PRODUCTION AND CHARACTERIZATION OF METAKAOLIN AS AN ADMIXTURE FOR IMPROVING COMPRESSIVE STRENGTH OF PORTLAND CEMENT IN BUILDING CONSTRUCTION

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

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DECLARATION

I hereby declare that this Thesis titled: “Production and characterization of metakaolin as an admixture for improving compressive strength of Portland cement in building construction” is a collection of my original research work and it has not been presented for any other qualification anywhere. Information from other sources (published or unpublished) has been duly acknowledged.

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CERTIFICATION

The Thesis titled: “Production and characterization of metakaolin as an admixture for improving compressive strength of Portland cement in building construction” by: AYUBA, Bijimi Caleb (MEng/SIPET/2018/8286) meets the regulations governing the award of the degree of Master of Engineering of the Federal University of Technology, Minna and it is approved for its contributions to scientific knowledge and literary presentation.

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DEDICATION

This research project is dedicated to Almighty God, my family and Mr. Timothy Yandang

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ABSTRACT

Kaolin is a precursor of metakaolin, rich in kaolinite, readily available and abundant. The production and characterization of metakaolin from kaolin deposit were carried out.

Metakaolin was successfully produced at dehydroxylation temperature of 550 C as confirmed by the X-Ray diffraction analysis. The physiochemical properties of kaolin and metakaolin were studied by X-ray fluorescence (XRF), thermogravimetry analysis (TGA) and X-ray diffraction (XRD) analysis. The properties of cements containing metakaolin were monitored and water cured. Metakaolin derived from indigenous Ahoko and Aloji Kaolin were used and Cement mortars alongside concretes with 0 %, 5 %, 10 %, 12.5 % ,15%, 20%,30% and 35% metakaolin's were studied. Water demand and setting time were determined in all samples. The cement containing metakaolin has an average percentage water demand of 26.5% compared to Portland cement with percentage water demand of 25%. Metakaolin cement has significantly more water demand than the relatively pure cement. Considering the compressive strength of metakaolin cements, a 10 % metakaolin content for both samples, seems to be generally, more favorable than the other mixed fractions considered. It was estimated from the material balance that 1276 kg/batch of raw kaolin is required to produce 1000 kg/batch of metakaolin with a five (5) batches operation per day to achieve 5 tons of metakaolin product per day. From the energy balance, 5486688.546 kJ/batch is required to achieve 95 % dehydroxylation of kaolin to metakaolin at 550 ^{o}C. The design of the calcination furnace was done successfully at a favorable condition and specifications of rotary furnace, material of construction- mild steel, Batch time-2hrs, batch operation, reactor type – rotary kiln, reactor volume per batch, length, diameter, thickness is 1.36458 L, 1380 mm, 300 mm,180 mm respectively. Minimum and maximum operating temperature of furnace is 500 ^{o}C and 900 ^{o}C respectively, for the start and end of dehydroxylation reaction.

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LIST OF ABBREVIATION

	Nomenclature	Unit
θ	Braggs diffraction angle	(^{o})
MK	Metakaolin	
K_1	Ahoko kaolin	
K_2	Aloji Kaolin	
XRF	X- ray Fluorecence	
XRD	X-ray Diffraction	
Å	Angstrom	0.1nm
TGA	Thermogravimetric Analysis	
V	Volume of furnace	(L)
m	Mass of kaolin/ Metakaolin	(kg)
t	Time	(minutes)
SCM	Supplementary Cementing Material	
PC	Portland Cement	

E	Energy	kJ
T	temperature	(K)

SEM Scanning Electron Microscopy

XRD X-Ray Diffraction
BET Brunaur-Emmett-Teller

GHG Greenhouse gas
C_3A tricalcium aluminate

C_4AF tetra calcium aluminoferrite
C_3S tricalcium silicate

C-S-H Secondary calcium Silicate hydrate CH

Calcium hydroxide.

FA Fly ash
SF Silica fume
GGBFS Ground granulated blast furnace slag

W/C: Water to cement ratio

PFD: Production Flow Diagram

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CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

Concrete is a building component needed to meet the demand for dwellings. Concrete is adaptable and performs well when put to use. The main raw ingredients are readily available, and compared to alternatives, it uses relatively little energy and has very little effect on the environment. However, cement is the main raw material which is regularly disproportionately costly in developing countries because of the significant energy consumption (Chakraborty, 2003).

To ascertain the concrete's flexural tensile strength and compressive ability, Aqeel, (2009), evaluated the effects of various additives, such as wood sawdust, rice husk, and silica gel, added to the mixture at various percentages. At 5 percent of adding materials, he found that the addition of these materials significantly changes the concrete's compressive as well as and flexural strength. In addition, when using composites made of husks from rice or iron splinter, additives are added to the concrete to produce less dense mixtures or extremely dense concrete, respectively (Garas and Kurtis, 2008).

The bulk of publications discussing the inclusion of metakaolin address strength in some way. In order to generate concretes with improved strengths at ages up to 365 days, Rashad, (2013) used Type I cement with metakaolin contents of 5 and 10 percent, respectively, with a weight per cubic meter of 0.40. These samples revealed strengths that were 10 to 15 percent higher on average than concrete that had silica fume added in the same amount. The specimens made with 5 percent Metakaolin demonstrated the highest strength of the group after 365 days. And when comparing sustainable building materials, he found that concrete is the most frequently made and used artificial material. Cement, which is a primary component of concrete, produces close to 8 percent of all anthropogenic CO_2 emissions in the world. These take place as a result of the high temperatures used during production and the reduction in carbon dioxide of limestone, which result in the creation of clinker, a key component of cement.

Fly ash and slag from blast furnaces, which are respective waste products of coal power stations and the iron sector, are the next two most commonly used SCMs. Even while they have an important influence on minimizing the ecological footprint nowadays, they only account for 15 percent of the cement that is now generated, and practically all of the sources are currently employed in cement or are eventually blended into concrete. Furthermore, as we stop burning coal and more steel is recycled, this quantity is probably going to go down in the future. However, there is significant potential for large-scale decreases in CO_2 through greater utilization of clays, which are readily available around the world and may produce a highly reactive supplemental cementitious substance when heated at a slightly lower temperature (Alujas et al., 2015).

The reduction of carbon dioxide is essentially taking place when baked clay (metakaolin) is added to Portland cement having high performance levels (Antoni et al., 2012). In order to achieve no emissions at all and achieve neutrality for climate change, decarbonization means the technique of lowering or finally eliminating the release of carbon dioxide from human operations in the atmosphere through SCM. In order to address the issue of climate change, it is predicted that by the year 2050, the global temperature will drop to less than 20 degrees Celsius, or 1.50C. Moreover, cement can reduce global CO_2 emissions by 15 to 30 percent (Gartner and Sui, 2018).

Human-related greenhouse gas emissions are to blame for both the increase in temperature and a changing climate. In order to slow the effects of climate change, these greenhouse gases must be decreased. Technology allows for significant cuts in greenhouse gas emissions worldwide during the decade that follows, and reaching net-zero world emissions by Midcentury would have a significant impact on subsequent climate change. Achieving zero emissions entails not releasing any greenhouse gases, into the atmosphere. In order to achieve net-zero emissions, some greenhouse gases must still be produced, but these must be balanced out by taking an equivalent amount from the earth's atmosphere and keeping it in soil, plants, or other forms of matter (Gartner and Sui, 2018).

According to Mohammed, (2017), clay goes through three stages of thermal behaviour, including dehydration, dehydroxylation, and recrystallization. According to Kakali et al., (2001), the dehydration stage ended around 100 ^{o}C, however Mohammed, (2017) stated that it might go up to 200 ^{o}C. Depending on the clay structure and minerals present, various degrees of temperature are required to complete the dehydroxylation and recrystallization processes. Unbound water molecule escape during the process of dehydration stage, but the molecular makeup of the clay remains unchanged. Around 400 ^{o}C and 600 ^{o}C, the dehydroxylation process of kaolinite takes place during this stage. To create metakaolin, covalently bound water must be removed. Dyhydroxylation reaction involves the removal of molecules of hydroxyl from the kaolin framework, which causes the basal spacing to break down and the crystallinity to disappear (Mohammed, 2017).

lately the microscopic makeup of metakaolin was determined using computational simulation approaches, and it was said to have advanced significantly. By gradually removing the hydroxyl molecules from a kaolinite system and applying the initial fundamental strategy of the density functional theory, the metakaolin composition was obtained. The outcome showed that the octagonal layer displayed significant structural change due to the depletion of 87.5 percent of the water content, which resulted to a decrease in crystalline characteristic. Nevertheless, there are certain issues with the created metakaolin composition that must be resolved. Since the model cell only has 272 atoms, it is unlikely that the metakaolin's extended instability configuration is going to be activated (White et al., 2010).

Many scholars have examined heat dehydroxylation of kaolinite, examining a mathematical an overview of the noticed rate of response, dehydroxylation of kaolinite via molecular dynamic research, related issue of the structural changes taking place throughout its conversion to Meta kaolinite, pozzolanic behaviour of metakaolin, and the impact on concrete and cement's properties (Begbroke, 2020). Although the depth of comprehension is adequate, only a handful of scholars examine the initial design simulation element of the metakaolin pilot plant.

This research work focus on, identifying an indigenous kaolin clay in Northern part of Nigeria, Kogi state, that has the ability to function as additional cementitious ingredients in concrete; investigate their dehydroxylaion behaviour experimentally, evaluate their pozzolanic activity and therefore, establish a preliminary design of pilot plant that will able to produce 5 tons per day of metakaolin.

1.2 Statement of the Research Problem

Cement represents the core and primary ingredient used in the creation of concrete, a material for construction used to fill housing needs as well as to fill abandoned oil drilling sites. However, using Portland cement (PC) in concrete has a considerable impact on greenhouse gas emissions, during the Portland cement production process and composition of $CaCO_3$ (Gartner and Sui, 2018).

Per 1kg Portland cement production, the CO_2 emission from limestone decomposition is 0.2938kg (Feng et al., 2016). This pollutes the environment and also lead to climate change. Hence, by partially addition of supplemental cemented ingredients to Portland cement, carbon dioxide emission in concrete can be decreased (Justice, 2005).

1.3 Aim and Objectives of the Study

This study's primary goal is to produce metakaolin and analyse the dehydroxylation behaviour of the metakaolin precursor, namely: Ahoko, and Aloji kaolin through experiments; evaluate their pozzolanic activity as an additive for improving compressive strength of Portland cement.

The objectives of this project are to:

1. Collect, purify and characterize the two different Nigerian Kaolin deposits, to know their structural, Physio-chemical and morphological properties.
2. Carry thermal studies via Thermogravimetric studies of the refined Kaolin deposit to determine the beginning and the ending of the dehydroxylation reaction
3. Determine the optimum percentage of metakaolin to replace cement in the formation of mortar and concrete

4. Develop flowsheet for metakaolin production; carryout material and energy balance across the flow sheet for the design Meta kaolinization plant.
5. Perform detailed design of the furnace/ calciner component of the plant to perform 5 ton per day of Meta kaolinization
6. Carry out the actual fabrication of the pilot plant calciner and perform testing and characterization of the metakaolin produce.

1.4 Justification of the Study

(1) Kaolin is a clay mineral, readily available and abundant.

(2) Owing to the unique properties of metakaolin, it is one of supplementary cementing material with good pozzolanic reactivity, lower influence on the environment and can be produced on lower expenditures.

(3) Metakaolin and Portland cement mixtures will help in decarbonization of environment starting from Nigeria

(4) The outcome of this study will exploit Ahoko and Aloji kaolin in concrete technology.

1.5 Scope of Work

This research wok is narrowed to Production and characterization of metakaolin as an admixture for improving compressive strength of Portland cement in building construction.

CHAPTER TWO

2.0 LITERATURE REVIEW

Concrete is the construction material having the greatest prospect for satisfying the rising housing demand in advanced countries. With a total of two billion tonnes utilized globally every year, concrete is one of the most common building materials in the globe (Mindess et al., 2003). Concrete can typically be made from materials that are easily obtainable nearby and moulded into a wide range of structural forms. There is pressure to minimize the use of cement through the use of supplemental materials as a result of CO_2 emissions linked to cement manufacturing's contribution to environmental degradation (Sabir et al., 2001).

2.1. Portland Cement Concrete

Portland cement comprises Portland cement clinker and gypsum. Portland cement clinker is made up of calcium silicates and calcium aluminates. During the last grinding process of Portland cement, gypsum is introduced in order to control the setting time. The chemical mixture of Ca, Si, Al, and Fe is very similar to gypsum. Portland cement is created by grinding Portland cement clinker with calcium sulfate (Lord and Syncrude, 2003).

According to Mindness et al. (2003), Portland cement is made by burning materials like lime, iron, silica, and alumina to temperatures between 1250 °C to 1400 °C in a rotary kiln while the clinker is then ground into a finely ground powder. The combustion that takes place in the kiln results in the formation of new chemical substances. The mass proportion, make-up, and temperature of the raw materials used to make lime, iron, silica, and alumina are what determine the cement's chemical makeup.

Lehne and Preston (2018) reported that concrete is the most practical construction material in the world and has been used for generations. In 2018, about 4 % of the carbon dioxide released in the globe comes from the production of 4 billion tonnes of cement yearly. The majority of concrete is made of cement, fine and coarse aggregate. The main component known as clinker is manufactured by high temperature (1250 - 1400 ^{o}C), fuel fire of clay, limestone, silica, aluminium, and iron oxides.

According to Mindness et al. (2003), Portland cement is a powder made up of gypsum and that has been finely pulverized. Alite, also known as C_3A or tricalcium silicate, and belite, also recognized as calcium silicate and C_2S, make up the majority of the four crystalline phases that make up clinker. Tetra calcium alumino ferrite, also known as C_4AF, and tricalcium aluminate, often known as C_3A, make up the other one-third of clinker. Low concentrations of metal oxides and impurities minerals are also present.

During cement hydration, there are a lot of stable and metastable layers created. The main binding gel phase that contributes to cement strength is the weekly crystalline calcium silicate

During cement hydration, there are a lot of stable and metastable layers created. The main binding gel phase that contributes to cement strength is the weakly crystalline calcium silicate hydrate gel. Portlandite is gotten in sufficient amount, during the formation of CSH and is does contribute to the strength of cement (Mindess et al., 2003).

2.1.1. Strength gain in portland concrete

The hydration of belite and elite causes the creation of secondary calcium silicate hydrates in Portland cement. Calcium silicate hydrate production is what leads to the gain in overtime strength (Taylor, 2014).

$$2Ca_3SiO_5 + 6H_2O \rightarrow 3CaO.2SiO_2.3H_2O + 3Ca\,(OH)_2 \qquad (2.1)$$

$$2C_2S + 4H_2O \rightarrow C_3S_2H_3 + CH \qquad (2.2)$$

Calcium hydroxide is a by- product form, from the hydration of elite and belite. The interfacial transition zone is a fragile region. The porous nature of the concrete paste in this area makes it easier for harmful barriers to penetrate (Hassan et al., 2000).

2.2 Supplementary Cementitious Materials (SCMs)

A pozzolanic substance known as supplementary cementing materials possesses significant quantities of silica and, in few instances alumina. it combines with calcium hydroxide from Portland cement hydration to yield calcium silicate hydrates. Portland cement often contains additional cement ingredients such as fly ash, ground granulated blast furnace slag, natural pozzolan and metakaolin (Lord and Syncrude, 2003).

According to their chemical and physical properties, supplementary cementitious elements make specific contributions to the strength and durability of concrete. the hydration reaction of the supplementary cementing material improved performance attributes (Hassan et al., 2000).

Supplementary cementing substances can have pozzolanic reactivity. A silicious material in the presence of water reacts with calcium hydroxide to yield cementitious compound. Natural pozzolan are volcano as and diatomaceous earth. Fly ash is a non-combustible left over from burning coal in power plants. the effluent gases are used in the production of silicon metal and alloys. The cementious compound were produced by the reaction with water (Sabir et al., 2001).

Metakaolin is created by heating virgin kaolin (Lord and Syncrude, 2003).it is made from a naturally occurring mineral and is utilize in cementing. the size of the particles is transformed during the manufacturing of metakaolin, and its colour is adjusted (Brooks and Johari, 2001; Ding and Li, 2002).

Even though metakaolin is more expensive to manufacture than clinker, it is widely utilized by organization for a variety of products (Dill, 2016).

According to Curcio et al. (2008), Metakaolin can enhance a number of concrete qualities while using less cement.

2.3. Structure of Kaolin

The majority of the clay that is known as kaolin, is pure white or can be processed to white. To make it suitable for use in white goods, it undergoes beneficiation (Lefond, 1983).

Plate I: Raw kaolin clay Source: (Justice and Kurtis, 2007)

The most popular white clay is kaolin, which can be used as a raw material for the manufacturing of white ceramics and as a covering for paper. the pollutants that lower the value of the deposit are oxides and hydroxide. (Manning, 1995).

According to Manning (1995), the parameters of formation, the method of formation, the materials found in the original rock, the location of formation, and the particles size of both the kaolinite and the impurities are some of the variables that affect the contaminants found in kaolinite. the degree to which the kaolin can purified is determined by the particle size. there are many purification procedures that can be used to separate out the coarsest particles because they can contain less than 10 percent kaolinite.

Kingery et al. (1976) observed that, small particles and a plate-like shape can be found in clay minerals, which have an inactive layer structure. the particles freedom to migrate over one another resulted in physical features such as a slippery sensation and ease of breaking.

2.3.1 Kaolinite

The majority of the clay is made of kaolinite. The hydrated aluminium disilicate is known as $Al_2Si_20_5(OH)_4$. Other minerals, like quartz and muscovite also contain kaolin (Moulin, 2001). The aluminosilicate hydrate complex is made up of an alumina crystal and a Si-O crystal (Varga, 2007).

The structure of the kaolin is broken apart by a mineral created by collapsing the kaolinite structure and releasing the minerals -OH chain to form an amorphous structure called metakaolin the structure of the material is created by the shared oxygen in the twodimensional plane of the silica tetrahedron rings in kaolinite and other clay. the material is joined to a different structure (Souri et al., 2015).

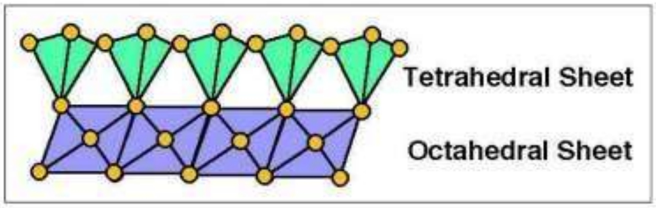


Figure 2.1: Kaolin Material Structure Source: (Souri et al., 2015).

kaolinite composition is ($Al_4(Si4O10)(OH)_8$), is:

- SiO_2 (46.54%)
- Al_2O_3 (39.5%)
- H_2O (13.96%)

(Weaver and Pollard, 1973).

A silia sheet is attached to layers of aluminum oxides or hydroxides in kaolinite particles. As a result, the silica sheet is composed of linked silicon atoms that are tetrahedrally coordinated with oxygen atoms, depicted below in Figure 2.1

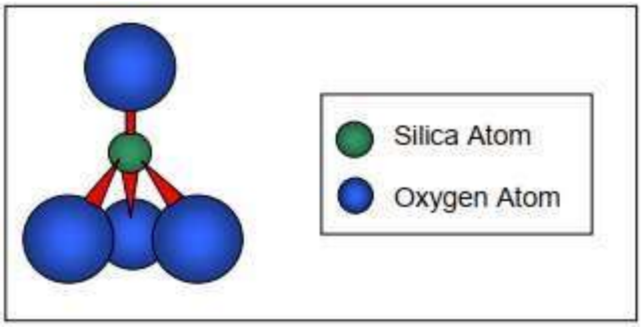


Figure 2.2: Silica Tetrahedral group Source:
(Justice and Kurtis, 2007).

Gibbsite layer is also refers to aluminium layer of kaolinite. A two-dimensional layer is subsequently created by the octahedral interconnection of the alumina molecules.

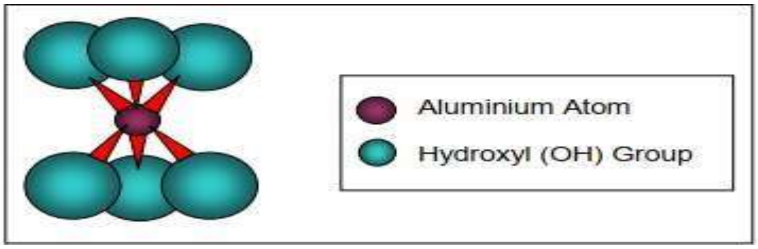


Figure 2.3: Octahedral alumina group Source:
(Justice and Kurtis, 2007).

The silica and alumina sheets come together to form a hexagonal strip that makes up the structure of kaolinite. Aluminium and silicon share two-thirds of their atoms in the layer that connects the octahedral and tetrahedral groups; as a result, they no longer form hydroxyl groups but rather become single oxygens. The arrangement of the atoms ensures that two aluminium atoms are separated by hydroxyl groups on both the top and bottom surfaces, with the other atoms remaining as hydroxyl groups. Therefore, the centre of the octahedral surface developed a hexagonal dispersion in only one plane (Justice and Kurtis, 2007).

