A STUDY ON THE PROPERTIES OF BLENDED REGENERATED SPENT CATALYST AND CEMENT SANDCRETE BLOCKS

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DECLARATION

This thesis is the result of research work undertaken by Emmanuel Kofi Amissah in the Department of Nuclear Engineering, School of Nuclear and Allied Sciences, University of Ghana, under the supervision of Dr. A. Abugre and Dr. K. A. Danso.

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DEDICATION

To my heavenly Father be all the glory, honour and praise. This work is dedicated to my parents, Mr. John Kojo Amissah and Madam Gifty Acquah, and my lovely wife Elizabeth Arkoh for having provide me with rich education and fulfilling my spiritual needs throughout their immeasurable support, encouragement, love, care and prayers.
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# TABLE OF CONTENTS

DECLARATION .......................................................................................................................... ii  
DEDICATION ........................................................................................................................... iii  
ACKNOWLEDGEMENT .............................................................................................................. iv  
TABLE OF CONTENTS ........................................................................................................ v  
LIST OF FIGURES ................................................................................................................ xi  
LIST OF TABLES .................................................................................................................. xii  
LIST OF ABBREVIATION ...................................................................................................... xiv  
LIST OF SYMBOLS .............................................................................................................. xiv  
ABSTRACT .............................................................................................................................. 1  
CHAPTER ONE ...................................................................................................................... 2  
INTRODUCTION ................................................................................................................... 2  
1.1 BACKGROUND .............................................................................................................. 2  
1.2 Statement of Problem .................................................................................................... 5  
1.3 Objectives ..................................................................................................................... 6  
1.4 Relevance and Justification .......................................................................................... 6  
1.5 Scope and Delimitation ................................................................................................. 7  
1.6 Organization of Thesis ................................................................................................. 7  
CHAPTER TWO ..................................................................................................................... 8  
LITERATURE REVIEW ......................................................................................................... 8  
2.1 Background ................................................................................................................... 8  
2.2 Catalytic Cracking ......................................................................................................... 9  
2.2.1 Catalyst Structure and Reaction Mechanism ....................................................... 10  
2.2.2 Characterization of Catalyst ................................................................................... 11  
2.2.3 Changes in Catalyst Properties ............................................................................. 12  
2.2.4 Pozzolanic Activity and Mechanical Properties ................................................... 12  
2.3 Sandcrete ...................................................................................................................... 16  
2.3.1 Composition of Sandcrete ..................................................................................... 17  
2.3.2 Properties of Sandcrete ......................................................................................... 19  
2.3.3 Useful Life ............................................................................................................. 22  
2.3.4 Impact of Modern Sandcrete Use ......................................................................... 22
2.4 Importance of Measurements & Expression of Results in Reporting ............................................. 24
  2.4.1 Quality Management Systems and Accreditation ................................................................. 25
  2.4.2 The Importance of Quality ................................................................................................... 27

2.5 Cement in Ghana ......................................................................................................................... 30
  2.5.1 Why Check the Quality of Cement ..................................................................................... 31
  2.5.2 Important Physical Properties of Cement ........................................................................... 32
  2.5.3 Chemical Properties of Cement .......................................................................................... 33
  2.5.4 Standard Strength of Cement ............................................................................................. 34
  2.5.5 Testing of Cement .............................................................................................................. 35
  2.5.6 Physical Test ....................................................................................................................... 36
  2.5.7 Standard Consistency Test .................................................................................................. 36
  2.5.8 Setting Times ....................................................................................................................... 37
  2.5.9 Fineness of Cement ............................................................................................................ 37
  2.5.10 Compressive Strength ....................................................................................................... 38
    2.5.10.1 Effect of Cement Variations on Compressive Strength .............................................. 38
  2.5.11 Deterioration of Cement with Storage .............................................................................. 38
  2.5.12 The Importance of Curing .................................................................................................. 39
  2.5.13 Quality Management System in the Testing Laboratory .................................................... 41
  2.5.14 Benefits of Standards ........................................................................................................ 41
  2.5.15 Evolution of ISO 17025 and Its Importance ....................................................................... 42
  2.5.16 Benefits of Accreditation and the Accreditation Process .................................................. 43
  2.5.17 Importance of Accurate and Reliable Data ........................................................................ 44

CHAPTER THREE ............................................................................................................................ 47
RESEARCH METHODOLOGY ............................................................................................................. 47
  3.0 Introduction ............................................................................................................................... 47
  3.2 The study Area .......................................................................................................................... 47
  3.3 The Research Approach and Strategy ....................................................................................... 48
  3.4 Research Procedure .................................................................................................................. 48
  3.5 Data Collection and Experimentations ....................................................................................... 49
    3.5.1 Primary Data Collection .................................................................................................... 49
    3.5.2 Secondary Data .................................................................................................................. 50
3.6 Data Collection Techniques .......................................................................................... 52
3.7 Methods for Testing and Determining of Strength of Samples .................................. 54
3.8 Laboratory and Equipment ......................................................................................... 54
  3.8.1 Mixer ................................................................................................................... 55
  3.8.2 Preparation of Sandcrete, Placing, Conditioning and Testing ............................. 55
  3.8.3 Determination of Setting Times and Standard Consistence Test ....................... 58
     3.8.3.1 Principles ...................................................................................................... 58
     3.8.3.2 Standard Consistence Test ........................................................................... 58
     3.8.3.3 Setting Times Test; Initial and Final ........................................................... 59
  3.8.4 Chemical Analysis of Cement ............................................................................. 60
     3.8.4.1 Blank Determinations .................................................................................. 60
     3.8.4.2 Preparation of Test Samples ....................................................................... 60
  3.8.5 Specification of Standard Sand ............................................................................ 61
  3.8.6 Analysis of Data .................................................................................................. 61

CHAPTER FOUR ................................................................................................................ 63
RESULTS AND DISCUSSIONS ...................................................................................... 63
  4.1 Compressive Strength ............................................................................................... 63
     4.1.1 Effect of Regenerated Spent Catalyst on Compressive Strength ..................... 65
  4.2 Setting Time ............................................................................................................. 66
     4.2.1 Effect of Regenerated Spent Catalyst on Setting Time .................................. 66
  4.3 Water Absorption ................................................................................................... 67
     4.3.1 Effect of Regenerated Spent Catalyst on Water Absorption ............................. 67
  4.4 Chemical Analysis .................................................................................................. 68
     4.5.1 Effect of Regenerated Spent Catalyst on Compressive Strength versus Setting .......................................................... 69
     4.5.2 Effect of Regenerated Spent Catalyst on Compressive Strength versus water 70
     4.5.3 Effect of Regenerated Spent Catalyst on Water Absorption versus Setting.... 71

CHAPTER FIVE ................................................................................................................ 72
CONCLUSIONS AND RECOMMENDATIONS .............................................................. 72
  5.1 Conclusion ............................................................................................................... 72
  5.2 Recommendations .................................................................................................. 73
REFERENCES ................................................................................................................. 74
APPENDIX ....................................................................................................................... 82
LIST OF FIGURES

Figure 2.1: ISO/IEC 17025 Requirements for Testing Laboratories ..................30

Figure 2.2: Some Cement Manufactured and Imported In Ghana ..................32

Figure 2.3: A Cement Properly and B. Improperly Stored ..........................40

Figure 2.4: Typical Process of Accreditation ....................................45

Figure 3.1: Flow chart for preparing and crushing sandcrete cubes ...............54

Figure 3.2: Mortar Mixer ........................................................................56

Figure 3.3: Compression Test Machine ................................................57

Figure 3.4: Set of sandcrete cubes ready for crushing ..............................57

Figure 3.5: Curing Tank for sandcrete cubes storage ..............................58

Figure 3.6: A Vicat Apparatus Accessories .........................................60

Figure 3.7: Sample being tested for its chemical composition ....................61

Figure 4.1: Compressive strength curve of various samples for 2daysCuring Period...64

Figure 4.2: Compressive strength curve of various samples for 7daysCuring Period...64

Figure 4.3: Compressive strength curve of various samples for 28 daysCuring Period...65

Fig 4.4: Setting Time curves for various samples .....................................66

Figure 4.5: Water absorption curve for various samples ...........................67

Figure 4.6 Chloride Content curve for various samples ...........................68

Fig.4.7. A graph of Compressive Strength against Setting Time at different RSC replacement in cement ..........................................................69

Fig.4.8. A graph of Compressive Strength against water Absorption at different RSC replacement in cement ............................................70
Fig.4.9. A graph of water Absorption against Setting Time at different RSC replacement in cement.
LIST OF TABLES

Table 2.1: Chemical Requirements of Cement ...............................................................35
Table 3.1: Proportion of materials in sandcrete mixture ..............................................51
Table 3.2: Chemical Composition of Portland Ghacem Cement .................................52
Table 3.3: Physical Properties Portland Ghacem Cement .............................................52
Table 3.4 Chemical Composition of Regenerated Spent Catalyst (RSC) .......................52
Table 3.5 Physical Properties of Regenerated Spent Catalyst (RSC) ..............................53
Table 3.6: Standard sand requirement ...........................................................................62
# LIST OF ABBREVIATION

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AR</td>
<td>Atmospheric Residue</td>
</tr>
<tr>
<td>AAS</td>
<td>Atomic Absorption Spectrometry</td>
</tr>
<tr>
<td>CDU</td>
<td>Crude Distillation Unit</td>
</tr>
<tr>
<td>CMT</td>
<td>Construction Materials Testing</td>
</tr>
<tr>
<td>ECAT</td>
<td>Equilibrium Catalyst</td>
</tr>
<tr>
<td>FCC</td>
<td>Fluid Catalytic Cracking</td>
</tr>
<tr>
<td>GHAIP</td>
<td>Ghanaian Italian Petroleum Company</td>
</tr>
<tr>
<td>LPG</td>
<td>Liquefied Petroleum Gas</td>
</tr>
<tr>
<td>LCO</td>
<td>Light Cycle Oil</td>
</tr>
<tr>
<td>MES</td>
<td>Minimum Efficient Scale</td>
</tr>
<tr>
<td>OCM</td>
<td>Organization Change Management</td>
</tr>
<tr>
<td>OD</td>
<td>Organization Development</td>
</tr>
<tr>
<td>OPC</td>
<td>Ordinary Portland Cement</td>
</tr>
<tr>
<td>PM</td>
<td>Pozzolanic material</td>
</tr>
<tr>
<td>PLC</td>
<td>Portland Lime stone Cement</td>
</tr>
<tr>
<td>RSC</td>
<td>Regenerated Spent Catalyst</td>
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</table>
RFCC  Residue Fluid Catalytic Cracking
TOR  Tema Oil Refinery
TQM  Total Quality Management
XRF  X-Ray Fluorescence
LIST OF SYMBOLS

%  percentage
μ  linear attenuation coefficient
A  ampere
mm  millimeter
mm²  square millimeter
wt  weight
MPa  mega Pascal
N  Newton
k  kilo
kN  kilo Newton
N/ mm²  Newton per square millimeter
min  minute
°C  degree celsius
K  Kelvin
g  grams
m²/kg  square metre per kilogram
cm³/g  cube centime per gram
m²/g  square metre per gram
kP  kilo Pascal
ABSTRACT

Sandcrete is widely used as building material. Its properties greatly depend on the properties and proportions of its constituents. The main binder material to produce sandcrete is the Portland cement. The uncertainty about future availability of commonly used Portland materials concomitantly with the environmental problems such as greenhouse gases emissions and high cost of clinker consumption are highlighting the need of identifying other materials for the construction industry, which will aid in minimizing the clinker consumption and reduce the greenhouse gas emissions and cost in the production of cement. The purpose of this study is to examine the properties of sandcrete blocks produced with blended Regenerated Spent Catalyst and cement. In this work, two different series of sandcrete mixtures in which cement was partially replaced with Regenerated Spent Catalyst (RSC) within the range of 5% to 20% (by mass) with an increment of 5%. 100% cement sandcrete was also prepared as reference sandcrete. The physical properties studied were compressive strength, water absorption and setting time. Chemical property studied was chloride content. Comparison of data between the control and that of cement with additives were made. The results obtained in this study clearly indicated that substituting Portland cement up to 20 wt. % RSC gave sandcrete strengths higher than the 32.5 N/mm$^2$, which corresponds to that of Portland cement. The replacement of Portland cement with 10 wt. % of RSC gave the highest strength of 34.0 N/mm$^2$. Thus, Regenerated Spent Catalyst may be utilized as effective mineral additive for designing durable sandcrete structures. The optimum amount of RSC recommended to be added as an additive to the Portland cement is 10%.
CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND

The Construction industry is important in the development of the building and construction sector in any economy, especially in the provision of infrastructure such as harbours, airports and roads, simple residential buildings, high rise buildings, and massive structures like dams, flyovers and bridges.

