PROPERTIES OF QUANTENARY BLENDED CEMENT MORTAR CONTAINING RICE HUSK ASH, CALCIUM CARBIDE WASTE AND METAKAOLIN

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(MTECH/SET/2017/7054)

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ABSTRACT

In order to reduce carbon dioxide (CO_2) emissions and increase the strength and durability of mortars/concrete in the cement industry, the use of pozzolanic materials has received increased interests in the last decades. Hence, with a view to producing a PC blended mortar with low carbon foot-print and also cut cost, this study presents three supplementary cementitious materials (SCMs), that were used to produce quaternary binders; Rice Husk Ash (RHA), Calcium Carbide Waste (CCW) and Metakaolin (MK) as partial replacement of Portland Cement (PC). To investigate and evaluate the combinations of the (SCMs) for optimal level of replacement as blending agent in cement, the materials were blended together in a varied combinations proportion of 5 %, 10 %, 15%,20 %, 25% and 30% of PC. RHA were employed for the blended mortar mix in all the mixes prepared. The properties of cements was investigated, their effects on standard consistency, setting times (initial and final), compressive strength and splitting tensile was evaluated. At the fresh state, the flow (workability) and the consistency of mortar mixtures were determined, while at the hardened state, the compressive strength and splitting tensile at the ages of 7, 21, 28, 56 and 90 days were evaluated. Test results showed that RHA significantly influenced the standard consistency of the quaternary binders as compared with CCW and MK. Water demand in the paste increases with increase in RHA content within the paste due to its porous nature as both the initial and final setting time was increased due to their combinations than in Portland cement mortar mix. The compressive and the splitting tensile strength properties of the mortar comparatively improved with rice hush ash, metakaolin and calcium carbide waste. The effect of the SCMs on consistency and setting times in quaternary binder is retardation of the setting time which can be overcome by activation. With the combined use of MK, RHA and CCW results revealed improvement in low early compressive strength development was overcome. Test results also revealed that the compressive strength of mortar cubes also increased as the RHA increased relative to the other pozzolan percentage in the mixes. Therefore, the combination of RHA, CCW and MK could be used as a supplementary cementitious material at 15% percentage replacement to produce cement-based material of higher quality of low carbon foot print.

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CHAPTER ONE

1.0 INTRODUCTION

1.1 Background of the Study

Mortar as materials in construction industry has many applications since it came to be. These include rendering, finishing of structural elements as well as repair material for same applications. This is due to its performance for bedding unit in Masonry, rendering, plastering with concrete when coarse aggregate is added. Apart from the degrading of the environment because of the carbon dioxide Co_2 emission, Mortar has become expensive due to the rising cost of cement.

The amount of emission globally of greenhouse gases, in the atmosphere, CO_2 is about 8–10% of the total gas emission, largely contributed during manufacture of Portland cement (Mehta, 2002 and Sudendro, 2014). According to Shafigh *et al* (2014) this emission made cement industry the most energy intensive industry globally which is worrisome and a thing of great concern as it's the third most energy intensive industry. In order to reduced rate of emissions of Co_2, manufacturing processes by regulating and decreasing the consumption fuel for production and the replacement mineral admixture by the additions of supplementary cementing materials (SCMs) as a clinker production unit or without the use of cement. However, some studies including (Mehta 2009) concluded that meaningful CO_2 emission reduction could be achieved if the heat of end product is reduced while the proportion of the admixtures in cement materials is maximized. Hence, agro-industrial waste is being carried out on production of cement and other cementitious materials.

Several research have been carried out on RHA to replace cement as Calcium Carbide and Metakaolin improved the properties of strength and durability of the materials. Ecosmart (2008) narrated supplementary cementitious materials (SCMs) of 30% of cement is used in its production,CO_2 emissions will be minimized also the disposal problems of byproducts with other forms global wastes that pollutes and degrade environment now employed in other forms greatly to help in the environmental balance. The binary blending method involves having cement mixed together with a cementitious material (SCM) and the technology of ternary blending also involves having cement mixed together with two supplementary cementitious materials (SCMs) while the quaternary blending is the mixing of three SCMs with cement; enhance performance of both mortar and concrete in general.

Supplementary cementitious materials (pozzolanic materials); Metakaolin (MK), blast furnace, FA, RHA etc, contribute to properties which affect cement in mortar and concrete while affecting the economy of construction in general (Amrutha *et al*., 2009). Particularly, some of the pozzolanic materials like the RHA are produced the world over, e.g. Nigeria, use RHA in sustainable built environment as construction material. High silicate content present in Rice Husk Ash (RHA) is highly reactive pozzolan hence recommended friendly and green and useful as it reduces the amount of cement constituent in mortar.

A Calcium Carbide Waste (CCW) is a by-product of an acetylene gas (C_2H_2), used as a fuel for lightening, welding and metal cutting etc.

The carbide residue is produced when water (H_2O) reacts with calcium carbide (CaC_2)as shown in equation (1.1) (Neville, 2012)

$$\mathrm{CaC_2} + 2\mathrm{H_2O} \rightarrow \mathrm{C_2H_2} + \mathrm{Ca(OH)_2} \quad (1.1)$$

Based on research work of Mehta & Monteiro (2006) that Pc reaction of Tricalcium Silicate (C_3S) when exposed in water gives Calcium-Silicate - Hydrate (C-S-H) and Calcium Hydroxide (CH)as expressed in the equation (1.2) and the Portland - pozzolan cement reaction in Equation (1.3)

$$\mathrm{C_3S} + \mathrm{H} \rightarrow \mathrm{C} - \mathrm{S} - \mathrm{H} + \mathrm{CH} \quad (1.2)$$

$$Pozzolan + \mathrm{CH} + \mathrm{H} \rightarrow \mathrm{C} - \mathrm{S} - \mathrm{H} \quad (1.3)$$

C = CaO, S = SiO_2 and H = (OH)

Calcium hydroxide ($\mathrm{Ca(OH)_2}$), an oxide of carbide waste is in a slurry form of hydraulic material. In Nigeria, Calcium carbide waste has accumulated, from road side mechanic workshops gives rise to calcium carbide residues widely spread across the country. Metakaolin (MK) is one of the pozzolanic thermally activated aluminosilicate when kaolin is calcined at temperatures ranging from 700.-850^{o}C (Shebi *et al.,* 2009; Sabir *et al.*, 1996). MK has been used in binary and ternary mixtures to enhance the behaviours of mortar and concrete because of the highly pozzolanic properties which is believed to have doubled the reactivity of most other pozzolans hence taken to be a viable admixture.

Siliceous or siliceous and aluminous materials are constituents of pozzolanic materials, calcium hydroxide ($\mathrm{Ca(OH)_2}$) with water in good form, can react with cement paste to form additional calcium silicate hydrate (C-S-H). This is because rich $Ca(OH)_2$ in Calcium carbide waste can react with these siliceous or siliceous and aluminous materials of RHA and MK to form more durable calcium silicate hydrate(C-S-H) as expressed in equation (1.3), that improves properties of Mortar.

Calcium Carbide waste (CCW), Rice Husk Ash (RHA) and Metakaolin (MK) an admixture containing $Al_2Si_2O_2$ influence strength development as material constitutes materials gets consumed while other are formed. RHA is also a highly reactive pozzolan with high content of SiO_2 which readily reacts with positive ions from other pozzolans but requires more water due to its porous nature which can be off-set with CCW. MK is also a very reactive pozzolan and its heat of hydration is a drawback but with CCW with water being produced as a by-product will hinder by lowering the heat of hydration from MK. Combining pozzolans to react with Portland Cement (PC), a better mechanical durability properties compared with binary and ternary blends will be produced.

1.2 Statement of the Research Problem

The enhancement of the properties of mortar is a major concern in construction companies. Studies have shown that property of mortar is improved with PC blended materials (SCMs) like RHA, MK and CCW Makaratat *et al.*, (2004). These materials have been used in replacing PC and it has been established that approximate optimum level is 15% to 20% by weight of PC (Ranan and Ganesan 2012). As the replacement of PC with these SCMs aforementioned increase, strength and durability of mortar are affected due to decrease in calcium oxide (CaO) content in PC and increase of other chemical components. This effect(s) on structural properties of mortar caused by decrease in CaO content of cement can be addressed in logical combination of some SCMs rich in CaO content as well as high content of other chemical components so as to neutralise the effect on the properties of construction materials afterword. In this light, RHA, MK and CCW were used to partly replace PC at varying proportions in quaternary mixes to enhance fresh and hardened properties of quaternary blended Mortar. Chemically, composition of CCW has 95.69% CaO content on reaction in presence of water produces calcium hydroxide ($Ca(OH_2)$) and water (Makaratat *et al.*,2011).

This Ca (OH_2) reacts with Ca and Al_2O_3 from disordered MK and form C-S-H with C-S-A-H early rather than wait for $Ca(OH_2)$ liberated from PC hydration. CCW contains alkaline solution with pH greater than 12 and readily aids reaction to continue (Sun *et al*., 2015). This shows that low water/cement ratio can be employed in the mix proportion. MK is reactive because of its disordered structure. It readily reacts with SiO_2 from RHA (73.60%) content to further produce C-S-H. Studies showed binary blended cement with MK has its draw back in increase in hydration energy while, while binary cement with RHA leads to increase in water content due to its reactive nature and low durability due to its porous nature.

This short coming has been taken care in the synergy between MK, RHA and CCW. CCW(Hydrolic Material) with 95.69 CaO react with water to produce $Ca(OH_2)$

.$Ca(OH_2)$ will react with Al2O3 in MK to produced C-S-H and C-S-A-H early rather than wait for $Ca(OH_2)$ liberation from Pc hydration. CCW with pH greater than 12 readily aid reaction to continue while MK readily react with SiO_2 to further produce C-S-H.

Chemical components of these SCMs, their combined effect certainly improved structural properties of mortar as the synergy between them is brought into play. For example, water from CCW takes care of high water demand of RHA due to its porous nature and heats of hydration from MK reaction. Hence this study focussed on the improvement of strength properties of quaternary blended binder incorporating RHA, CCW and MK, Thus mitigating the environmental degradation caused by these waste materials.

1.3 Aim and Objective of the Study.

Research work is to evaluating the properties of binary, ternary and quaternary blended cement Mortar of Rice Husk Ash (RHA), Calcium Carbide waste (CCW) and Metakaolin (MK) with a view to producing a low carbon foot print blended PC.

The specific objectives of the study are to;

i determine the physiochemical properties of the constituent materials (RHA, CCW and MK)

ii establish optimum mix proportion for the blended cement.

Iii evaluate the fresh properties of the blended cement mortar from various mixes.

vi evaluate the hardened properties of mortar from various mixes

1.4 Significance of the Study

Research project work gives information of binder which is beneficial and serves as foundation for model development for a binder. It also helps the results of existing standards to either adjust or modify code of practice.

The use of the pozzolans and incorporating materials in no-doubt contribute to the reduction of the waste burden in the environment, thereby reducing environmental degradation and other related problems.

Also, the hydraulic properties provide and pave way for further researchers to develop a better mortar/concrete work.

The results of data obtained will provide information to prospective researchers, scholar's areas of further research work and also help policy makers or standards created.

1.5 Scope of the Study

The properties of quaternary blended cement mortar containing Rice Husk Ash, Calcium Carbide waste and Metakaolin were evaluated in both the preliminaries of fresh properties that involves the setting time, soundness, workability (flow rate), Standard consistence and hardened properties (compressive and tensile) at 5% interval to 30% replacement levels of PC of each admixture and test specimens cured at 7,14,21,28,56 and 90 days hydration.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Mortar.

Mortar is bond together using binding medium comprising inert materials that are composite. It's material of fine aggregate (sand), water, and cement as binder that bond all its constituent together, air and sometimes admixture. When the paste is form by the combination of cement, water, air and sand mixed together in good proportion having the strength of mortar is dependent on the type of binder (Abebe *et al*., 2002). The mortar is placed in a plastic state to develop strength (harden), a process under proper curing conditions, may continue for years (Ramasamy and Biswas, (2008).

A hardened and fresh property of mortar is a fundamental knowledge for construction industries to ensure good performance in masonry walls (Vladimir *et al.*, 2011). Emmitt and Gorse (2014) puts simply, proportioned mix ratio of cement and sand with conditioned water content to form a paste.It explained that the basic requirement of mortar to include its ability to remain hardened without crushing while remaining sufficiently plastic as it is laid to take the varying sizes of bricks. It is also expected that the mortar should have the porosity that is identical to the bricks so that it will not deteriorate to the weathering action of rain or frost.

Sand has good strength in resisting crushing, cheap and also attains plasticity when mixed with water. Furthermore, sand has impervious grains as such it is able to resist the action of rain and frost. The materials that bind the grains of sand together into a solid mass are lime, cement and water known as the Matrix.

2.1.1 Types of mortar

Classifications are in four major classes, namely; Types M, Types S ,Types N and Types O (being in ASTM C 270, 2002). Types M, Types S, and Types N are specifications typically required and used by building codes. Some mortars are restricted by Building codes for particular applications. Such as in the design of foundation walls with categories Types M or Types S mortar. The glass unit masonry requires categories Type N or S mortar while the Seismic design categories of types D, types E, and types F Portland cement/lime or mortar cement mortar Types S or Types M is also required

Table 2.1 Property specification requirements in ASTM C270-02

Types	Average Compre. Stght at 28 curing Days min. psi (Mpa)	Mortar
M	250 (17.2)	Mortar Portland Cem.
S	1800(12.4)	Mortar Portland Cem.
N	750(5.2)	Mortar Portland Cem.
O	350(2.4)	Mortar Portland Cem.

Source (ASTM C270-02)

2.1.2 Constituents of mortar

The Design mix of mortar is made up Portland cement, fine aggregate, water, Rice husk ash and calcium carbide waste. Constituents of mortar are;

2.1.2.1 Portland cement

This is discussed extensively because the topic deals with partial replacement of binders. Hydraulic cement hardens when reacting with water will also forms water-resistant product. Pulverizing clinkers made-up of hydraulic calcium silicates containing more forms of calcium sulfate as ground addition is the Portland cement as defines by ASTM C150(2012).

Hydraulic material in Portland cement is made of two-thirds mass of calcium silicates, ($3CaO{\cdot}SiO_2$, and $2CaO{\cdot}SiO_2$), other materials of aluminium- and iron-of clinker phases with other compounds. CaO to SiO_2 in ratio having not less than 2.0. Oxide of Magnesium (Mg O) is also not exceeding 5.0% by mass. Clinkers above 90% with limited calcium sulfate ($CaSO_4$, controls setting time), with about 5% of other constituents (fillers) that are minor are in various standards. Clinkers are nodules (diameters, 0.2–1.0 inch 5.1–25.4 millimeters) of a sintered material are heated to high of predetermined temperature to produce a new mixture of raw material composition. Portland cement is define (>1,300 °C (2,370 °F)) as belite (Ca_2SiO_4) combines with calcium oxide (CaO) to form alite (Ca_3SiO_5) during chemical reaction from other hydraulic limes at high temperatures.

The classification of cement with general features according to ASTM C150(2012) into various uses, types, with its constituents in the Table 2.2 below. Mortar needed for small work is hand mixed and bigger works are mixed using mixing machine during preparation.

Table 2.2: Portland cement and its features.

Classification	Characteristics	Types	Applications
One use for General purpose	For strght dev. At early stage C_3S	1	Employed in Bridges,pavement and pre- cast units.
Moderatelly Sulphate resistance	We have C_3A low which is less than 8%	2	Works exposed to sulphate ions of soil or water.
Early dev. Of high strength.	Grounded finely and has slightly more C_3A	3	Employed where Rapid constn. Work,also in cold weather concreting.
Has heat Hydration low with slow reaction.	Also C_3A is low which is less 50%	4	Employed in Big Structures such as Dams.
The Sulphate resistance is high.	And has very low C_3A of less than 5%	5	For strucures in region and expose to very high level of sulphate ions
Has its colour White	With low MgO an no presences of C3AF	White	For decorative units and also tend to have same with 1

Source; Shetty(2005); Mehta & Monteiro (2006) & Neville (2012).

Well ground powders will enable chemical reaction (hydration) takes place when mixed with water will have the important property.

a) Binding medium that is strong and hard of aggregate particles is produced by Hydration.

b) The amount of cement required in concrete or mortar is selected based on Particular properties required.

2.1.2.2 Chemical composition of Portland cements

The materials of silica, lime, alumina and iron oxide are used and 90% cement Oxides, while other oxides of Portland cement is;

Table 2.3 Oxide composition of PC

The Oxide	Name	Abbreviation	Composition limits (%)
CaO	Lime	C	60-66
SiO_2	Silica	S	19-25
Al_2O_3	Alumina	A	3-8
Fe_2O_3	İron oxide	F	1-5
MgO	Magnesia	M	0-5
Na_2O	Sodium	N	0.5-1
K_2O	Potassium	K	0.5-1
$S0_3$	Sulfur trioxide	S	1-3

Table 2.4: Typical chemical analysis of PC.

Item	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	Na_2O	K_2O	LOI	residue	Free
Per (%)	63.6	20.7	6.0	2.4	1.4	2.4	0.1	0.7	1.2	0.3	1.1

i) The source of SO_3 (sulfur trioxide) is ($CaSO4 \cdot 2H2O$) from gypsum whose amount is derived by approximately multiplying the amount of SO_3 by 2.15.

ii) Detrimental expansion of MgO can be control if it is limited to 5%. (Expansion of hardened concrete as a result of hydration of free MgO).

iii) MgO and CaO are free but CaO is undesirable as they are oxides that hydrate much later than other compounds of cement. After hydration, large volume of expansion is noticed resulting in disintegration of hardened concrete $CaO + H_2O$ $Ca(OH)_2$. Specified limiting value of alkali oxides for cements are usually used aggregates that are reactive to prevent disruptive expansion that is create from alkali-aggregate reaction.

i) L.O.I. (loss on ignition):

Shown before hydration or carbonation storing improperly for long time.