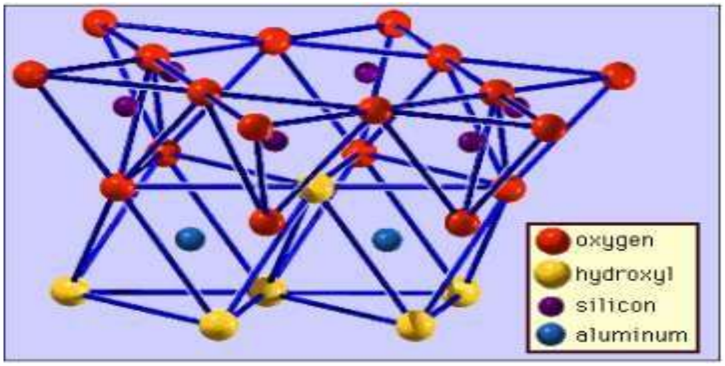


Figure. 2.4: kaolinite Structure

Three surfaces make up each kaolinite particle: an edge surface made up of aluminum, silicon, oxygen, and hydroxyl groups; an aluminum, silicon, oxygen, and hydroxyl layer; and a silicon, oxygen sheet.However, dickite and nacrite were also included in the kaolinite category of minerals, in addition to kaolinite. The three minerals are polymorphs, meaning their structures are different despite sharing a similar chemical makeup (Manning, 1995).

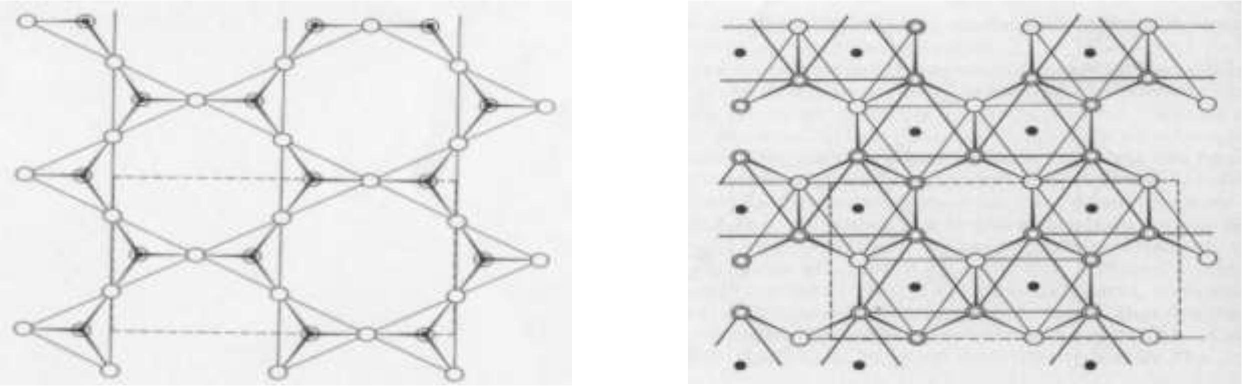


Figure 2.5: Collection of Atoms (a) Si_2O_5 and (b) $AlO(O)_2$ Sheets. Si(a) or Al(b); Oxygen;Hydroxyl
Source: (Justice and Kurtis, 2007).

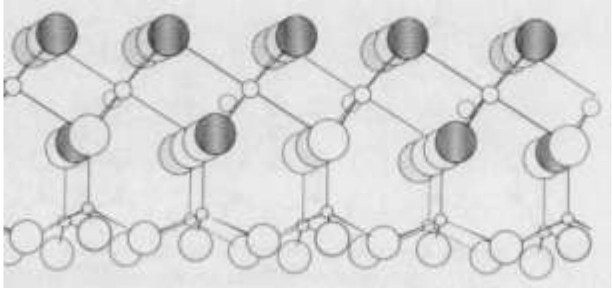


Figure 2.6: kaolinite with Si-O and Al-O Source:
(Justice and Kurtis, 2007).

Under typical ambient circumstances, kaolin is remarkably stable. When heated to a temperature of 650-900 ^oC, it loses 14 percent of its mass in bonded hydroxyl ions. By applying heat to the kaolin, calcination (heat treatment) breaks down the structure in a manner that causes the alumina and silica layers to contract and lose their long-term order. An extremely reactive transition phase known as Metakaolin is created as a result of the process of heat treatment (Bich et al., 2009).

2.3.2. Low-grade kaolin (LGK)

Low -grade kaolin can be found in tropically worn top-sequences and deposits. the manufacturing of bricks, building supplies, and constructing roads are all offered at extremely low prices in Nigeria (Dill., 2016).

Plate II: Low- grade kaolin (Brondon, 2020)
Source: (Brondon, 2020)

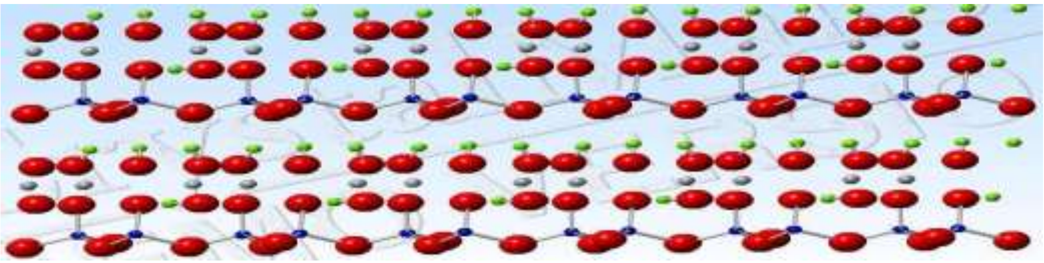
Plate III: High-grade Kaolin Source: (Brondon, 2020)

2.3.3. Kaolin structural disorder

The reactivity of the heated clay in the clay framework is affected by the proportion of order/disorder of the clay mineral (Tironi et al., 2012; Bich et al., 2009). In comparison to using a more crystalline clay with the same kaolin percentage, increased pozzolanic reactivity and compressive strength arise as the structural disorder of kaolin rises.

Despite the fact that Tironi et al. (2012) found that a higher kaolin level and structural disorder influence cementitious system strength obtained at later ages (3–7 days), the authors also found that structurally disordered kaolin particles possess significantly more surface area and porosity than those that are extremely crystalline.

Clay minerals like bentonites, palygorskites, and sepiolite have two silicate sheets connected to each other's face of an octahedral sheet, according to Aparicio et al. (2006). He continued by stating that the polymorph clay minerals Kaolinite, dickite, nacrite, halloysite, and live site are included in the kaolin group. While kaolinite, dickite, and nacrite are rarely encountered in natural deposits, they nonetheless form a "composite kaolin layer" that has twisted Si-O and Al-O bond lengths, flaws brought on by unoccupied cation locations, and is susceptible to layer translations with arbitrary variations.



Blue – Silicon, Silver – Aluminum, Red – Oxygen, Green – Hydrogen.

Figure 2.7: Stick model on Crystal Maker; kaolinite
Source: (Begbroke, 2020)

According to Brigatti et al. (2006) the different levels of disorder can be described in the form of a variety of faults or defects in the crystal structure. the characteristic explains for the well-known propensity of kaolin minerals to form an extensive array of regular and disordered polytypes. According to Brooks and Johari (2001) a small amount of dickite-like stacking in the lattice could be the cause of the disorder.

2.4. Metakaolin

Metakaolin is an additional cementing ingredient that is created by heating kaolin clay at a temperature between 550 to 900 degrees Celsius. The metakaolin might become less reactive once it gets above this temperature. It is a mineral additive that converts calcium hydroxide into cementitious materials (Michael, 2009).

Plate IV: Metakaolin sample

Georgia is known as the world's largest manufacturer of kaolin products. Georgia produces over eight million metric tons of kaolin each year and has a market worth more than $1 billion (Michael, 2009).

According to studies by Poon et al. (2006); Zhang and Malhotra (1995), the Jupia Dam in Brazil was the first place where metakaolin in concrete was used. It had been on the market for a long time. The negative minerals include Fe_2O_3, TiO_2, CaO and MgO.

Metakaolin is white. Since the processing is regulated, Metakaolin powders are uniform in appearance and function (Ding and Li, 2002).

2.4.1 Effect of metakaolin on compressive strength

Metakaolin strengthens concrete in two distinct manners that are advantageous:

- It causes secondary calcium silicate hydrate to form when it interacts with calcium hydroxide crystals
- Metakolin serves as an additive

Supplemental cementing ingredients are produced in an extremely alkaline environment which reacts with calcium hydroxide to create calcium alkali silica hydrate, which greatly increases the strength of the cementing material. the blain fineness of supplemental cementing ingredients is similar to or greater than that of standard cement. An anhydrous grains improve density and prolong the fine aggregate grade. There are two categories for supplementary cementing materials. Slow-reacting and rapid-reacting. Slow-reacting materials are fly ash and slag, while rapid-reacting materials are metakaolin and silica fume. fly ash concrete is expected to function better over the long term than Portland concrete, but with lower initial strength. In the initial stage, metakaolin has higher initial strengths (Powers, 1958).

The effects of metakaolin on concrete strength were demonstrated by Ding and Li (2002) and Khatib (2008). the degree of improvement depends on the w/cm ratio and the grade of the metakaolin. the strength of concrete can be improved due to the fact that metakaolin has a better impact on the strength of concrete than conventional Portland cement. It can fit into smaller spaces to improve packing. They said that metakaolin reacts with calcium hydroxide to create secondary calcium silica hydrate, which will strengthen and reduce the porosity of the paste.

According to Guneyisi et al. (2017), the ideal replacement rate is around 15 percent, and any additional additions will tend to ruin the concrete mixture.

2.4.2 Effect of metakaolin on setting time

The term "setting time" describes how long it takes for concrete to go from being a viscous liquid to a solid. By using a known force and measuring penetration, it is determined. Wet sieving the large coarse aggregate is typically used to evaluate the setting of concrete, and mortar is used for the setting test. Pre-induction phase, induction period, acceleration stage, and post-acceleration stage are the four parts that make up the setting curve. Pre-induction happens during the first few seconds of soaking, and all phases quickly hydrate as significant amounts of calcium hydroxide escape into the resulting solution (Taylor, 2014).

Minimal hydration happens during the induction stage, runs for a few hours, and is related to the level of calcium, hydroxyl, and sulfate ions in solution. After mixing, C_3S begins to react quickly, and C_2S hydration also occurs, which is when the acceleration period starts. As a result, as the concentration of sulfate drops, ettringite production uses up all of the sulfate ions. As for the post-acceleration time, it is characterized by the onset of C_2S hydration as well as a consistently slower rate of C_3S hydration as the rate of diffusion decreases. Ettringite also starts to react with any extra C_3A to generate mono sulphate due to the sulphate loss. Therefore, setting depends on the w/cm ratio, the mix's amount of cement, the temperature, the chemistry of the cement, the particle size of the cement, and the additives (Taylor, 2014).

According to Brooks and Johari (2001), additional cementing elements take longer to set because they are less hydraulic than Portland cement, which results in a lower percentage of a functional adhesive in the first couple of hours. The results obtained by Brooks and Johari, (2001) are shown in Table 2.2, it demonstrates that metakaolin samples increase the initial set time by up to 28 percent, but this trend reverses after the 10 percent replacement, the point at which the setting time starts to drop once more. He noted that the most likely cause of the change in behaviour was an increase in the amount of aluminate, which led to the creation of more aluminate phase and accelerated the loss of fluidity in the mixture.

Justice and Kurtis (2007) also reported using the Vicat setting test indicated that, the metakaolin addition of 8 percent reduced the final time by approximately 40 percent. Comparing the two studies, metakaolin samples don't differ significantly except in fineness. Therefore, the metakaolin of Brook's (15m^2/g) was in between the two-fineness used by Justice (25m^2/g) and (11m^2/g), it was assumed fineness might not necessary be the issue. The disparity in the two findings in terms of level of replacement is probably due to the result of the various testing procedures.

Table 2.1: Setting time of varying cementious material replacement.

Mixture of concrete	Initial setting (h)	Relative (h)	Final setting (h)
OPC	5	1	7.7
SF5	6.27	1.25	8.38
SF10	6.7	1.34	8.72
SF15	8.82	1.76	10.9

MK5	6.42	1.28	8.82
MK10	6.98	1.40	9.42
MK15	6.45	1.29	9.31
FA10	5.97	1.19	8.93
FA20	6.1	1.22	9.37
FA30	7.83	1.57	11.6
GGBS20	7.88	1.58	12.9
GGBS40	11.53	2.31	17.8
GGBS60	12.43	2.49	21.55

Source: (Brooks and Johari, 2001).

2.5 Calcination Reaction of Kaolinite

Calcination, which results in the loss of water, is the process of heating a substance below its fusion point. This important method frequently improves the qualities and values of kaolin. After being calcined, the kaolin transforms to white and become chemically inert, being used in an array of products like paper, paint, rubber, and plastic (Drzal et al., 2003).

The main reaction that took place was the breaking apart of the kaolinite. Kaolinite is the main component. $Al_2Si_2O_5(OH)_4$ is an aluminosilicate compound made up of a layer of alumina crystal and a layer of silicon oxide. the formation of metakaolin is caused by the thermal reaction which collapse the kaolinite structure and removing the -OH group from the mineral (Varga, 2007).

According to Ptacek et al. (2014), the thermal conversion of kaolinite to Meta kaolinite occurs in three stages

- First stage: The long-ordered structure of the kaolinite begins to disintegrate as the heating process commences. As the heating temperature reach 400 ^{o}C, dehydroxylation of hydroxyl structure in the layer brings about evaporation of the water crystal from the main structure.
- Second stage: second stage and first stage occurred simultaneously, water crystal compound is being released when kaolinite structure in kaolin mineral has broken down at temperature between (100 ^{o}C – 200 ^{o}C) and continuously till the temperature reach 400 ^{o}C. H^{+} and O^{2-} will be produced by the -OH group. During the process, the second -OH group joins the protons to create water molecule.

when the following stages of reformation takes place, the O^{2-} will still be present.

- Third Stage: At a temperature of 600 ^{o}C, the shattered layer structure of the kaolinite will be rebuilt. The crystal structure will also be rebuilt to create meta kaolinite. The balanced chemical equation for the dehydroxylation reaction of kaolinite to form Meta kaolinite is written below:

$$Al_2Si_2O_5(OH)_4 \rightarrow Al_2Si_2O_7 + 2H_2O \quad (2.3)$$

At higher temperature (925 ^{o}C - 950 ^{o}C) of calcination, metakaolin structure that will be formed become aluminium-spinel structure ($Si_3Al_4O_{12}$).

$$\underset{\text{Metakaolin}}{2Al_2Si_2O_7} \rightarrow \underset{\text{spinel phase}}{Si_3Al_4O_{12}} + \underset{\text{free silica}}{SiO_2} \quad (2.4)$$

At higher temperature (1050 °C) will make the spinel structure to transform to mullite structure ($3Al_2O_3.2SiO_2$) and on continue heating to temperature of 1200 °C, the amorphous SiO_2 structure will transform to cristobalite SiO_2, as shown below:

$$2Si_3Al_4O_{12} \rightarrow 2Si_2Al_6O_{13} + 5SiO_{2(amor.)} \qquad (2.5)$$

$$SiO_{2(amor.)} \rightarrow SiO_{2(cristobalite)}$$

Rashad (2013) found that the kaolin's calcination process produces metakaolin with various characteristics depending on the temperature and duration of the calcination process. He said that 550 °C to 800 °C was the best temperature range employed by earlier researches. Due to the varied kaolin sources, calcination times vary. As a result, the composition and minerals found in different kaolin sources vary.

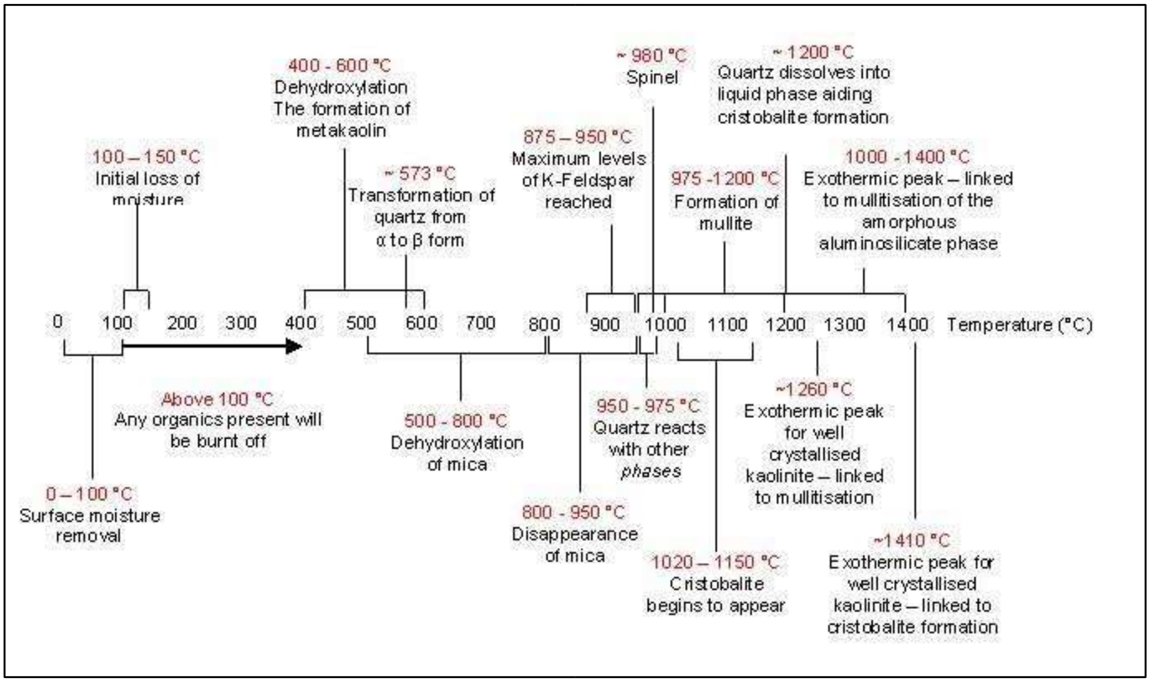


Figure 2.8: Temperature-dependent development of the calcination physiochemical response and transformation for kaolin and related mineral entities

(Source: Drzal et al., 2003).

There are additional reactions that take concurrently at higher temperatures. Any mica that is found in the kaolin will be dehydroxylated between 500 and 800 ^{o}C

Dunham et al. (1992) noted that the temperature at which the mica disappeared was between 800 and 950 ^{o}C. the results of the calcination showed that the feldspar content eventually dropped off or vanished. the temperature at which mica has vanished tends to be higher than the one at which the greatest potassium feldspar content occurs. the raw components were created by the breakdown of the mica. the phase change from the typical alpha form to the less stable and less dense alpha form. As a result, the temperature was determined using the Differential Scanning Calorimetry (DSC) traces. The beta-quartz also begins to react with potassium-rich phases between 950 and 975 ^{o}C, which causes them to dissolve. At a temperature of about 1200 ^{o}C, any quartz residues could dissolve in the liquid state. consequently, encourage cristobalite production.

Traces from differential scanning calorimetry discovered a little exothermic peak occurring at about 1410 ^{o}C. there is a sign that cristobalite is foaming in kaolin when the amount of quartz is low. the temperature of cristobalite production can be lowered at 200 ^{o}C
(Chakraborty, 2016).

When the temperature is higher than the spinel phase, the kaolinite keeps reacting. According to the type of kaolin, mullite starts developing between 975 ^{o}C and 1200 ^{o}C. When the mineral mullite ($3Al_2O_3SiO_2$) forms, silica from the structure starts to discharge (Schneider et al., 1994).

Chakraborty (2016), reported that, there are two stages to the production of mullite. Two exothermic peaks appeared on the trace when analysed with differential scanning calorimetry. First peak was broad and occurred throughout a wide temperature range of 1000 to 1400 ^{o}C

2.6 Dehydroxylation Reaction of Kaolinite

Clay undergoes three stages of thermal behaviour which includes: dehydration, dehydroxylation and recrystallization (Mohammed, 2017). Kakali et al. (2001), reported that, Mohammed (2017) stated that the dehydration stage can last up to 2000 ^{o}C, although the end of it occurred at 1000 ^{o}C. Depending on the clay structure and minerals present, different temperature ranges are required for the dehydroxylation and recrystallization processes. Free water molecules escape during the dehydration stage, but the structure of the clay remains unchanged.

The dehydroxylation process of kaolinite takes place between 400 ^{o}C and 600 ^{o}C. The elimination of water is needed to create metakaolin.

the crystallinity of the kaolin structure is destroyed by the removal of hydroxyl groups during the dehydroxylation reaction (Mohammed, 2017). Pozzolanic character of clay minerals resulted from this thermal changes.

Rahier et al. (2000) proposed a formula which was used to calculate the degree of dihydroxylation (DTG) from thermogravimetry analysis (TGA) measurement.

$$DTG= \qquad (2.6)$$

In the equation above, m and m_0 signifies residual and maximum mass loss, respectively

Before TGA analysis, Rahier et al. (2000) dehydrated the kaolin samples at 2500 ^{o}C; the water from dehydration was not regarded as a component of m and m0. All kaolin minerals undergo modifications during the dehydroxylation stage (Teklay et al., 2014).

$$Al_2O_3.2SiO_2.2H_2O \rightarrow Al_2O_3 .2SiO_2 + 2H_2O_{(g)} \qquad (2.7)$$

The creation of aluminium during the calcination process has led to the dehydration of kaolin, which can be used in concrete. According to Bich et al. (2009), the ratio of the declining to rising slopes of the TGA mass loss curve's first derivative over a range of temperatures or times can provide insight into the surface imperfections found in kaolin.

The apparent impacts include the nature and quantity of kaolin impurities, vacant hydroxyl ion locations or amorphous substance that is present in kaolin due to natural formation conditions.

According to Bich et al. (2009), there should be no or few flaws coupled with numerous effects with a slope ratio (SR) of 1 and a slope ratio (SR) greater than 1 correspondingly. Additionally, they demonstrated that the slop ratio value and presence of surface defects increase with the degree of dehydroxylation of a kaolin.

