅/150kVp/PC	I₃₀/150kVp/PC	I₄₅/150kVp/PC
52.0	0.0	6.0	3.0
57.0	9.0	6.0	4.5
65.0	9.0	6.0	6.0
63.0	9.0	6.0	4.5
60.0	9.0	6.0	3.0
68.0	9.0	6.0	3.0
63.5	9.0	5.0	3.0
63.5	9.0	3.0	2.5
63.0	9.0	3.0	0.5
63.0	9.0	3.0	0.0
Average: 61.8	Average: 8.1	Average: 5.0	Average: 3.0

Table G 7: X-Ray data for 50% PPC

I₀/150kVp/PC	I₁₅/150kVp/PC	I₃₀/150kVp/PC	I₄₅/150kVp/PC
52.0	9.5	9.0	8.0
57.0	2.5	0.0	0.0
65.0	3.5	0.0	0.0
63.0	2.5	0.0	0.0
60.0	0.0	1.0	0.0
68.0	0.0	1.5	0.0
63.5	2.0	3.0	0.0
63.5	3.0	0.0	0.5
63.0	3.0	0.0	2.0
63.0	3.0	0.0	3.0
Average: 61.8	Average: 2.9	Average: 1.45	Average: 1.35

Table G 8: X-Ray data for 60% PPC

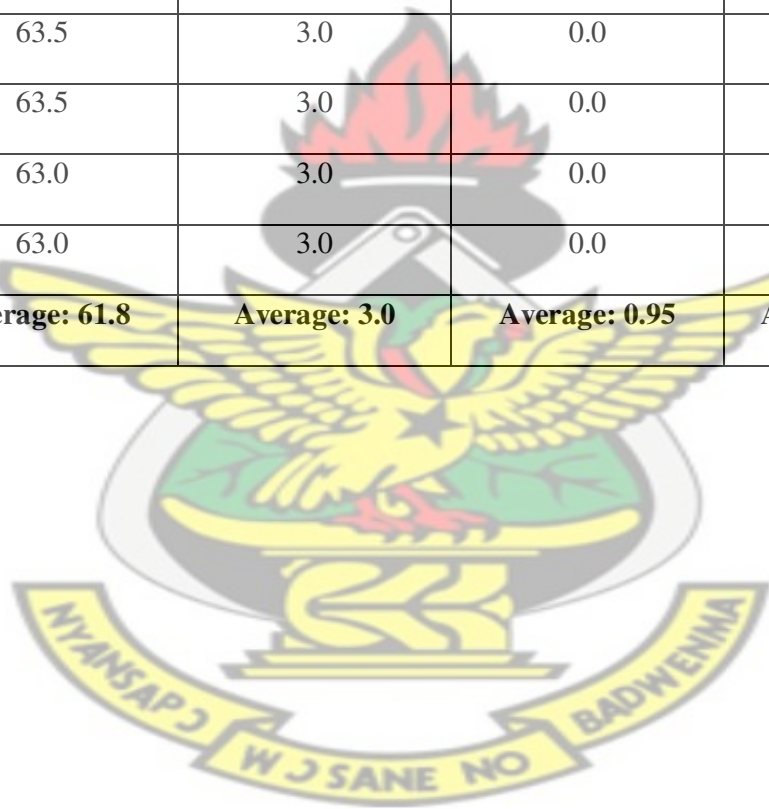
I₀/150kVp/PC	I₁₅/150kVp/PC	I₃₀/150kVp/PC	I₄₅/150kVp/PC
52.0	5.5	3.5	0.0
57.0	4.0	3.0	0.5
65.0	6.0	5.5	5.5
63.0	4.0	0.5	0.5
60.0	4.0	1.0	1.0
68.0	4.0	1.0	1.0
63.5	4.0	3.0	3.0
63.5	4.0	3.0	3.0
63.0	4.0	3.0	3.0
63.0	4.0	3.0	3.0
Average: 61.8	Average: 4.35	Average: 2.65	Average: 1.35

Table G 9: X-Ray data for 70% PPC

I₀/150kVp/PC	I₁₅/150kVp/PC	I₃₀/150kVp/PC	I₄₅/150kVp/PC
52.0	1.5	6.5	1.0
57.0	9.0	6.0	1.0
65.0	9.5	6.0	1.0
63.0	9.0	6.0	3.0
60.0	9.0	6.0	3.0
68.0	9.0	6.0	3.0
63.5	9.0	6.0	3.0
63.5	9.0	4.5	3.0
63.0	9.0	3.0	3.0
63.0	9.0	6.0	3.0
Average: 61.8	Average: 8.3	Average: 5.0	Average: 2.4