The loss in weight in cement heated at 1000°C. i.e. LOI ≤ 3% ASTM C311 (2020)

ii) Insoluble residue. (I.R): These are the materials of cement that is not soluble in HCl acid, they are non-reactive silica that form silicate compounds in kiln. These show completeness of reactions in the kiln.

iii) IR ≤ 0.75% ASTM C311 (2020)

Cement clinker formed of typical composition is illustrated below;

Table 2.5 The composition of chemical in the clinker

Formula	**Compound**	**Shorthand form**	**% by weight**
$Ca_3Al_2O_6$	Tricalcium aluminate	C_3A	10
$Ca_4Al_2Fe_2O_{10}$	Tetracalcium aluminoferrite	C_4AF	8
Ca_2SiO_5	Belite or dicalcium silicate	C_2S	20
Ca_3SiO_4	Alite or tricalcium silicate	C_3S	55
Na_2O	Sodium oxide	N	Up to 2
K_2O	Potassium oxide	K	
$CaSO_4.2H_2O$	Gypsum	$C\underline{S}H_2$	5

2.1.2.3 Properties of cement compounds

i) , C_3A, - Tri-calcium aluminate :-

The compound liberates heat during the initial stages of hydration, and contributing low strength while retarding the hydration rate of C_3A by gypsum as cement is sulfate resistant.

ii) C_3S- Tri-calcium silicate:-

The result of the initial set and early strength gain of Portland cement is from the rate of hydration and it's rapidly hardening.

iii) C_2S- Di-calcium silicate:-

strength gain is as from a week which is largely responsible for its slow hardening and hydration.

iv) Ferrite, C_4AF:

This is an agent (Fluxing agent) that hinders melting temperature of the cement materials in the kiln from 3,000º F to 2,600º F and strength it contributed is small but hydrates rapidly

Methods of mixing by the Manufacturers to suit several construction environments.

i) Complex compounds are formed when Oxides interact with each other. The measurement of conventional chemical methods is not possible.

ii) Four basic chemical compounds of Portland cements below.

$3CaO·SiO_2 = C_3S$ Tri-calcium silicate.

$2CaO\cdot SiO_2 = C_2S$ Di-calcium silicate.

$3CaO.\ Al_2O_3 = C_3A$ Tri-calcium aluminate.

$4CaO.Al_2O_3.Fe_2O_3 = C_4AF$ Tetra-calcium aluminoferrite

Table 2.6 PC main chemical compounds

Usual Abbr.	Chemical Composition	Compounds Name	Percentage(%)
C_3S	$3CaO.SiO_2$	TricalciumSilicate	51
C_2S	$2CaO.SiO_2$	DicalciumSilicate	23
C_3A	$3CaO.Al_2O_3$	Tricalciumaluminate	8
C_4AF	$4CaO.Al_2O_3Fe_2O_3$	Tetracalcium aluminoferrite	9

The fundamental stages in cement production are ;

i. Materials are reduced to finer size particle.

ii. Silica, iron oxide, calcium carbonate and alumina are materials with uniform chemical composition that are well mixed and blended.

The end user has to know both the physical and the characteristics of cement before being employed for use which are normally specified as follows below according to ASTM C150; 2012).

2.1.2.4 Manufacture of Portland cement

The kiln containing mixture of cement materials heated to a temperature degree of 700°C having 1,450°C temperature fusion for most resent cements manufacturer used in transforming it into clinker of constitutes :- alite, belite, tri-calcium aluminate, and tetra-calcium alumino ferrite. Lower temperature are form by present of flux allowing the calcium silicates bring about presence of aluminum, iron, and magnesium oxides contributing little to the strength. Limestone ($CaCO_3$) mixed alumino-silicate a clay source are major materials for clinker. Limited amount of tricalcium aluminate are needed for low heat cements in that categories respectively as Sulfate resistant (SR) types, ($3CaO·Al_2O_3$) are formed. Limestone with CaCO3 constituents could be low as 80% depending on its purity. The $CaCO_3$ content of this can be as low as 80% depended on the purity of the limestone. Materials of secondary raw material such as Ashes of coal and those used are clay, shale, sand, iron ore, bauxite, fly ash, and slag, by the time cement kiln is heated by coal.

2.1.2.5 Hydration of cement

i) Cement hydration and water produces a binding medium aggregate particles in concrete that is hard and strong.

ii) Normal construction of structural sizes members that are small produces heat that poses no problems.

iii) While in cold weather concreting heat is advantageous.

iv) Thermal cracks are formed and generated heat in hot weather when it is in mass concrete construction should be avoided.

Determination of cement hydration is measurement by;

i) $Ca(OH)_2$ is presence in the paste,

ii) It generated heat during hydration.

iii) Cement paste unhydrated Specific gravity noted,

iv) Chemical combined water amount noted,

v) Unhydrated amount of cement paste.

2.1.2.6 Fineness of cement

Cement material fineness with water is highly determined to know degree of reaction of cement as far as water is concern. It is because, finely binder material has more revealed surface area when compared coarsely fine one. Total surface area of materials of cement represents the rate by which hydration takes place and depends. on the fineness of the cement particles that produced no effects in strength development and the early heat evolution is as a result of early hydration (Rahhal and Talero, 2010; Neville, 2012). The behaviour of cement is measured from specific surface of cement determined with air permeability apparatus where typical value of PC specific surface is $300m^2kg$.

2.1.2.7 Soundness of cement

When we have paste cement i.e. in its slurry state, hardens without any cracking or disintegrating its material constituent, is sound cement (Klemm, 2005). Cement particles surrounded by lime (CaO), magnesia (MgO) and sulphates (SO_4) caused unsoundness of cement by preventing easy hydration of free lime (uncombined lime) and other materials during normal setting time. If you require cement to be sound, it requires thoroughly mixed, well burnt and also well ground Maximum of 0.80 per cent for all Portland cement types while NIS 444:2003 specifies a maximum of 10mm ASTM C 150 (2012) standard of Portland cement .Le Chaiterlier apparatus is widely used to checking the soundness of cement (Shetty, 2009).

Soundness of test Specimen, cement paste mix ratio 1:3 for mortar was prepared in Building Laboratory in a mould and was immediately immersed in water and was removed after 24 hours. The harden mortar in Le Cheiterlier apparatus for boiling. Time placed in water and when its removal was noted and then later removed after 3hours of boiling. A check of expansion of the size of the hardened mortar was carried out and recorded. The same procedures used for the cement paste for the determination of soundness was also implored for the binder paste placed in Le Cheiterlier mould with water filled while noting when it was placed in water and when its was removed at elapse of 24 hour noting also the change in expansion. The change result from expansion was measured and with Vernier calliper. Apparatus use for this test, Le Cheiterlier mould and bath, glass tray, spatula and Vernier calliper of Model No EL 38 – 3400 by ELE according to BS EN 196-3-2005.

2.1.2.8 Setting of cement

i) Initial set a stiffening process in cement paste noticed initially.

ii) Initial set is the last hardening of cement process responsible for its strength known as final set.

iii) Addition water after the set of to both the initial and final set is known as the setting times.

iv) Setting time of cement changes when the cement composition, cement fineness, rate of hydration, and the ambient temperature affect setting times of cement measured with the Vicat apparatus was employed in measuring setting time which has different penetrating attachments. BS EN 196-3: 2005 has laid the procedure of the initial set, with a diameter 1.13 ± 0.05 mm needles used. Standard consistency will have its needle of the Vicat penetrates the cement paste in a mould by its weight. As cement paste stiffens, penetration of 5 ± 1mm occurs. When this happens, we then say the initial set has taken place which is expressed as time elapsed when mixing water was added to the cement. Usually according to standards 60 minutes period minimum is prescribed by BS EN 197-1: 2000 for grade 42.5 N/mm^2 cement while 75 minutes for 52.5N/mm^2 cements strength and 45 minutes for higher strengths. These are all standard prescribes in the American Standard ASTM C 150 (2012) where initial set using the apparatus is 45 minutes a minimum time in ASTM C 191(2008).

2.2 Fine Aggregate (Sand)

A constituent of Mortar, aggregate materials that passes 4.75µm (No 4) sieve, retained on 75µm sieve number 200 of ASTM C125 (2015). A lower size limit of 0.07mm or little less is usually considered. If these properties are constant, the flowing ability and segregation resistance is improved, because well graded fine sand improves the flowing ability, packing density which subsequently enhances the strength and durability of Mortar.

2.3 Water.

This is obviously useful liquid in preparing Mortar/Concrete work. Its quality is highly important because many substances dissolve more or less easily in water because of the impurities sometime affects the setting time and subsequently influence the mix of Mortar/Concrete materials after all. It seriously affects concrete as it leads to corrosion of reinforcement as a result of chemical attack. Therefore it is important and highly suitable to have water for mixing, curing and for construction work portable. Portable water is generally safe for work while water isn't potable may be satisfactory and can be consider for mixing of Mortar/Concrete. Water of high concentration of sodium or potassium is not suitable for Mortar/Concrete mixing and a potential of alkali aggregate reaction.

2.3.1 Water absorption

Water absorbed by mortar determined its durability. When more water is absorbed by mortar/concrete less its durability. Measure of permeability known as absorption is defined as process of porous medium materials of a solid body that liquid passes through, such as paste, mortar or concrete (ASTM C125, 2015).

2.3.2 Workability.

Strength development and good workability have been the favorite features. However; it is often difficult to overcome concurrently using conventional water-to-cement ratio, The paste property influence the homogeneity of mixed, placed and consolidated or simply the energy to overcome friction while compacting or the relative ease by which mortar or concrete can be mixed. Workability of mortar or concrete is increased when water cement ratio increases. Hence workability of cement can be determined by the materials used in the mixing of cement. Such as water, sand and aggregate properties such as size, shape, grading, mix design ratio and use of admixtures.

Pozzolans and water when combine with lime at ordinary temperatures will form stable insoluble compounds possessing cementing properties. Workability for a given flow increased its specific surface, which lower water demand of cement paste in pozzolans.

2.3.3 Flow rate

Flow rate is a measure of workability for Mortar and it is the ratio of diameter increase by original diameter multiplied by 100.

$$\frac{100 * M_1 - M_2}{M_1} \qquad (2.1)$$

If flow is in the range of 75% to 80% the compressive strength of Mortar is maximum. This is used to determining the workability of mortar paste in building. It is made of Flow Table Test; Flow table a 30 cm. diameter ground with steel plate polished and has three inscribed annular circles of 7,11 and19cm diameter. Its top is The apparatus is designed to have a fall of 12.5 mm by a cam action. The components of the flow table; one brass conical mould, 65 mm ID at base and 40mm ID at top and mould height is 90 mm.

Figure 4.5 Flow table concrete test

2.4 Segregation

Segregation could come about during mix and having its internal factors not well proportioned with its workability not too well workable mix. External factors as well improper transportation, placement and vibration or adverse weather conditions could cause segregation.

2.5 Density

Specimens was surface dry and weighed with a weighing balance so as to determine its. BS EN 12390-7 (2009) Specified. Mortar density is got using equation.

$$Density\ D = \frac{\mathrm{M}}{\mathrm{V}} \qquad (2.2)$$

Where D- density of mortar (kg/m$^{3)}$

M- Mass of mortar (kg)

V- Vol. of mortar (m$^{3)}$

2.6 Compressive Strength

Samples (cubes) was removed from curing basin placed outside for surface dry, weighed and positioned and set for compression machine to crushing. The mortar samples were subject under load of crushing machine till it failed. BS EN 196-1:2016 is a specification used for prescribing compressive strength test on mortar specimens of European Standard.

Compressive strength – 50mm cubes

Compressive strength $$F = \frac{P}{\mathrm{A}} \qquad (2.3)$$

Where: F – Is the strength (KN/mm^{2,}) and P- Is the load (KN) and A - cross-sectional area of the specimen.

2.7 Abrasion Resistance

The resistance to wearing by rubbing or friction of a surface is abrasion resistance. (Scott and Safiuddin 2015). They are factor that are highly of important in construction industries because of the kind of treatments floors, roads, or pavements desires for them to be durable. Surface materials with enough curing, good finishing and aggregates that are hard, well mixed in good proportion to enhance paste bond, placing and compaction are factors responsible for abrasion resistance. Abrasion resistance can be improve by some SCMs such as FA,GGBFS,SF ,super plasticizers and other forms of SCMs once its porosity and void content are change .(Haque and Kayali, 1998; Ozbay *et al* (2010) used about 40% GGBFS in his study to have an increment of about 20% in abrasion resistance in concrete. In their conclusion that abrasion resistance can greatly be enhanced in presence of GGBFS in concrete mixture at a marginal level of 20% to 40%. Another research work by Rashad *et al* (2014) who conducted abrasion test on high-volume FA (HVFA) concrete where FA and GGBFS at 10% and 20% was used to have a result indicating higher abrasion resistance of HVFA blended with GGBFS. Rao *et al* (2016)

For abrasion resistant mortar/concrete, factors such Sound, hard aggregates be selected with materials are dense and would result in a, robust mortar/concrete. Having high strength and low w/c is critical for concrete to resist stresses imposed by abrasion. Having good finishing methods and well cured material will help to maximize surface hardness of the mortar are paramount factors in respective of their constituents will maximizing abrasion resistance. The performance of mortar against abrading can be determined using a suitable test method given by ASTM.

Many researchers agreed that a general trend between the two , enhance the strength of concrete reduces the effects of abrasion. Liu, *et al* (2005), Dhir *et al* (1991) , Atis (2002) .

Table 2.7 Los angeles abrasion test- LAA

Mass of Charge	Number Spheres	Grading
5020 ± 5	12	A
4606 ± 5	11	B
3345 ± 5	8	C
2510 ± 5	6	D

2.7.1 Steps of sample preparation

1) Washed and oven-dried the sample at 105ºC to 110ºC to substantially constant mass.

2) Separated the samples into individual size fractions and recombined to the grading.

3) Prior mass to test on the sample to the nearest 1g (m_1)

2.7.2 Abrasion test procedure

1) Rotate the Los Angeles testing machine after placing charge and sample in the machine moving at 30 to 33 rpm for 500 revolutions.

2) Sample material are separated from the sample after 5000 revolutions in the testing machine and sieve using 1.70 mm sieve for a finer portion on a 1.70 mm sieve.

3) Sample material more than 1.70 mm sieve in size are wash .

4) Over-dry at 105ºC to 110ºC

2.8 Bleeding

This is a situation where water in Mortar /concrete mix rise through the pore to the surface of the placed material resulted from the water that failed to be absorbed by the higher components present in the concrete when the mixing is finally through. Bleeding of water in mortar/concrete seizes when cement paste finally hardened enough as the sedimentation end. Types of cement in mortar can influence bleeding capacity, increased of cement in proportion. Presence of finer aggregate (smaller than 150µm) in good proportion reduces bleeding. Similarly polypropylene micro-fibers are known to reduce bleed. Remixing the bleed water during finishing of top surfaces where it occurs weaken such surfaces. Finishing can be delay to prevent bleeding as water will evaporated from the surfaces of placement. Where we have the evaporation of the surfaces faster than the rate of bleed, plastic shrinkage cracking may result.

2.9 Rice Husk Ash (RHA)

RHA is manufactured through processes involving burning of rice husk either through methods of open field or incineration of the husk while temperature are controlled to specified time duration. High carbon content in RHA results from open burning that adversely affects the performance of mortar that makes the structure highly crystalline form (Hwang and Chandra, 2016). Thus, the amorphous the form of silica of RHA in is potentially employed for structural concrete. There are different Ash production methodologies of which have merits and demerits.

At higher burning temperature the Ash increases with Silica present Rice husks Ash (RHA) with high reactivity and pozzolanic property.

RHA under burning temperatures of between 600°C and 700°C period of 2 hours, contains 90-95% SiO_2, 1-3% K_2O and < 5% un-burnt carbon. At controlled burning temperature Mehta (1992), RHA contains silica in amorphous and highly cellular form, with 50-1000 m^2/g surface area. Workability and stability are improve with RHA partial replaced then combined with cement as it reduces heat evolution, thermal cracking and plastic shrinkage with increased strength development.

When Pozzolanic reaction of RHA takes place, $Ca(OH)_2$ present in a hydrated Portland cement paste are consumed which reduces susceptible to acid attack and improves resistance to chloride penetration. This reduces large pores and porosity resulting in very low permeability. The reaction of the pozzolanic materials with RHA inflence the lime present in the cement paste that affect the reaction by decreaseing the permeability of the system and improves overall resistance to CO_2 attack which enhances resistance to corrosion of steel in concrete.

Use of these materials in mortar /concrete will assist to lessen the adverse effects it has on the environment from myriad of activities that take place in the construction industry, by the replacement of cement with this pozzolan. In his research work (Nehdi *et al*., 2003), Materials of RHA from a temperature of 650°C for 60 min is considered non-crystalline and also save the RHA production time, while it is recommended that the burning temperature should be at 700°C for a period of two hours.

Table 2.8 Chemical composition of rice husk ash (RHA)

Sample	Si_2O	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	LOI	SAF
RHA	87.2	0.15	0.16	0.55	0.35	0.24	-	-	5.44	87.51

Swaminathen and Rava (2016)

2.10 Calcium Carbide Waste (CCW)

Researchers like; Amnadnua *et al.,* 2013 used FA and other forms of cementing agent for manufacturing mortar, concrete and masonry units without PC. This shows the use of CCW as partial or full replacement of PC, where CCW blended as pozzolanic materials that improve the engineering properties of soils with deficiency.

Jaturapitakkul *et al* (2003). Calcium carbide waste and rice husk ash with high compressive strength could be used as cementitious material in mortar production. Krammart *et al.*,(2004) used calcium carbide waste as part of cement raw material, and found that the compressive strength of calcium carbide waste cement mortars was close to that of the control cement. Here in Nigeria, CCW is about 70-80% calcium hydroxide $Ca(OH)_2$ with its constituents as copper, lead, iron, manganese, nickel and zinc (Semikolennykh *et al*., 2012; Chukwudebelu *et al*., 2013).

Table 2.9 Chemical Composition of Calcium Carbide Waste

ChemicalComposition	CaO	MgO	Al_2O_3	Fe_2O_3	SiO_2	SO	LOI
CCW	61.41	0.80	1.78	0.17	2.69	0.36	32.51

Source (Manesseh and Joseph, 2016)

2.11 Metakaolin

Metakaolin (MK) is use have being of grate interests in recent years (Siddique and Klaus, 2009). Kaolin deposits can be nearly pure kaolinite or may contain impurities that affect the characteristics of the clay with a melting point of 1750 °C and is a clay mineral that has a chemical composition of $Al_2Si_2O_5$.Also they are clay forming with hydrous aluminum silicates (AL_2 ($Si2O_5$) $(OH)_4$) which are initiated by the decomposed Aluminous compounds. Among the decomposed aluminous compounds is Feldspars. Incorporating Metakaolin as admixture adds to the properties of concrete. In paste of mortar/concrete, it eliminates or minimizes the bleeding, reduces segregation. As the microscopic structure improved, the materials becomes durable and the replacement of Metakaolin accelerates the strength gain in concrete at early age.

2.11.1 Source location of high purity kaolin

One of the sources, Alkaleri is a little settlement 74 kilometers East of Bauchi, Bauchi-Gombe road. Kaolin deposits of this material are at the Jalgalwa River bank at the two sides of the bridge over the river at Alkaleri. The deposit outcrops along the bank of Jalgalwa River is dry from May to October.