According to Amin et al. (2015), the most reactive clay mineral is calcined kaolinite, which is followed by the other kaolin polymorphs, montmorillonite, illite.

2.6.1 Degree of dehydroxylation of kaolin and effect on reactivity

Bich et al. (2009), examined how the degree of dehydroxylation affected the reactivity of kaolinite clays. They determined the degree of dehydroxylation from the differential scanning calorimetry measurement and altered the time it took to do so. Among the clay samples, the dehydroxylation levels ranged from 50 to 100 percent. The scientists found that clays with a high degree of order required less time to complete dehydroxylation than clays with a low degree of order. The hydroxyl ion is easier in a disorganized structure. Lime consumption in a paste made of 50 percent calcium hydroxide and 50 percent calcined material was used to gauge the reactivity of clays. the scientists concluded that there was no correlation between lime consumption and the degree of dehydroxylation during the first seven days. At 28 days, a strong association between these characteristics was seen.

complete dehydroxylation. Kaolin's disordered structure takes 45 minutes, whereas its ordered structure takes 5 hours. So, in a disordered structure, the hydroxyl ion is considerably easier. To gauge the reactivity of calcined clays, lime consumption in a paste made of 50 percent calcium hydroxide and 50 percent calcined material was utilized. The scientists came to the conclusion that there was no correlation between lime consumption and the degree of dihydroxylation during the first seven (7) days. Even yet, at twenty-eight (28) days, a strong association between these characteristics was seen. Even after 28 days, there was still a lot of changes in the data. the authors theorize that factors other than the degree of dehydroxylation may affect baked clay reactivity.

2.7. Pozzolans and Pozzolanic Reactivity

2.7.1 Pozzolans

A pozzolan is a compound with cementitious capabilities when it combines with calcium hydroxide in the presence of water. The amount of iron oxide and alumina should be greater than 70% (ASTM C618-17 International, 2017). Pozzolans should also be classified based on whether they were formed naturally or artificially. The term "natural pozzolans" describes substances that result from an original rock being altered through time or by an ad hoc natural occurrence, such as a volcanic explosion, and are unrelated to human activity. Tuffs and zeolites are examples of naturally occurring pozzolans that are formed from weathered consolidated sediments, volcanic ash, diatomaceous earth, and unburned clays.

Artificial pozzolans are materials that originate from coal-fired power plants and the silicon industry. Rice husks are an example of artificial pozzolans that have to be burned in order to remove the organic material and obtain a pozzolan with a high concentration of silica. the metakaolin is made specifically. The common factor among all artificial pozzolans is that they were all created through a process involving heat that either disrupted the long-term order of the source material (calcined clays, silica fume), or removed the organic matter to create fly ash, which is largely composed of the original material and is high in alumina or silica (Fernandez et al., 2011).

2.7.2 Pozzolanic reactivity

The application of pozzolanic substance in Portland cement depends on the alkaline medium created by the hydrating cement (Bye, 2019).

Pozzolanic refers to a property that indicates a substance’s capability to react with calcium in the presence of water to produce compound with cementitious material qualities. Direct measurement and indirect measurement are the two ways of determining a material’s properties. Direct technique based on the use of an instrument to measure the properties of the material, with or without the requirement to be aware of the pozzolanic reaction that occurs to metakaolin. The indirect method modifies the cement’s mechanical properties by adding metakaolin (ASTM C618-17 International, 2017).

The cement hydration generated has a similar composition as the original calcium silicate hydrate.

Depending on how much rice husk ash was replaced for cement, the calcium silicate ratio might be different. the silicate ratio is not as high as it could be (Taylor, 2014).

General formula for pozzolanic reaction:

$$xCa\,(OH) + SiO_2 + (x\text{-}y)\,H_2O \rightarrow (CaO)_x\,SiO_2\,(H_2O)_y \qquad (2.8)$$

Alumina to calcium ratio of calcium silicate hydrate is increase, when a natural pozzolan or calcined clay contains appreciable quantities of reactive alumina, which tends to favour the formation of calcium aluminate.

CHAPTER THREE

3.0 MATERIALS AND METHODS

The materials and chemicals, process for characterization of metakaolin prepared from kaolin deposit as an admixture to cement were listed and briefly described.

3.1 Materials, Reagents and Equipment

The following are lists of the items that were used for this research project;

3.1.1 Reagents and materials

Table 3.1 below lists the ingredients and chemicals used to characterize metakaolin, a cement additive made from kaolin deposits:

3.1.2 Equipment

Table 3.2 Indicate the equipment used for the manufacturing of metakaolin from kaolin deposit.

Table 3.1: Reagents and materials.

S/N	Materials and Reagents	Manufacturer	Purity
1	Kaolin	Kogi state	95.0
2	Acetone	JTB Baker Company	99.5
3	Iso propanol	JTB Baker Company	99.7
4	Portland cement	Dangote cement	
5	Distilled water	Commercial grade	

Table 3.2: Equipment and apparatus

S/N	Equipment/Apparatus	Model	Source
1	Electrical Furnace	Gallanhamp, UK	Material science FUT Lab
2	Oven	S-87K027	Civil Engr. FUT Lab
3	Compressive Strength Machine	12399890	Building Depart. FUT Lab
4	Sieve	ISO3310	Civil Engr. FUT Lab
5	Vicat apparatus		Civil Engr. FUT Lab
6	Digital weighing balance	TT 12F	Civil Engr. FUT Lab
7	Beakers	Pyrex England	Civil Engr. FUT Lab
8	Measuring Cylinder	Pyrex, England	Civil Engr. FUT Lab
9.	Morta and pistol	-	Civil Engr. FUT Lab
10	Fine aggregate (Sand)	-	Minna
11.	Coarse aggregate(gravel)	-	Minna

3.2 Methods

The methodology for the Characterization of Metakaolin Prepared from Kaolin Deposit as admixture to Cement is described below:

3.2.1 Purification of kaolin

An indigenous raw kaolin (Ahoko and Alolji) samples were collected from Eastern part of Kogi in Nigeria. The two kaolin samples were mechanically treated using manual crushing.

Thereafter, purified by sedimentation (Kovo et al., 2009). The samples were then dried in the oven for 12 hours at 125 ^{o}C to less than 0.5 percent moisture content.

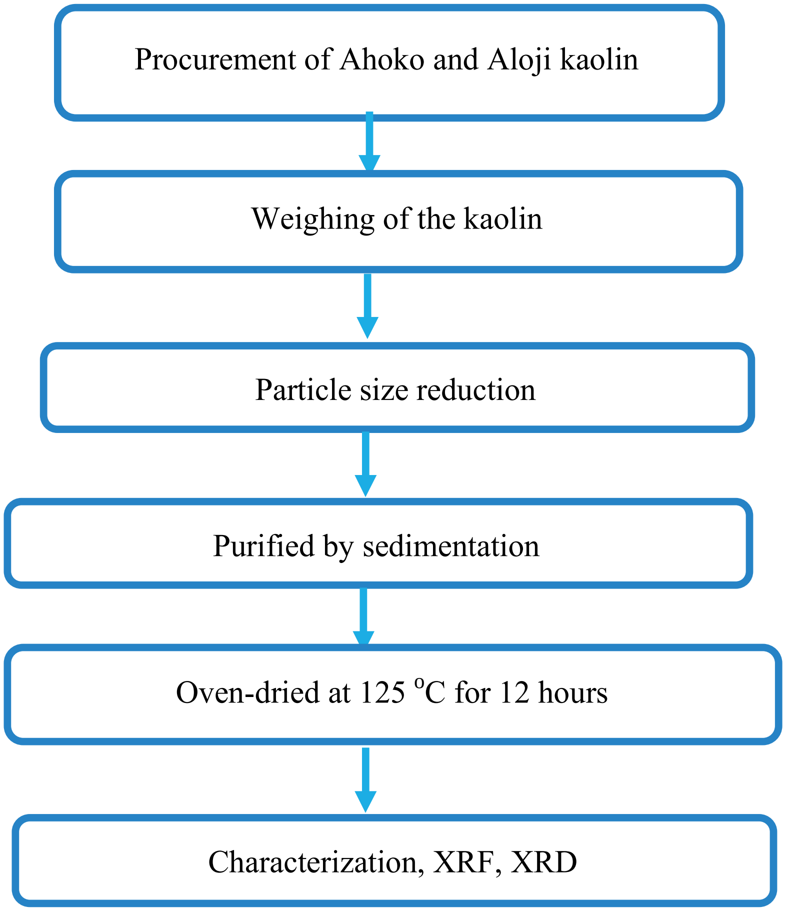


Figure 3.1: Methodology for purification of raw kaolin. Source: (Kovo et al., 2009)

3.2.2 Metakaolin production

The kaolin, Ahoko, and Aloji were processed in a pilot-plant furnace at 550 ^{o}C for 1 hour for complete dehydroxylation. The full conversion of kaolinite to metakaolinite was verified by X-ray diffraction. the metakaolins were developed from Ahoko and Aloji.

$$Al_2Si_2O_5(OH)_4 \rightarrow Al_2Si_2O_7 + 2H_2O \qquad (3.1)$$

Kaolinite metakaolin water.

Equation above represents dehydroxylation reaction of kaolinite to metakaolin

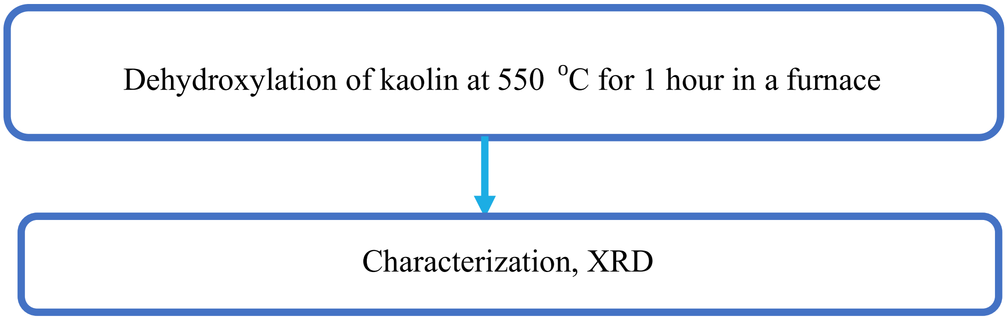


Figure 3.2: Methodology for Meta kaolinization.

Source: (Kovo et al., 2009)

3.3 Production of Mortar and Concrete

The water to cement ration of 0.52 was used to make concrete and mortar. the concrete created by Ahoko and Aloji was formed in a large cubic mold of 100 by 1000 by 100 mm. The mortar was cast in a smaller mold of 50 by 50 by 50mm. The specimen was kept in the molds for 5 hours before being put in containers and water-cured at 20 degrees celsius. For 7, 28, 56days, samples were hydrated. Portland cement was replaced with 5, 10, 12 and 15 percent w/w for the production of mortar.5, 10, 15, 20, 30, and 35 percent w/w are for concrete manufacture. The strength, setting time, and water demand of mortar and concrete were determined.

3.3.1 Production of blended concrete

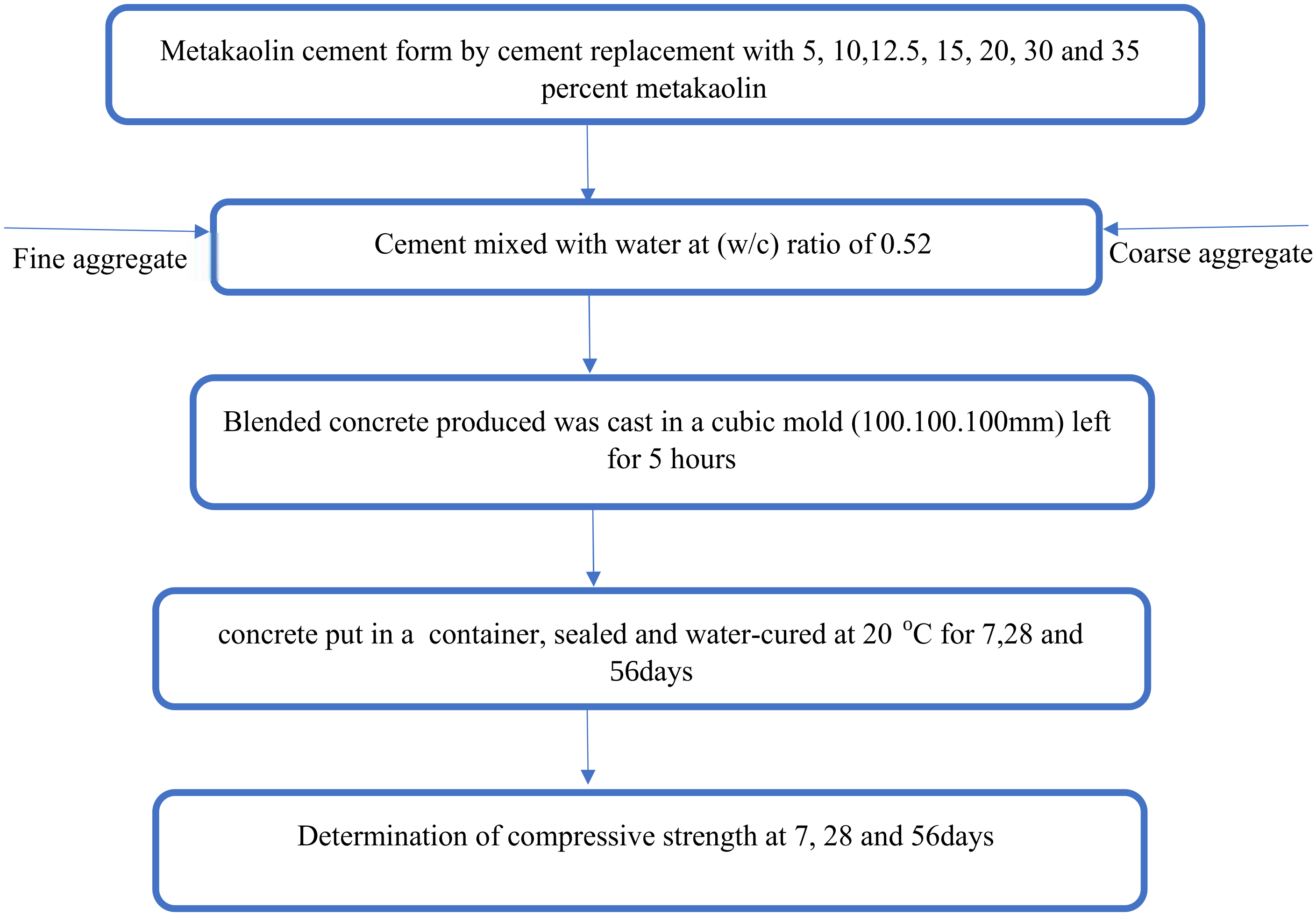


Figure 3.3: Methodology of blended concrete production

Source: (Badogiannis et al., 2005)

3.3.2 Production of blended Mortar

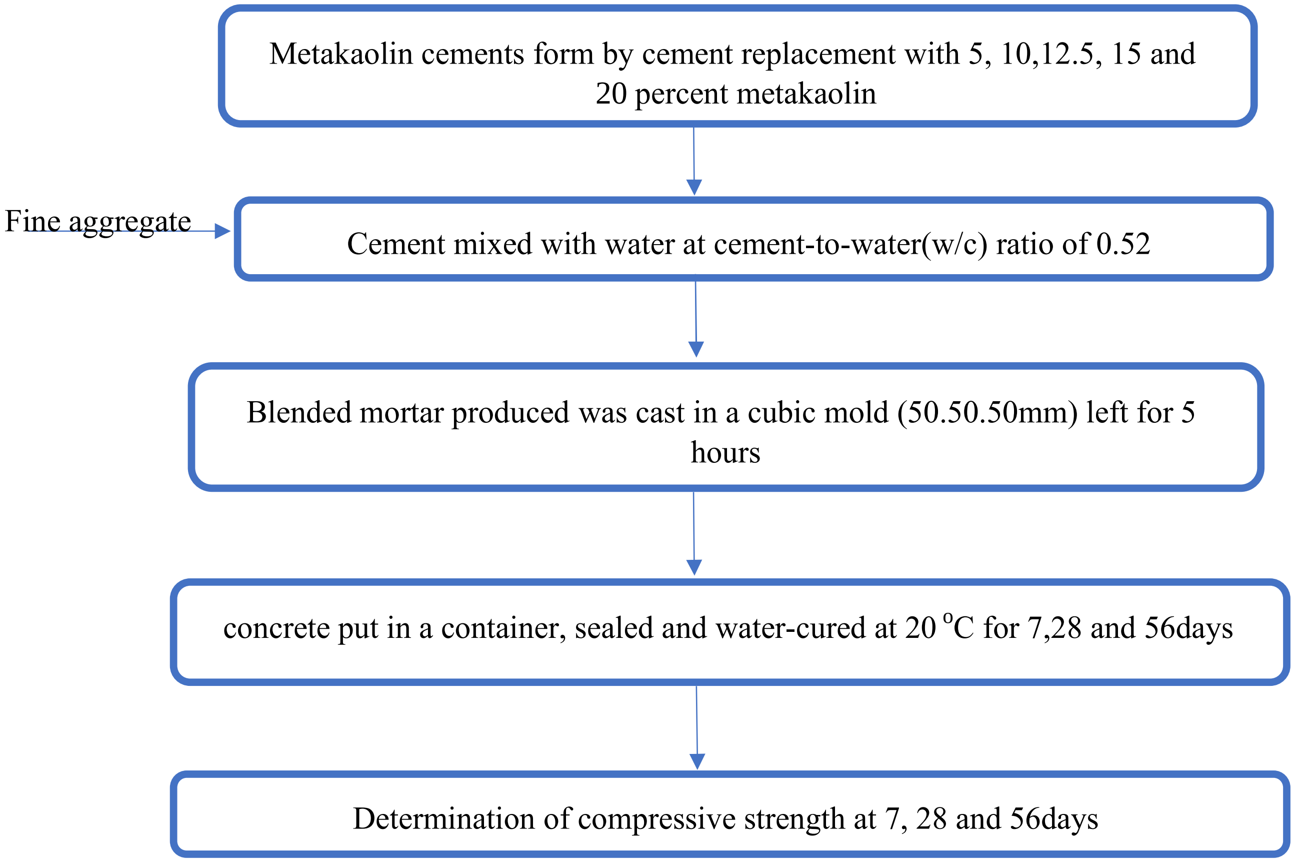


Figure: 3.4: Methodology of blended mortar production

Source: (Badogiannis et al., 2005)

Plate V: Concrete and mortar produced before curing

Plate VI: Concrete and mortar at 7 days of curing

Plate VII: Concrete at 28 days of curing

Plate VIII: Concrete and mortar at 56 days of curing

3. 4 Hydration Stoppage and Pozzolanic Evaluation

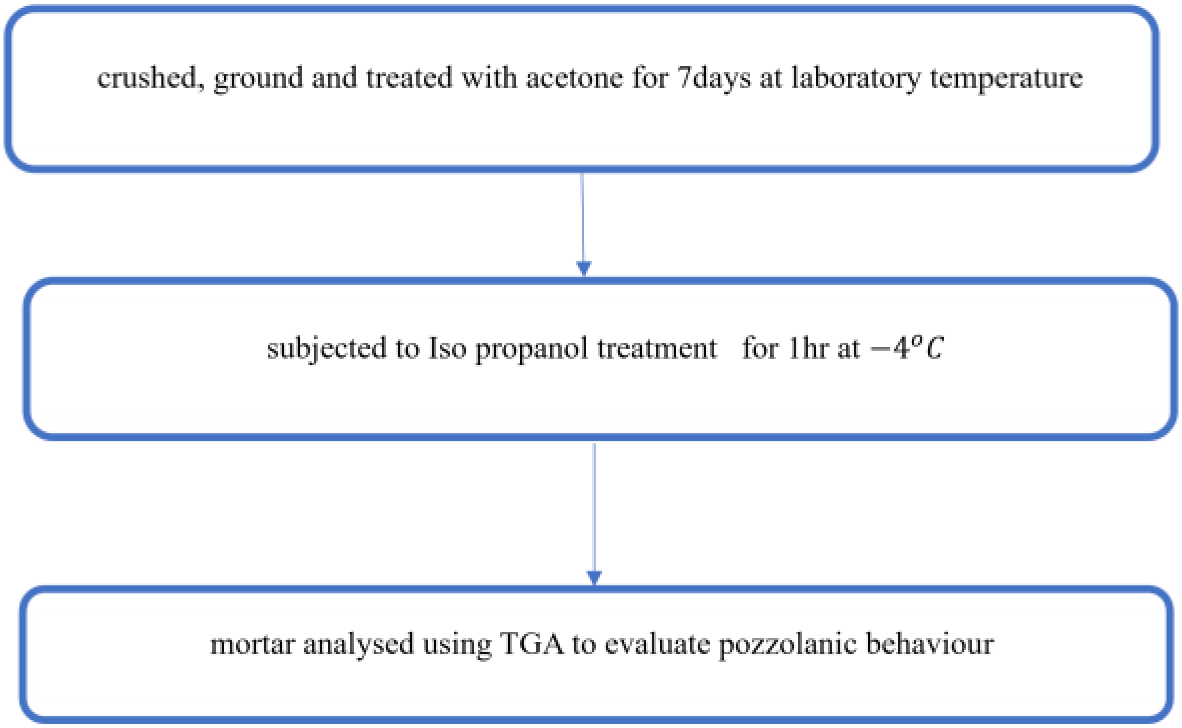


Figure 3.5: flow chart for hydration stoppage and pozzolanic evaluation

Source: (Badogiannis et al., 2005)