The widely used building material is sandcrete or concrete. Its properties greatly depend on the properties and proportions of its constituents. As cement is the major component of concrete or sandcrete, the choice of its proper type and use has vital importance in obtaining the right balance of its desired properties in the most economical way.

Sandcrete is a composite material composed of aggregate bonded together with fluid cement which hardens over time. Most use of the term "concrete" refers to Portland cement concrete or concretes made with other hydraulic cements, such as cimentfondu. Type I/II Portland cements, which provide sufficient levels of strength and durability, are the most common cements used by sandcrete or concrete users. However, road surfaces are also a type of concrete, "asphaltic concrete", where the cement material is bitumen.

In Portland sandcrete (and other hydraulic cement sandcrete), when the aggregate is mixed together with the dry cement and water, they form a fluid mass that is easily molded into shape. The cement reacts chemically with the water and other ingredients to form a hard matrix which binds all the materials together into a durable stone-like material that has many uses. Often, additives (such as pozzolans or superplasticizers) are
included in the mixture to improve the physical properties of the wet mix or the finished material. Most sandcrete or concrete is poured with reinforcing materials (such as rebar) embedded to provide tensile strength, yielding reinforced concrete or sandcrete

Reinforced sandcrete is still the protagonist in the construction of structures, due to its numerous advantages over other materials (Fernández, 2002). It is the most widely used material in the construction industry in developed and developing countries; compared with other materials it is cheaper and has adequate strength and durability (Malhotra & Mehta, 1996).

The main binder material to produce sandcrete or concrete is the Portland cement; however, its production generates large amounts of greenhouse gases (Gartner, 2004). The reduction of these gaseous emissions can be achieved by increasing the efficiency in the cement production process through reduction in fuel consumption and in the use of clinker (Price et al., 1999).

The reduction in the use of clinker can be achieved through the incorporation of mineral admixtures at the time of manufacturing sandcrete or concrete, besides, the use of mineral admixtures is an alternative to improve concrete resistance to different aggressive environments (Lothenbach et al., 2011). For this reason it has become a common practice to use additions of cementitious materials, which usually are byproducts of industrial processes or other materials of natural origin (Sánchez de Rojas et al., 1996).

There are several benefits associated with the use of pozzolans in sandcrete and concrete, including improved mechanical strength and increased durability (Lothenbach et al.,
Additionally, the use of pozzolans brings in many cases economic and environmental benefits. It is considered that in the future, a concrete without pozzolanic or cementitious additions will be an exception to the rule (Malhotra & Mehta, 1996). The use of these pozzolanic waste materials brings environmental two-fold benefits: on one hand the substitution of a portion of cement, and on the other hand, the consumption of waste materials from other industrial processes, which when not used, would have to be stored in landfills (Roskovic & Bjegovic, 2005).

There is another pozzolanic material that is able to improve the performance of binders. It is the Spent Catalyst in the process of Catalytic Cracking of oil (FCC - Fluid Catalytic Cracking) which has been used on research in pastes, sandcretes and concrete (Castellano, 2014). It has been found that this material acts as a very active pozzolan since the early ages of curing, as indicated by Soriano, 2008; Payá et al., 2001; Antiohos et al., 2006. Its activity is due to its ability to react with the calcium hydroxide (portlandite) released in the hydration of Portland cement and form compounds with hydraulic properties. This behavior causes additional increase in the mechanical strength of sandcrete and concrete (Borrachero et al, 2002). Since FCC catalyst has a high reactivity at an early age, it may improve the mechanical properties and durability of Portland cement sandcretes or concretes when added as cementing material (Morozov et al., 2013). From environmental and economic points of view, the use of this residue could contribute to the conservation and preservation of the environment as well as obtaining better performance sandcrete or concrete.
This research presents a study on the properties of sandcrete blocks with Regenerated Spent Catalyst blended cement. It compares also optimal ratios for maintaining cement quality or properties and identifies and compares the relevant quality properties.

1.2 Statement of Problem

The main binder material to produce sandcrete or concrete is the Portland cement; however, its production generates large amounts of greenhouse gases (Gartner, 2004). The reduction of these gaseous emissions can be achieved by increasing the efficiency in the cement production process, through reduction in fuel consumption and the use of clinker (Price et al., 1999).

The reduction in the use of clinker can be achieved through the incorporation of mineral admixtures at the time of manufacturing sandcrete or concrete, besides, the use of mineral admixtures is an alternative to improve sandcrete resistance to different aggressive environments (Lothenbach et al., 2011).

For this reason, it has become a common practice to use additions of cementitious materials, some of which are byproducts of industrial processes or other materials of natural origin.

However as a result of the increase in cement demand in the Ghanaian economy, there is the need for the use of supplementary cementing material which will aid in minimizing the clinker consumption and reduce the greenhouse gas emissions and cost in the production of cement.
It is against this background that this work is identifying additional materials which bring both environmental and financial benefits such as the substitution of a portion of cement which is able to comply with standard construction materials and their performance target as well, reduction in the cost of production of clinker and greenhouse gas emissions particularly CO$_2$ and to add value to the Regenerated Spent Catalyst (RSC) obtained from Residue Catalytic Cracking Unit of Tema Oil Refinery (TOR) Limited, which when not used, would have to be environmentally disposed off.

1.3 Objectives

This research presents a study on the properties of sandcrete blocks produced with regenerated spent catalyst blended cement.

The specific objectives are:

1. Establish the Compressive Strength of the sandcrete blocks
2. Establish the Initial and Final setting times of the sandcrete mixture
3. Evaluate the Water Absorption of the control sandcrete blocks
4. Evaluate the chloride content of the sandcrete mixture
5. To compare the calculated values with the standard specification.
6. To Identify optimal ratios for maintaining cement quality or properties

1.4 Relevance and Justification

The uncertainty about future availability of commonly used Portland materials concomitantly with the environmental problems such as greenhouse gases emissions and high cost of clinker consumption are highlighting the need of identifying other materials for the construction industry.
1.5 Scope and Delimitation

The area of study is within the capital Accra where samples for testing was collected. This research work will be limited to few parameters such as Compressive Strength, Setting Time, Water Absorption, and chloride content due to the period allocated for conducting this research work. The experiment was conducted in the Engineering Department of the Ghana Standard Authority (GSA) since they are legally mandated to ensure safety and standard of goods.

1.6 Organization of Thesis

The thesis is arranged in five chapters. Chapter One provides an introduction including problem statement, objectives, relevance and scope of the study. Chapter Two reviews the existing literature relevant to the research problem. Chapter Three focuses on the materials and the analytical techniques used in the study. The results obtained are presented and discussed in Chapter Four. Conclusion of the study, recommendations and suggestions for further study are presented in chapter five.
CHAPTER TWO

LITERATURE REVIEW

2.1 Background

The refinery is in Tema, the most industrialized city in Ghana. It is 24 kilometres (15 mi) from Accra, the capital of Ghana. The refinery was first named the Ghanaian Italian Petroleum Company (GHAIP). It was licensed as a public limited liability company in 1960 as a fully owned Italian company – ANIC Societa per Azioni and AGIP Societa per Azioni of Italy were its major shareholders. In 1977, the Government of Ghana became the sole shareholder. The name of the refinery was changed to the Tema Oil Refinery in 1991.TOR is authorized by law to operate both as a refiner of crude oil and seller of petroleum products.

In the mid 1970’s and 1990’s it was realised that the capacity of the Refinery will be unable to support the growing increase of the market demand for white petroleum products( Gasoline/ Petrol, Diesel/ Gas oil and kerosene) and Liquefied Petroleum Gas (LPG) respectively. Therefore the Refinery embarked on a Modernisation Programme in Phases in 1966.

In Phase I, the Crude Distillation Unit (CDU) was revamped. The revamp project increased the processing capacity of the CDU from 28,000 Barrels Per Stream Day (BPSD), an equivalent of 1,250,000 metric tons of crude oil per year to 45,000 BPSD, that is 2,000,000 metric tons of crude oil per year.

Tema Oil Refinery (TOR) Limited undertook the Phase II of the Modernisation
Programme by constructing and commercially commissioning the Residue Fluid Catalytic Cracking (RFCC) unit in 2002.

The RFCC Unit converts low value Atmospheric Residue (AR) into high value Petroleum products namely LPG, Gasoline and Light Cycle Oil (LCO) using large quantities of Catalytic Cracking Catalysts. The operation of the RFCC unit generates 2-3 metric tons of used catalyst termed Regenerated Spent Catalyst (RSC) daily. The RSC is considered as a waste product.

2.2 Catalytic Cracking

Catalytic cracking is a process aimed at modifying the molecular structure of certain hydrocarbons, to obtain high quality petroleum products. The catalyst used for cracking process is a material consisting of a type Y zeolite which is a microporous crystalline aluminosilicate consisting of tetrahedra \([\text{SiO}_4]^4\) and \([\text{AlO}_4]^{5-}\). The catalyst undergoes rigorous treatments in the regenerator during the catalytic cracking process which changes its behaviour, forming an amorphous material with consequent loss of activity for the cracking process (AgamezPertuz et al., 2006). Some of these Equilibrium Catalyst(ECAT) are removed in the cracking units as they present low activity and are replaced by new catalysts.(Garcia et al., 2006).

Studies on materials manufactured with this by-product have shown that they are not dangerous as they comply with environmental requirements (Furimsky, 1996, Su et al., 2000).
2.2.1 Catalyst Structure and Reaction Mechanism

The first cracking catalysts were natural clays. These were used in their original forms after activation by acid treatment. Acid treatment increased the surface area and porosity of the clays and this considerably modified their physical and chemical properties. Crystalline clays of bentonite type were mostly used. The main component of bentonite is montmorillonite, $\text{Al}_2\text{O}_3.4\text{SiO}_2.2\text{H}_2\text{O}.n\ \text{H}_2\text{O}$. Activated minerals of kaolinite $\text{Al}_2\text{O}_3.2\text{SiO}_2.2\text{H}_2\text{O}$ and halloysite $\text{Al}_2\text{O}_3.2\text{SiO}_2.2\text{H}_2\text{O}.\text{H}_2\text{O}$ were also used. Natural clays were soon replaced with synthetic catalysts consisting of amorphous alumina-silica. Comparatively, amorphous alumina-silica, with better thermal stability and mechanical strength produced higher octane gasoline.

As far as gasoline production is concerned, the technology of catalytic cracking underwent a drastic change in 1962 with the advent of the third generation catalyst known as zeolites and based on crystalline alumina-silica with better kinetic properties (activity, selectivity and stability) are 10-100 times more active than the conventional catalysts. Mordenite and faujasite are examples. Some zeolites occur naturally whilst the synthesized are known as synthetic zeolites.

The basic structural element of alumina –silica is a tetrahedron, the nucleus of which is a silicon ($4\text{Si}^{4+}$) or aluminium ($\text{Al}^{3+}$) ion with an oxygen atom at each corner. The association of elementary tetrahedral $[\text{SiO}_4]^{4-}$ and $[\text{AlO}_4]^{5+}$ with common oxygen atom linkages form complex three-dimensional structures. The isomorphic displacement of $\text{Si}^{4+}$ and $\text{Al}^{3+}$ ions due to negative charge of the framework is compensated by cations, such as $\text{Na}^+$ however, zeolites crystalline in nature have more pronounced three dimensional structure with repetitive identical structural patterns. In the petroleum
industry the most important of such patterns are based on a sodalite cage, a hollow truncated octahedron with 8 hexagonal rings and 6 square rings on its surface.

Solid phase reaction, occurring between the zeolites and matrix, can be represented as follows:

\[(\text{Me}^{3+}\text{Na}^{+})\text{Y} + \text{H.Gel (Sol)}^{3/4} \rightarrow (\text{Me}^{3+}\text{H})\text{Y} + \text{H.Gel (Sol)}\].

The Na+ Gel (Sol) is distributed throughout the matrix. As a rule, modern cracking catalysts contain 10-20 wt.% of zeolites type REHY that is the active component during synthesis.

2.2.2 Characterization of Catalyst

Knowledge of chemical, structural and surface characteristics of catalyst is essential for understanding the catalytic phenomena taking place on its surface controlling reaction the measurement of catalyst surface area and pore volume is the first step in characterizing the catalyst behavior. The well-known BET method, developed by Brunauer et al (1938) is used in determining the adsorption isotherm graph from which the catalyst surface area, pore size, shape and volume can be extracted from the isotherm.