They are broadly in two types:

a) With the alteration of feldspar-rich rocks, Primary (residual) kaolin are developed.

b) While accumulated deposits, products of eroded residual materials forms the secondary kaolin. Found in stream, lake and basin environments.

Table 2.10 Proven reserve of high purity kaolin in Alkaleri

Spot Identification	Area (m^2)	Average overburden (m)	Kaolin thickness (m)	Specific gravity	Reserve (tonnes)
Area 1	46.920	6.5	13.2		1,610,294.4
Area 2	169.764	5.5	16.0	2.25	7,062.182.4
Total	216,687	-	-		8,672,476.8

Source; Journal of Emerging Trends in Engineering and Applied Sciences (JETEAS).Geological investigation of kaolin deposit, Bauchi State, Nigeria.

Kaolin uses are multiple and diversified. Its whiteness nature and plasticity behavior create its great demand hence becomes extensive as a raw material in refractories, catalyst, cement and fibre glass industries. It is unique as is chemically inert with wide pH range that provides good covering when employed as a pigment or extender in coated films and filling applications. Kaolin is soft, non-abrasive and has a low conductivity of heat and electricity.

The Origin of the material can be alter by beneficiation and blending, hence the need of calcination of 700°C to 800°C.

2.11.2 Durability properties of metakaolin

Flexural, compressive, tensile strength and durability are enhancement with the incorporation of MK (Justice *et al*., 2005). This comes as a result of Interfacial Transitional Zone (ITZ) present primarily to improve the path of flow. ITZ with more pores to allow capillary action of water to cementitious materials. Chemical composition of the ITZ and its mineralogical defer from the bulk paste material of Metakaolin that hinders the chemistry and microstructure ,the ITZ, is responsible in the transportation of ion that are decreased into concrete materials then later improve the concrete durability.

The enhancement of strength of MK is dependent on purity; it is a effective pozzolan because of its low drying shrinkage and higher durability when in used with cement based system.

2.12 Properties of Binary Blended Cement

Mortars paste of 18 different mixtures of binary blends comprising Rice Husk Ash (RHA) and Portland cement (PC) with three series of mix was produced. PC in the mixtures (5%–30%) partially replaced with RHA by weight of the binder at fresh state, flow(workability) was determined, also at hardened state, is the compressive and Split Tensile strength at the ages of 7, 28, and 90 days was determined. A change in properties with the reduction of flow of mortar was observed from the results when the combined use of RHA compared to PC is separately employed. Compressive strength development is enhanced with about 5%-10% RHA added to the content. The results of this combination of PC and RHA which produced higher quality can effectively be employed as supplementary material of cement-based material.

2.12.1 Blended cement

Pozzolanic material additions to Portland cement known as blended cement are so significant because of their various beneficial effects. It is used in many concrete structures liable to failure such as corrosion in hot climates (a condition prevailing in some part of the World) thereby making it virtually necessary.

It is a mix of Portland Cement (PC) with blending materials as Metakaolin, Rice Husk ash and Calcium Carbide Waste to enhance its properties for different uses.

They are cements produced by uniformly mix of Portland cement and blending materials of by-product.

First stage of Blended cements. Blending of Rice Husk Ash with Portland cement at percentages of 5%, 10%, 15%, 20%25% and 30% with CEMI42.5N grade, and w/c of 0.5 as binary. 2nd is blended cement pastes RHA/CCW- at percentages of 5%, 10%, 15%, 20%25% and 30% with CEMI42.5N grade as ternary and the final is blended cement pastes RHA/CCW/MK at percentages of 5%, 10%, 15%, 20%25% and 30% with CEMI42.5N grade as quaternary. Mixture was mixed with water: cement: sand at ratio of 1:3. The blended material cement improves properties such as the chemical resistance in concrete also the workability and strength.

2.12.2 Ternary blended cement

The replacement of PC at 5% –30% using three blended materials i.e two SCMs with cement to improve the performance of Mortar in general. The properties of mortar mixtures at fresh and hardened state were determined at the ages of 7, 21, 28, 56 and 90 days comprising compressive, Splitting Tensile strength and the workability (flow).

As RHA percentage increased relative to other pozzolans percentage in the ternary blends, it is observed, RHA increased compared to the ternary blends, the Bulk Densities increased. After 28 days curing, the Compressive Strengths of mortar cubes for ternary cement mixes with 70% Portland Cement (PC) and 30% of combined Rice Husk Ash (RHA) 15.16 N/mm^2.The optimum strength at 90% PC combined with 5% RHA and 5% Calcium carbide waste is 19.09 N/mm^2.

2.12.3 Quaternary blended cement

This is a mix of three SCMs and PC at 5%–30% by ratio using quaternary enhances the performance of Mortar. The properties for 7,21,28,56 and 90 days curing was determined such that the compressive and Splitting Tensile strength and flow (workability) are known. Standard consistency of MK quaternary binder increases with increasing in percentage levels of MK, as a result of the pore high surface area and high water demand of MK. Small replacement such as 5% of PC by some binders shows a consistency that small control effect as partial replacement of PC (50%) by RHA, CCW and MK affect the consistency much more because increased could also be in limit and almost similar to PC. The property of MK and RHA which is the demand of water is very similar to that of PC. However, ternary (PC–MK–RHA) binder setting properties increase in quaternary concrete (PC-MK-RHA) with addition of CCW. The results showed Hydration process is increased with pozzolanic materials increase as well as the setting time period.. The advantage of Delayed in setting is an advantage because it give concrete time of work, transport while mixing and usage. The behaviors help to reduce risk of cold joints in larger concrete areas.

2.13 Pozzolans

A siliceous material, Pozzolan a replacement material used in cement mortar are either made naturally or Man-made. The lowered heat generated during hydration that improves the properties workability and durability during reaction with calcium hydroxide and alkalis that may exist sulfate and alkali-silica reactions. It has its benefits when use in big and large concrete works like dams and bridges (Gibbons 1997).

Pozzolan are;

Naturally deposits are ash-like whose origins are of volcanic activity, usually around Europe and Middle East. We have also the organic ash pozzolan which is weak when compared to type, produced by burning coal or lime, they are not incourage in brick/mortar construction works. Pozzolans produced by crushing rock and sand, that is been used in mortar throughout history but are not commonly used today (Gibbons 1997).

Most pozzolans are plentiful and because current uses for them are limited, they represent a potential source of inexpensive construction material. Some pozzolans can be processed into a material with characteristics similar to Portland cement, so it is feasible that a significant portion of cement in a concrete mixture may be replaced by pozzolan.

2.13.1 Activities of pozzolanic materials

Ordinary Portland cement reacts with silica present in pozzolans ie, by reacting with free lime present in cement. Used with lime, to make it hydraulic it is generally added during mixing.

Principle pozzolans in this study; Rice Husk Ash contains about 80 per cent silica varying with the type of rice husk. When burnt with free access to atmosphere, then the resulting silica is crystalline but when burnt under controlled condition with limited access to air to temperature of about 700 for 2 to 3 hours, then the silica produced is non-crystalline but amorphous. This rice husk ash has very good pozzolanic properties.

Synergy arise in ternary mixtures when different pozzolan that are reactive is employed, their results is higher than those in binary mixtures is known as synergic effect Isaia,(1997). This mean that having more than one SCM is likely change the behavior properties of both the mechanical and structural by improving cement matrix.

Pozzolanic materials falls in two groups classified as: Natural pozzolanas are of volcanic origin with volcanic ash referred to by Neville (2012) original Pozzolan. According to Parhizkar *et al* (2010) .

Natural pozzolans are reactive silica or alumina as a natural material with no binding properties but in the presence of Portland cement will react with lime and water will = harden and set the cement material. These pozzolans are group into four based on lime reactive constituent present Ramezanianpour (2014).

Artificial type are materials got from heat treatment and materials with low pozzolanic activity and need further treatments to achieve pozzolanic activity; it results from chemical or structural modifications of materials originally having no or only weak pozzolanic properties (Ramezanianpour, 2014).Some artificial pozzolans as stated by (Shetty, 2009) are; Fly ash, Blast furnace slag, Silica Fume, Rice Husk ash, Guinea corn husk ash and Matakaoline.

2.14 Admixtures

An admixture “material ingredients use to enhance properties of freshly mixed mortar during setting, or hardened. Chemical admixture “an admixture in form of suspension or in water-soluble solid"(ASTM C125-15). ASTM C494 (2015) group the chemical admixtures in seven different forms.

ASTM C494 (2015) classified as,

i) Type A: The water-reducing which accelerates the admixtures setting time that results in early development of strength in concrete which is known as Water reducing admixture.

ii) Type B: These delay the setting of concrete known as the Retarding admixtures.

iii) Type C: The admixtures required to producing a given consistency got by the reduction of quantity of mixing water required to produce concrete is known as Accelerating admixture.

iv) Type D: The admixtures required producing a given consistency by 12% or more is achieved by the reduction of quantity of mixing water required to produced concrete is known as Water-reducing and retarding admixture.

v) Type E: The admixtures required to produce a given consistency and accelerating the setting time with early concrete strength development is got by the reduction of quantity of mixing water required to produce concrete is known as Water- reducing and accelerating admixture.

vi) Type F: Admixtures required producing a concrete of a given consistency and retards the setting of concrete by reducing quantity of water used for mixing is known as Water – reducing, high range.

vii) Type G: The admixtures required producing a given consistency by 12% or more and retards setting of concrete is achieved by the reduction of quantity of mixing water required to produced concrete is known as Water-reducing, high range, and retarding admixtures.

Adsorption depends on type of cement and the presence of amixture. Uchikawa *et al*., (1992) determined the degree of adsorption of β-naphthalene sulfonic acid condensate admixtures and lignosulfonate admixtures on eight different cements including blended cements with SCMs such as fly ash and slags. The adsorption of both admixtures varied with the type of cement. They also observed that admixtures were adsorbed preferentially to the interstitial phase and free lime.

Composition for adsorption occurs when several chemical admixtures are present in cement, admixtures with similar chemical structures. In this case, adsorption of admixtures is done preferentially with high anionic charge density will first, preventing low anionic charge density admixtures (Plank and winter, 2008).

2.15 Overview of Related Work

The properties of pozzolans as it act on mortar/concrete was studied by Nattapong *et al* (2010), The addition of agrochemicals to cement material will provide the required performance of stabilities and strength development of Mortar / concrete. The result showed that binder content with lower w/b percentages of admixtures will produce compressive strength that is higher 24.3N/mm^2 at older age.

The properties of SCC are enhanced by Metakaolin in first 14days of curing because the introduction of this material results in lower absorption with tensile strength and electrical resistivity improved. Lower percentage of the material is regarded as a suitable replacement.

Also, the research work carried out by Chopra and Siddique (2015) on the partial replacement with RHA that flows by its own weight, SCM's in SCC while noting the fresh flow, mechanical strength and durability properties at the strength testing days of 7, 28 and 56. When the study was carried at different percentage , 15% RHA influence the strength of both compressive and split tensile while the penetration Chloride and porosity values were subsequently reduced when RHA were added and formation of more C-S-H gel as the root cause of the increase in strength were shown in the XRD and SEM.

Archana *et al* (2013) investigates the strength of concrete containing locally available industrial and agricultural waste. The study undertaken to investigate the influence of partial replacement of cement with, Rice Husk Ash (RHA) and Sand with Stone Dust (SD).Its study shows that 10% RHA+20% FA and 30% SD in concrete gives maximum strength.

2.16 Summary of Research Gap

There are many research works on admixtures of RHA, MK and CCW. Some of the review literature, it is clear that rigorous efforts were made by different authorities to establish metakaolin as a SCMs in construction industries as a replacement of cement, especially in HPC, SCC and other kinds of special made Mortar and Concretes and in combination with metakaolin as partial replacement was also use to enhance their mechanical and durability properties.

Carbon dioxide (CO_2) emission, waste disposal and environmental degradation resulting from cement raw materials source has necessitate research in the utilization of RHA/CCW and MK as total replacement of cement, but of importance, introduction of metakaolin explored the use of other raw material that is not being utilised effectively when compared to its abundance of its massive exposures of kaolin.

The generation of agro-industrial wastes such as RHA, SHA, FA, SF and CCW in commercial quantity has prompted research in that direction. Particularly, reducing the CO_2 emission from clicker manufacturing process of cement, groundwater pollution resulting from waste disposal, environmental degradation as results of fills Rice Hush Ash and Calcium Carbide as raw materials source production. This research attempts to fill the knowledge gaps by carrying out extensive experimental study on RHA/MK/CCW as partial replacement of PC.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

Portland Cement (PC), Rice Husk Ash (RHA), Calcium Carbide Waste (CCW), High purity Metakaolin (MK), Sharp Sand and Water are the materials used.

3.1.1 Portland cement (PC)

Dangote (3X) (CEM 1 42.5 N) of cement has average specific gravity 3.17 was obtained from cement sellers in Minna, Niger state was used for the research work. The basic ingredient of mortar is the Portland cement widely used around the world for construction purposes that complied with BS EN 196- 1: 2016.

3.1.2 Sharp sand

Natural, clean and air-dried river sand of specific gravity 2.38 of BS 882-2 used as fine aggregate. Larger particles such as stone, and other harmful substances like leaves, broken glass, wooden particles and silts particles was removed.

3.1.3 Rice husk ash (RHA)

Rice husk ash (RHA) was heated for 2 hours at 700°C, this was conducted at National cereals research institute (NCRI) Laboratory Bida. The RHA produced through incineration conditions has high performance. The average specific gravity of Rice husk ash 2.25 showing a lighter material when compared with that of cement.

3.1.4 Calcium carbide waste (CCW)

Calcium Carbide Wastes (CCW) was obtained from a local Panel beating shops along Ketarin Gwari road, Minna, Niger State. The CCW was air-dried, calcined at 700^0C for 2 hours and sieved through 75µm sieve in the laboratory. The average specific gravity of CCW 2.33 compared to 3.16 of cement indicating that CCW is lighter than cement.

3.1.5 Metakaolin (MK)

MK was sourced from three locations because the materials are categorized into three types; high purity, medium purity, low purity. High purity MK was sourced from sedimentary kaolin deposit at Alkaleri, Bauchi-Gombe road, 74 kilometers east of Bauchi town. The medium purity MK was sourced in Kontagora, Niger state and the low purity MK was sourced in Kutigi, Niger state. The kaolin collected is crude kaolin. After collection, it was then open air dried, crushed, and sifted (sieved). Then it was taken for calcination at a temperature of 700°C for 2 hours in National Cereal Research Institute (NCRI) laboratory in Bida. The purities was determined by a Technologist in Geology Department Federal University of Technology Minna.

3.1.6 pH values

pH values was conducted at Departmental laboratory, Chemistry of the Federal University of Technology Minna with a pH meter Model Kent EIL 7045/46. The machine is calibrated with buffer Solution of 47 and 9. The machine was engaged and allowed to warm-up for at least 15 minutes to 20 minutes. The pH was connected to electrode with samples as solutions.

3.2 Detail Method of Mortar Mix

The binary blending of Supplementary Cementitious Material (SCM) is the method where a material is mixed with cement and ternary blending of Supplementary Cementitious Material (SCM) is the method where two SCMs are mixed with cement and quaternary blending of Supplementary Cementitious Material (SCM) is mixing three SCMs with cement all the three to improve performance of mortar. It involves having materials blended together in a binary, ternary and quaternary cement mortar mixed in a varied combination proportion of 5%, 10%, 15%, 20% 25%, to 30% replacement to cement. Total number of cube cast during this mix ratio is 756 cubes for compression test and 168 cylinders for split tensile.

3.2.1 Mixing, casting and curing water

Table 3.1 Binary, ternary and quaternary mix details of number of cube cast

Days	CONTROL	**95% CEM 5% RHA/CCW**	**90% CEM 10% RHA/CCW**	**85% CEM 15% RHA/CCW**	**80% CEM 20% RHA/CCW**	**75% CEM 25% RHA/CCW**	**70% CEM 30% RHA/CCW**
7	3	3	3	3	3	3	3
14	3	3	3	3	3	3	3
21	3	3	3	3	3	3	3
28	3	3	3	3	3	3	3
56	3	3	3	3	3	3	3
90	3	3	3	3	3	3	3
TOTAL	18	18	18	18	18	18	18
G/TOTAL					126		

756 cubes were cast and total numbers of 168 cylinders were cast for the experiment.

3.2.1.1 Mixing

Potable water from the borehole of School of Environmental Technology (SET) of the University (FUTM) was used for the preparation of the specimen for mixing and curing. The water is clean, odourless, colourless, tasteless qualities and free from organic materials and the size/amount of mix in this study is small since the volume of mortar is not much and the mechanical mixer is not needed because of its size.

Table3.2 Binary, ternary and quaternary of binders (Admixtures) blended cement mortar mix proportion (kg/m³)

MIX DESIGNATION	CEM	MK/CCW/RHA	SAND	WATER
Pc (Control)	250	-	750	125
95% Pc/(5%Binder) 1.6%MK/1.6%CCW/1.6%RHA	237.5	12.5	750	125
90% Pc/10% 3.33%MK/3.33%CCW/3.33%RHA	225	25	750	125
85% Pc/15% 5%MK/5%CCW/5%RHA	212.5	37.5	750	125
80% Pc/ 20% 6.66%MK/6.66%CCW/6.66%RHA	200	50	750	125
75% Pc/ 25% 8.33%MK/8.33%CCW/8.33%RHA	187.5	62.5	750	125
70% Pc/ 30% 10%MK/10%CCW/10%RHA	175	75	750	125

Binders; PC, RHA, CCW and Mk

Table 3.3 Mix proportion of specimen materials (kg/m³) of cylinders (Splitting Tensile) for binders cast.

TEST TYPE	CEM	MK/CCW/RHA	SAND	WATER
CONTROL MIX	100%	-	860	170
TEST (1) 5%	95%	5%		
1.6%MK/1.6%CCW/1.6%RHA	237.5	12.5	860	170
TEST (2)	90%	10%		
10%3.33%MK/3.33%CCW/3.33%RHA	225	25	860	170
Test (3)	85%	85%	860	170
15% 5%MK/5%CCW/5%RHA	212.5	37.5		

Binders; PC, RHA, CCW and MK

3.2.1.2 Casting

After the uniform mix was achieved, casting the mortar into an already oiled cube mould to reduce friction and assist removal of the hardened cubes was done immediately. Cast cube are then vibrated to remove voids, the surface of the mortar in the mould is levelled, smoothed and marked for the purpose of identifications.