Table G 10: X-Ray data for 80% PPC

I₀/150kVp/PC	I₁₅/150kVp/PC	I₃₀/150kVp/PC	I₄₅/150kVp/PC
52.0	3.0	2.0	1.5
57.0	3.0	2.0	1.0
65.0	3.0	1.5	1.0
63.0	3.0	1.5	1.0
60.0	3.0	1.0	1.0
68.0	3.0	1.5	1.0
63.5	3.0	0.0	0.0
63.5	3.0	0.0	0.0
63.0	3.0	0.0	0.0
63.0	3.0	0.0	0.0
Average: 61.8	Average: 3.0	Average: 0.95	Average: 0.65



APPENDIX (H)

Derived Data for Calculating Linear attenuation Coefficients

Table H 1: Fraction transmitted (FT) & Natural log of FT for 0% PPC

$x(\text{cm})$	0	15	30	45
$\frac{I}{I_0}$	1	0.121	0.081	0.049
$\ln\left(\frac{I}{I_0}\right)$	0	-2.11	-2.51	-3.03

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Table H 2: Fraction transmitted (FT) & Natural log of FT for 10% PPC

$x(\text{cm})$	0	15	30	45
$\frac{I}{I_0}$	1	0.113	0.040	0.016
$\ln\left(\frac{I}{I_0}\right)$	0	-2.18	-3.21	-4.12

Table H 3: Fraction transmitted (FT) & Natural log of FT for 20% PPC

$x(\text{cm})$	0	15	30	45
$\frac{I}{I_0}$	1	0.097	0.073	0.008
$\ln\left(\frac{I}{I_0}\right)$	0	-2.33	-2.62	-4.82

Table H 4: Fraction transmitted (FT) & Natural log of FT for 30% PPC

$x(\text{cm})$	0	15	30	45
$\frac{I}{I_0}$	1	0.008	$0.00 \approx (10^{-10})$	0.008
$\ln\left(\frac{I}{I_0}\right)$	0	-4.82	$\infty; (-23.03)$	-4.82

Table H 5: Fraction transmitted (FT) & Natural log of FT for 40 % PPC

x(cm)	0	15	30	45
$\frac{I}{I_0}$	1	$0.00 \approx (10^{-10})$	0.049	$0.00 \approx (10^{-10})$
$\ln\left(\frac{I}{I_0}\right)$	0	$\infty; (-23.03)$	-3.03	$\infty; (-23.03)$

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Table H 6: Fraction transmitted (FT) & Natural log of FT for 50 % PPC

x(cm)	0	15	30	45
$\frac{I}{I_0}$	1	$0.00 \approx (10^{-10})$	$0.00 \approx (10^{-10})$	$0.00 \approx (10^{-10})$
$\ln\left(\frac{I}{I_0}\right)$	0	$\infty; (-23.03)$	$\infty; (-23.03)$	$\infty; (-23.03)$

Table H 7: Fraction transmitted (FT) & Natural log of FT for 60 % PPC

x(cm)	0	15	30	45
$\frac{I}{I_0}$	1	0.065	0.008	$0.00 \approx (10^{-10})$
$\ln\left(\frac{I}{I_0}\right)$	0	-2.74	-4.82	$\infty; (-23.03)$

Table H 8: Fraction transmitted (FT) & Natural log of FT for 70 % PPC

x(cm)	0	15	30	45
$\frac{I}{I_0}$	1	0.024	0.049	0.016
$\ln\left(\frac{I}{I_0}\right)$	0	-3.72	-3.03	-4.12

Table H 9: Fraction transmitted (FT) & Natural log of FT for 80 % PPC

x(cm)	0	15	30	45
$\frac{I}{I_0}$	1	0.049	$0.00 \approx (10^{-10})$	$0.00 \approx (10^{-10})$
$\ln\left(\frac{I}{I_0}\right)$	0	-3.03	$\infty; (-23.03)$	$\infty; (-23.03)$

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Table H 10: Linear Attenuation Coefficients Determined from the Data

