# **OPTIMISATION OF CARBON DIOXIDE (CO<sub>2</sub>) SEQUESTRATION USING SANDCRETE-TALC COMPOSITE**

BY

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# A THESIS SUBMITTED TO THE POSTGRADUATE SCHOOL FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA, NIGERIA IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF THE DEGREE OF MASTER OF ENGINEERING IN THE DEPARTMENT OF CHEMICAL ENGINEERING

NOVEMBER, 2019

#### ABSTRACT

Global interest on CO<sub>2</sub> emission reduction has triggered alternative cement formulation using non-classical raw materials. This work presents a study on optimisation of CO<sub>2</sub> uptake by sandcrete-talc composite material. The morphology, mineralogical and oxide composition of the materials were characterized using Scanning Electron Microscopy (SEM), X-ray Diffractormeter (XRD) and X-ray fluorescence (XRF). The result obtained from XRF indicate that sand consist 50.213% SiO2.and 16.001% Si with other diminish amount of oxide and metals, while mineral talc consist of 48.932% SiO<sub>2</sub> and 24.86% MgO and cement contain 52.911% CaO, 15.122% Ca and 11.55%  $SiO_2$  with minute quantities of Alkalis and other metallic oxides. The mathematical model for the determination of CO<sub>2</sub> concentration and compressive strength of sandcrete-talc composite was developed and optimized by the application of two-level four factorial design. The result showed that as curring age increases with increase in talc and sand components at constant cement component, the CO<sub>2</sub> concentration and compressive strength also increases. The developed predicted model showed that the optimal concentration of CO<sub>2</sub> and compressive strength obtained were 0.289mol/dm<sup>3</sup> and 3.506N/mm<sup>2</sup> respectively after 28 day curing at an ambient condition.

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#### **CHAPTER ONE**

#### INTRODUCTION

#### **1.1 Background of the Study**

1.0

It is a well-known fact that carbon dioxide plays an important role in the natural greenhouse warming of the Earth's atmosphere. The idea of climate warming related to  $CO_2$  increases, as propounded by Arrhenius among others in the late nineteenth century. In the 1930s and 1940s Guy Stewart Calendar at Imperial College (London) revived the warming theory and by the 1970s it was generally accepted that global surface temperatures would increase as  $CO_2$  concentrations increased(Zhong and Haigh, 2013). It was on this note that World leaders gathered in Kyoto, Japan, in December 1997 to consider a world treaty restricting emissions of "greenhouse gases," chiefly among them is carbon dioxide ( $CO_2$ ), that are thought to cause "global warming", severe increases in Earth's atmospheric and surface temperatures, with disastrous environmental consequences; this is however evident in the melting of ice caps, increased sea level rise and extreme weather conditions (Robinson *et al.*, 1998). To be sure,  $CO_2$  levels have increased substantially since the Industrial Revolution, and are expected to continue doing so. Greenhouse gases cause plant life, and the animal life that depends upon it, to survive (Zhong and Haigh, 2013).

Currently one of the most pressing and globally recognised challenges is how to mitigate the effects of global environmental change brought about by increasing emissions of greenhouse gases, especially  $CO_2$  (Woodward *et al.*, 2009). A number of strategies have been proposed to deal with this problem. The most obvious way in which  $CO_2$  emissions can be reduced is by switching from burning fossil fuels to using non-fossil-fuel sources of energy such as nuclear energy, wave and wind power, and geothermal sources. In response, a variety of schemes have been proposed to either draw-down the amount of  $CO_2$  in the atmosphere or mitigate the effects of global warming through energy efficiency, alternative energy sources, energy conservation as well as carbon capture and sequestration. Hyseni (2017), stated that carbon sequestration strategies can be a clear path in mitigating climate change as well as neutralizing the excess  $CO_2$  in the atmosphere emitted through anthropogenic sources. There are several other mitigation strategies for  $CO_2$  that have been studied and are currently being used in various pilot projects to further increase the incentive to study this field.

Different methodologies and technologies have been developed for the sequestration of atmospheric carbon dioxide (Sunho *et al.*, 2009). However  $CO_2$  capture and sequestration using sandcrete blend is probably the best short to long term solution for the reduction of atmospheric acidic emission in developing nations. The effects of admixture on sandcrete carbonation have not been highlighted by most researchers. Significant knowledge gap exits. This work presents a study on the optimization and analysis of  $CO_2$  sequestration using 2-factorial design.

The rapid growth in any country's economy and population requires additional physical infrastructures to accommodate additional various component of the Gross National Product. These physical infrastructures include residential and commercial buildings, agricultural and health facilities, on the other hand it requires the integration of engineering, project, and production management techniques. A Sandcrete block is an important material in building construction. It is widely used in Nigeria, Ghana, and other African countries as load bearing and non-load bearing walling units. British Standard 6073: 1981 Part 1 defines a block as a masonry unit of larger size in all dimensions than specified for bricks but no dimension should exceed 650mm nor should the height exceed either its length or six times its thickness. The quality of sandcrete blocks is influenced by so many factors such as the

quality of constituent materials, the process adopted in manufacture, duration of curing, forms and sizes of blocks.

Ngally *et al.* (2014) reported that talc is an hydrated magnesium silicate with chemical formula  $Mg_3Si_4O_{10}(OH)_2$ . Talc is hydrophobic material that easily blends and disappears within organic media including polymer. It is widely used in reinforcing filler in several industrial products such as, papers, paints, rubbers, polymers, ceramics and refractory materials. The composition of talc make it potential raw material in cement formulation, since magnesium can interact with phosphate to yield cementitious materials as in phosphate magnesia cement. They can be roughly divided into two deposits types, Talc-carbonate (dominantly talc with variable amount of chlorite, dolomite and or magnesite), and Talc-chlorite (dominantly talc with chlorite).

One of the key durability indicators of sandcrete is the degree of carbonation. Understanding sandcrete micro-structure as it affects durability is the hallmark for most sandcrete researchers (Odigure, 2002). The degree of porosity, pore type and size, pore connectivity and their distributions coupled with mineral crystal sizes and arrangement in the solid matrix significantly affect physicochemical properties of cement-based materials (Naik and Kumar, 2010).

Rostami *et al.* (2012) studied micro- structure of cement paste subject to early age carbonation curing with aim of understanding the mechanism of concrete carbonation through micro-structure development. They found that early carbonation curing could accelerate the strength gain and increases durability of concrete. Research has shown that the three polymorphs (Calcite, Aragonite or Vaterite) of calcium carbonate could be formed during carbonation as  $CO_2$  ingress into the concrete. The micro-structure of

calcite formed during carbonation is characterized by small closely crammed crystals of a circular shape with particle size of less than 3µm (Fermandez *et al.*, 2004).

In view of the above background, this research studies the uptake of  $CO_2$  and performance characterization of sandcrete-talc composite at early age of carbonation curing, as it is evidence that talc is a raw material for the formation of the cementitious material by increasing binder phase formation held in the matrix and reduction of pores will be probably observed. However, this study demonstrated how  $CO_2$  capture and sequestration using sandcrete-talc composite can be employed as a short to long term solution for the reduction of atmospheric emissions.

#### **1.2** Statement of the Research Problem

The environment is under increasing threat from global carbon dioxide ( $CO_2$ ) emissions deriving from cement industry and human sources. Statistics have shown that Cement manufacturing industries are among the largest industries that emit  $CO_2$  to atmosphere, for every 1 ton of cement produced, 1.25 tons of  $CO_2$  is released to the atmosphere (IPCC, 2015). Rising atmospheric  $CO_2$  is also increasing the absorption of  $CO_2$  by seawater, causing the ocean to become more acidic, with potentially disruptive effects on marine plankton and coral reefs. Technically and economically feasible strategies are needed to mitigate the consequences of increased atmospheric  $CO_2$ . This project seeks to find way of mitigating global warming using talc-cement composite as  $CO_2$  sequestration materials, thereby saving energy and creating an environmentally friendly condition.

## 1.3 Aim and Objectives of the study

This research work optimised carbon dioxide (CO<sub>2</sub>) uptake by Sandcrete-Talc composite at ambient conditions.

This aim was achieved through the following objectives:

- i. Characterization of the Sandcrete-Talc composite produced at ambient conditions using XRD, XRF and SEM.
- ii. Evaluation of the effect of carbonation on the compressive strength after 28 days.
- iii. Evaluating the optimal process condition that maximizes  $CO_2$  uptake and compressive strength.

# **1.4** Justification of the Study

Increased burning of fossil fuels, deforestation, land degradation and various industry practices have resulted in high atmospheric CO<sub>2</sub> levels creating global concern because of its implications towards climate change. These sources have increased global emissions of CO<sub>2</sub> by 48% more than two decades ago thus accelerating the greenhouse gas effect and increasing the overall temperature of the planet by 0.8°C in 2013. Recently, global leaders committed to keep the overall temperature rise of the planet to under 2°C at the 21<sup>st</sup> Conference of Paris (COP21) in Paris promising to mitigate climate change by moving toward a carbon free future (Hyseni, 2017).

Researchers in building sector have indicated that between 50 to 60% of the total construction input goes into building materials especially cement. Also, the construction industry has witnessed a higher level of criticism and controversies over the issues of building, the main problem being the issue of using substandard building materials and construction process or methods. It is necessary to enhance the cement quality and to find new cementitious raw material to overcome the global warming challenge due to the

production of the largely used Portland cement. This research area arouses interests, which focuses on the reduction of the  $CO_2$  emission and for alternative cement formulation using non-classical raw materials. Indeed, the Portland cements are considered to be a cause of the global warming increase due to the large amount of  $CO_2$  that is produced by the related industries. Another reason to seek for alternative cement formulation is the related energy cost for the production of hydraulic cements. In addition, the need of cement products to be used in humid or acidic environments has aroused interest in studies that focused on the development of cements with low setting time. This research will assist, in solving the problems resulting from release of green-house gases (global warming), when talc is used to substitute cement partially at different percentage of replacement. Cement can be made cheaper, and cement raw materials may be preserved. Hence the outcome of this would attempt to optimize the use of sandcrete buildings as an infrastructure for  $CO_2$  sequestration in Nigeria.

This research will assist, in solving the problems resulting from the release of green-house gases (global warming), when talc is used to substitute sand partially at different percentage of replacement.

#### **1.5** Scope of the Study

The scope of this study was limited to the evaluation and optimisation of the process parameters affecting the uptake of  $CO_2$  by sandcrete-talc composite and it effect on compressive strength.

#### **CHAPTER TWO**

# 2.0 LITERATURE REVIEW

# 2.1 Portland Cement Production

Ordinary Portland Cement (OPC) is mainly composed of calcium silicate minerals. OPC is produced out of limestone rock, chalk or clay. These minerals are then crushed and some minerals (from waste or by-products) are added to ensure the correct chemical composition for cement production. At this stage, the minerals are calcined inside a kiln. At 900-1350°C the dicalcium silicate ( $C_2S$ ), tricalcium aluminate ( $C_3A$ ) and calcium aluminium iron oxide ( $C_4AF$ ) are formed. Above 1350°C, the clinker melts and there is formation of tricalcium silicate ( $C_3S$ ). Finally, the clinker is cooled and stored before a small amount of gypsum is added to regulate the cement setting. The cement is grinded before use (Lerigoleur, 2014).