3.2.1.3 Curing water

The mortar cubes was cured for 24 hours after being moulded, the blended mix to set and hardened after de-mould. After successful removal of the cubes from the moulds, then placed in water for varying hydration period of 7. 14. 21, 28, 56, and 90 days. Also specimen mortar cubes of the splitting tensile was cured for 24 hours in the cylindrical moulds for both control and the blended mix to set and hardened before de-moulding and applying same procedure as the cube mortar.

3.2.2 Preliminary tests

Preliminary test, fresh/ early age performance of mortar and mechanical properties was conducted in the Departmental Laboratory, Building of Federal University of Technology, Minna. Tests that were not obtainable in the department such as flow test for Mortar was conducted in Material Science Laboratory of Building Department Federal Polytechnic Bida. Also, the admixtures were tested for acidity or alkaline level at the chemistry department of the University.

3.2.3 Specific gravity

Is a ratio of a given volume of a material weight with equal weight of volume of water displaced as specified by BS EN 1097:2003. The specific gravity test on suitability of the materials was determined.

Equation 3.6 of specific gravity (G_s) of the materials was calculated using

$$\text{Specific gravity } G_s = \frac{w_2 - w_1}{(w_4 - w_1) - w_3 - w_2} \qquad 3.6$$

Also, moisture contents of fine aggregates were determined. These involved oven-drying known weights of the aggregate sample for 24 hours at a temperature above

105^{o}C. Their weights were taken after drying to determine the weight of water evaporated and that of the dry sample (Neville, 2012).

$$Moisture\ content = \frac{\boldsymbol{Initial\ wt\ of\ sample - Dry\ wt\ of\ sample}}{\boldsymbol{Dry\ wt\ of\ sample}} \qquad 3.7$$

3.2.4 Consistency

Percentage of water required for blended sample material of cement for normal consistency. Apparatus used are,Vicat Apparatus , Plunger of 10mm diameter, Trowel, Weighing balance, Weight box and measuring cylinder as outlined in the standard BS EN 196-3:2016. All materials were tested for consistency. The water required of paste to actualised suitable standard consistence as advocated (Neville, 2012)

3.2.4 Setting Time

Using the Vicat apparatus that has penetrating tools, use to measure initial and final setting time. Initial set is expressed as the time elapsed when mixing water was added to the cement or proportion and for final was determined by a 1mm square needle fitted with a metal attachment hollowed out so as to leave a circular cutting edge 5mm in diameter and set 0.5 behind the tip of the needle. Final set time was said to have taken place when the needle, gently lowered to the place, an impression is made on it without any impression of the circular cutting edge, following procedures as outlined in the standard (BS EN 196-3:2005). As water is added to the cement during mixing and where the proportion is required by the relevant B.S not exceeding ten hours for rapid-hardening, low heat and blast-furnace Portland cement (Neville, 2006). Materials were mixed proportionately so as to determine their setting times.

3.3 Method to Actualise the Physiochemical Properties

i) The first objective was achieved by carrying out the physiochemical properties of MK, CCW and RHA, this was achieved by carrying out test of specific gravity, the chemical analysis with the Bulk density and other preliminary tests in the Department and samples of constituent materials were well packaged and sent to a standard laboratory in Ewekoro Factory, Lafarge Cement Lagos where the Oxide composition were obtained.

ii) In order to establish and achieve the appropriate mix proportions of the constituent materials for the blended cement mortar, appropriate combinations of the materials were carried out with the ratio of 1:3 for Cement/ Sand and water 0.5 employed to obtain mortars at various mixes.

iii) The blended binder combination of the binary, ternary and quaternary mix were prepared in comparison with a control of PC grade of (CEM 42.5N) at 1:3 cement /sand mix for strength test.

iv) Determination of the compressive strength of specimens was carried out at 7,14,28,56 and 90 days of curing at surface dry condition as stated in European Standard BS EN 12390-3:2002. Each category of specimens was tested and the mean compressive strength of specimen was considered as the compressive strength of the specified category using equation (4.1).

$$FC = {}^{P}/_{A}$$

(4.1)

Where P = Crushing Load, A = Area of cross-section for all the mix.

The tensile splitting determined with Universal Testing machine, model number No ET 882816 A and having capacity of 200 KN. Mortar cubes size 50 mm at curing at 7, 14,21,28,56 to 90 days were tested and average result of three replicates were recorded while selected cylindrical size 45mm for tensile tests were carried out at curing days of 7, 14, 21 and 28 days.

The Los Angeles Abrasion test- LAA was conducted in the Engineering Laboratory Federal Polytechnic, Bida, Niger state. All test results were recorded, analysed and discussed accordingly.

3.4 Mortar

142.5N grade Portland cement having specific gravity 3.40 the SCMs of Rice husk ash with specific gravity 2.25, Metakaolin with specific gravity 2.50 and Calcium carbide waste with specific gravity 2.33 was employed for the blended mortar mix in all the mixes prepared.

The categories of the blended quaternary mix are the Binary, Ternary and the Quaternary mixes.

3.4.1 Mortar preparations

This involved the weighing out the appropriate constituent materials and ensuring thoroughly mixed in stages; Stage 1. Blended cements prepared by blending of R H A / Pc with Sand at 5%, 10%, 15%, 20%25% and 30% with CEMI42.5N cement, and water/cement ratio of 0.5 as binary. Stage2. Preparation of cement pastes of RHA/CCW-blended cements and sand at percentages of 5%, 10%, 15%, 20%25% and 30% with CEMI42.5N cement as ternary and the last cement pastes with all the three blended RHA/CCW/MK with sand at percentages of 5%, 10%, 15%, 20%25%

and 30% with CEMI42.5N cement as quaternary, The ratio of 1:3 blended cement /sand and water.

3.4.2 Compressive strength of mortar

In determining strength cube of 50mm, mortar specimen that were produced and cured at different curing age. The mortar cube due for the compressive strength test are taken to the departmental Digital Universal Machine (DUTM-20) to crush in order to assess the strength development after being weighed on a top balance.

Determination of compressive strength was carried out on 7,14,28,56 and 90 days of curing, surface dry condition (European Standard BS EN 196-1:2016). The specimen mean compressive strength of the specimen was considered compressive strength of the specified category.

The, tensile splitting Machine with model number No ET 882816 A was used and having capacity of 200 KN. Mortar cubes size 50mm curing at 7, 14,21,28,56 and 90 days were tested and the result of average three samples were recorded while selected cylindrical size 45mm for tensile tests were determined at curing ages of7, 14, 21 and 28.

3.4.3 Tensile strength of mortar

The determination of strength of Splitting Tensile of 45mm by 90mm mortar specimen cylinder that were produced and cured at curing ages in the laboratory. The cylinder mortar specimen due for the splitting tensile strength determination was picked from the curing tank and taken to the same Departmental Digital Universal Testing Machine (DUTM-20) to crush to assess the development of splitting tensile strength after being weighed on a top balance.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Physical and chemical properties of constituent materials

4.1.1 Physical properties

Physical and chemical properties of PC, CCW and MK in Table 4.1 respectively. The results of the average specific gravity of RHA, CCW and MK showed that CCW with a specific gravity of 2.19 is very light and fine compared with other constituents like in PC of specific gravity 3.15. RHA and MK are both lighter than PC with a specific gravity of 3.15. The specific surface area of CCW, RHA and MK are higher than that of PC which means that they are much finer than PC and thus occupy more space than PC. Generally, the specific gravity of mineral admixture is less than that of PC therefore more volume is obtained.

4.1.2 Chemical properties

From Table 4.1, SiO_2 + Al_2O_3 +Fe_2O_3 (SAF) that exceeds 70 % and thus met ASTM C 618-00 Class F and ASTM C Class C provisions for pozzolanic materials. Amount of SiO_2 and Al_2O_3 in RHA and MK depicts their reactive capability with the primary hydrate of cement. It is interesting to note that CCW with less SiO_2 and Al_2O_3 has the greatest content of CaO 95.69 % ensuring that the Ca/Si ratio is maintained (kept at not less than pH 12) for reaction to continue. Except for CCW with an LOI of 21 %, the values for MK and RHA are within specifications.

Table 4.1: Physical and chemical properties of materials

Properties	Material (Wt. %)			
	PC	RHA	CCW	MK
Physical				
Sp. Gt (g/cm^3)	3.15	2.25	2.19	2.52
B. Fineness (cm^2/g)	3045			7483
Loss on Ignition	2.98	2.93	21.71	1.22
Chemical				
CaO	64.19	1.11	65.59	0.14
SiO_2	19.57	86.75	8.69	57.53
Al_2O_3	5.47	0.68	1.10	27.12
Fe_2O_3	3.24	0.94	0.30	2.25
MgO	2.01	0.92	0.05	0.10
M_2O_5	1.25	0.33	0.03	0.08
Na_2O	0.26	0.12	0.00	0.00
K_2O	0.45	2.37	0.10	0.31
SO_2	2.74	0.03	0.10	0.00
P_2O_5	-	3.53	0.16	0.07
TiO_2	-	0.31	0.12	1.73

4.1.3 Particle size distribution

PSD curve in Figure 4.1for sand was observed to conform to the fine aggregate prescribed in BS 812- 103. 1:1985(2000). It has Fineness modulus (FM) of 2.46. PSD determined how workable Mortar/concrete is, as the Sand requires water more than coarse aggregate having same size (Neville 1981). From the figure, D_{30} is the particle size diameter passing 30% and D_{10} size diameter passing10%. D_{60} is the diameter passing 60% particle. To determine coefficient of curvature (C) and the coefficient of uniformity (C_u), Equations (3.3) and (3.4) were used.

$$\text{Uniformity Coefficient (Cu)} = \frac{D_{60}}{D_{10}} \quad (3.3)$$

$$\text{Coefficient of Curvature (Cc)} = \frac{(D_{30})^2}{D_{10} \times D_{60}} \quad (3.4)$$

Well graded requirements were thereby presented as: Cu ≥ 4 for coarse aggregate. Cu ≥ 6 for fine agg. and Cc = 1 to 3 for all type of soil. While Shetty (2005) presented limits for Fineness Modulus (F.M.) of fine aggregate for concrete works. Fine Sand, Medium and Coarse Sand F.M are F.M.: 2.2 – 2.6, F.M.: 2.6 – 2.9 F.M.: 2.9 – 3.2 but the F.M. > 3.2 of sand will be unsuitable for good mortar.

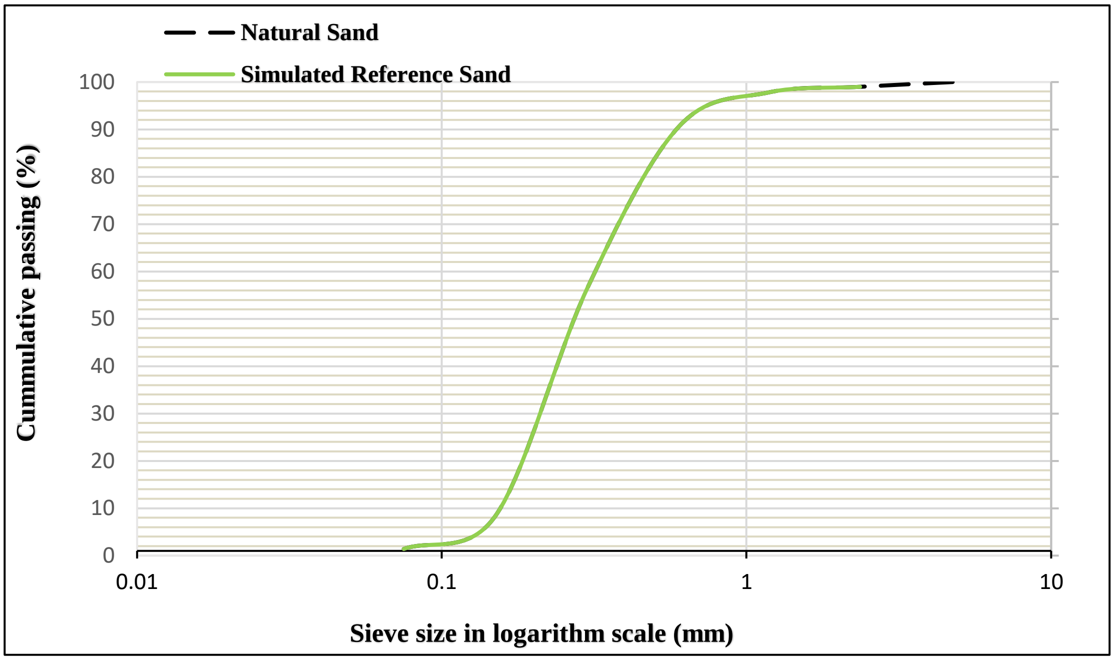

Figure 4.1: Particle size distribution of sand

The combined fine aggregate used for the study has a FM of 2.46, fine aggregate (Shetty, 2009).

4.2 Standard Consistency and Setting Times

4.2.1 Standard consistency of PC,-CCW,-RHA,-MK

Standard consistency of PC-CCW-RHA-MK at different percentage for quaternary replacement levels is shown in Figure 4.2. The work of Nochaiya, *et al* (2010) on OPC-FA-SF at 20FA 10SF and 30FA 10 SF replacement of PC showed that as there is a change in the increase level of replacement, also need for water to increase as a result of the high degree of fineness.

From this study, it was found that as CCW, RHA and MK were added as replacement of PC to make quaternary binder, the water and the replacement level of increases one increased. These behaviors results due to high fineness of RHA and MK compared to that of PC. Their specific surface value is much higher than that of PC (Table 4.1).

At 5 % replacement level of PC with CCW-RHA-MK binder, its water demand increased by 9 %, and 21 %, 39 % at 10 % and 20 % replacement levels, when compared to PC value (Figure 4.1). For the binary binders (PC-CCW, PC-RHA, PC-MK), consistency is same with that of PC (control) value (Table 4.3) at 5 % and 10 % replacement levels but increased by about 5 % at 20 % replacement level except for PC-CCW binder whose consistency is same with that of PC at all replacement levels.

CCW does not demand much water due to its low pozzolan city compared to that of RHA and MK. The increase in consistency for the quaternary binder is fruitful as it facilitates more reactions between constituents and the primary hydrate of PC as it consumes Ca(OH₂) and produces other components that are resistible to acid and Sulphate attack.

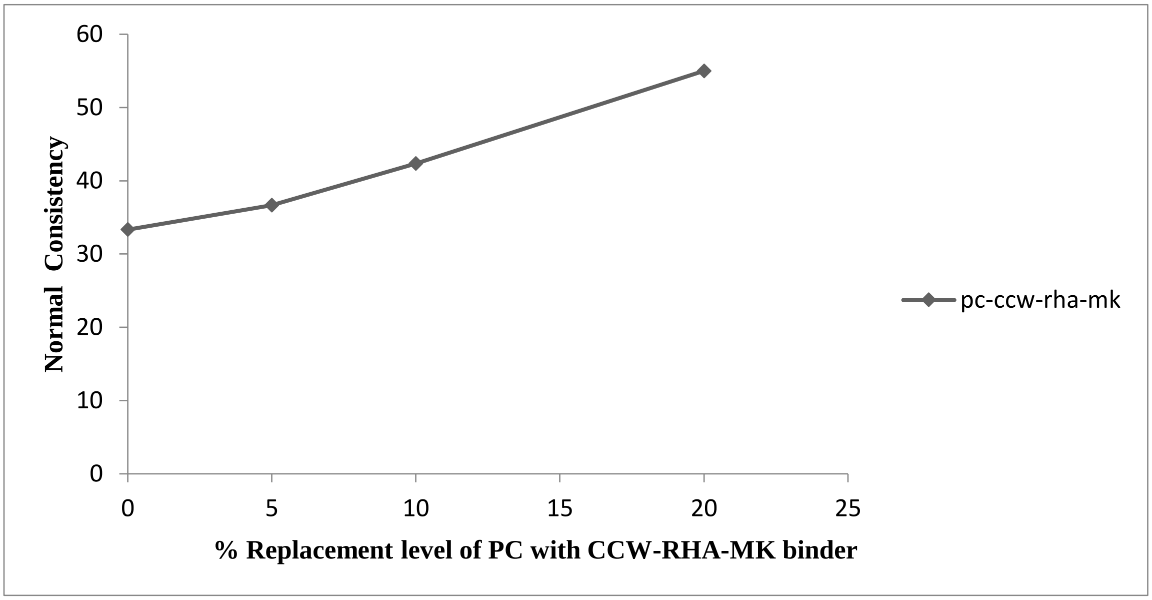

Figure 4.2: Consistency of PC-CCW-RHA-MK

4.2.2 Setting time

4.2.2.1 Initial and final setting times of PC-CCW-RHA-MK binders

For Quaternary binders containing PC, CCW, RHA and MK, the setting times, initial (IS) and final (FS) are shown in Figures 4.3 and 4.4. It was observed that the admixtures prolonged the hardening process (setting times) of the quaternary binders when compared with control value.

The observed retardation in setting times is mainly due to the effect of lower cement content. It was also observed that the setting times for binary and ternary binders increased as percentage replacement level of PC with the admixtures increased.

This showed that the admixtures used in this study retarded the setting times of binary, ternary and quaternary binders Berg and Kukko, (1991) ; Rao, G.A.,(2003).

If Quaternary binder (PC-CCW-RHA-MK) is compared with ternary binder (PC-CCW-MK), (Table 4.3 and Figure 4.3) at 5 % replacement level, IS decreased by 27 %, at 10 % replacement level, increased by 8 % and then decreased again at 20 % replacement level by 24 %.