The chemical characterization refers to the examination of the compositional, structural, morphological and surface properties of catalyst. Techniques such as Atomic Absorption Spectrometry (AAS) and X-flourescence (XRF) are used for this purpose. Whilst AAS uses the the absorption of incident radiation by the vaporized atoms of the elements being analyzed, XRF is based on the emission of characteristic secondary X-rays (fluorescence) when the sample is irradiated with X-ray beam. The structural and morphological
information can also be determined by use of electron microscopy techniques such as Scanning Electron Microscopy (SEM) and transmission Electron Microscopy (TEM).

### 2.2.3 Changes in Catalyst Properties

The industrial Catalytic Cracking Units, the catalyst operates continuously in a cycle, reactor-regenerator – reactor and undergoes significant changes in activity, selectivity, composition, mechanical strength, apparent density, pore structure and other properties. During operations the catalyst undergoes changes in structure and texture and active sites due to deactivation by coke deposits and poisoning by metal- organic sulphur and nitrogen containing compounds (A. Abugre, 1986).

The development of a more effective catalyst for a given feedstock can significantly increase the performance of catalyst cracking unit, i.e. increase the conversion of feed stock and consequently yield large volumes of high quality products (Stiles, 1987).

Examples of some cracking catalysts for residual oils, gas oils and mixtures:

- DAB, GX, RC, Super-D, KMZ, ULTRASIN, MAGNASIV for gas oil, residue and mixtures thereof
- XI, MRZ, HFZ, HEZ, DOC, HA, and HAY for gas oil cracking

### 2.2.4 Pozzolanic Activity and Mechanical Properties

A pozzolanic material is by definition capable of binding calcium hydroxide in the presence of water. Therefore, the chemical measurement of this pozzolanic activity
represents a way of evaluating pozzolanic materials. This can be done by directly measuring the amount of calcium hydroxide a pozzolan consumes over time.

The pozzolanic activity is a measure for the degree of reaction over time or the reaction rate between a pozzolan and \( \text{Ca}^{2+} \) or \( \text{Ca} (\text{OH})_2 \) in the presence of water. The rate of the pozzolanic reaction is dependent on the intrinsic characteristics of the pozzolan such as the specific surface area, the chemical composition and the active phase content.

The rate of the pozzolanic reaction can also be controlled by external factors such as the mix proportions, the amount of water or space available for the formation and growth of hydration products and the temperature of reaction. Therefore, typical blended cement mix design properties such as the replacement ratio of pozzolan for Portland cement, the water to binder ratio and the curing conditions strongly affect the reactivity of the added pozzolan.

Mechanical evaluation of the pozzolanic activity is based upon a comparison of the Compressive Strength of sandcrete or mortar bars containing pozzolans as a partial replacement for Portland to reference mortar bars containing only Portland cement as binder. The sandcrete or mortar bars are prepared, cast, cured and tested following a detailed set of prescriptions. Compressive Strength testing is carried out at fixed moments, typically 2, 7, and 28 days after mortar preparation. A material is considered pozzolanically active when it contributes to the compressive strength, taking into account the effect of dilution.

The following are the results of investigations characterising the residue and its inclusion as a replacement for cement to measure its reactivity.
Pacewska compared the lime fixation in several cement paste pozzolans, including FCC, silica fume (SF) and fly ash (FA) (Pacewska et al., 1998).

They concluded that setting time seemed to have been accelerate by the presence of pozzolans, especially FCC. They found that 28 days of curing fixed lime was similar for HS and FCC. Pastes having added FCC resistance showed improved mechanical strength after 7 days of curing, this being superior to the other pastes tested.

In a later study, Pacewska studied the influence of different percentages regarding FCC substitution for cement (Pacewska et al., 2000). They concluded that a small addition of FCC (5-10% cement substitution) acted as a system accelerator. However, higher additions of catalyst (over 10%) led to the heat released after 72 hours decreasing, probably because of smaller amounts of calcium silicate hydrate (CSH) phase being formed.

Studies have also been conducted on the influence of particle size from the two wastes generated in the process Equilibrium Precipitator Catalyst (EPCAT) and Equilibrium Catalyst (ECAT). Pacewska concluded from calorimetry studies that if the substitution of cement by EPCAT was between 5%-10%, then the hydration process became accelerated (Pacewska et al., 2002).

Wang-Lung characterised EPCAT and studied lime fixation in pozzolan-added cement pastes (Wang-Lung et al., 2003). Following characterisation, the authors reported that the residue contained irregular shaped particles and were mainly composed of faujasite, silica, kaolinite and mullite. They concluded that lime fixation increased percentage EPCAT substitution. Similar results were found by Hsiu-Jung, Wu et al., (2003). Kung-
Chung et al. (2005) found that mortars containing this residue exhibited greater resistance to compression due to their high reactivity.

Yun-Sheng subjected the material to 650°C to increase ECAT reactivity (Yun-Sheng et al., 2005). The authors reported an increase in mechanical strength of between 8% and 18% for mortars and 7% and 11% in added concrete compared to reference samples.

Similarly, Nan Su studied EPCAT and ECAT properties (Su et al., 2000) and reported that mortars replaced by EPCAT (5% to 15%) showed greater resistance than mortars replaced with ECAT because EPCAT particles are much smaller than those of ECAT. The same conclusion was reached by Hsiu-Liang (Hsiu-Liang et al., 2004).

Pacewska has studied the benefits it can have on FCC milling (Pacewska et al., 2002 pp.133-142). The authors found that the pastes added to the catalyst powder consumed more calcium hydroxide Ca(OH)$_2$ than control sample and those added to the original catalyst. Other authors have recommended reducing FCC particle size to increase the compressive strength of mortars containing it (Payá J et al., 1999; Basaldella E et al., 2006; Pacewska B et al., 2002). Torres made a preliminary study of the pozzolanic activity of a catalyst residue from a Colombian petroleum industry (Torres et al., 2008, 2009). Compressive strength and lime consumption were measured.

The authors concluded that this material presented good reactivity, thereby making it suitable for producing pozzolan-added sandcretes and concretes.

Currently, oil-refineries worldwide generate around 500,000 metric tons/year of waste catalyst in their fluid catalytic cracking units and, therefore, it is available in significant amounts. FCC catalysts are typically “taylor-made” for each oil-refinery based on the feed composition and desired products spectra. Despite not being a commodity, the
chemical-mineralogical and physical properties—namely the particle size distribution—of a given wFCC catalyst generated in the same oil-refinery remain almost constant for some years. As such, this by-product can be a steady supply for the construction materials industry.

By-products such as silica fumes, fly ashes and ground blast-furnace slags have been extensively used in past decades to replace part of the cement as PMs for different uses in construction industry Aïtcin, P-C. (2007) Siddique, R. and Khan, M.I. (2011). However, the uncertainty about future availability of commonly used PMs concomitantly with the aforementioned environmental and technological reasons are highlighting the need of identifying additional PMs able to comply with construction materials standards, performance targets as well as market needs. In this scope, the waste catalyst originated in the fluid catalytic cracking (RFCC) units by oil-refinery companies is being investigated. This study focuses on this purpose.

2.3 Sandcrete

Sandcrete is a composite material composed of aggregate bonded together with a fluid cement which hardens over time. Most use of the term "concrete" refers to Portland cement concrete or to concretes made with other hydraulic cements, such as cimentfondu. However, road surfaces are also a type of concrete, "asphaltic concrete", where the cement material is bitumen.

In Portland cement sandcrete (and other hydraulic cement concretes), when the aggregate is mixed together with the dry cement and water, they form a fluid mass that is easily moulded into shape. The cement reacts chemically with the water and other ingredients to
form a hard matrix which binds all the materials together into a durable stone-like material that has many uses. Often, additives (such as pozzolans or superplasticizers) are included in the mixture to improve the physical properties of the wet mix or the finished material. Most sandcrete is poured with reinforcing materials (such as rebar) embedded to provide tensile strength, yielding reinforced concrete.

Famous concrete structures include the Hoover Dam, the Panama Canal and the Roman Pantheon. The earliest large-scale users of concrete technology were the ancient Romans, and sandcrete or concrete was widely used in the Roman Empire. The Colosseum in Rome was built largely of sandcrete or concrete, and the concrete dome of the Pantheon is the world's largest unreinforced concrete dome. Today, large sandcrete or concrete structures (for example, dams and multi-storey car parks) are usually made with reinforced sandcrete or concrete.

After the Roman Empire collapsed, use of sandcrete or concrete became rare until the technology was redeveloped in the mid-18th century. Today, concrete is the most widely used man-made material (measured by tonnage).

2.3.1 Composition of Sandcrete

Sandcrete is made up of three main ingredients: water, Portland cement, and aggregates. The ratio of the ingredients changes the properties of the final product, which allows the engineer to design sandcrete that meets their specific needs. Admixtures are added to adjust the sandcrete mixture for specific performance criteria.
Aggregate consists of large chunks of material in a concrete mix, generally a coarse gravel or crushed rocks such as limestone, or granite, along with finer materials such as sand which help increase the strength of the concrete beyond what cement can provide on its own.

Cement, most commonly Portland cement, is associated with the general term "concrete." A range of materials can be used as the cement in sandcrete or concrete. One of the most familiar of these alternative cements is asphalt. Other cementitious materials such as fly ash and slag cement, are sometimes added as mineral admixtures (see below) - either pre-blended with the cement or directly as a concrete component - and become a part of the binder for the aggregate.

To produce sandcrete or concrete from most cements (excluding asphalt), water is mixed with the dry powder and aggregate, which produces a semi-liquid that workers can shape, typically by pouring it into a form. The concrete or sandcrete solidifies and hardens through a chemical process called hydration. The water reacts with the cement, which bonds the other components together, creating a robust stone-like material.

Sandcrete has relatively high compressive strength, but significantly lower tensile strength, and as such is usually reinforced with materials that are strong in tension (often steel). The elasticity of concrete or sandcrete is relatively constant at low stress levels but starts decreasing at higher stress levels as matrix cracking develop. Sandcrete has a very low coefficient of thermal expansion, and as it matures concrete or sandcrete shrinks. All concrete structures will crack to some extent, due to shrinkage and tension. Sandcrete, which is subjected to long-duration forces, is prone to creep.
Tests can be made to ensure the properties of sandcrete correspond to specifications for the application. The density of concrete varies, but is around 2,400 kilograms per cubic metre. As a result, without compensating, concrete would usually fail from tensile stresses even when loaded in compression. The practical implication of this is that concrete elements subjected to tensile stresses must be reinforced with materials that are strong in tension.

Sandcrete or concrete can also be prestressed (reducing tensile stress) using internal steel cables (tendons), allowing for beams or slabs with a longer span than is practical with reinforced sandcrete alone. Inspection of existing concrete structures can be non-destructive if carried out with equipment such as a Schmidt hammer, which is sometimes used to estimate relative concrete strengths in the field.

Admixtures accomplish a variety of goals. This can be as simple as adding a pigment to colour the sandcrete. Other admixtures are used for faster curing times in cold weather or as may require in some cases, creating extremely high-strength sandcrete, or for increasing the flowable nature of sancrete without compromising the strength.

2.3.2 Properties of Sandcrete

Sandcrete has relatively high compressive strength, but much lower tensile strength. For this reason it is usually reinforced with materials that are strong in tension (often steel). The elasticity of concrete is relatively constant at low stress levels but a start decreasing at higher stress levels as matrix cracking develops. Sandcrete has a very low coefficient
of thermal expansion and shrinks as it matures. All concrete structures crack to some extent, due to shrinkage and tension. Sandcrete that is subjected to long-duration forces is prone to creep.

The ultimate strength of concrete is influenced by the water-cementitious ratio \((w/cm)\), the design constituents, and the mixing, placement and curing methods employed. All things being equal, concrete with a lower water-cement (cementitious) ratio makes a stronger concrete than that with a higher ratio. The total quantity of cementitious materials (portland cement, slag cement, pozzolans) can affect strength, water demand, shrinkage, abrasion resistance and density. All concrete will crack independent of whether or not it has sufficient compressive strength. In fact, high Portland cement content mixtures can actually crack more readily due to increased hydration rate. As concrete transforms from its plastic state, hydrating to a solid, the material undergoes shrinkage. Plastic shrinkage cracks can occur soon after placement but if the evaporation rate is high they often can actually occur during finishing operations, for example in hot weather or a breezy day. In very high-strength concrete mixtures (greater than 70 MPa) the crushing strength of the aggregate can be a limiting factor to the ultimate compressive strength. In lean concretes (with a high water-cement ratio) the crushing strength of the aggregates is not so significant. The internal forces in common shapes of structure, such as arches, vaults, columns and walls are predominantly compressive forces, with floors and pavements subjected to tensile forces. Compressive strength is widely used for specification requirement and quality control of concrete. Engineers know their target tensile (flexural) requirements and will express these in terms of compressive strength.
Different mixes of sandcrete ingredients produce different strengths. Sandcrete strength values are usually specified as the compressive strength of either a cylindrical or cubic specimen.