Plate IX: Compressive strength machine

3.5 Flow Chart Diagram for Metakaolin Production Incorporating Cement Replacement by Metakaolin for Concrete and Mortar Production

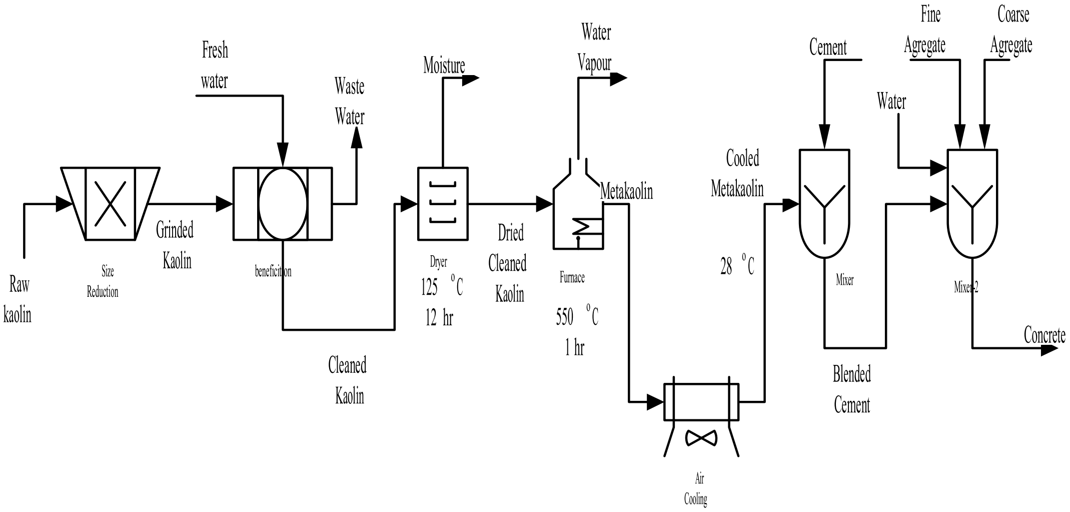


Figure 3.6: Flow chart of blended concrete production

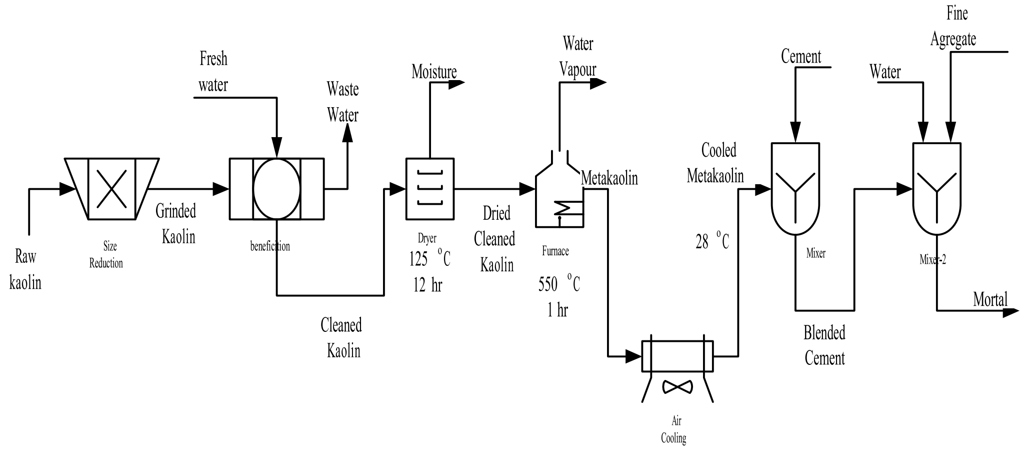


Figure 3.7: Flow chart of blended mortar production

Plate X: Fabricated furnace

Plate XI: Fabricated furnace side view

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Results

The results gotten from the characterization of metakaolin prepared from kaolin deposit as an additive to cement in construction industry are presented and discussed. The oxide composition of the kaolin deposits and metakaolin were presented and discussed. Also, the result of the thermogravimetry of kaolin deposits, X-Ray diffraction analysis of metakaolin, the setting time, water demand, the compressive strength of metakaolin concrete and mortar was given and discussed. Furthermore, the material balance, energy balance, detail design of the calcination furnace and description of the fabrication of the pilot plant calciner was carried out.

4.2 Oxide Composition of Kaolin

Two kaolin, K1 (Ahoko) and K2 (Aloji), with different chemical composition were determined and their compositions are stated in Table 4.1.

Table 4.1: Chemical composition of kaolin (% w/w)

Constituents	K1(/Ahoko) (%w/w)	K2 (Aloji)(%w/w)
SiO_2	54.039	54.05
Al_2O_3	40.827	26.21
Na_2O	0.109	0.04
K_2O	0.537	0.18
Fe_2O_3	2.009	-
MgO	0.177	0.22
FeO	-	2.94
CaO	-	3.61
TiO_2	-	2.37

L.O.I. (loss on ignition)	2.07	10.37

The result shown in Table 4.1 the chemical composition of Aloji refined kaolin, is based on the phase analysis, the titanium oxide in the kaolin emanates from anatase, potassium and sodium are closely related to muscovite and illite. The FeO is suspected to be from iron containing mineral like goethite, or colloidal particle from kaolin crystal structure (Liu and Dai, 2003). The alumina and silica contents are 26.21 and 54.05 percent respectively. The alumina-silica ratio is 0.48. The chemical composition is similar to that of kaolin deposits studied by (Liu and Dai, 2003). The fluxes (TiO_2, K_2O, Na_2O, CaO and MgO) are suspected to have lowered the refractory value of the kaolin, but because the alumina content is relatively high, this effect is minimal. Table 1 demonstrates that the Ahoko refined kaolin has a decrease in the percentage composition of SiO_2 and rise in the percentage composition of Al_2O_3. A high concentration of Al_2O_3 in the refined sample suggests that Ahoko is a kaolinite rich clay.

4.3 Oxide Composition of Produced Metakaolin

Table 4.2 is the result of oxide composition of metakaolin. Metakaolin is an amorphous alumina silicate structure formed by controlled heating of origin kaolin that break off -OH chain from the kaolinite to form pozzolan (Mohammed, 2017). Pozzolans can generate cementitious compounds by chemically reacting with calcium hydroxide and water in the reacting environment. However, metakaolin obtain from dehydroxylation of kaolinite comprised majorly oxide of aluminium and silica. From the table below, it is noticed that the summation of percentage composition of silica and Aluminum for Ahoko metakaolin is 95.965 percent and 82.259 percent for Aloji metakaolin respectively. This indicate that the metakaolin produced is a good pozzolana since these values are greater than 70 percent.

Table 4.2: Metakaolin chemical composition. (w/w)

	MK1(/Ahoko) (%w/w)	MK2(Aloji) (%w/w)
SiO_2	54.195	54.15
Al_2O_3	41.477	28.11
Na_2O	0.066	0.04
K_2O	O.537	0.18
Fe_2O_3	0.887	-
MgO	0.54	0.22
FeO	-	0.84
CaO	-	3.61
TiO_2	-	2.47

4.4 Thermo Gravimetric Analysis

The TGA analysis result shows a gradual loss of weigh as the kaolin samples were heated to a temperature of 300 ^oC, a spontaneous weight lost was observed afterword until it reached a temperature of 500 ^oC, finally there were insignificant weight lost at temperature above 500 ^oC which can be observed from Figure 4.1 and 4.2, this implies that the samples can undergo dehydroxylation reaction between the temperature of 500 ^oC to 900 ^oC , this is also in line with the result obtained by Kovo et al. (2009).

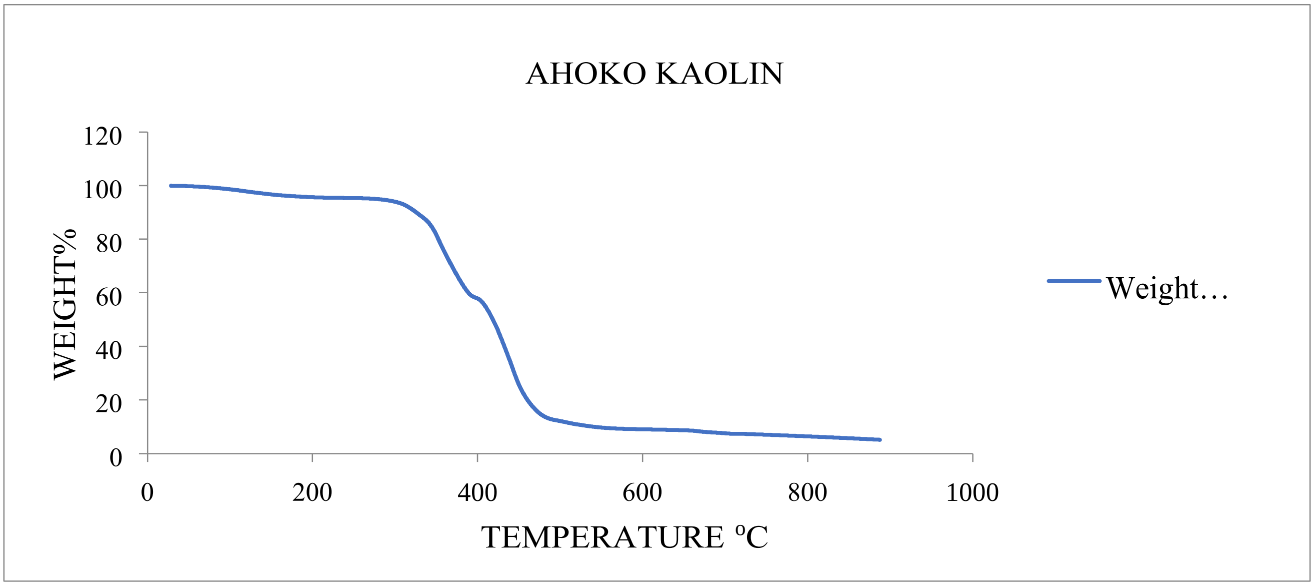


Figure 4.1: TGA Analysis for Raw Ahoko Kaolin

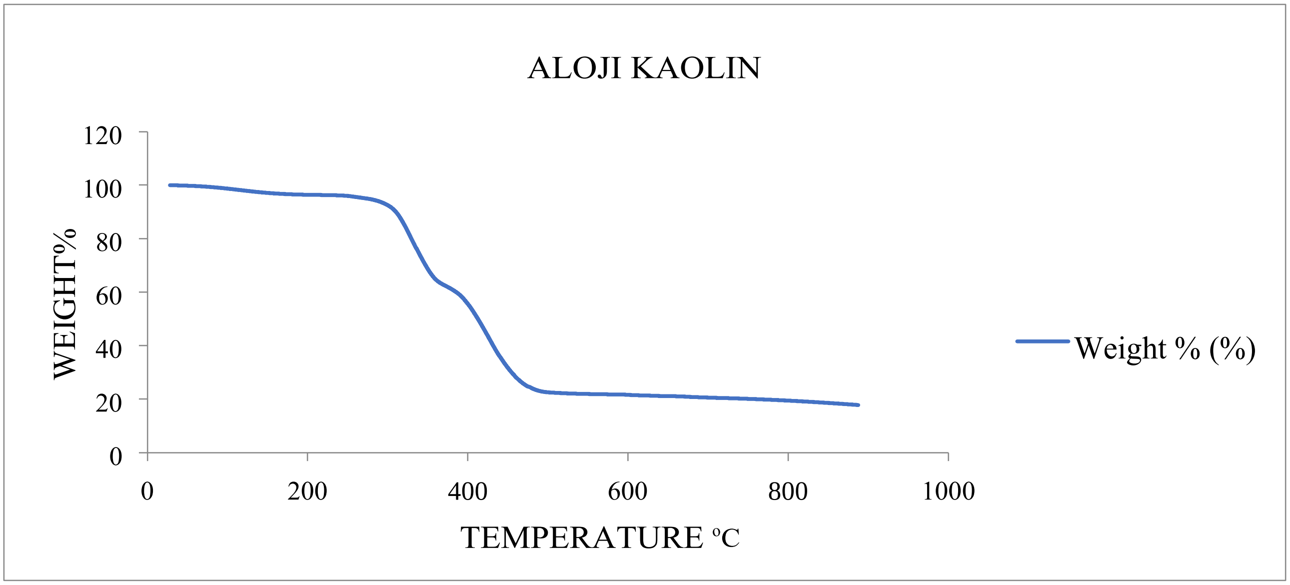


Figure 4.2: TGA Analysis for Raw Aloji Kaolin

4.4 Analysis of X- Ray Diffraction

4.4.1 X- ray diffraction of refined kaolin

Figure 3 shows an X- ray Diffraction result. the structural properties of the samples under study can be verified with the help of the XRD. the dispersion phenomenon allows crystals to act as a grating toward an X-ray. The atoms in the disperse the X-rays in all directions and in a material-specific fashion. The extent of crystallinity and crystallite size are revealed by the XRD pattern of refined kaolin samples. At 2 = 12.37o (d= 7.15), it showed a strong and shallow peak with a bottom reflecting. This corresponds to the top region in the refined Aloji kaolin sample. This was done to find out the quantity of quartz and kaolinite in the specimen.

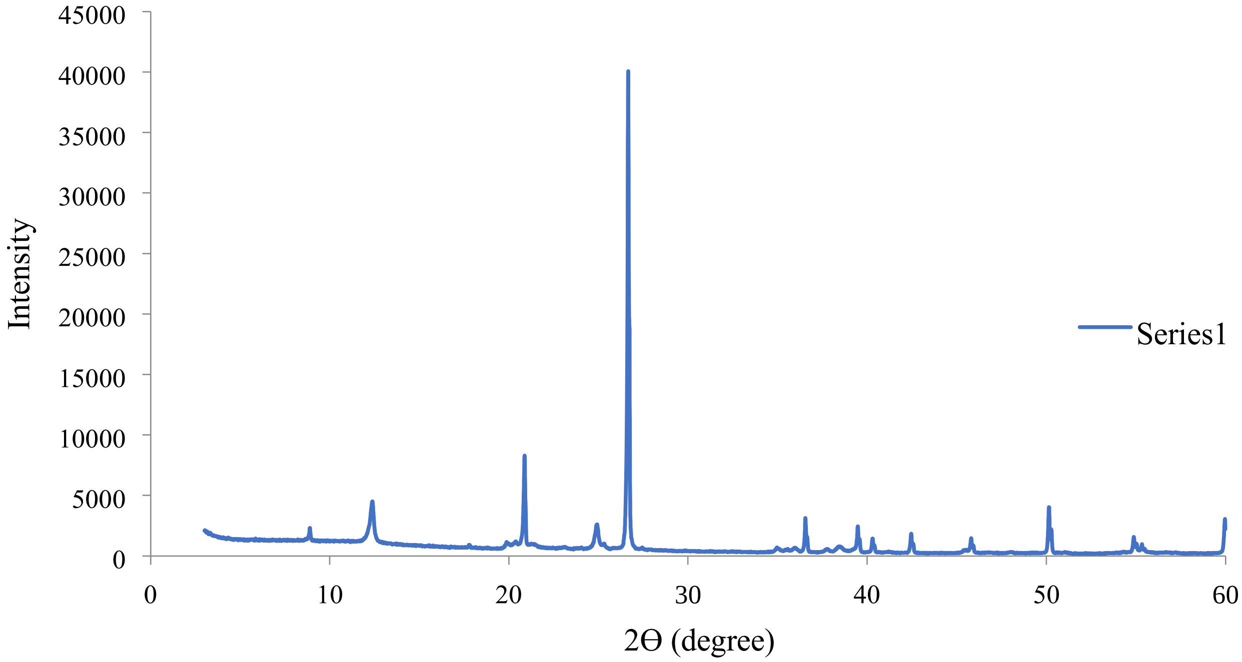


Figure 4.3a: XRD Pattern of Refined Ahoko kaolin

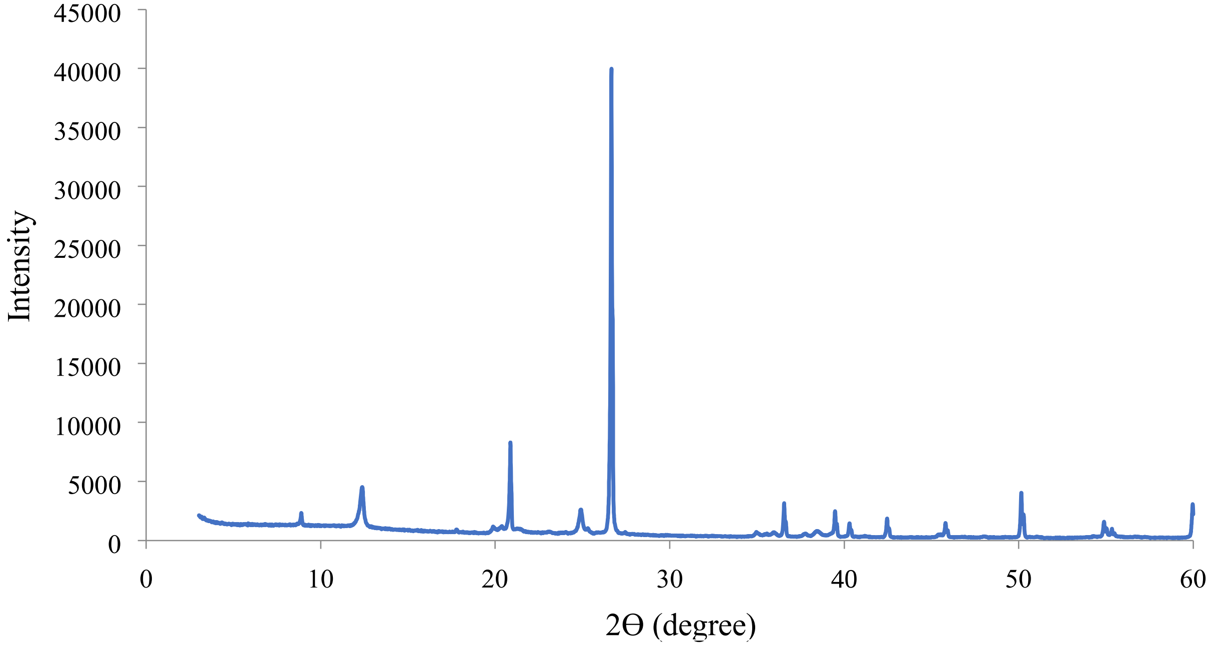
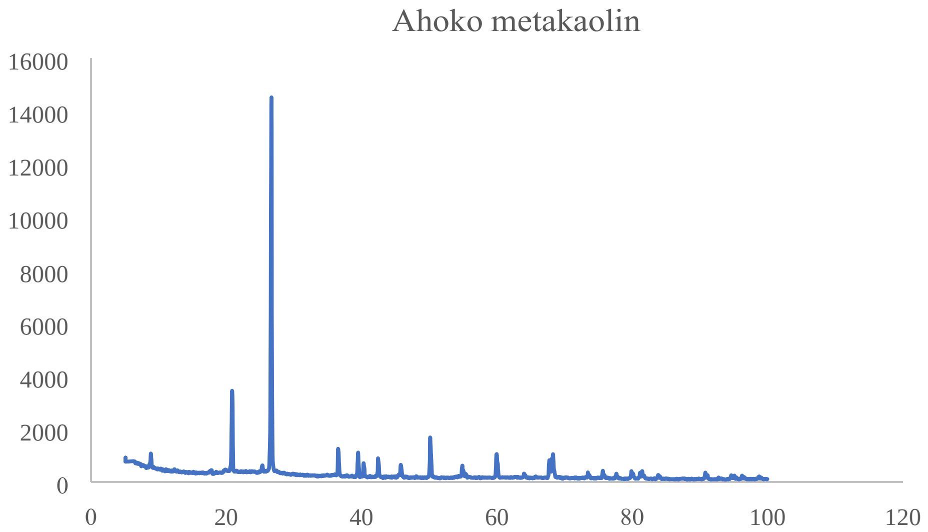


Figure 4.3b: XRD Pattern of Refined Aloji Kaolin 4.4.2

X- ray diffraction of metakaolin



. Figure 4.4a.: XRD Pattern of Ahoko Metakaolin

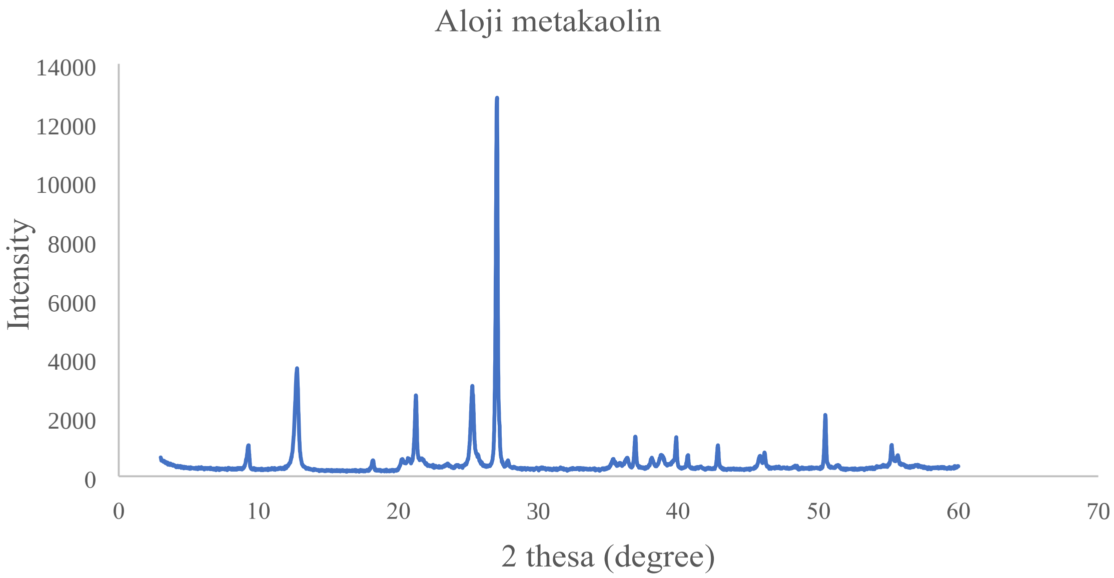


Figure 4.4b: XRD of Aloji metakaolin

The main reflections of Ahoko and Aloji kaolin sample were increased by the increased intensity of the XRD pattern shown in Figure 4.4a and 4.4b. the loss of hydration water may result in a better ordering in the structure of the kaolinite molecule. there was a small halo which indicated the main reflection was lost. metakaolin produced can be described as an amorphous material containing free silica, free alumina as reported by Brigatti et al. (2006).