## 2.1.1 Portland cement properties and use

Depending on the application, different types of Portland cement are used. The type I is used for general purpose. It contains a fairly high  $C_3S$  content in order to ensure a good early strength. It is used for general construction such as buildings, bridges, pavements and precast units. The precast application made it suited for early age carbonation curing because it is easier to apply the method on precast units.

Type I cement is not sulfate resistant. Therefore, type II and V cements have been developed when structures are exposed to soil or water containing sulfate ions. Moreover, type I Portland cement does not have a high early strength as the type III cement. To improve the early strength steam curing has been used for years but it is a very expensive process. Hence, early age carbonation curing has been developed. A recent study has found that early age carbonation curing also enhances the durability of the concrete and sandcretes (sulfate attack, water absorption and chloride ion penetration) (Rostami, *et al.*, 2011, and Azara, 2013).

# 2.1.2 Hydration

During hydration of Portland cement, the major products formed are the CSH, the calcium hydroxide (CH), the sulphoaluminate and the ettringite. CSH is known to be the main binding phase in all Portland cement-based systems; its exact nature is central to the science of cement and concrete. Since late 19th century, the first studies were devoted to understand CSH, which is central for hydration of Portland cement. Numerous works studied CSH chemical composition (Taylor, 1997). However, the poor crystalline structure of CSH lend to difficulties to characterize the CSH phase.

The chemical equations of Portland cement hydration concerning CSH could be described as (Lerigoleur, 2014):

$$2C_3S + 6H_2O \rightarrow C_3S_2H_3 + 3CH$$
 (2.1)

$$2C_2S + 4H_2O \rightarrow C_3S_2H_3 + CH \tag{2.2}$$

#### 2.1.3 Weathering carbonation

In the field, concrete is exposed to environment. This means attack from different type, such as freeze and thaw, chloride and carbonation. This carbonation after full hydration and under a low  $CO_2$  pressure is called weathering carbonation. Groves (1991) noted that, for a W/C ratio of 0.5, the CaCO<sub>3</sub> is mixed within the CSH structure densifying it. Studies showed that sandcrete strength could be enhanced (Richardson, 1993, Cheng-Feng and Jing-Wen, 2005 and Jerga, 2004). Nevertheless, the overall impact is detrimental because the drop of pH inside the pore solution can lead to depassivation and then corrosion of

rebars. Moreover, with high W/C ratio, the overall strength is lower (Diamond, 2005). The reactions responsible for weathering carbonation are:

$$CH + CO_2 \rightarrow CaCO_3 + H_2O \tag{2.3}$$

$$C_3S_2H_3 + 3CO_2 \rightarrow 2SiO_2 + 3CaCO_3 + 3H_2O$$

$$(2.4)$$

The CaCO<sub>3</sub> that occurs is mainly in 3 different crystalline structures: calcite, vaterite and aragonite (Taylor, 1997). Nevertheless it can also be amorphous in CSH phase. To study weathering carbonation, high pressure for accelerated tests and weathering carbonations were done to compare the laboratories testing with the field conditions (Groves, 1991). These studies are interesting for this work, because they deal with pure  $CO_2$ . Pure  $CO_2$  is also used in early age carbonation.

# 2.1.4 Carbonation curing

Due to improved strength in weathering carbonated concrete and the possibility to store  $CO_2$  that arose during the past decades , attempts to use carbon dioxide right after mixing, also referred to as carbonation curing, to enhance its early and overall strength has been conducted (Rostami*et al.*, 2011). First, the carbonation curing was decided to be directly after mixing, thus the two reactions that occurred are:

$$2C_3S + 3CO_2 + 3H_2O \rightarrow C_3S_2H_3 + 3C\overline{C}$$

$$(2.5)$$

$$2C_2S + CO_2 + H_2O \rightarrow C_3S_2H_3 + C\overline{C}$$
(2.6)

As it can be seen from Equations (2.5) and (2.6), the carbonation curing generates CSH and calcium carbonate, and no CH. The strength is enhanced within a few minutes to a few hours. Therefore, the use of carbonation curing has been successfully studied to

replace partially the steam curing (Rostami *et al.*, 2011). A better durability has been found.

Nevertheless, the carbonation applied immediately after mixing faces one problem: free water remains at the surface of the grains that hinders the carbon uptake and thus the strength gain. Therefore, a new process was developed, referred to as early age carbonation curing. It is a carbonation curing process but has a new approach because of a presetting in control conditions. It allows some water evaporation leading to a higher carbonation. After carbonation, the water loss is retrieved and the samples are sealed for subsequent hydration (until 28 days). It appears that carbonation curing is less expensive and more environmental friendly than steam curing (Rostami *et al.*, 2011).

The main difference between carbonation curing and the weathering carbonation is that it started with hydration, followed by carbonation and finished with rehydration again. It could be related to the tests done in laboratories to study weathering carbonation, except that weathering carbonation works were performed with completely hydrated paste. For this method all the equations (2.1) to (2.6) occur: the two first equations (2.1) and (2.2), then the equations (2.3) to (2.6) occur at the same time (corresponding to carbonation) and then again equations (2.1) and (2.2).

Carbonations of  $C_3A$  and  $C_4AF$  are not assumed because they are not  $CO_2$ -reactive in a short period of time (Shtepenko, 2006). They were not reactive during the first five hours. Moreover, carbonation of ettringite will be also out of the scope because it has a very slow kinetic reaction with carbon dioxide (Glasser, 2000).

### 2.2 Carbon Capture and Storage (CCS)

Carbon capture and storage (CCS) includes a large range of technologies that involve different processes for CO<sub>2</sub> capture, separation, transport, storage and monitoring. They are usually defined as the removal of CO<sub>2</sub> that would otherwise be emitted in the atmosphere. CCS can reduce emissions by 80%-95% from very large point source emission sources such as industry power plants specifically for the cement or energy industries (Leung *et al.*,2014). Using this approach, the carbon is captured precombustion and post-combustion, separated from the sorbent, stored or reutilized industrially (Leung et al., 2014). The most used and applicable reservoirs for these types of technologies are depleted oil and gas reservoirs, un-minerable coal beds as well as deep saline formations (Farrelly *et al.*, 2013).

As reported by the International Energy Agency (2011), if no proactive mitigation action is taken, energy-related CO<sub>2</sub> emissions are likely to be 40 - 110% higher in 2030 than they were in 2000 (23.5 Gt CO<sub>2</sub> per annum). The latest figures indicated that world CO<sub>2</sub> emissions from fuel combustion were 29.4 Gt in 2008. By 2100, atmospheric CO<sub>2</sub>concentrations could reach 540-970 ppm resulting in a global mean temperature rise of 1.8 - 4°C (Houghton *et al.*, 2001). A temperature increase of such enormity would have extreme implications for water and food availability, human health, ecosystems, coastlines and biodiversity which sum up the concept of climate change in the study conducted by (Bernstein *et al.*, 2007).

Based on scientific study, it is evident that modern-day emission of  $CO_2$  is primarily from the burning of fossil fuels in large combustion units such as those used for electric power generation. There is a need to effectively manage the large point sources of  $CO_2$  emission from power plants and large industrial processes in order to significantly mitigate climate change. This process forms a fundamental part of the concept of carbon capture and storage (CCS). A report by the International Panel for Climate Change (2002), considers CCS as an option in the portfolio of mitigation actions for stabilization of atmospheric greenhouse gas concentrations.

Praetorius and Schumacher (2009), report that CCS is not a new concept. They further state that technologies and practices associated with carbon capture and transport, and some types of carbon sequestration, geologic storage for example, have been in commercial operation within various industries for 10-50 years.  $CO_2$  capture systems are present in coal and natural gas fired power generation, coal gasification facilities and various industrial facilities. CCS is attracting growing interest around the world, particularly in countries where electricity generation and export income are heavily dependent on fossil fuels, such as China, Western Europe, Australia, Canada and the US (van Alphen *et al.*, 2010). Analysis by Metz *et al.*, (2005) indicate that CCS may contribute up to 15-55% of the cumulative global climate change mitigation effort by 2100 and that the inclusion of CCS in a mitigation portfolio could reduce the costs of stabilizing atmospheric  $CO_2$  concentrations by close to 30%.

## 2.2.1 Carbon capture

Due to the fact that carbon dioxide is formed during various combustion processes, specific techniques must be put into place to choose the appropriate removal. The main  $CO_2$  systems that are associated with these different processes include pre-combustion, post-combustion, oxyfuel combustion. Table 2.1 gives an overview of the advantages and disadvantages of each of the technologies and the area of application.

<b>Table 2.1:</b>	The	advantages	and	disadvantages	s of th	ne different	technologies	used	for
carbon caj	pture	•							

Capture Process	Application Area	Advantages	Disadvantages
Post combustion	Coal-fired and gas fired plants	Technology more mature than other alternatives; can easily retrofit into existing plants;	Low $CO_2$ concentration affects the capture efficiency;
Pre-combustion	Coal-gasification plants	High CO <sub>2</sub> concentration enhance sorption efficiency: fully developed technology, commercially deployed at the required scale in some industrial sector: opportunity for retrofit to existing plant;	Temperature associated heat transfer problem and efficiency decay issues associated with the use of hydrogen-rich gas turbine fuel: high parasitic power requirement for sorbent regeneration; inadequate experience due to few gasification plants currently operated in the market: high capital and operating costs for current sorption system
Oxyfuel combustion	Coal-fired and gas- fired plants	Very high CO <sub>2</sub> concentration that enhances absorption efficiency: mature air separation technologies available: reduced volume of gas to be treated, hence required smaller boiler and other equipment.	High efficiency drop and energy penalty: cryogenic O <sub>2</sub> production is costly: corrosion problem may arise;
Chemical looping combustion	Coal-gasification plants	$CO_2$ is the main combustion product, which remains unmixed with $N_2$ , this avoiding energy intensive air separation:	Process is still under development and inadequate large scale operation experience

Source: (Leung et al., 2014).

#### 2.2.2 Pre-combustion

Pre-combustion capture normally occurs for coal or natural gas, where the fuel is pre treated in order to release the minimal amount of carbon dioxide (Leung *et al.*, 2014). Coal undergoes the gasification process where it is pretreated in a gasifier under low oxygen levels creating carbon monoxide and hydrogen gas (Leung *et al.*, 2014). The CO will then be converted to carbon dioxide through a water-gas shift reaction and subsequently captured. This technique can be applied to the Integrated Gasification Combined Cycle power plants that use coal as fuel. Furthermore, natural gas can also be treated through a pre-combustion process as it mainly contains methane. It can be reformed to synthesis gas that contains both carbon monoxide and hydrogen gas using the reaction below (Leung *et al.*, 2014).

$$CH_4 + H_2O \rightarrow CO + H_2$$
 (Leung *et al.*, 2014) (2.7)

#### 2.2.3 Post-combustion CO<sub>2</sub> capture

This is the capture of  $CO_2$  from flue gases produced by combustion of fossil fuels and biomass in air. With this type of capture, flue gas is passed through equipment, separating most of the  $CO_2$  instead of the traditional practice of discharging it directly to the atmosphere. The  $CO_2$  is then fed to a storage reservoir and the remaining flue gas is discharged to the atmosphere as shown in Figure 2.1. Post-combustion capture can best be applied to oil, coal and gas power plants (IPCC, 2002; IEA WEO, 2004; IEA CCC, 2005).