Different strengths of sandcrete are used for different purposes. Very low-strength - 14 MPa (2,000 psi) or less - concrete may be used when the concrete must be lightweight. Lightweight concrete is often achieved by adding air, foams, or lightweight aggregates, with the side effect that the strength is reduced. For most routine uses, 20 MPa (2,900 psi) to 32 MPa (4,600 psi) concrete is often used. Readily commercially available concrete is 40 MPa (5,800 psi) as a more durable, although more expensive option. Higher-strength concrete is often used for larger civil projects. Strengths above 40 MPa (5,800 psi) are often used for specific building elements. For example, the lower floor columns of high-rise concrete buildings may use concrete of 80 MPa (11,600 psi) or more, to keep the size of the columns small. Bridges may use long beams of high-strength concrete to lower the number of spans required. Occasionally, other structural needs may require high-strength concrete. If a structure must be very rigid, concrete of very high strength may be specified, even much stronger than is required to bear the service loads. Strengths as high as 130 MPa (18,900 psi) have been used commercially for these reasons.
2.3.3 Useful Life

Sandcrete can be viewed as a form of artificial sedimentary rock. As a type of mineral, the compounds of which it is composed are extremely stable. Many concrete structures are built with an expected lifetime of approximately 100 years, but researchers have suggested that adding silica fume could extend the useful life of bridges and other concrete uses to as long as 16,000 years. Coatings are also available to protect concrete from damage, and extend the useful life. Epoxy coatings may be applied only to interior surfaces, though, as they would otherwise trap moisture in the sandcrete.

2.3.4 Impact of Modern Sandcrete Use

Sandcrete is widely used for making architectural structures, foundations, brick/block walls, pavements, bridges/overpasses, highways, runways, parking structures, dams, pools/reservoirs, pipes, footings for gates, fences and poles and even boats. Concrete is used in large quantities almost everywhere mankind has a need for infrastructure.

Reinforced concrete is one of the most widely used materials today due to its numerous advantages. It has been concluded that the product most consumed by man, after water, is concrete by simple comparison between overall concrete production and the world's population (Fernández, 2002).

The main cementitious material is currently Portland cement; however, its production produces large amounts of greenhouse gas (Gartner, 2004). The emission of these gases
can be decreased by reducing fuel consumption or reducing clinker production by incorporating mineral admixtures when manufacturing mortar or concrete (Price et al., 1999). It is thus a common practice to use additions to and replacement for cementitious material in mixtures which are usually by-products from other processes or natural materials.

It is generally accepted that the incorporation of pozzolanic materials (PMs) in cement based materials plays a crucial role towards sustainable development of the construction industry by addressing environmental and performance issues in an integrated way. In fact, partial replacement of cement with PMs reduces the use of non-renewable raw materials and energy as well as decreases the CO$_2$ footprint in cement plants, (Aïtcin, P-C. (2007). In addition, if the surrogate material is a waste from other industry it would also contribute to mitigate solid waste disposal of in landfills and to turn a polluting waste from one industry into a product with added-value for cement industry, Naik, T.R. and Moriconi G. (2005). Furthermore, PMs, typically, lead to performance and durability enhancement due to the formation of additionally strength-providing hydrated calcium aluminosilicates (C-A-S-H) products during PMs reaction with the Ca(OH)$_2$ (liberated upon cement hydration), Massazza, F. (2004).

The most noteworthy benefits obtained by using pozzolans are improved mechanical strength in mortars and concretes and increased durability (ACI 201, 2001). It is considered that using concrete lacking adding pozzolanic or cementitious materials will be an exception to the rule in the future (Malhotra and Mehta, 1996).
Using waste pozzolanic materials brings a double environmental benefit, first by replacing cement and other waste material consumption from other industrial processes and being stored in landfills when not used (Roskovic and Biegovic, 2005).

Sandcrete properties greatly depend on the properties and proportions of its constituents. As cement is the major component of concrete, the choice of its proper type and use has vital importance in obtaining the right balance of its desired properties in the most economical way.

Type I/II Portland cements, which provide sufficient levels of strength and durability, are the most common cements used by concrete users. The selection of a type of cement, involves the exact knowledge of the connection between the cement and performance required and, in particular, between the kind of cement and either strength or durability or both properties of concrete (Akroyd, 1962).

During the literature review, the author Akroyd, 1962 came to the conclusion that there is hardly any work carried out by researchers to investigate the application and benefits of ISO 17025 in relation to the evaluation of cement in Ghana, however there is enough literature available on the implementation, benefits, and improvement of the quality of cement. Studies carried out by researchers in the related area are highlighted below.

2.4 Importance of Measurements & Expression of Results in Reporting

Institutions used to operate the ISO/IEC Guide 25 and EN 45001 but the introduction of the ISO 17025 brought in a new approach to the testing industry (Anon, 2001). The later
had no processes for the expression of opinions and the interpretation of results and did not also allow for the inclusion of such opinions but the ISO 17025 came to correct that and had uniform rules to the expression of personal opinion and interpretation of results and the onus now lies on individual laboratories to get accreditation for that or not. Expression of opinions and interpretations relating to results are considered to be an inherent part of testing and calibration.

A range of measurements are dependent on socio-economic activities such as food safety, health, environmental protection and chemical analyses (F & Balgobin, 2011). Confidence in chemical measurements can only be boosted by their accuracy as we rely heavily on them. Laboratory accreditation, achieved through the implementation of the ISO/IEC 17025:2005 standard is the process that determines the competence of staff and laboratories in delivering accurate results consistently. Using accurate and reliable data, obtained from an internationally recognised accredited laboratory, eliminates the need for retesting.

2.4.1 Quality Management Systems and Accreditation

The use of quality management systems and laboratory accreditation is now acquiring significance in various sectors and quite a number of Companies are now aware of the benefits of implementing quality management systems and acquiring accreditation. Though the process of implementation and maintenance involves a lot of investment, organisations have become a lot more conscious of the fact that customer satisfaction is of top most importance to their businesses. Most government agencies are often more likely to seek the services of an accredited laboratory knowing that their decisions will be
more accurate and reliable. Unlike the previous papers reviewed, this one indicates the benefits of implementing the ISO/IEC 17025:2005 in testing laboratories. Accredited laboratories benefit from international recognition of their results which ultimately represents an advantage for those accredited laboratories. Standard (SPIN, 2012), dwelt on the methods and procedures of testing cement to the same standard across Africa. Each laboratory was provided with the same set of cement samples and tests were conducted on them using the same set of standards and procedures.

Results obtained indicated that most laboratories did not perform the required tests in the proficiency testing scheme under the approved standard temperatures but conclusion drawn indicated that 89.29% of all tests conducted on the cement sample fell within the limits given.

Malik & Khan, 2011, focused on the cement industry, specifically measuring the critical factors of Total Quality Management (TQM). The findings showed the extent and intensity of mapping the total quality management practice in the Pakistani cement industry. They also found that the cement industry needed to be careful for practices and putting in place improvement plans. The tools identified, will have to be utilized to compare and evaluate within the organizations the Total Quality Management practices. As compared to the previous studies reviewed, the study used only a small sample size and was limited to only the cement industry.

Similarly R & Chakrabarty, 2011 states that TQM and business performance has a positive and significant relationship. This relationship is based on the three concepts underpinning the theory of management; Organization Management (OM), Organization Change Management (OCM), and Organization Development (OD). Organizational
Management refers to the management of organizational activities towards meeting the customers’ requirements while addressing other aspects of the organization.

Organizational Management according to Tunaly, 1992, is used for improving efficiency and effectiveness of an organization. Organization Change Management covers the entire aspects of organizational performance measures. Planned and emergent changes are reviewed under this theory. Planned change is further divided into two sub-categories, namely evolutionary and revolutionary under which we have various other approaches (Wiklund, 1999). Organization Development on the other hand, is a common approach to managing change in organizations and helps improve the functioning of the total organization by educating all stakeholders how to continuously improve their own functioning. Total Quality Management (TQM) practices and performance in the cement industry have important relationships to assure and enhance the level of performance of management through the effective and efficient application of TQM including processes for continuous improvement and implementation of the quality system and procedure in accordance with the customers’ requirements. (R & Chakrabarty, 2011)

2.4.2 The Importance of Quality

The Indian Concrete Journal, (Sarkar, 2007) looked at the importance of quality and the fact that quality is a philosophy rather than an attribute. It further indicated that the application of quality management is now not only common, but now mandatory in the construction industry. Knowing some quality control methods is just not good enough. Adoption and implementation of all the control methods and tools is now the key to the success of the organization. Quality control thus means a control of all resources, and the
implementation of appropriate procedures. The journal sums up the causes of poor quality as ignorance, poor materials, poor detailing and poor workmanship, and lack of technical knowledge in the construction industry.

Cement can thus be defined as a fine mineral powder which when mixed with water, transforms into a paste that binds and hardens when submerged in water. Cement is the main component of concrete and it is an economical, high-quality construction material used in construction projects across the country.

The Construction Materials Testing ISO/IEC 17025 application document (2013) provides a criteria for the application of ISO/IEC 17025 in the field of Construction Materials Testing (CMT) for both applicant and accredited facilities. Applicant and accredited facilities must also comply with standard application documents and, policies (NATA, 2013) in providing testing and calibration laboratories with superior accreditation services.

Timely response is of top priority. Most laboratories are tied to time-sensitive mandates, schedule and timetables. The goal of every laboratory is to process reports and issue certificates with very little delays. (ISO/IEC, 2015)
In the testing of building materials, one will pride himself on being able to provide answers for virtually any construction-related testing question. A range of test facilities will have to be acquired, and experts employed to test this individual whole buildings, materials and products, engineering structures and, building systems, using well established methods, research tests and simulations. Tests are typically carried out against published standards, and where standards do not exist, new ones can be developed with dedicated procedures for new, unique or innovative products. There is often the responsibility of civil engineers specifying, manufacturing and designing the materials with which structures are built. Studies into building construction materials are often intended to make transportation, structural, and foundation engineers aware of the fundamental properties of the materials they use.
2.5 Cement in Ghana

Types of building materials i.e. Portland cements are manufactured to meet various physical and chemical requirements for purposes, such as durability and high-early strength etc. Cement which is a common building material is made from rock and widely used in the construction of most buildings in the world. The market studied, according to K & R Singh, 2010 is the market for Ordinary Portland Cement (OPC), Portland Lime stone Cement (PLC). PLC is cheaper to produce and generally durable for most construction work and is more commonly used in most developing countries than OPC.

An important input for construction and infrastructure development, the commodity cement, underpins growth and industrialisation. Thus the price and availability of cement is very important. The cement sector is one that often suffers from limited competition and has been a source of concern for authorities in many countries across the world. There is significant scope for anti-competitive practices in the cement industry to result in much higher prices than would otherwise be the case, with negative economic consequences.

In the cement market there is a high Minimum Efficient Scale (MES) which means that large players have a significant cost advantage, which generates a concentrated market structure with relatively few players. However, having fewer players in the market reduces competition, and makes the sector vulnerable to anti-competitive practices, which is likely to result in higher prices. The high risk of anti-competitive practices suggests there is a need for careful monitoring of the cement sector by competition authorities.

Prices of cement doubled in 2007 causing great concern in the building industry. Stakeholders alleged that because the few companies producing in Ghana enjoys strong
market power they are able to set higher prices without fear of losing customers. Most Stakeholders have also alleged that the dominant players block their attempts to import cement into the country through informal means. This assertion is however not true because the monopoly Ghacem cement had, had been broken about fifteen years ago.

