PROPERTIES OF ALKALI-ACTIVATED MILLET HUSK ASH - CALCIUM CARBIDE WASTE BINDER BASED-MORTAR

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ABSTRACT

Alkali-activation of agro-industrial wastes as alternative binder to Portland cement (PC) is receiving more consideration most especially in the developed nations due to the issue of green-house gas (CO_2) emission from the production process of (PC) and the need to improve the fresh and hardened properties of agro-industrial waste-based mortar and concrete. This research focused on alkali-activated millet husk ash (MHA)-calcium carbide waste (CCW) binder-based mortar for appropriate characterization of the materials and evaluating the fresh / hardened properties of the mortar produced. MHA – an agricultural by-product and CCW – an industrial by-product was examined as possible materials to be combined as alternative to PC. Three combination proportions of MHA – CCW (40:60, 45:55 and 50:50) was determined from CaO and SiO_2 content), activated with 5, 10, 15 and 20 molar concentrations of Sodium Hydroxide (NaOH) and mortar produced at 1:3 binder/sand (b/s) and 0.5 water/binder (W/B) examined for binding, strength development and water absorption at varied curing age (3, 7, 14, 28, and 56 days respectively) in accordance to BS EN 196-1: 2016. The chemical analysis of the supplementary cementitious materials via X-ray fluorescent (XRF) revealed MHA having 73.4 % silica (SiO_2) content and the aggregation of the main oxides ($SiO_2 + Al_2O + Fe_2O_3$) gives 88.1 % which is above 70 % minimum limit stipulated in ASTM C618 (2015) standard while CCW primarily contain CaO (66.1 %). The fresh properties examination of the binder pastes results revealed 45-55 (MHA-CCW) at 15M NaOH molarity possessing similar fresh properties as the control (PC). The tested mortar samples exhibited increasing performance for both properties examined with increasing NaOH concentration up to 15M but decreased performance at 20M for all combination proportions. The 7- and 14-days strength for 45-55 MHA-CCW activated with 15M NaOH were 10.72 (67 %) and 15.96 N/mm^2 (79 %) respectively of the PC-based sample at same age. The 28days strength for the 45-55 MHA-CCW, 15M NaOH sample showed higher strength gain (29 %) as against 23 % strength gain of the PC-based mortar. Further curing of the alkali-activated MHA-CCW mortar till 56days resulted in additional 24 % strength gain over the 28^{th} day as compared to the 7 % increase of the control`. The trend is same for all the alkali-activated MHA-CCW mortar studied up to 15M NaOH concentrations. The 15M NaOH activation of 45-55 MHA-CCW is considered by this study as the indicated proportion for good strength. However, recommends the utilization of 45-55 MHA-CCW, 15M NaOH in 1:3 binder/sand at 0.5 W/C for masonry work and further studies into the examination of the morphology and nature of the hydration as well as the formed microstructure should be undertaken.

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ABREVIATIONS AND SYMBOLS

CS- Compressive strength

$CaCO_3$ – Calcium Carbonate

CO_2 - Carbon Dioxide

GHG - Green-House Gas

CaO – Calcium Oxide

Fe_2O_3 – Ferric Oxide

MHA – Millet Husk Ash

NaOH – Sodium Hydroxide

Na_2SiO_2 - Sodium Silicate

PVC - Polyvinyl Chloride

MPa - Mega Pascal

UK – United Kingdom

PSD - Particle Size Analysis

RHA - Rice Husk Ash

ASTM - America Society for Testing and Materials

BS - British Standard

SDS - Saw Dust Ash

^oC - Celsius

CEM II 42.5N – Dangote Portland cement

Gs - Specific gravity

C_3S – Tri-calcium Silicate

CCA - Corn-Cob Ash

CCW - Calcium Carbide Waste

CH - Calcium Hydroxide

C-S-H - Calcium Silicate Hydrate

D_{10} – Cumulative 10 % Passing

D_{30} – Cumulative 30 % Passing

D_{60} – Cumulative 60 % Passing

EDX – Energy Dispersive X-ray

EDXRF - Energy Dispersive X-Ray Fluorescence test

CEN - European Committee for Standardization

FA - Fly Ash

H - Water

LOI - Loss on Ignition

MK - Metakaolin

PC - Portland cement

DUTM - Digital Universal Testing Machine

W/C – water cement ratio

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

Mortar, binders and concrete are crucial substances for expeditious urbanization. Binder is known as one of the key components in the social, economic, financial and infrastructural advancement of human society of any nation. Cement is a significant component in all kinds of construction as binder; and in previous years the cement market has been filled by one item, Portland cement (PC) (Tsado *et al.*, 2014). In numerous countries, PC is very costly and this has seriously restricted the construction of moderate housing (Didel *et al.*, 2014). Thus, developing alternative binder to PC is a magnificent choice at much lower cost toward making critical contribution in making low-cost building materials available and thereby results to less expensive shelters (Abdullahi *et al*, 2013). Olawuyi *et al. (*2017) reported that PC based binder is one of the most significant materials used in construction across the globe and is manufactured through calcination of calcium carbonate ($CaCO_3$) in an oven at elevated temperature above 900^oC to liberate calcium oxide (CaO) as carbon dioxide (CO_2) content of it was emitted to the atmosphere as shown in Equation (1.1) and thus added about 5 % of the globe CO_2 (Nattapong *et al*., 2011; Rubenstein, 2012).

$$CaCO_3 \xrightarrow{heat\ (>900\ oC)} CaO + CO_2 \qquad (1.1)$$

This development has made it necessary to look for alternative to PC as a way of making building materials less expensive and abate green-house gas (GHG) emission occurring from PC manufacturing processes. Over years, research bearings on alternative to PC concentrated mainly on incomplete replacement while report on complete cement

replacement in mortar / concrete production with an activator to facilitate its setting time and strength advancement are limited in literature (Habeeb & Mahmud, 2010).

The major constituents of a clean PC are silica (SiO_2), alumina (Al_2O_3) and ferric oxide (Fe_2O_3) with strength determinant being the SiO_2 which joined with CaO in the addition of water at any given temperature (Neville, 2012; Mehta & Monteiro, 2014) as shown in Equation (1.2). This results in the formation of calcium silicate hydrate - $CaO\text{-}SiO_2\text{-}H_2O$ (C – S – H), the final result for strength improvement as cement hydration advances after water contact.

$$CO_2 + SiO_2, + Al_2O_3 + Fe_2O_3 \xrightarrow{heat\ (>1450\ ^{o}C)} CaO, SiO_2, Al_2O_3, Fe_2O_3 \qquad (1.2)$$

MHA is a decent wellspring of silica content (Jimoh *et al.,* 2013) while CCW has been accounted to be of high calcium oxide-showed as CaO in the chemical formula (Olawuyi *et al.,* 2017). These were joined together and activated with alkaline in this research at different proportions as calculated from the molar concentration of the MHA/CCW as accounted in Olonade & Bello (2017). As indicated by Yunusa (2015), CCW produce calcium hydroxide when completely mixed in water as appeared in Equation 1.3 As indicated by Yunusa (2015),

$$CaC_2 + 2H2O \rightarrow C_2C_2 + Ca(OH)_2 \qquad (1.3)$$

As reviewed by Mehta and Monteiro (2014), Pozzolanic responses in the similar way as that of Portland cement using Tri-Calcium-Silicate (C_3S) in the presence of water (H) to liberate Calcium-Silicate-Hydrate (C-S-H) and Calcium Hydroxide (CH).

These (Portland- Pozzolan) cement responses are illustrated in equation (1.4) and (1.5) as follows:

$$C_3S + H \rightarrow C - S - H + CH \quad (1.4)$$

$$\text{Pozzolan} + CH + H \rightarrow C\text{-}S\text{-}H \quad (1.5)$$

Where C = CaO, S = SiO_2 and H = $(OH)^-$

The reaction in Equation (1.4) is considered to be quick and lime delivering while the response in Equation (1.5) is somewhat moderate or idle, contingent upon the properties of the pozzolanic material and doesn't require nearness of PC (Auta *et al*., 2015 and Jimoh *et al.,* 2013) yet a functioning wellspring of CaO, hence the thought for preference source of CaO to upgrade pozzolanic response with an agricultural waste ash (MHA) as SiO2 source is the focal point of the current investigation. Likewise, the utilization of alkali-activation can definitely improve the pace of the response and hence improve its workability (Ka'ase et al., 2018).

Alkali activation as clarified by Martinez & Palomo (2001) is the chemical procedure where indistinct structure is changed into a skeletal structure that displays cementitious properties. The material containing responsive SiO_2 or Al_2O_3 can be enacted as appeared in condition (1.6) and (1.7).

$$NaOH + SiO_2 \rightarrow \text{N-S-H (Gel) as in geopolymer} \quad (1.6)$$

$$NaOH + Al_3O_2 \rightarrow \text{N-S-H (Gel) as in geopolymer} \quad (1.7)$$

Where N-S-H (Gel) is Sodium-Silicate-Hydrate gel.

Early strength development is a basic measure in construction industry since it decides the speed of construction. In this way, low early strength advancement is a deterrent in advancing Pozzolan utilization as PC substitution. In order to avoid the less early strength advancement and high setting occasions in pozzolanic materials, methods like alkali-activation have been recommended by (Kwabena, 2012).

Bakharev (2006) disclosed that concrete manufactured with the aid of alkali-activated fly ash with NaOH had a two-day development strength of 10 N/mm^2 meanwhile at 28-days strength development was 60 N/mm^2. Moreover, He also makes fly ash more active in reaction by adding Sodium Silicate ($Na_2SiO_{2)}$ to attain two-day strength development of 2 N/mm^2 and a 28-day compressive strength of 45 N/mm^2.

1.2 Statement of the research problem

The quest to improve the setting time and early age strength development of agricultural and industrial waste as revealed by Kisharet *et al.* (2013); Olawuyi, *et al.* (2017); Egwuda *et al.* (2018); Enejiyon *et al.* (2018) and Okwute (2018) has made it necessary to search for reasonable activators to improve the workability of these materials. But, in previous years, researchers had shone their searchlight on numerous researches on the quest for preference binder which focused mainly on the partial substitution of the PC in mortar or concrete due to the sensitization on the atmospheric change credited to a worldwide temperature alteration (Abdullahi, 2013).

According to Enejiyon *et al.* (2018), at 56 days of curing agricultural / industrial waste (RHA/CCW) based mortar and PC showed that the strength of RHA/CCW based mortar (9.52N/mm^2 is three times of PC-based mortar (30.72N/mm^2). Therefore, recommended for

the utilization of set accelerator other than superplasticizer to improve strength gaining/fast track the setting time, early/later strength advancement of agricultural/industrial waste-based mortar to attain strength that is almosautat the same to that of PC based mortar. Also, Okwute (2018) reported that MHA/CCW based mortar compressive strength of 5.08N/mm^2 and 8.38N/mm^2 against that of PC (18.44N/mm^2 and 22.25N/mm^2 at 28days and 56days respectively. Therefore, suggested the use of set accelerator admixture to improve the early and later strength advancement of agricultural/ industrial waste-based mortar.

1.3 Aim and Objectives of the Study

This research is aimed at the development of alternative binder for mortar using alkali-activated millet husk ash (MHA) and calcium carbide waste (CCW) with a view to establishing the good performance level in strength and durability of mortar made from such binders.

The specific objectives are to:

i. Study the chemical and physical properties of the constituent materials (MHA, CCW and sand).

ii. Determine optimum mix proportions of MHA and CCW with NaOH-activation for mortar production.

iii. Evaluate the fresh and the early- age characteristics of the alkali-activated mortar.

iv. Investigate the hardened properties and the effect of alkali-activator (NaOH) on the strength properties (density, compressive strength and water absorption) of the various mix proportion of mortar.

1.4 Scope of the Study

This study focused mainly on experimental toward making a preference binder for mortar by utilizing NaOH to facilitate agro-industrial waste materials reaction (MHA– as SiO_2 source and CCW – as CaO source) at different mix proportions (40:60, 45:55 and 50:50) respectively as determined from the molar concentration of the binder contents. Meanwhile, the molarities of NaOH used for activation of this agro-industrial waste vary from 0M to 20M at 5M step intervals. It involved appropriate characterization of the materials and determining the fresh and hardened properties of the binder pastes and mortar samples. Mortar cubes of 1:3 cement/sand (s/s) and 0.5 water/cement (w/c) showed by BS EN 196-1:2016 as control were examined in comparison with MHA-CCW based-mortar at different mix proportions. The tests conducted were chemical property (X-Ray Fluorescence – XRF); The fineness of the binders examined using 75 µm sieve, particle size distribution (PSD) and BET specific surface area for even distribution; Fresh properties (standard consistency, setting time, soundness); strength advancement and water assimilation of the hardened mortar. These mortar cubes prepared were tested for strength advancement and water assimilation at different curing ages (3, 7, 14, 28 and 56 days) with their densities evaluating accordingly.

1.5 Justification for the Study

The end result of this study offers well explained technical information on the alkali-activation of the preference binder with decent carbon foot print and continually making environment eco-friendly. The outcome of this study can be used in designing the mix proportion for specified compressive strengths and achieving a complete PC usage in mortar cubes production with decent workability data that affirmed the use of these

environmental-friendly innovation on the large scale for word impact and maintaining eco-system friendly environment.

The uses of alkali-activated MHA-CCW binder will drastically diminish over dependent on PC usage reduce both agricultural and industrial waste disposals, diminish of hazard due to alkaline pollution and continually usage of materials with low CO_2 emission (Jimoh *et al.*, 2013; Abdulfatai *et al.*, 2013; Bakharev, 2006).

The strive for developing alternative binder cannot be over emphasized in the face of the critical changing of economic realities, carbon blue print and the need for sustainable eco-friendly environment. The problem therefore is not only to look for preference cementitious materials but to resolve the chemically combined issues of these pozzolans with a view to establishing the early strength advancement achieved and the rate of water assimilation of mortar/concrete made from that kind of binders. Integrating alkali-activation on the agro-industrial waste usage results to early setting time and quick strength advancement of the mortar/concrete leads offers contribution towards improving knowledge in concrete technology and development of our nation, Nigeria. To justify the use of these, potentially more energy efficient technology on the large scale and to have global impact, it is necessary to develop adequate performance data that can bring changes to building codes and standard.

Nigeria's drive for Agriculture with millet creation as one fundamental harvest and Niger State being a significant millet delivering State in Nigeria with tremendous measure of millet husk loaded at dump destinations as waste is the inspiration for this investigation (Jimoh *et al.*, 2013; Auta *et al.*, 2015; Anowai & Jones 2015. This investigation is an

endeavour to investigate the use of alkali to activate MHA-CCW as an preference binder for mortar and concrete manufacture, towards the provision of less expensive housing, diminish over dependent on PC, reduction of indiscriminate waste disposal in both urban and rural area (Utsev & Taku, 2012; Abdulfatai *et al.,* 2013) and furthermore help to diminish natural risk presented by CO_2 gas outflow during PC manufacture (Abdul-Wahab *et al.*, 2016; Olawuyi *et al.*, 2017; Razi *et al.,* 2016; Zhang *et al.,* 2014). The use of the alkaline to activate on these materials in mortar manufacture will result in energy preservation with economic, ecology and technical merit.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Mortar

Mortar is a workable paste used to holding building blocks such as stones, bricks, and concrete masonry units together firmly, fill and seal the irregular gaps between them, and sometimes add decorative colours or patterns in masonry walls (Neville, 2012). It is one of the constituents of the composite anisotropic material called stonework. Mortar is in charge of making a uniform pressure conveyance, repairing the anomalies of squares and obliging distortions related to warm extension and shrinkage (Vladimir *et al.*, 2011). Emmitt & Gorse (2014) defined cement mortar as the mixture of cement and sand at a proportioned mix ratio and conditioned water content to form a paste. Mortar qualities are influenced by its constituent materials which are mainly sand, binders and water. Sand as a characteristic material is sensibly shabby and when mixed with water can be made plastic but then has great quality in resisting crushing. Its grains are likewise basically impenetrable to the activity of ice and rain. The vital ingredients to tie the particles of sand together into a strong form of a frame is a grout glue called binder and two materials utilized for these processes are cement and lime.

ASTM C270 (2004) generally categorized mortar into four major types, namely; M, S, N and O. But, building codes (USA) require types M, S, and N. The codes describe the utilization of a few mortars for specific applications. For example, type M or S mortar is required for exact foundation walls design and type N or S mortar is used for glass unit masonry. Types S or M mortar is used basically for seismic design.

Table 2.1: Property Specification Requirements for Mortar

`	Type	Average compressive at 28 days min. N/mm^2
Mortar cement	M	19.2
	S	14.4
	N	6.2
	O	3.3

Source: ASTM C270:2004

2.2 Constituent of Mortar

The major constituents of mortar are binder (PC or lime), fine aggregate and water and sometimes admixtures to give desire properties.

2.2.1 Cement

Cement was discovered by Joseph Asphidin (1824) in UK (as cited in Neville & Brooks, 2010) and he called his product Portland cement (PC). It is a binder developed by intimately mixing together argillaceous and calcareous, or other alumina, iron and silica oxide bearing materials, calcinating them in a clinker at high temperature up to around 1400 ^{o}C (2550 ^{o}F) and grinding the subsequent clinker (Neville & Brooks, 2010). The raw materials utilized for the production of PC comprise of four noteworthy mixes; iron oxide (Fe_2O_3), Silica (SiO_2), lime (CaO), and alumina (Al_2O_3) and two minor mixes; magnesia (MgO) and gypsum ($CaSO_4$. $2H_2O$). The calcareous (CaO) materials can be found in calcium carbonate (limestone), marl, calcite, or shale and the argillaceous (SiO_2 and Al_2O_3) materials found from sand, shale, and mud.

ASTM C150 (2012) describe PC as hydraulic cement manufactured by pulverizing clicker comprising essentially of hydraulic calcium silicates and a little quantity of one or more

forms of calcium sulphate solution. Clinkers are 5 to 25 mm-diameter nodules of sintered material that is made when a raw mixture of predetermined composition is heated to high temperature as illustrated in Mehta & Monteiro (2014). According ASTM C150 (2012), PC was classified into different types, uses and by constituents. Table 2.2 summarizes the types of cement and its uses.

Table 2.2: General properties of the main types of Portland cement

Types	Classification	Characteristics	Applications
Type I	General purpose	Fairly high C_3S content for good early strength development	General construction (most buildings, bridges, pavements, precast units etc.)
Type II	Moderate sulphate resistance	Low C_3A content (<8%)	Structures exposed to soil or water containing sulphate ions
Type III	High early strength	Ground more finely, may have slightly more C_3S	Rapid construction cold weather concreting
Type IV	Low heat of hydration (slow reaction)	Low content of C_3S (<50%) and C_3A	Massive structure such as dams.
Type V	High sulphate resistance	Very low C_3A content (<5%)	Structures exposed to high levels of sulphate ions
White	White colour	No C_4A, low MgO	Decorative (otherwise has properties similar to type I)

Sources: Enejiyon, (2018); Mehta & Monteiro, (2014)

Cement paste is an important component of mortar/concrete and thus; Rasa *et al*. (2009) stated that the mechanical characteristics of cement are exceptionally affected by the thickness and compressive quality of binder. The quality of materials used determines the

life span of the structure. The testing of cement to judge its quality is essential in civil engineering and the construction industry as a whole. Findings on the compound arrangement and fineness are endorsed in European Standards BS EN 196-1: 2016 and additional tests are recommended by BS EN 196 – 1: 2016 for common and fast solidifying PC.