Figure 2.1 : A schematic diagram showing the types of CO<sub>2</sub> capture systems (Source : Amponsah-Dacosta, 2017).

# 2.2.4 Oxy-fuel combustion CO<sub>2</sub> capture

In oxy-fuel combustion, nearly pure oxygen is used for combustion instead of air, resulting in a flue gas that is mainly  $CO_2$  and  $H_2O$ . If fuel is burnt in pure oxygen, the flame temperature is excessively high, but  $CO_2$  and  $H_2O$ -rich flue gas can be recycled to the combustor to moderate this. Oxygen is usually produced by low temperature (cryogenic) air separation and novel techniques to supply oxygen to the fuel, such as membranes and chemical looping cycles are being developed. The power plant systems of reference for oxy-fuel combustion capture systems are the same as those noted above for post-combustion capture systems (IPCC, 2002).

# 2.2.5 CO<sub>2</sub> Separation from energy related sources

 $CO_2$  has been captured from industrial process streams for 80 years, although most of the  $CO_2$  that was captured was vented to the atmosphere because there was no incentive or

requirement for storage (Amponsah-Dacosta, 2017). Presently, capture is generally required to be able to economically transport and store the  $CO_2$  (Rubin, 2003; Herzog, 2010). Metz *et al.* (2005) gave details of a number of  $CO_2$  separation technologies. Observations made during their study stated that  $CO_2$  may be captured post-combustion, pre-combustion, from oxy-fuel combustion or from industrial process streams. Technologies used in these systems include the separation of  $CO_2$  with sorbents, membranes, cryogenic distillation and chemical-looping with chemical absorption being the most common method used.

#### 2.2.6 Transportation of CO<sub>2</sub> captured

Once  $CO_2$  has been separated and captured as part of CCS, it must be transported to a storage area. Practical modes of overland transport include motor carrier (tankers), rail, and pipeline. The most economic method of transport depends on the locations of capture and storage, distance from source to sink, and the quantities of  $CO_2$  to be transported (Figure 2.2) (Sarv, 2001 and Praetorius and Schumacher, 2009). An infrastructure must be developed to move  $CO_2$  from its source to the storage site. Transporting large quantities of  $CO_2$  is most economically achieved with a pipeline (Sarv, 2001). An important technical consideration in the design of  $CO_2$  pipelines is that the  $CO_2$  should remain above its critical pressure. Recompression is often needed for pipelines over 150 km length. However, it may not be needed if a sufficiently large pipe diameter is used (Hattenbach *et al.*, 1999).



# Figure 2.2: CCS of CO<sub>2</sub> transportation modes (Source: Norisor *et al.* 2012)

Natural gas pipelines are a good analogue to a  $CO_2$  pipeline network for purposes of understanding costs. First, for a given pipeline diameter, the cost of construction per unit distance is generally lower, the longer the pipeline. Second, pipelines built nearer to populated areas tend to be more expensive. Finally, road, highway, river, or channel crossings and marshy or rocky terrain also greatly increase the cost (de Figueiredo *et al.*, 2007). Seiersten (2001), states that a transportation infrastructure that carries carbon dioxide in large enough quantities to make a significant contribution to climate change mitigation will require a large network of pipelines. There have been few studies that have addressed the cost of  $CO_2$  transport in detail. However, earlier work by Svensson et al., (2004), identified pipeline transport as the most practical method to move large volumes of  $CO_2$  overland. As discussed in the section (2.2),  $CO_2$  has already been separated and presumably captured into metal canisters. The transportation would therefore take a different approach. It would be impractical to re-channel the already captured  $CO_2$  into the pipelines. An appropriate suggestion would be to transport the jars of  $CO_2$  to the storage area by road using trucks.

#### 2.2.7 CO<sub>2</sub> storage

Storage or sequestration of  $CO_2$  can be accomplished through geological storage, ocean storage, industrial use and mineral sequestration.  $CO_2$  is generally proposed to be injected to depths greater than 800 m, where it is in a super critical state (Matter and Keleman, 2009).

# 2.2.8 Geological storage

The storage of  $CO_2$  within geological formations is regarded as a natural process in the Earth's upper crust.  $CO_2$  derived from biological activity, igneous activity and chemical reactions between rocks and fluids accumulates in the natural subsurface environment as carbonate minerals, in solution or in gaseous or supercritical form, either as a gas mixture or as pure  $CO_2$  (Bachu and Shaw, 2005). According to Liu and Zhao (1999), the lithosphere acts as an overwhelmingly dominant natural  $CO_2$  sink with 99% of the carbon trapped in the rocks of the Earth's crust. Yuan (1997), further states that these carbonate rocks cover an area of about 22 million km<sup>2</sup> worldwide, with over 90% of rock-forming carbonates consisting of calcite (CaCO<sub>3</sub>) and dolomite (CaMg(CO<sub>3</sub>)<sub>2</sub>).

As a result of the ever-increasing emission of  $CO_2$ , (Heinrich *et al.*, 2004) have valid proof that there are opportunities available to store  $CO_2$  in geological formations in a variety of underground geological settings in sedimentary basins, oil fields, depleted gas fields, deep coal seams and saline formations are all possible storage formations as shown in Figure 2.3 by (Priestnall, 2013). Metz *et al.*, (2005) also concluded that there was available evidence that suggested that there was a technical potential of at least about 2000 Gt  $CO_2$  of storage capacity in geological formations. This is a large number, about two orders of magnitude greater than total annual worldwide  $CO_2$  emissions, indicating the potential of CCS to be a significant  $CO_2$  mitigation strategy.





#### Source: (Amponsah-Dacosta, 2017)

A study conducted by Surridge and Cloete (2009) claimed that South Africa should concentrate its efforts on geological storage of  $CO_2$ . Their study targeted areas such as the 55m thick Vryheid Formation, located in KwaZulu Natal, which forms part of the coal deposits and made up of carbonaceous shale and sandstone with dull coal seams. Other targeted areas included geological formations within the Free State province. A combination of the above mentioned potential storage sites was estimated to store a total of 287 Gt  $CO_2$ .

#### 2.2.9 Ocean storage

The Earth's oceans cover over 70% of the Earth's surface with an average depth of about 3,800 meters; hence, there is no practical physical limit to the amount of anthropogenic  $CO_2$  that could be placed in the ocean. However, the amount that is stored in the ocean on the millennial time scale depends on oceanic equilibration with the atmosphere.

Deductions from ocean modeling suggest that injected  $CO_2$  will be isolated from the atmosphere for several hundreds of years and that the fraction retained tends to be larger with deeper injection. Additional concepts to prolong  $CO_2$  retention include forming solid  $CO_2$  hydrates and liquid  $CO_2$  lakes on the sea floor, and increasing  $CO_2$  solubility by, for example, dissolving mineral carbonates. Over centuries, large scale ocean water mixing can disturb isolation of injected  $CO_2$  and cause leakage to the atmosphere (Akai *et al.*, 2004).

Experiments show that added  $CO_2$  could harm marine organisms. Effects of elevated  $CO_2$  levels on individual organisms have mostly been studied on time scales up to several months near the ocean surface. Observed phenomena include reduced rates of calcification, reproduction, growth, circulatory oxygen supply and mobility as well as increased mortality over time. In some organisms these effects are seen in response to small additions of  $CO_2$ . Immediate mortality is expected close to injection points or  $CO_2$  lakes (Hill*et al.*, 2004).

# 2.3 Mineral Carbonation

Mineral carbonation is based on the reaction of  $CO_2$  with metal oxide bearing materials to form insoluble carbonates, with calcium and magnesium being the most attractive metals. In nature such a reaction is called silicate weathering and takes place on a geological time scale. It involves naturally occurring silicates as the source of alkaline and alkaline-earth metals and consumes atmospheric CO<sub>2</sub>. However, with mineral carbonation, where high concentration CO<sub>2</sub> from a capture step is brought into contact with metal oxide bearing materials with the purpose of fixing the CO<sub>2</sub> as carbonates (Seifritz, 1990 and Dunsmore, 1992). Suitable materials may be abundant silicate rocks, serpentine and olivine minerals for example or on a smaller scale alkaline industrial residues, such as slag from steel production or fly ash. In the case of silicate rocks, carbonation can be carried out either ex-situ in a chemical processing plant after mining and pretreating the silicates, or in-situ, by injecting CO<sub>2</sub> in silicate-rich geological formations or in alkaline aquifers Industrial residues on the other hand can be carbonated in the same plant where they are produced. It is worth noting that products of in-situ mineral carbonation and geological storage may be similar for the fraction of the CO<sub>2</sub> injected for geological storage that reacts with the alkaline or alkaline-earth metals in the cap rock leading to 'mineral trapping'.

In terms of material and energy balances, mineral carbonation can be schematized as illustrated in Figure 2.3, which applies to a power plant with  $CO_2$  capture and subsequent storage through mineral carbonation. With respect to the same scheme for a power plant with capture and either geological or ocean storage two differences can be observed. First, there is an additional material flux corresponding to the metal oxide bearing materials; this is present as input and also as output, in the form of carbonates, silica, non-reacted minerals and for some input minerals product water. Secondly, for the same usable energy output, the relative amounts of fossil fuels as input and of energy rejected as lower grade heat are different.



Figure 2.4: Material and energy balances through the system boundaries for a power plant with CO<sub>2</sub> capture and storage through mineral carbonation

# In-situ carbonation is an operation similar to geological storage, while ex-situ carbonation involves processing steps requiring additional energy input that are difficult to compensate for with the energy released by the carbonation reaction. With present technology there is always a net demand for high grade energy to drive the mineral carbonation process that is needed for: (i) the preparation of the solid reactants, including mining, transport, grinding and activation when necessary; (ii) the processing, including the equivalent energy associated with the use, recycling and possible losses of additives and catalysts; (iii) the disposal of carbonates and byproducts. The relative importance of the three items differs depending on the source of the metal oxides, for example whether they are natural silicates or industrial wastes.

Despite this potential energy penalty, interest in mineral carbonation stems from two features that make it unique among the different storage approaches, namely the abundance of metal oxide bearing materials, particularly of natural silicates, and the permanence of storage of  $CO_2$  in a stable solid form. The fossil fuel input provides energy both to the power plant that produces  $CO_2$  and to the mineralization process (either directly or indirectly via the power plant). The other materials input serve all processes

within the system boundaries and includes the metal oxide bearing materials for mineralization.

# 2.4 Supplementary Cementitious Materials

Cementitious materials are the product of complex chemical reactions that take place when cement (primarily tricalcium and dicalcium silicates) reacts with water to form various hydration products with nanoscale features that are arranged in a multi-scale fashion in a three-dimensional space as alite and belite ( $C_3S$  and  $C_2S$ ) comprise over 80% of most cements, their hydration products dominate in terms of volume. Both silicate phases react with water to form a hydrated version of calcium silicates (C-S-H) and calcium hydroxide (CH or Portlandite):

# $C_3S/C_2S+H\rightarrow C-S-H+CH$

The poorly crystalline, highly porous and non-conductive nature of the material makes it complex to study and as a result the actual mechanisms that govern the formation and its properties remain unidentified to date. In fact, the hyphens in C-S-H reflect its uncertain stoichiometry Cementitious products comprise the glue that holds concrete together. These materials include traditional Portland cement and other cementitious materials, such as fly ash, ground granulated blast furnace slag (GGBS), limestone fines and silica fume. These materials are either combined at the cement works (to produce a composite cement) or at the concrete mixer when the concrete is being produced (the cementitious product is called a combination in this case).