![Figure 2.2: Some Cement Manufactured and Imported In Ghana](image)

2.5.1 Why Check the Quality of Cement

Savitha (2012) gives the importance of testing materials in particular cement. In the building industry. This is because an incorrect assessment of a material would ultimately be harmful to people and to the environment. The infrastructural development of a nation, eventually leads to the growth of that country. The use of high quality construction materials leads to high quality infrastructures. The quality of such materials should be assessed properly in an accredited and an accepted laboratory, using standard test
methods and specifications. Building materials should be chosen to comply with the relevant standards i.e. BS, EN, IS, GS and ASTM standards.

2.5.2 Important Physical Properties of Cement

ASTM C150, 2012 has identified some requirements regarding the physical properties for cement. These properties comprise: fineness, specific gravity, soundness, and standard consistency, setting time, compressive strength, heat of hydration and loss of ignition. In general sense, cements are cohesive and adhesive materials in nature which are normally capable of bonding together particles of solid matter into a solid durable mass. For civil engineering works, they are restricted to calcareous cements containing compounds of lime as their chief constituent; the primary function is to bind the fine and coarse aggregate particles together. In the use of cement in the construction industry, one can classify it as hydraulic or non-hydraulic. The latter does not set and harden in water such as non-hydraulic lime or which are unstable in water, e.g. Plaster of Paris. Hydraulic cement sets and hardens in water and gives the product which is stable. Care must be exercised in proportioning the raw materials so that the clinker of proper constitution may be obtained after burning.

The properties of cement conforming to the GS: 22: 2011 Standard shall when appropriately batched and mixed with aggregates and water be capable of producing mortar or concrete which retains its workability for a sufficient time and shall after a defined period attain specific strength levels and also possess long term volume stability.
The sum of the proportions of reactive calcium oxide and reactive silicon dioxide shall not be less than 50 % (m/m). Cement conforming to the standard shall consist of individual small production, in particular grinding and homogenisation processes.

In the manufacture of cement, clinker shall be made by burning, a specific mixture of raw materials which shall consist of not less than two thirds (2/3) by mass of calcium silicate, some aluminium, iron oxide and other oxides. The ratio by mass of calcium and silicon oxide shall not be less than 2.0 and magnesium oxide shall also not exceed 5.0% m/m by mass (Viyors, 2014) there are other additional minor constituents i.e. granulated blast furnace slag, pozzolana and fly ash. These additional constituents should not promote corrosion of the reinforcement or impair the properties of the cement or of the mortar or concrete made from the cement.

Calcium sulphate or gypsum has to be added in small quantities in order to control the setting time of the cement, and the total sulphates shall not exceed 3 to 5% m/m of the final cement. (Papadakis, 2005)

2.5.3 Chemical Properties of Cement

According to Anon, 2013, The Chemical Properties of cement are also checked to determine presence of certain properties like the Loss on ignition where a known weight of cement is heated until a constant weight is obtained. A high loss on ignition shows that the cement was inappropriately stored. This document also describes the reference methods and, in certain cases, an alternative method which can be considered to be equivalent. The loss on ignition shows the extent of carbonation and hydration of free lime and free magnesia due to the exposure of cement to the atmosphere. The insoluble residue, determined by treating with hydrochloric acid, is a measure of adulteration of
cement, largely arising from impurities in gypsum (Anon, 1996), limits the insoluble residue to 1.5 per cent of the mass of cement. The chemical properties when tested shall also conform to the table below:

Table 2.1

<table>
<thead>
<tr>
<th>Property</th>
<th>Test reference</th>
<th>Strength class</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Loss in ignition</td>
<td>ISO 680</td>
<td>All</td>
<td>&lt; 5.0 wt.%</td>
</tr>
<tr>
<td>Insoluble residue</td>
<td>ISO 680</td>
<td>All</td>
<td>&lt; 5.0 wt.%</td>
</tr>
<tr>
<td>Sulphate content</td>
<td>ISO 680</td>
<td>32.5N, 32.5R, 42.5N</td>
<td>&lt; 3.5 wt.%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>42.5R, 52.5R, 52.5N</td>
<td>&lt; 4.0 wt.%</td>
</tr>
<tr>
<td>Chloride content</td>
<td>EN 196-21</td>
<td>All</td>
<td>&lt; 0.10 wt.%</td>
</tr>
</tbody>
</table>

Source: GS -22, 2011

2.5.4 Standard Strength of Cement

The standard strength of cement is the compressive strength at 28 days and shall conform to the three classes of standard strength; class 32.5, class 42.5, and class 52.5
The early strength of cement is the compressive strength at two(2) days or seven(7) days. Two classes of early strength are included for class of standard. A class with ordinary early strength is classified as N and a class with high early strength is indicated by R.

2.5.5 Testing of Cement

There are two methods for testing cement, one performed in the laboratory, and the other performed on the field. According to Anon, 2010, Field Tests are a convenient way of initial inspection of cement when used in small scale works or when decision has to be made quickly during purchase. The following steps can be used to ensure a good quality cement.

1. There should not be any lumps formation inside the bag when it is first opened for visual inspection.

2. Put your hand inside the bag of cement. This should give you a feel of a cool sensation around your hands.

3. A sample of cement in your hand, rubbed in between your fingers should give you a feel of smoothness.

4. Take another handful sample of cement and throw it on the surface of a bucket full of water. Particles of cement should float a while before sinking down.

The Field Tests only indicate that cement is not bad and can be used for small scale works. More detailed tests is conducted in the laboratory to determine the quality of
cement. Laboratory tests according to (ISO/IEC, 2005) are necessary to confirm that the cement is good in nature and can be used. The following tests are conducted on cement in the laboratory.

2.5.6 Physical Test

The physical properties tested for are fineness which can also be termed as the particle size distribution of the cement. This affects the rate of hydration and directly impacts the rate of strength gain. Another property is the soundness which is the ability of a hardened cement paste to retain its volume after it has set. The presence of excessive hard burnt free lime results in a lack of soundness.

2.5.7 Standard Consistency Test

Consistency of cement refers to the ability of the cement paste to flow and is said to have normal consistency when the plunger of the Vicat apparatus penetrates it by 10 plus or minus one (10 ±1). Consistency indicates the degree of density of cement. Therefore, it is necessary to determine the amount of water for a given cement to get a mixture of the required consistency. Consistency of cement is measured by the Vicat apparatus. The paste is said to be of standard consistency, when the penetration of the plunger, is 33-35 mm. (Neville, 2010).
2.5.8 Setting Times

To enable the concrete to be laid in position properly the initial setting of cement should not start too quickly. Once the concrete is laid it should harden rapidly so that the structure could be put to use early. Initial setting of cement is that step in the process of setting, after which if any cracks appear do not reunite and the final setting is that time when sufficient strength and hardness is attained. Setting thus, describes “The strength of the cement paste”. During setting, the paste attains some strength. But it is different from hardening, which refers to the increase of strength of a set cement paste. The setting process is associated with temperature changes in the cement paste. Initial set is related to a quick increase in temperature and final set to the highest temperature. Akroyd, 1962.

2.5.9 Fineness of Cement

Fineness of cement is the total surface area of cement grains available for hydration. It also determines the level of grinding of cement clinker and affects the rate of hydration. The Greater the fineness, the more the surface available for hydration, resulting in greater early strength and a more rapid release of heat. The fineness of cement is measured as specific surface by observing the time taken for a fixed quantity of air to flow through a compacted cement bed of specified dimensions and porosity. Under standardized conditions the specific surface of cement is proportional to $\sqrt{t}$ where the time is for a given quantity of air to flow through the compacted cement bed. The number and size range of individual pores in the specified bed are determined by the cement particle size distribution, which also determines the time for the specified air flow, (Anon, 2013).
2.5.10 Compressive Strength

The compressive strength test of cement is defined in Mega Pascal (MPa) or Newton (N/mm²) and is carried out to check the strength of cement. For example, Portland cement of grade 32.5R, the strength should be 35MPa or 2 N/mm² in 28 days. In this case, the cement is mixed with sand and water to the standard measurement, the paste is then put in the mould, demoulded after 24 hours and cured for 28 days. After which it is crushed and the strength determined.

2.5.10.1 Effect of Cement Variations on Compressive Strength

The strength of concrete largely depends on the water-cement ratio, the quality and characteristics of cement, the degree of compaction, curing and the age of the concrete. Strength increases, as the concrete becomes old. Generally, the strength of concrete is largely dependent of the type of aggregate used and the mix proportions or ratio. These factors influence the amount of water required to produce. The degree of flow of concrete affects its strength indirectly. Too much water will destroy your concrete (Akroyd, 1962).

2.5.11 Deterioration of Cement with Storage

Cement readily absorbs moisture from the atmosphere or from any damp material in contact with it. When cement is stored in sacks, absorption takes place from the air and strength of the cement is considerably reduced. The absorption by cement, of 1 or 2
percent of water, has no appreciable effect, but further amounts of absorption retard the hardening of cement and reduce its strength.

Cement stored in bulk or in air-tight containers does not deteriorate. It can be stored in covered barrels, bins and polythene bags. Cement thus stored up to about 2 meters or more in depth can lie for longer than a year with no more damage than the formation of a crust on the surface about 5 centimetres (cm) thick, which should be removed before it is taken for use. Portland cement may be stored indefinitely without loosening its quality, if it is protected from moisture. (Akroyd, 1962)

![Cement properly and improperly stored](image)

Figure 2.3: A. Cement Properly and B. Improperly Stored

2.5.12 The Importance of Curing

Usual concrete must be cured. No one has doubts about it. But this does not mean that concrete is always well cured, if it is cured at all. For usual concretes, water curing is necessary to ensure the highest degree of hydration possible in order to obtain the highest
strength and the lowest permeability (Neville, 1995). Uncured concrete dries more or less rapidly, depending on its water/binder ratio, since it never even approaches its full strength and durability potential. Early curing is always better than late curing, and in the case of usual concretes late curing is better than no curing at all.

There are different ways to water-cure concrete: fog misting, wet burlap, water ponding, water spraying and curing membranes. No one will argue that it is absolutely necessary to protect the top surface of the deck of a bridge built with concrete from drying shrinkage and early drying. Nobody will argue for long that it is very useful to wet-cure the top surface of column made of concrete that is exposed to drying for 7 days or more after being cast. It is absolutely necessary to understand what is happening when the hydration reaction develops in high concrete in order to take the appropriate measures to reduce as much as possible the shrinkages that will develop in any uncured concrete.

The concrete skin is always the first line of attack, if there is an attack, its weakening due to the lack of appropriate curing can be critical for the durability of the structure. So it is always important to evaluate the risk taken in weakening the concrete skin when there is improper curing. In any case, too much curing is always better than no curing at all, but it must also be recognized that proper curing is not always practical if it has not been well planned before the start of the construction. When it has been planned well ahead of schedule, there is never a problem in carrying out proper curing, because it only involves adding an almost free material to concrete: water.

Curing is carried out for two reasons: to hydrate as much as possible the cement present in the mix and, what is sometimes forgotten, to minimize shrinkage (Aitcin, 1998).
2.5.13 Quality Management System in the Testing Laboratory

A quality management system in a testing laboratory is a way of indicating that the quality of the final test results can be relied upon. The use of appropriate procedures and management methods, guarantees clients that errors in test results are minimal. The quality management system provides the laboratory with measurement traceability systems, and the use of preventive actions provides the possibility of initiating corrective action when errors are detected (F &Balgorbin, 2011).

2.5.14 Benefits of Standards

A standard is a document that provides requirements, specifications, guidelines or characteristics that can be used consistently to ensure that materials, products, processes and services are fit for their purpose (Anon, 2015). In essence, a standard is an agreed way of doing something and is designed for voluntary use so it’s up to you - you’re offered ways to do your work better.

Standards ensure the safety of products and services are of good quality and are reliable. For business, they are strategic tools that reduce costs by minimizing waste and errors, and increasing productivity. A standard helps companies to enter new markets and provides a level playing field for developing countries thus facilitating free and fair global trade (Anon, 2015). Standards cover a wide range of subjects, they can be very specific to a particular type of product, or general such as management practices.

The aim of a standard is to provide a very reliable basis for people to share the same expectations about a product or service from construction to nanotechnology, from
energy management to health and safety and from cricket balls to goalposts. This helps to facilitate trade, provide a framework for achieving economies, efficiencies and enhance consumer protection and confidence. (British Standards Institution, 2015)

2.5.15 Evolution of ISO 17025 and Its Importance

The first edition (1999) of this international Standard was produced as a result of extensive experience in implementation of ISO /IEC Guide 25 and EN 45001, both of which are replaced. It contained all of the requirements that testing and calibration laboratories have to meet if they wish to demonstrate that they operate a management system, are technically competent and are able to generate technically valid results. These standards have been superseded by ISO9001:2000, which made an alignment of ISO/IEC 17025 necessary. In the second edition, clauses have been amended or added only when considered necessary in the light of 9001:2000. Accreditation bodies that recognize the competence of testing and calibration laboratories use this standard as the biases for their accreditation. Clause four specifies the requirements for sound management and clause 5 specifies the requirements for technical competence for the type of tests and/or calibrations the laboratory undertakes.