4.5 Setting Time and Water Demand of Metakaolin Blended Mortar

Table 4.3 gives the water demand and the setting times of the cement mortar. The ‘‘water demand’’ is the quantity of water that is required in order to prepare a cement paste or mortar of standard consistency as specified in EN 196-3. The blended cements demand significantly more water than the relatively pure cement. With a metakaolin content of 5 percent, the water demand varies from 28.0 to 25 percent for Ahoko and 27 to 24 percent for Aloji, while the

Portland cement has a water demand of 25 percent. With a metakaolin content of 5 to 10 percent for Ahoko sample, the water demand increases from 28 to 29 percent. MK1 and MK2 show the best behaviour from 5 to 10 percent.

The initial and final setting time of metakaolin cements is affected by the metakaolin content. Cements with 10 percent metakaolin, generally, exhibit similar initial setting times but different final setting time with delay. while for 12.5 percent metakaolin content there is not delay in the setting time.

Table 4.3: Physical properties of metakaolin cements

Sample	Metakaolin (%w/w)	Water demand (%w/w)	Initial setting time	Final setting time
PC	-	25	40	256
Mk1-5	5	28	112	200
Mk1-10	10	29	110	260
Mk1-12.5	12.5	27	50	180
Mk1-15	15	25	40	250
Mk2-5	5	27	80	243
Mk2-10	10	29	110	310
Mk2-15	15	23	131	263
Mk2-20	20	24	121	269

4.6 Compressive Strength of Metakaolin Concrete

4.6.1 Aloji metakaolin concrete

Table 4.4a. Below shows the compressive strength development of Aloji metakaolin concrete and cement replacement level. The concrete was produced with 10, 20, 30 and 35 percent metakaolin by weight with water-to-cement ratio (w/c) of 0.52 which showed enhanced strength at ages up to 56days. At 56days, the concrete prepared with 10 percent metakaolin showed the highest strength of the group, 284 N/mm^2. At 28 days, the concrete prepared with 10 percent metakaolin also showed the highest strength, 268.7 N/mm^2, followed by 30 percent metakaolin of compressive strength 263.33N/mm^2 at 56days., then decrease to 249.23 at 35 percent replacement. Control specimens had the lowest strengths at all ages. Other researchers, at 365 days determined the compressive strength of metakaolin cement at 10 percent to be 310.2N/mm^2. Whereas Ding and Li (2002) further investigated 10 percent replacement with metakaolin was approximately 305N/mm^2.

Table 4.4a: Compressive strength of Aloji blended concrete.

	COMPRESSIVE STRENGTH (N/mm^2)				
AGE(DAYS)	0%	10%	20%	30%	35%
7	119.63	221.73	206.47	129.47	212.77
28	140.57	268.7	238.9	185.37	219.77
56	187.8	284	258.63	268.33	249.23

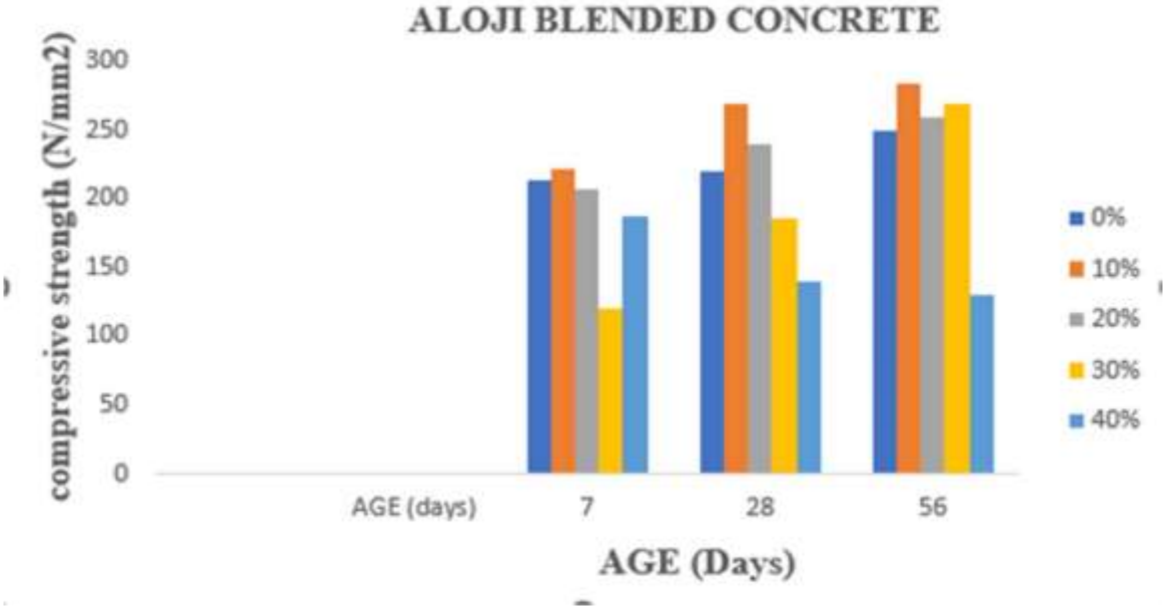


Figure 4.5: Compressive strength of Aloji blended concrete in relation to curing age.

4.6.2 Ahoko metakaolin concrete

Table 4.4b below shows the compressive strength development of Ahoko metakaolin concrete and cement replacement level. The concrete was produced with 5, 10, 12.5, 15 percent metakaolin by weight with water-to-cement ratio (w/c) of 0.52 which showed enhanced strength at ages up to 56days. the concrete prepared with 10% metakaolin showed the highest strength of the group 356.5 N/mm^2 at 56 days. followed by 352.4N/mm^2 at 28days then 314.56 N/mm^2 at 7 days. Control specimens had the lowest strengths at all ages. Similar results were reported by Badogiannis et al. (2005). Who tested concrete ranging from one to 180 days in age, produced at a W/C 0.4. He found that 10 percent replacement with metakaolin produced 282N/mm^2 which was optimal for achieving maximum long-term strength.

Table 4.4b: Compressive strength of Ahoko blended concrete

	COMPRESSIVE STRENGTH (N/mm^2)				
AGE(DAYS)	0%	5%	10%	12.5%	15%
7	55.8	276.95	314.55	160.5	114.45
28	74.05	306.7	352.45	196.05	144.85
56	80.05	202.35	356.5	198.9	121.7

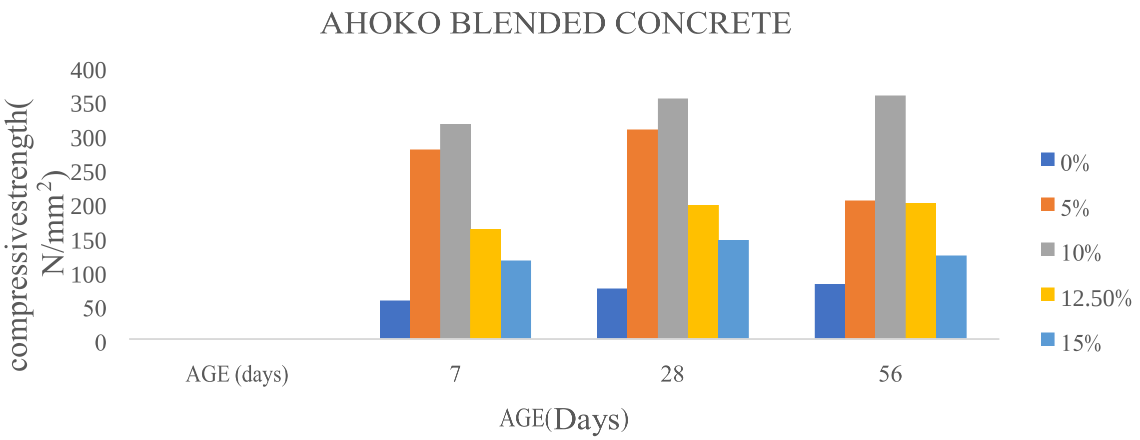


Figure 4.6: Compressive strength of Ahoko Blended concrete in relation to curing age.

4.7 Compressive Strength of Metakaolin Mortar

4.7.1 Aloji metakaolin mortar

Table 4.5 shows the compressive strength growth of Aloji metakaolin mortar, as well as the cement replacement level. The mortar was made with 5, 10, 15, and 20 percent metakaolin by weight with a (w/c) of 0.52 and shown improved strength at ages of up to 56 days. The strength enhancement caused by metakaolin in the mortar is greatest between 28 and 56 days and lowest at 7 days. The mortar containing 10 percent metakaolin had the maximum strength at 56 days, 70.7 N/mm^2. Control specimens exhibited least strengths at age 7. According to the results, the compressive strengths of the Aloji mixed mortar rise significantly as metakaolin concentration increases from 5-10 percent to 15-20 percent. Despite the fact that Cancio et al. (2017) evaluated both compressive and tensile strength of morta including MK is 9.21N/mm^2, compressive strength was shown to rise significantly as MK content increased. By three days of age, samples with 15 percent replacement had increased in strength. The strength of samples containing 10 and 15 percent Metakaolin was greater three days than the strength of the control samples. Curcio et al. (2008) reported that after three days, mortars had 8N/mm^2 achieved 79 percent of their 28-day strength

Table 4.5: Compressive strength of Aloji metakaolin mortar.

	COMPRESSIVE STRENGTH (N/mm^2)				
AGE(DAYS)	0%	5%	10%	15%	20%
7	30.07	32.9	37.8	36.83	32.9
28	42.63	59.57	62.9	53.6	45.2
56	43.8	47.53	70.7	57.03	54.4

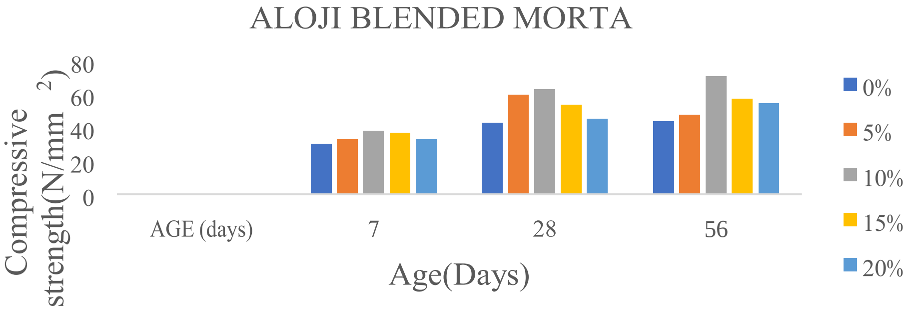


Figure 4.7: Compressive strength of Aloji blended mortar in relation to curing age.

4.7.2 Ahoko metakaolin mortar

Table 4.6 illustrates the strength development of Ahoko metakaolin mortar and cement replacement amount. The mortar was made with 5, 10, 12.5, and 15 percent metakaolin by weight with a (w/c) ratio of 0.52 and shown improved strength at ages up to 56 days. The strength augmentation of the mortar due to metakaolin is similarly stronger between 28 and 56 days, and reduced at 7 days. At 7 days and 56 days, the compressive strength of the mortar made with 10 and 12.5 percent metakaolin was 37.7 N/mm^2 and 42.3.7 N/ mm^2, respectively. Control samples showed the least strength at all ages through 56 days. Nevertheless, the concrete made with 10 and 12.5 percent metakaolin had the maximum strength of the group after 56 days. This conclusion is consistent with the findings of Curcio et al. (2008), who also investigated the evolution of strength in mortars. At ages up to 28 days, samples produced with three of the four metakaolin materials indicated faster rates of strength evolution than controls.

Table 4.6: Strength of Ahoko metakaolin mortar.

	COMPRESSIVE STRENGTH (N/mm^2)				
AGE(DAYS)	0%	5%	10%	12.5%	15%
7	26.25	32.09	37.50	37.50	36.00
28	27.55	39.95	40.60	38.0	46.20

56	30.90	37.55	42.30	42.30	42.00

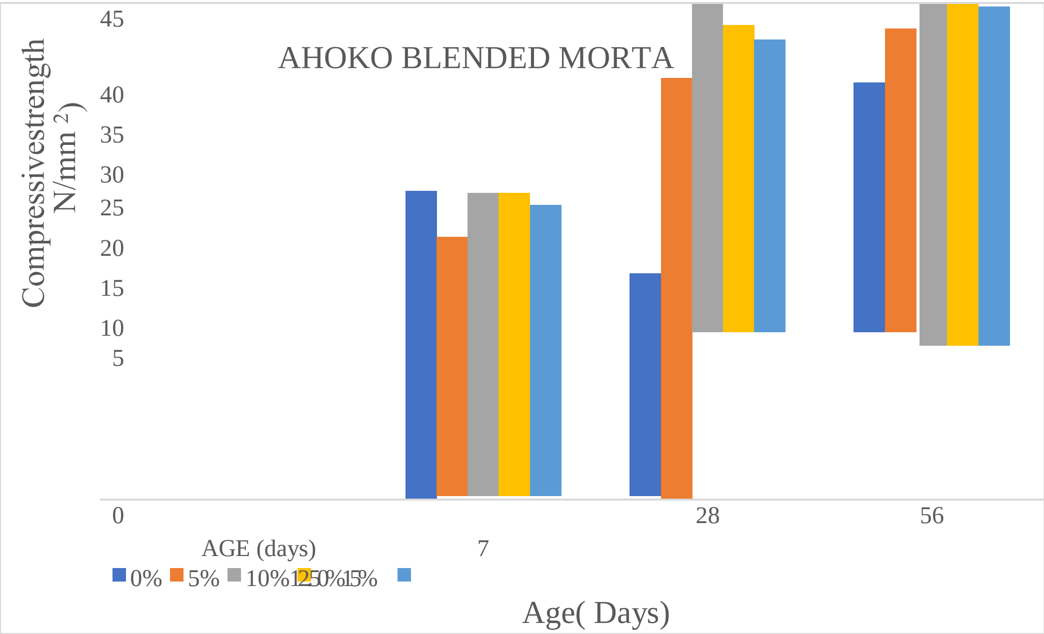


Figure 4.8: Compressive strength of Ahoko Blended mortar in relation to curing age.

4.7.3 Blended mortar 10 percent.

Table 4.7 indicates the strength development of metakaolin mortar in conjunction with metakaolin type and cement replacement amount. the Ahoko and Aloji mixed mortar was superior to the Portland cement at all ages up to 56 days. And between 28 and 56 days, metakaolin has the biggest effect on strength. The metakolinte content is one of the variables that influence metakaolin cement strength growth.

Table 4.7: Compressive strength of metakaolin mortar at 10 percent replacement.

	COMPRESSIVE STRENGTH(N/mm^2)		
AGE (Days)	PC	AHOKO	ALOJI
7 26.25 37.5 37.80 28 27.55 40.60 62.90			
56	30.90	42.30	70.7

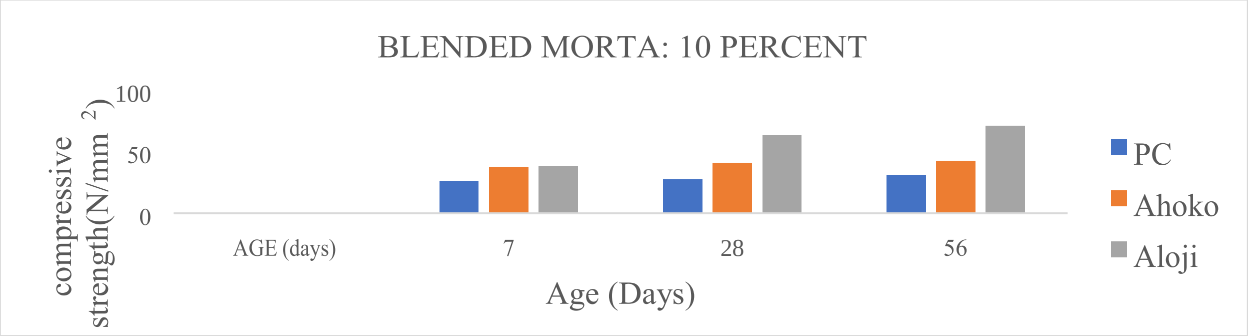


Figure 4.9: Compressive strength of metakaolin mortar at 10 percent replacement.

4.7.4 Blended concrete 10 percent.

Table 4.8 illustrates the compressive strength of metakaolin concrete at 10 percent. With a 10 percent substitution of metakaolin for Portland cement, the blended concrete of Ahoko and Aloji demonstrated greater strength than Portland cement at all ages up to 56 days. The largest increase in strength due to metakaolin occurs at 56 days. The Ahoko mixed concrete is stronger than the Aloji blended concrete.

Table 4.8: Compressive strength of metakaolin concrete at 10 percent replacement

	COMPRESSIVE STRENGTH(N/mm^2)		
AGE (Days)	PC	AHOKO	ALOJI
7	114.45	276.95	221.73
28	121.7	352.45	268.7
56	144.85	356.5	284

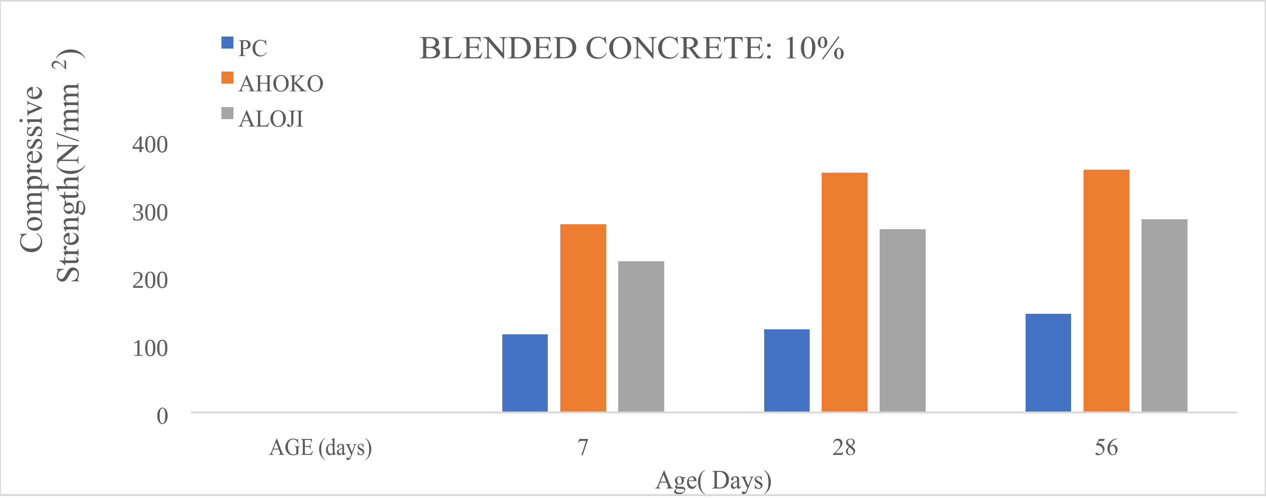


Figure 4.10: Compressive strength of metakaolin concrete at 10 percent replacement.

4.7.5 Relative strength of blended mortar (10 percent metakaolin)

Table 4.9 indicates the relative strength of blended metakaolin mortar in relation to curing age, metakaolin type, and cement replacement amount. The ratio of metakaolin cement strength to Portland cement strength is called relative strength. The ratio of metakaolin cement strength to Portland cement strength is called relative strength. the cement development rate in blended metakaolin mortar is determined by the mixture of Portland cement hydration and metakaolin pozzolanic activity. the plots of relative strength vs time provide information on the pace of reaction in the mixed metakaolin in comparison to Portland cement species. The key elements that determine metakaolin's contribution to strength, according to Khatib et al. (1996) are the filler effect, the dilution effect, and the pozzolanic reaction of metakaolin with calcium hydroxide (lime). Justice (2005) investigated Greek metakaolin and discovered significant variances in metakaolinite composition. As a result, metakolinte content is an additional variable influencing the strength development of a mixed metakaolin system. The filler effect, which brought about an early acceleration of

Portland cement hydration, is primarily responsible for the higher relative strength at 7 days for Ahoko and Aloji blended mortar. The relative strength of the Ahoko mortar is reduced at 28 days because of the dilution impact. A rise in relative strength is caused by the metakaolin's pozzolanic response. the relative strength of the Aloji mortar increased from 7 to 56 days due to the pozzolanic reaction of the metakaolin with calcium and changes in metakaolinite.

Table 4.9: Relative Strength of blended metakaolin mortar.