PPC%	0	10	20	30	40	50	60	70	80
μ	0.140	0.145	0.155	0.218	0.327	1.533	0.474	0.248	0.593



APPENDIX (I)

XRD Data

Scan Parameters

Raw Data Origin:	XRD measurement (*.XRDML)
Scan Axis:	Gonio
Start Position [$^{\circ}2\theta$.]:	20.0050
End Position [$^{\circ}2\theta$.]:	70.0050
Step Size [$^{\circ}2\theta$.]:	0.0100
Scan Step Time [s]:	0.1500
Scan Type:	Continuous
Offset [$^{\circ}2\theta$.]:	0.0000
Divergence Slit Type:	Fixed
Divergence Slit Size [$^{\circ}$]:	1.0000
Specimen Length [mm]:	10.00
Receiving Slit Size [mm]:	0.1000
Measurement Temperature [$^{\circ}\text{C}$]:	25.00
Anode Material:	Cu
K-Alpha1 [$\text{\AA}$]:	1.54060
Generator Settings:	40 mA, 45 kV
Goniometer Radius [mm]:	240.00
Dist. Focus-Diverg. Slit [mm]:	91.00

Table I 1: Peak List for OPC (Control): 0% PPC

<u>Pos. [°2Th.]</u>	<u>Height [cts]</u>	<u>FWHMLeft [°2Th.]</u>	<u>d-spacing [Å]</u>	<u>Rel. Int.</u>
		<u>[%]</u>		
20.7755	349.02	0.1440	4.27212	24.05
22.9684	24.87	0.3840	3.86895	1.71
26.5076	1451.52	0.0840	3.35986	100.00
26.7152	1320.51	0.0960	3.33423	90.97
26.8144	571.22	0.0600	3.32211	39.35
27.4474	65.60	0.0720	3.24692	4.52
27.6789	5.31	0.5760	3.22029	0.37
29.2811	284.63	0.2400	3.04762	19.61
34.1800	20.90	0.5760	2.62119	1.44
36.6335	231.58	0.4320	2.45107	15.95
39.4379	159.55	0.1920	2.28300	10.99
40.2746	77.18	0.2400	2.23748	5.32
42.3162	51.92	0.3360	2.13413	3.58
43.1368	44.14	0.2880	2.09541	3.04
45.7558	92.06	0.4320	1.98138	6.34
47.1416	54.30	0.5760	1.92632	3.74
48.3890	42.78	0.3840	1.87953	2.95
50.0647	268.62	0.0960	1.82048	18.51
54.8084	61.74	0.2400	1.67360	4.25
57.2326	13.26	0.6720	1.60834	0.91
59.8465	220.15	0.1200	1.54418	15.17
64.3975	8.65	1.1520	1.44560	0.60
67.7976	153.23	0.3840	1.38114	10.56
68.2393	142.37	0.3840	1.37327	9.81

Table I 2: Peak List for 10% PPC

Pos. [°2Th.]	Height [cts]	FWHMLeft [°2Th.]	d-spacing [Å]	Rel. Int.
		[%]		
20.9310	397.76	0.0960	4.24072	17.97
22.9738	132.12	0.0720	3.86806	5.97
26.4376	1432.25	0.0840	3.36861	64.70
26.6221	2213.51	0.0720	3.34567	100.00
26.8160	686.85	0.0600	3.32192	31.03
28.0360	26.18	0.5760	3.18007	1.18
29.3569	176.33	0.4320	3.03993	7.97
34.0351	35.23	0.4800	2.63202	1.59
36.5497	139.39	0.3360	2.45650	6.30
39.3630	260.52	0.0720	2.28717	11.77
39.5520	231.86	0.1200	2.27667	10.47
40.2703	37.89	0.2880	2.23771	1.71
42.4998	90.87	0.3360	2.12533	4.11
43.1455	27.79	0.2880	2.09501	1.26
44.2180	48.86	0.1440	2.04666	2.21
45.7471	80.46	0.3840	1.98174	3.64
47.2385	36.76	0.7680	1.92259	1.66
48.4700	27.57	0.4320	1.87658	1.25
50.0840	316.50	0.0960	1.81982	14.30
54.8392	64.69	0.2880	1.67273	2.92
57.3501	19.99	0.5760	1.60532	0.90
59.8142	144.38	0.3360	1.54494	6.52
64.0188	35.71	0.2880	1.45323	1.61
67.6598	106.44	0.2400	1.38362	4.81
68.1643	183.32	0.3360	1.37460	8.28