2.2.1.1 Fineness

The fineness of cement is one of the properties of cement that determine the degree of reaction of the materials with water, the finer the material the higher the rate of the reaction with water and other constituent materials (Rahhal & Talero, 2005). The fineness of cement is measured by the sieving or wet method. For neat PC, the fineness is in m^2/kg and should be less than 225 m^2/kg. ASTM C150 (2012) specified a minimum of 280 m^2/kg for fineness of PC. According to Neville (2012) & Shetty (2009), early strength development of PC has a fineness of above 325 m^2/kg.

2.2.1.2 Consistency (Water demand)

This is the process of determining the percentage of water required for workable paste (normal consistency) for a given sample of the binder. The Vicat apparatus with the plunger of 10 mm diameter, trowel, weighing balance, weight box and measuring cylinder are used as specified by ACI EI 99-1:2000. It involved determining the water demand of the paste which will give the suitable standard consistency as also affirmed by Neville (2012).

2.2.1.3 Chemical composition

This is the basis of characterization of cement which can affect all properties of cement except the fineness of cement (Neville & Brooks, 2010). The high content of certain cement

of constituents' compound in proportion to others may lead to retardation or acceleration of the rate of setting and hardening, thus the constituents need to conform to standard specification. Excess of any compound could affect the rate of heat evolution as the cement hydrates and to guide against this, the ratios of lime, silica, alumina, iron oxide, alkali and its sulphur contents should be maintained based on the standard specification. Calcium sulphate (gypsum) and calcium chloride are utilized as admixtures to retard or accelerate the setting rate of cement paste (Neville & Brooks, 2010). According to Olawuyi, *et al.* (2017), Energy Dispersive X-Ray Fluorescence test (EDXRF) is one of the methods that can be used for checking the chemical composition of cement. Report of XRF analysis of typical PC (Olawuyi *et al.*, 2017) is presented below in Table 2.1

Table 2.3: Oxide Composition of Portland cement

Oxide content	CaO	MgO	Al_2O_3	Fe_2O_3	SiO_2	SO_3	AR	SR	LOI
CCW	64.0	2.9	5.2	1.2	21.5	4.5	4.5	3.4	0.0

Source: Olawuyi *et al. (2017)*

2.2.1.4 Soundness

Cement paste is the mixture of binders and water. When it sets and hardens normally without any cracking or disintegrating such paste cement is said to be sound (Klemm, 2005). Hydration of free lime (CaO), magnesia (MgO) and sulphates (SO_4) surrounded by cement particles caused unsoundness of cement by preventing easy hydration of free lime (un-combined lime) and other materials during the normal setting period. For a cement to be free from unsoundness it should be thoroughly mixed, burnt and ground. The standard specification of PC according to ASTM C150 (2012) specifies a maximum of 0.80 percent

for all types of PC and specified a maximum of 10mm i.e., Chartelier apparatus is the instrument widely used for checking the unsoundness of cement in the laboratory as reported by Shetty, 2009.

2.2.1.4 Setting time

Setting time (both initial and final) is usually determined with the aid of Vicat apparatus. In this experiment a 1mm diameter needle is allowed to penetrate into a prepared part of the cement/Pozzolan paste. Initial setting time is recorded when the needle does not visibly penetrate the surface and final setting time noted when the circular attachment is lowered over the surface with the centre needle make an impression while the circular cutting edge fails to do so (Shetty, 2009 and Neville, 2012). Most PC has an initial setting time which range from 2 – 4 hours and final setting which ranges from 5 – 8 hours. ASTM C191-13 specified that the initial setting time should not below 30 min and final setting time not to be less than 10 hours

2.2.2 Sand

Fine aggregate is also a vital ingredient for mortar and concrete production. The well-known fine aggregate used for normal mortar/concrete production being sand and are generally referred to as aggregates passing sieve 4.75 mm but are retained on sieve 0.075 mm (ASTM C128, 2015). They enhance the flowing ability and segregation resistance (workability) when mixed in an appropriate proportion (Bhattacharya, *et al*., 2008; Okamura & Ozawa, 1995; Su, *et al*., 2002). A well graded fine aggregate improves the workability of Mortar (Hu & Wang, 2005; Benabed, *et al*., 2012). In addition, according to

Tasi, *et al*. (2006), a well-graded fine aggregate enhances the packing density and thereby increases the strength development and durability of mortar or concrete.

2.2.3 Water

Water is one of the basic elements used in construction industry. Its quality and quantity has much effect on the strength development of mortar and concrete in construction work. In mortar /concrete production, the water used in mixing should not contain impurities that may affect some properties of concrete, thus, water from tap is recommended. This does however mean that water that is unsuitable for drinking is not appropriate for concrete production as affirmed by Aneje & Damen (2014), but must be free from contaminated substance like salt, sugar, oil, organic materials, vegetable growth and other substance that may be deleterious to stone, bricks, concrete or steel.

Salt or brackish water can cause dampness and white deposits of precipitated salts on the surface of the concrete, increased risk of corrosion (rust) damage to embedded reinforcement and damage to painted parts. It is therefore advisable not to use such water for durable concrete work. Use of salt water in construction works is generally avoided (Savitha, 2012; Dunn, *et al.,* 2011).

2.2.4 Millets and Millet Husk Ash

Millets are a group of highly variable small-seeded grasses, widely grown around the globe as cereal crops or grains for fodder and human food. Millets are important crops in the semi-arid tropics of Asia and Africa (especially in India, Mali, Nigeria and Niger), with 97% of millet production in developing countries (Anowai & Jones, 2015). According to FOA, (2016), about twelve species of millet are available with a total product of 38.38

million tons, out of which 13.27 million tons (45%) are produced in Africa. Nigeria produces about 42%

of the millet produced in Africa and was rated as the second largest producer of millet in the World.

Millet husk ash (MHA) is a by-product of agricultural waste generated or obtained from burning of the millet husk produced after processing using a control burning method. Anowai & Jones (2015) reported that 40% of the weight of the harvested millet is removed as husk from the stroke. The husk is of no known economic value and sometimes used as filling materials.

Ogork *et al.* (2014) investigated the influence of MHA on the properties of plain concrete and reported that increased MHA content led to a decrease in the concrete workability. It was also revealed that compressive strength of the concrete decreased when MHA content increase beyond 5 % with and inferred that MHA can be used as a Pozzolan cement-based material. The study reported that MHA has cementitious compound which contribute in the strength improvement of mortar and concrete. It has been noted that the useful oxides (SiO_2 + Al_2O_3 + Fe_2O_3) of the composition of MHA is about 80 % which is greater than the ASTM C618 (2015) minimum limit of 70% for a good Pozzolan. Other studies suggest that the MHA is very reactive and have the possibility of pozzolanic reactions when blended with PC (Ogork *et al.,* 2014). Other recent studies (Jimoh *et al.,* 2013**;** Anowai & Jones**,** 2015; Okwute, 2018) reveal the chemical properties of MHA (Table 2.4) as having useful oxides (SiO_2 + Al_2O_3 + Fe_2O_3) contents of 73.05 %, 70.10 %; 79.91 % respectively and satisfies the 70 % content required by ASTM C618 (2015) for chemical constitution of a

Pozzolan. The results also satisfied the ASTM requirement for the loss on ignition put at a maximum of 12 %.

Table 2.4 Oxide Composition of MHA (Oxides were arranged according to the other of the source).

Oxide content	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	MgO	K_2O	LOI	P_2O_5	SiO_2 $+Al_2O_3$ $+Fe_2O_3$
[1]	73.10	0.025	4.2	10.5	1.30	0.44	7.50	11.04	0.44	77.72%
[2]	63.70	3.80	2.60	0.02	2.06	0.03	20.10	11.20	4.10	70.10%
[3]	74.08	1.64	4.17	1.64	0.50	0.73	5.21	4.01	1.19	79.89%

Source: [1]Jimoh, *et al.*, 2013; [2]Anowai & Jones, 2015; [3]Okwute, 2018

Auta *et al.* (2015) investigated the compressive strength of concrete with MHA as partial replacement of cement (0 %, 10 % and 20 %) with water cement ratio of 0.65 at 35 days of curing. The compressive strengths attained are 32.00, 25.56 and 23.18N/mm^2 respectively. This then revealed that MHA could be blended in small amounts between 0% and 10% replacement by weight or volume of cement in concrete production and concrete works under strict supervision for major concrete works.

2.2.5 Calcium Carbide Waste (CCW)

Calcium carbide waste (CCW) is the residue of acetylene gas generated from calcium carbide used in the production of Polyvinyl Chloride (PVC) and in welding steels, especially in the auto industry. CCW in Nigeria is reported to be about 70-80 % calcium hydroxide $Ca(OH)_2$ with the impurities in it listed as copper, lead, iron, manganese, nickel and zinc (Chukwudebelu *et al*., 2013; Olawuyi, *et al.*, 2017). The oxide composition of

CCW indicated that it was predominantly CaO (65.8 %) and low combined SiO_2, Al_2O_3 and Fe_2O_3 content of 3.14 %. This shows that CCW is cementitious and may combined with certain Pozzolan containing high SiO_2, Al_2O_3 and Fe_2O_3 in a hydrated mix and due to pozzolanic reactions yield final products that are similar to those obtained from cement hydration process (Wang, *et al.*, 2013*).*

Vichan & Raachan (2013) and Horpibulssuk *et al.*,2011) described CCW as a by-product of acetylene manufacturing, which dissolves in water to liberate $Ca(OH)_2$. CCW and hydrated lime are similar in their chemical and mineralogical composition except for the presence of carbon in CCW. Therefore, ground CCW by ball mill to increase its fineness before mixing with fly ash (FA) at a ratio 30/70 (CCW/FA) as a binder without PC. The initial and final setting time of CCW/FA paste in the study was reported to be much longer than the normal mortar or concrete with compressive strength values between 19.0 and 24.7 MPa at 28-days and 90-days respectively. The lower the W/B ratio, the higher the compressive strength of the CCW/FA concrete produced. The hardened concrete was reported to possess the same properties as that of the normal concrete. Table 2.5 presents the report the XRF analysis of CCW by Olawuyi et al (2017) and Manasseh & Joseph (2016).

Table 2.5: Oxide Composition of CCW

Oxide content	CaO	MgO	Al_2O_3	Fe_2O_3	SiO_2	SO_3	KO_2	LOI
[1]	65.8	0.2	1.6	1.3	3.6	0	1	26.4
[2]	61.4	0.84	1.78	0.78	2.69	0.36	32.51	-

Source: [1]Olawuyi *et al.*, (2017); [2]Manesseh & Joseph (2016)

Makaratat *et al*., (2010) studied calcium carbide residue – fly ash (CR–FA) concrete, for a weight ratio of 30: 70 (CR: FR) used as a binder to cast concrete. The CR was reported to

have specific gravity 2.32, a similar value as reported other studies (Makaratat, *et al*., 2010; Horpibulsuk *et al.,* 2014).

2.2.6 Properties of Fresh and Hardened Mortar

The principal features of fresh and hardened mortar of concern to this research are workability, bleeding, segregation, density, compressive strength and water absorption.

2.2.6.1 Workability

Neville & Brooks (2010) defined workability of a fresh mortar as the capacity of the mortar blend to be set inside the formwork, around any support, and to be effectively compacted by hand or mechanical means to expel trapped air pockets. Factors influencing workability of fresh mortar are: mix proportion, water-cement ratio, size of aggregates, grading of aggregates, shape of aggregates and the nature of alkaline activation used.

2.2.6.2 Segregation

According to Shetty (2009) defined segregation as the disintegrating of the constituent materials of mortar and concrete which makes their distribution to no longer be consistent. A quality mortar or concrete is one in which all the constituent materials are well circulated to make a consistent blend. Segregation is a state of concrete at which the constituents will be separated from one another and therefore the goal of mix proportioning and good concrete production will not be achieved.

2.2.6.3 Bleeding

Bleeding is an aspect of segregation referred to as water gain in which certain of the water in the mix has a tendency to float to the surface of freshly placed mortar. The main factors

causing bleeding of mortar are the failure of the strong components of the blend to hold the majority of the blending water when they settle downwards.

2.2.6.4 Density

Kazjonovs *et al.* (2010) described the density of mortar and concrete as mass per unit volume. Density is also referred to as unit weight or unit mass in air. Density can be ascertained in the laboratory via the procedure offered in BS EN 934-2:2009. Hypothetically, density is the quantity of masses of all the constituents of a batch of mortar divided by the volume occupied by the mortar. Mortar samples with higher density than 2000 kg/m^3 are known as normal weight mortar (ASTM C140: 2003).

2.2.6.5 Compressive strength

European Standard BS EN 196-1:2016 endorsed a compressive strength test in mortar. The specimens are tested as 40 mm equivalent cubes; they are gotten from 40 by 40 by 160 mm prisms, which are first tested in flexure so as to break into halves. Also, an optional flexural Centre point test over a span of 100 mm is possible. The test is carried out on mortar of fixed composition, produced with 'CEN standard sand'. CEN has been the abbreviation of the French title of the European Committee for Standardization. The sand is normal, siliceous and can be obtained from different sources. It is not of uniform size but is graded between 80 µm and 1.6 mm. The mortar is required to be of 1:3 sand/binder ratios and 0.5 water/binder ratios. Neville (2012) recommends mixing in a cake mixer and compacted on a bumping table with a drop of 15 mm. A vibrating table in like pattern can be used, provided it results in similar compaction.

2.2.6.6 Water absorption

Absorption simply means measure of permeability and is defined as a process through which liquid penetrates into and fills porous medium within a solid body, such as paste, mortar or concrete (ASTM C128, 2015). Castro *et al*. (2011) expressed that water absorption is a critical factor in measuring the solidness of a cementitious frameworks. Water absorption chiefly relies upon the aggregate volume of pores, filler composes, thickness and saturation systems of the concrete (Rajput, 2006; Nambiar & Ramamurthy, 2007). Castro *et al*. (2011) described water absorption as the ability of concrete samples to take in water by means of capillary suction. The concrete of low water absorption will give better protection to reinforcement within it (Nwaubani & Poutos, 2013). It was further stated that the average absorption of the concrete test specimens shall not be greater than 5 % and individual unit not greater than 7 %. Also, (Saraswathy *& Song,* 2007) performed water absorption test according to ASTM C642-06 for concrete containing RHA up to 30 % and reported that coefficient of water absorption reduced in concrete containing RHA for all percentages of cement replacement.

2.2.7 Alkaline activation

According to Angel & Ana (2011), described alkali-activation as a powdery substance: Alumino-silicate for example, FA, RHA and MHA blended in with an alkali-activator to create glue equipped for setting and solidifying within a sensibly brief timeframe. The strength, acid and fire resistance and shrinkage of the paste produced depends on the Alumino-silicate used and the activation process varies. The reaction of a solid Alumino-silicate with a highly concentrated aqueous alkali hydroxide or silicate solution produce a synthetic alkaline Alumino-silicate materials which may perform comparably to

traditional cementitious binders in a range of applications, emits significantly less green-house gas than in PC production. Depending on the raw materials and processing conditions used, alkali-activated binders may feature high compressive strength, low shrinkage, and fast or slow setting, acid and fire resistance, and thermal conductivity

Barbosa, *et al*. (2000) and Fernandez- Jimenez *et al*. (2005) reported that sodium silicate (Na_2SiO_3) solution in combination with sodium hydroxide (NaOH) is an effective alkaline activator which enhances the setting and strength development within a short period of time. Also, Hardjito, *et al.* (2004) conducted research on the effects of NaOH concentration on the compressive strength of FA-based geopolymer mortar and reported that alkaline concentration was proportionate to the compressive strength of geopolymer mortar. They also claimed that higher concentration of NaOH solutions results in higher compressive strength of geopolymer mortars.

Martinez & Palomo (2001), additionally depict soluble base enactment is concoction process where formless structure is changed into a skeletal structure that displays cementitious properties. Once a material contains reactive silica or alumina, it could be considered for activation using appropriate alkali solution such as NaOH, Sodium carbonate (Na_2CO_3), Sodium Silicate (Na_2SiO_3), potassium oxide (KO), Sodium oxide (Na_2O). Also, Olonade & Bello (2017) and Qureshi & Ghoshi, (2013) on the other hand worked on alkali-activated Cocoa shell ash.

Ka'ase *et al.* (2018) conducted study on the effects of different molarities (M) on the ambient temperature cured Metakaolin - based geopolymer concrete and reported that geopolymer concrete with the 8M and 16M concentrations of NaOH has a good

compressive strength (27.46 N/mm^2 & 34.01 N/mm^2) at 28 days curing and poor rate of water absorption.

It was proposed by Barbosa (2001) that an alkaline liquid made of sodium or potassium hydroxide (NaOH or KOH and sodium or potassium silicate (Na_2SiO_3 or K_2SiO_3) reacts with Pozzolans of geological origin or industrial/agricultural by-products which are rich in silica and alumina (such as fly ash, rice husk ash, millet husk ash, Corn cob ash, Pumice and Sorghum etc.) to produce a geopolymer.

Morenoa *et al.* (2008) activated class F fly ash with NaOH from different source and attained a 28-days compressive strength of between 29 N/mm^2 and 66 N/mm^2. Another study conducted by Fernandez-Jimenez *et al.*, 2004 used class F fly ash with one or more of these components: Na_2CO_3, Na_2SiO_3 and NaOH, to achieve 7days compressive strength between 35 N/mm^2 to 44 N/mm^2 and 28-day compressive strength in the range of 47 N/mm^2 and 57 N/mm^2.

The Spanish group – Eduardo Torroja Institute has accumulated considerable experience with these alkali-activated systems over the 20 years and has contributed to the growing body of literature on the systematic characterization of the element (Fernandez-Jimenez *et al.*, 2004).

2.3 Pozzolanic Materials

Pozzolans are fine materials that is made up of SiO_2 and/or Al_2O_3 which on their own have little or no binding property but when mixed with lime in the presence of water, will set and hardened like cement (ASTM C618, 2015; Abdulfatai *et al*., 2013). The utilization of pozzolanic materials such as saw dust ash (SDS), rice husk ash (RHA), Metakaolin (MK),

fly ash (FA), silica fume (SF), Blast-furnace Slag (BFS), and natural Pozzolans as partial cement clinker replacement will help to minimize CO_2 emissions during cement production, (Damtoft *et al*., 2008). Research trends on alternative binder had focused on the utilization of natural Pozzolans i.e. volcanic ash (Hossain 2003 & 2005; Hassan, 2006; Olawuyi & Olusola, 2017) or agricultural waste ashes such as RHA (Okpala, 1987; Chaowat, 2001; Abalaka & Okoli, 2013), SDA (Elinwa & Mahmood, 2002), corn-cob ash [CCA] (Raheem, 2010), MHA (Jimoh *et al.*, 2013;) and palm kernel nut ash [PKNA] (Joshua *et al.*, 2015) amongst others as partial PC replacement in mortar or concrete..