#### 2.4.1 Rice husk ash (RHA)

Rice husks are by-products that are accountable for 20% of the Rice husks ash (RHA) weight after the husks have been burned. This ash with its high specific surface area and

non-crystalline features, can result in highly pozzolanic reactions, hence, this ash is considered a better alternative for Portland cement in fillers for high-performance concrete (Owaid *et al.*, 2012). One of the most important causes of the highly pozzolanic reaction is due to the fineness of RHA after grinding. According to (ASTM C 618-94, 1994), these rice husk ashes should act in accordance with the current standards, such as ASTM C 618-84. Rice husk ash is selected as a class F pozzolan because the amount of CaO in the rice husk ash is less than 10 per cent. Furthermore, with such large size particles, it is possible for RHA particles to promote high surface area because of their cellular properties. In addition, it has been found that, although other supplementary materials (e.g., slag and fly ash) are all equally responsible for concrete strength development, rice husk ash has better pozzolanic reactivity, which leads to earlier pozzolanic reactions at a controlled temperature. This feature is why rice husk ash is used as an additive in high-performance concrete.

#### 2.4.2 Fly ash (FA)

Fly ash (FA) or pulverized-fuel ash (PFA) is a pozzolanic material that is used in cementbased materials to improve long-term strength, workability, resistance to sulfate attack and durability in concrete. When harvested from electric power generation plants, FA can exhibit high pozzolanic activity because it is produced from the burning of pulverized coal (Owaid *et al.*, 2012). Upon ignition in the furnace, a large amount of the volatile matter and carbon in the coal are burned off. During combustion, the coal's mineral impurities (such as clay, feldspar, quartz, and shale) fuse in suspension and are passed away from the combustion chamber by the exhaust gases. In the process, the fused material cools and solidifies into spherical glassy particles called fly ash. The fly ash is then collected from the exhaust gases by electrostatic precipitators or bag filters. Fly ash is a finely divided powder resembling Portland Cement (Kosmatka *et al.*, 2003). The fly ash particles are fine-grained particles normally with sized between less than 1µm to over 100µm. On the other hand, the diameter of the average fly ash particle is smaller than 20µm in size, and only 10-30% of all fly ash particles have a size of more than 45µm. The surface area of fly ash, typically varies from 300 to 500 m<sup>2</sup>/kg, with 200 m<sup>2</sup>/kg as the lowest surface area and 700 m<sup>2</sup>/kg highest surface area. The density of fly ash also ranges from 540 to 860 kg/m<sup>3</sup>. The bulk density for FA under close-packed storage or vibration ranges from 1120 to 1500 kg/m<sup>3</sup>.Fly ash is primarily silicate glass containing silica, alumina, iron, and calcium. Minor constituents are magnesium, sulfur, sodium, potassium, and carbon. Crystalline compounds are present in small amounts. The relative density (specific gravity) of fly ash generally ranges between 1.9 and 2.8. Depending upon its chemical and mineral constituents, the color may vary from tan to dark gray.

# 2.4.3 Ground granulated blast furnace slag (GGBFS)

Ground Granulated Blast Furnace Slag (GGBFS) is a by-product material formed from the blast-furnaces used to make iron, at a temperature of around 1600°C, a mixture of iron ore, coke and limestone in the furnace can yield two products, namely, molten iron and molten slag. Due to its light weight, molten slag floats on top of the molten iron. Chemically similar to Portland cement, molten slag comprise mostly of silicon dioxide (30% - 40%) and CaO (40%). Silicates and alumina, are the main ingredients in molten slag, and then cooled down by high-pressure water jets. Ultimately, these rapidly waterquench process forms a glassy granulate particles. These particles are further processed by drying and grinding to the required size to make a fine, glassy powder known as granulated blast furnace slag. The results can be different and depend very much upon the speed of cooling of the slag melts. In the case of slow cooling, for example, the resulting material is composed of a stable solid with Ca-Al-Mg silicates. However, the granulated slag is ground to a size less than 45µm and has a surface area of approximately 400 to 600  $m^2/kg$ . Granulated blast furnace slag was first developed in Germany, which has also been used for general purpose concrete in North America. Since its beginning, ground granulated blast furnace slag has been widely used as a cementitious materials (Owaid *et al.*, 2012).

In concrete, the GGBFS rate of replacement vary from 35 to 65 per cent, which at last results in a reduction of 0.5 ton of CO<sub>2</sub> per half replacement of each ton of Portland cement. Some components of slag can have more than 70 per cent cementitious properties. According to ASTM C 989 (AASHTO M 302), slag can be classified by it increasing level of reactivity into three strength grades, namely, Grade 80, 100, or 120, (ASTM C 989-93, 1993). Studies show that the early strengths in concrete containing GGBFS, when it is compared with Portland cement are lower due to its initial reaction rate. Hence, when water is blended withslag Portland cement, the hydration mechanism is activated in the component of Portland cement, which is then followed by the immediate reaction of slag, thereby release calcium and aluminum ions. This process is completed by the formation of a C-S-H gel through the reaction of slag with alkali hydroxide and  $Ca(OH)_2$ . This compound of calcium plays a vital rolein slag hydration because it can increase the cementitious properties. In addition, gypsum is another slag hydration activator that is released by  $C_3S$  and  $C_2S$  hydration.

# 2.4.4 Silica fume (SF)

Silica fume (SF) has been widely used as a supplementary cementing material for producing high performance concrete elements and is formed from the smelting of condensed silica fume and volatilized silica in submerged-arc electric furnaces. SF is also known as silica dust, condensed silica fume, micro silica or volatilized silica and is usually a grey or premium white color. In the ferro-silicon metal industry, this process is usually performed at a temperature of 2000°C, which leads to the reduction of high-purity

quartz to silicon. This process, in turn, produces SiO<sub>2</sub>vapors that, in the process of condensing and oxidizing, change to non-crystalline silica. Its by-product materials that result from the production of silicon metal and ferrosilicon alloys at approximately 75% silicon and contain 85-95% non-crystalline silica. When this amount is reduced to 50% silicon in the production of ferrosilicon alloys, the silica content is reduced and the by-product is less pozzolanic (Owaid *et al.*, 2012). In its non-crystalline form, condensed silica fume is basically silicon dioxide. Similar to fly ash, SF has a very fine spherical shape, can be sold in the form of powder or liquid; however, there is more accessibility to its latter form. Silica fume is usually used at 5% to 10% (by mass) of the total cementitious material (Kosmatka *et al.*, 2003). Similar to RHA, silica fume should also meet ASTM C 1240. The common SF particles are very small and less than 1µm in diameter. When SF is added to cement/concrete, it acts as a filler to fill the gaps between cement particles resulting in finer pore structure. Also more CSH gel can be formed in SF concrete due to the reaction that occurs between the silica in SF and the Ca(OH)<sub>2</sub> in hydrating cement.

#### 2.4.5 Metakaolin (MK)

Metakaolin (MK) is a pozzolanic material, which can provide many specific features. Metakaolin is available in many different varieties and qualities. Some of them also provide special reactivity. Metakaolin is a valuable admixture for concrete or cement applications. Usually 8% - 20% (by weight) of Portland cement had been replaced by metakaolin. Such a concrete exhibits favourable engineering properties (Narmatha and Felixkala, 2016). Unlike the other materials, it is not a by-product because it is obtained under carefully controlled conditions by calcinations of pure kaolinite clay, for the kaolinite to break down and produce an amorphous material for pozzolanic and latent hydraulic reactivity, a temperature between 650 and 900°C is required. It is only within

this temperature range that the calcium silicate hydrate (CSH) in cement paste can be produced as a result of the reaction of metakaolin with  $Ca(OH)_2$ , it is in this region that a high concentration of associated  $Ca(OH)_2$  crystals can result in increased porosity and lower strength (Owaid*et al.*, 2012). Compared with silica fume systems, it has been reported that metakaolin systems have a higher initial reactivity due to their higher rates of pozzolanic reaction and  $Ca(OH)_2$  consumption. Moreover, Metakaolin is used as an additive to concrete (approximately 10% of the cement mass) when very high strength and very low permeability are needed in special applications (Kosmatka *et al.*, 2003). Narmatha and Felixkala, (2016) drawn the following conclusions when Metakaolin in concret is partial replace with cement;

(1) The strength of Metakaolin concrete mixes over shoot the strength of OPC.

(2) 15% cement replacement by Metakaolin is superior to other mixes.

(3) The increase in Metakaolin content improves the compressive strength and split tensile strength up to 15% cement replacement.

(4) The results support the use of Metakaolin, as a pozzolanic material for partial replacement in producing high performance concrete.

# 2.5 Talc

Talc is an hydrated layered magnesium silicate with chemical formula  $Mg_3Si_4O_{10}(OH)_2$ . It is a hydrophobic material that easily blends and disperses within organic media including polymers.Talc is a phyllosilicate mineral known for having a hardness of one. It is a monoclinic mineral and occurs as a hydrothermal alteration of magnesium silicates that lack aluminum. Talc can occur in many different colors but is commonly light green or white and often has a greasy, "soapy" texture to it, giving it the common name soapstone. It forms closely with pyrophyllite and is often associated with asbestos minerals. Talc is an important industrial mineral that is mined using open-pit mining techniques in the United States. Talc is a mineral silicate mineral that forms through hydrothermal alteration. It is mined throughout the world, with America being the second largest producer. Talc is used in many different everyday products because its physical and chemical properties make it desirable for many different uses. Talc is widely used as reinforcing filler in several industrial products such as paper, paints, rubbers, polymers, ceramics, and refractory materials (Ngally *et al.*, 2014).

# 2.5.1 Talc as cementitious material

Ngally *et al.* (2015) carried out a research on Talc- based cementitious product: Effect of talc calcination and reported that The calcination is used to enhance the availability of magnesium from talc to react with phosphate for cement phase formation. It was shown that previous calcination of talc leads to products having enhanced mechanical performance due to the formation of more cement phase than in products based on raw talc. Talc fired at 900°C was found to be the one in which magnesium release was maximal. Firing at temperature higher than 900°C leads to the stabilization of enstatite, which decreased the magnesium availability. The cement phase is struvite, which was better detected on the X-ray patterns of the products involving fired talc. All the products have very rapid setting time and low shrinkage. Recently, a wide range of applications of magnesium phosphate cements (MPCs) were developed in civil engineering (Ngally *et al.*, 2015).

Yang *et al.* (2014) research on preparation, hydration, and properties of magnesium phosphate cement (MPC). MPC is the phosphate-bonded inorganic material derived from reactions between phosphate and magnesium oxide. The hydration reaction in the MgO- $NH_4H_2PO_4$  system is strongly exothermic, and the main product is struvite ( $NH_4MgPO_4 \cdot 6H_2O$ ). The setting time and mechanical properties depend on the proportion and
characteristics of raw materials, water to binder ratio, the addition of retarders, and admixtures. Moreover, the MPC demonstrates favorable durability performance compared with Portland cement. MPCs are among promising products. MPCs are chemically bonded phosphate ceramics, which were discovered and developed as dental cement in the late 19th century. In these materials, the chemical bonding is formed by a through solution acid-base reaction between dead burned magnesia and phosphate. MPCs also exhibit improved characteristics such as low setting time, ability to set and harden at low temperature as  $-20^{\circ}$ C high bonding strength, and very good durability including resistance to chemical attack and permeation.