Table I 3: Peak List for 20% PPC

Pos. [°2Th.]	Height [cts]	FWHMLeft [°2Th.]	d-spacing [Å]	Rel. Int.
		[%]		
20.9080	173.95	0.3840	4.24534	7.07
26.3937	1784.16	0.0480	3.37411	72.52
26.5406	2460.36	0.0720	3.35576	100.00
26.6084	2318.78	0.0480	3.34736	94.25
26.7436	2414.07	0.0840	3.33074	98.12
27.8159	26.32	0.5760	3.20474	1.07
29.2550	171.57	0.3360	3.05028	6.97
34.0668	25.44	0.3840	2.62964	1.03
36.4988	71.34	0.2880	2.45981	2.90
39.4565	220.63	0.0960	2.28196	8.97
40.2525	59.25	0.2880	2.23865	2.41
42.4916	62.40	0.3360	2.12573	2.54
43.1807	23.73	0.2880	2.09339	0.96
45.5692	201.90	0.0720	1.98906	8.21
45.7375	359.26	0.0960	1.98213	14.60
45.8886	236.63	0.0720	1.97595	9.62
47.2457	27.89	0.7680	1.92232	1.13
48.4190	24.49	0.3840	1.87844	1.00
50.1263	138.30	0.3840	1.81839	5.62
54.8808	65.49	0.1920	1.67156	2.66
59.9003	204.58	0.1440	1.54292	8.32
63.9740	48.37	0.3840	1.45414	1.97
68.3549	107.08	0.3840	1.37123	4.35

Table I 4: Peak List for 30% PPC

Pos. [°2Th.]	Height [cts]	FWHMLeft [°2Th.]	d-spacing [Å]	Rel. Int.
		[%]		
20.9098	1061.12	0.1080	4.24498	85.28
21.0349	828.91	0.0480	4.22000	66.62
21.9477	41.89	0.2880	4.04652	3.37
26.4033	611.77	0.0720	3.37289	49.17
26.5825	1244.29	0.1200	3.35057	100.00
29.3181	207.07	0.4800	3.04386	16.64
36.4409	214.88	0.0960	2.46359	17.27
36.6241	173.84	0.0960	2.45168	13.97
39.3497	191.60	0.1200	2.28791	15.40
40.2432	91.21	0.1920	2.23915	7.33
42.5061	900.05	0.1200	2.12503	72.33
42.6457	471.62	0.0480	2.11840	37.90
45.7630	60.70	0.3840	1.98109	4.88
47.2094	31.09	0.7680	1.92371	2.50
48.4489	35.48	0.5760	1.87735	2.85
50.0588	208.42	0.3360	1.82068	16.75
54.8282	38.00	0.3840	1.67304	3.05
57.3460	13.96	1.1520	1.60542	1.12
59.9262	150.23	0.4320	1.54232	12.07
64.0122	26.99	0.3840	1.45337	2.17
65.8422	111.63	0.1200	1.41734	8.97
67.7052	268.54	0.0960	1.38280	21.58
68.2734	301.56	0.1440	1.37267	24.24
68.4794	118.51	0.0720	1.36904	9.52

Table I 5: Peak List for 40% PPC

<u>Pos. [°2Th.]</u>	<u>Height [cts]</u>	<u>FWHMLeft [°2Th.]</u>	<u>d-spacing [Å]</u>	<u>Rel. Int.</u>
		<u>[%]</u>		
20.6953	352.66	0.0720	4.28849	12.48
20.8509	494.90	0.0960	4.25684	17.52
21.0146	436.89	0.0720	4.22404	15.46
26.3902	1400.58	0.0600	3.37454	49.58
26.5874	2825.10	0.0720	3.34996	100.00
26.7060	2656.67	0.0720	3.33536	94.04
27.8914	40.10	0.2880	3.19623	1.42
29.1994	188.88	0.4800	3.05596	6.69
36.6065	132.61	0.3360	2.45282	4.69
39.5161	103.22	0.3840	2.27866	3.65
40.2625	34.31	0.3840	2.23812	1.21
42.4707	246.22	0.1200	2.12672	8.72
45.6567	60.32	0.3840	1.98545	2.14
47.2381	27.38	0.7680	1.92261	0.97
48.2928	32.10	0.6720	1.88305	1.14
50.1184	272.66	0.2880	1.81865	9.65
54.8481	102.16	0.4320	1.67248	3.62
55.8992	62.01	0.0720	1.64350	2.19
59.8522	299.91	0.1200	1.54405	10.62
64.0397	35.97	0.2880	1.45281	1.27
68.2317	385.92	0.0720	1.37340	13.66

