INFLUENCE OF SILICA-NANO PARTICLES ON PROPERTIES OF RICE HUSK ASH AND CALCIUM CARBIDE WASTE BINDER BASED-MORTAR

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ABSTRACT

In the recent time effort has been made to replace Portland Cement (PC) wholly, thus producing an alternative binder. This innovation could be due to environmental challenges attributed to the green-house gas emission coupled with energy intensity and consumption associated with PC production. The use of Rice Husk Ash (RHA) and Calcium Carbide Waste (CCW) as a binder in the production of mortar has revealed positive results with some drawbacks such as poor initial setting time (IST), final setting time (FST) and early strength gain. This study therefore target at inclusion of silica-Nano particle (SNP) and superplasticizer in RHA-CCW binder with a view to evaluate the influence of SNP inclusion of its performance characteristics. Silica-Nano Particles were extracted by chemical leaching of the RHA and so was the physical and chemical properties of the constituents materials determined before the assessment of the fresh properties of the binder paste. Mortar specimen were thereby made with 60:40 RHA-CCW binder containing varied SNP contents (i.e. 0 to 5 % at 0.5 step increment) for 1:3 binder/sand mix of 0.5 W/B ratio. This was tested for compressive strength, water absorption and abrasion resistance at various curing ages in accordance with BS EN 196-1:2016 for Strength of cement and other established standards. The results shows mortar specimen of 60:40 RHA-CCW binder containing 3% SNP has the best performance in compressive strength (having 28 56 and 90 days values of 10.94, 13.50 and 15.75 N/mm^2) representing an increase in strength 40, 63 and 80 % when compared to the RHA-CCW without SNP. 3% SNP addition in 60:40 RHA-CCW binder at 60/40 1:3 binder sand mortar, with 0.5W/B and 1.5% Superplasticizer by weight of binder was thereby recommended for masonry operations as it conforms to Class N of ASTM C270.

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CHAPTER ONE

1.0 INTRODUCTION

1.1 Background of the Study

Concrete is the most widely used building material. With rapid industrialisation and the growth of infrastructure, the demand for cement production is growing, leading to CO_2 emissions into the atmosphere, which have a negative effect on the environment (Rao and Navaneethawma, 2016). Studies on alternative binders have been the subject of research for a reduction in the use of cement in construction which will lead to a reduction in CO_2 emissions. Extensive work is aimed at the production of some agricultural and industrial waste that is thought to possess certain pozzolanic content.

Recent studies on the production of eco-friendly binders by Enejiyon (2018) and Olawuyi *et al.* (2017) have shown that Calcium carbide waste and Husk Ash from rice has a binder possibility in mortar / concrete. Ash from rice is created by the use of rice husk, which is a rice milling by-product. Reportedly this accounts for 20-24 percent of the produced raw rice. Several research findings on RHA's efficiency as cement substitutes have been published (Oyetola & Abdullahi, 2006 & Obilade 2017)

Likewise, waste from Carbide is a derivative from the processing of acetylene gas (C_2H_2). It consists mainly of lime ($Ca(OH)_2$) and solid caustic compounds, and, when pure, is white. The reaction between calcium carbide and water produces a trace of acetylene gas that is used in oxyacetylene welding and Calcium hydroxide

$$CaC_2 + 2H_2O \longrightarrow C_2H_2 + Ca(OH)_2 \qquad (1.1)$$

However, in many fields of application the use of Nanoparticles in recent years has received particular attention in formulating ingredients with new functionalities, and it

has become contemporary area of research in the field of concrete technology (Reddy & Vardhan 2017).

Nanotechnology has been applied for construction purposes to the study of cement and concrete, and is moving concrete science into a new age. Countless Nanomaterials (including Nano-sized powders, tubes and fibres) have been developed and incorporated in cement materials. Nano-Silica and Nano-Titanium Oxide have done significant work to date. Nano-SiO_2 or NS have not been published. Studies in Nanosilica, Nano-Calcareous (Nano-$CaCO_3$), Nano- Cement, Nano-Iron (Nano-Fe_2O_3) Nano-Carbon and Nano-Aluminum (Nano-Al_2O_3) application studies (Kumar and Singh 2018) are the (Sobolev *et al.,* 2009: Huang *el.al,* 2015)

Studies have shown that well-dispersed Nanomaterials can speed up the hydration of cement, i.e. decrease the setting time of paste and mortar (Kumar and Singh, 2018). It further refines micropores concrete and transforms the inner structure of C – S – H gel (like the silicate chains), hence substantially decreasing permeability and enhancing the reliability and durability of the properties (Huang *et al.,* 2015) Nonetheless, one inadequacy of cement-based materials affected by Nanomaterial's is due to high specific surface area, the Nano-material will unfavourably affect the workability.