	RELATIVE STRENGTH	
AGE (Days)	AHOKO	ALOJI
7	1.3562	1.44
28	1.3204	2.0356
56	1.53	2.5662

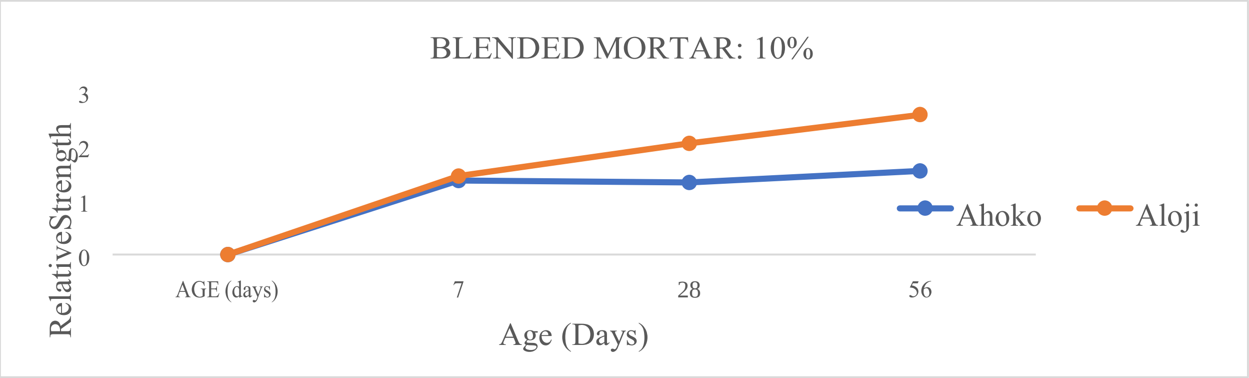


Figure 4.11: Relative Strength of metakaolin mortar at 10 percent replacement.

4.7.6. Relative strength of blended concrete (10 percent metakaolin)

Table 4.10 indicates the results, of the relative strength of both mixed metakaolin concrete at

10% replacement which increases linearly from 7 to 56 days. This demonstrated that metakaolin had a positive effect on cement strength. Ahoko and Aloji metakaolin concrete and mortar appear to benefit from a 10 percent content.

Table 4.10: Relative Strength of blended metakaolin concrete.

	RELATIVE STRENGTH	
AGE (Days)	AHOKO	ALOJI
7	2.4198	1.44
28	2.4332	2.0356
56	2.9293	2.3336

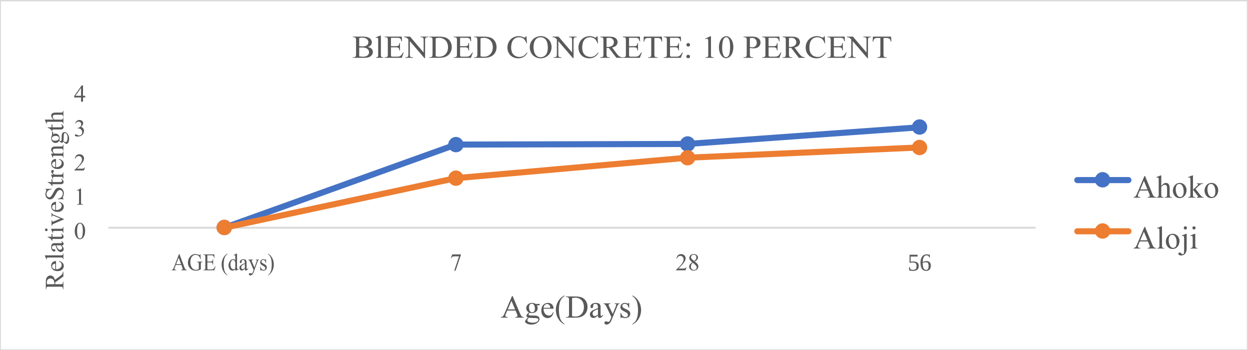


Figure 4.12: Relative Strength of metakaolin concrete at 10 percent replacement.

4.8 Hydration Rate of Blended Metakaolin Morta

The PC mortar, MKAloji-10, and MKAhoko-10 were examined using a TG analyser. The weight loss at 550 °C was compared to the total amount of water added to the mortar. the $Ca(OH)_2$ content is related to silicate hydration in the Portland cement. The pozzolanic reaction occurs when there is a decrease in calcium hydroxide in blended mortar as compared to Portland cement alone. the breakdown of $CaCO_3$ which is converted to $Ca(OH)_2$ resulted to loss in weight between 600 and 700 °C. Carbonation of the mortar can occur during the manufacturing of the mortar or during the grinding process prior to the TG measurement. Water content in hydration products besides calcium hydroxide is combined to correlate to weight loss up to 300 °C. Any variations in this value show that there were variations in the types, amounts, and relative proportions of the hydration products.

The pozzolanic reaction results in a considerable decrease in calcium hydroxide content in metakaolin samples compared to the PC control. The observed effect is related to metakaolin's increased fitness.

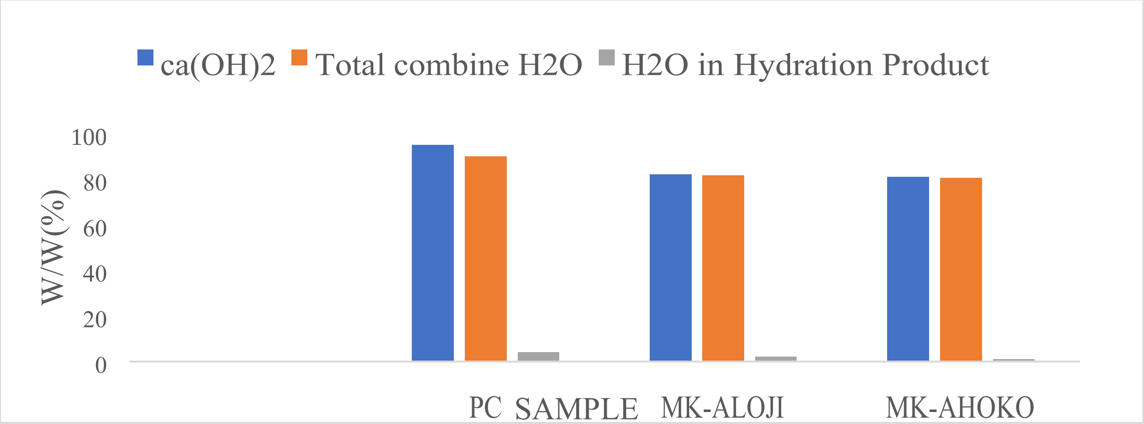


Figure 4.13: water in the hydration products, Calcium hydroxide and total combined water at hydration period of 28days

4.9 Process Flow Diagram of Metakaolin Production Plant.

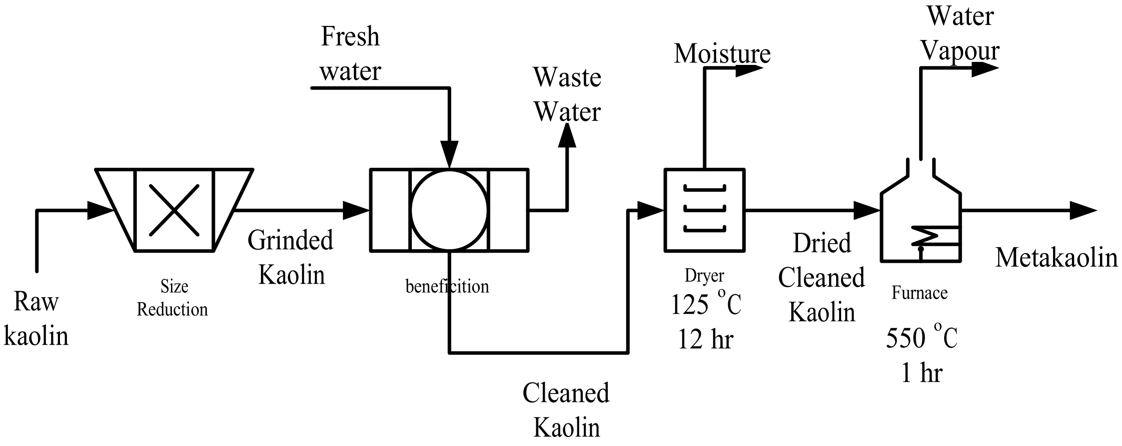


Figure 4.14: PFD of metakaolin production plant.

4.10 Material Balance

The foundation for process design is material balance. The quantities of raw materials needed and the products produced will be established by applying a material balance to the entire process. Balances over specific process units determine the process streams and compositions. A solid understanding of material balance assessment is important in process design. Material balances are useful for analysing factory operations (Sinnott, 2005).

For each of the unit operations, mass balances for every kind have been calculated as shown in appendix A. based on the law of conservation of mass. The components involved in the design include kaolin, metakaolin, impurities, steam, water. In the design, a basis of 1000 kg/batch of metakaolin was used and the corresponding quantity of water, kaolin and impurities required were determined based on specification given in the literature referenced. In order to meet the production requirement of 5 metric tons per day, a scaling up factor was determined (1000) based on the basis chosen and was used to multiply the basis (1000 kg/batch metakaolin) to achieved the target production rate (5 tone/day). Appendix A contains information on the material balance and the summary of material balance across the individual units are presented in Tables 4.11 to 4.14. From the material balance presented in Tables 4.11 – 4.14, it was estimated that 1275.49 kg/batch of raw kaolin is required to produce 1000 kg/batch of metakaolin with a five (5) batches operation per day to achieve 5 tons of metakaolin product per day.

Table 4.11: Material Balance Summary throughout the Size Reduction Unit

	Input	Output
	Inlet Stream (kg/batch)	Outlet Stream (kg/batch)
Component	Stream 1	Stream 2
Kaolin	1211.716	1211.716
Impurities	63.775	63.775
Water	0.000	0.000
Metakaolin	0.000	0.000
Steam	0.000	0.000
Total	1275.49	1275.49

Table 4.12: Material Balance Summary throughout the beneficiation unit

	Input (kg/batch)		Output (kg/batch)	
Component	Stream 2	Stream 3	Stream 4	Stream 5
Kaolin	1211.716	0.000	115.130	60.586
Impurities	63.775	0.000	1.275	62.500
Water	0.000	1913.235	478.309	1434.926
Metakaolin	0.000	0.000	0.000	0.000
Steam	0.000	0.000	0.000	0.000
Total	1275.49	1913.235	1630.714	1558.011

Table 4.13: Material Balance Summary throughout the dryer unit

	Input (kg/batch)		
Component	Stream 4	Stream 6	Stream 7
Kaolin	115.130	0.000	1151.130
Impurities	1.275	0.000	1.275
Water	478.309	430.478	47.831
Metakaolin	0.000	0.000	0.000
Steam	0.000	0.000	0.000
Total	1630.714	430.478	1200.236
		Output (kg/batch)	

Table 4.14: Material Balance Summary throughout the calcination unit

	Input (kg/batch)	Output (kg/batch)	
Component	Stream 7	Stream 8	Stream 9

Kaolin	1151.130	0.000	57.557
Impurities	1.275	0.000	1.275
Water	47.831	0.000	0.000
Metakaolin	0.000	0.000	941.168
Steam	0.000	200.493	0.000
Total	1200.236	200.493	1000.000

4.11 Energy Balance Calculation

Energy, like mass, can be regarded preserved in all but nuclear processes. However, energy conservation varies from mass conservation in that energy can be produced or used in a chemical process. If energy is generated or used in the operation, the total enthalpy of the output stream will not be equivalent to that of the entrance stream. Energy balances are used in process design to estimate a process's energy requirements, such as the amount of heating, cooling, and power needed. For this design, the following assumptions are made in the course of performing energy balance:

i. State operation prevails throughout the plant.
ii. Heat loss in the course of energy balance is neglected.
iii. Pressure effect on enthalpy is also neglected. iv. Heat capacity for all components is at a given operating temperature.
v. Reference temperature is assumed 25^{o}C.

Appendix B contains information on the material balance and the summary of material balance across the individual units are presented in Table 4.15 to 4.16. Table 4.15 shows that 10744.847 kJ/batch of energy is required to achieve desired drying while Table 4.16 shows that 5486688.546 kJ/batch is required to achieve 95 percent dehydroxylation of kaolin to metakaolin at 550 ^{o}C.

Table 4.15: Summary of energy Balance across the dryer unit

	In ut (kJ/batch)	Output (kJ/ba ch)	
Component	Stream 4	Stream 6	Stream 7
Kaolin	2175.636	0	108781.815
Impurities	2.010	0	100.815
Water	3137.742	140560.337	15617.815
Metakaolin	0	0	0
Steam	0	0	0
Total	5315.388	140560.337	124500.101

Table 4.16: Summary of energy Balance across the calcination unit

	Input (kJ/batch)	Output (kJ/batch)	
Component	Stream 7	Stream 8	Stream 9
Kaolin	- 26204146.725	0	- 1287091.209
Impurities	3515.919	0	3943.081
Water	- 743238.42	0	0
Metakaolin	0	0	- 20464888.492
Steam	0	290855.940	0
Total	- 26943869.2261	290855.939	- 21748036.6196

4.12 Fabricated Metakaolin Equipment and Specification

4.12.1 Insulation material and refractory

Constant energy is needed to maintain a certain temperature, and insulation lowers this energy loss. The thermal conductivity of the material in question as well as the temperature drive determine the rate at which heat transfer happens. Standard laboratory kilns can be equipped with a variety of options, including plain tubes in ceramic, quartz, or heatingresistant metal, shaped vessels in metal or quartz, and incorporating agitation blades. The proper choice of cylindrical material is determined by process temperature, process compatibility, and physical size.

4.12.2 Constructed material

Mild steel is utilized for the building of steel supports since it is the least expensive of the steel groups obtainable. It is tough, ductile, malleable and has good tensile strength and easy to use in fabrication, although it has a lesser corrosion resistance property as compared to other steels, it’s generally used in processes that are mildly corrosive (Gavin and Sinnott, 2008). It possesses a melting point between 1300 ^{o}C to 1520 ^{o}C and a tensile strength of 430 MPa), so that it can withstand the working condition utilised in this study while galvanised steel is selected as the material of construction for the cylindrical rotary kiln (Sinnott, 2005).

4.12.3 Fabrication of rotary calciner

The rotary calciner was fabricated using carbon steel material of construction while the galvanized steel was used for the external cover. 60 percent raw kaolin, 15 percent fired kaolin, 20 percent plasticized and talc clay were used to developed the rotary calciner refractory and insulator. The insulator thickness utilised is 120 mm to minimized heat loss from the refractor to the environment. The fabrication of the calciner was carried out according to design specification and dimensions.

4.13 Performance Evaluation of the Fabricated Furnace

4.13.1 Inspection and testing

All parts of the fabricated furnace (from the stand/support, motor seat, bearing seat & support, rotary gear & motor, mechanical parts, casing and insulating materials) were inspected to ensure proper connection. To eliminate any electrical bridges, all electrical connections, and equipment were verified. the temperature regulator and controller were tested for 2 hours after the furnace was turned on with a maximum temperature above 500 ^{o}C.

The temperature was checked in the furnace. the reading obtained from the temperature regulator is considered authentic because it is digital. An external probe is used to calibrate the temperature controller, ensuring accurate temperature readings.

4.13.2 Operation procedure

The furnace was powered on, and the rotary motor's knob was switch on, the furnace was heated to 500 ^{o}C, and the material to be heated was placed in the rotary furnace while adhering to safety precautions and holding for the design reaction time (1 hr), after which the equipment was shut down and detached from the main power supply, and the generated metakaolin was removed from the calciner's product line for fumigation.

4.13.3 Performance evaluation

The effectiveness of the furnace was measured using the functioning of the furnace, extent of dehydroxylation, maintainability, safety, and cost analysis.

4.13.4 Furnace functionality

The effectiveness of temperature sensing was also evaluated. The temperature is checked on a regular basis using an external device to calibrate the temperature controller, ensuring accurate temperature reading. Applying a kaolin clay insulator improves the prevention of heat loss from the calciner walls.

4.13.5 Heating rate

The heating rate of the furnace is a measure of the time required to reach the specified temperature. The temperature of the rotary furnace was raised to 550 ^{o}C in 113 minutes. This test protocol was done five times in a row, with the same average needed to achieve the temperature of 600 ^{o}C each time. This heating rate of 600 ^{o}C/113 minutes is comparable to 5 ^{o}C/min (0.0811 ^{0}C/sec). This heating rate is slightly lower than that of commercially available muffle furnaces, such as those developed by the Park Thermal Equipment Company in Canada, that require 150 minutes to heat up to a temperature of 870 ^{o}C, corresponding to a heating rate of 5.8 ^{o}C/min (0.096 ^{o}C/s) (Alaneme and Bodunrin, 2017). When compared to regular furnaces of various makes, the manufactured rotary furnace is said to be more effective in service. The rotation of rotary calciner/furnace, also aide in almost uniform temperature distribution in the calciner.

The capacity to maintain a steady temperature and sensitivity is significant in the performance evaluation test (Alaneme and Bodunrin, 2017). The effectiveness in preventing heat loss to the environment was assessed by measuring the temperature variation while the furnace was running. the furnace heating environment has temperature changes in the steam generated during the reaction may have caused a slight pressure build-up in the calciner.

However, the rise in pressure in the calciner is not significant to the extent that it threatens the safety of the operator and the fabricated furnace.

4.13.6 Fabricated furnace maintenance

Simple routine upkeep and adherence to safety precautions provided for furnace operations increase the durability of service life and effectiveness of the constructed rotary calciner. After each operation, thoroughly clean the entire constructed calciner, as well as all metallic parts that made contact with the starting material or steam generated during the operation. The use of an external digital temperature sensor at regular intervals (after 5 operations) helps to calibrate and ensure the correctness of the temperature measurements. The outer casing of the fabricated calciner is also coated using heat resistant paint to prevent against corrosion.

4.13.7 Operational safety measures

The manufactured furnace is placed in a well-ventilated location to limit the chance of dehydration caused by a high ambient temperature rise to a maximum of 39 ^{o}C. there is no risk of heat radiation in the calciner environment. The calciner was constructed to prevent leakage or emission and does not damage the environment with any type of pollutants or cause skin burns.

Equipment design specifications obtained from the design calculation for calciner unit are presented in Table 4.17.

Table 4.17:

PARAMETER	DIMENSION
Type	Rotary
Function	Meta kaolinization
Operation	Batch
Batch time	2 hrs
Reactor type	Electric Heater Rotary Kiln
Rotary Kiln Reactor Volume	0.45486 X 3 /batch
Reactor Length (mm)	460 by 3
Reactor Diameter (mm)	100 by 3
Rotary Kiln Reactor thickness (mm)	60 by 3
Gear power	1.928 x 3 hp
Reactor Controller	Digital temperature controller
Operating temperature	500^0 C
Maximum operating temperature	900^0 C
Material Of Construction	Mild Steel
Rotary Kiln Refracting Heating Element	Kanthal Super 1900
Pinion Rotation speed	60 r p m
Number of teeth	36
Pinion Material	S A E 1040/ 245 B H D
Gear Operating Period	14 hours/ day
Gear Material	Cast Steel 035/045 and carbon 210
Refractory Material of construction	Catable refractory Material
Heating element	Kanthal super 1900

Summary of Fabricated metakaolin Equipment and Specification

Temperature sensor	k- type

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

When Metakaolin mixed with Portland cement, it exhibits a remarkable pozzolanic behaviour.

- The metakaolin examined had a very positive influence on cement strength after 7 days, particularly at 28 and 56 days. Mixed cement requires considerably more water than comparably pure cement, with the water demand rise reaching 10 percent replacement.
- The pozzolanic reaction of metakaolins increases between 7 and 28 days, followed by a significant drop in $Ca\ (OH)_2$.
- Based on the mechanical and physical qualities of metakaolin cements, a 10 percent metakaolin content is recommended. content seems to be, generally, more favourable than the other percentages replacement.
- It was estimated from the material balance, that 12756 kg/batch of raw kaolin is required to produce 1000 kg/batch of metakaolin with a five (5) batches operation per day to achieve 5 tons of metakaolin product per day.
- 1t was Also estimated from the energy balance, that 5486688.546 kJ/batch is required to achieve 95% dehydroxylation of kaolin to metakaolin at 550 ^{o}C.
- The furnace design specifications are: rotary furnace, material of construction- mild steel, Batch time-2hrs, batch operation, reactor type – rotary kiln, reactor volume per batch,

length, diameter, thickness is 1.36458L,1380mm,300mm,180mm respectively. Minimum and maximum operating temperature is 500 ^oC and 900 ^oC respectively.

5.2 Recommendations

The following are my recommendations

1. Optimization of the cement replacement percentage should be extended to 50 percent by other researchers
2. The Partial replacement of clinker by Metakaolin and fine limestone at it ground state should be done by other researchers.

5.3 Contribution to Knowledge

The study found that an indigenous kaolin (Ahoko and Aloji) from Kogi State Nigeria, produced metakaolin at a temperature of 550 ^oC and they are good pozzolans for improving compressive strength of Portland cement in building construction. The average compressive strength of mortar and concrete produced were 56.5 N/mm^2 and 320.25 N/mm^2 respectively better than the average compressive strength gotten from others researchers from literature (8.60N/mm^2 for mortars and 299N/mm^2 for concrete). However, the optimum percentage replacement ratio of cement to metakaolin is 1:1.