Table I 6: Peak List for 50% PPC

<u>Pos. [°2Th.]</u>	<u>Height [cts]</u>	<u>FWHMLeft [°2Th.]</u>	<u>d-spacing [Å]</u>	<u>Rel. Int.</u>
		<u>[°]</u>		
20.7805	415.68	0.4800	4.27109	15.81
26.4145	1787.57	0.0600	3.37149	67.98
26.5580	2629.46	0.0720	3.35361	100.00
26.7548	1890.72	0.0720	3.32938	71.91
27.8171	101.02	0.0720	3.20460	3.84
28.4149	166.38	0.0480	3.13852	6.33
29.4023	200.11	0.3840	3.03534	7.61
36.5237	155.70	0.0960	2.45819	5.92
39.3548	305.46	0.0720	2.28763	11.62
39.5182	282.22	0.0960	2.27855	10.73
40.1663	44.06	0.3840	2.24326	1.68
42.2799	146.75	0.1440	2.13588	5.58
42.4641	170.12	0.1440	2.12704	6.47
45.7463	79.75	0.2880	1.98177	3.03
47.2152	30.40	0.7680	1.92348	1.16
48.4115	31.05	0.5760	1.87871	1.18
49.6723	963.61	0.0480	1.83394	36.65
50.1011	606.82	0.0960	1.81924	23.08
50.2569	473.49	0.0840	1.81396	18.01
54.8279	63.96	0.2880	1.67305	2.43
57.4142	20.34	0.5760	1.60368	0.77
59.8120	384.14	0.1200	1.54499	14.61
64.0537	11.90	1.1520	1.45253	0.45
67.7134	183.46	0.1920	1.38265	6.98
68.2333	123.12	0.3360	1.37338	4.68

Table I 7: Peak List for 60% PPC

Pos. [°2Th.]	Height [cts]	FWHMLeft [°2Th.]	d-spacing[Å]	Rel.Int. [%]
20.6232	751.57	0.0720	4.30331	21.33
20.7588	899.75	0.0600	4.27552	25.54
20.8363	1106.07	0.0600	4.25978	31.40
20.9316	733.23	0.0480	4.24060	20.81
26.3765	1730.54	0.0480	3.37627	49.12
26.5258	3522.97	0.0600	3.35760	100.00
26.5850	3345.31	0.0480	3.35026	94.96
26.7075	2088.26	0.0720	3.33517	59.28
26.8778	353.52	0.0360	3.31442	10.03
29.3296	136.60	0.4320	3.04269	3.88
36.3984	166.91	0.0960	2.46636	4.74
36.5820	174.42	0.0720	2.45441	4.95
39.5193	134.16	0.3360	2.27848	3.81
40.2177	49.84	0.2880	2.24051	1.41
42.4278	96.82	0.3360	2.12877	2.75
45.6928	60.93	0.3840	1.98397	1.73
47.2544	23.30	0.5760	1.92198	0.66
48.4041	21.85	0.5760	1.87898	0.62
50.0583	396.36	0.0960	1.82070	11.25
50.2129	345.90	0.1200	1.81545	9.82
54.6505	433.44	0.0720	1.67806	12.30
54.8087	570.60	0.0840	1.67359	16.20
54.9844	256.31	0.0480	1.66866	7.28
59.8850	147.72	0.3360	1.54328	4.19
64.0551	23.41	0.2880	1.45250	0.66
68.1799	132.02	0.3840	1.37432	3.75