Growth in the use of management systems generally has increased the need to ensure that laboratories which form part of larger organizations or offer other services can operate to a quality management system that is seen as compliant with ISO 9001 that are relevant to the scope of testing and calibration services that are covered by the laboratories management system. Testing and calibration laboratories that comply with this International Standard will therefore also operate in accordance with ISO 9001.
The acceptance of testing and calibration results between countries should be facilitated if laboratories comply with this international standard and if they obtain accreditation from bodies which have entered into mutual recognition agreements with equivalent bodies in other countries using these International Standards. The use of International Standard facilitates cooperation between laboratories and bodies that assist in the exchange of information and experience and in the harmonisation of Standards and Procedures.

2.5.16 Benefits of Accreditation and the Accreditation Process

Accreditation is the process by which the quality of a laboratory or an institution, public or private is formally recognized as having met certain pre-determined minimal criteria or standards. The result of this process is usually the awarding of a status of recognition with sometimes a license to operate within a period of validity (VLĂSCEANU, 2007). Accreditation gives confidence in results obtained. There is public and industry acceptance of results, there is also an increase in competitiveness and market share and there is also an Assurance to customers of good laboratory practices. Competence and experience of staff is assured, and there is an Integrity and traceability of equipment and materials.

After receiving the application for accreditation, a quotation is sent to the laboratory and arrangements made for a preliminary assessment. This assessment is then conducted by a team of technical assessors, who are drawn from the technical community, either within the country or outside. When the laboratory has demonstrated compliance with all necessary requirements, it is recommended for accreditation. Once approved, a certificate is given for accreditation (Anon, 2014).
2.5.17 Importance of Accurate and Reliable Data

Accurate, timely and reliable data is very important for effective decision making in virtually all aspects of human endeavour irrespective of who undertakes it, be it in an organisation, individual or government. According to Eslake, 2006, it is very important and an essential component in persuading businesses, individuals and governments to make decisions from the ones which they might make in the absence of a particular piece of information. It is an integral part of any attempt to hold those who make decisions, accountable for the consequences of the decisions they make. It is important to know how ISO 17025 2005 benefits the laboratory. It ensures we provide products and services that support safety world-wide, it identifies laboratories specific competencies, there is an assurance of acceptance of data, also, there is an assurance to meet the quality managements requirements of consumers, and lastly it establishes minimum competency.
2.5.18 Importance of Testing, Personnel and Equipment in Cement Analysis

Several tests are performed to determine the characteristics of cement and its compatibility with other materials in a concrete mix design. Testing of mortar cubes at 2-days, 7-days and 28-days is used to observe the development of strength gained over time. An analysis of the cement provides a reasonable estimate of the composition of the cement (Anon, 2008).

Portland cements are characterized by their physical properties for quality control purposes. Their physical properties can be used to classify and compare Portland cements. The challenge in physical property characterization is the development of physical tests methods that can satisfactorily characterize these properties. These are just a few of the tests that can be used to determine the quality of the cement.

These physical properties are the Setting Time which according to Anon, 2009 is determined using the Vicat apparatus with a plunger properly marked, calibrated and a Vicatmould. The cement is weighed and made into a paste and the plunger is dropped to determine the initial and final set times.

In checking the Soundness of cement, the Le Chatelier apparatus is used. The mould shall be of spring brass with indicator needles. A pair of plane glass base and cover plates shall be provided. There is the need for a water bath and a humidity chamber adequate enough to maintain a temperature of 20°C and not less than 98% relative humidity. The cement paste is placed in the slightly oiled mould without shaking the mould, level the top and cover the mould. After 24 hours, measure to find if there is any change in dimensions. Heat for 3 hours, allow cooling and measuring again. This assesses the possible risk of expansion due to hydration.
Fineness: the method of checking for the fineness of cement is by weighing an amount of cement to the nearest 0.01g (gram) and place it on the sieve. It is then agitated until no fines passes through it. The residue is then weighed and expressed as a percentage of the quantity placed on the sieve to the nearest 0.1 % (Anon, 2014).

The strength of the cement is tested by preparation of mortar cubes and curing them. After which they are crushed to determine their strength as declared.

In order to gain accreditation for laboratory, a person must be available who has relevant qualifications (e.g. a geologist) or has extensive experience in Construction Materials Testing identifying, quarry and 'source material'. Where the qualification requirements are not met, accreditation may be granted for a single source only. This will be included in the scope of accreditation, NATA 2013.
CHAPTER THREE

RESEARCH METHODOLOGY

3.0 Introduction

In this work, Regenerated Spent Catalyst (RSC) from the Residue Catalytic Cracking (RFCC) Unit of Tema Oil Refinery (TOR) Limited located in the city Temawas used as part replacement by 5 to 20% wt. of Portland cement from Ghacem. The desirable properties of Regenerated Spent Catalyst are: Average thermal stability, average activity, large pore sizes, average resistance to attrition and low coke formation.

Studies on the properties of replacement samples were carried out. The physical properties studied were Compressive Strength, Setting Times and Water Absorption. Chemical property studied was the chloride content. Comparison of data between the Control, that is 100% cement and with additive was made. The procedure adopted in achieving the goals and objectives of this research are listed in the Chapter One. This procedure includes the methodology adopted for the research, data collection and lastly the method of analysing the data.

3.2 The study Area

The Civil Engineering Laboratory under the testing division of the Engineering Department of Ghana Standards Authority (GSA) was chosen for this study. The Engineering Department is involved mainly in the testing of building materials for the Building and Construction Industry, Consultants in the industry, pre-mix concrete supply Companies and Research institutions in the industry.
3.3 The Research Approach and Strategy

This research is to study on the properties of sandcrete blocks produced with Regenerated Spent Catalyst. All Testing procedures relevant to the research were carried out in order to make a detailed analysis of the data obtained from the experiment. The Civil Engineering Laboratory provided the tests methods used to determine the properties of the prepared samples. This topic was chosen because of the importance of cement in the Ghanaian economy and also to add value to the Regenerated Spent Catalyst, generated as waste from Tema Oil Refinery (TOR) Ltd.

3.4 Research Procedure

The research was carried out by the collection of data relevant to the work, namely information from dissertations/ thesis, technical reports, articles, foreign and local standards, specification, the internet and some codes related to the field of study.

Standards, Codes or Best Practices used included the following:
- GS 766 2011 - Building and construction materials- Specification for Portland Limestone Cement CEM II
- BS EN197-1:2011 – Cement Composition, Specifications and Conformity Criteria for Common Cements
- DIN EN196-3 2009 – Determination of Setting Times and Soundness.
- ASTM C 778-06 – Standard Specification for Standard Sand
- DIN EN196-2 2013 – Methods of Testing Cement; Chemical Analysis of Cement
- DIN EN196-1 2005 – Methods of Testing Cement, Part I; Determination of Strength.

3.5 Data Collection and Experimentations

3.5.1 Primary Data Collection

Primary data involved laboratory specimen tests for the physical and chemical properties of sandcrete sampled. Sandcrete were prepared from Ghacem 32.5R cement (600 g), standard quartz-sand (1800 g) and tap water (300 mls). In case of sandcrete with addition, part of the cement was replaced by 5 to 20% mass of Regenerated Spent Catalyst. A total of 45 sandcrete cubes measuring 70mm x 70mm x 70mm sandcrete moulds were prepared, cast and cured at 2 days, 7 days and 28 days. The prepared samples were tested in the laboratory.

In the determination of the setting time 600 grams of cement and the Regenerated Spent Catalyst was sampled for use. Chemical analysis on the control, 5% RSC, 10% RSC, 15% RSC and 20% RSC samples at the Chemistry Laboratory to determine the chloride content.
Table 3.1

Proportion of Materials in Sandcrete Mixture

<table>
<thead>
<tr>
<th>Samples</th>
<th>Cement (g)</th>
<th>Regenerated Spent Catalyst (g)</th>
<th>Sand (g)</th>
<th>Water (mil)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control 100% cement</td>
<td>600</td>
<td>0</td>
<td>1800</td>
<td>300</td>
</tr>
<tr>
<td>5% RSC, 95% cement</td>
<td>570</td>
<td>30</td>
<td>1800</td>
<td>300</td>
</tr>
<tr>
<td>10% RSC, 90% cement</td>
<td>540</td>
<td>60</td>
<td>1800</td>
<td>300</td>
</tr>
<tr>
<td>15% RSC, 85% cement</td>
<td>510</td>
<td>90</td>
<td>1800</td>
<td>300</td>
</tr>
<tr>
<td>20% RSC, 80% cement</td>
<td>480</td>
<td>120</td>
<td>1800</td>
<td>300</td>
</tr>
</tbody>
</table>

3.5.2 Secondary Data

Secondary data refers to the statistical material which is not originated by the research person but obtained from someone else’s record. Secondary data helps to improve the understanding of the problem, prevent any repetition or duplication of studies carried out on the topic under consideration and provides basis for comparison for data that is collected by the researcher. According to M. M. Blair, “secondary data are those already in existence for some other purpose than answering of the question in hand”

Relevant data and information from the internet, dissertation/thesis, specification, and technical papers/reports, local and foreign documents on standards, text books, some selected codes of practise and local /foreign journals are extracted. Some of the documents used are namely
- GS 22 2011 building and construction materials-specification for Portland cement

CEM I
- DIN EN 196 1-6 Methods of testing cement

### Table 3.2

**Chemical Composition of Portland Ghacem Cement**

<table>
<thead>
<tr>
<th>Properties (wt. %)</th>
<th>SiO$_2$ Content</th>
<th>Al$_2$O$_3$ Content</th>
<th>Fe$_2$O$_3$ Content</th>
<th>CaO Content</th>
<th>MgO Content</th>
<th>SO$_3$ Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>21.9</td>
<td>6.9</td>
<td>3</td>
<td>63</td>
<td>2.5</td>
<td>1.7</td>
</tr>
</tbody>
</table>

### Table 3.3

**Physical Properties Portland Ghacem Cement**

<table>
<thead>
<tr>
<th>Property</th>
<th>Specific surface (m$^2$/kg)</th>
<th>Specific gravity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity</td>
<td>370</td>
<td>3.15</td>
</tr>
</tbody>
</table>

### Table 3.4

**Chemical Composition of Regenerated Spent Catalyst (RSC)**

<table>
<thead>
<tr>
<th>Properties (wt. %)</th>
<th>Al$_2$O$_3$ Content (wt. %)</th>
<th>Fe$_2$O$_3$ Content (wt. %)</th>
<th>CaO Content (wt. %)</th>
<th>Hydrogen Content (wt. %)</th>
<th>Carbon Content (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>48.5</td>
<td>0.54</td>
<td>1.01</td>
<td>0.242</td>
<td>0.04</td>
</tr>
</tbody>
</table>
Table 3.5

Physical Properties of Regenerated Spent Catalyst (RSC)

<table>
<thead>
<tr>
<th>Property</th>
<th>Pore Volume (cm$^3$/g)</th>
<th>Specific Surface (m$^2$/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity</td>
<td>0.33</td>
<td>90</td>
</tr>
</tbody>
</table>

3.6 Data Collection Techniques

The technique adopted to collect data was by direct observation of the processes involved in the preparation of sandcrete cubes from the selected samples. Some parameters or conditions observed at the cube preparation stages were the general temperature conditions under which the cubes were being prepared, the measuring of the cement and Regenerated Spent Catalyst samples, storage temperature, that is ambient temperature (298.15K), pressure (100kP), mixing procedures, sandcrete preparation for vibration, curing procedures and lastly the crushing process. Other investigations conducted on sample included the Setting Time Test, Chemical Test and the Test for distilled water. Cement test as already described in the standard has to follow particular procedures. These procedures which are guided by ISO 17025 and the various standards are listed below:

- Check the temperature of the room and record
- Condition the cement sample
- Weigh the cement, sand and water
- Replaced part of the cement was replaced by 5 to 20% mass of Regenerated Spent Catalyst
- Oil the moulds to be used
- Transfer the samples and water into the mixing bowl and mix for 90s
- Transfer the mortar into the mould, vibrate and cover the sandcrete for 24 hours
- Demould the sandcrete cubes, weigh and transfer them into the Curing Tank for specific period
- On the due date 2, 7 and 28 days, remove the cube from the Curing Tank, weigh and crush to determine Compressive Strength.
- Record the crushing forces and calculate the Compressive Strength by dividing force by area.
- Check for markings
- Calculate results

The flow chart showing the stages of preparing sandcrete cubes, crushing by exerting a load on it and calculating the Compressive Strength of samples is described below.
3.7 Methods for Testing and Determining of Strength of Samples

The samples were cast from a batch of sandcrete containing one(1) part by mass of cement or the Regenerated Spent Catalyst, three(3) parts of standard sand and one half(1/2) part of water. The sandcrete was prepared by mechanical mixing and compacted in a mould using a Jolting Apparatus. These samples were kept in the mould in a moist atmosphere for 24 hours, then and demoulded and stored under water until they were due for testing.