If Nano Particles are combined with paste, mortar, and concrete in Portland cement (PC), products with different characteristics are obtained from conventional materials (Rao & Navaneethawma, 2016). The performance of these cement-based materials depends heavily on solid Nano Particles, such as particles of calcium silicate hydrates and Nano-sized solid porosity are interfacial in between cement and sand particles. Energy, durability and shrinking are some of properties of Nano-sized particles (Alireza *et al.,* 2010)

Nevertheless, in view of past studies, an in-depth understanding of the Nano-Silica incorporation mechanism on the hydration of a cement binder and its role in microstructural evolution and in gel chemistry is still lacking (Memon *et al,* 2010). Likewise, a thorough understanding of the chemistry of CCW combined with RHA to form an agro-industrial waste binder has yet to be developed (Reddy and Vardham. 2017). Hence, this study aims to establish understanding of fresh properties and early-age strength properties of Nano-silica-mortar, inclusion in CCW and RHA-based binders.

1.2 Statement of Problem

Because of its adaptability, versatility, durability and ease of manufacture, Concrete is the world's most popular construction material. The challenge the concrete industry worldwide is therefore to meet the demand of enormous infrastructure requirements, coupled with rapid industrial development and economic growth, as well as urbanization. (Rao & Navaneethawma, 2016) on the other hand, it is sadly found that PC, which forms concrete aggregate's blending agent, contributes tremendously to the global warming syndrome. Technically, ecologically and economically, there is a need to modify the current concrete production methods in terms of the application of cement.

A significant amount of work has been done on how to remove or reduce the use of PC in concrete without compromising the critical characteristics of cement composite materials. Materials from Pozzolanine like the, Fly Ash, Silica Fume, granulated slag from the earth, RHA and Industrial Timber Ash (ITA) or Saw Dust Ash (SDA) have been found to offer the technological advantages of replacing PCs in terms of new, hardened condition and toughness of concrete. (Ferraro et al., 2017)

The eco-friendly advantage accompanying the use of this material is that it acts as another method of waste disposal, thereby turning the waste into a viable building material. This practice has multiple advantages; the consumption of industrial waste in an environmentally friendly manner, the conservation of natural resources, the moderation of greenhouse gas emissions and, above all, the improvement of the properties of concrete is the culmination of a sustainable community growth. While cost savings have been made in replacing the PC with this waste, the burden associated with its disposal is also reduced.

Recently, attempts have also been made to replace PC using CCW wholly in combination with Sorghum Husk Ash (SHA) or RHA in mortar and concrete production (Egwuda, 2017 & Enejiyon, 2018).While the analysis yielded a promising result, features such as workability and early strength gain on the SHA / RHA-CCW binder were adversely affected. Therefore, this research aims to include silica Nano particulate matter and superplasticizer in RHA-CCW in order to strengthen the deficiencies of slow hydration and low strength gain found in earlier studies.

1.3 Aim and Objectives

The aim of the research is to study silica and Nano particles' impact on the properties of Rice Husk Ash and Calcium Carbide Waste binder based mortar in order to develop an effective proportional combination for good strength output and adequate RHA-CCW bonding hydration. The Objectives are to:

i. Evaluate the physio-chemical properties of the RHA, CCW and SNP.
ii. Determine appropriate mix proportions of the constituent materials for fresh properties of mortar made with RHA-CCW and SNP.
iii. Assess the effect of SNP inclusion on pozzolanic mortar properties made using RHA-CCW.

iv. Examine the microstructure of the hardened RHA-CCW and SNP binder-based mortar.

1.4 Research Scope of the Study

This Research concentrated on using RHA CCW and SNP as an alternative binder base mortar. With gradual replacement of SNP from 0.5 to 5 percent. The analysis highlights the benefit in fresh and early strength as well as the microstructural properties of alternative binder-based materials, which are believed to be within the limits set by the objectives. The study findings cannot be extended in general terms except for RHA and CCW which have the same index features. The cost efficacy of this analysis was not considered. This does not, of course, intend to neglect the study of the economy to the background, but rather it is believed that performance assessment must be understood and perfected before determining the economic aspect of the study.

1.5 Justification of the Study

Rice husk is one of the common wastes produced from North Central and North West Nigeria rice milling centres. Such wastes are actually disposed of as land-fill material without any economic gain in return, and substantial money is expended on disposal operations. RHA and CCW can be used properly; considerably enrich the mortar and concrete products and other associated building materials to reduce the burden on PC domestic and industrial use (Obilade, 2017).

The use of RHA and CCW as an alternative binder would help to reduce the carbon dioxide (CO_2) emissions associated with PC production (Olawuyi *et al.,* 2017). Essentially, a substantial amount of CO_2 is released into the atmosphere for every PC output. As this study is aimed at alternative binders in mortar and concrete, it will ideally help balance the environment, reduce disposal costs, reduce the pressure on PC use and conserve the natural resources used in PC production (Obilade, 2017;

Brown, 2017). If the RHA-CCW technology with SNP as additive is properly expressed, the total construction cost can be that, thereby generating green mortar and concrete and making construction affordable. The study is expected to reveal fundamental properties with RHA-CCW and SNP for producing sustainable, high ultimate strength and high durability concrete.