Table I 8: Peak List for 70% PPC

Pos. [°2Th.]	Height [cts]	FWHMLeft [°2Th.]	d-spacing [Å]	Rel. Int. [%]
20.7213	324.46	0.1680	4.28315	21.18
26.4630	1238.54	0.0720	3.36542	80.86
26.6327	1531.64	0.1200	3.34436	100.00
26.7912	656.42	0.0720	3.32494	42.86
29.3020	64.73	0.3840	3.04550	4.23
35.0537	113.60	0.0600	2.55783	7.42
36.4969	57.59	0.3360	2.45994	3.76
39.4739	114.78	0.1440	2.28100	7.49
40.2793	39.18	0.2880	2.23723	2.56
42.3843	84.77	0.1920	2.13086	5.53
45.7777	54.25	0.2400	1.98049	3.54
50.0569	148.63	0.2400	1.82074	9.70
54.8083	137.28	0.1200	1.67360	8.96
59.8422	103.41	0.3840	1.54428	6.75
64.0738	22.62	0.5760	1.45212	1.48
68.2069	88.11	0.4800	1.37384	5.75

Table I9: Peak List for 80% PPC

Pos. [°2Th.]	Height [cts]	FWHMLeft [°2Th.]	d-spacing[Å]	Rel.Int. [%]
20.8020	314.55	0.3840	4.26672	10.33
21.7872	44.81	0.5760	4.07596	1.47
25.4642	318.78	0.0600	3.49512	10.46
26.3408	845.11	0.1200	3.38076	27.74
26.5838	3046.22	0.1440	3.35041	100.00
26.7500	2114.07	0.0960	3.32997	69.40
26.8835	229.13	0.0480	3.31373	7.52
27.8759	152.22	0.0960	3.19797	5.00
29.3012	66.62	0.3840	3.04558	2.19
32.7468	22.56	0.7680	2.73257	0.74
36.4791	212.13	0.1200	2.46109	6.96
38.4824	205.10	0.0720	2.33746	6.73
39.3539	187.81	0.1920	2.28768	6.17
40.2606	43.24	0.3840	2.23822	1.42
42.3744	81.38	0.2400	2.13134	2.67
45.7173	46.07	0.2880	1.98296	1.51
47.0725	17.91	0.5760	1.92898	0.59
50.0014	315.00	0.0720	1.82263	10.34
50.1630	334.68	0.0960	1.81714	10.99
50.3309	164.10	0.0720	1.81147	5.39
54.8136	199.90	0.1200	1.67345	6.56
59.8468	147.21	0.2880	1.54417	4.83
68.1262	486.76	0.1440	1.37527	15.98
68.2837	489.75	0.0960	1.37249	16.08

APPENDIX (J)

CALCULATIONS

J1 Calculation of Compressive Stress

$$\text{Compressive stress } (\delta) = \frac{\text{Force (P)}}{\text{Area (A)}} = \frac{\text{N}}{\text{mm}^2} = \text{MPa} \quad (\text{J1.1})$$

For a concrete cube of size 150 mm:

$$\text{Cross sectional area} = 150 \text{ mm by } 150 \text{ mm} = 22,500 \text{ mm}^2 \quad (\text{J1.2})$$

$$\text{Therefore, Compressive stress } (\delta) = \frac{\text{Crushing Force (P)}}{22,500 \text{ mm}^2} = P/A \quad (\text{J1.3})$$

This calculation was used to determine the compressive strength of the concrete specimens. The value is for the 20% PPC cubes of 0.5 w/c cured for 28 days.

$$\sigma = \text{compressive strength} = P/A$$

$$P = 742.50 \text{ kN}$$

$$A = 150 \text{ mm} \times 150 \text{ mm} = 22,500 \text{ mm}^2$$

$$\sigma = 33.00 \text{ MPa}$$

J2 Flexural Strength (Modulus of Rupture) Calculation

This calculation was used to determine the flexural strength of cylindrical concrete specimens (20% PPC).

$$\text{Flexural strength } (\delta_f) = \frac{PL}{\pi R^3} ; \quad \text{for a circular cross section} \quad (\text{J2.1})$$