Table 4.2: Consistency, initial and final setting time results

S/NO	Mix ID	Materials m) PC	RHA	CCW	MK	Std Consistency	Mins) IS	(Mins) FS
	100 % PC	300				33.33	45	165
Group 1b, BM	95PC- 5MK	285			15	33.00	80	295
	90PC-10MK	270			30	34.00	110	295
	80PC-20MK	240			60	34.33	85	295
Group 2, BM	95PC-5RHA	285	15			32.67	85	225
	90PC-10RHA	270	30			33.67	85	250
	80PC-20RHA	240	60			35.00	75	325
Group 3, BM	95PC-5CCW	285		15		31.67	105	280
	90PC-10CCW	270		30		32.00	60	250
	80PC-20CCW	240		60		32.33	70	255
Group 4, TM	95PC-5CCW-5MK	285		15	15	33.00	123	265
	90PC-10CCW-10MK	270		15	15	34.67	115	275
	80PC-20CCW-20MK	240		30	30	39.00	145	305
Group 5, QM	95PC-5CCW-5RHA-5MK	255	15	15	15	36.67	90	250
	90PC-10CCW-10RHA-10MK	210	30	30	30	42.33	125	270
	80PC-20CCW-20RHA-20MK	120	60	60	60	55.00	110	301

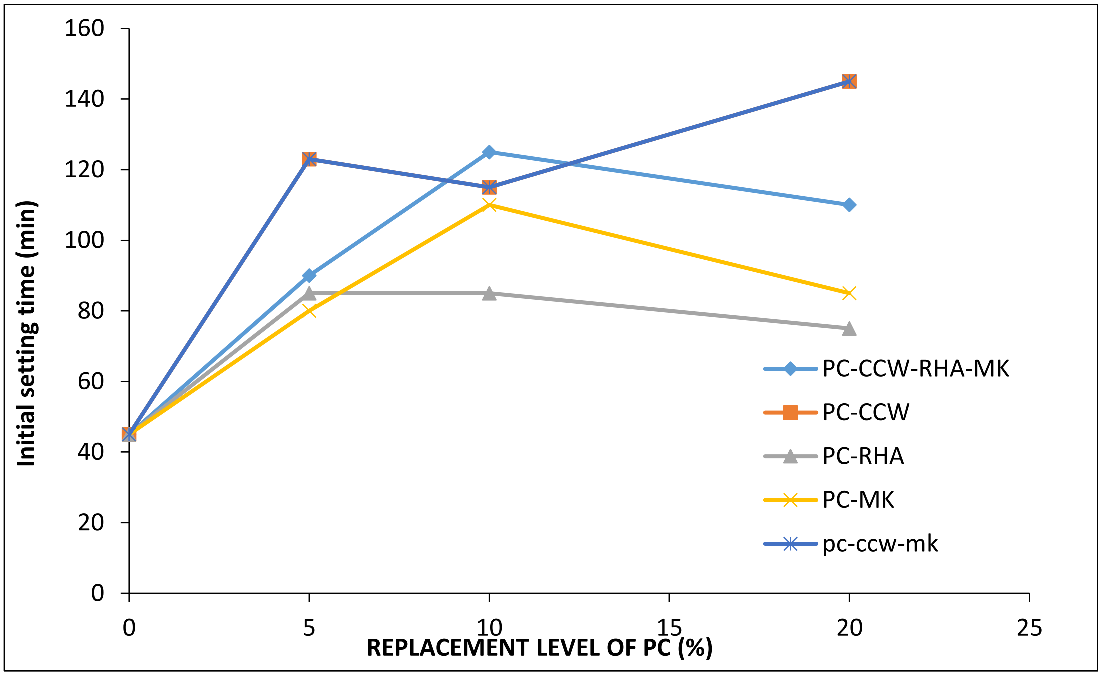

Figure 4.3: Initial setting times of binders

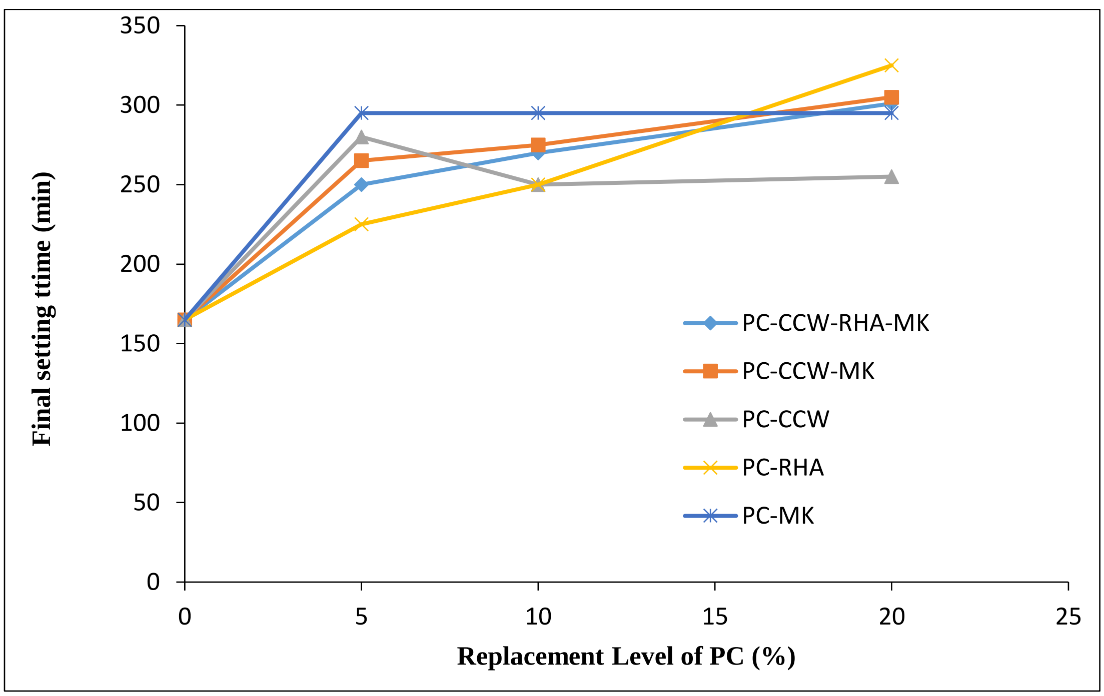

Figure 4.4: Final setting times of binders

This effect of RHA on IS depict disorder; but on FS, at 5 % level of replacement, it decreased by 6 %; at 10 % level by 2 % and by 1 % at 20 % replacement level which showed that RHA progressively decreased FS of binders studied. This is attributed to pozzolanic reaction of RHA at later stage. Similarly, when PC-CCW-MK is compared with PC-CCW, at 5 % level of replacement, decreased FS by 5 %, at 10 % level of replacement decreased by 1 % and then increased by 26 %. The effect of MK decreased FS by 5 % and 1 % at 5 and 10 % replacement levels but increased it by 26 % at 20 % replacement level. Furthermore, comparison between PC-CCW-MK with PC-MK revealed that CCW prolonged IS by 35 %, 4 % and 41 % but decreased FS by 10 %,7 % and 3 % at 5, 10 and 20 % replacement levels respectively. Theses test results showed that as % replacement level increased IS increased and then decreased FS which implies that reaction becomes faster at later stage of hydration.

4.3 pH value of Constituent Materials

pH values of Cement (PC) = 12.03%, Rice Husk Ash (RHA) = 12.73%, Metakaolin (MK) = 9.77% and Calcium carbide waste (CCW) = 7.98%

The result are alkaline with Rice husk ash having 12.73% having the highest, followed by Cement with 12.03% while Metakaolin has 9.77% alkaline meaning the reactions were basic reactions that took place and the constituent materials reacted with each other. (Fereshte *et al*., 2015).

Table 4.3: pH value of constituent materials

Materials	pH value
CEM	12.03
RHA	12.73
CCW	7.98
MK	9.77

Moisture content for fine aggregates is between 0 to 10% and according to ASTM618-05 moisture content for binders should be between 0 to 3% which presents moisture test result of fine aggregate and binders conformed to standard (Table 4.3).

Table 4.4: Sample materials for moisture content

Materials	Wt of material before oven dry (kg)	Weight of material after oven dry (kg)	Moisture content (%)
CEM 1	0.5	0.499	0.2
RHA	0.5	0.499	0.4
CCW	0.5	0.488	2.4
MK	0.5	0.499	0.0
Sand	0.1	1.0	0.0

4.4 Flow Table Test

Result of flow test values of fresh mortar for control mix (PC) and mixes with varying proportions of blended cements is shown in figure 4.5. Flow values are influence by a decrease in flow as the replacement level increase. The fresh mortar mixtures with blended cements have lower Flow values when compared with reference value. This is not unconnected with the influence of physical properties of

SCMs used for the study such as specific gravity, specific surface and particle size on consistency of mixes. (Mlinarik, (2016). The flow value of the fresh mortar for quaternary mix is close to that for the reference value, compared with binary and ternary values. This result shows that, studying the fresh properties 9such as consistence) is, therefore important in practical applications where SCMs are used in cement mortars.

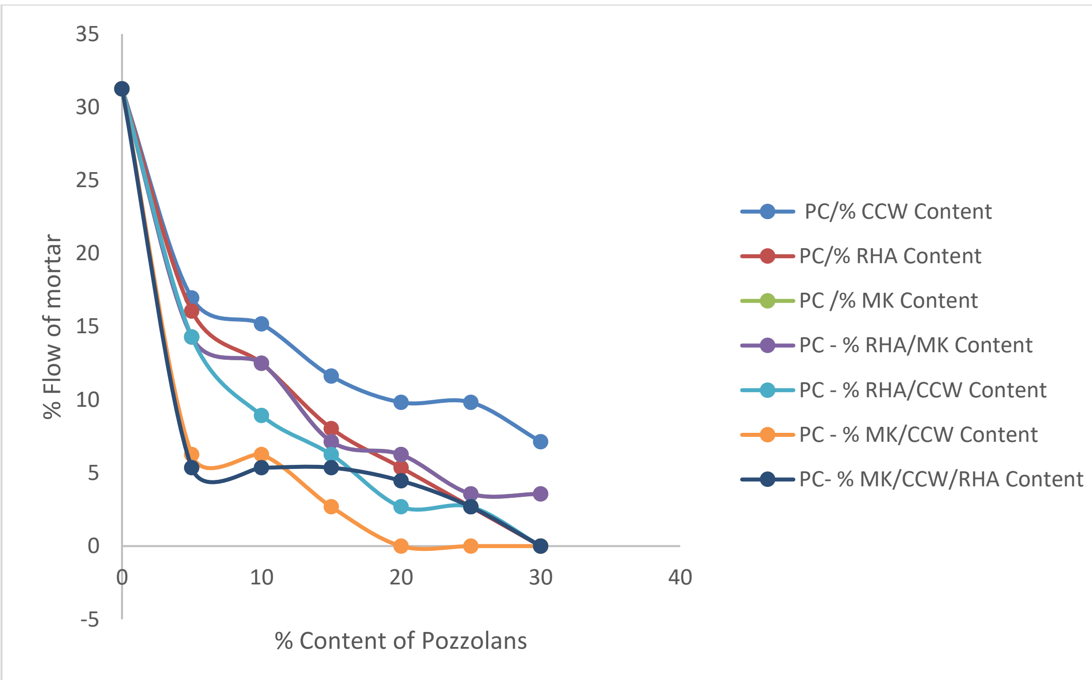

Figure 4.5: % flow values verses % content of Mixes

4.5 Compressive Strength of Mortar

The mechanical properties for the aforementioned ages for binary, ternary and quaternary mixes of control and blended mixes at varying contents is evaluated and tabulated in Figures 4.6 to 4.11.

Results of values for the compressive strength of binary mixtures at all curing ages are lower compared with control (PC) values. This is because of the reduction in PC content, leading to reduction of C_2S content responsible for early hydration. However, this trend improved for ternary mixes and the compressive strength values improved when compared with control values due to the synergy effect between the SCMs studied. Furthermore, for the quaternary mix, the compressive strength values further improved to level and even surpassed that of control values due to more synergistic effects between CEM/RHA/CCW/MK and in addition the increase of pozzolanic reactions and Ca $(OH)_2$, the by-product of PC hydration at later curing ages. From figure 4.11, Quaternary mix (CEM85/RHA5/CCW5/MK5) has the highest performance which evened and surpassed control values at 28 and beyond curing ages compared with all other mixes. This indicated optimum synergetic effect among the SCMs studied. This is reflected in Ternary mixes (CEM85/RHA7.5/MK7.5) and CEM80/RHA 10/CCW10) respectively.

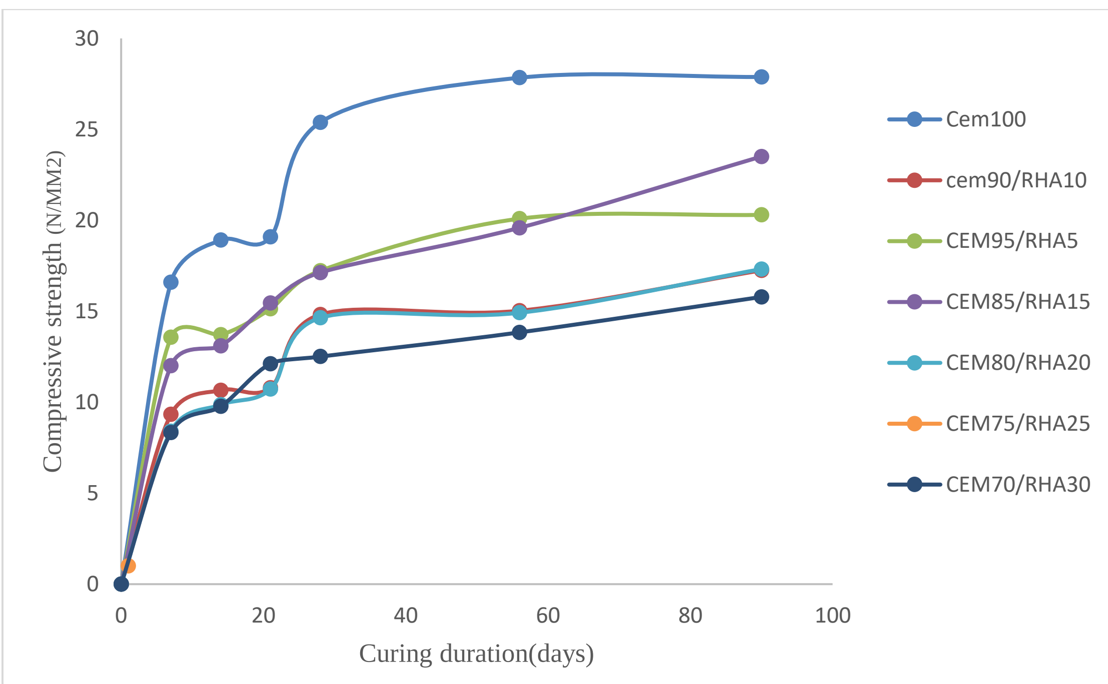

Figure 4.6: Compressive strength of binary mix (CEM/RHA)

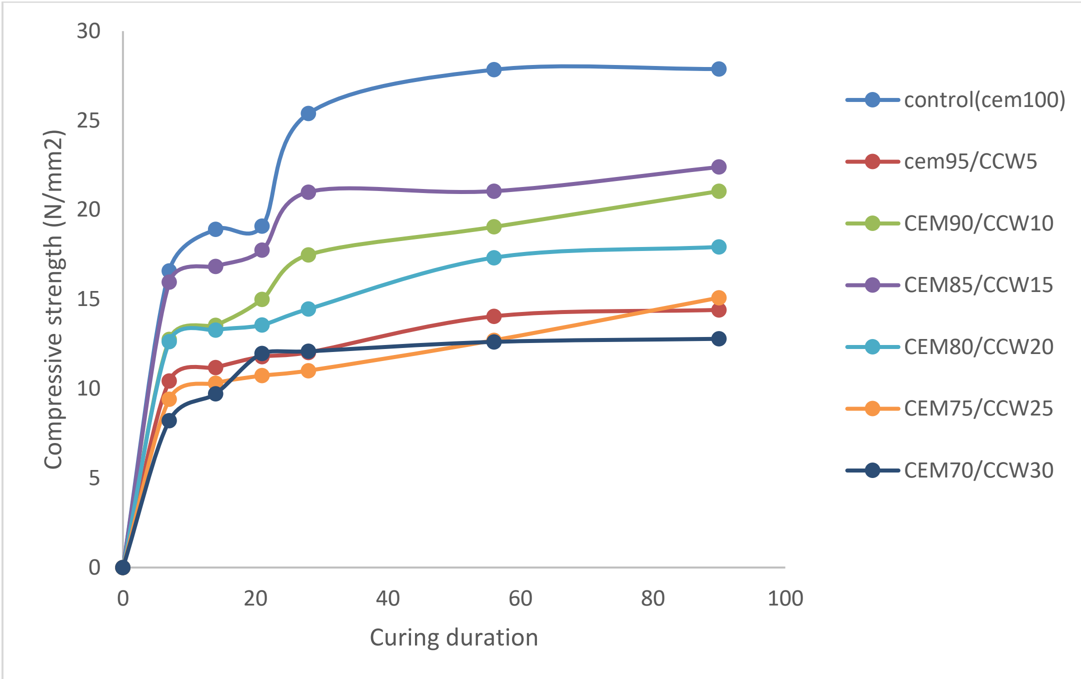

Figure 4.7: Compressive strength of binary mix (CEM/CCW)

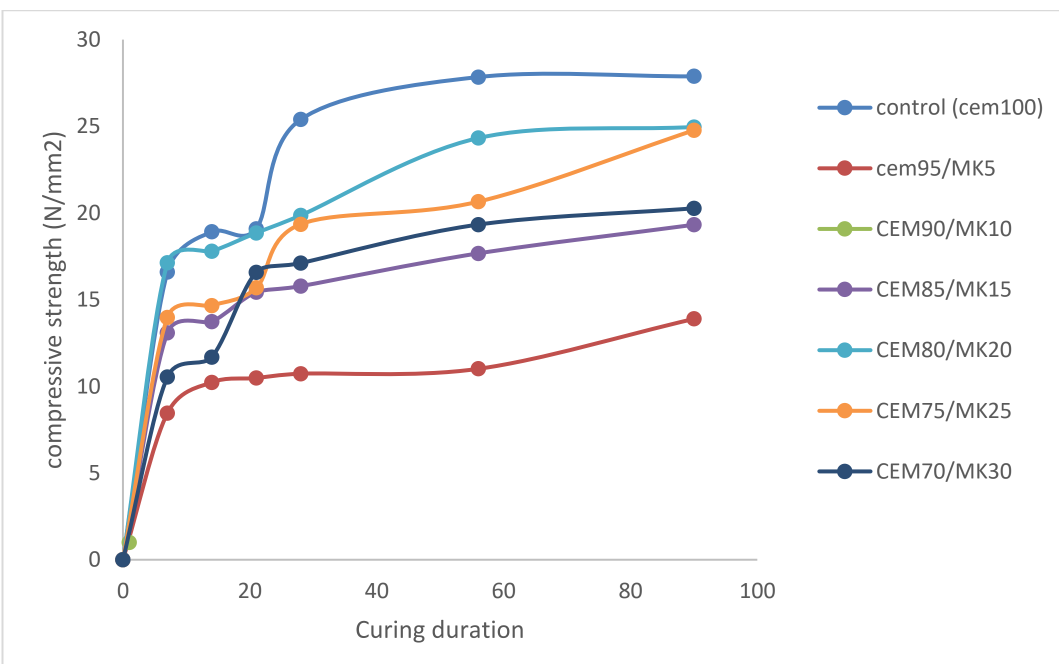

Figure 4.8: Compressive strength of binary mix (CEM/MK)