Evidences has shown that magnesium from the talc structure can be involved in a reaction with phosphate to form cementitious products. However, the products performance, namely compressive strength, was low and when talc was partially replaced by magnesia (MgO) up to 10%, the performance was increased. This was an indication that the mechanical response of the products could be increased if the magnesium contained in talc is made more available for the reaction process. Thermal treatment of the talc was proposed as a potential way to increase magnesium availability. In the present study, talc was thermally treated at various temperatures and used for cementitious products formulation with potassium dihydrogenophosphate. Fourier transform infrared spectroscopy (FTIR), X-ray powder diffraction (XRPD), and scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM/EDS) were used to analyze the products. Setting time, linear shrinkage, and compressive strength are the mechanical parameters that are evaluated (Ngally *et al.*, 2015).

Ngally *et al.* (2014), investigate Talc as raw material for cementitious products formulation. They reported the characterization of a talc from Cameroon as a possible source material for cement formulation. To that end, the talc sample was characterized

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and mixed with a solution of sodium polyphosphate to formulate the cementitious products. Addition of magnesia (MgO) was done to analyze the effect of available MgO on the products. Fourier transform infrared, X-rays diffraction, linear shrinkage, compressive strength and scanning electron microscopy were used to analyze the products. The compressive strength increased with addition of MgO and the linear shrinkage decreased. All the analyses indicate that talc is a raw material of interest in cementitious products formulation; however, they concluded that MgO is a key factor for a better performance of the products. The composition of talc makes it a potential raw material in cement formulation since magnesium can beneficially interact with phosphate to give cementitious material, as in phosphate-magnesia cements. Talc-based cementitious material is already used as prosthesis in dental surgery and also used as a super plasticizer for the formulation of cement poor concrete to be used for boreholes sealing in rocks.

#### 2.6 Sandcrete

Sandcrete is a yellow-white building material made from a binder (typically Portland cement), sand in a ratio of 1:8, and water. Sometimes other ingredients may be added to reduce the amount of expensive Portland cement such as pozzolanas and rice husk ash. Sandcrete is similar but weaker than mortar, for which the ratio is 1:5. Soil cement and landcrete are similar materials but use other types of soil and hydraform blocks which are compressed, stabilized, earth blocks. Sandcrete is usually used as hollow rectangular blocks similar to concrete masonry units, often 45 centimetres (18 in) wide, 15 centimetres (5.9 in) thick, and 30 centimetres (12 in) long with hollows that run from top to bottom and occupy around one third of the volume of the block. The blocks are joined together with mortar.

ASTM C618 (2008) defined pozzolana as a siliceous or siliceous and aluminous materials, which, in itself, possesses little or no cementitious value but which will, in finely divided form in the presence of moisture react chemically with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties. A way of reducing the cost of cement in sandcrete blocks is by partially replacing it with some cheaper and locally available materials, referred to as pozzolana which in Nigeria are abundant. Cement is an essential ingredient serving as a binder in almost all forms of construction. In many countries, particularly in the third world countries, Ordinary Portland Cement (OPC) is expensive. This has severely limited the construction of affordable houses. The growing concern of resource depletion and global pollution has challenged many researchers to seek and develop new materials relying on renewable resources. Ashes derived from the industrial and agricultural wastes or residues such as Fly Ash, Rice Husk, Ground Granulated Blast Furnace Slag, Corn Cob, Periwinkle Shell and so on, have been found by various researchers to be pozzolanic materials containing reactive silica or alumina which, when mixed with water, possesses cementitious properties (Umoh and Olusola, 2012). These by-products contribute to the improvement of thermal, strength and durability performances of mortar or sandcrete blocks performance as well as reduction of waste energy and carbon dioxide in the production of cement (Nagataki, 1994).

Abdullahi *et al.* (2015), carried out study on preparation, characterization and predictive model of Cement-Based composite for atmospheric  $CO_2$  sequestration. The mineralogical compositions and microstructure of the composites as carbonation progressed were characterized using X-ray Diffractometer (XRD), X-ray Fluorescence (XRF), Scanning Electron Microscopy (SEM) and Elemental Diffraction Spectrometer. The work determined the sequestration of carbon dioxide of the composite using accelerated carbonation curing and phenolphthalein spraying techniques, also concentration of carbonates was determined using back titration method.

At 20% replacement of siliceous sharp sand with kaolin higher carbon dioxide sequestration for both experimental and simulated results were observed at 30  $^{\circ}$ C (0.029 kg/m<sup>3</sup>; 0.02992 kg/m<sup>3</sup>), it was observed that at 50% replacement of siliceous sharp sand with talc at 35  $^{\circ}$ C (0.0406 kg/m<sup>3</sup>; 0.0425 kg/m<sup>3</sup>) while for standard sandcrete (S-S) it was observed at 30  $^{\circ}$ C (0.0128 kg/m<sup>3</sup>; 0.0134 kg/m<sup>3</sup>).

From the results, carbon dioxide sequestration analysis showed that S-K sequestratered 56%  $CO_2$  while S-T adsorbed 68.5% when compared with the reference samples (S-S). The use of kaolin and talc to replace siliceous sharp sand in sandcrete increases compressive strength at 7 and 28 days of curing due to formation of C-S-H gel.

Younsi *et al.* (2013), investigate accelerated carbonation of concrete with high content of mineral additions. Their research focus on studying the accelerated carbonation of concrete mixtures with high substitution rates of cement by fly ash or blast-furnace slag. This study was aimed at determining the durability of the sample mixtures, particularly their resistance against carbonation and their equivalence of performances. Their results show that mixtures with high substitution rates of cement by fly ash (50%) or blast-furnace slag (75%) could replace mixtures in accordance with the current standard.

Their results showed low hydration rates and low Portlandite (CH) contents when Portland cement was substituted by fly ash or BFS. As reported, these concretes are more affected by drying which tends to stop hydration and degrades the concrete external layer (higher porosity, lower Portlandite content), as shown by numerical simulations. Also, high substitution concretes are more sensitive to water curing. Kolovos *et al.* (2016), examined the properties of sandcrete mixture with metakaoline. Their investigation considered using metakaolin as a mineral admixture in the synthesis of sandcrete mixture.

The results showed that the mechanical property exhibited by the mixture makes it a good option for soil stabilization or a better material for structural element construction in civil Engineering projects.

It was concluded that metakaolin can be used in the production of sandcrete to improve it mechanical properties. Higher values for average compressive strength of sandcrete samples were recorded not only at medium but also at low and high water to binder (W/B) ratio, in comparison to the reference samples. The higher the W/B ratio, the higher the reduction for compressive strength of sandcrete samples at 28 days. In sandcrete samples produced with 50% w/w of the modified binder, metakaolin exhibits a pronounced pozzolanic reactivity leading to improved compressive strength for all designed W/B, especially in low and medium ones.

However, the optimization process used in production of the sampled mixture is complex and prone to error. Also, metakaoline is costly material and not readily affordable especially in local areas, adding to the fact that the quantity of it availability is also limited considering the statistics of civil Engineering projects in recent times.

Marangu *et al.* (2019), investigate carbonation resistance in hydrated Cement based materials their research shows that blended cements are more subjected to degradation due to carbonation than OPC, when  $CO_2$  is ingress within the porous mortar matrix diffusion process is controlled. Subsequent chemical reaction between  $CO_2$  and cement hydration products (mostly calcium hydroxide [CH] and calcium silicate hydrate [CSH]) results in degradation of cement based materials. CH offers the buffering capacity against

carbonation in hydrated cements. Partial substitution of OPC with pozzolanic materials however decreases the amount of CH in hydrated blended cements. Hence, low amounts of CH in hydrated blended cements make them more subjected to degradation as a result of carbonation compared to OPC. The magnitude of carbonation affects the service life of cement based structures significantly. It is therefore apparent that sufficient attention is given to carbonation process in order to ensure resilient cementitious structures.

Rostami et al. (2012), carried out studied on Microstructure of Cement paste subject to early carbonation curing; they examine the effect of early carbonation on performance of paste at different ages. The study investigates the mechanism of concrete carbonation at early age through the microstructure development of its cement paste. Early carbonation was carried out after 18-hour initial controlled air curing. It was found that early carbonation could accelerate early strength while allowing later hydration. The short term carbonation created a microstructure with more strength-contributing solids than conventional hydration. Calcium hydroxide was transformed to calcium carbonates, and calcium-silicate-hydrate became intermingled with carbonates, generating an amorphous calcium-silicate-hydro carbonate binding phase. Carbonation modified C-S-H to retain its original gel structure. The re-hydration procedure applied after carbonation was essential in increasing late strength and durability. However, the early carbonation of precast concrete could reduce the carbonation shrinkage of assembled concrete structures in service. On the other hand early carbonation curing is a  $CO_2$  sequestration process that is capable of utilizing carbon dioxide recovered from cement kiln flue gas, allowing for carbon recycling within the cement industry.

#### 2.7 Mechanism of Carbonation Reaction in Sandcete-Talc Composite

Carbonation mechanism of cement-based materials is affected by a several parameters relating to the material properties and its surrounding environment. In particular, carbonation is governed by the diffusivity of  $CO_2$  within sandcrete and chemical reactions between  $CO_2$  and carbonated products. Two main phenomena affect the carbonation process: cement hydration and drying. Hydration is the main source of carbonatable products such as Portlandite and it governs the formation of pore structure, and thus the gas diffusivity. Drying affects both cement hydration and water saturation degree, thus gas diffusivity and the aqueous chemical reactions of carbonation (Younsi et al., 2013).

The atmosphere contains substantial amounts of carbon dioxide. However, gaseous  $CO_2$  cannot react directly with the hydrates of the cement paste. Thus the  $CO_2$  gas must first dissolve in water and form carbonate ions that in turn will react with the Ca ions of the pore water. When  $CO_2$  comes into contact with water at neutrality it forms bicarbonate.

$$CO_2 + H_2O \rightarrow H_2CO_3$$
 (Dissolution of  $CO_2$  in pore solution) (2.8)

Dissolution of talc in acidic solution and subsequent reaction with cement hydrated product (Abdullahi *et al.*, 2016).

$$Mg_{3}Si_{4}O_{10}(OH)_{2} + 5H_{2}CO_{3} + 3CaO.2SiO_{2}.3H_{2}O \rightarrow Mg(HCO_{3})_{2} + 2Mg(OH)_{2} + 3CaCO_{3}$$
  
+ 6SiO\_{2} + 6H\_{2}O (2.9)

Reaction between talc dissolution product with Portlandite (Abdullahi et al., 2016).

$$Mg(HCO_3)_2 + 2Ca(OH)_2 \rightarrow 2CaCO_3 + Mg(OH)_2 + 2H_2O$$
(2.10)

Direct carbonation of mineral talc inside the sandcrete-talc composite occur with the release of silica and magnesite as expressed by equation (2.11) (Abdullahi *et al.*, 2016).

$$Mg_{3}Si_{4}O_{10}(OH)_{2} + CO_{2} \rightarrow 3MgCO_{3} + 4SiO_{2} + H_{2}O \text{ (Direct sequestration)}$$
(2.11)

Reaction between cement hydrated product with hydrated carbon dioxide (Abdullahi et al., 2016).

$$Ca(OH)_2 + H_2CO_3 \rightarrow CaCO_3 + 2H_2O$$
(2.12)

Sequestration of dissolve carbon dioxide with cement binding gel (Abdullahi et al., 2016).

$$3CaO.2SiO_2.3H_2O + 3CO_2 \rightarrow 3CaCO_3 + 2SiO_2 + 3H_2O$$

$$(2.13)$$

# CHAPTER THREE

#### 3.0 MATERIALS AND METHODS

# 3.1 Equipment and Material

Table 3.1 and Table 3.2 shows the list of equipments and materials used for the experimental work.