Moreover, Mehta & Monteiro (2014) and Neville (2012) stressed further that the silica must be amorphous, as a result crystalline silica has low reactivity. Duggal (2008) reported that the term Pozzolan is gotten from Pozzulini, a town in Italy on the Bay of Naples close to Mount Vesuvius. The sand (volcanic clean) around this town, when mixed with hydrated lime, was found to have water driven properties. In those days prior to the advent of PC, Pozzolan was utilized with lime to make concrete in those days. Also, Hewlett (2006) said that Pozzolan has a particular implication which reveals it as pyroclastic rocks, basically smooth. All Pozzolans by characteristic are rich in silica and alumina and contain just a little amount of antacids (Duggal, 2008).

Pozzolanic materials are classified into two basic forms, this are-

i. Natural pozzolans
ii. Artificial pozzolans

i. **Natural Pozzolans** are of volcanic origin and volcanic powders are usually obtained as the first Pozzolan as reported by Neville (2012). Parhizkar *et al*., (2010) expressed that Natural

Pozzolans can likewise be characterized as regular materials containing receptive silica or alumina, which, alone, have next to zero reactive property, however when blended with PC react with calcium hydroxide ($Ca(OH)_2$) in presence of water will set and solidify like concrete. Natural Pozzolans are characterized in four classes in view of the foremost lime responsive constituent present (Ramezanianpour, 2014). They are volcanic tuff and pumices, Un-modified volcanic glass, Calcined earth or shale and Raw or calcined opaline silica.

ii. **Artificial Pozzolans** are Pozzolans which can be acquired by warm treatment of characteristic materials and furthermore, they are materials with low pozzolanic reaction and need proper treatment to accomplish pozzolanic action (Ramezanianpour, 2014). Examples of artificial Pozzolans as highlighted by (Shetty, 2009) includes; FA, MHA, BFS, MK, SF, RHA and ashes of other cereal grain husk.

2.3.1 Standard Specifications and Test of Pozzolans

ASTM C618 (2015) classified Pozzolans into the following classes;

i. Class N: Raw or calcined typical pozzolans that consent to the germane requirements for the class, for instance, some diatomaceous earths; shales, cherts and opaline; tuffs and pumicites or volcanic slag, (calcined or un-calcined); and distinctive materials anticipating that calcination should incite appealing properties, for examples soils and shales.

Therefore, for a material to be a pozzolanic, it needs to satisfy ASTM C618-15 standard chemical and physical requirements as presented in Table 2.6 and 2.7.

Table.2.6: Standard Chemical Requirements

Material contents	Mineral Admixture Class		
	N	F	C
$\Sigma(SiO_2 + Al_2O_3 + Fe_2O_3)$ min, %	70.0	70.0	50.0

SO_3 max, %	4.0	5.0	5.0
Moisture content	3.0	3.0	3.0
Loss on ignition, max, %	6.0	6.0	6.0

Source: ASTM C618:2015

Table 2.7 Standard Physical Requirements

	Mineral Admixture Class		
	N	F	C
Fineness:			
Amount retained when wet-sieved on 45 μm (No. 325) sieve, max, %	34	34	34
Strength activity index:			
With Portland cement, at 7 days, min, percent of control	75C	75C	75C
With Portland cement, at 28 days, min, percent of control	75C	75C	75C
Water prerequisite, max, percent of control	115	105	105
Soundness: Autoclave expansion or contraction, max, %	0.8	0.8	0.8
Uniformity requirements: The fineness and density of independent samples shall not vary from the average established by the ten preceding tests, or by all preceding tests if the number is less than ten, by more than:	5	5	5
Density, max variation from average, %			
Percent reserved on 45-μm (No. 325), max variation, rate focuses from average	5	5	5

Source: ASTM C618:2015

Class F Fly ash, this is usually conveyed from expending bituminous coal or anthracite that qualifies the material specified for this group. This class of fly ash possess pozzolanic properties.

iii. Class C Fly ash are usually obtained from sub-bituminous coal that qualifies the material specified for this group. This class of fly slag possess some cementitious properties.

2.3.2 Pozzolanic Activity

Duggal (2008) states that when Pozzolan is combined with ordinary PC, the SiO_2 present in the Pozzolan reacts with the free $Ca(OH)_2$ discharged during cement hydration process and this is known as a pozzolanic activity. The pozzolanic activity happens because of the nearness of finely isolated polished $Ca(OH)_2$ and SiO_2 which yields calcium silicate hydrate (C-S-H) similar to products of PC hydration. The SiO_2 in the Pozzolan reacts with the $Ca(OH)_2$ residue from PC hydration process and adds to quality improvement. Hydration of PC may be illustrated as given in equation 2.1

$$C_3S + H_2O \longrightarrow \underset{\text{(Calcium silicate hydrate)}}{\text{C-S-H}} + \underset{\text{(Lime)}}{Ca(OH)_2} \qquad (2.1)$$

Calcium hydroxide formed from equation 2.1 combines with silica of the Pozzolan to produce calcium silicate hydrate as shown in equation 2.2

$$Ca(OH)_2 + \underset{\text{(Silica)}}{SiO_2} \longrightarrow \text{C-S-H} \qquad (2.2)$$

Silica of undefined shape respond with calcium hydroxide promptly than those of crystalline frame and this establishes the distinction concerning amorphous Pozzolans and similar materials which display little pozzolanic activity. Since pozzolanic action can happen only in the presence of water, enough water needs to be provided for complete pozzolanic activity. It is regularly felt that $CaO - SiO_2$ reaction is the principle or the main reaction that happens, however later data demonstrates that Al_2O_3 and Fe_2O_3 also participates in the reaction process as found in FA (Dwivedi *et al*., 2006; Duggal, 2008; Massaza, 2005).

2.4 Summary of previous work on MHA and CCW

In this chapter, concerted efforts were made to review the literature related to the study. Based on the review of related literature, it is clear that rigorous efforts were made by different researchers on utilisation of agro-industrial wastes as a partial cement replacement in mortar/concrete. These agro-industrial wastes were used in concrete or mortar to improve and enhance strength development and durability properties of mortar.

Existing studies reveals that:

MHA and CCW, MHA has been separately utilized as a partial replacement in both mortar and concrete as reported by Jimoh *et al.,* 2013; Ogork *et al.,* and 2014; Anowai *& Jones,* 2015. MHA was also combined with CCW and revealed 30% and 70% as the optimum of MHA to CCW content combination in mortar / concrete (Okwute, 2018). CCW was utilized in combination with other agro-waste and showed significant improvement in strength development as the curing age's increases (Olawuyi, *et al*., 2017; Enejiyon *et al*. 2018). They also reported that 30/70% of FA to CCW as an optimum combination proportion for good strength development.

The utilization of MHA and CCW solves the problem of chemical (sulphate, chloride) attack on mortar/concrete. There is high water required when MHA is used with cement in mortar/concrete production (Vinchan & Ranchan, 2012; Yunusa, 2015).

The knowledge gap in the study

i. Report on the total replacement of PC with alkali-activated MHA/CCW binder in mortar/concrete is scarce in literature.

ii. Report on approaches to enhancing the setting time and early strength development of agro-industrial waste by using alkali-activators is not common in the literature

iii. Indiscriminate disposal of waste generated from agricultural waste and over dependency on cement for construction activities will drastically reduce and thus minimized the quantum of CO_2 release to atmosphere during cement production.

What the study intends to do in filling the gap

i. Activating MHA-CCW binder by using alkali-activator (NaOH) to enhance early strength development of mortar made from this binder as means of solving slow rate of strength gains of agro-industrial binders.

ii. Developing a suitable mix proportion of MHA-CCW (using molar concentration of the constituent materials) to produce mortar with suitable strength.

iii. Development of alternative binder using MHA-CCW as a means of solving or reducing the waste disposal problem and thus leads to the production of environmental-friendly binder with low CO_2 emission.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

The materials used for this research are, Portland cement (PC), millet husk ash (MHA), calcium carbide waste (CCW), fine aggregate and water, and sodium hydroxide (NaOH).

3.1.1 Portland cement (PC)

The PC (CEM I 42.5 N) used for this research is the Dangote (3X) brand of Portland cement. The cement was obtained from the local cement dealers in Minna. Precaution was taken to ensure that the cement is of recent supply and free of adulteration. It is stated by the manufacturer as complying with BS EN 196-1:2016.

3.1.2 Calcium carbide waste (CCW)

Calcium carbide waste (CCW) was obtained in slurry form as a waste of acetylene gas production from the disposal area of a local automobile welder (Panel Beater) workshop in the Mechanical Village, Keteren-Gwari, Minna, Niger State. The CCW was sun dried for a day and calcined in a furnace at a temperature of 600^{o} C to obtain its amorphous form before use as CaO source. It was ground and sieved with 75 µm sieve and the particles passing used as the CCW sample as shown in appendix (plate IV).

3.1.3 Fine aggregate

The fine aggregate used for this research was clean and saturated surface dried river sharp sand. It was sieved and the materials retained between 1.18mm and 75µm was used for this

experiment which is consonance with BS EN 196-1:2016 (sand prescription for strength test of a binder).

3.1.4 Mixing and curing water

The water used for the production and curing of mortar samples of this research work is clean potable water available at the tap behind the Convocational Square of Federal University of Technology, Minna.

3.1.5 Sodium Hydroxide (NaOH)

NaOH in pallet form was purchased from Panlac Chemical Laboratory, Minna and different concentrations of 5, 10, 15, 20M were prepared for the study as shown in Appendix A21

3.1.6 Mortar mix details

The mortars were prepared using 50 mm cubes size. Appropriate combination proportions of MHA and CCW were determined through the molar concentration of SiO_2 content of MHA and CaO content of CCW. This was done by dividing reactive mass of SiO_2 and CaO by their molar masses to arrive at number of moles of the MHA and CCW and then divide each number of the mole by the summation of the number of the mole of MHA and CCW. The three combinations proportions of MHA to CCW that were determined from the molar concentration of SiO_2 and CaO are 40:60; 45:55 and 50:50 MHA: CCW. For the control mortar mix, (PC) CEM II 42.5N (Dangote (3X) brand) was used. 1:3 (c/s) at 0.5 water/cement (w/c) ratio as specified by BS EN 196-1:2016 for the PC as control, while the alkali-activation involved varied molar concentrations (0M to 20M at 5M steps) (NaOH). Thirty cubes were cast from each mix giving a total of 480 cubes specimen cast and cured for the varied curing ages (3, 7, 14, 28 and 56 days respectively). From the observation of

the setting times test in section 4.2.1. The samples with alkali-activator were de-moulded as determined from the setting time (49, 38, 34, 30, 25 and 24 hours) respectively and those without activators were kept in the mould covered with jute sacks and cured by water sprinkling for 72 hours before de-moulding and further cured in water by immersion**.** Table 3.1 & appendix A21 & A20 presents the details of the mortar mixes for respective specimen designation.

Table 3.1: Mix Details for Mortar Samples (kg/m^3)

Specimen	CEM I	MHA	CCW	Sand	Water	Molarities of NaOH Solution				
						0M	5M	10M	15M	20M
Control	900	0	0	2220	444	0	0	0	0	0
40/60 MHA/CCW	0	300	450	2220	449	0	123	245	367	490
45/55 MHA/CCW	0	405	495	2220	454	0	123	245	367	490
50/50 MHA/CCW	0	450	450	2220	459	0	123	245	367	490

3.2 Method

The method adopted for this study includes experimental plan, material characterization (physical and chemical properties), determination of the fresh and early-age properties and the long-term strength properties.

3.2.1 Experimental plan

The experimental procedure in actualizing the study objectives of this research work as outlined in section 1.3 was channelled to take after the work plan as laid out in this study and the details are listed in the following subsection:

Work plan one

Work plan one was designed to actualize objective one which centred on investigating the physical and chemical properties of the respective constituent materials (PC, MHA, CCW and Sand).

i. Samples used were examined in the laboratory for sieve analysis, specific gravity, bulk density, and other physical properties.

ii. Samples used were packaged and sent to different laboratory for X-ray Fluorescent (XRF) analysis information on the oxide composition: MHA (analysed at the National Geoscience Laboratory, Kaduna, Kaduna State); PC and CCW (analysed at Ewekoro Works Department of Lafarge Cement.

3.2.1.1 Work plan two

This is in line with objective two which is concerned with developing appropriate mix proportions of MHA and CCW for suitable binding properties with varied molarities of NaOH.

Appropriate combination proportions of MHA and CCW was determined through the SiO_2 content (73.4%) of MHA and CaO content (66.1%) of CCW. This was done by dividing reactive mass of SiO_2 and CaO by their molar masses (60 g/mol. & 56 g/mol.) to arrive at number of moles of the MHA and CCW and then divide each number of the mole by the summation of the number of the mole of MHA and CCW to arrive at the percentage mix proportion of 45/55 as shown in Appendix A21. Thus, this was varied to arrive at various combination proportions of MHA to CCW (40:60; 45:55; 50:50) respectively.

3.2.1.2 Work plan three

Work plan three was designed to achieve objective three which dealt with the evaluation of the fresh and early - age properties of the mortar made with/without alkali-activated binders.

Various tests (i.e., consistency, setting time, soundness and fineness test) were carried out on the binder combination types and the control.

The varied binder combinations – three in number as stated in above was prepared in comparison with the PC at 1:3 binder: sand mix in compliance with the BS standards for strength test on cement and determination of the early-age properties (density and compressive strength at 3, 7 days of curing).

3.2.1.3 Work plan four

Work plan four was designed to evaluate the hardened properties and assessing the effect of alkali-activator (NaOH) on the strength development of the various mix proportion of the mortar.

These properties were investigated by using the following tests and sample sizes:

i. Compressive strength – 50 mm mortar cubes at curing age of 14, 28 and 56 days.
ii. Water absorption – 50 mm mortar cubes at curing age of 28 and 56 days.
iii. Density- 50 mm mortar cubes at curing age of 14, 28 and 56 days.

3.2.2 Material characterization

The material characterizations for this research work are the chemical and physical properties of constituent materials (MHA, CCW, PC and NaOH).

3.2.2.1 Chemical properties

The oxide composition was determined using X-Ray Fluorescence (XRF) analysis. Samples (MHA, PC and CCW) used were packaged and sent to National Geoscience Laboratory, Kaduna, Kaduna State and Ewekoro Works Department of Lafarge Cement using XRF Analyser connected to a computer system for details information on oxide composition.

3.2.2.2 Physical properties of the constituent materials

The physical properties of the samples used that was examined in this study include: particle size distribution (PSD) of the fine aggregate, BET and Surface area of the binders (fineness), specific gravity and moisture content of the various samples.

a. Particle size distribution analysis (PSD)

Particle size distribution (PSD) of the available natural sand was carried out using the dry-sieve approach in accordance with BS EN 196-1:2016 for proper classification of the available natural sand. The apparatus used are the sieve of different sizes (4.75mm, 2.36mm, 1.18mm, 600µmm, 300µmm, 150µmm, 75µmm, and pan), sieve brush, weighing balance, scoop, watch, and mechanical sieve shaker. The particles retained on the 75µm sieve were used for the mortar mixture for the strength test.

The results from particle size distribution of the fine aggregate were plotted on a semi-log graph with sieve particle diameter or size on the X-axis with the logarithmic axis while the Y-axis indicates the percentage passing. Figure 4.1 provides a perfect picture of the particle size distribution. As stated in Olawuyi & Olusola (2017), D_{60} is the particle diameter

passing 60%; D_{30} is the particle diameter passing 30% and D_{10} is the particle diameter passing 10% were determined for further analysis to obtain the coefficient of uniformity (C_u) and coefficient of curvature (C_c) (Equation 3.1 and 3.2) and hence the classification of the sand particles.

$$\text{Uniformity Coefficient } (C_u) = \frac{D60}{D10} \quad (3.1)$$

$$\text{Coefficient of Curvature } (C_c) = \frac{D30}{D10\ X\ D6} \quad (3.2)$$

Well graded requirements using the C_u and C_c were presented by Vandevelde (2008) as $C_u \geq$ 4 for gravel; $C_u \geq$ 6 for sand and C_c= 1 to 3 for all type of soil. Soils having a $C_u <$ 2 are classified as uniformly graded.

Shetty (2009) presents the following boundaries to be taken as control for Fineness Modulus (F.M) of sand for concrete works.

F.M for Fine Sand: 2.2 – 2.6

F.M for Medium Sand: 2.6 – 2.9

F.M for Coarse Sand: 2.9 – 3.2

A sand having F.M. >3.2 will be unfit for producing suitable concrete.

b. Specific Gravity Test of Binder and the Aggregate Used

The specific gravity of a material is the ratio of the weight of a given volume of that material to the weight of an equal volume of water displaced. Thus, the test was carried out as specified by BS EN 1097: 2003. The apparatus used in conducting the test includes a funnel, weighing balance, density bottle, spatula and stopper.

Therefore, the Specific gravity (Gs) of the materials was determined by using equation 3.3

$$\text{Specific gravity Gs} = \frac{(w2-w1)}{(w4-w1)-(w3-w2)} \quad (3.3)$$

w1 is the weight of bottle

w2 is the weight of the bottle + dry sample

w3 is the weight of the bottle + sample + water

w4 is the weight of the bottle + water

c. Fineness

Fineness test was also carried out on the PC and the varied combination of binders (MHA: CCW) via dry-sieving method as postulated by BS EN 196-6:2016 using a 75μm sieve available in the Building Concrete Laboratory.

d. Bulk Density Test of Binders and the aggregate Used

The bulk density of the fine aggregate was calculated in accordance with ASTM C642 2006. The apparatus used was density cube (wooden), trowel, rammer and weighing balance. The bulk density for aggregate sample was computed through the equation 3.4

$$D = \frac{M}{V} \quad (3.4)$$

Where;

D = density of the aggregate sample in kg/m^3

M = mass of the aggregate sample in kg

V = volume of the aggregate sample in m^3

Meanwhile, the mass of the aggregates sample was determined by subtracting the weight of empty container from the weight of container plus aggregate sample using equation 3.4

$$M = H - G \quad (3.5)$$

Where;

M = mass of the aggregate sample in kg

G = weight of the empty container in kg

H = weight of container plus aggregate sample in kg

e. BET specific surface area of the Binders

This was conducted to ascertain the reactivity of the particles of the binders (MHA & CCW). It was carried out with the use of Malvern Zetasizer Instrument at the Centre for Genetic Engineer and Bio-informatics Technology, Minna, Niger State.

f. Total Moisture Content Determination

The test step for the determination of total moisture content is covered by ASTM C566: 2013; Neville, 2012. These involved drying the known weights of the material sample in the oven for a period of 24 hours at a temperature over 105 ^{o}C as show in appendix (Plate VII). The weights of the dry sample were noted after oven-drying to determine the weight of evaporated water. The formula for moisture content is shown in equation (3.6).

$$\text{Moisture content (P)} = \frac{Initial\ weight\ of\ sample - Dry\ weight\ of\ sample}{Dry\ weight\ of\ sample} \quad (3.6)$$

3.3 Fresh and Early-Age Properties

The fresh and early-age strength properties conducted includes; consistency, setting time, and soundness tests of the pastes and determination of the early age strength development of the alkali-activated mortar.