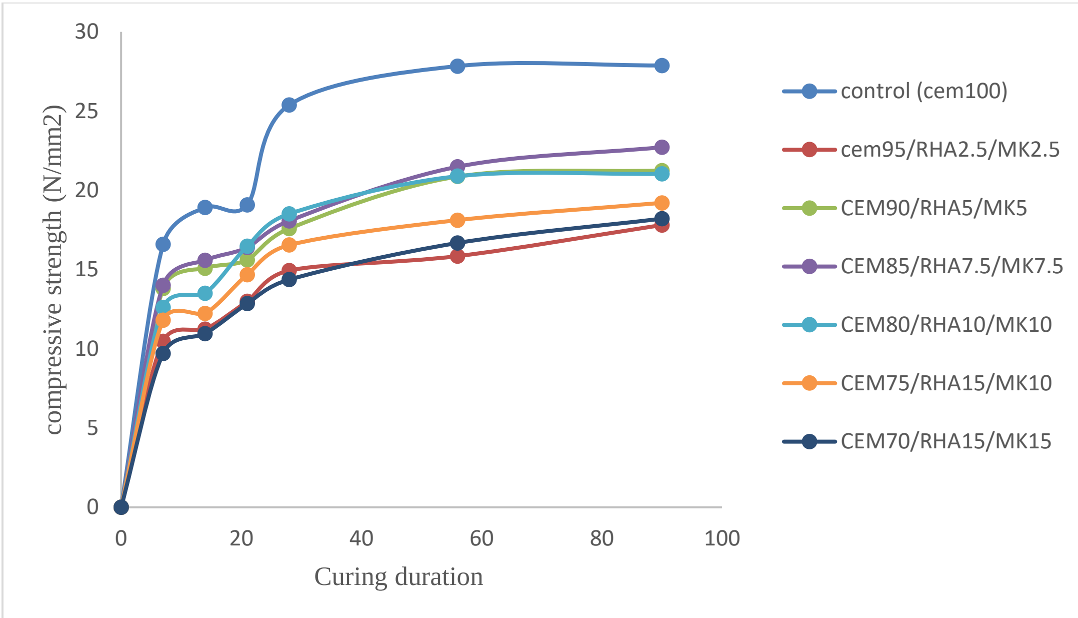

Figure 4.9: Compressive strength of ternary mix (CEM/RHA/MK)

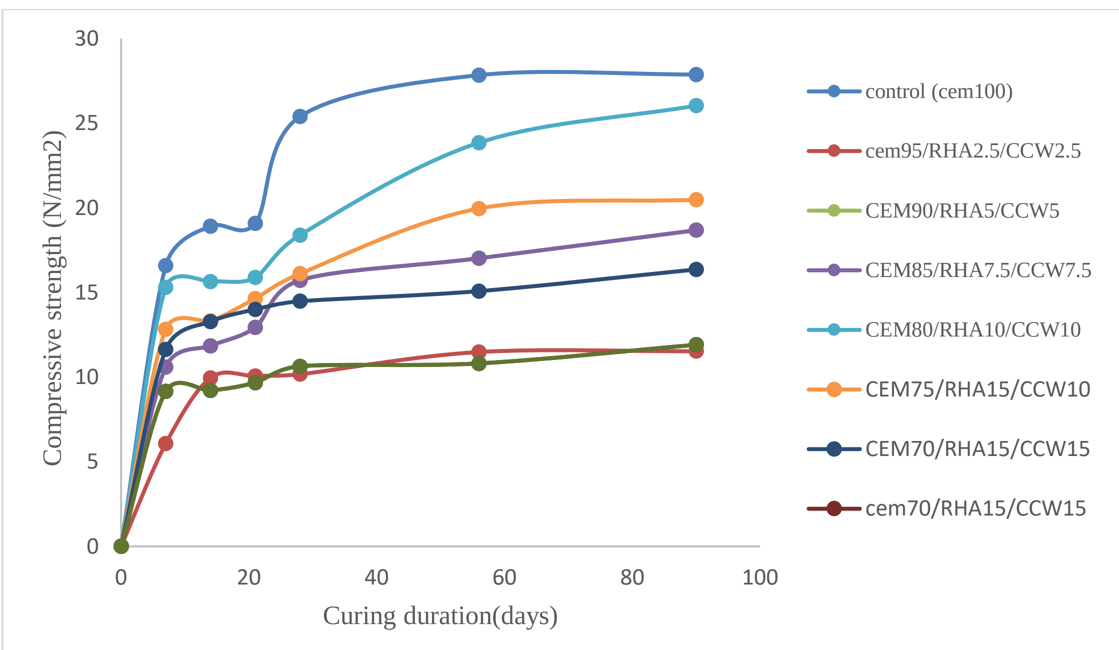

Figure 4.10: Compressive strength of ternary mix (CEM/RHA/CCW)

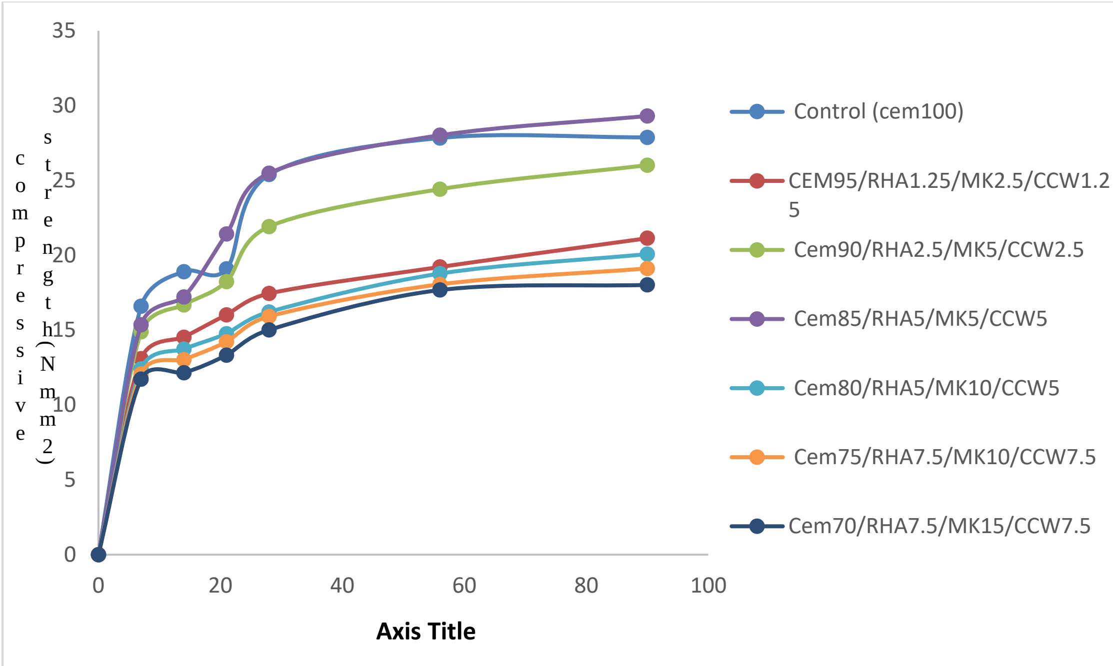

Figure 4.11: Compressive strength of quaternary mix (CEM/RHA/CCW/MK)

4.6 Tensile Strength of Splitting Mortar Cube

Figure 4.12 to 4.16 shows the results of splitting tensile strength tests conducted on cylindrical mortar specimens for PC and PC blended mixes. Tensile strength increase with increase in curing ages as expected. This is because splitting tensile strength is related to compressive strength. For control specimens, there is an increase of 31 % in 28 days from 7 days value and 3% in 90 days from 28 days value. Maximum strength increase of 3.25 N/mm^2 was obtained for Quaternary Mix (CEM85/5RHA/5MK/5CCW) for 90 days as compared with 3.21N/mm^2 for control mix. This shows that split tensile strength of PC is also influenced by synergetic effect of Quaternary Mix.

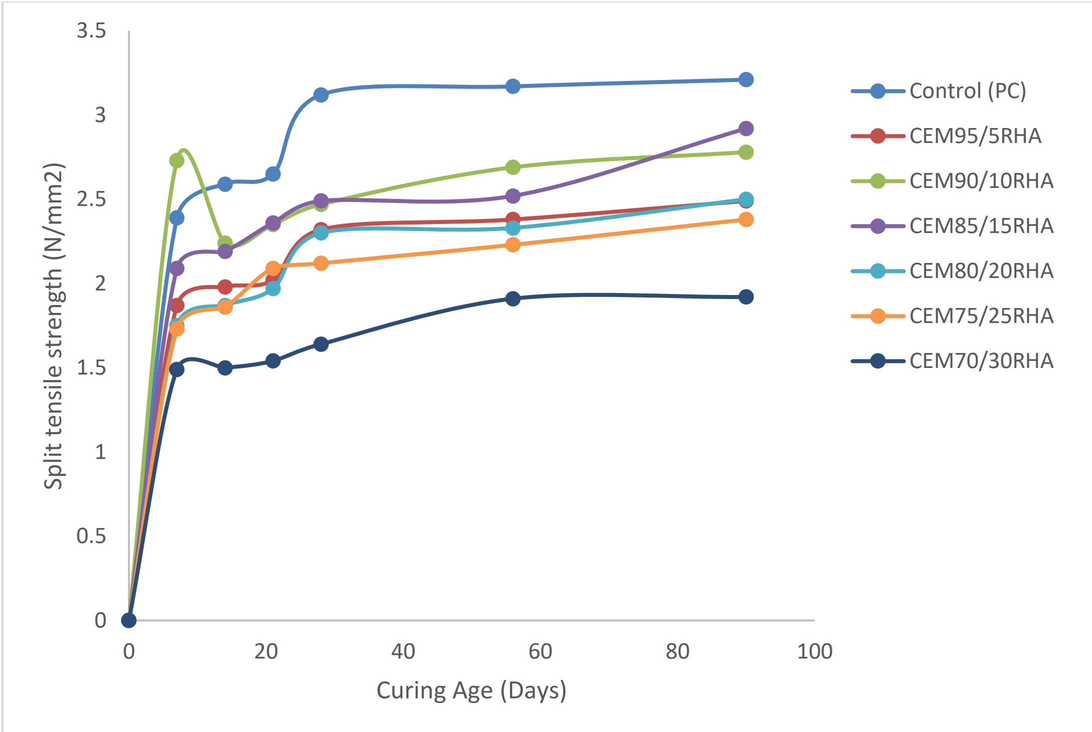

Figure 4.12: Tensile strength of binary (CEM/RHA) Mix

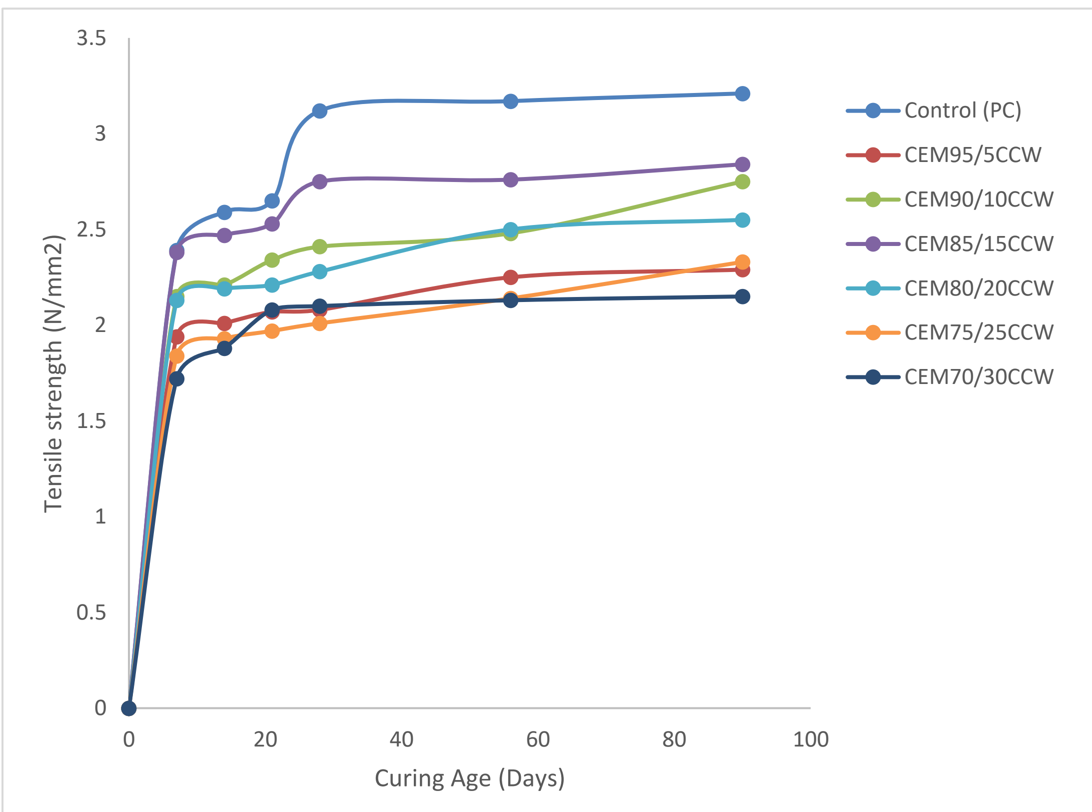

Figure 4.13: Tensile strength of binary (CEM/CCW) Mix

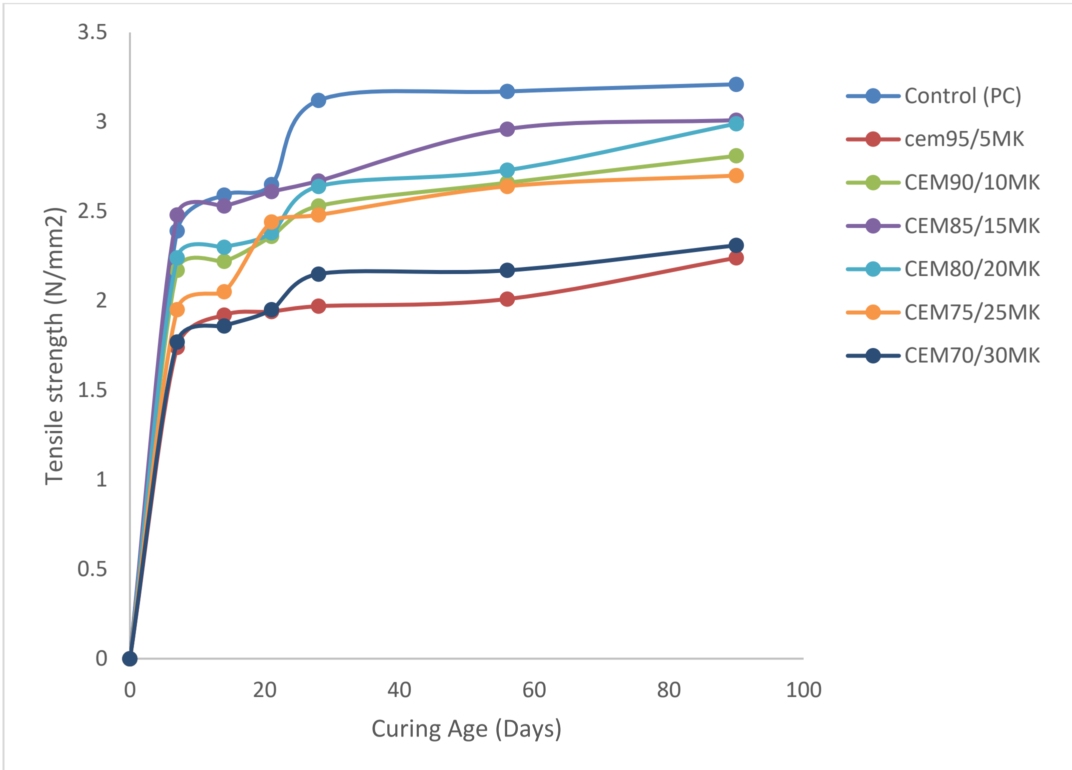

Figure 4.14: Tensile strength of binary (CEM/MK) Mix

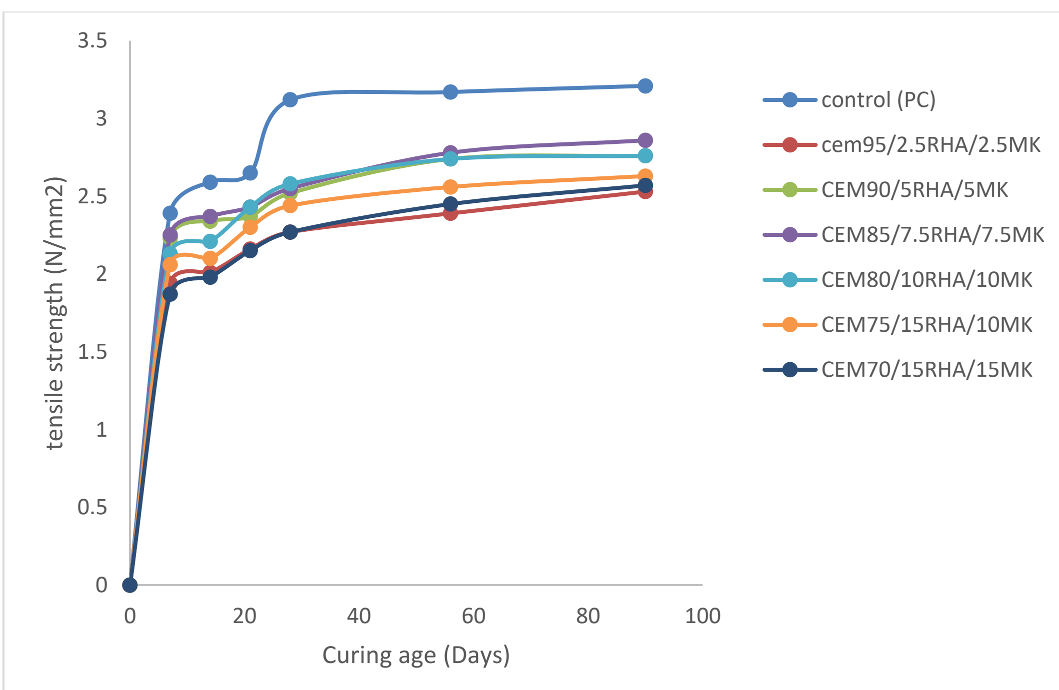

Figure 4.15: Tensile strength of ternary mix

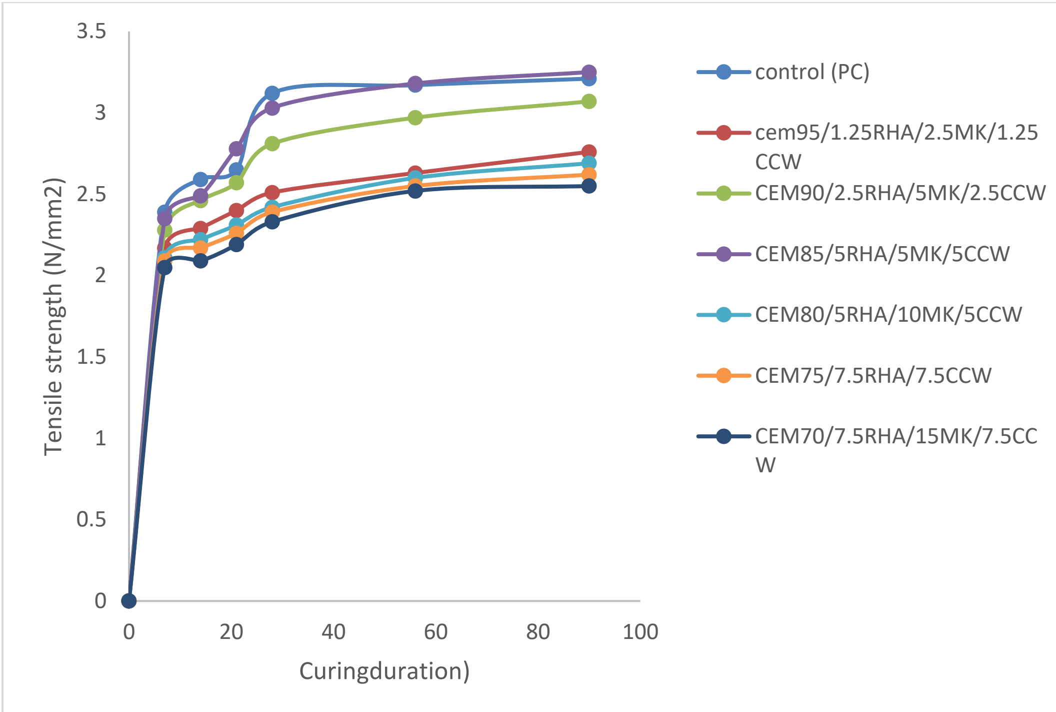

Figure 4.16: Tensile strength of quaternary mix

Samples of blended cement that their compressive strength properties are high were prepared for split tensile to test the extent and size of cracking in mortar.