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APPENDIX A

A.0 MATERIAL BALANCE

Definition: Species indices in the order Kaolin, Dirty (as ordinary clay), Water,

Metakaolin, Steam. A.1 Stream initialization:

s □□ 1□□ 5 ORIGIN □ 1 j □□ 1□□ 9 kmol □□ 1000□mol

Mass flow rate (Amount) of a component s in a stream j is:

kg

n_s j□□ □□ 0□

$\overline{}$
hr

Total Mass flow rate (Total Amount) of a stream j is:

kg
N_j □□ 0□$\overline{}$
hr

Mass composition of a stream s in a stream j is: x_s

j□□ □□ ^{0}

Molar flow rate of a component s in a stream j is:

kmol
z_s j□□ □□ 0□$\overline{}$
hr

Total Molar flow rate of a stream j is:

kmol
Z_j □□ 0 $\overline{}$
hr

Molar composition of a component s in stream j is:

y_s j□□ □□ ^{0} kJ □ 1000□J

kg kJ
Input_j □□ 0□ $\overline{}$ □ s □□ 0□$\overline{}$
hr kmol

A.2 Molecular weights of Components

The Molecular weights and densities of components in kg/kmol is:

	Mw_s	$density_s$
Kaolin	258.1	2650
Dirty clay	76.083	890
Water	18.0153	997
Metakaolin	222.13	1005
Steam	18.0153	2.95

$Mw_s := Mw_s \cdot \frac{kg}{kmol}$ $\rho_s := density_s \cdot \frac{kg}{m^3}$

A.3 Design Consideration

This design project is aimed at producing 5 tons metakaolin/day, which is equivalent to 5 batches of 1000 kg/batch of metakaolin. Considering the low-capacity nature of the plant, together with the flexibility associated with batch process for such low production capacity, a batch process is selected for the choice of method

A.4 Batch Time

In batch operation there will be periods when product is being produced (productive period), followed by non-productive periods when the product is discharged and the equipment prepared for the next batch. The rate of production will be determined by the total batch time, productive plus non-productive periods (Sinnott, 2005).

A.4.1 Productive period

This includes the period for loading of feed material and preparing the system.

A.4.2 Loading time

This includes feed pre-treatment and loading time

$t_{loading} := 30\,min$

A.4.3 Calcination time

Smaller plants often use batch reactors. Kaolin is feed in to the calciner and calcined for approximately one hour at 550°C.

$t_{calcination} := 60\,min = 1\cdot hr$

A.4.4 Total productive time

$t_{productive} := t_{loading} + t_{calcination} = 1.5\cdot hr$

A.4.5 Non-productive time

This includes product purification and treatment where need, discharged and equipment preparation for the next batch. This nonproductive time is considered as 30 min

$t_{non\_productive} := 30\,min$

Therefore, the total batch time for the calcination of a batch is

$t_{batch} := t_{productive} + t_{non\_productive} = 2\cdot hr$

$batch = 2\,hr$

Target metakaolin production capacity per batch

kg

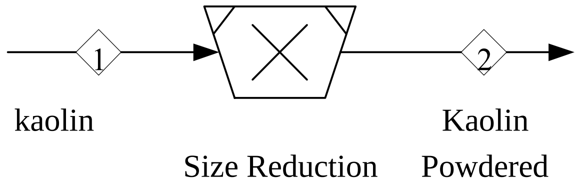
$\text{Target\_Capacity}\square\square\ 1000\ \frac{}{\text{batch}}$

Basis:

Feed Rate of kaolin:

$$\text{feed}\ \ \square\square\ \ \text{SF}\square 1000\ \frac{\text{kg}}{\text{batch}}$$

A.5.1 Size Reduction



Assumptions

1. 100% material recovery
2. The feedstock is expected to contain impurity (dirty clay) of not more than 5% percentage purity. This design considers 5% impurity in the kaolin.

Stream 1

Feedstock compositions of stream 1 in terms of mass fraction

$X1\ 1\square\square\ \square\square\ 0.95$ $\qquad$ $X2\ 1\square\square\ \square\square\ 0.05$

$\square\ \text{Kaolin}\ \square\ xs\ \square\square^{1}\ \square$

$$\begin{array}{l} \\ \text{Dirty clay(} \\ \\ \text{Water} \\ \\ \text{Metakaolin} \\ \\ \text{Steam} \end{array} \begin{pmatrix} 0.95 \\ 0.05 \\ 0 \\ 0 \\ 0 \end{pmatrix})$$

Total mass fraction of stream 1

$$x1 := \sum_{s} xs_{,1} = 1$$

Mass flow rates of components in feedstock

$$ns_{,1} := xs_{,1} \cdot feed$$

$$ns_{,1} = \begin{array}{l} \text{Kaolin} \\ \text{Dirty clay( )} \\ \\ \text{Water} \\ \\ \text{Metakaolin} \\ \\ \text{Steam} \end{array} \text{kg} \begin{pmatrix} 1211.716 \\ 63.775 \\ 0 \\ 0 \\ 0 \end{pmatrix} \frac{}{\text{batch}}$$

Total mass flow rate of stream 1

$$N1 := \sum_{s} ns_{,1} = 1275.49 \frac{\text{kg}}{\text{batch}}$$

Molar flow rates of components in stream 1

$$zs\,\Box\Box_{1}\,\Box\Box\,\frac{ns\,\Box\Box_{1}}{Mw_{s}}$$

zs □□1 □

□ Kaolin	4.695	□kmol
□ Dirty clay( )	0.838	□ □batch
□ □	0	
□ Water □	0	
□ □ Metakaolin	0	
□ □		
□ Steam □		

Total molar flow rate of stream 1 is

$$Z1\,\Box\Box\,\sum_{s}\Box\, zs\,\Box\Box^{1}\,\Box\,5.53298\,\Box\,\frac{kmol}{batch}$$

Molar composition of components in stream 1 is

$$ys\,\Box\Box_{1}\,\Box\Box\,\frac{zs\,\Box\Box_{1}}{Z1}$$

□ Kaolin □ ys □□^{1} □

□ □
Dirty clay( )
□ □
□ Water □
□ □
Metakaolin
□ □
□ Steam □

0.8485
0.1515
0
0
0

Total mole fraction of component stream 1 is

y1 □□ □ ys □□$_1$ □ 1
s

Stream 2

No separation taking place in the size reduction unit, hence, input is equal to output Mass flow rates of components in stream 2 is:

ns □□2 □□ ns □□1

ns □□2 □
□ Kaolin □kg
□ Dirty clay( ) □ □batch
□ □
□ Water □
□ □
Metakaolin
□ □
□ Steam □

1211.7157
63.77451
0
0
0

Total amount of stream 2 is:

$$N2 := \sum_{s} ns_{s,2} = 1275.49 \, \frac{kg}{batch}$$

Mass Composition of components in stream 2 is:

$$xs_{s,2} := \frac{ns_{s,2}}{N2}$$

$$\begin{pmatrix} Kaolin \\ \text{Dirty clay} \\ Water \\ Metakaolin \\ Steam \end{pmatrix} \quad xs_{s,2} = \begin{pmatrix} 0.95 \\ 0.05 \\ 0 \\ 0 \\ 0 \end{pmatrix}$$

Total mass fraction of component in stream 2

$$x2 := \sum_{s} xs_{s,2} = 1$$

Molar flow rates of components in stream 2 is:

$$zs_{s,2} := \frac{ns_{s,2}}{Mw_{s}}$$

$$zs_{s,2} =$$

$$\begin{pmatrix}\text{Kaolin}\\ \text{Dirty clay( )}\\ \text{Water}\\ \text{Metakaolin}\\ \text{Steam}\end{pmatrix} \begin{array}{|r|}\hline 4.69475\\ \hline 0.83822\\ \hline 0\\ \hline 0\\ \hline 0\\ \hline\end{array} \frac{\text{kmol}}{\text{batch}}$$

Total molar flow rate of stream 2 is

$$Z2 = \sum_{s} zs(2) = 5.53298\,\frac{\text{kmol}}{\text{batch}}$$

Molar composition of components in stream 2 is

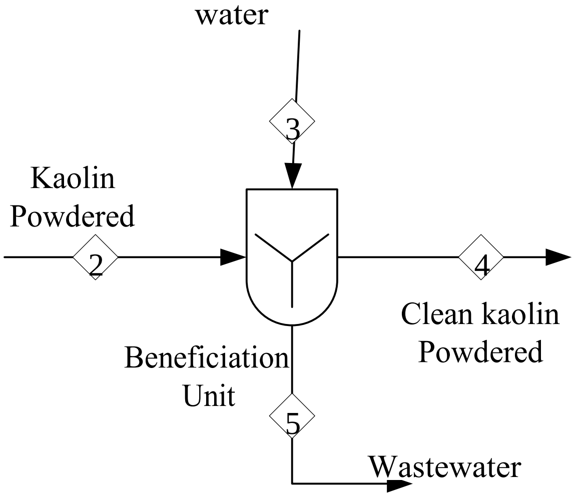
$$ys(2) = \frac{zs(2)}{Z2}$$

$$\begin{pmatrix}\text{Kaolin}\\ \text{Dirty clay( )}\\ \text{Water}\\ \text{Metakaolin}\\ \text{Steam}\end{pmatrix} ys(2) = \begin{array}{|r|}\hline 0.8485\\ \hline 0.1515\\ \hline 0\\ \hline 0\\ \hline 0\\ \hline\end{array}$$

The total mole fraction of component in stream 2 y2 □□ □ ys □□$_2$ □ 1

s

A.5.2 Beneficiation Unit



The powdered kaolin is beneficiated in order to obtain pure kaolin free of dirty by addition of water to wash off all the dirty and impurities using a water flow rate of 1.5 times the flowrate of raw kaolin

Assumptions

1. 100% material recovery
2. 5% of kaolin is recovered in stream 4
3. 98% of dirty is recovered in stream 5
4. 70% of water is recovered in stream 5

Stream 3

Mass flow rates of components in stream 3 n3 3□□ □□ 1.5□N1

ns □□3 □

□ Kaolin □kg

□ Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

0
0
1913.23532
0
0

□ □batch

Total mass flowrate of stream 3

kg

N3 □□ □ ns □□3 □ 1913.235□ batch

s

Mass composition of components in stream 3

ns □□3

xs □□3 □□ ——

N3

□ Kaolin □ xs □□3 □

□ □

Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

0
0
1
0
0

Total mass fraction oc component in stream 3

$$x3 := \sum_{s} xs^{\langle 3\rangle} = 1$$

Molar flow rates of components in stream 3

$$zs^{\langle 3\rangle} := \frac{ns^{\langle 3\rangle}}{Mw_{s}}$$

$$zs^{\langle 3\rangle} = \begin{array}{l} \text{Kaolin} \\ \text{Dirty clay( )} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{array} \begin{pmatrix} 0 \\ 0 \\ 106.20058 \\ 0 \\ 0 \end{pmatrix} \frac{\text{kmol}}{\text{batch}}$$

Total molar flow rate of stream 3

$$Z3 := \sum_{s} zs^{\langle 3\rangle} = 106.20058 \frac{\text{kmol}}{\text{batch}}$$

Molar composition of components in stream 3

$$ys^{\langle 3\rangle} := \frac{zs^{\langle 3\rangle}}{Z3}$$

□ Kaolin □ ys □□^{3} □

□ □

Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

0
0
1
0
0

Total mole fraction of component in stream 3

y3 □□ □ ys □□$_3$ □ 1

s

Stream 4

Mass flow rates of components in stream 3

n1 4□□ □□ 95% n□ □ 1 2□□ □

n2 4□□ □□ 2% n□ □ 2 2□□ □

n3 4□□ □□ 25% n□ □ 3 3□□ □

ns □□4 □

□ Kaolin □kg

□ Dirty clay( ) □ □batch

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

1151.12992
1.27549
478.30883
0
0

Total mass flowrate of stream 4

$$N4 \square\square \underset{s}{\square} ns \square\square 4 \square 1630.714 \square \frac{kg}{batch}$$

Mass composition of components in stream 4

$$xs \square\square 4 \square\square \frac{ns \square\square 4}{N4}$$

$$\begin{array}{l|r} \square\ \text{Kaolin}\ \square\ xs & 0.70591 \\ \square \qquad \square\ \text{Dirty clay(} & 0.00078 \\ \square \qquad \square & 0.29331 \\ \square\ \text{Water}\ \square & 0 \\ \square \qquad \square\ \text{Metakaolin} & 0 \\ \square \qquad \square & \\ \square\ \text{Steam}\ \square & \end{array} \ \square\square^{4} \square \ )$$

Total mass fraction of component in stream 4

$$x4 \square\square \underset{s}{\square} xs \square\square 4 \square\ 1$$

Molar flow rates of components in stream 4

$$zs \square\square 4 \square\square \frac{ns \square\square 4}{Mw}$$

$$s\ zs \square\square 4$$

$$\square$$

$$\begin{bmatrix} \text{Kaolin} \\ \text{Dirty clay( )} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{bmatrix} \begin{array}{|r|} \hline 4.46002 \\ \hline 0.01676 \\ \hline 26.55015 \\ \hline 0 \\ \hline 0 \\ \hline \end{array} \; \frac{\text{kmol}}{\text{batch}}$$

Total molar flow rate of stream 4

$$Z4 = \sum_{s} zs^{(4)} = 31.02692 \, \frac{\text{kmol}}{\text{batch}}$$

Molar composition of components in stream 4

$$ys^{(4)} = \frac{zs^{(4)}}{Z4}$$

$$\begin{bmatrix} \text{Kaolin} \\ \text{Dirty clay(} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{bmatrix} ys \begin{array}{|r|} \hline 0.14375 \\ \hline 0.00054 \\ \hline 0.85571 \\ \hline 0 \\ \hline 0 \\ \hline \end{array} \;)^{(4)}$$

Total mole fraction of component in stream 4

y4 □□ □ ys □□4 □ 1
s

Stream 5

Mass flow rates of components in stream 5

n1 5□□ □□ 5% n□□ 1 2□□ □ n2 5□□ □□ 98% n□□ 2 2□□ □

n3 5□□ □□ 75% n□□ 3 3□□ □

ns □□5 □

□ Kaolin □kg

□ Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

60.58579
62.49902
1434.92649
0
0

□ □batch

Total mass flowrate of stream 5

kg

N5 □□ □ ns □□5 □ 1558.011□ batch
s

Mass composition of components in stream 5

ns □□5
xs □□5 □□ ——
N5

$$xs_{s,5} = \left(\begin{array}{l|r} \text{Kaolin} & 0.03889 \\ \text{Dirty clay( )} & 0.04011 \\ \text{Water} & 0.921 \\ \text{Metakaolin} & 0 \\ \text{Steam} & 0 \end{array}\right)$$

Total mass fraction of component in stream 5

$$x5 := \sum_{s} xs_{s,5} = 1$$

Molar flow rates of components in stream 5

$$zs_{s,5} := \frac{ns_{s,5}}{Mw_s} \quad zs_{s,5}$$

$$= \left(\begin{array}{l|r} \text{Kaolin} & 0.23474 \\ \text{Dirty clay( )} & 0.82146 \\ \text{Water} & 79.65044 \\ \text{Metakaolin} & 0 \\ \text{Steam} & 0 \end{array}\right) \frac{\text{kmol}}{\text{batch}}$$

Total molar flow rate of stream 5

$$Z5 = \sum_{s} zs_{5} = 80.70663 \frac{kmol}{batch}$$

Molar composition of components in stream 5

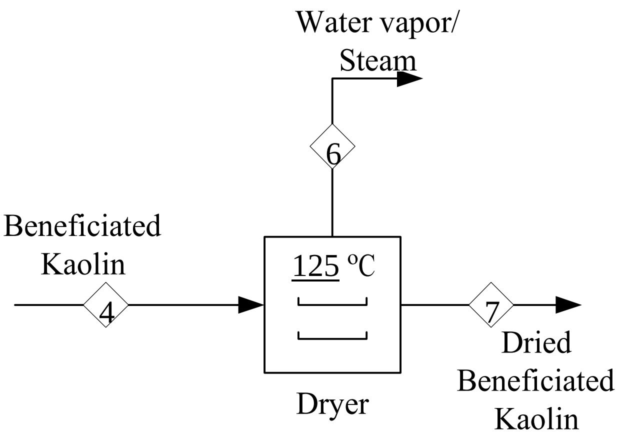
$$ys_{5} = \frac{zs_{5}}{Z5}$$

$$ys_{5} = \left(\begin{array}{l|r} \text{Kaolin} & 0.00291 \\ \text{Dirty clay} & 0.01018 \\ \text{Water} & 0.98691 \\ \text{Metakaolin} & 0 \\ \text{Steam} & 0 \end{array} \right)$$

Total mole fraction of component in stream 5

$$y5 = \sum_{s} ys_{5} = 1$$

A.5.3 Dryer



The beneficiated powdered kaolin is expected to be dried in a dryer to obtain dried beneficiated kaolin to have not more than 5% water in the dried product.

Assumptions

1. 100% material recovery
2. 0% of kaolin is recovered in stream 6
3. 0% of dirty is recovered in stream 6
4. 90% of water is recovered in stream 6

Stream 6

Mass flow rates of components in stream 6

n1 6□□ □□ 0% n□ □
1 4□□ □

n2 6□□ □□ 0%
n□ □ 2 4□□ □

n3 6□□ □□ 90%
n□ □ 3 4□□ □

ns □□6 □

$\boxed{0}$

$$\begin{pmatrix} \text{Kaolin} \\ \text{Dirty clay( )} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{pmatrix} \text{kg} \begin{array}{|r|}\hline 0 \\ \hline 430.47795 \\ \hline 0 \\ \hline 0 \\ \hline \end{array} \frac{\quad}{\text{batch}}$$

Total mass flowrate of stream 6

$$N6 := \sum_{s} ns^{\langle 6 \rangle} = 430.478 \frac{\text{kg}}{\text{batch}}$$

Mass composition of components in stream 6

$$xs_{\langle 6 \rangle} := \frac{ns^{\langle 6 \rangle}}{N6}$$

$$\begin{pmatrix} \text{Kaolin} \\ \text{Dirty clay(} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{pmatrix} xs^{\langle 6 \rangle} = \begin{array}{|r|}\hline 0 \\ \hline 0 \\ \hline 1 \\ \hline 0 \\ \hline 0 \\ \hline \end{array})$$

Total mass fraction of component in stream 6

$$x_6 := \sum_{s} x_{s,6} = 1$$

Molar flow rates of components in stream 6

$$z_{s,6} := \frac{n_{s,6}}{Mw_s}$$

$$z_{s,6} = \begin{pmatrix} \text{Kaolin} \\ \text{Dirty clay( )} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{pmatrix} \begin{array}{|r|}\hline 0 \\ \hline 0 \\ \hline 23.89513 \\ \hline 0 \\ \hline 0 \\ \hline \end{array} \frac{\text{kmol}}{\text{batch}}$$

Total molar flow rate of stream 6

$$Z_6 := \sum_{s} z_{s,6} = 23.89513 \frac{\text{kmol}}{\text{batch}}$$

Molar composition of components in stream 6

$$y_{s,6} := \frac{z_{s,6}}{Z4}$$

$$y_{s,6} = \begin{pmatrix} \text{Kaolin} \\ \text{Dirty clay} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{pmatrix} \begin{pmatrix} 0 \\ 0 \\ 0.77014 \\ 0 \\ 0 \end{pmatrix}$$

Total mole fraction of component in stream 6

$$y_6 = \sum_s y_{s,6} = 0.77014$$

Stream 7

Mass flow rates of components in stream 7

$n_{1,7} = 100\%\, n_{1,4}$ $\qquad$ $n_{2,7} = 100\%\, n_{2,4}$

$n_{3,7} = 10\%\, n_{3,4}$

$$n_{s,7} = \begin{pmatrix} \text{Kaolin} \\ \text{Dirty clay} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{pmatrix} \begin{pmatrix} 1151.12992 \\ 1.27549 \\ 47.83088 \\ 0 \\ 0 \end{pmatrix} \frac{\text{kg}}{\text{batch}}$$

Total mass flowrate of stream 7

$$N7 := \sum_{s} ns^{\langle 7 \rangle} = 1200.236 \, \frac{kg}{batch}$$

Mass composition of components in stream 7

$$xs^{\langle 7 \rangle} := \frac{ns^{\langle 7 \rangle}}{N7}$$

$$\begin{array}{l} \text{Kaolin} \\ \text{Dirty clay} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{array} \; xs^{\langle 7 \rangle} = \left(\begin{array}{r} 0.95909 \\ 0.00106 \\ 0.03985 \\ 0 \\ 0 \end{array} \right)$$

Total mass fraction of component in stream 7

$$x7 := \sum_{s} xs^{\langle 7 \rangle} = 1$$

Molar flow rates of components in stream 7

$$zs^{\langle 7 \rangle} := \frac{ns^{\langle 7 \rangle}}{Mw}$$

s zs □□7

□

□ Kaolin	4.46002	□kmol
□ Dirty clay( )	0.01676	□ □batch
□ □	2.65501	
□ Water □	0	
□ □	0	

Metakaolin

□ □

□ Steam □

Total molar flow rate of stream 7

$$Z7 \;□□\; \underset{s}{\boxed{}}\; zs\;□□^{7}\;□\;7.13179\;□\;\frac{kmol}{batch}$$

Molar composition of components in stream 7

$$ys\;□□_{7}\;□□\;\frac{zs\;□□7}{Z7}$$

□ Kaolin □ ys		□□^{7} □
□ □	0.62537	
Dirty clay(	0.00235	)
□ □	0.37228	
□ Water □	0	
□ □	0	

Metakaolin

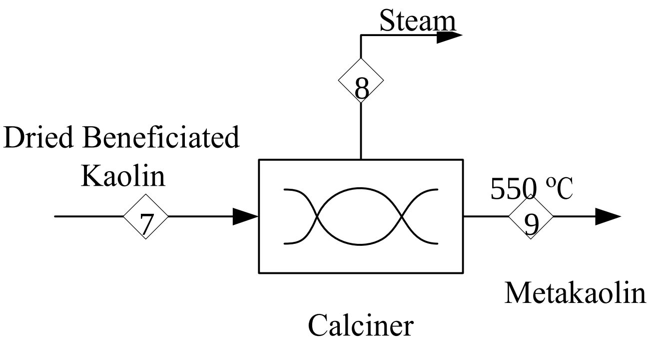
□ □

□ Steam □

Total mole fraction of component in stream 7

y7 □□ □ ys □□7 □ 1
s

A.5.4 Calciner Unity



Assumption

1. 100% material recovery

Stream 8

The dehydroxylation reaction in the calciner takes place at 550 ºC according to the reaction below;