3.3.1 Consistency

It is the measure of the water require for a standard paste in accordance with ASTM C566-2013 with the use of Vicat apparatus with the plunger of 10 mm diameter, trowel, weighing balance, weight box and measuring cylinder. This involved determining the water demand to make a paste which will give the suitable standard consistency (Neville, 2012). Vicat apparatus Model No EL 38 - 2010 by ELE was used for measuring the consistency following the procedures as outlined in the standard (BS EN 196-3:2016). 300 g of the binder was weighted carefully and 25% water added. Care was taken to ensure that the time of mixing is not less than 3 min and gauging recorded from the time when the water was added. The paste was then filled into the mould with excess paste trimmed off and shaking done to eliminate air foams. The 10 mm diameter plunger was fixed in the moving rod and brought down to meet with the paste and the plunger released. The process was repeated till it penetrated 33 – 35 mm from the top & water demand noted.

The percentage of water (p) was computed by weight of dry cement require to prepare cement paste of standard consistency

$$P = \frac{W}{B} X\ 100 \qquad 3.7$$

Where: M = Quantity of water added

B = Quantity of Cement used

3.3.2 Setting Time of Binders

These involves determine the initial and final setting time of the binders paste. The Vicat apparatus, needle, stop watch, non-porous plate, weighing balance, measuring cylinder and weight box was used. The initial and final setting times for the PC and the various mix proportion combinations of MHA/CCW with alkali-activated were determined using neat pastes of standard consistency in accordance to BS EN 196-1:2016.

3.3.2.1 Initial Setting Time

The is the period elapsed between when the water was added to the binder and the time at which the needle (1 mm^2 cross-section needle) gives a reading between 4-7mm from the bottom of a standard Vicat apparatus. This is known as initial setting time of that particular binder's paste as illustrated in table 4.5.

3.3.2.2 Final Setting Time

This is the period elapsed between the initial contact of binder and water and the time when the smaller needle (1 mm^2 cross-section and 0.5 mm deep) completely penetrates into the paste and the outer angular metal attachment of 5 mm diameter does not leave an impression on the binders paste. The final setting time results recorded in this experiment was illustrated in table 4.5.

3.3.3 Soundness of Binder

This is to assess the performance of the binder paste from the period of dipping in water for roughly 24 hours ± 4 hours and to determine the extent of expansion after the sample has been boiled for about 3 hours. It was conducted on paste of standard consistency for

various binder combination proportions. The apparatus that was used are Le-Chatelier mould, spatula, veneer caliper, glass tray and bath. Le-Chatelier apparatus Model No EL 38 – 3400 by ELE according to BS EN 196-1-2016 was used. The Le-Chatelier mould was oiled, placed on an oiled glass sheet, filled with prepared binder paste and covered with another oiled glass sheet. The whole samples were immersed in water and kept for 24 hours. The whole assemblage was detached from water bath and the distance separating the indicator points measured to the nearest 0.5mm (B_1) and further immersed the whole assembly in water bath and kept boiled the samples for 3 hours. After completion of 3 hours, the temperature of the water bath was allowed to cool down to a room temperature and then the whole sample was detached from the water bath. The distance between the two indicator points was again measured to the nearest 0.5mm (B_2).

$$\text{Expansion of the binder paste} = B_1 - B_2 \qquad 3.8$$

Where;

B_1 = Measurement taken after 24 hours of submerged in water

B_2 = Measurement taken after 3 hours of immersion in boiling water

3.3.4 Preparation of specimen in the laboratory

The mortar sample mix proportion of 1.3 that is binder to sand with water cement ratio of 0.5 was adopted in conforms to the strength of cement. The varied molarities of NaOH (5, 10, 15 and 20M) were dissolved in the water ratio calculated to make alkaline water. 1mole of NaOH is equal to 40g as was revealed in previous literature. The cement – sand, MHA/CCW- sand / normal water was used as control while normal water and cement were complete replaced alkaline water and MHA/CCW with the following proportions 40/60, 45/55 and 50/50 and each mixed proportion was activated with 5, 10, 15 and 20M of

NaOH. The binder and fine aggregate were batch by weight and mixing was done by manual mixing due to the quantity of the constituent materials. The fresh mortar from these mixes was cast into 50mm cube moulds. The mortar cubes were cured in water for various curing ages (3, 7, 14, 28 and 56days) before the hardened tests were carried out. After the curing period, the cubes were removed and air dried, three cubes were then randomly picked and tested for water absorption and compressive strength. The average value of each was noted and recorded.

3.3.4.1 Mixing of mortar Constituent Materials

The mixing of mortar constituent materials was done by manual mixing since the volume of the mortar to be cast was small and do not require mechanical mixer. In this study, the mixing of mortar was carried out by manual method. The batching method employed for this research work was batched by weight. The amount of sand was first weighed by weighing balanced and poured into the mixing tray, and then measured amount of binder was thoroughly mixed with the sand until uniformity was attained. Finally, the required quantity of mix alkaline water / water was poured into the measured sand-binder and thoroughly mixed for about 3minute to achieve uniform paste mortar.

3.3.4.2 Casting of Cube Samples

The casting of the cube samples was carried out immediately after achieving the uniform mix of the binders. The mortar was cast into an already oiled 50mm cubes mould to reduce friction and aid easy de-moulding of the cubes. The moulds were filled and compaction was achieved by the use of electrical vibration table for about three minutes so as to achieve a mortar with fewer voids. The surface of the moulds was levelled, smoothed and marked for

identification. After this, the PC mortar samples were left in the mould for 24 hours 35 minutes (95minuties) and 77, 68, 53, 38, 30 and 25 hours was used for MHA/CCW mortar cube with water and alkaline water due to the differences in their setting time so as to achieve some hardening of the cubes before de-moulding and commencement of the curing as shown in Table 4.5. A total of 480 cubes were cast and cured in water to determine their strength at 3, 7, 14, 28 and 56 days respectively.

3.3.4.3 Curing

The method of curing used in this study was by immersion of the cubes in water and this was for specified ages of 3, 7, 14, 28 and 56 days for strength, density and 28 and 56 days for water absorption as postulated by ASTM C 266-2008.

3.4 Long Term Strength Properties

The long-term strength properties studied are density; compressive strength and water absorption of the hardened mortar at the respective curing ages.

3.4.1 Density

The mortar samples were brought out of the curing tank and placed in open-air to surface dry, then the mass of the mortar samples was determined using weighing balance in compliance with BS EN 934 – 2: 2009. The density of mortar sample was then calculated, using equation 3.9.

$$\text{Density (D)} = \frac{M}{V} \qquad 3.9$$

Where

D = Density of the mortar sample in kg/m^3

M = Mass of the mortar sample in kg

V = Volume of the mortar sample in m^3

3.4.2 Compressive strength

The mortar samples (cubes) were carried out from the curing tank and put in open-air to surface drying, weighed and placed at the centre of hydraulic manual compression machine for crushing (Digital Universal Testing Machine - DUTM) as shown in appendix plate VI. The mortar samples were subject under load for testing and the force was applied to the sample by swinging the handle of the crushing machine till it failed to check its behaviour under load. The compressive strength test on mortar sample was determined in consonance with BS EN 196 – 1 (2016)

Compressive strength – 50mm cubes

$$\text{Compressive strength } F = \frac{P}{A} \qquad 3.10$$

Where;

F = Compressive strength in N/mm^2

P = Maximum load at failure, in N

A = Cross-sectional area, in mm^2.

3.4.3 Water absorption test

The mortar samples (cubes) were removed from the curing tank and allowed and then placed in the electronic oven to oven-dry at 105 ^oC for 72 hours. The specimens were brought out of the oven after being allowed to cool to room temperature before measurement was taken to determine the initial weights (w_1). The final weights (w_2) were determined after immersing the mortar samples in the curing medium for 30 minutes,

removed, dried and re-weighed again. The values obtained were thereby used to calculate the water absorption rate of the mortar sample in accordance with BS 1881-122 (2011) using Equation 3.11.

$$\text{Water absorption} = \frac{w2-w1}{w1}\ x\ 100 \qquad 3.11$$

Where W1 = weight of the samples after removing from oven

W2 = weight of the sample after 30 minutes immersion in water

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Characterization of the constituent Materials

The experiments conducted for characterization of the constituent materials (MHA, CCW, PC and NaOH) includes physical (PSD, specific gravity, Bulk density, moisture content and BET surface area) and chemical properties (determination of oxide compositions).

4.1.1 Particle Size Distribution (PSD)

Particle size distribution (PSD) of the natural sand used for the experiment is presented in Figure 4.1. The summary of the sieve analysis (Table 4.1) reveals the sand used to be well graded and of fine classification (C_c = 2.5, C_u = 0.96 and FM = 2.3) in consonance with Shetty (2009) specification (fine sand classification). The PSD and BET specific surface area tests conducted reveals the MHA as meeting the quality criteria of even dispersal giving an average particle size of 180.23 nm and BET specific surface area value of 360.5m^2/g. The data quality of the CCW sample was observed to be fairly poor and poly-disperse for distribution analysis and this is due to large sedimenting particles and florescence/absorbent nature of the sample shown in Appendix (Table A17 and 18). It has an average particle size of 109.01 nm and BET specific surface area of 414.24 m^2/g.

Table 4.1: Summary of the Sieve Analysis

D_{10}	D_{30}	D_{60}	C_c	C_u	FM
0.22	0.34	0.55	2.50	0.96	2.30

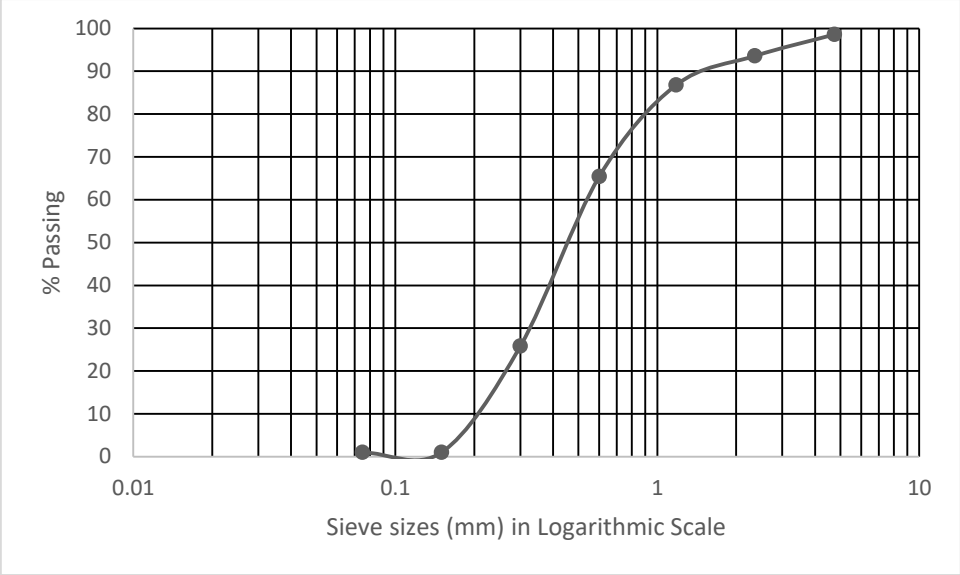

Figure 4.1: Plot of Particle Size Distribution of Natural Sand

4.1.2 Specific Gravity

The result of average specific gravities of the constituent materials (natural sand and binder) used for this research work is presented in Table 4.2. The results indicate that the values fit well with the previous report in literature (Neville, 2012) and it was conducted according to the specification of BS EN 1097-6:2013.

Table 4.2: Specific Gravities of Constituent Materials (kg/m^3)

Samples	Average Gs
Cement	3.14
MHA	2.20
CCW	2.09
NaOH	1.65
Sand	2.62

Bulk density refers to the weight of aggregate needed to fill a container of unit volume. Both compacted and uncompact (loose) bulk density test conducted on the fine aggregate

used revealed that the result of loose sand was 1558 kg/m^3 and that of compacted was 1594 kg/m^3 as shown in Appendix, Table A19. These results revealed that the sample satisfactorily fit into the description of normal weight aggregate presented in ASTM C642, 2006 as aggregate having bulk density range from 1120 kg/m^3 – 1920 kg/m^3

4.1.3 Moisture Content

Moisture Content of the fine aggregates should be within 0 to 10 % as postulated by ASTM C618: 2015. Also, according to ASTM C618: 2015 moisture content limits for binders should be within 0 to 3%. From Table 4.3 the value of the moisture is within the specified acceptable limits.

Table 4.3: Moisture Content of constituent Materials

Materials	Average Mc (%)
CEM 1	0.0
MHA	0.0
CCW	0.3
Sand	1.3

4.1.4 Chemical Analysis of the Cementitious Materials

The oxides composition of the different cementitious materials was conducted at National Geoscience Laboratory, Kaduna and the Ewokoro Works Department of Lafarge Cement. The major component of MHA is SiO_2 (74.2 %). MHA is thus classified as a class F Pozzolan since the aggregation of the main oxides (SiO_2 + Al_2O + Fe_2O_3) gives 88.1% which is above 70 % minimum limit stipulated in ASTM C618 (2015) standard as presented in Table 4.4. Also, SO_3 is below 4% and Loss on Ignition (LOI) is less than 10%

conforming to the codes specification. The CCW was observed to contain 66.1 % CaO similar values to the CaO content (65.0 %) of the PC and lower SiO_2 and Al_2O_3. The LOI of 28.2 % of CCW was observed in Table 4.4 which is above the specified 10% maximum and similar to that reported by Manesseh & Joseph (2016) of 32.51%. This shows that some more heat is require for effective performance.

Table 4.4: XRF Analysis for Oxide Composition of Cementitious Materials

Samples	SiO_2	Al_2O_3	LOI	Fe_2O_3	MgO	CaO	K_2O	TiO	MnO	SrO	CuO	ZnO	BaO	SO_3	$SiO_2+Al_2O+Fe_2O_3$
MHA	72.4	14.2	2.0	1.5	1.0	6.4	0.2	0.0	0.5	0.0	0.0	0.2	0.1	0.1	88.1
CCW	5.5	1.8	28.2	0.3	0.1	66.1	0.1	0.1	0.0	0.0	0.0	0.0	0.1	0.2	7.6
CEM1	21.3	5.1	0.0	1.1	2.8	65.0	0.0	0.1	0.0	4.4	0.0	3.5	0.0	4.5	27.5

4.2 Fresh and Early Strength Properties

The experimental test conducted to determine the fresh and early strength properties of the mortar pastes of alkali-activated MHC/CCW and control (PC) are consistency, setting time and soundness. The summary of water demand, setting times and soundness of the binders (i.e., PC and MHA: CCW) pastes with different molarities of NaOH contents are presented in Table 4.5

4.2.1 Consistency (Water Demand)

Table 4.5 reveals that the PC recorded the least standard consistency of 28.3% and that of alkali-activated MHA/CCW is 46.6% of mix proportions 45/55 and 50/50% at 0 and 5M respectively.

It is clear that water required to form a workable paste increase with increase in CCW content and various molarities of NaOH. This progressive in water require is due to the smaller particle size of CCW and MHA which increase surface area of the whole mix proportion translating into higher water required (ASTM C128: 2015; Neville, 2012), therefore, the water demand to make a workable paste of alkali-activated MHA-CCW is twice that of PC (control).

Table 4.5: Summary of Water demand, setting times and soundness test of the Binders

Specimen No	Binder Type	Water Demand (%)	Penetration (mm)	Soundness Expansion (mm)	Initia Setting Time (mins.)	Final Setting Time (mins)
PC	CEM 1	28.3	5.0	0.0	60	95
40/60	0 M	56.6	6.0	0.5	305	270
	5 M	56.6	5.5	0.0	135	210
	10 M	56.6	5.5	0.0	105	150
	15 M	63.3	5.0	1.0	90	110
	20 M	63.3	5.0	0.5	105	120
45/55	0 M	46.6	5.5	0.5	270	305
	5 M	40.2	6.0	0.0	120	152
	10 M	36.3	5.5	0.5	90	120
	15 M	31.5	5.0	1.5	75	100
	20 M	35.4	5.5	1.0	90	135
50/50	0 M	44.6	5.5	0.5	240	375
	5 M	42.6	6.0	0.0	150	195
	10 M	42.0	5.5	0.5	120	145
	15 M	40.0	5.0	1.5	90	115
	20 M	45.0	5.5	1.0	105	135

4.2.2 Setting Times of Binder Pastes

Table 4.5 reveals the initial and final times of cement /agro-industrial with or without alkaline activator. It was observed that there are progressive decreases in both initial and final setting time of Pozzolan cements as the molarities of NaOH increased from 5 to 15M but experienced a sharp increase at 20M across all mix proportions.

For 40:60 (MHA: CCW) mix proportion, the initial and final setting of the Pozzolan cement paste was recorded as 305 and 207 minutes as compare to control (PC based mortar). As the NaOH molarities increased for the Pozzolan cement paste, there exists progressive decrease in both the initial and final setting as shown in Table 4.5. The 40:60 (MHA: CCW) at 15M NaOH molarity possessed fresh properties close to that of control (PC based mortar).

For 45:55 (MHA: CCW) mix proportion, the initial and final setting was noted to be 270 and 305 minutes as compare to PC based-mortar (28.3 and 95 minutes). As the NaOH molarities increased, there was a progressive decrease in both the initial and final setting as shown in Table 4.5. The specimen at 15M NaOH molarity revealed similar fresh properties to that of PC based-mortar.

For 50: 50 MHA/CCW mix proportion, there was a progressive decrease in both the initial and final setting time as the activator dosage increased from 5 to 15M as shown in the mix proportions above.

Optimum mix composition for acceptable setting time of the alkali-activated MHA: CCW is adjudged to be 45: 55 (MHA: CCW) activated with at 15M of NaOH since it possessed fresh properties closest to that of the PC based mortar.

4.2.3 Soundness of Binder Pastes

The soundness test conducted revealed that all the binder combinations with NaOH or without conform to the 10 mm maximum expansion prescribed by BS EN 1881-122: 2011

as shown in Table 4.5. This means that all the binder mixes were suitable for mortar productions on basis of soundness consideration alone.

In reference to Section 3.1.6 (Table 3.1 and Appendix (Figure C1) and the fresh properties studied, it can therefore be inferred that 45: 55 (MHA: CCW) at 15M NaOH activation is the appropriate mix proportion of the MHA-CCW binder for effective performance in mortar.

4.3 Hardened properties of the Mortar

The hardened properties of mortar samples studies from the varied mixed proportion combinations of alkali-activated MHA-CCW in comparison with the PC based-mortar are density, compressive strength and water absorption.

4.3.1 Density of Mortar Samples

The average density of the control and various combination proportions of alkali-activated MHA-CCW at 28 and 56 days respectively are presented in Figures 4.2 to 4.4 and Appendix (Table A2 to A6). The average density of the alkali-activated MHA-CCW mortar samples and the control varies from 2099 kg/m^3 to 2182 kg/m^3. It showed that the density increased as the curing age increased. Mortar samples with density above 2000 kg/ m^3 are considered as normal weight according to ASTM C642 (2006).