The blended materials were mix specimen in binary, ternary and quaternary mixes with high compressive strength where the effects of cracking

4.7 XRF Analysis for Oxide Composition of Cementitious Materials

The result value of XRF conducted at Lafarge Cement in Ewekoro shows 87.90% oxide composition of cementious material indicating the constituent materials are very reactive. The major component of RHA sample is silicon oxide of having mass contents of 83.79%. Main oxide SiO_2 is a reaction that happens in pozzolans and is also responsible of the secondary hydration in mortar/concrete with pozzolanic materials. Other key oxides percentages such as Alumina ((Al_2O_3) and Iron oxide

(Fe_2O_3) are present reasonably to buttress the strength development in the reaction. From the table above, it is revealed that Rice Husk ash a Class N Pozzolan sum-up to SiO_2+Al_2O_3+Fe_2O_3 > 70%, where SO_3 is as low as 2% and LOI < 10%. The Calcium carbide waste in the material contain 69.64% CaO, however, it is observed that calcium carbide waste are high when compared to that of cement and other constituent materials in the blended mix. The essence of the pozzolan materials apart from improving the properties also enhances mortar/concrete durability; limit the cement in production of mortar. Metakaolin which is introduced as substitute of cement paste to limit cement use in mortar has SiO_2 +Al_2O_3 + Fe_2O_3 which is 86.90% in the material constituent will be an added advantage in the reaction of the blended paste.

The relatively high LOI of Metakaolin and Calcium carbide waste will not affect the reactions of the quaternary because they are both lower by 10% was noted to be below the specified 10% maximum, an indication that some heat treatment might be required for more effective performance of the material.

Table 4.5: XRF results for oxide materials .Composition of cementitious materials.

Properties	Wt. % of Materials			
	PC	RHA	CCW	MK
Physical				
Specific Gravity (g/cm^3)	3.6	2.25	2.19	2.52
Loss on Ignition (LOI)	2.98	2.93	21.71	1.22
Chemical				
CaO	64.19	1.11	65.59	0.14
SiO_2	19.57	86.75	8.69	57.53
Al_2O_3	5.47	0.68	1.10	27.12
Fe_2O_3	3.24	0.94	0.30	2.25
MgO	2.01	0.92	0.05	0.10
M_2O_3	1.25	0.33	0.03	0.08
Na_2O	0.26	0.12	0.00	0.00
K_2O	0.45	2.37	0.10	0.31
SO_2	2.74	0.03	0.10	0.00
P_2O_3		3.53	0.16	0.07
TiO_2		0.31	0.12	1.73

4.8 Fresh Properties of Blended Cement with Binder

Results conducted for the soundness in the Table 4.30, 4.31 and 4.32 revealed all the binder combinations of RHA/CCW, RHA/MK and MK/CCW with10 mm expansion (max.) specified EN 197-1:2000 of BS which provides us with the behaviours of the pozzolans in terms of their relativities as the surface area is a factor that governs water required by paste to a much greater extent than any other admixture.

Table 1.6: Fresh properties of blended cement with RHA and CCW

Specimen	Consistency RHA/CCW		Soundness Expansion (mm)[0.04 in] RHA/CCW
	Water Demand (%)	Penetration (mm)	
CEM 1	100	5.0	0.0
BINDER 95%/5%	98	6.0	0.0
BINDER 90%/10%	107	6.0	0.5
BINDER 85%/15%	111	5.0	0.5
BINDER 80%/20%	123	7.0	1.0
BINDER 75%/25%	129	7.0	1.0
BINDER 70%/30%	133	7.0	1.0

BINDER; RHA/CCW

Table 2.7: Fresh properties of blended cement with MK and CCW

Specimen	Consistency MK/CCW		Soundness Expansion (mm)
	Water Demand (%)	Penetration (mm)	MK/CCW
CEM 1		5.0	0.0
BINDER 95%/5%	105	5.0	0.0
BINDER 90%/10%	113	5.0	0.5
BINDER 85%/15%	121	7.0	0.5
BINDER 80%/20%	130	7.0	1.0
BINDER 75%/25%	136	7.0	1.0
BINDER 70%/30%	141	7.0	1.0

BINDER; MK/CCW

Table 3.8: Fresh properties of blended cement with MK and RHA

Specimen	Consistency MK/RHA		Soundness Expansion (mm)
	Water Demand (%)	Penetration (mm)	MK/RHA
CEM 1	100	5.0	0.0
BINDER 95%/5%	105	5.0	0.0
BINDER 90%/10%	111	6.0	0.5
BINDER 85%/15%	118	5.0	0.5
BINDER 80%/20%	131	7.0	1.0
BINDER 75%/25%	139	7.0	1.0
BINDER 70%/30%	140	7.0	1.0

BINDER; MK/RHA

Table 4.9: Fresh properties of blended cement with MK/RHA/CCW

Specimen	Consistency MK/RHA/CCW Water Demand (%)	Penetration (mm)	Soundness Expansion (mm)[0.04 in] MK/RHACCW
CEM 1	100	5.0	0.0
BINDER 95%/5%	110	6.0	0.0
BINDER 90%/10%	113	6.0	0.5
BINDER 85%/15%	140	6.0	0.5
BINDER 80%/20%	146	7.0	1.0
BINDER 75%/25%	153	7.0	1.0
BINDER 70%/30%	165	7.0	1.0

BINDER; MK/RHA/CCW

4.9 Abrasion Resistance Test Result

Results of figures 4.17 - 4.22 of the abrasion resistance for control and cement blended mortar mixes cured and tested for strengths at 28, 56 and 90 days the average durability values of 86 to 90 % for control, 86 to 88 % for binary, 89 to 90 % for ternary and 92 % for quaternary mixes at 15 % replacement level respectively. as curing increases, a marginal change in durability of increment was noticed.