Al2SiO5(OH)4 ========> Al2Si2O7 + 2H2O

(kaolin) (Metakaolin) (steam)

The degree of dehydroxylation of kaolin to metakaolin reported in the literature is 80 - 97% for a calcination period of 45 - 90 min. This design considers a 95% conversion of kaolin to metakaolin

Kaolin converted

kaolinconverted □□ 95% z□□ 1 7□□ □
Unconverted kaolin

kmol

kaolinunconverted□□ 5% z□□ 1 7□□ □ □ 0.223□——batch

z1 9□□ □□ kaolinunconverted

From the stoichiometry of the reaction above, 1 mole of kaolin produces 1 mole of metakaolin, hence

metakaolinproduced □□ kaolinconverted

z4 9□□ □□ metakaolinproduced

From the stoichiometry of the reaction above, 1 mole of kaolin produces 2 mole of water as steam, hence

Steamproduced □□ 2□kaolinconverted

□z5 8□□ □ □□ Steamproduced □ z3□□7

Other component that enters the calciner and do not partake in the reaction such as the trace dirty will also leave with the solid stream

z2 9□□ □□ z2□□7

Stream 8

Molar flow rates of components in stream 8

□

□ Kaolin □

□ □

Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

zs □□8

0	kmol batch
0	
0	
0	□——
11.12904	

Total molar flow rate of stream 8

kmol

Z8 □□ □ zs □□^{8} □ 11.12904□——batch

s

Molar composition of components in stream 8

zs □□8

ys □□$_8$ □□ ——

Z8

□ Kaolin □ y □□^{8} □

s

□

□ Kaolin □

□ □

Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

□ □

Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

0
0
0
0
1

Total mole fraction of component in stream 8

y8 □□ □ ys □□8 □ 1
s

Mass flow rates of components in stream 8

ns □□8 □□ zs □□8□Mw
s

ns □□8

0
0
0

□

□ Kaolin □

□ □

Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

0	kg batch
200.49305	

□___

Total mass flowrate of stream 8

kg

N8 □□ □ ns □□^{8} □ 200.493□___batch

s

Mass composition of components in stream 8

ns □□8

xs □□$_8$ □□ ___

N8

□ Kaolin □ xs □□^{8} □

□

□ Kaolin □

□ □

Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

□ □

Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

0
0
0
0
1

Total mass fraction of component in stream 8

x8 □ □ $\boxed{}_{s}$ xs □ □8 □ 1

Stream 9

Molar flow rates of components in stream 9

zs □ □9

0.223
0.01676
0
4.23701
0

kmol batch

□

□

□ Kaolin □
□ □
Dirty clay( )
□ □
□ Water □
□ □
Metakaolin
□ □
□ Steam □

Total molar flow rate of stream 9

$$Z9 \;\square\square\; \sum_{s} \; zs\,\square\square^{9} \;\square\; 4.47678\,\square \frac{\text{kmol}}{\text{batch}}$$

Molar composition of components in stream 9

$$ys\,\square\square_{9}\;\square\square\;\frac{zs\,\square\square_{9}}{Z9}$$

□ Kaolin □ $y\,\square\square^{9}$ □

0.04981
0.00374
0
0.94644
0

s

□ □
Dirty clay( )
□ □
□ Water □

□

□ Kaolin □
□ □
Dirty clay( )
□ □
□ Water □
□ □
Metakaolin
□ □
□ Steam □
□ □
Metakaolin
□ □
□ Steam □

Total mole fraction of component in stream 9

y9 □□ □ ys □□$_9$ □ 1
s

Mass flow rates of components in stream 9

ns □□9 □□ zs □□9□M_W
s

ns □□9

57.5565	kg batch
1.27549	
0	
941.16801	□_____
0	

Total mass flowrate of stream 9

□

□ Kaolin □
□ □
Dirty clay( )
□ □
□ Water □
□ □
Metakaolin
□ □
□ Steam □

$$N9 \; □□ \; \underset{s}{\boxed{}} \; ns \; □□9 \; □ \; 1000 \; □ \; \frac{kg}{batch}$$

Mass composition of components in stream 9

$$xs \; □□_9 \; □□ \; \frac{ns \; □□9}{N9}$$

□ Kaolin □ xs □□^{9} □
□ □
Dirty clay( )
□ □
□ Water □
□ □
Metakaolin
□ □
□ Steam □

0.05756
0.00128
0
0.94117
0

Total mass fraction of component in stream 9

□

□ Kaolin □

□ □

Dirty clay( )

□ □

□ Water □

□ □

Metakaolin

□ □

□ Steam □

x9 □□ □ xs □□9 □ 1

s

A.6 Determination of Scaleup Factor

Given Amount of metakaolin = 1000 kg/batch

$$\text{Target} \;\square\square\; 1000\,\frac{\text{kg}}{\text{batch}}$$

Calculated
metakaolin

$$\text{Calculated}\square\square\;\; \text{N9} \;\; \square \;\; \frac{\text{kg}}{}$$

1000□batch

$$\text{SF} \;\; \frac{\text{Target}}{\text{Calculated}} \;\square\square \qquad \square\, 1$$

The process is a batch operation and 1000 kg of metakaolin is produced per batch for a 2 hr batch time. Hence, the number of batches required to meet 5 tons metakaolin per day is

$$\frac{\text{tonne}}{\text{day}} \qquad \frac{\text{kg}}{\text{batch}} \;\; \text{Design\_Capacity}\square\square\, 5 \qquad \square\, 416.667\square$$

$$\text{Nbatch} \qquad \frac{\text{Design\_Capacity}}{} \;\; \square\square \;\; \text{N9} \;\; \square\, 0.417$$

APPENDIX B

B.0 ENERGY BALANCES

B.1 THERMODYNAMICS PROPERTIES

B.1.1 HEAT CAPACITY COEFFICIENTS OF THE COMPONENTS

The heat capacity coefficients of the components involved in this project are as outlined below. Denoting the matrix of the heat capacity coefficients by c, we have

$$\begin{bmatrix}\text{Kaolin}\\ \text{Dirty clay}(\,)\\ \text{Water}\\ \text{Metakaolin}\\ \text{Steam}\end{bmatrix} \quad c = \begin{bmatrix} 0.945 & 0 & 0 & 0\\ 0.788 & 0 & 0 & 0\\ 3.2243 & 1.9238\times10^{-4} & 1.0555\times10^{-6} & -3.596\times10^{-9}\\ 1.065 & 0 & 0 & 0\\ 33.46 & 0.688\times10^{-4} & 0.7604\times10^{-6} & -3.593\times10^{-9}\end{bmatrix}$$

The heat capacity is in kJ/kg

B.1.2 HEAT OF FORMATION OF THE COMPONENTS

The heat of formation of the components involved in this project are as outlined below: Denoting the matrix of the heat capacity coefficients by hf, we have

$$\begin{bmatrix}\text{Kaolin}\\ \text{Dirty clay}(\,)\\ \\ \end{bmatrix} \qquad \begin{bmatrix} \\ -228583.482\\ 2677.724 \end{bmatrix}$$

□MetakaolinWater □hf □□ □□ □□$^{223032.644}$$_{15865.403}$□□□□kJkg

□ □

□ □ □

□ Steam □ □ □15865.403□

kJ

Hs □□j □□ 0

—

hr

B.2 FORMULATION OF ENERGY BALANCE EQUATION

The equation to be used for the energy balance is given as

$$\Box H = n\Box \overset{\Box}{\Box} \Box \; cpdT \quad \underset{\Box Tr}{\overset{Ts \; \Box}{}}$$

where

H = Enthalpy

Cp = Heat capacity

T = Temperature

n = Amount

Tr = Reference temperature

Ts = System temperature

If a reaction is involved, the equation becomes

$$
\begin{array}{l}
\quad Ts \\
\quad \square \\
\square H = n\square \, \square \, \square \quad cpdT \; \square \; hf \\
\quad \square Tr
\end{array}
$$

where hf = heat of formation

It should be noted that Cp is given in terms of heat capacity coefficients as

$$Cp = a\ b\ T\square\square\square\ cT\square^{2}\square\ d\ T\square^{3}$$

where a, b, c and d are heat capacity coefficients (constants).
So, the enthalpy balance equation will then become

$$
\begin{array}{llll}
\square\ i = & T\ \square\ ni\square\square\ CpidT = & \quad T & \\
& & \square\ \square & \qquad\qquad ^{2}\qquad ^{3}\square \\
& \square Tr & ni\square\square\ \ A\ \square\ B\ T\square\square\ C\ T\square\square\ D\ T\square & \\
& & \qquad dT & \\
& & \square Tr &
\end{array}
$$

Integration of above equation gives

$$
\begin{array}{l}
\square\ ini\square\square\square A\ T\square\square\ \overline{B\ T}\square^{2}\square\ \overline{C\ T}\square^{3}\square\ \overline{D\ T}\square^{4}\square\ \square \\
\quad = \\
\qquad \square \qquad\quad 2 \qquad\quad 3 \qquad\quad 4\ \square
\end{array}
$$

$$
\begin{array}{l}
\square\ ini\square\square\ \square \qquad B\ T\square\ \square\ 2\ \square\ Tr2\ \square\ C\ T\square\ \square\ 3\ \square\ Tr3\ \square\ D\ T\square\ \square\ 4\ \square \\
Tr4\ \square\ \square\ \square \\
\quad = A\ T\square\ \square\ \square\ Tr\square\ \square \qquad\quad \square \qquad\qquad \square \\
\qquad \square \qquad\qquad\qquad 2 \qquad\qquad\quad 3 \qquad\qquad\quad 4 \qquad \square
\end{array}
$$

The equation above is used to estimate the enthalpy of each stream

Were

T = Stream temperature

Tr = Reference temperature

B.2.1 When a Reaction Takes Place

To account for the energy change cause by a reaction (Heat of reaction) in the energy balance, the enthalpy of individual constituent is incorporate as an additional quantity termed the standard heat of formation, ΔH^o_f . The superscript 'o' denotes 'standard state' and the subscript 'f' denotes 'formation'. For a single species without any pressure effect on the enthalpy and without phase changes, the enthalpy change when a reaction is involved at temperature, T, is thus;

$$H_i = n\left[\Delta H^o_f + \int_{Tr}^{T_s}\left(a + bT + cT^2 + dT^3\right)dT\right]$$

The same equation is applied to calculate for the enthalpy change of all other species involved in the reaction and those involved in the product as well.

Therefore, enthalpy changes over the input and output stream (Energy Input and output) is thus;

Energy Input

The total energy input to a system is the sum of enthalpy of all component in the input stream at the input stream temperature

$$\square H_{\text{input}} = \square\ \square H_1 \square H_2 \square H_3 \square \square \square \square \square \square \square \square \square \square \square \square H_n \square$$

Energy Output

The total energy output from a system is the sum of enthalpy of all component in the output stream at the output stream temperature

$$\square H_{\text{output}} = \square\ \square H_1 \square H_2 \square H_3 \square \square \square \square \square \square \square \square \square \square \square \square H_n \square$$

Therefore, the thermal energy duty over any unit operation is the difference between the total output enthalpy and the total input enthalpy. That is,

Duty (Q) = Total Enthalpy of products - Total Enthalpy of Reactants

Duty (Q) = Total Output Enthalpy - Total Input Enthalpy

B.3 Energy Balances Calculations Energy

Balance:

Evaluation of Species Enthalpies: the reference state is the elements at 25 ^{0}C.

Tr □□ 25°C □ 298.15K

□cs □□0 cs □□1 □ kJ

□□

h s(□□□) = zs □□1□□ ___1□ □T □ Tr□ □

___2□ □ T^2 □ Tr^2 □ □ mol

□□□ □

□ □

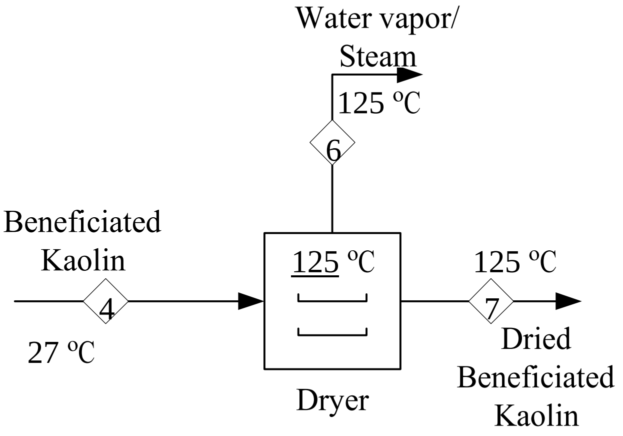
□□ ___s3□□2 □ □ T3 □ Tr3 □

□ ___cs4□□3 □ □ T4 □ Tr4 □

c

□

B.1 Energy Balance Around Dryer



□□□ □□□□□□ T□□ □ □□Tr□□□□ □ 2 □ □

□□ cs □□2□□□□□□□ T □□2 □ □□ Tr□ □□ □□

Hs □□4 □□ ns □□4□□□□ cs □□1□ □□□ kJ□

□□□ 1 □ □□K □ □K□□ □ 2 □ □□K□□ —□ K □ □ □

4 kg□

□□□□□□ cs□□□□□□ □3□ □□ □ Tr□ □ □

Tr□□3□□ □ □□ cs □□4□□□□□□□ T □□ □ □

□□4

□ □□3□ T □ □ □ —□ □ □

□ K □

□□ □ 3 □ □□K□□K □ □ □ 4 □ □□K □ □

Hs □□4 □

Kaolin

Enthalpy of components in stream 1 is:

T □□ 27°C □ 300.15K T □□ T

4 4

	2175.63555
	2.01017
	3137.74243
	0
□ Steam □	0

Total energy in stream 4

kJ

H4 □□ □ Hs □□4 □ 5315.3881□——batch

s

2

□ Tr□ □□

□□□

Enthalpy of components in stream 6 is: □ □ —□

□ K □ □

4□ □ □

T6 □□ 125°C □ 398.15K T □□ T6

□
□kJ
□ Dirty
clay( )

□□ □
□ c □□
Hs □□6 □□ ns □□6□□□□ s □□1 □□□□□□□ T □□ □ kJ□
□□ Tr□□□□ □ □□ cs □□2□□□□□□ T □□2 □
□□□ ^{1}□ □□ K □ □ K □□ □ ^{2}□ □□ K □ kg□
□□ □ ^{3}□ □ □ □
□□ cs □□3□□□□□□ T □□3 □ □□Tr□□ □ □ □ □
□□ cs □□4□□□□□□ T □□4 □ □□ Tr□□ □ □ □
□□□
□□ □ ^{3}□ □□ K □□ K □ □ □ ^{4}□ □□ K □□ K □ □

□ □batch
□ □
□ Water □
□ □
Metakaolin
□ □

Hs □□6 □

□ Kaolin □kJ	0
□ Dirty clay( )	0 □ □batch
□ □	140560.337
□ Water □	0
□ □	0

Metakaolin

□ □

□ Steam □

Total energy in stream 6

kJ

H6 □□ □ Hs □□6 □ 140560.3366□——batch

s

Enthalpy of components in stream 7 is:

T7 □□ 125°C □ 398.15K T □□ T 7

Hs □□7 □□ ns □□7□□□□c □ □□ T □ □□ Tr□□□□ — □ □□ — cs □□2□□□□□□□ T □□2 □ □□ Tr□□2□□ □□□ □□□kJ□□

□□□ s □□1□□□□ □ □

□□□ 1 □ □□K □ □K □□ □ 2 □ □□K □□K □ □ □ kg□

□□□□□ □ □ cs □□3 □ □□3 □ Tr□3□□ □ □□ cs □□4□□□□□□□ T □□4 □ □□ Tr□□4□□ □□ □□

□□ □□ T □□ □ □ —□ —

□□ □ 3 □ □□K □□K □ □ □ 4 □ □□K □□K □ □ □ □

$$H_s(s,7) = \begin{pmatrix} \text{Kaolin} \\ \text{Dirty clay()} \\ \text{Water} \\ \text{Metakaolin} \\ \text{Steam} \end{pmatrix} \begin{pmatrix} 108781.777 \\ 100.509 \\ 15617.815 \\ 0 \\ 0 \end{pmatrix} \frac{\text{kJ}}{\text{batch}}$$

Total energy in stream 7

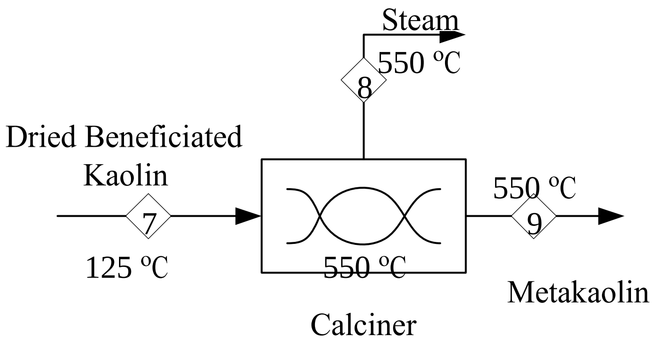
$$H7 = \sum_{s} H_s(s,7) = 124500.1011 \frac{\text{kJ}}{\text{batch}}$$

Dryer heat duty

$$Q_{dryer} = (H6 + H7) - H4 = 10744.847 \frac{\text{kJ}}{\text{batch}}$$

This implies that, the estimated amount of heat must be added to the dryer to attain desired drying

B.2 Energy Balance Around Calciner



Enthalpy of components in stream 7 is:

T7 □□ 125°C □ 398.15K T □□ T7

□ □

Hs □□7 □□ ns □□7□□hf □ □□□□ cs1□□1□□□□□□□□□□□ KT □□□ □ □□□ TKr□□□□□□□ □ □□□ cs2□□2□□□□□□□□□□□ KT □□□2 □ □□□ TKr□□□2□□□ □□□ □□□□kgkJ□□□

□ s □

□□ □□□ □ □□□3 □ Tr□3□□ □ □□ cs □□4□□□□□□□□ T
□□4 □ □□ Tr□□4□□ □□ □□ □ cs □□3□ □□□ T
□□ □ □ □

□ □ □ 3 □ □□ K □ □ K □ □ □ 4 □ □□ K □ □ K □ □ □ □
Hs □□7 □

□ Kaolin □kJ	-26204146.72533	
□ Dirty clay( )	3515.91939	□ □batch
□ □	-743238.42016	
□ Water □	0	
□ □	0	

Metakaolin

□ □

□ Steam □

The total energy balance entering calciner is as follows

$$H7 \square\square \boxed{}_{s} Hs \square\square 7 \square \square 26943869.2261 \square \frac{kJ}{batch}$$

Enthalpy of components in stream 8 is:

$T8 \square\square 550°C \square 823.15K$ $T \square\square T^{8}$

Hs □□8 □□ ns □□8□□ □□ c □ □□ T □ □□ Tr□□□□ □ □□ cs □□2□□□□□□□ T □□2 □ □□ Tr□□2□□ □□□ □□□ kJ□□

□□hfs □ □□□□ s1□□1□□□□□□□ K □□ □ □ K □□ □ 2 □ □□ K □□ K □ □ □ kg□

□□□□□ □ cs □□3 □ □□3 □ Tr□3□□ □ □□ cs □□4□□□□□□□ T □□4 □ □□ Tr□□4□□ □□ □□

□□ □□ T □□ □ □ □

□ □ □ 3 □ □□ K □ □ K □ □ □ 4 □ □□ K □ □ K □ □ □ □

Hs □□8 □

□ Kaolin □kJ	0
□ Dirty clay( )	0
□ □	0
□ Water □	0
□ □ Metakaolin	290855.93911

□ □batch

□ □

□ Steam □

Total energy balance from stream 8

kJ

H8 □□ □ Hs □□8 □ 290855.9391□ batch

s

Enthalpy of components in stream 9 is:

T9 □□ 550°C □ 823.15K T □□ T9

□ cs □□1□

Hs □□9 □□ ns □□9□□□□hfs □ □□□□□ 1 □□ □□□□□ KT

□□□ □□□ TKr□□□□□□ □ □□□ cs2□□2□□□□□□□□□□ KT □□□2 □

□□□ TKr□□□2□□□ □□□ □□□□kJkg□□□

□ □ □

□□ □□□ □□ c □□3□□□□□□□ T □□3 □ □□

Tr□□3□□ □ □□ cs □□4□□□□□□□ T □□4 □ □□

Tr□□4□□ □□□□ s

□ □ □ 3 □ □□ K □ □ K □ □ □ 4 □ □□ K □ □ K □ □ □ □

Hs □□9 □

□ Kaolin □kJ	-1287091.20859
□	3943.08106
Dirty clay( )	0
□ □	-20464888.4921
□ Water □	0

□ □batch

□ □

Metakaolin

□ □

□ Steam □

The total energy in stream 9

$$H_9 \; \square\square \; \square \; H_s \; \square\square_9 \; \square \; \square 21748036.6196 \square \; \frac{kJ}{s}$$

$$H_{4\,9} \square\square \; \square \; \square 20464888.492 \; \frac{kJ}{batch}$$

Calciner heat duty

$$Q_{Calciner} \square\square \; \square \; H_8 \; \square \; H_9 \square \; \square \; H_7 \; \square$$

$$5486688.546 \square \frac{kJ}{batch}$$

batc
h

This implies that, the estimated amount of heat must be added to the calciner to maintain the

at temperature at 550 ^{o}C.