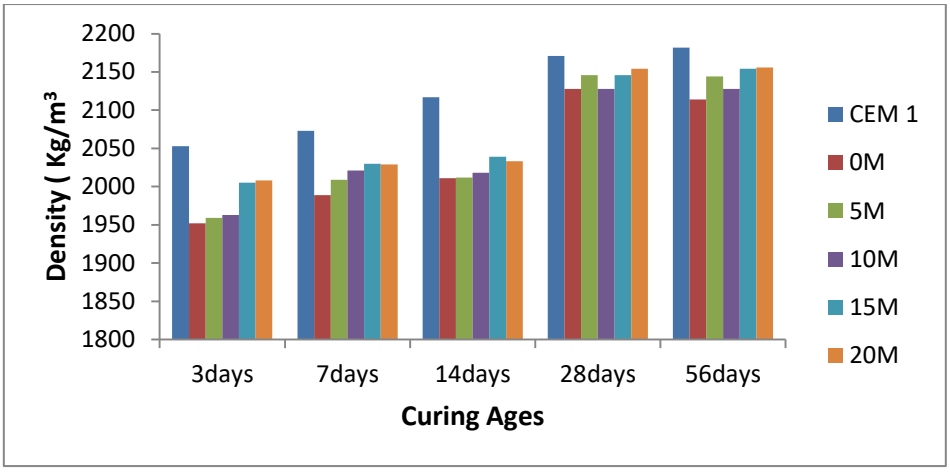

Figure 4.2: Average density of 40:60 (MHA: CCW) mortar samples cured in water

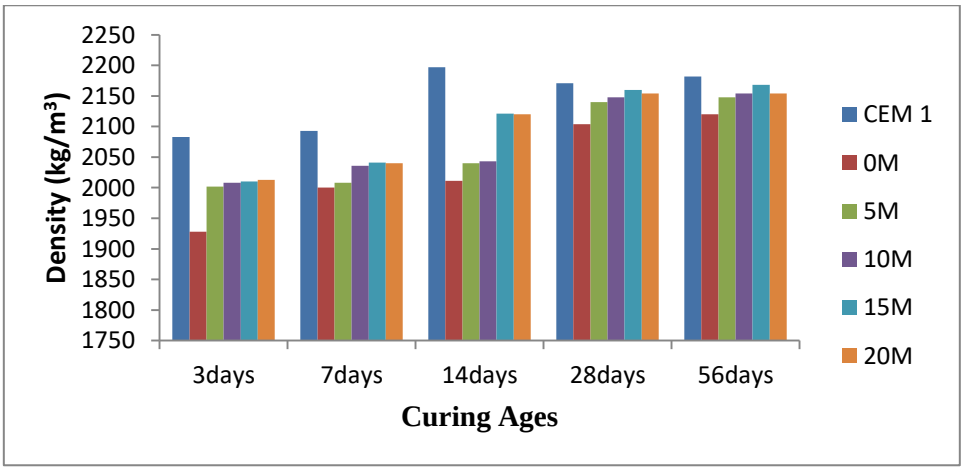

Figure 4.3: Average density of 45:55 (MHA: CCW) mortar samples cured in water

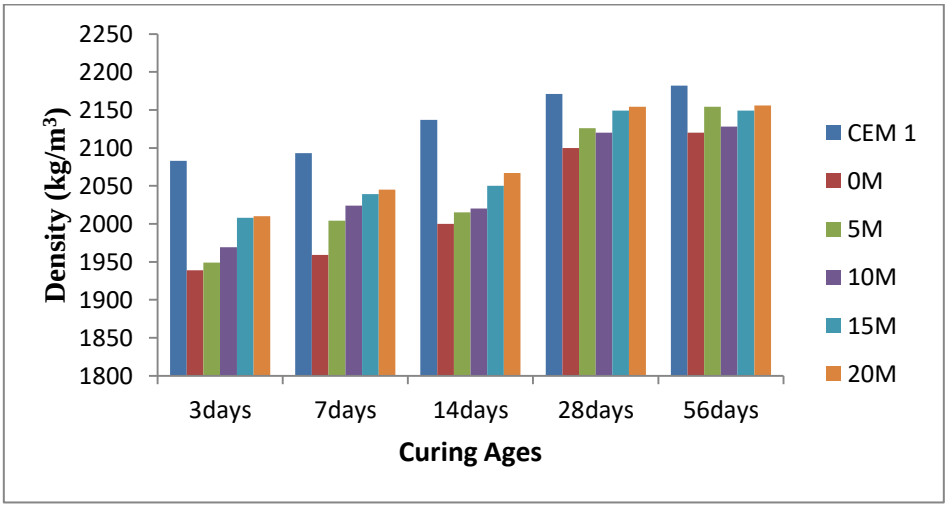

Figure 4.4: Average density of 50:50 (MHA: CCW) mortar samples cured in water

4.3.2 Compressive Strength of Alkali-Activated MHA-CCW Mortar

The compressive strength of Alkali-activated mortar at varied molar concentrations of NaOH solution as shown in Table 4.6 and Figures 4.5 to 4.7 and Appendix (Table, A7).

Table 4.6: Compressive Strength of Alkali-Activated MHA-CCW Mortar

Specimens		NaOH (M)	Compressive Strength (N/mm^2)					CS_{28} Factor				
			3days	7days	14days	28days	56days	3days	7days	14days	28days	56days
CEM II		0	10.24	16.02	20.26	26.29	28.04	0.39	0.61	0.77	1.00	1.07
MHA: CCW	40:60	0	1.80	1.96	3.33	7.44	7.92	0.24	0.26	0.45	1.00	1.06
		5	2.68	3.38	5.35	9.76	11.48	0.27	0.35	0.55	1.00	1.18
		10	4.59	6.98	9.71	13.84	15.28	0.33	0.50	0.70	1.00	1.10
		15	6.52	9.00	15.33	18.45	22.66	0.35	0.49	0.83	1.00	1.23
		20	4.00	5.85	10.17	16.02	18.52	0.25	0.37	0.63	1.00	1.16
	45:55	0	1.96	2.08	3.84	7.68	8.20	0.26	0.27	0.50	1.00	1.07
		5	2.80	3.91	5.44	10.28	12.20	0.27	0.38	0.53	1.00	1.19
		10	4.77	7.78	10.19	14.69	16.44	0.32	0.53	0.69	1.00	1.12
		15	6.92	10.72	15.96	19.77	24.52	0.35	0.54	0.81	1.00	1.24
		20	4.72	6.12	10.56	16.45	20.64	0.29	0.37	0.64	1.00	1.25
	50:50	0	1.84	2.00	3.76	7.23	8.04	0.25	0.28	0.52	1.00	1.11
		5	2.72	3.87	5.41	10.00	11.56	0.27	0.39	0.54	1.00	1.16
		10	4.65	7.53	9.95	14.36	15.88	0.32	0.52	0.69	1.00	1.11
		15	6.80	9.95	15.48	19.32	23.24	0.35	0.52	0.80	1.00	1.20
		20	4.20	5.96	10.21	16.25	19.88	0.26	0.37	0.63	1.00	1.22

At early age of 3 days, the 45:55 (MHA: CCW) activated with 15M of NaOH produced the highest strength of 6.92 N/mm^2 (i.e., 35 % of its 28th day strength – CS_{28}) which is higher than the stipulated minimum strength of 3 N/mm^2 for load bearing Sandcrete blocks as postulated by the Nigeria Industrial Standard (NIS, 2004).

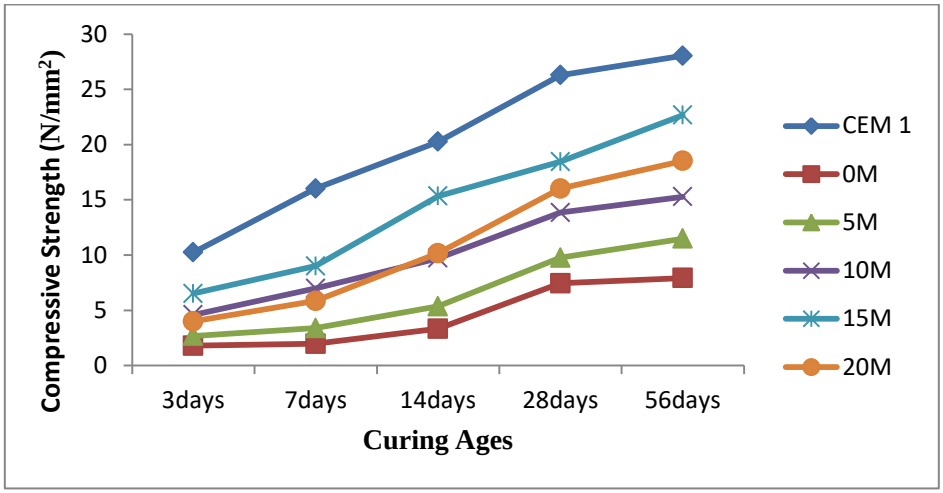

Figure 4.5: Compressive Strength of 40:60 (MHA: CCW) mortar samples

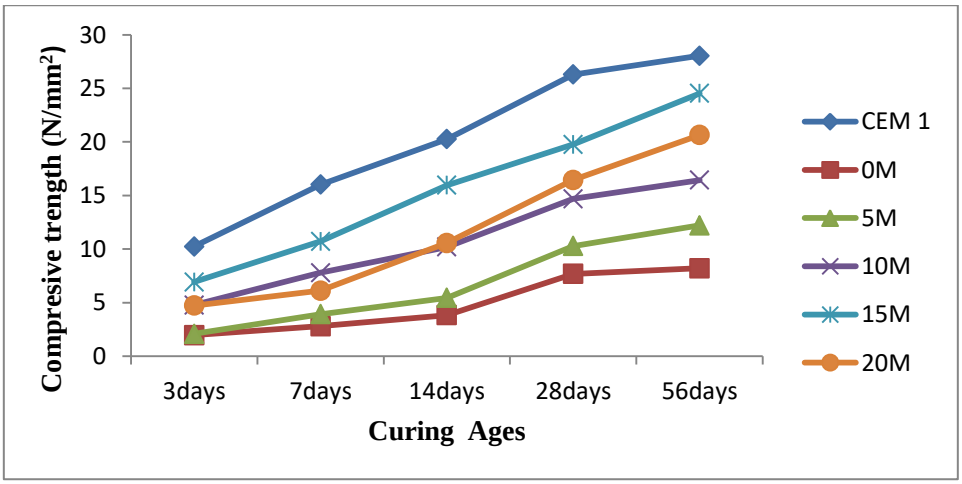

Figure 4.6: Compressive Strength of 45:55 (MHA: CCW) mortar samples

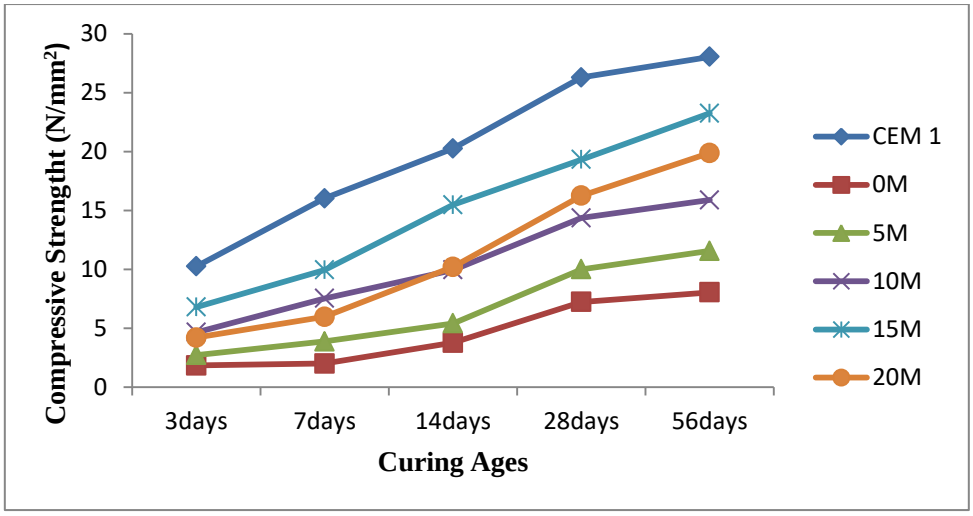

Figure 4.7: Compressive Strength of 50:50 (MHA: CCW) mortar samples

As the curing age of alkali-activated MHA-CCW increased, it was observed that rate of strength development increased with increase in concentration of NaOH from 5M to 15M but at 20M, slight decrease in strength was observed. This implies that the maximum strength recorded for the alkali-activated MHA-CCW on the 56th day was at 45:55 proportion combination activated with 15M of NaOH, similar value was observed for the 50:50 samples also at 15M NaOH. This conforms to the finding by Ramujee & Potharaju (2014) which reported that compressive strength of alkali-activated binder increases as the molar concentration of NaOH increased up to 15M. This concentration of NaOH resulted in good bonding between the aggregate and the mortar paste until the maximum concentration for activation is achieved.

The strength of the MHA-CCW binder-based mortar also increased as the curing ages increased for all the activated combination proportions of MHA-CCW as earlier affirmed by Barbosa, *et al*. (2000) and Fernandez-Jimenez *et al*. (2004). The 7- and 14-days strength for 45:55 (MHA: CCW) activated with 15M NaOH were 10.72 N/mm^2 (67 %) and 15.96 N/mm^2 (79 %) respectively of the PC-based sample at same age as shown in Appendix

(Table A8 and A9). The 28days strength for the 45:55 MHA-CCW, 15M NaOH sample showed higher strength gain (29 %) as against 23 % strength gain of the PC-based mortar. Further curing of alkali-activated MHA-CCW mortar till 56 days resulted in additional 24 % strength gain over the 28^{th} day as compared to the 7 % increase of the control. The trend is same for all the alkali-activated MHA-CCW mortar studied up to 15M NaOH concentrations. The 15M NaOH activation of 45:55 (MHA: CCW) is considered by this study as the indicated proportion for good strength.

4.3.3 Water absorption

Figures 4.8 – 4.10 and Appendix (Table A11 – A15) presents the results of water absorption of the MHA-CCW and PC based mortars with or without alkali-activation.

The water absorption of the MHA-CCW mortar was observed to be more than that of the PC-based mortar. This is likely due to the porous nature of both MHA and CCW as reported by Okwute, (2018) and also affirmed by the consistency results presented by Onuche *et al.,* 2019.

As the various combinations of MHA-CCW were activated with alkaline water (NaOH), the rate of water absorption by mortar cube decreases across the various combination proportions up to 15M but shows slightly increase at 20M NaOH activation on the 28 days and 56 days of curing respectively. The result shows that rate of water absorbed by mortar cubes at 15M (45:55) is similar to that of PC-based mortar.

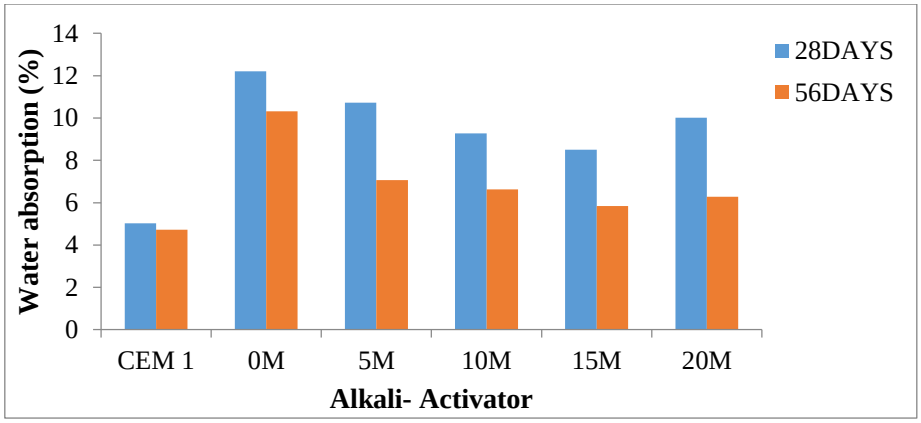

Figure 4.8: Average water absorption of 40/60 MHA/CCW binder combinations

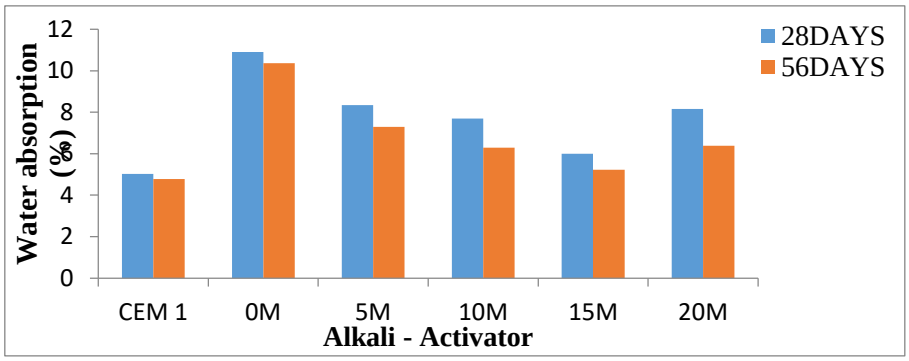

Figure 4.9: Average water absorption of 45/55 MHA/CCW binder combinations

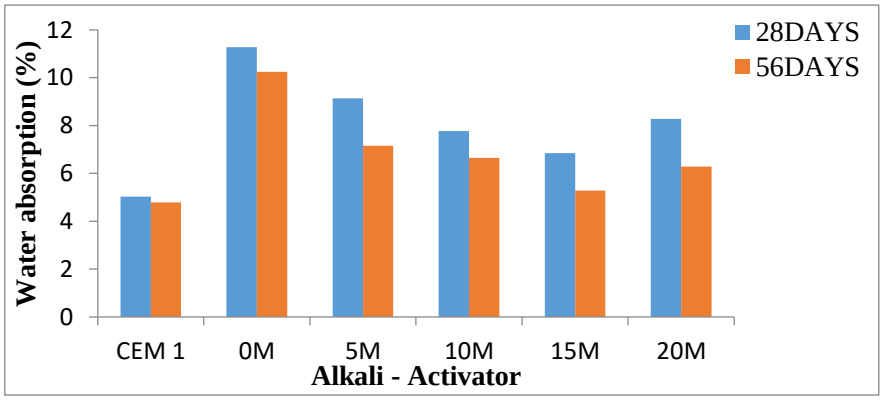

Figure 4.10: Average water absorption of 50:50 (MHA/CCW) binder combinations

4.4 Statistical Analysis and Implication of Results

Figure 4.11 reveals the influence of NaOH molarities on the compressive strength of the alkali-activated MHA-CCW binder based-mortar. The compressive strength generally increased as the NaOH molarity increases up to the 15M NaOH concentration. The NaOH

molarity effect on the mean compressive strength further affirms the 15M NaOH as the optimum concentration of NaOH for best performance in strength for the NaOH activated MHA-CCW binder-based mortar.