$$\delta_f = \frac{PL}{\pi R^3}$$

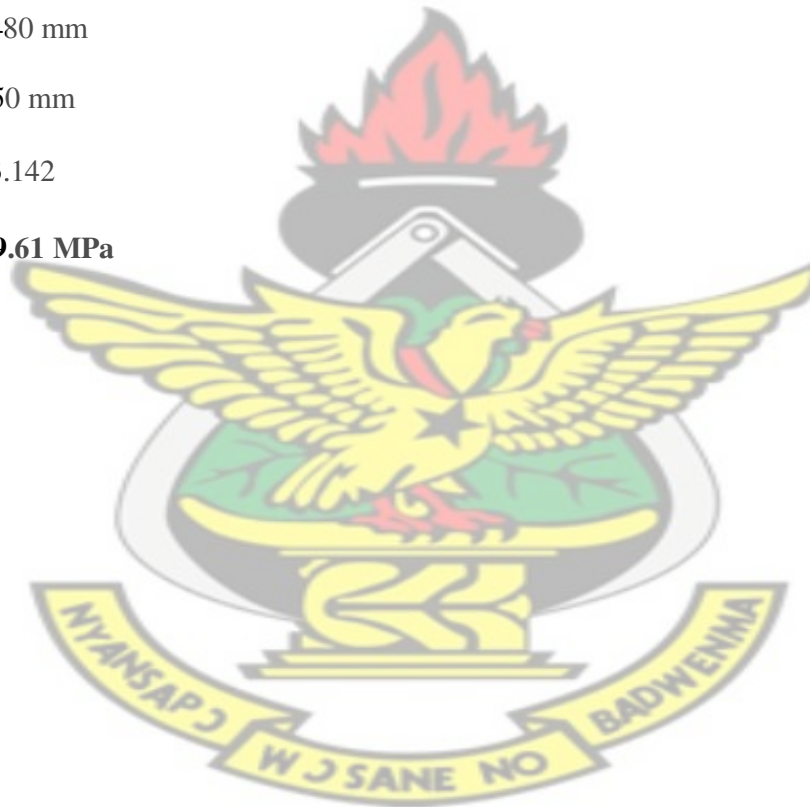
$$P = 7,862.20 \text{ N}$$

$$L = 480 \text{ mm}$$

$$R = 50 \text{ mm}$$

$$\pi = 3.142$$

$$\delta_f = \mathbf{9.61 \text{ MPa}}$$



Note:

- $1 \text{ N/mm}^2 = 1 \text{ MPa}$
- $1 \text{ m}^2/\text{kg} = 10 \text{ cm}^2/\text{g}$

J3 Shielding Calculation Analysis

This calculation was used to determine whether or not Pozzolanic materials have a better shielding property than the ordinary Portland cement (OPC).

For gamma-ray and X-ray shielding analysis, linear attenuation coefficients μ_1 , [cm^{-1}] is considered, the higher the μ_1 , the better the material for shielding. The relation is given by

$$\frac{I}{I_0} = B e^{-\mu_1 X} = B_T = \frac{Pd^2}{WUT} \quad (J3.1)$$

Whereas, for neutron shielding, total macroscopic cross section Σ_T , [cm^{-1}] is considered the higher the Σ_T , the better the material for neutron shielding. The relation is given by:

$$\Phi(x) = \Phi_0 e^{-\Sigma_T X} \quad (J3.2)$$

All symbols used are of usual definitions from literature.

J4 Calculation of Sample (Concrete) Mass Estimation

Density (ρ_{OPC}) of ordinary Portland cement = 2.3 g/cm^3

Density (ρ_{Poz}) of Pozzolana = 1.4 g/cm^3

For 15 cm^3 volume (V) of OPC and Pozzolana

$$\begin{aligned} M_{\text{OPC}} &= \rho_{\text{OPC}} V = 2.3 \text{ g/cm}^3 \times (15 \text{ cm})^3 \\ &= 2.3 \times 3375 \\ &= 7,726.5 \text{ g} \approx 7.7 \text{ kg} \end{aligned}$$

$$\begin{aligned} M_{\text{OPC}} &= \rho_{\text{Poz}} V = 1.4 \text{ g/cm}^3 \times (15 \text{ cm})^3 \\ &= 1.4 \times 3375 \\ &= 4,725 \text{ g} \approx 4.7 \text{ kg} \end{aligned}$$

$$\begin{aligned} \text{Average mass (Pozzo and OPC) per cube} &= \frac{7.7 + 4.7}{2} \\ &= 6.2 \text{ kg} \end{aligned}$$

A volume of $(15 \text{ cm})^3$ is equivalent to 6.2kg of mass of materials admixture.

Composition: 0% to 80% Pozzolana partial replacement in a step of 10.

This implies nine (9) samples.

Therefore total number of concrete cubes (three each): $3 \times 9 = 27$ samples

Total mass (OPC + Pozzo) per batch = $27 \times 6.2 \text{ kg}$

$$= 167.4 \text{ kg}$$