# Table 3.1: List of Equipment

S/N	EQUIPMENT	SOURCE	MODEL
1	X-ray Diffractometer (XRD)		Model: DY614
			Empyrean by
			Panalytical
2	X-ray Fluorescence (XRF)	Shimadzu	MiniPal4
3	British Standard sieve	ADDIS Nigeria	H-4325
4	Weighing Balance (Mettler)	Shimadzu	JJ200

# Table 3.2: List of Materials and Reagents

Materials	SOURCE
Ordinary Portland Cement	Bida Central Market, Niger State
Fine Sand	River Ladzun, Bida, Niger State
Talc	Kagara, Rafi Local Government of Niger State
Water	Federal Polytechnic bore-hole in Bida Niger State
Beaker	Biochemistry Lab, FederalPolytechnic,Bida
Spatula	Biochemistry Lab, FederalPolytechnic,Bida
Measuring cylinder	Biochemistry Lab, Fed Poly Bida

#### 3.2 Characterization of Starting Materials

Natural Talc, Cement and Sand were characterized using X-ray Diffractometer (XRD) (Model: DY614 Empyrean by Panalytical) and X-ray Fluorescence (EDXRF) (Model: MiniPal4 embedded with X'pertHighScorePlus Software) to determine the mineralogical compositions and oxides compositions respectively.

#### **3.3 Design of Experiment**

The full factorial design criteria utilising two level-four factor  $(2^4)$  to investigate the degree of CO<sub>2</sub> sequestration and compressive strength of S-T composite for 7 and 28-day was carried out. The mass fractions of components and the experimental range and levels of independent variables are presented in Table 3.3.

Component	ID	Minimum(-1)	Maximum (+1)
Sand(g)	А	171.95	202.29
Talc (g)	В	10.12 (5%)	30.34 (15%)
Age (Day)	С	7	28
Cement (g)	D	33.70	33.71
Water (g)		16.85	16.85
Water/Cement		0.5	0.5

Table 3.3: Range of Mix Design Proportion for LDPE-Sandcrete Composite

The effect of each component on  $CO_2$  sequestration and compressive strength coupled with their interactions were calculated using Equation (3.1).

$$Effect(M) = \frac{\Sigma Y_{+}}{n_{+}} - \frac{\Sigma Y_{-}}{n_{-}}$$
(3.1)

Where  $Y_+$  represent a positive response,  $Y_-$  represent a negative response,  $n_+$  and  $n_-$  represent data points at each experimental level.

The main and interaction effects give a quantitative estimation of the influence the components (factors), or the interaction of the components (factors), have upon the response variable (CO<sub>2</sub> concentration and compressive strength). The number of main and interaction effects in a  $2^k$  full factorial design is  $2^k - 1$  with  $2^k - 1$  degree of freedom.

Table 3.4 shows the full factorial mix design proportions for factorial experiment (in grams of sandcrete) in coded values (-1 for low values and +1 for high values).

Design ID	Run	Factor 1	Factor 2	Factor 3	Factor 4
	order	A=Sand (g)	B=Talc (g)	C=Age (day)	D=Cement
					(g)
13	1	-1	-1	1	1
12	2	1	1	-1	1
6	3	1	-1	1	-1
7	4	-1	1	1	-1
1	5	-1	-1	-1	-1
8	6	1	1	1	-1
4	7	1	1	-1	-1
11	8	-1	1	-1	1
5	9	-1	-1	1	-1
16	10	1	1	1	1
9	11	-1	-1	-1	1
3	12	-1	1	-1	-1
14	13	1	-1	1	1
2	14	1	-1	-1	-1
10	15	1	-1	-1	1
15	16	-1	1	1	1

 Table 3.4: Factorial Design Showing Combination in Coded Factors

#### 3.4 Materials

The Ordinary Portland cement (OPC) with ISO 9001:2008 obtained from Dangote Cement Company meeting NIS 444-1:2003-cem 11/B-L 42.5R was used for the experiment. The fine aggregates were natural sharp sand conforming to American Standard for Testing and measurement (ASTM C33) with a specific gravity of 2.66 with moisture content 0.4%. Talc was obtained from Kagara in Rafi Local Government, Niger State. Suitable water conforming to NIS 554:2007 was used for the experiment to aid hydration of cement and talc and to facilitate the binding.

#### 3.5 **Production Procedure**

All mixes generated by Design Expert version 11 is presented in Table 3.4, each approximately  $1.25 \times 10^{-4}$  m<sup>3</sup> in volume, were prepared in the laboratory based on Abdullahi *et al.*, (2016) method of sandcrete production. All samples produced were made up of fixed cement-to-aggregate ratio of 1:6 and water-to-cement ratio (W/C) of 0.5 (Anosike and Oyebade, 2012). The casted cubes were demoulded after 24 hours and tested for compressive strength at 7- and 28- day respectively using Hydraulic Powered Press Machine Model No. 526, OGAWA SEIKI CO., LTD., Tokyo, Japan.

#### **3.6** Quantification of CO<sub>2</sub> Sequestered

The carbon dioxide sequestered by S-T specimens was determined using quantitative analysis. 157 g of pulverised carbonated sandcrete specimen was weighed into a volumetric flask. 20 ml of 0.1 M HCl acid was added and shaken thoroughly for  $CO_2$  to effervescence. After effervescence has stopped, distilled water was added to make it up to 1 dm<sup>3</sup>0.05 M NaOH was used to titrate against the standard solution of carbonated

sandcrete to neutralize the HCl acid using phenolphthalein indicator (Abdullahi *et al.*, 2016).

# 3.7 Compressive Strength Test

The compressive strength test for S-T specimens was carried out at 7 and 28-day cured in ambient conditions using Hydraulic Powered Press Machine Model No. 526, OGAWA SEIKI CO., LTD., Tokyo, Japan.

#### **CHAPTER FOUR**

#### 4.0 RESULTS AND DISCUSSION

#### 4.1 Characterization of Composite Materials

The chemical compositions of the starting materials (cement, talc and sand) are presented in Table 4.1, the mineralogical compositions are shown in Figures 4.1 and 4.2 respectively and morphological and elemental compositions are shown in Figure 4.3 to 4.1

Element	Sharp sand %	cement %	Talc %	
Na <sub>2</sub> O	1.697	0	0	
MgO	1.189	0	24.86	
$Al_2O_3$	8.003	2.796	1.165	
SiO <sub>2</sub>	50.213	11.55	48.932	
$P_2O_5$	0.091	0	0.049	
K <sub>2</sub> O	3.071	0.561	0.001	
CaO	2.493	52.911	0.309	
SO <sub>3</sub>	0	3.22	0	
$Cr_2O_3$	0.373	0	0	
Fe <sub>2</sub> O <sub>3</sub>	3.402	2.701	0.7	
MnO	0.001	0.045	0.064	
TiO	0	0.068	0.019	
Ti	0	0.023	0.001	
Ni	0	0	0.095	
CuO	0	0.003	0	
Al	4.24	1.246	0.411	
Na	0.91	0	0	
Mg	0.71	0	6.928	
Si	16.001	6.409	13.011	
Zn	0.001	0.001	0.133	
Ba	0	0.055	0.071	
Р	0.04	0	0	
S	0.008	1.211	1.354	
Κ	2.5	0.462	0.091	
Ca	1.793	15.122	0	
Cr	0.61	0	0	
Pb	0.001	0	1.293	
Fe	2.64	1.567	0.113	
Mn	0.001	0.035	0.013	
TOTAL	99.988	99.986	99.613	

# Table 4.1: Oxide Composition Using X-Ray Fluorescence (XRF)

Results as presented in Table 4.1 indicate that the Portland cement consists of 52.911% CaO and 11.55% SiO<sub>2</sub> with minute quantities of other metallic oxides. Abdullahi*et al.*, (2016) also reported approximately the same results with minor variations. The slight disparitycould be attributed to the chemical composition of cement raw materials and other mineral additives used during production. The sand consists of 50.213% SiO<sub>2</sub> while the mineral talc consists of 48.932% SiO<sub>2</sub>, 24.86% MgO and 1.165% Al<sub>2</sub>O<sub>3</sub> with traces of other oxides as depicted by the XRF analysis shown in Table 4.1.



Figure 4.1: XRD analysis of mineral talc



Figure 4.2: XRD analysis of Sand and Cement

Figures 4.3.and 4.4 illustrate the scanning electron micrographs and elemental analysis of cement used for production of composite. The micrograph shows a very fine homogeneous sized cement grains while elemental analysis showed that 66.68% of calcium (Ca) constitute the compound with moderate amounts of other elements ranging from 7.6% down to 0.52% (Abdullahi *et al.*, 2016).



Figure 4.3: SEM Micrograph of Cement



Figure 4.4 EDX of Cement

Figures 4.5 and 4.6 depict the scanning electron microscopy (SEM) and elemental analysis of Talc. It can be observed that the materials have a homogenous feature in which platy disc particles characteristic of talc could be observed, while elemental analysis indicate presence carbon (C) and calcium (Ca) with minute amount of other element (Ngally *et al.*, 2014).



Figure 4.5: SEM Micrograph of Talc



Figure 4.6 : EDX OF TALC

Figure 4.7 indicates micrograph of sand material. There are a lot of groves and holes on the surface of the material. These characteristics are obvious at low and high magnification. These features indicate that this material will accommodate the guest fluid into its intrinsic part while Figure 4.8 shows elemental analysis with higher percentage of Silicon (Si) with moderate amount of other element (Abdullahi *et al.*, 2016).



Figure 4.7: SEM Micrograph of Sand



Figure 4.8 EDX OF Sand

The morphological aspect of the sandcrtet-talc composite is presented in figure 4.9. It can be observed that the structure is homogenous with holes associated to tearing out of some remaining entatite grains, which are imprisoned in cement matrix. Other holes are attributed to a porosity which is associated to organisation of struvite grains that composed the composite. EDX analysis in figure 4.10 showed major amount of calcium (Ca) with moderate amounts of other elements (Ngally *et al.*, 2015).