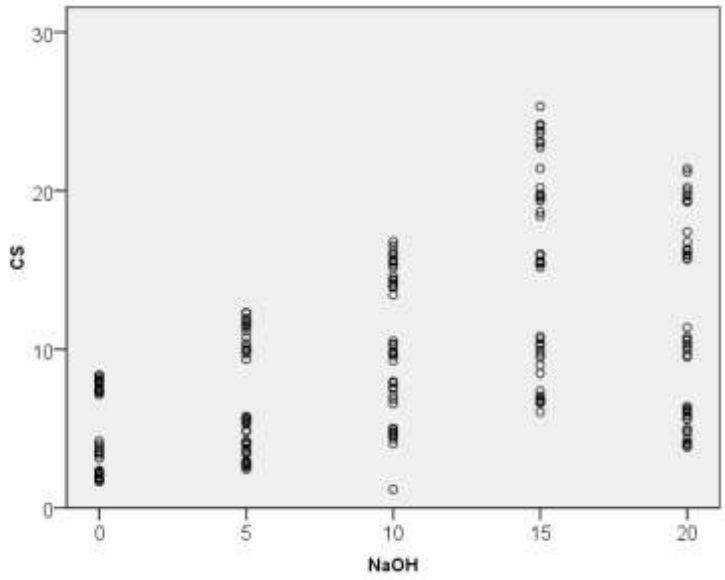

Figure 4.11: Influence of NaOH contents on Compressive Strength

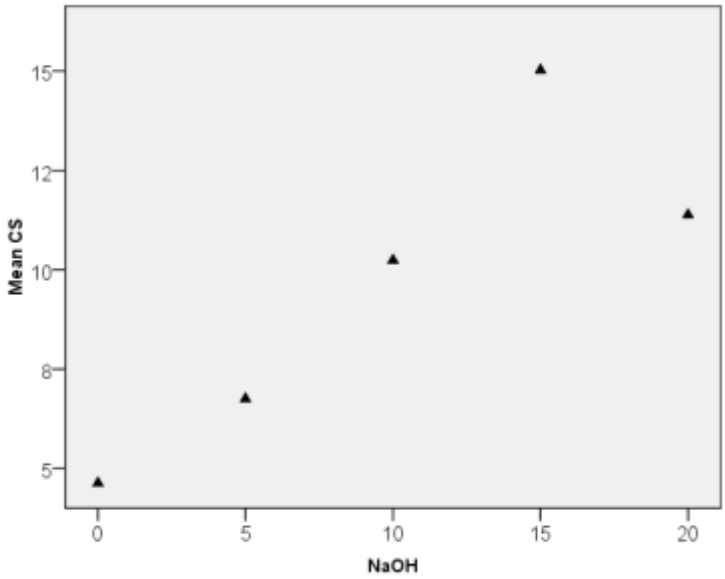

Figure 4.12: Influence of NaOH Molarity on Mean Compressive Strength Values

Influence of the NaOH molarity on the mean water absorption of the alkali-activated MHA-CCW binder-based mortar also affirm the 15M NaOH as the optimum concentration of the mortar for best performance in the durability property examined.

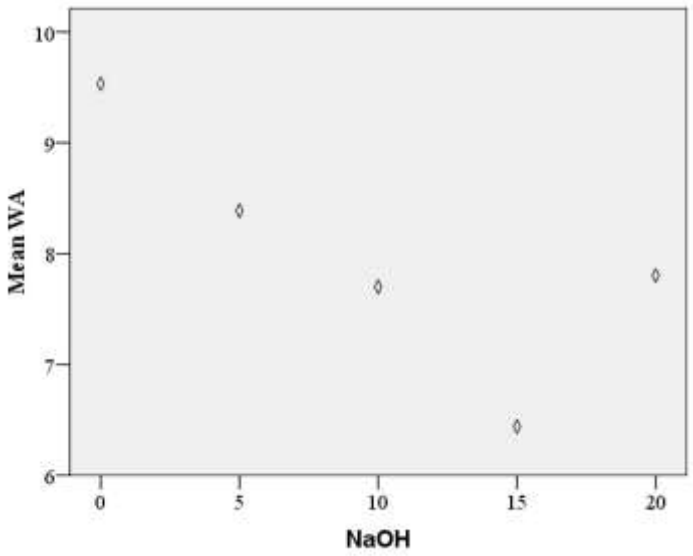

Figure 4.13: Influence of NaOH Molarity on Mean Water Absorption Values

Influence of MHA, CCW, NaOH molarity and Curing age (called independent variables) were examined on the compressive strength and water absorption (dependent variables) of the alkali-activated MHA-CCW binder-based mortar as reported in Table 4.7 using statistics (general linear model - multivariate). This is to examine which of the independent variables had significant effect on compressive strength and water absorption of the mortar. The statistical analysis indicated that all the independent factors when considered individually had significant effect on both the properties examined at 95 % confidence level ($\alpha = 0.05$). The test between-subject effects also reveal significant effects except combined effect of NaOH and curing ages (as highlighted) at the two-factor analysis level which is not significant. This implies that whenever any of the factors (MHA, CCW, NaOH molarity, and curing age) vary, the compressive strength and water absorption also changes.

The degree of variation is proportional to the magnitude of the change. The coefficient of determination (adjusted R-Square value) obtained from the analysis was 0.992 (99.2 %). This suggests strong statistical association between each independent variable and the dependent variables.

Table 4.7: Tests of Between-Subjects Effects

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	CS*	3233.150[a]	31	104.295	399.624	.000
	WA*	368.081[b]	31	11.874	57.756	.000
Intercept	CS	22159.182	1	22159.182	8.491E4	.000
	WA	4064.914	1	4064.914	1.977E4	.000
NaOH	CS	2170.132	4	542.533	2.079E3	.000
	WA	209.853	4	52.463	255.193	.000
CAges***	CS	98.210	1	98.210	376.306	.000
	WA	33.489	1	33.489	162.897	.000
NaOH * Cages	CS	38.714	4	9.678	37.084	.000
	WA	3.253	4	.813	3.956	.006
Error	CS	16.703	64	.261		
	WA	13.157	64	.206		
Total	CS	26130.698	96			
	WA	6632.744	96			
Corrected Total	CS	3249.853	95			
	WA	381.238	95			

a. R Squared = .995 (Adjusted R Squared = .992)
b. R Squared = .965 (Adjusted R Squared = .949)

*CS = Compressive Strength; WA = Water Absorption; CAges = Curing Ages; df = degrees of freedom, F = F-ratio, Sig. = exact significance level.

The Duncan's multiple range post-hoc test (Tables 4.8 and 4.9) – which is a multiple comparison procedure in statistics used to examine set of means – was also conducted for comparison of the sets of means amongst the dependent variables (Compressive strength and water absorption) as influenced by NaOH molarity for the alkali-activated MHA-CCW

binder based mortar The test reveals that the mean compressive strength and water absorption does not show any significant difference for the various groupings.

Table 4.8: Duncan Analysis for NaOH Molarity Influence on Compressive Strength

		Subset				
NaOH	N	1	2	3	4	5
5	18	10.8844				
0	24		12.5933			
10	18			15.0800		
20	18				18.1333	
15	18					21.4489
Sig.		1.000	1.000	1.000	1.000	1.000

Means for groups in homogeneous subsets are displayed. Based on observed means.
The error term is Mean Square (Error) = .261.

Table 4.9: Duncan Analysis for NaOH Molarity Influence on Water Absorption

		`			
NaOH	N	1	2	3	4
15	18	6.4400			
10	18		7.7000		
20	18		7.8028		
5	18			8.3861	
0	24				9.5321
Sig.		1.000	.488	1.000	1.000

Means for groups in homogeneous subsets are displayed. Based on observed means.
The error term is Mean Square (Error) = .206.

4.5 Summary of Findings

Based on the outcomes of the experiment performed on the characterization (physical and chemical properties) and the evaluation of the fresh, early age and hardened properties of paste and mortar samples for strength properties of alkali-activated MHA-CCW with PC. The highlights of the major findings are as follows:

i. The result of PSD of the sharp sand revealed that it was uniformly graded since it have C_u, C_c and FM values of 0.98, 2.5 and 2.3 respectively. The fineness modulus falls between the range of 2.2 – 2.6, an indication for fine sand with an average moisture content of 1.3%.

ii. The result of PSD and BET surface area of MHA and CCW showed that MHA meets the quality criteria of even dispersal giving an average particle size of 180.23 nm and BET surface area value of 360.5 m^2/g. The result revealed that CCW contained particles with an average particle size of 109.01 nm and BET surface area 414.24 m^2/g.

iii. The XRF analysis revealed that MHA is a Class F Pozzolan (72.4 % SiO_2 content), while the aggregation of the main oxides (SiO_2 + Al_2O + Fe_2O_3) gives 88.1 % which is above 70 % minimum limit stipulated in ASTM C618 (2015) standard. SO_3 content is below 4 % and Loss on Ignition (LOI) below the specified 10 % maximum. The CCW was observed to contain 66.1 % CaO, a similar value to the CaO content (65.0 %) of the PC but low in SiO_2 and Al_2O_3. The LOI of CCW is above the specified 10% maximum, implying that more heat might be required for effective performance.

iv. The PC, MHA, CCW, NaOH and sand have specific gravity of 2.20, 2.09, 1.65 and 2.62 respectively. An indication of values falling within the range of 2.4 – 2.9 as prescribed by BS EN 1097-6:2013.

v. The study revealed that initial and final setting times of MHA-CCW mix proportion decreased significantly due to the alkali-activation as the NaOH molarities increases.

vi. The optimum mix composition for acceptable setting of the alkali-activated MHA-CCW binder based-mortar is adjudged to be 45: 55 (MHA: CCW) activated with 15M NaOH. This mix revealed fresh properties similar to the PC based mortar.

vii. The water absorption results were observed to follow a similar trend as the inference drawn from the compressive strength of alkali-activated MHA-CCW binder based-mortar.

MHA-CCW activated with 15M NaOH were 10.72 N/mm^2 (67 %) and 15.96 N/mm^2 (79 %) respectively of the PC based samples at the 7th and 14th day of curing.

viii. The compressive strength at 28th days of curing showed strength gain (as revealed by the CS_{28} Factor, Table 4.6) of 3.81N/mm^2 (19 %) as against 6.03N/mm^2 (23 %) strength gain of the PC based mortar and at 56th days addition of 4.75N/mm^2 (24 %) strength gain over 28th days as compared to the 1.75N/mm^2 (7 %) increase of the PC.

ix. The water absorption by mortar cubes increases across the various combination proportions up to 15M but shows slight drop at 20M NaOH. The result shows that the rate of water absorbed by mortar samples at 15M (45: 55) is similar to that of PC based mortar.

x. The Duncan's multiple range post-hoc test conducted for comparison Compressive strength and water absorption as influenced by NaOH molarity for the alkali-activated MHA-CCW binder-based mortar reveals that the mean compressive strength and water absorption does not show any significant difference for the various groupings.

CHAPTER FIVE

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

Based on the results of this research work, the following conclusions were drawn:

The chemical analysis shows that MHA can be classified as Class F Pozzolan of high SiO_2 content (72.4 %) while CCW is a good CaO source of similar percentage concentration (66.1 %) as the PC. The physical properties (PSD, BET surface area, specific gravity, bulk density, soundness, moisture content of PC, MHA and CCW were in accordance with BS EN- 197: 2011. Therefore, the fresh properties result from the study reveals that the alkali-activated MHA-CCW possesses good binding properties.

The study reveals that mortar samples from alkali-activated MHA-CCW possess normal density and similar water absorption as PC based mortar in conformance with ASTM C642 (2006) specification. Mortar samples made with 45: 55 MHA-CCW activated with 15M NaOH at of 1:3 binder/sand and 0.5 W/B gave the best performance out of the various proportion combinations studied and resulted in setting times and compressive strength values similar to the PC based mortar samples of same binder/sand and W/B.

5.2 Recommendation

Alkali-activated MHA-CCW (45:55) at 15M NaOH at 1:3 binder/sand mortar and 0.5 W/B is hereby recommended for masonry work based on the 28- and 56-days compressive strength values noted to be similar to that obtained for the PC based mortar and classified as class M mortar by 28-day compressive strength as specified by BS EN 196 – 1 (2016).

5.3 Suggestion for Further Research

i. Future studies on products of hydration should be conducted using scanning electron microscopy and X-ray diffraction analysis.

ii. Investigating the comparative cost benefit of using these alternative binder as against the PC in mortar or concrete construction.

iii. Further studies on the influence of temperatures slightly above ambient temperature (i.e., 40 – 90^{o}C) on the effectiveness of NaOH activation in MHA-CCW binder.

iv. The Alkali-activated MHA-CCW should be further explored using different optimization approach to produce higher strength for structure

v. Utilisation of the new binder in actual concrete work should be explored in further studies.

5.4 Contribution to knowledge

The study established 15M NaOH alkali-activation of MHA-CCW (45: 55) at 1: 3 binder/sand of 0.5 W/B as the suitable mix proportion for good binding and strength development. Adopting the utilization of alkali-activated 15M NaOH (45:55 of MHA: CCW) proportion combination will reduce over dependency on PC and improve concrete technology knowledge. Activating the agro-industrial waste (MHA-CCW) with 15M NaOH improved its workability and compressive strength progressively as compare to PC based mortar.

REFERENCES

Abalaka, A. E., & Okoli, O. G. (2013). Comparative effects of air and water curing on concrete containing optimum rice husk ash replacement. *Journal of Emerging Trends in Engineering and Applied Sciences*, 4(1), 60-65.

Abdulfatai, A. M., Mustapha A. G., & Bashir, A. U. (2013). Performance of corn cob ash filler in hot mix asphalt using stone dust as control. *Proceedings of 3rd Biennial Engineering Conference*, School of Engineering and Engineering Technology, Federal University Technology Minna, Niger State, Nigeria, pp. 268-275.

Abdullahi, A., Abubakar, A., & Afolayan, A. (2013). Partial Replacement of Sand with Sawdust in Concrete Production. *Proceedings of 3rd Biennial Engineering Conference, School of Engineering and Engineering Technology*, Federal University Technology Minna, Niger State, Nigeria, pp. 333-338.

Abdul-Wahab, S. A., Al-Rawas, G.A., Ali, S. & Al-Dhamri, H. (2016). Impact of the addition of oil-based mud on Carbon dioxide emissions in a Cement plant. *Journal of cement production*, 112, 4214-4225.

American Concrete Institute *(ACI EI-99)* (2000). Cement and Concrete Terminology. *American Concrete Institute (ACI EI-99)*, Farmington Hills, Michigan, USA.

Aneje, J. A. & Damen, R. M. (2014). Rate of Strength Development of Concrete made using Selected Nigerian Cement Brands, *International Journal of Technology Enhancements and Emerging Engineering Research*, 2(12), 48-51.

Angel, P. & Ana, F. J. (2011). Alkaline activation, Procedure for transforming fly ash new materials. Part 1: *Application of World of Coal (WOCA) Conference, USA*. http://www.flyash.info/.

Anowai, S. I., & Jones, E. (2015). Millet husk ash as partial replacement of cement in sandcrate block. *International Research journal of Engineering & Technology*. Vol.04./ss. 07, pp. 677-680

ASTM C140 (2003). Standard Test Methods for Sampling and Testing Concrete Masonry Units and Related Units. *Annual Book of ASTM Standards, Vol.04.02* (pp. 4). Philadelphia, USA: American Society for Testing and Materials.

ASTM C266 (2008). Standard Test Method for Time of Setting of Hydraulic-Cement Paste by Gillmore Needles. *Annual Book of ASTM Standards, Vol.04.02* (pp. 8). Philadelphia, USA: American Society for Testing and Materials.

ASTM C128 (2015). Standard Terminology Relating to Concrete and Concrete Aggregates. *Annual Book of ASTM Standards, Vol.04.02* (pp. 9). Philadelphia, USA: American Society for Testing and Materials. 76.

ASTM C150 (2012). Standard Specification for Portland Cement, *Annual Book of ASTM Standards, Vol.04.02* (pp. 9). Philadelphia, USA: American Society for Testing and Materials.

ASTM C 566 (2013). Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle. *Annual Book of ASTM Standards, Vol.04.02* (pp. 8). Philadelphia, USA: American Society for Testing and Materials.

ASTM C270 (2004). Mortar and Masonry Grout Mortar Cement. Portland Cement Association. *Annual Book of ASTM Standards, Vol.04.02* (pp. 8). Philadelphia, USA: American Society for Testing and Materials.

ASTM C618 (2015). Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete, *Annual Book of ASTM Standards, Vol.04.02* (pp. 2). Philadelphia, USA: American Society for Testing and Materials.

ASTM C642 (2006). Standard Test Method for Density, Absorption, and Voids in Hardened Concrete, *Annual.*

Auta, S. M., Shiwua, A. J., Tsado, T. Y., (2015). Compressive strength of concrete with millet husk ash as partial replacement for cement. *Magazine of Civil Engineering*. Vol. 07.

Bakharev, T. (2006). Thermal behaviour of Geopolymers prepared using Class F fly ash and elevated temperature curing. Cement and Concrete Research. 36, 1136-1147.

Barbosa, V. F. F., Mackenzie, K. J. D., Thaumathugo, C. (2000). Synthetic and Characterization of materials based on inorganic polymers of alumina and silica: sodium polysialate polymer. *International Journal of inorganic materials,*2(4) 309-317.

Benabed, B., Kadri, E.-H., Azzouz, L., & Kenai, S. (2012). Properties of self-compacting mortar made with various types of sand. *Cement and Concrete Composites.* 34(10), 1167-1173.

Bhattacharya, A., Ray, I., & Davalos, J. F. (2008). Effects of Aggregate Grading and Admixture/Filler on Self-Consolidating Concrete. *The Open Construction and Building Technology Journal.* 2, 89-95.

British Standard Institution – BS EN 1097-6 (2013). *Tests for mechanical and physical properties of aggregates - Determination of Particle density and water absorption – Part 6, London, British Standard* Institution (BSI).

British Standard Institution – BS EN 196 – 1 (2016). *Methods of testing cement - Part 1: Determination of strength*. London, BSI.

British Standard Institution (2003). *Tests for mechanical and physical properties of aggregates - Determination of loose bulk density and voids.* (BS EN 1097:2003), London, British Standard Institution.

British Standard Institution (2009). *Admixtures for concrete, mortar and grout. Concrete admixtures - Definitions, requirements, conformity, marking and labelling*. (BS EN 934 – 2:2009), London, British Standard Institution.

British Standard Institution (2011). *Testing concrete. Method for determination of water absorption*. (BS 1881-122:2011), London, British Standard Institution (BSI).

Castro, J., Bentz, D., & Weiss, J. (2011). Effect of Sample Conditioning on the Water Absorption of Concrete. *Cement and Concrete Composites*, 33(1), 805-813.

Chaowat, N. (2001). Properties of Portland cement mixed with Rice Husk Ash and Quicklime. Unpublished M.Sc. Thesis, Department of Civil Engineering Education, King Mongkuts Institute of Technology, North Bangkok.

Chukwudebelu, J.A., Ige, C.C., Taiwo, O.E, & Tojola O.B. (2013); "Recovery of pure slaked carbide sludge; A Case Study of Lagos State, Nigeria. *Environmental Science and Technology,* 7(6), Pp 490-495.