3.8 Laboratory and Equipment

The laboratory test conditions were maintained at a temperature of (20±2) °C and a relative humidity not less than 50%. The temperature of the room was recorded at least once a day during working hour’s whiles the humidity of air was recorded every 4 hours.
The tolerance of the equipment used was considered as it was important for the correct operation of the equipment. The control measures were carried out regularly.

### 3.8.1 Mixer

The mixer consists essentially of a stainless steel bowl with a blade revolving about its own axis at a controlled speed and scarpers.

![Mortar Mixer](image)

**Figure 3.2: Mortar Mixer**

### 3.8.2 Preparation of Sandcrete, Placing, Conditioning and Testing

By means of a balance weigh the water and sample, place both in the mixing bowl, taking care to avoid loss of water or sample. Immediately start the mixer at low speed. After 30 seconds of mixing, add the sand steadily during the next 30 seconds. Switch the mixer to high speed and continue for another 30 seconds. Mould the sample immediately after the preparation of the sandcrete paste and compact using the Jolting Apparatus.
Lift the moulds gently after wiping off excess sandcrete and mark or label the moulds respectively for identification purposes. Cover each mould with a woollen material without delay and wait for 24 hours.

Submerge the marked samples in tap water for curing at (20±1.0) ºC. Remove the samples from water 15 minutes before each test is due. Subject the samples to stress using the Compressive Test Machine increasing the load progressively. Where the load increase is regulated by hand, care must be taken when making adjustments for the decrease of the loading rate near fracture load as this could affect the results. (DIN EN 196-1,2005)

![Compression Testing Machine](http://ugspace.ug.edu.gh)

Figure 3.3: Compression Test Machine
Figure 3.4: Set of sandcrete cubes ready for crushing

Figure 3.5: Curing Tank for sandcrete cubes storage
3.8.3 Determination of Setting Times and Standard Consistence Test

3.8.3.1 Principles

Cement paste or the Regenerated Spent Catalyst paste of Standard Consistence has a specified Resistance to penetration by a Standard Plunger.

The water required for such a paste was determined by trial penetrations of paste with different contents. The Setting Times were thus determined by observing the penetration of a needle into the cement paste of Standard Consistence until it reached a specific value.

3.8.3.2 Standard Consistence Test

The manual Vicat Apparatus with a plunger was used to weigh a total of 300±1 g. The movement of the Vicat Apparatus with plunger was kept truly vertical. The mould to contain the paste is made of rubber or brass placed on glass flat base. In mixing the cement paste, weigh to an accuracy of ±1g by means of the balance, 500g of cement or Regenerated Spent Catalyst and a quantity of water e.g. 125g. The weighed items were mixed using the mixer at low speed. The paste produced was immediately transferred into a lightly oiled mould; any voids were removed in the paste by gently tapping the sides of the mould and levelling to the top surface.

Immediately after levelling the paste, it was transferred onto the Vicat Apparatus and the plunger dropped intermittently. This was repeated until the distance between the plunger and paste was (6±2) mm.
3.8.3.3 Setting Times Test; Initial and Final

The filed mould and base plate were placed in a container filled with water at a controlled temperature of 20±1 °C. After a period of time the sample was removed and placed under the Vicat and the needle dropped gently onto the paste. The scale was read when penetration had ceased or 30 seconds after the release of the needle. The scale readings, which indicated the distance between the needle and the base plate, were recorded. The penetration was repeated on the same sample until the time where the distance between the needle and the base plate was 6 ± 3 mm measured to the nearest minute. This is the Initial Setting Time of the cement or Regenerated Spent Catalyst and is reported to the nearest 5 minutes.

In the determination of the Final Setting Time, the paste in mould used for the Initial Setting Time was inverted and again immersed under water. This time the needle was dropped until a reading of 0.5 mm was recorded. This indicates the Final Setting Time has been achieved. (DIN EN 196-9, 2009)

![Figure 3.6: A Vicat Apparatus Accessories](image-url)
3.8.4 Chemical Analysis of Cement

3.8.4.1 Blank Determinations

Blank determinations were carried out without a sample, following the same procedure and using the same amounts of regents and results obtained were corrected to the analytical determinations accordingly.

3.8.4.2 Preparation of Test Samples

The samples were treated to a homogenous test sample by taking approximately 100g by means of a sample divider or by quartering. This portion was sieved on a 150µm and 125µm standard sieve until residue remained constant. Metallic iron particles were removed from the metal retained in the sieve by means of a magnet. The iron free fraction of the retained material was ground so that it passed the 150µm and 125µm Standard Sieve. The sample was then transferred to a clean dry container with an air tight closure and vigorously mixed thoroughly. This was now sent to the X-ray Florescence for analysis. (DIN EN 196-2, 2013)

Figure 3.7: Sample being tested for its chemical composition
3.8.5 Specification of Standard Sand

Standard sand shall meet the requirements in the table below with respect to grading, source of sand and absence of air entraining characteristics (ASTM C778-06, 2006).

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>20-0 sand</th>
<th>Graded Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grading sieve sizes</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.18mm (No. 16)</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>850µm (No.20)</td>
<td>85 to 100</td>
<td></td>
</tr>
<tr>
<td>600µm (No.30)</td>
<td>0 to 5</td>
<td>96 to 100</td>
</tr>
<tr>
<td>425µm (No.40)</td>
<td></td>
<td>65 to 75</td>
</tr>
<tr>
<td>300µm (No.50)</td>
<td></td>
<td>20 to 30</td>
</tr>
<tr>
<td>150µm um (No.100)</td>
<td></td>
<td>0 to 4</td>
</tr>
<tr>
<td>Source of Sand</td>
<td>Ottawa, IL OR Le Suer, MN</td>
<td>Ottawa, IL</td>
</tr>
</tbody>
</table>

Source: ASTM C778-06

3.8.6 Analysis of Data

The data was analysed using simple percentage statistical method of data analysis to indicate the strength and significance of each set of mortar cube crushed.
Data was analysed through the whole case study and was not limited to any one chapter. In the analysis made, discussions of data obtained from the experiments conducted was analysed. Additional use of tables and graphs were made.
CHAPTER FOUR

RESULTS AND DISCUSSIONS

4.1 Compressive Strength

In order to check if the strength of the sandcrete cube achieved is in conformity with the standard, the crushing strength value recorded in kN is divided by the area (mm²) of the sandcrete cube.

In all, forty five (45) cubes were prepared, cast, cured and crushed according to the BS 1881 Part 116. (BS,1983). At two, seven and twenty-eight days, due samples were removed dried and crushed to the point of failure by a load and the value at failure recorded. This is used in the calculation of the Compressive Strength using the formula

\[ f_c = \frac{P}{A} \]

Where,

\( f_c \) Compressive Strength (N/mm²)

P load at failure (N)

A surface area of the sandcrete cube (mm²)

The results obtained for the various samples are presented below:
Figure 4.1: Compressive strength curve of various samples for 2 days Curing Period

Figure 4.2: Compressive strength curve of various samples for 7 days Curing Period
4.1.1 Effect of Regenerated Spent Catalyst on Compressive Strength

Figure 4.1, 4.2 and 4.3 showed the Compressive Strength curves of various samples for the two (2) days, seven (7) days and twenty eight (28) days curing periods. Comparison of the data for 2,7 and 28 days of curing period showed that the Compressive Strength increased when the Regenerated Spent Catalyst was added to sandcrete, especially as a 10% replacement for cement. The Regenerated Spent Catalyst (RSC) activity is due to its ability to react with the calcium hydroxide (portlandite) released in the hydration of Portland cement and form compounds with hydraulic properties. This behavior causes additional increase in the mechanical strength of sandcrete (Borrachero et al, 2002).

Also the high specific area of Regenerated Spent Catalyst (RSC) led to a number of nucleation sites for additional hydration products. This increased the hydration heat all
over the period of testing. This hydration heat increase was due to the pozzolanic effect, pozzolanic material (RSC) react with calcium hydroxide released in the hydration of Portland sandcrete, increasing the hydration heat due to the exothermic effect of the pozzolanic reaction, thereby the resulting in relatively higher Compressive Strength is higher than the reference sandcrete.

4.2 Setting Time

4.2.1 Effect of Regenerated Spent Catalyst on Setting Time

From the figure 4.4, it can be seen that the Initial Setting Time measured for sandcretes with Regenerated Spent Catalyst (RSC) was found to be lower than that of the control; also the Final Setting Time measured for control was higher than the sandcretes with RSC. This phenomenon was as a result of the increased in the hydration heat resulting from the replacement of the cement with RSC. The sandcretes with RSC solidifies and
hardens faster through this chemical process called hydration thereby reducing the Setting time.

4.3 Water Absorption

![Water Absorption vs RSC Replacement Content in Cement](image)

Figure 4.5: Water Absorption curve for various samples

4.3.1 Effect of Regenerated Spent Catalyst on Water Absorption

From the figure 4.5, it could be observed that the Water Absorption for the sandcretes with RSC was found to be lower compared to control sandcrete. This phenomenon is as a result of the molecules of water in the hydrated form in zeolites of the RSC, when the RSC addition was incorporated in the sandcrete and also due to increased hydration heat resulting in reduced amount of water retained.
4.4 Chemical Analysis

![Chloride Content versus RSC Replacement Content in Cement](image)

Fig 4.6: Chloride Content curve for various samples

From the figure 4.6, it could be observed that the samples with RSC showed lower Chloride Content values when compared with their equivalent mixture without addition. The chloride content for the control sandcrete and sandcretes with the RSC were all within the acceptable levels. (EN 196-21).
4.5.1 Effect of Regenerated Spent Catalyst on Compressive Strength versus Setting Time

From figure 4.7 above, it could be observed that as the Compressive Strength increased to maximum strength at 10% RSC replacement content in cement and begun to decrease whilst the Setting Time decrease throughout the specified period. This was due to the fact that increases in the RSC replacement content in the cement increased the hydration heat resulting in increased compressive strength thereby reducing the Setting Time. The decreased in the compressive strength beyond the 10% RSC replacement content in cement was due to the fact that the optimum for replacing the cement with the RSC had been attained and beyond this lead to a reduction in the better binder which is the cement than the RSC.
4.5.2 Effect of Regenerated Spent Catalyst on Compressive Strength versus Water Absorption

From figure 4.8 above, it could be observed that as the Compressive Strength increased to maximum strength at 10% RSC replacement content in cement and begun to decrease whilst the Water Absorption decrease throughout the specified period. This was due to the fact that increases in the RSC replacement content in the cement increased the hydration heat resulting in increased compressive strength thereby reducing the Amount of water retained. The decreased in the compressive strength beyond the 10% RSC replacement content in cement was due to the fact that the optimum for replacing the cement with the RSC has reached and beyond this lead to a reduction in the cement which is a better binder than the RSC.
Fig. 4.9. A graph of Water Absorption and Setting Time versus RSC Replacement Content in Cement

4.5.3 Effect of Regenerated Spent Catalyst on Water Absorption versus Setting Time

From figure 4.9 above, it could be observed that as the amount of water retained in the sandcrete decreases, the setting time also decreases as the RSC replacement content in cement increases. This is due to the fact that an increase in the RSC content in the sandcrete increased the hydration heat resulting in less amount of water and the sandcretes with RSC solidifies and hardens faster through this chemical process called hydration as such reducing the setting time.
CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

- Comprehensive experimental/ laboratory and field analysis indicate RSC, also referred to as ECAT consist mainly of Silica (49 wt. %), alumina (45wt. %) and minerological constituents of zeolites, kaolinites and quartz. RSC has some prozzolanic properties and can be used for part cement substitution.