Figure 4.9: SEM Micrograph of sandcrete-talc composite



Figure 4.10: EDX of Composite

# 4.2 Optimization of CO<sub>2</sub> Concentration and Compressive Strength of S-T Composite

#### 4.2.1 Mathematical model and verification

The results of 7 and 28 day  $CO_2$  concentrations and compressive strengths for all 16 mix proportions is shown in Table 4.2.Predictive mathematical model is then developed as a function of sandcrete components via full factorial analysis generated by Design-Expert 11.0 (stat ease Inc. USA). The predictive model attributed to the four independent components and their interactions is depicted according to Equation (4.1).

$$CS(\gamma) = \alpha_0 + \alpha_1 A + \alpha_2 B + \alpha_3 C + \alpha_4 D + \alpha_5 A B + \alpha_6 A C + \alpha_7 A D + \alpha_8 B C + \alpha_9 B D + \alpha_{10} C D + \alpha_{11} A B C + \alpha_{12} A B D + \alpha_{13} A C D + \alpha_{14} B C D + \alpha_{15} A B C D$$
(4.1)

Where  $\gamma$  = optimized CO<sub>2</sub> concentration or compressive strength (N/mm<sup>2</sup>)

 $\alpha_0$  = model constant

 $\alpha_1 - \alpha_{15}$  = coefficient associated with variable A, B, C and D

Standard order	Run	CO <sub>2</sub> concentration (mol/dm <sup>3</sup> )	Compressive strength (N/mm <sup>2</sup> )
13	1	0.923	7.43
12	2	0.298	2.31
6	3	0.895	7.54
7	4	0.756	7.59
1	5	0.768	7.62
8	6	0.278	3.21
4	7	0.267	2.98
11	8	0.213	2.43
5	9	0.456	7.31
16	10	0.205	2.43
9	11	0.257	3.16
3	12	0.447	6.43
14	13	0.458	6.53
2	14	0.209	2.12
10	15	0.452	6.75
15	16	0.211	2.22

 Table 4.2: Experimental Compressive Strength at Varying Age

The predictive model is validated by checking the analysis of variance (ANOVA) and the model terms were accepted or rejected based on p-values with 95% confidence level. The model is used to confirm the effect of the various components and the degree of significance on  $CO_2$  concentration and compressive strength of sandcrete-talc composite. The model is significant in view of the acceptable p-and F-values coupled with high correlation coefficients ( $R^2$ , adjusted  $R^2$  and predicted  $R^2$ ).

Figures 4.11 and 4.12 showed the degree of significant of the main components and their interaction matrices on  $CO_2$  concentration and compressive strength of sandcrete-talc composite at different p-values. The results show that Talc (B), Age (C) and their interaction (BC) are the most important components that have great effect on the concentration of  $CO_2$  sequestered, while Sand (A), Talc (B) and Age (C) have significant effect on the compressive strength. The results also showed that components B and C contribute 87.27% while A, B and C contributes 99.34% to the degree of  $CO_2$  adsorbed and the compressive strength of the S-T composite respectively. Other factors and interactions such as AB, AD, BD, ABD, ACD and ABCD were used in estimation of error in the analysis of variance hence did not appear in model equation.







Figure 4.12: Pareto Chart Showing the Effect of Sandcrete Components on Compressive Strength

To affirm the findings in Figures 4.11 and 4.12 on the effect of components and their interactions on carbonation and compressive strength ,analysis of variance was carried out. The analysis of variance (ANOVA) in Table 4.3 revealed that the compressive strength data obtained experimentally is best fitted into a quadratic model at 95% confidence level. The p-values less than 0.05 as depicted in Tables 4.3 and 4.4 implies that the linear regression model, coupled with components B, C and its interaction matrices BCwhile A, B and C are significant and have effect on the  $CO_2$  concentration and compressive strength respectively. This is corroborated by the calculated F-values shown in Tables 4.3 and 4.4.

The developed model equations for predicting the  $CO_2$  concentration and compressive strength of sandcrete-talc composite in terms of actual values are given in Equations 4.2 and 4.3 respectively.

 $CO_{2} Conc. (\gamma) = 0.161834 + CEMENT - 6.2E - 5 *SAND + 1.2819E - 2 *TALC + 3.824E - 3 *$ AGE -7.9E -5 \* SAND \*TALC + 7.74E - 4 \* TALC \* AGE (4.2)

Comp. Strength ( $\gamma$ ) =-1.39419+ 0.011811 × SAND + 0.010265 × TALC + 0.207972 × AGE -1.05E-5 × SAND × AGE + 4.12E-4 × TALC × AGE + 5.1214E-6 × SAND × TALC × AGE (4.3)

Table 4.4 shows the regression analysis with model coefficient terms. The result indicates that compressive strength increases with age and decreased as LDPE pellet was added with decreasing sand content in the composite. This finding is corroborated by Al-Manaseer and Dalal (1997). In addition, the interaction between sand, cement and LDPE pellet showed negative effect on the sandcrete composite. This could be attributed to poor bond characteristics between the components (Thorneycroft *et al.*, 2016). The mass

compactness observed could be attributed to the formation of calcium-silicate hydrate gel in the composite matrix as cement reacts with water.

Source	Sum of	df	Mean	<b>F-value</b>	p-value
	squares		square		
Model	0.9544	8	0.1193	51.08	< 0.0001
<b>B-TALC</b>	0.2005	1	0.2005	85.84	< 0.0001
C-AGE	0.6468	1	0.6468	276.94	< 0.0001
D-CEMENT	0.0000	1	0.0000	0.0195	0.8929
AB	0.0066	1	0.0066	2.83	0.1366
AD	0.0000	1	0.0000	0.0060	0.9403
BC	0.1003	1	0.1003	42.96	0.0003
CD	0.0001	1	0.0001	0.0328	0.8615
BCD	0.0000	1	0.0000	0.0077	0.9324
Residual	0.0163	7	0.0023		
Cor Total	0.9707	15			

 Table 4.3: Effect of Components on CO<sub>2</sub>Sequestration

Source	Sum of	df	Mean	<b>F-value</b>	p-value
	squares		square		
Model	85.26	9	9.47	159.81	<0.0001 Significant
A-SAND	0.5402	1	0.5402	9.11	0.0234
<b>B-TALC</b>	1.97	1	1.97	33.30	0.0012
C-AGE	82.54	1	82.54	1392.35	<0.0001
D-CEMENT	0.0900	1	0.0900	1.52	0.2640
AD	0.0004	1	0.0004	0.0067	0.9372
BC	0.0306	1	0.0306	0.5166	0.4993
BD	0.0004	1	0.0004	0.0067	0.9372
ACD	0.0784	1	0.0784	1.32	0.2939
ABCD	0,0100	1	0.0100	0.1687	0.6955
Residual	0.3557	6	0.0593		
Cor Total	85.62	15			

# Table 4.4: Effect of Components on the Compressive Strength

# 4.2.2 Model validation

To validate the developed model, experimental data produced in Table 4.2 were inserted into Equations 4.2 and 4.3 and the predictive  $CO_2$  concentrations and compressive strengths associated with each run were obtained as depicted in Tables 4.5 and 4.6 respectively.

Table 4.5: Comparative Analysis of Experimental and Predictive CO2Concentrationof Sandcrete-Talc Composite

Run Order	Actual value	Predicted value	Residual	Standard order
	(mol/dm <sup>3</sup> )	(mol/dm <sup>3</sup> )	(mol/dm <sup>3</sup> )	(mol/dm <sup>3</sup> )
1	0.9230	0.8598	0.0632	15
2	0.2980	0.2978	0.0002	3
3	0.8950	0.8518	0.0432	7
4	0.7560	0.8212	-0.0651	8
5	0.7680	0.8093	-0.0413	16
6	0.2780	0.2541	0.0239	12
7	0.2670	0.2928	-0.0258	11
8	0.2130	0.2313	-0.0183	10
9	0.4560	0.4773	-0.0213	14
10	0.2050	0.2283	-0.0233	2
11	0.2570	0.2553	0.0017	4
12	0.4470	0.4311	0.0159	5
13	0.4580	0.4348	0.0232	13
14	0.2090	0.1888	0.0202	9
15	0.4520	0.4698	-0.0178	6
16	0.2110	0.1896	0.0214	1

Table 4.6: Comparative Analysis of Experimental and Predictive CompressiveStrength of Sandcrete-Talc Composite

Run Order	Actual value	Predicted value	Residual	Standard order
	(N/mm <sup>2</sup> )	( <b>N/mm<sup>2</sup></b> )	(N/mm <sup>2</sup> )	( <b>N/mm</b> <sup>2</sup> )
1	7.43	7.34	0.00887	15
2	2.31	2.56	-0.2512	3
3	7.54	7.38	0.1587	7
4	7.59	7.55	0.0412	8
5	7.62	7.91	-0.2888	16
6	3.21	3.09	0.1213	12
7	2.98	2.90	0.0788	11
8	2.43	2.51	-0.0837	10
9	7.31	7.06	0.2512	14
10	2.43	2.45	-0.0237	2
11	3.16	3.11	0.0513	4
12	6.43	6.55	-0.1213	5
13	6.53	6.59	-0.0613	13
14	2.12	2.23	-0.1062	9
15	6.75	6.82	-0.0687	6
16	2.22	2.01	0.2138	1

The developed models show that the maximum CO<sub>2</sub> concentration and compressive strength after 28-day curing in ambient condition obtained were 0.289 mol/m<sup>3</sup> and 3.506 N/mm<sup>2</sup> respectivelyat 95% confidence level with the following conditions:198.357g of sand, 12.378 after 11.45-days (Figures 4.13 and 4.14). Analysis of the statistics of goodness of fit was carried out. The analysis showed that the correlation coefficient  $R^2$  was 0.9553 which indicates a highly positive correlation between the experimental and predicted compressive strengths. Also the predicted  $R^2$  obtained was 0.7663 which is in reasonable agreement with the adjusted  $R^2$  of 0.9042.



Figure 4.13: Optimized CO<sub>2</sub> concentration at Varying Sandcrete-talc Components



Figure 4.14: Optimized Compressive strength at Varying Sandcrete-talc Components

#### **4.3** Optimization of Sandcrete Components (Desirability Optimization Approach)

Numerical optimization method utilizing desirability function used by Kharazi et al. (2013) was adopted. The method involves the transformation of individual responses into desirability function that ranged from  $0 \ll di \ll 1$ . If di = 1, the response is in its acceptable range, however, if di =0, the response is outside its acceptable range. The individual desirability functions could be defined as: minimum, maximum, target, in range and equal to, depending on the response objective. The software (Design Expert) can also be use to set the lower and upper limit of each sandcrete components. Figure 4.15 shows that the components and their combinations have a desirability equal to 1 indicating that the concentration of CO<sub>2</sub> sequestered from the atmosphere and compressive strength (response) are within the acceptable range hence validating the developed models. The sandcrete components that was selected to maximize desirability (D = 1) has the

following proportion: sand = 198.357 g, talc = 12.378 g cement = 33.70 g , Age = 11.447day and w/c =0.5. The slight variations in  $CO_2$  concentrations and compressive strengths shown in Table 4.5 could be attributed to temperature and humidity variation during curing stage (Kharazi *et al.*, 2013; Abdullahi *et al.*, 2016).