Damtoft, J. S., Lukasik, J., Herfort, D., Sorrentino, D., & Gartner, E. M., (2008). Sustainable development and climate change initiatives. *Cement and Concrete Research*. 38(2), 115–127.

Didel, M. J., Matawa, D.S. and Ojo, E. B. (2014). Cost comparative analysis of compressed stabilized Blocks and sandcrete Blocks in affordable Housing provision in Nigeria. *Quarterly Newsletter of the Nigerian Building and Road Research Institute.* Vol. 2, No. 5, June, 2014.

Duggal, S. K. (2008). Building Materials. *New Age International Publishers, New Delhi, India*. 142-300.

Dunn, D. S., Pulvirenti, A. L., & Hiser, M. A., (2011). Containment Linear corrosion Operating experience summary. *Washington DC- Nuclear Regulatory Commission.*

Dwivedi, V. N., Singh, N. P., Dass, S. S., & Singh, N. B. (2006). A New Pozzolanic Material for Cement Industry: Bamboo Leaf Ash. *International Journal of Physical Sciences*, 1 (3), 106-111.

Egwuda, C.I., Hassan, I. O., Olawuyi, B. J. & Enejiyon, M. O. (2018). Early-Age Properties of Sorghum Husk Ash and Calcium Carbide Waste Binder in Mortar. *Paper presented at School of Environmental Technology International Conference (SETIC) 2018*, Federal University of Technology, Minna, Nigeria, 10^{th} – 12^{th} April.

Elinwa, A. U. & Mahmood, Y. A. (2002). Ash from Timber Waste as Cement Replacement Material. *Cement and Concrete Composites*, 24(2), 219-222. 78

Emmitt, S., & Gorse, C. A. (2014). Barry's Advanced Construction of Buildings (3rd ed.), Ckhichester, UK: Wiley-Blackwell.

Enejiyon, M. O., Olawuyi, B. J**.,** Hassan, I. O., and Egwuda, C.I. (2018). Effect of Superplasticizer on Setting Time and Strength of Mortar made using Rice Husk Ash (RHA) and Calcium Carbide Waste (CCW) as Binder. *Paper presented at School of Environmental Technology International Conference (SETIC) 2018*, Federal University of Technology, Minna, Nigeria, 10^{th} – 12^{th} April..

Fernandez-Jimenez, A., Paloma, A. & Criado, M. (2004). Microstructure development of alkali-activated fly ash cement- A descriptive model. Cement and Concrete Research, 35(6), 1204-1209

FOA (2016). Food and Agriculture Organization; *World Millet Production: Economic and Social Department, the Statistics Division, United Nation, Corporate Statistical Database, Retrieved* 21 May 2017.

Habeeb, G.A. & Mahmud, H. B. (2010). Study on properties of Rice Husk Ash and its constituents. Material Research, 185 – 189.

Hardjito, D. & Wallah, S. E., Summajouw, D. M. J. & Rangan, B. V. (2004). On the development of fly ash-based geopolymer concrete. *ACI Materials Journals*, 101 (6) 467-472.

Hassan, I. O. (2006). Strength properties of concrete obtained using volcanic ash pozzolan as partial replacement of cement, Unpublished M.Sc. Thesis, Department of Building, University of Jos.

Hewlett, P. C. (2006). *Lea's Chemistry of Cement and Concrete.* Elsevier Publishers, Oxford, UK, 471-475.

Horpibulsuk, S., Phetchuay, C. & Chinkulkijniwat, A. (2011). Soil stabilization by Calcium Carbide Residue and Fly Ash. *Journal of Materials in Civil Engineering,* 24(2), 184-193.

Hossain, K. M. A. (2003). Blended cement using volcanic ash and pumice. *Cement and Concrete Research*. 33, 1601-1605.

Hossain, K. M. A. (2005). Chloride induced corrosion of reinforcement in volcanic ash and pumice based blended cement. *Cement and Concrete Composites*. 27, 381-390.

Hu, J., & Wang, K. (2005). Effects of aggregate on flow properties of mortar. *Paper presented at the Mid-Continent Transportation Research Symposium.*

Jimoh, R.A., Banuso, R.O. & Oyeleke, M. (2013). Exploratory assessment of strength characteristics of millet husk ash (MHA) blended cement laterized concrete, *Advances in Applied Science Research*, 2013, 4(1):452-457.

Joseph Asphidin (1824). Concrete Technology- Neville and Brook Second Edition- WWW.personed .Co.UK.

Joshua, O., Ogunde, A.O., Omuh, I.O., Ayegba, C. & Olusola, K.O. (2015). *Exploring the Pozzolanic Potential of Blend of Palm Kernel Nut Ash (PKNA) with Cement towards a Sustainable Construction*. *International Conference on African Development Issues (CU-ICADI), Material Technology Track,* Pp 135-140.

Ka'ase, E. T., Adeojo, J. E. & Adeojo, A. O., (2018). Effect of different Molarities on Ambient Temperature cured Metakaolin-based Geopolymer Concrete. Department of Building, Ahmed Bello University Zaria. *Nigerian Building and Road Research Institute International Conference.*

Kazjonovs, J., Bajare, D., & Korjakins, A. (2010*).* Designing of High Density Concrete by Using Steel Treatment Waste. *Paper Presented on Modern Building Materials, Structure and Techniques International Conference,* May 19-21, 2010. Vilnius, Lithuania, 138-142.

Kisharet, D. A., Ahmed, M. R. & Essam, A. T. (2013) Effect of activators on the early strength Development and the Hydration Characterization of Ground Clay Bricks Cement paste. *Beni-Suef University Journal.www.Sciencedirect.*

Klemm, W. A. (2005). Cement soundness and the autoclave expansion test: An update of the literature, R&D serial No. 2651, Portland Cement Association, Skokie, Illinois, USA, pp.13-20.

Klemm, W. A. (2005). Cement soundness and the autoclave expansion test: An update of the literature, R&D serial No. 2651, Portland Cement Association, Skokie, Illinois, USA, pp.13-20

Kwabena A. O. (2012). Improvement of Setting Time and Early Strength Development of Pozzolans Cement through Chemical Activation, Thesis, Kwame Nkrumah University of Science & Technology Collage of Engineering. *Published Online.*

Manasseh, J., & Joseph, E. E. (2016). Calcium Carbide Residue-Hydrated Lime Blend as Stabilizing Agent. *International Journal of Science and Technology (IJST)*, 5 (12), 606 – 614.

Marakaratat, N., Jaturapitakkul, C., & Laosamathikul, T. (2010). Effects of Calcium Carbide residue-fly ash binder on mechanical properties of Concrete, *Journal of Materials in Civil Engineering*, 22(11), 1164-1170.

Martinez, S. & Palomo, A., (2001) Microstructure studies on Portland cement pastes obtained in highly alkaline environments. Cem. Con. Res., 31(11): 1581-1585.

Massaza, F. (2005). Chemistry of pozzolanic addictions and mixed cements, chapter 10, lea's chemistry of cement and concrete (4th Ed.). Elsevier, oxford, u.k.

Mehta, P. K., & Monteiro, J. M. (2014), *Concrete microstructure properties and materials* (4th ed.), U.S.A.: McGraw-Hill Education.

Morenoa, S. Dieza, E. V. & Barra, M. 2008. Environmental, physical and structural characterization of geopolymer matrixes synthesized from coal co combustion fly ashes. *Journal of Hazardous Materials*, 154 1-3, 175-185.

Mudasiru, I. (2015). Durability Characteristics of Portland Cement/Volcanic Ash Concrete Exposed to Chemically Aggressive Environments. *Published M.Sc. Thesis*, Department of Building, Ahmadu Bello University, Zaria

Nambiar, E. K. K., & Ramamurthy, K. (2007). Sorption Characteristics of Foam Concrete. *Cement and Concrete Research Journal*, 1-7. Retrieved from http://www.sciencedirect.com.

Nattapong. M., Chai, J., Charin, N., & Vanchai, S. (2011). Effects of binder and CaCl2 contents on the strength of calcium carbide residue-fly ash concrete. *Cement and Concrete Composites*, 33, 436–443.

Ndububa, E. E., & Nurudeen Y. (2015). Effect of Guinea Corn Husk Ash as Partial Replacement for Cement in Concrete, 12(2), 40-41.

Neville, A. M. (2012). *Properties of concrete* (5th Ed.). England: Pearson Educational Limited.

Neville, A. M., & Brooks, J. J. (2010). *Concrete Technology*, (2nd Ed.). London, Longman Publishers.

Nigeria Industrial Standard: NIS (2004). Standard for Sandcrete Blocks, *Standard Organization of Nigeria* (Approved).

Nwaubani, S. O., & Poutos, K. I. (2013). Influence of Waste Glass Powder Fineness on the Properties of Cement Mortar. *International Journal of Application or Innovation in Engineering and Management*, 2(2), 110-116. Retrieved from http://www.ijaiem.org

Ogork, E.N., Uche, O.A. and Elinwa, A.U. (2014). Performance of Groundnut Husk Ash (GHA), Rice Husk Ash-(RHA) Modified Concrete in Acidic Environment. *Journal of Engineering Research and Applications*, 4(11), (Version 1): 71-77.

Okamura, H., and Ozawa, K. (1995). Mix design for self-compacting concrete. *Concrete Library of JSCE*. 25, 107-120.Pitroda, J., & Shah, D. S. (2014). An Experimental Study on Durability and Water Absorption Properties of Pervious Concrete. *International Journal of Research in Engineering and Technology*, 3(3), 439-444. Retrieved from http://www.ijret.org

Okpala (1987)**.** Rice Husk Ash (RHA) as Partial Replacement of Cement, *Concrete Procedure. Nigerian Society of Engineers (NSE) AGM.*

Okwute, E. (2018). Tenile Strength Properties of Millet Husk Ash Calcium Carbide Waste based Mortar containing Egg Shell Powder and Dilute Nitric Acid as Admixture. *Unpublished B.Tech.. Dissertation*. Department of Building, Federal University of Technology, Minna.

Olawuyi, B. J. & Olusola, K. J. (2017). Strength Characteristic of Volcanic Ash Blended Cement Laterized Concrete. *LAP Lambert Academic Publishing*. pp.160.

Olawuyi, B. J., Joshua, O., Hassan, I. O., Enejiyon, M. O., & Egwuda, C. I. (2017). Exploratory Study on Agro-Waste Ashes Combination with Industrial Waste as

Alternative Binders in Concrete. *Nigerian Building and Road Research Institute International Conference.*

Olonade, K. A. and Bello, T. (2017). Performance Evaluation of Alkali-Activated Coconut Shell Ash as Binder in Mortar. *Nigerian Building and Road Research Institute (NBRRI) International Conference.*

Olutoge, F. A., Bhashya V., Bharatkumar B. H., & Sundar K. S. (2010). *Comparative Studies on Fly Ash and GGBS High Performance Concrete*. Proceeding of National Conference on Recent Trend and Advance in Civil Engineering-TRACE.

Onuche G., Olawuyi B.J. & Saka, R.O. (2019). Material Characterization and Fresh Properties of Alkali-Activated MHA/CCW Based Mortar. *18th Nigeria International Materials Congress,* Pp 72.

Parhizkar, T., Najimi, M., Pourkhorshidi, A. R., Jafarpour, F., Hillemeir, B., and Herr, R. (2010). Proposing a New Approach for Qualification of Natural Pozzolans. *Scientica Iranica Journal,* 17(6), 450-456. Retrieved from http://www.SID.ir.

Qureshi, N. M & Ghoshi, S. (2013). Alkali activated blast furnace slag as a green construction material. Pp. 24 (8).

Raheem, A. Y. (2010); An Investigation of Corn Cob Ash Blended Cement for Concrete Production, *Unpublished Ph.D. Thesis*, Department of Building, Obafemi Awolowo University, Ile-Ife.

Rahhal, V., & Talero, R. (2005). Early hydration of Portland cement with crystalline mineral additions. *Cement and Concrete Research*, 35, 1285–1291.

Rajput, R. K. (2006). *Engineering Materials*, S. Chand & Company Limited, New Delhi, India.

Ramezanianpour, A. A. (2014). Cement Replacement Materials. *Properties, Durability, Sustainability.* Doi:101007/978-3- 642-36721-2.

Ramujee K. and potharatu V. K., (2014), Abrasion Resistance of Geopolmer and its Composite. 3rd International Conference on Materials Processing and Characterization (ICMPC).

Rasa, E., Ketabchi, H., & Afshar, M. H. (2009). Predicting Density and Compressive Strength of Concrete Cement Paste Containing Silica Fume Using Artificial Neutral Networks. *Scientia Iranica,* 16(1), 33-42. Retrieved from http://www.SID.ir.

Razi, P. Z., AbdulRazak, H., & Khalid, N.H.A.,(2016). Sustainability, Eco-Point and Engineering Performance of Different Workability OPC Fly-Ash Mortar Mixes. Materials, 12 (5), 341.

Rubenstein, M. (2012). Emission from the Cement Industry, "State of the Planet" a publication of the Earth Institute, University of Columbia accessed online on 6th February, 2017.http://blogs.ei.columbia.edu/2012/05/09/emissions-from-the-cement-industry.

Saraswathy, V., & Song, H. (2007). Corrosion Performance of Rice Husk Ash Blended Concrete. Construction and Building Materials, 21(8): 1779-1784.

Savitha, A. (2012). Importance of quality assurance of materials for construction work. Building Materials Research and Testing Division, 1-5.

Shetty, M. S. (2009). *Concrete Technology; Theory and Practice*, S Chad and Company, New Delhi, India, 174-418.

Su, J. K., Cho, S. W., Yang, C. C., & Huang, R. (2002). Effect of sand ratio on the elastic modulus of self-compacting concrete. *Journal of Marine Science.* 10(1), 8-13.

Tasi, C. T., Li, S., & Hwang, C. L. (2006). The effect of aggregate gradation on engineering properties of high performance concrete. *Journal of ASTM International.* 3(3), 1-12.

Tsado, T. Y., Yelwa, M., Yamma, S. and Yewa, F. (2014). Comparative Analysis of Properties of Some Artificial pozzolana in Concrete Production. *International Journal of Engineering and Technology.* Vol. 4 No. 5.Uche, O.A.U., Adamu, M. and Bahuddeen, M.A. (2012). Influence of Millet Husk Ash (MHA) on the Properties of Plain Concrete. *Journal of Epistemics in Science, Engineering and Technology,* 2(2): 68-73.

Utsev, J. T., & Taku, J. K. (2012). Coconut shell Ash as partial replacement of ordinary Portland cement in concrete production. *International Journal of Scientific and Technology Research, 1(8).*

Vandevelde, P. E. (2008). Structural Mechanics (Version Fall, 2008), GEM Engineering Inc. (pdf. accessed online on 10/9/2010 by 4.49pm).

Vinchan, S., & Rachan, R. (2012). Chemical Stabilization of Soft Bangankok Clay Using the Blend of Calcium Residue and Biomass Ash. Soils and Foundations, 53 (2) 272-281.

Vladimir, G. H., Graca, V., & Paulo, B. L. (2011). Influence of aggregates grading and water/cement ratio in workability and hardened properties of mortars.

Wang, Y. L., Dong, S.J., Liu, L. L., & Ciu, S. P. (2013). Using calcium carbide slag as one of calcium containing raw materials to produce cement clinker. *Energy Environmental Material*. 171, 743-744.

Yunusa, S. A. (2015). Investigation into the use of calcium carbide waste as partial Replacement of Cement in Concrete. *International Journal of Engineering and Management Research, 5(2), 675 – 680.*

Zhang, J., Liu, G., Chen, B., Song, D., Qi, J., & Liu, X. (2014). Analysis of CO2 Emission for the cement manufacturing with alternative raw materials- A LCA-based framework. Energy Procardia, 61, 2541-2545.

APPENDIX A (DETAIL TABLES)

Table A1: Sieve Analysis Fine Aggregate

Sieve size mm	Weight of sieve (g)	Sample sieve (g)	Wt. of sieve + retained (g)	%Retained	Cumulative % retained	% Passing
4.75	377.0	384.0	7.0	1.4	1.4	98.6
2.36	396.0	421.1	25.0	5.0	6.4	93.6
1.18	366.0	400.0	34.0	6.8	13.2	86.4
600	380.0	477.0	97.0	19.6	32.6	67.4
300	367.0	565.0	198.0	39.6	72.2	27.4
150	334.0	458.0	125.0	25.6	97.2	2.8
75	376.0	376.0	9.0	1.8	99.0	1.0
Pass	310.0	315.0	5.0	1.0	100	0.0
Total			500	100		

D60 = 0.55mm, D30 = 0.34mm and D10 = 0.22mm

Coefficient of uniformity (Cu) = $\frac{D60}{D10}$ = 0.96

Coefficient of curvature (Cc) = $\frac{D30}{D10\ X\ D6}$ = 2.50

Fineness Modulus (FM) = 2.30

Table A2: Average density of mortar cubes (g/m^3) for 3 days curing

Samples		MHA/CCW		
	PC	40/60	45/55	50/50
PC	2083	0	0	0
0M	0	1952	1928	1939
5M	0	1959	2002	1939
10M	0	1963	2008	1969
15M	0	2005	2010	2008
20M	0	2018	2013	2010

Table A3: Average density of mortar cubes (g/m^3) for 7 days curing

Samples		MHA/CCW		
	PC	40/60	45/55	50/50
PC	2093	0	0	0
0M	0	1989	2000	1959
5M	0	2008	2008	2004
10M	0	2021	2036	2024
15M	0	2030	2041	2039
20M	0	2029	2040	2045

Table A4: Average density of mortar cubes (g/m^3) for 14 days curing

Samples		MHA/CCW		
	PC	40/60	45/55	50/50
PC	2197	0	0	0
0M	0	2001	2011	2015
5M	0	2014	2040	2031
10M	0	2018	2043	2010
15M	0	2139	2151	2149
20M	0	2133	2137	2123

Table A5: Average density of mortar cubes (g/m^3) for 28 days curing

Samples		MHA/CCW		
	PC	40/60	45/55	50/50
PC	2171	0	0	0
0M	0	2099	2104	2128
5M	0	2138	2148	2146
10M	0	2146	2160	2128
15M	0	2162	2162	2146
20M	0	2157	2152	2154

Table A6: Average density of mortar cubes (g/m^3) for 56 days curing

Samples		MHA/CCW		
	PC	40/60	45/55	50/50
PC	2182	0	0	0
0M	0	2106	2128	2114
5M	0	2138	2146	2144
10M	0	2146	2154	2128
15M	0	2162	2168	2154
20M	0	2156	2154	2157