- Substituting Portland cement up to 20wt. % RSC gave sandcrete strengths higher than the 32.5 N/mm$^2$, which corresponds to that of Portland cement. This phenomenon is largely attributed to the structure and texture of the RSC due to bonding between the zeolites and the alumina – silica matrix during manufacture. The replacement of Portland cement with 10 wt. % of RSC gave the highest strength of 34.01 N/mm$^2$.

- The setting times of RSC-Portland mixture (sandcrete) decreased with increased substitution of Portland cement with RSC. This was as a result of the increased in the hydration heat in the sandcrete with RSC which solidifies and hardens faster through a chemical process called hydration. However, they were within limit specified by (GS 22 and EN 197-1).

- Water absorption of RSC-Portland cement mixtures (sandcrete) decreased with increased substitution of Portland cement with RSC. This phenomenon is as a result of the molecules of water in the hydrated form in zeolites of the RSC, which is at 235 for faujasite[(Na$_2$K$_2$Ca)$_{29.5}$ (AlO$_2$)$_{59}$ (SiO$_2$)$_{133}$ ] and 264 each for zeolite X [( Na$_{86}$ (ALO$_2$)$_{86}$ (SiO$_2$)$_{106}$ ]and zeolite Y[ ( Na$_{56}$ (ALO$_2$)$_{56}$]
(SiO$_2$)$_{136}$](A.G Oblad, Oil and Gas Journal 1972, and C. Marcilly, Les tamimoleculaires, IFP Report 18859, 1971 and Les zeolithes, description et principles applications en sorption et catalyse, IFP Report 28913, 1981). In addition the structural patterns of the zeolites produce more complex structures, resulting in the formation of crystalline solids with high surface area and high standard-size porosity.

- The chloride content of RSC-Portland cement mixtures (sandcrete) decreased as the quantity of RSC replacing Portland cement increased within the limits studied. However, the chloride content were within the limits specified by (EN 196-21).

Regenerated Spent Catalyst can be used to replace up to 20 wt. % of Portland cement without compromising the properties and consequently the quality of sandcrete used in manufacturing blocks for construction.

Consequently, this is of immersed economic benefit to the construction industry and reduction in the use of clinker in cement production as well disposal quantities of RSC

### 5.2 Recommendations

It is recommended that further studies be carried out on the:

- Durability of Regenerated Spent Catalyst as a cementitious material.
- Aggregate substitution of RSC in sandcrete masonry systems.
- Use of RSC as a glazing material in ceramic wares.
- Health hazards associated with the use of RSC.
REFERENCES


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28. DIN EN196-2 2013 – Methods of Testing Cement; Chemical Analysis of Cement


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64. SPIN, 2012. Cement Proficiency Testing Scheme, Dares Salaam: PTB.


## APPENDIX

### Appendix A

**Chemical Requirements of Cement**

<table>
<thead>
<tr>
<th>Property</th>
<th>Test reference</th>
<th>Strength class</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Loss in ignition</td>
<td>ISO 680</td>
<td>All</td>
<td>&lt; 5.0 % wt.</td>
</tr>
<tr>
<td>Insoluble residue</td>
<td>ISO 680</td>
<td>All</td>
<td>&lt; 5.0 % wt.</td>
</tr>
<tr>
<td>Sulphate content</td>
<td>ISO 680</td>
<td>32.5N, 32.5R, 42.5N, 42.5R, 52.5R, 52.5N</td>
<td>&lt; 3.5 % wt.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>&lt; 4.0 % wt.</td>
</tr>
<tr>
<td>Chloride content</td>
<td>EN 196-21</td>
<td>All</td>
<td>&lt; 0.10 % wt.</td>
</tr>
</tbody>
</table>

Source: GS-22, 2011

### Appendix B

**Proportion of Materials in Sandcrete Mixture**

<table>
<thead>
<tr>
<th>Samples</th>
<th>Cement (g)</th>
<th>Regenerated Spent Catalyst (g)</th>
<th>Sand (g)</th>
<th>Water (mil)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control 100% cement</td>
<td>600</td>
<td>0</td>
<td>1800</td>
<td>300</td>
</tr>
<tr>
<td>5% RSC, 95% cement</td>
<td>570</td>
<td>30</td>
<td>1800</td>
<td>300</td>
</tr>
</tbody>
</table>
### Chemical Composition of Portland Ghacem Cement

<table>
<thead>
<tr>
<th>Properties</th>
<th>SiO₂ Content (wt. %)</th>
<th>Al₂O₃ Content (wt. %)</th>
<th>Fe₂O₃ Content (wt. %)</th>
<th>CaO Content (wt. %)</th>
<th>MgO Content (wt. %)</th>
<th>SO₃ Content (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition (wt. %)</td>
<td>21.9</td>
<td>6.9</td>
<td>3</td>
<td>63</td>
<td>2.5</td>
<td>1.7</td>
</tr>
</tbody>
</table>

### Physical Properties of Portland Ghacem Cement

<table>
<thead>
<tr>
<th>Property</th>
<th>Specific surface (m²/kg)</th>
<th>Specific gravity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity</td>
<td>370</td>
<td>3.15</td>
</tr>
</tbody>
</table>

### Chemical Composition of Regenerated Spent Catalyst (RSC)

<table>
<thead>
<tr>
<th>Properties (wt. %)</th>
<th>Al₂O₃ Content (wt. %)</th>
<th>Fe₂O₃ Content (wt. %)</th>
<th>CaO Content (wt. %)</th>
<th>Hydrogen Content (wt. %)</th>
<th>Carbon Content (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>48.5</td>
<td>0.54</td>
<td>1.01</td>
<td>0.242</td>
<td>0.04</td>
</tr>
</tbody>
</table>
### Appendix F

**Physical Properties of Regenerated Spent Catalyst (RSC)**

<table>
<thead>
<tr>
<th>Property</th>
<th>Pore Volume (cm$^3$/g)</th>
<th>Specific Surface (m$^2$/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quantity</td>
<td>0.33</td>
<td>90</td>
</tr>
</tbody>
</table>

### Appendix G

**Standard Sand Requirement**

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>20-0 sand</th>
<th>Graded Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grading sieve sizes</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.18mm (No. 16)</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>850µm (No. 20)</td>
<td>85 to 100</td>
<td></td>
</tr>
<tr>
<td>600µm (No. 30)</td>
<td>0 to 5</td>
<td>96 to 100</td>
</tr>
<tr>
<td>425µm (No. 40)</td>
<td></td>
<td>65 to 75</td>
</tr>
<tr>
<td>300µm (No. 50)</td>
<td></td>
<td>20 to 30</td>
</tr>
<tr>
<td>150µm um (No. 100)</td>
<td></td>
<td>0 to 4</td>
</tr>
</tbody>
</table>

Source of Sand
- Ottawa, IL OR Le Suer, MN
- Ottawa, IL

Source ASTM C778-06
### Appendix H

**Compressive Strength Test Results Obtained for two (2) days Curing Period**

<table>
<thead>
<tr>
<th>Samples</th>
<th>Crushing value (kN)</th>
<th>Crushing value (kN)</th>
<th>Crushing value(kN) (Average)</th>
<th>Area of sample (mm²)</th>
<th>Compressive strength (N/mm²)</th>
<th>Standard specification (N/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control 100% cement</td>
<td>95</td>
<td>90</td>
<td>91.7</td>
<td>4900</td>
<td>18.7</td>
<td>≥10</td>
</tr>
<tr>
<td>5% RSC, 95% cement</td>
<td>100</td>
<td>95</td>
<td>95.0</td>
<td>4900</td>
<td>19.4</td>
<td>≥10</td>
</tr>
<tr>
<td>10% RSC, 90% cement</td>
<td>95</td>
<td>100</td>
<td>98.3</td>
<td>4900</td>
<td>20.1</td>
<td>≥10</td>
</tr>
<tr>
<td>15% RSC, 85% cement</td>
<td>95</td>
<td>95</td>
<td>100</td>
<td>4900</td>
<td>19.7</td>
<td>≥10</td>
</tr>
<tr>
<td>20% RSC, 80% cement</td>
<td>95</td>
<td>90</td>
<td>93.3</td>
<td>4900</td>
<td>19.0</td>
<td>≥10</td>
</tr>
</tbody>
</table>

### Appendix I

**Compressive Strength Test Results Obtained for seven (7) days Curing Period**

<table>
<thead>
<tr>
<th>Samples</th>
<th>Crushing value(N)</th>
<th>Crushing value(kN)</th>
<th>Crushing value(kN) (Average)</th>
<th>Area of sample (mm²)</th>
<th>Compressive strength (N/mm²)</th>
<th>Standard Specification (N/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control 100% cement</td>
<td>110</td>
<td>105</td>
<td>110</td>
<td>4900</td>
<td>22.1</td>
<td>-</td>
</tr>
<tr>
<td>5% RSC, 95% cement</td>
<td>110</td>
<td>110</td>
<td>115</td>
<td>4900</td>
<td>22.8</td>
<td>-</td>
</tr>
<tr>
<td>10% RSC, 90% cement</td>
<td>115</td>
<td>115</td>
<td>115</td>
<td>4900</td>
<td>23.5</td>
<td>-</td>
</tr>
<tr>
<td>15% RSC, 85% cement</td>
<td>110</td>
<td>115</td>
<td>115</td>
<td>4900</td>
<td>23.1</td>
<td>-</td>
</tr>
</tbody>
</table>
## Appendix J

### Compressive Strength Test Results Obtained for twenty-eight (28) Curing Period

<table>
<thead>
<tr>
<th>samples</th>
<th>Crushing value(kN)</th>
<th>Crushing value(kN)</th>
<th>Crushing value(kN)</th>
<th>Area of sample (mm²)</th>
<th>Compressive strength (N/mm²)</th>
<th>Standard specification (N/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Test 1</td>
<td>Test 2</td>
<td>Test 3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Control 100%cement</td>
<td>160</td>
<td>160</td>
<td>160</td>
<td>160.0</td>
<td>4900</td>
<td>32.7</td>
</tr>
<tr>
<td>5% RSC, 95%cement</td>
<td>165</td>
<td>160</td>
<td>165</td>
<td>163.3</td>
<td>4900</td>
<td>33.3</td>
</tr>
<tr>
<td>10% RSC, 90%cement</td>
<td>170</td>
<td>165</td>
<td>165</td>
<td>166.7</td>
<td>4900</td>
<td>34.0</td>
</tr>
<tr>
<td>15% RSC, 85%cement</td>
<td>165</td>
<td>165</td>
<td>165</td>
<td>165.0</td>
<td>4900</td>
<td>33.7</td>
</tr>
<tr>
<td>20% RSC, 80%cement</td>
<td>160</td>
<td>165</td>
<td>160</td>
<td>161.7</td>
<td>4900</td>
<td>33.0</td>
</tr>
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</table>

## Appendix K

### Setting Time Test Results of Samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Initial Setting Time (min)</th>
<th>Final Setting Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0% RSC, 100%cement</td>
<td>135</td>
<td>300</td>
</tr>
<tr>
<td>5% RSC, 95%cement</td>
<td>131</td>
<td>297</td>
</tr>
<tr>
<td>10% RSC, 90%cement</td>
<td>129</td>
<td>295</td>
</tr>
<tr>
<td>15% RSC, 85%cement</td>
<td>126</td>
<td>293</td>
</tr>
</tbody>
</table>
Appendix L

Result of Amount of water contained for 28 days Curing Period

<table>
<thead>
<tr>
<th>Samples</th>
<th>Amount of water absorbed after 28 days (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0% RSC, 100% cement</td>
<td>0.0043</td>
</tr>
<tr>
<td>5% RSC, 95% cement</td>
<td>0.0038</td>
</tr>
<tr>
<td>10% RSC, 90% cement</td>
<td>0.0035</td>
</tr>
<tr>
<td>15% RSC, 85% cement</td>
<td>0.0031</td>
</tr>
<tr>
<td>20% RSC, 80% cement</td>
<td>0.0028</td>
</tr>
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Appendix M

Chemical Analysis on the Control

<table>
<thead>
<tr>
<th>Tests Conducted</th>
<th>Chloride Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control (100%cement)</td>
<td>0.07</td>
</tr>
<tr>
<td>5% RSC,95% cement</td>
<td>0.06</td>
</tr>
<tr>
<td>10% RSC,90% cement</td>
<td>0.05</td>
</tr>
<tr>
<td>15% RSC,85% cement</td>
<td>0.03</td>
</tr>
<tr>
<td>20% RSC,80% cement</td>
<td>0.02</td>
</tr>
</tbody>
</table>