**Figure 4.15: Desirability optimization of sandcrete components** 

#### **CHAPTER FIVE**

### 5.0 CONCLUSION AND RECOMMENDATIONS

# 5.1 Conclusion

The starting materials and composite produced were characterised using the following analytical techniques, XRF, XRD and SEM. XRF analysis revealed that Silica and it oxide as the main constituent for sand and talc particle. The crystalline peak from the XRD pattern depict Magnesium Silicate as the predominant oxide for talc and Silica for cement and sand, while SEM illustrate morphological feature of the samples. The mathematical models for the determination of CO<sub>2</sub> concentration and compressive strength sandcrete-talc (S-T) composite was developed and optimized by application of 2level factorial design. The interactive effects Age (7 - 28-day), Talc (5 - 15%), cement and sand with constant w/c ratio of 0.5 were investigated. The statistical analysis (ANOVA) showed that Age and talc have great effect on the degree of CO<sub>2</sub> sequestered from the atmosphere at ambient condition, while age, talc and sand have significant effect on the compressive strength of the sandcrete-talc composite. The result showed that as curing age increases with increase in talc content, the concentration of carbon dioxide increases. Also, the compressive strength increased as the age, talc and sand components increase at constant cement composition. The developed predicted model showed that the optimal concentration of  $CO_2$  and compressive strength obtained were 0.289mol/m<sup>3</sup> and 3.506N/mm<sup>2</sup> after 28-day curing at ambient condition. The developed predicted equations could be used in the design of S-T mix composite for structural buildings and to minimize the effect of greenhouse gas.
### 5.2 **Recommendations**

The following recommendations could be made for further clarification and better understanding of  $CO_2$  sequestration using metallic mineral such as talc:

- 1. Further work should be carried out to investigate the adsorption of CO<sub>2</sub>utilizing different talc samples obtained from different geo-political zones.
- 2. Optimizing this technology, field studied should be carried to better characterize the rates of  $CO_2$  adsorption and strength development at different technological conditions.
- 3. Finally, I recommend blended BAC for pharmaceutical industries and other related industries for the treatment of effluent before discharge as it is relatively economical and effective.

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#### APPENDIX

# DETERMINATION OF CARBON DIOXIDE CONCENTRATION ( $C_{CO2}$ ) USING TITRIMETRIC ANALYSIS AT AMBIENT CONDITIONS

### EXPERIMENTAL RUN 1

Mass of pulverised sandcrete Mp = 157gm Mp = 0.157kg

Average Titre value  $V_A = 12.735 \text{ cm}^3$ 

Molar Concentration of base  $C_B = 0.05 \text{ mol/LMolar mass}$   $M_{HCl} = 36.4607 \text{gm/mol}$ 

Volume of base  $V_B = 25 \text{ cm}^3$ 

 $\frac{CAVA}{CBVB} = \frac{na}{nb}$ 

 $NaOH + HCl \rightarrow NaCl + H_2O$ 

1

$$C_{A} = \frac{CB.VB.na}{VA.nb}$$

1

$$C_A = 0.098 \text{ mol/L}$$

Concentration of HCl in g/dm3  $M_{HCl} = Molar mass_{HCl} \cdot C_A$ 

 $M_{HCl} = 3.57879 \text{ m}^{-3}.\text{kg}$ 

Mass of HCl reacted with NaOH  $M_{HCl} = 3.579 \text{ m}^{-3}$ .kg

Since 0.1M of HCl = 3.64607 g/L

Mass of HCl that reacted with pulvrised sandcrete  $m_{HCl} = 3.64607 \text{ g/L} - M_{HCl}$ 

$$m_{HCl} = 0.067 \text{ kg/m}^3$$

To determine the mass concentration of CaCO<sub>3</sub> that reacted in the pulverised sandcrete

$$CaCO3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2$$

1 2 1

 $MM_{HCl} = 72.9214 \text{ gm/mol}$   $MM_{CO2} = 44.0098 \text{ gm/mol}$   $MM_{CaCO3} = 100.0872 \text{ gm/mol}$ 

### Mass of calcite in pulverised sandcrete

$$m_{calcite} = \frac{mHCl.MMCaCO3}{MMHCl}$$

 $m_{calcite}=0.092 \ kg/m^3$ 

### Mass of carbon dioxide absorbed

$$m_{co2} = \frac{mHCl.MMCO2}{MMHCl}$$
$$m_{co2} = 0.041 \text{ kg/m}^3$$
$$C_{CO2} = \frac{mco2}{MMCO2}$$
$$C_{CO2} = 0.923 \text{ mol/m}^3.$$

## DETERMINATION OF CARBON DIOXIDE CONCENTRATION ( $C_{CO2}$ ) USING TITRIMETRIC ANALYSIS AT AMBIENT CONDITIONS

### **EXPERIMENTAL RUN 2**

Mass of pulverised sandcrete Mp = 157 gm Mp = 0.157 kgAverage Titre Value  $V_A = 12.575 \text{ cm}^3$ Molar concentration of base  $C_B = 0.05 \text{ mol/L}$ Molar mass<sub>HCl</sub> = 36.5 gm/mol Volume of base  $V_B = 25 \text{ cm}^3$  $\frac{CA.VA}{CB.VB} = \frac{na}{nb}$  $NaOH+HC1 \rightarrow NaC1 + H_2O$ 1 1  $C_A = \frac{CB.VB.na}{VA.nb}$  $C_A\ = 0.099\ mol/L$ concentration of HCl in  $g/dm^3$  M<sub>HCl</sub> = Molar mass<sub>HCl</sub>. C<sub>A</sub>  $M_{HCl} = 3.62823 \text{ m}^{-3}.\text{kg}$ Mass of HCl reacted with NaOH  $M_{HCl} = 3.62823 \text{ m}^{-3} \text{.kg}$ Since 0.1M of HCl = 3.64607 g/LMass of HCl that reacted with pulverised sandcrete  $m_{HCl} = 3.65 \text{ gm/L} - M_{HCl}$ 

 $m_{HCl} = 0.022 \text{ m}^{-3}$ . Kg

To determine the mass concentration of CaCO<sub>3</sub> that reacted in the pulverised sandcrete CaCO<sub>3</sub> + 2HCl  $\rightarrow$  CaCl<sub>2</sub> + H<sub>2</sub>O + CO<sub>2</sub> 1 2 1 MM<sub>HCl</sub> = 73 gm/mol MM<sub>CaCO3</sub> = 100 gm/mol

 $MM_{CO2} = 44 \text{ gm/mol}$ 

Mass of calcite in the pulverised sandcrete

$$m_{calcite} = \frac{mHCl \cdot MMCaCaCO3}{MMHCl}$$

 $m_{calcite}\ =\ 0.03\ kg/m^3$ 

Mass of Carbon dioxide absorbed

$$m_{co2} = \frac{mHCl.MMCO2}{MMHCl}$$

$$m_{co2} = 0.013 \text{ kg}/\text{m}^3$$

$$C_{CO2} = \frac{mCO2}{MMCO2}$$

 $C_{CO2} = 0.298 \text{ mol/m}^3$ .

## DETERMINATION OF CARBON DIOXIDE CONCENTRATION ( $C_{CO2}$ ) USING TITRIMETRIC ANALYSIS AT AMBIENT CONDITIONS

### **EXPERIMENTAL RUN 3**

Mass of pulverised sandcrete Mp = 157 gm Mp = 0.157 kgAverage Titre Value  $V_A = 12.728 \text{ cm}^3$ Molar concentration of base  $C_B = 0.05 \text{ mol/L}$ Molar mass<sub>HCl</sub> = 36.5 gm/mol Volume of base  $V_B = 25 \text{ cm}^3$  $\frac{CA.VA}{CB.VB} = \frac{na}{nb}$  $NaOH+HC1 \rightarrow NaC1 + H_2O$ 1 1  $C_A = \frac{CB.VB.na}{VA.nb}$  $C_{A} = 0.098 \text{ mol/L}$ Concentration of HCl in  $g/dm^3$  M<sub>HCl</sub> = Molar mass<sub>HCl</sub> . C<sub>A</sub>  $M_{HC1} = 3.58462 \text{ m}^{-3} \text{ .kg}$  $M_{HCl} = 3.585 \text{ m}^{-3}.\text{kg}$ Mass of HCl reacted with NaOH Since 0.1M of HCl = 3.64607 g/LMass of HCl that reacted with pulverised sandcrete  $m_{HCl} = 3.65 \text{ gm/L} - M_{HCl}$  $m_{HCl} = 0.065 \text{ m}^{-3}$ . Kg

To determine the mass concentration of CaCO<sub>3</sub> that reacted in the pulverised sandcrete  $CaCO_3 + 2HC1 \rightarrow CaCl_2 + H_2O + CO_2$ 1 2 1

 $MM_{HCl} = 73 \text{ gm/mol}$ 

 $MM_{CaCO3}\ =\ 100\ gm/mol$ 

 $MM_{CO2}\ =\ 44\ gm/mol$ 

Mass of calcite in the pulverised sandcrete

$$m_{\text{calcite}} = \frac{mHCl \cdot MMCaCaCO3}{MMHCl}$$

 $m_{calcite}~=~0.09~kg/m^3$ 

Mass of Carbon dioxide absorbed

$$m_{co2} = \frac{mHCl.MMCO2}{MMHCl}$$

$$m_{co2} = 0.039 \text{ kg} / \text{m}^3$$

$$C_{CO2} = \frac{mCO2}{MMCO2}$$

 $C_{CO2} = 0.896 \text{ mol/m}^3$ .

## DETERMINATION OF CARBON DIOXIDE CONCENTRATION ( $C_{CO2}$ ) USING TITRIMETRIC ANALYSIS AT AMBIENT CONDITIONS

### **EXPERIMENTAL RUN 4**

Mass of pulverised sandcrete Mp = 157 gm Mp = 0.157 kgAverage Titre Value  $V_A = 12.692 \text{cm}^3$ Molar concentration of base  $C_B = 0.05 \text{ mol/L}$ Molar mass<sub>HCl</sub> = 36.5 gm/mol Volume of base  $V_B = 25 \text{ cm}^3$  $\frac{CA.VA}{CB.VB} = \frac{na}{nb}$  $NaOH+HC1 \rightarrow NaC1 + H_2O$ 1 1  $C_A = \frac{CB.VB.na}{VA\,nb}$  $C_A = 0.098 \text{ mol/L}$ Concentration of HCl in  $g/dm^3$  M<sub>HCl</sub> = Molar mass<sub>HCl</sub>. C<sub>A</sub>  $M_{HCl} = 3.59478 \text{ m}^{-3} \text{ .kg}$  $M_{HCl} = 3.595 \text{ m}^{-3}.\text{kg}$ Mass of HCl reacted with NaOH Since 0.1M of HCl = 3.65 g/LMass of HCl that reacted with pulverised sandcrete  $m_{HCl} = 3.65 \text{ gm/L} - M_{HCl}$  $m_{HCl} = 0.055 \text{ m}^{-3}$ . Kg

To determine the mass concentration of CaCO<sub>3</sub> that reacted in the pulverised sandcrete CaCO<sub>3</sub> + 2HCl  $\rightarrow$  CaCl<sub>2</sub> + H<sub>2</sub>O + CO<sub>2</sub>

 $MM_{HCl} = 73 \text{ gm/mol}$ 

 $MM_{CaCO3} = 100 \text{ gm/mol}$ 

 $MM_{CO2}\ =\ 44\ gm/mol$ 

Mass of calcite in the pulverised sandcrete

$$m_{\text{calcite}} = \frac{mHCl \cdot MMCaCaCO3}{MMHCl}$$

 $m_{calcite}\ =\ 0.076\ kg/m^3$ 

Mass of Carbon dioxide absorbed

$$m_{co2} = \frac{mHCl.MMCO2}{MMHCl}$$

$$m_{co2} = 0.033 \text{ kg}/\text{m}^3$$

$$C_{CO2} = \frac{mCO2}{MMCO2}$$

 $C_{CO2} = 0.756 \text{ mol/m}^3$ .