Table A7: 3days Compressive Strengths value

	A		B		C	
			40/60 MHA / CCW			
SAMPLES	KN	N/mm^2	KN	N/mm^2	KN	N/mm^2
PC	25.10	10.94	25.4	10.16	26.30	10.52
0M	1.10	1.64	4.40	1.76	5.60	2.24
5M	7.20	2.88	6.10	2.44	6.80	2.72
10M	11.80	4.72	10.10	4.04	12.50	5.00
15M	15.20	6.04	16.70	6.68	17.00	6.80
20M	9.90	3.96	10.00	4.00	10.30	4.12
			45/55% of MHA and CCW			
PC	25.10	10.94	25.4	10.16	26.30	10.52
0M	4.50	1.80	5.40	2.16	4.60	1.84
5M	7.30	2.92	6.50	2.66	7.30	1.84
10M	12.00	4.80	11.30	4.54	12.50	5.00
15M	18.40	7.36	16.60	6.64	17.50	7.00
20M	12.50	5.00	10.80	4.32	12.10	4.84
			50/50% of MHA and CCW			
PC	25.10	10.94	25.4	10.16	26.30	10.52
0M	4.80	1.92	5.40	2.16	4.70	1.88
5M	6.50	2.60	6.90	2.76	7.10	2.84
10M	10.90	4.36	11.50	4.60	12.50	5.00
15M	16.90	6.76	16.6	6.64	17.50	7.00
20M	11.90	4.76	9.60	3.84	10.00	4.00

Table A8: 7days Compressive Strengths value

	A		B		C	
	40/60% of MHA and CCW					
SAMPLES	KN	N/mm^2	KN	N/mm^2	KN	N/mm^2
PC	39.4	15.76	38.30	15.32	45.50	17.00
0M	5.20	2.08	4.50	1.80	5.90	2.36
5M	8.50	3.40	10.10	4.04	10.40	4.16
10M	18.90	7.56	17.10	6.86	16.40	6.56
15M	21.20	8.48	23.80	9.52	22.50	9.00
20M	14.50	5.80	14.00	5.60	15.40	6.16
	45/55% of MHA and CCW					
PC	39.76	15.76	38.30	15.32	45.50	17.00
0M	4.70	1.88	5.50	2.20	5.60	2.20
5M	8.90	3.56	9.90	3.96	10.50	4.20
10M	19.90	7.96	18.80	7.52	19.70	7.88
15M	25.90	10.36	27.00	10.80	26.60	10.64
20M	15.60	6.24	16.00	6.40	15.00	6.00
	50/50% of MHA and CCW					
PC	39.76	15.76	38.30	15.50	45.50	17.00
0M	5.60	2.24	5.70	2.28	5.60	2.24
5M	8.8	3.52	9.30	3.72	10.40	4.16
10M	19.80	7.92	18.90	7.56	17.80	7.12
15M	24.80	9.92	10.24	25.60	24.20	9.68
20M	14.00	5.60	15.30	6.12	15.40	6.16

Table A9: 14 days Compressive Strengths value

	A		B		C	
			40/60 MHA / CCW			
SAMPLES	KN	N/mm^2	KN	N/mm^2	KN	N/mm^2
PC	50.40	20.16	52.10	20.84	49.50	19.80
0M	8.40	3.36	7.80	3.12	8.80	3.52
5M	14.30	5.27	12.00	4.80	13.80	5.52
10M	24.00	9.60	23.20	9.28	25.60	10.24
15M	38.50	15.40	37.90	15.16	38.60	15.44
20M	24.10	9.64	23.80	9.52	28.40	11.36
			45/55 MHA / CCW			
PC	50.16	20.16	52.10	20.84	49.50	19.80
0M	8.90	3.56	10.50	4.20	9.00	3.60
5M	13.20	5.28	14.00	5.60	13.50	5.40
10M	26.30	10.52	24.70	9.88	25.40	10.16
15M	39.90	15.96	40.00	16.00	39.80	15.92
20M	26.90	10.76	25.80	10.32	26.50	10.60
			50/50 MHA / CCW			
PC	50.16	20.16	52.10	20.84	49.50	19.80
0M	9.30	3.72	9.50	3.80	10.00	4.00
5M	14.00	5.60	12.20	4.88	14.4	5.57
10M	24.50	9.80	25.80	10.32	24.30	9.72
15M	38.60	15.44	38.90	15.56	38.60	15.44
20M	26.40	10.40	24.90	9.96	25.30	10.12

Table A10:28 days Compressive Strengths value

	A		B		C	
			40/60 MHA / CCW			
SAMPLES	KN	N/mm^2	KN	N/mm^2	KN	N/mm^2
PC	65.80	26.32	64.40	25.76	67.00	28.80
0M	19.00	7.60	18.30	7.32	18.50	7.40
5M	25.00	10.00	23.40	9.36	24.90	9.96
10M	33.60	13.44	35.40	14.16	34.80	13.92
15M	49.30	19.72	46.00	18.40	48.5	19.40
20M	39.90	15.96	40.50	16.20	39.80	15.92
			45/55 MHA / CCW			
PC	65.80	26.32	64.40	25.76	67.00	28.80
0M	17.90	7.16	18.50	7.40	18.70	7.48
5M	26.90	10.74	25.50	10.20	24.80	9.92
10M	35.90	14.36	38.30	15.32	36.00	14.40
15M	50.50	20.20	46.60	18.64	49.20	19.68
20M	40.60	16.24	39.30	15.72	43.50	17.40
			50/50 MHA / CCW			
PC	65.80	26.32	64.40	25.76	67.00	28.80
0M	18.40	7.36	19.60	7.84	19.70	7.88
5M	25.90	10.36	24.80	9.92	24.30	9.72
10M	34.80	13.90	37.90	15,16	35.00	14.00
15M	47.00	19.60	48.50	19.40	49.40	19.76
20M	40.80	16.32	41.90	16.76	39.20	15.68

Table A11: 56 days Compressive Strengths value

	A		B		C	
			40/60 MHA / CCW			
SAMPLES	KN	N/mm^2	KN	N/mm^2	KN	N/mm^2
PC	70.60	28.24	69.30	27.72	70.50	28.20
0M	19.80	7.92	19.50	7.80	20.10	8.04
5M	29.50	11.80	27.90	11.16	28.80	11.52
10M	39.10	15.64	36.50	14.60	38.90	15.56
15M	53.50	21.40	56.80	22.72	59.50	23.80
20M	48.30	19.32	48.50	19.40	50.00	20.00
			45/55 MHA / CCW			
PC	70.60	28.24	69.30	27.72	70.50	28.20
0M	20.50	19.90	19.90	7.96	20.90	8.36
5M	30.70	12.28	30.80	12.32	29.90	11.96
10M	39.90	15.96	42.00	16.80	41.30	16.52
15M	60.20	24.08	60.50	24.20	63.30	25.32
20M	53.50	21.40	48.30	19.32	52.90	21.16
			50/50 MHA / CCW			
PC	70.60	28.24	69.30	27.72	70.50	28.20
0M	20.90	8.36	20.50	8.20	20.10	8.04
5M	28.90	11.56	28.50	11.40	29.30	11.72
10M	40.50	16.20	38.80	15.52	39.90	15.96
15M	57.80	23.12	59.00	23.60	57.50	23.00
20M	49.20	19.68	49.30	19.72	50.50	20.20

Table A17: PSD of MHA

Size Distribution Report by Intensity

v2.2

Malvern

Sample Details

Sample Name:	Sample M H A		
SOP Name:	ZETASIZER.sop		
General Notes:	Average result created from record number(s): 1 2 3		
File Name:	ZETASIZER.dts	**Dispersant Name:**	Water
Record Number:	172	**Dispersant RI:**	1.330
Material RI:	1.59	**Viscosity (cP):**	0.8872
Material Absorbtion:	0.010	**Measurement Date and Time:**	02 November 2019 10:08:55

System

Temperature (°C):	25.0	**Duration Used (s):**	60
Count Rate (kcps):	468.5	**Measurement Position (mm):**	4.65
Cell Description:	Disposable sizing cuvette	**Attenuator:**	7

Results

			Size (d.n...	% Intensity:	St Dev (d.n...
Z-Average (d.nm):	90.86	**Peak 1:**	186.8	38.4	54.74
PdI:	0.302	**Peak 2:**	14.68	37.6	4.951
Intercept:	0.934	**Peak 3:**	38.95	22.9	12.86
Result quality	Good				

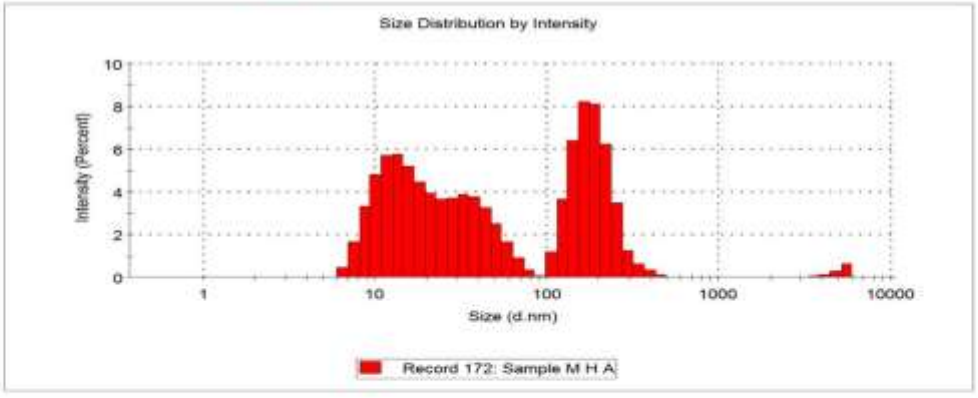

Table A18: PSD of CCW

Size Distribution Report by Volume

v2.2

Malvern

Sample Details

Sample Name:	CCW (Calcium C. W)
SOP Name:	D S L.sop
General Notes:	Average result created from record number(s): 155 158 172

File Name:	ZETASIZER.dts	**Dispersant Name:**	Water
Record Number:	173	**Dispersant RI:**	1.330
Material RI:	1.59	**Viscosity (cP):**	0.8872
Material Absorbtion:	0.010	**Measurement Date and Time:**	02 November 2019 10:09...

System

Temperature (°C):	25.0	**Duration Used (s):**	60
Count Rate (kcps):	353.8	**Measurement Position (mm):**	1.25
Cell Description:	Disposable sizing cuvette	**Attenuator:**	11

Results

			Size (d.n...	**% Volume:**	**St Dev (d.n...**
Z-Average (d.nm):	993.8	**Peak 1:**	10.22	13.3	2.729
PdI:	0.512	**Peak 2:**	36.53	31.9	11.93
Intercept:	0.426	**Peak 3:**	199.5	28.5	80.30
Result quality	Refer to quality report				

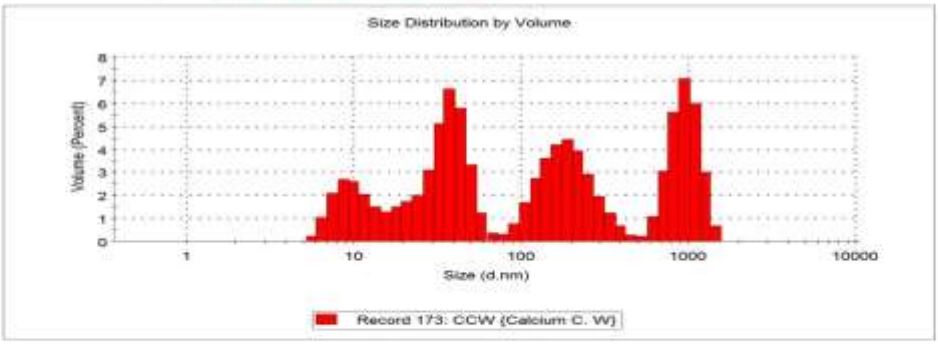

Table A19: Bulk Density of Fine Aggregate

TEST	COMPACTED	UNCOMPACTED
Wt. of mould based plate + base plate (g) W1	8676	8676
Wt. of empty plate + baesd plate + Fine aggregate (g) W2	12901	12805
Volume of the mould (m3)	0.00265	12805
Bulk Density = $\frac{Net\ Wt,of\ Aggregate}{Volume}$ $Kg/m3$	1594	1558

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Table A11: 28 DAYS WATER ABSORPTION

	PC			40: 60 OF MHA-CCW														
				0M			5M			10M			15M			20M			
	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
Before oven dry	281	275	280	260	263	264	258	256	259	249	259	249	261	259	260	256	259	253
After Oven dry	256	250	257	222	224	226	226	224	230	220	228	223	235	231	234	228	229	277
After Absorption	271	263	271	254	255	258	254	251	256	243	252	246	255	252	254	253	255	248
Water Absorption (%)	5.53	4.94	5.16	12.59	12.15	12.4	11.02	10.75	10.15	9.46	9.92	9.34	7.84	8.33	7.87	9.88	10.19	8.46
Average water Absorption (%)		5.21			12.38			10.64			9.57			8.01			9.51	

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Table A12: 28 DAYS WATER ABSORPTION

				45/55 OF MHA/CCW													
	PC			0M			5M			10M			15M			20M		
	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
Before oven dry	281	275	280	260	254	255	261	265	260	258	259	250	261	257	258	255	257	260
After Oven dry	256	250	257	224	220	222	235	237	235	233	233	223	235	233	234	233	230	237
After Absorption	271	263	254	254	247	249	257	258	256	252	253	243	251	249	249	250	251	254
Water Absorption (%)	5.53	4.94	5.16	11.81	10.93	10.84	8.56	8.13	8.20	7.53	7.90	8.23	6.37	6.42	6.02	6.80	8.36	6.69
Average water Absorption (%)		5.21			11.19			8.29			7.88			6.27			7.28	

Table A13: 28 DAYS WATER ABSORPTION

				50/50 OF MHA/CCW													
	PC			0M			5M			10M			15M			20M		
	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
Before oven dry	281	275	280	261	259	260	258	259	260	255	259	258	263	263	265	257	258	257
After Oven dry	256	250	257	226	226	226	228	229	228	224	226	224	229	229	229	226	227	224
After Absorption	271	263	271	257	255	254	252	253	251	246	246	245	247	245	246	246	249	246
Water Absorption (%)	5.53	4.94	5.16	12.06	11.37	11.02	9.52	9.48	9.16	8.94	8.16	8.16	7.28	7.66	6.53	8.13	8.83	8.94
Average water Absorption (%)		5.21			11.48			9.38			8.42			7.15			8.63	

Table A14: 56 DAYS WATER ABSORPTION

				40/60 OF MHA/CCW														
	PC			0M			5M			10M			15M			20M		
	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
Before oven dry	278	273	275	257	258	260	255	253	256	250	255	250	259	253	258	255	258	251
After Oven dry	257	253	256	277	228	229	228	226	230	224	227	224	228	226	230	230	231	224
After Absorption	270	265	269	254	255	256	246	244	248	241	244	241	243	241	245	246	247	240
Water Absorption (%)	4.81	4.52	4.84	10.54	10.58	10.54	7.31	7.37	7.25	7.05	6.96	7.05	6.17	6.22	6.12	6.50	4.47	6.66
Average water Absorption (%)		4.72			10.55			7.31			7.02			6.17			6.54	

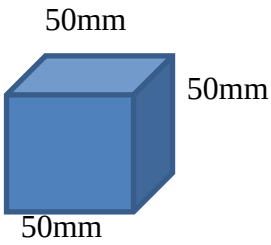
Table A15: 56 DAYS WATER ABSORPTION

				45/55 OF MHA/CCW														
	PC			0M			5M			10M			15M			20M		
	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
Before oven dry	278	273	275	256	252	253	253	254	258	254	258	250	259	255	258	253	254	259
After Oven dry	257	253	256	227	222	224	226	228	231	228	232	225	231	228	229	227	227	232
After Absorption	270	265	269	253	249	250	244	245	250	244	248	241	245	242	242	243	243	247
Water Absorption (%)	4.81	4.52	4.83	10.27	10.84	10.40	7.37	6.93	7.60	6.55	6.45	6.63	5.71	5.78	5.37	6.58	6.58	6.07
Average water Absorption (%)		4.72			10.50			7.30			6.54			5.62			6.41	

Table A16: 56 DAYS WATER ABSORPTION

				50/50 OF MHA/CCW													
	PC			0M			5M			10M			15M			20M		
	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
Before oven dry	278	273	275	257	255	258	254	256	259	253	256	258	260	259	261	255	257	253
After Oven dry	257	253	256	225	225	227	227	230	232	228	230	231	232	231	233	228	229	227
After Absorption	270	265	254	254	253	253	245	248	251	246	246	247	248	246	248	250	249	248
Water Absorption (%)	4.81	4.52	4.84	11.02	11.06	10.27	7.34	7.25	7.56	7.31	6.50	6.47	6.45	6.09	6.04	8.80	8.03	8.46
Average water Absorption (%)		4.72			11.02			7.30			6.76			6.19			8.43	

Appendix A20: Computation for Mix



Volume of cube = 50*50*50mm = 0.000125m^3

Mass = Volume *Density, where density of mortar = 2165kg/m^3

= 0.000125*2165

= 0.27kg

= 270g

Ratio of cement to aggregate = 1:3, W/c = 0.5

For cement: $\frac{1}{4} * 270 = 67.5 = 68g$

For water: C * 0.5 = 68 * 0.5 = 33.75 = 34g

For fine aggregate: 270 – (68+34)g = 168g

Allow 10% wastage

Therefore, Cement = 75g, Sand = 185g, Water = 37g.

Ratio of MHA to CCW: 40 to 60% of MHA and CCW,

40% = $\frac{40}{100} * 75 = 30g$ & 60% = 45g.

45% = $\frac{55}{100} * 75 = 34g$ & 55% = 41g,

50% = $\frac{50}{100} * 75 = 38g$

Appendix A21: the molar ratio of millet husk ash and calcium carbide

MHA= Millet husk ash

CCA=Calcium carbide

$SiO_2 + CaO + H_2o \longrightarrow Cao.SiO_2.H_{2O}$

The molar mass of Sio_2 = 20+(16*2)=60g/mol.

The molar mass of CaO = 40 + 16 = 56g/mol.

The of molar mass H_2O = (1*2)+16 = 18g/mol.

Therefore, the molar mass of $Cao.SiO_2.H_{2O}$ = 134g and $CaO + SiO_2$ = 116g

But, concentration of MHA in (g):

SiO_2 = 72.4% = 72.4/100 = 0.73 = 1/0.72 = 1.38g

Concentration of CCW in (g):

CaO = 66.1% = 66.1/100= 0.66= 1/0.66 = 1.51g

Therefore, number of mole= reactive mass/molar mass

= 1.38/60 and 1.51/56

= 0.023*100and 0.027*100

= 2.3 and 2.7

Therefore, the ratio of MHA to CCW = 2.3: 2.7

= 2.3+2.7 = 5.0

2.3/5.0: 2.7/5.0

45%: 55%

Proposed mix proportion for consideration based:

45 % of the MHA to 55% of the CCW

Sodium Hydroxide- NaoH

$SiO_2 + SiO_2 + CaO\ H_2O + 2NaoH \longrightarrow Na_2O.CaO.SiO_2\ .2H_2o$

Molar weight of NaOH = 40g

If 1 mole of NaoH = 40g and molar concentration of NaOH = 40g/mol.

Therefore,

5moles = 200g, 10moles = 400g, 15moles = 600g, 20moles = 800g

APPENDIX B (PLATES)

Plate I: Incinerator

Plate II: Burning of MHA

Plate III: Sieved Millet Husk Ash

Plate IV: Processed CCW

Plate VI: DUTM & Uncrushed Cubes

Plate VII: Oven & Mortar Cube