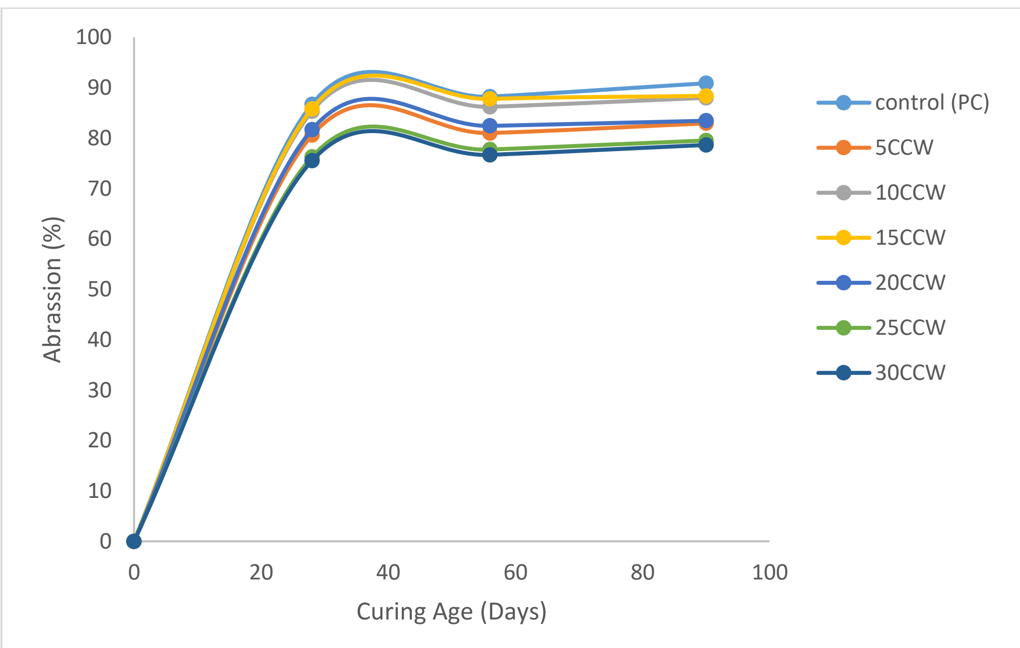

Figure 4.17: Abrasion resistance for binary (PC-CCW) Mix

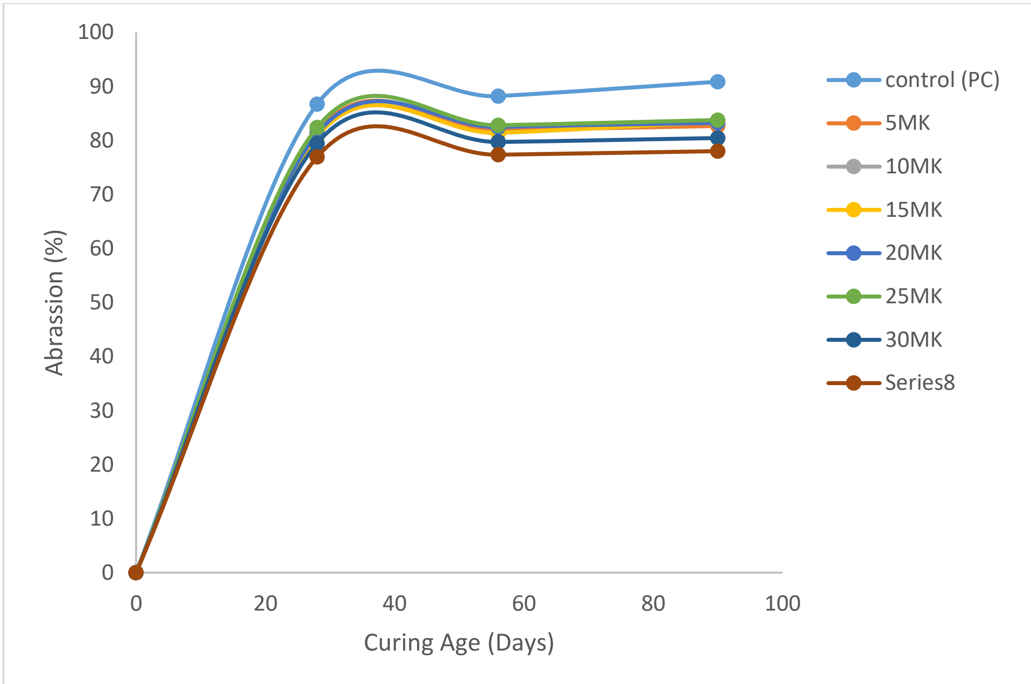

Figure 4.18: Abrasion resistance for binary (PC-MK) Mix

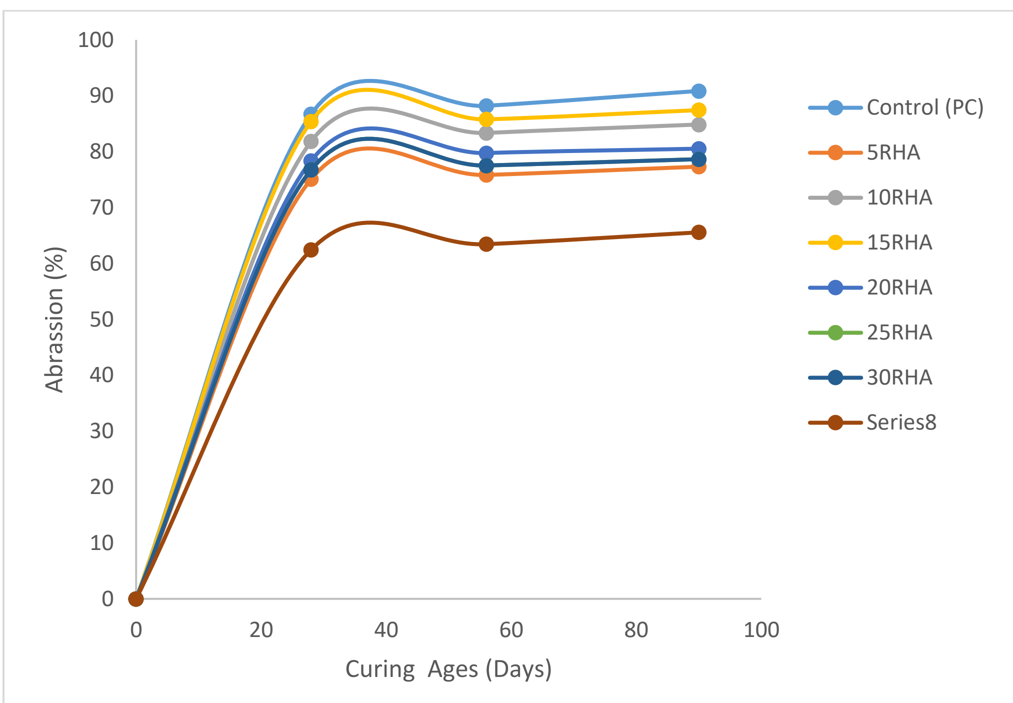

Figure 4.19: Abrasion resistance for binary (PC – RHA) Mix

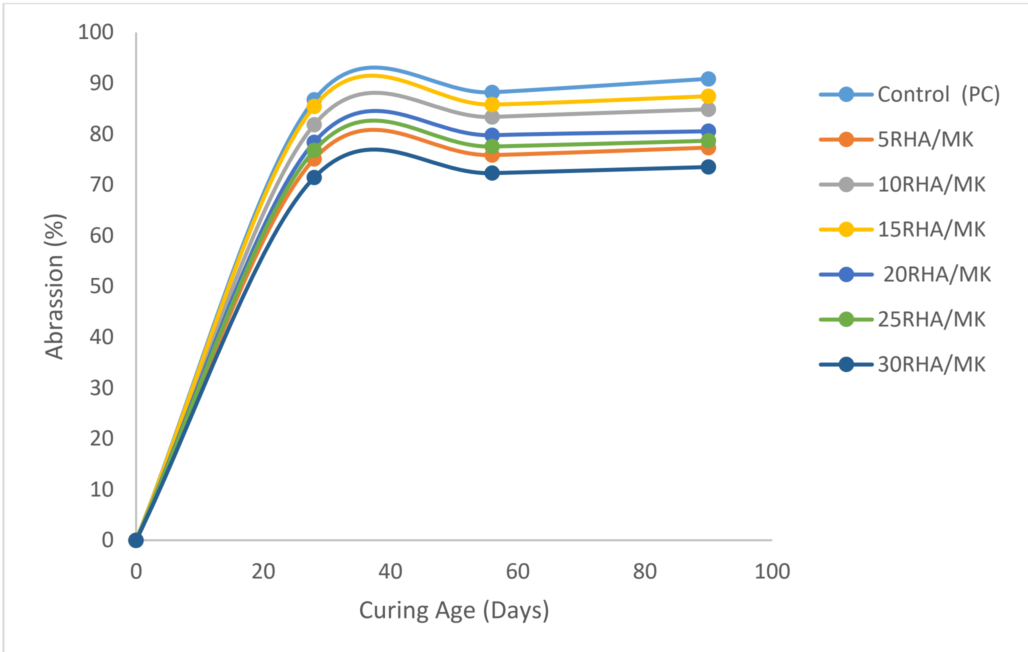

Figure 4.20: Abrasion resistance for ternary (PC/RHA/MK) Mix

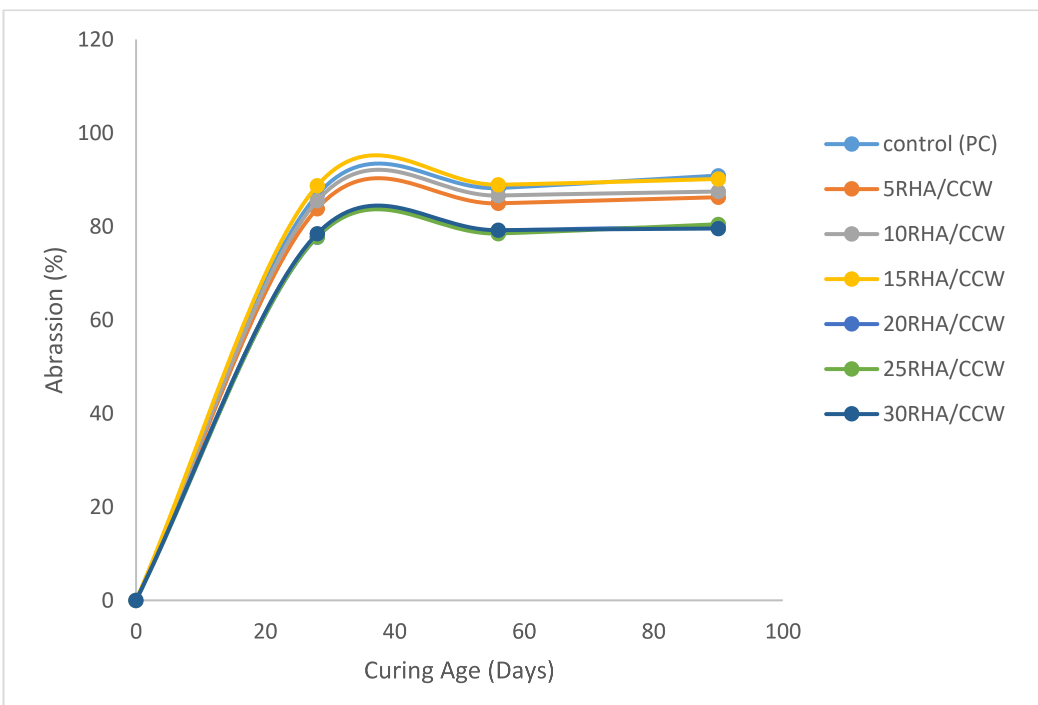

Figure 4.21: Abrasion resistance for ternary (PC/ RHA/CCW) Mix

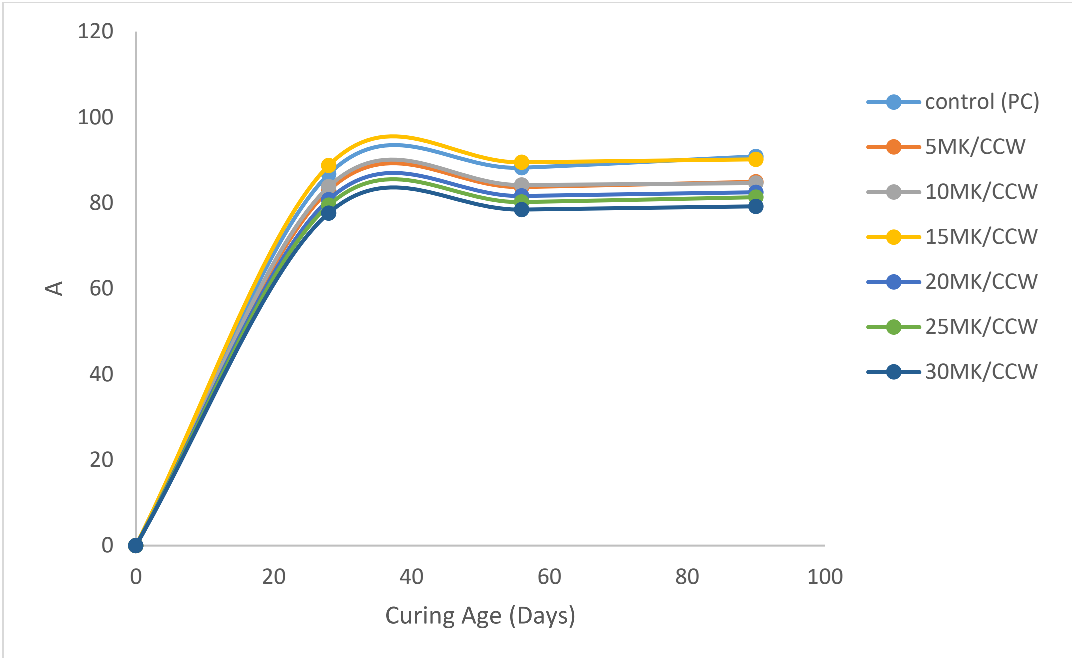

Figure 4.22: Abrasion resistance for ternary (PC/ MK/CCW) Mix

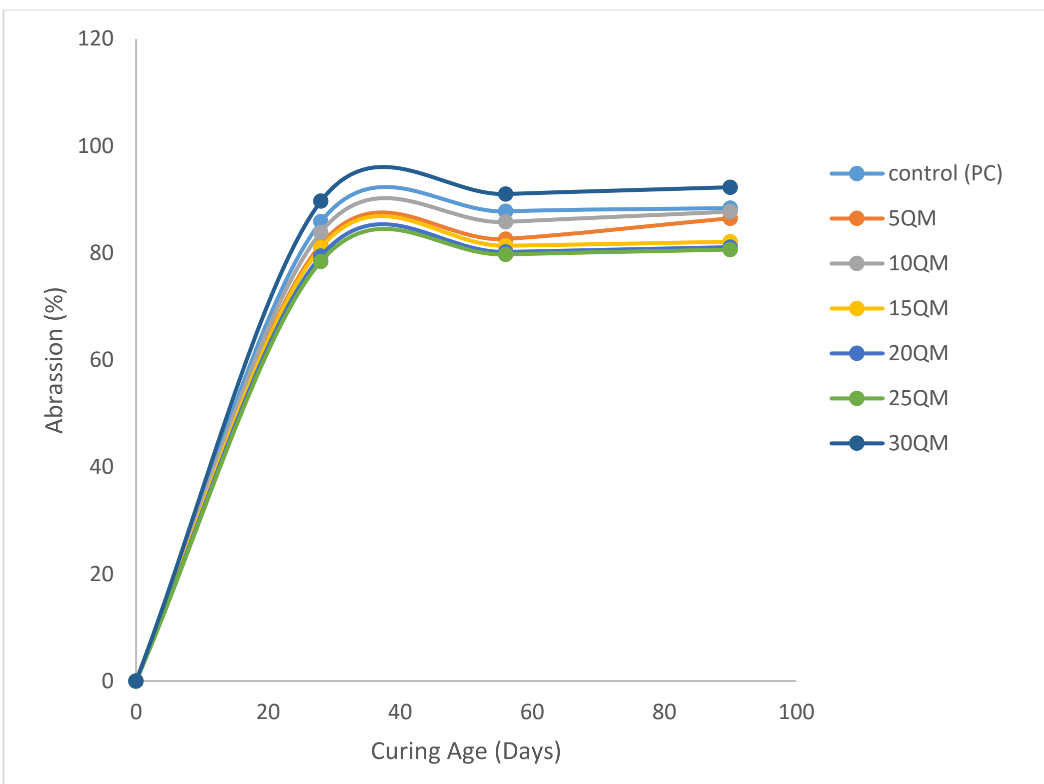

Figure 4.23: Abrasion resistance for quaternary (PC/RHA/MK/CCW) Mix

4.10 Summary of Findings

The study determined the properties of the constituent materials for mortar interms of both physical and chemical. All fresh and mechanical properties; consistency, setting times, flow rate, compound and tensile strengths and abrasion resistance were for each binder mix determined. This is imperative to determine and assess the feasibility of its usage in the anticipated place of exposure to wind, rain and other wear and tear effects. The summary results findings are:

The FM of fine aggregate 2.46 implies that it is medium sand.

1The combined oxide composition of SiO_2, Al_2O_3 and Fe_2O_3 for RHA, MK and CCW is 88.37 5, 86.90 % and for CCW, it is mainly 65.59 % CaO. The LOI of PC, RHA and MK met ASTM C 618 requirements except CCW which has a value of 21.71 %.

1) The properties of both initial and final setting times of blended mixes exceeded value of PC by about twice Pc values. The flow value decreased for cement blended mortar mix with increase in replacement levels compared with target value.

2) When the curing age increased, compressive strength increased (Figure 4.6 to 4.11). The strength of the cement blended mortars evened and surpassed control values after 28 curing age. It was observed same trend with the splitting tensile strength (Figures 4.12 to 4.16.)

3) Test results for the abrasion resistance for all the mixes are as shown (Figures 4.17 to 4.22). For control mix, an abrasion resistance value of 86 % to 90 % was obtained and for the quaternary mix, it is 92 %. It was also observed that abrasion resistance values slightly increased with curing age.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From tests results and analysis, the following conclusion is drawn:

1) The Oxide compaction(C.A.F) for RHA and MK is more from 20% which met ASTM C 618 provision while CCW S.A.F value did not but however, is rich in CaO with a65.59 % which enhanced the value for PC. Physical properties of the constituent materials also met all relevant code provisions.

2) Quaternary mix with 15% replacement of PC with RHA,MK and CCW is the optimum mix that produced cement based mortar binder with high quality.

3) Mortar produced from Quaternary mix with aforementioned SCMs used for the study had their setting times enlongated and flowability decreased.

4) The compressive and tensile strengths of their binder mortar obtained from quaternary mix improved over control values.

5) The PC- blended mortars are retarders and thus find application of reduction in cold joints in larger concrete pours in the production of Reinforced Marine Concrete (RMC) and in construction works in marine areas with RMC where longer period is required for its placing.

5.2 Recommendations

Based on the findings in this research work, the following is recommended:

1. Since the synergy of RHA, MK and CCW enhanced the properties of Quaternary binder containing these admixtures, it is imperative to investigate

hydration behaviour of the binder.

2. The PC – blended mortar containing RHA, MK and CCW belong to type M mortar (ASTM C 270 – 02) and hence can be used for empirical design of foundation walls.

3. The mortar can be used in construction walls in marine areas with reinforced marine concrete (RMC).

5.3 Further Studies

i) More research on Metakaolin in quaternary combined with PC, incorporating superplastiser to determine the influence of water reduction.

ii) Comparative cost benefits of these binders to other sources of partial replacement should be determined.

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APPENDIX A (Characterisation of Material)

Table A1: Sieve Analysis of Fine Aggregates

Sieve size	**Wt. of empty sieve**	**Residue + sieve**	**Mass of mat. retained**	**% mass retained**	**% cum. retained**	**% of cum passing.**
4.75	375	377	2	0.4	o.4	99.6
2.36	366	38.3	17	3.4	3.8	96.20
1.18	367	412	45	9	12.8	87.2
600	379	546	167	33.4	46.2	53.80
300	363	619	256	51.2	97.4	2.60
150	356	319	13	2.60	100.0	0
75	364	364	0	0	100.0	0
Pan	310	310	0	0	100.0	0
			500			

D60 = 610 mm, D_{30} =330 mm and D_{10} = 210mm

Coefficient of uniformity (Cu) = $D60/_{D10}$ = 2.9

Coefficient of curvature (Cc) = $(D30)2/_{D60(D10)}$ = 0.823

Table A2: Specific Gravity/ Bulk Density of Constituent Materials (kg/m^3)

Materials	Specific gravity	Bulk density (kg/m3)
CEM	3.40	3145
RHA	2.25	2430
CCW	2.33	2320
Sand	2.38	2570
MK	2.50	2495

Table A3: Compressive strengths values

1ST BINARY (RHA) Test Result IN N/mm

DAYS	7	14	21	28	56	90
CONTROL	18.91	16.59	25.39	19.09	27.88	27.84
CEM95%/RHA5%	15.13	13.71	13.57	17.23	20.30	20.09
CEM90%/RHA10%	9.33	10.8	10.66	15.03	14.82	17.00
CEM85%/RHA15%	14.13	13.09	13.45	14.59	12.01	17.32
CEM80%/RHA20%	8.41	10.72	9.89	14.64	14.92	23.51
CEM75%/RHA25%	8.33	9.77	12.51	12.51	15.79	13.84
CEM70%/RHA30%	6.24	5.8	10.12	6.6	10.21	7.55

Table A4: Compressive strengths values 2nd binary (CCW) in n/mm²

DAYS	7	14	21	28	56	90
CONTROL	18.91	16.59	25.39	19.09	27.88	27.84
CEM95/CCW5	15.96	22.40	21.00	17.75	16.84	21.05
CEM90/CCW10	12.75	15.00	19.05	13.55	17.49	21.67
CEM85/CCW15	11.80	10.44	14.41	14.05	12.03	11.19
CEM80/CCW20	13.56	13.28	17.33	12.64	14.46	17.93
CEM75/CCW25	11.01	10.73	12.71	15,09	10.31	9.41
CEM70/CCW30	9.72	12.09	12.62	8.21	11.97	12.79

Table A5: Compressive strengths values 3rd binary (MK) in n/mm²

DAYS	7	14	21	28	56	90
CONTROL	18.91	16.59	25.39	19.09	27.88	27.84
CEM95/MK5	17.12	19.86	24.77	18.85	17.19	24.32
CEM90/MK10	15.48	13.08	19.32	13.73	15.43	17.67
CEM85/MK15	11.01	8.45	10.23	13.89	10.43	10.40
CEM80/MK20	13.98	41.66	19.35	15.68	20.65	24.96
CEM75/MK25	9.64	10.61	14.8	12.89	13.07	8.72
CEM70/MK30	10.53	16.57	17.11	11.69	19.33	20.27

Table A6: Compressive strengths values 1st ternary (RHA/MK) in n/mm²

DAYS	7	14	21	28	56	90
CONTROL	18.91	16.59	25.39	19.09	27.88	27.84
CEM95/RHA2.5/MK2.5	11.24	13.76	14.81	14.25	13.00	10.47
CEM90/RHA5/MK5	12.81	18.01	16.09	15.61	15.09	16.87
CEM85/RHA7.5/MK7.5	14.08	10.03	13.39	18.72	14.49	10.01
CEM80/RHA10/MK10	5.70	7.68	6.85	6.60	9.21	7.37
CEM75/RHA15/MK10	8.61	9.51	12.04	11.89	10.47	10.47
CEM70/RHA15/MK15	6.24	5.80	10.12	6.60	10.21	7.55

Table A7: Compressive strengths values 2nd ternary(RHA/CCW) in n/mm²

DAYS	7	14	21	28	56	90
CONTROL	18.91	16.59	25.39	19.09	27.88	27.84
CEM95/RHA2.5/CCW2.5	15.31	18.39	15.88	15.64	26.04	23.85
CEM90/RHA5/CCW5	12.83	14.64	16.11	13.32	19.96	20.48
CEM85/RHA7.5/CCW7.5	6.07	11.48	11.53	9.95	10.17	10.07
CEM80/RHA10/CCW10	10.60	11.99	11.84	14.12	13.61	14.68
CEM75/RHA15/CCW10	14.00	11.63	15.08	14.48	16.36	13.27
CEM70/RHA15/CCW15	5.73	7.79	11.12	15.16	11.52	10.83

Table A8: Compressive strengths values 3rd ternary (MK/CCW) in n/mm²

DAYS	7	14	21	28	56	90
CONTROL	18.91	16.59	25.39	19.09	27.88	27.84
CEM95/MK2.5/CCW2.5	8.35	14.91	13.84	14.16	11.24	10.79
CEM90/MK5/CCW5	10.64	10.33	14.77	13.16	13.43	10.08
CEM85/MK7.5/CCW7.5	11.07	12.33	10.51	11.44	15.72	15.41
CEM80/MK10/CCW10	12.59	13.57	10.73	14.29	10.88	12.00
CEM75/MK15/CCW10	9.73	11.43	10.24	12.28	10.20	10.07
CEM70/MK15/CCW15	9.15	10.64	11.91	10.81	9.21	9,67

Table A9: Compressive strengths values quantenary (RHA/CCW/MK) in n/mm²

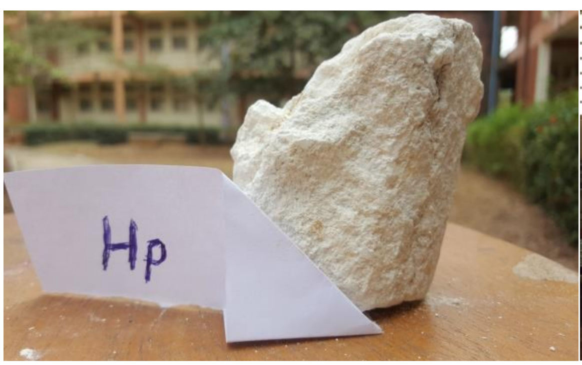
DAYS	7	14	21	28	56	90
CONTROL	18.91	16.59	25.39	19.09	27.88	27.84
CEM95/RHAI.25/MK2.5/CCW1.25	10.92	14.53	11.24	12.69	15.03	13.09
CEM90/RHA2.5/MK5/CCW2.5	10.91	12.04	11.80	16.97	19.13	19.19
CEM85/RHA5/MK5/CCW5	15.97	18.88	13.75	22.09	21.40	16.51
CEM80/RHA5/MK10/CCW5	14.77	12.16	13.73	15.75	13.97	11.97
CEM75/RHA7.5/MK10/7.5CCW	9.59	9.39	12.85	10.41	12.56	13.44
CEM70/RHA7.5/MK15/CCW7.5	7.36	10.91	8.60	11.09	13.91	14.3

TABLE A10: results Los Angeles abrasion test with 3 balls at 250 revolution

Sample Description	Mass of Sample M_1 (g)	Mass Retained on sleeve 1.20mm M_2(g)	Mass Passing Sleeve 1.20mm M_1-M_2 (g)	Abrasion Value $\frac{M_1-M_2}{M_1}\times 100$ (%)
CONTROL(PC)	272.4	247.6	24.8	9.10
5%CCW	270	238.6	31.4	11.63
5%RHA/MK	261.6	225.6	36	13.76
5%RHA	258	225.6	32.6	12.64
5%MK	261.5	217.2	44.3	13.76
5%RHA/CCW	271.4	241.5	29.9	11.02
5%RHA/CCW/MK	264.5	220.7	43.8	16.56
10% RHA/CCW/MK	268.7	225.3	43.4	16.15
10%MK/CCW	260	218.3	41.7	16.04
10%RHA/MK	257.5	216.2	41.3	16.04
10%RHA	269.4	228.6	40.8	15.14
10%CCW	269.7	225	44.7	16.57
10%MK	270.2	222.7	47.5	17.58
15% RHA/CCW/MK	261	217.8	43.2	16.55
15%RHA/CCW	255.5	200.3	55.2	21.60
15%RHA	264.4	204.6	55.2	20.88

15%MK	215.9	158.2	57.2	26.49
15%CCW	264	198.9	65.1	24.66
15%MK/CCW	277.8	204.5	73.3	26.39
20%RHA	246.1	198.2	47.9	19.46
20%CCW	246.3	216.7	47.6	19.33
20%RHA/MK	244	125.1	114.9	47.09
20% RHA/CCW/MK	268.6	206.5	67.8	25.24
20%MK	274.3	229.8	44.5	16.22
20%MKCCW	255.3	201.6	53.7	21.03
20%RHA/CCW	257.3	199.9	57.4	22.31
25%RHA	242.5	145.4	97.1	40.04
25%MKCCW	258.2	188.5	69.7	26.99
25%RHA/MK	274.9	206.8	68.1	24.77
25%CCW	262.7	208.9	53.8	20.48
25%RHA/CCW	261	199.5	61.5	23.56
25%MK	258.8	176.9	81.9	31.65
30MK	279.4	217	62.4	22.33
30%MC	259.6	179.6	80	30.82
30%RHA/MK	214.8	154	60.8	28.31

APPENDIX B



B1: Sample of High purity Metakaolin B2: Preparations of Mortar cubes and de-mould cubes

B3: Cubes for Testing

B4: UTM Compressive Machine

B5: UTM Compressive Machine