CHAPTER TWO

2.0 LITRETURE REVIEW

2.1 Binder in Concrete and Mortar

2.1.1 Cement

Cement is a binder that sets, strengthens and adheres to certain materials to tie them together. It can also be described as an adhesive that can unite fragments or masses of solid matter into a compact whole (Hewlett, 2006). Building materials and the techniques of architectural design are gradually developed prior to using cement: people designed the foundation of masonry with the laying of large flat piles, which were then filled with small stones and used mud as a binder (Rasa *et al.*, 2009).

Developments in construction sectors have put in tougher materials. Lime and gypsum were several examples of materials that came into use for adhesive or cement. Natural cement rock was discovered in later days during the 1818 Erie Canal geological survey Close to Chittenango, New York (Shan, 2001) Therefore, hydraulic cements is one of the natural cement.

Hydraulic cement is one that acquires binding properties when there is water. Joseph Aspdin first found Portland cement in 1824. Today cement in Portland is the most common binder in the construction sector.

2.1.2 Lime

Lime is calcium that includes inorganic minerals, mainly consisting of oxide and hydroxide, typically calcium oxide or calcium hydroxide. Calcium carbonate is normally produced through calcination. One of the naturally occurring sources of calcium in this cycle High temperature carbonate is subjected to the separation of Carbon dioxide from calcium carbonate as seen from the equation below. $CaCO_3$ occurs naturally in various forms, including granite, chalk and calcareous (Nattapong, *et al.,* 2010).

$$CaCO_3 \longrightarrow CaO + CO_2 \quad (2.1)$$

2.1.3 Gypsum

Gypsum is a soft mineral sulphate with the chemical formula CaSO4.2H2O consisting of calcium sulphate-dihydrate. Pure gypsum is a completely hydrated lime sulfate also called calcium sulphate dehydrated. This is the mineral sulfate most commonly present in many locations and types (Shan, 2001). Gypsum is commonly mined and used as an agricultural fertilizer and as many types of cement, black board side walk chalk and dry wall.

2.2 Natural cement

Natural cement is formed by the natural calcination, at a temperature at which the calcareous and argillaceous material mixtures are smeared ("USGS mineral programme, cement program report" 2011). Natural cement types include the following: the British "roman" cement, the former American rock cement and the Belgian "pure Portland cement."

2.2.1 Portland cement

In today's construction practice, Portland cement is the most frequently used cement type. In 2010 the worldwide hydraulic cement output amounted to 3.3 billion tons ("USGS Cement Program Survey" 2011). China generates 1800 million tons, India produces 220 million tons, while the United States produces more than half the world cement ("USGS mineral program cement survey" 2011) China produces more than 63.5 million tons of cement.

2.2.2 Composition of Portland cement

Portland cement (PC) consists of complex oxides. The composition of PCs is significant for their properties:

PC is made up of various oxides with calcium oxide amounting to about 64%. The amount of the compounds contained in cement varies greatly except for a relatively minor change in the oxide composition of the raw materials. It is absolutely important to track closely the composition of Oxide of the raw materials for the production of cement with a compound composition specified (Shan 2001).

Table 2.1: Typical Composition of Portland cements

Oxide formula	% in PC
CaO	64.10%
Al_2O_3	5.50%
Fe_2O_3	3.00%
SiO_2	22.00%
MgO	1.40%
SO_3	2.10%

Source: Shetty (2015)

2.2.3 Properties of Portland cement

Cement paste is a vital part of mortar and, as a result, concrete strength characteristics are strongly affected by cement paste density, according to Rasa *et al.*(2009). However, it is important to carry out acceptability tests or, more often, to analyse the properties of a cement used for a particular resolution by the buying or independent laboratory. Chemical and physical composition measures such as fineness, soundness, setting time, hydration pressure, etc. The BS EN 196-1:2016 European Norms suggest these, and further testing for normal and fast hardening cements is recommended by BS EN 196-6:2010.

2.2.3.1 Fineness

The fine consistency of the cement influences the rate of water reaction as it has a higher surface area than coarse concrete. One of the last steps in the concrete manufacturing process should be the grinding of gypsum clinkers. Since hydration starts at the cement particle level, the total cement surface reflects the hydration

content. Thus, the hydration rate is dependent on the fineness of the cement particles and a high fineness is required for quick strength development. Higher early hydration also poses an increased risk of early heat production (Rahhal & Talero, 2010; Neville, 2012). This cement feature can be extracted from a certain cement surface, measured using the air permeability process, with the average surface value of the PC defined being 300 m2 / kg.

2.2.3.2 Setting Time

This is the term used to explain the change in condition of the fresh cement paste. Setting usually refers to moving from a fluid to a static state (Neville, 2012). Replacement of PC with pozzolanic content influences the setting characteristics of concrete and cement paste. In general, impact of concrete and cement paste setting time is more pronounced when a large percentage of Pozzolan is used (Senff *et al.*, 2009). While the broad application can be detrimental to the pasting and concrete environments, plasticizers can be used to restore the output required in terms of setting time. Paste and concrete temperature and other environmental factors also lead to the setting impact.

2.2.3.3 Workability

The favourite characteristics of concrete were early development of strength and strong workability. However, using the standard ratio of water to cement is always challenging to overcome at the same time. Those concrete properties depend on the concrete mixture's ratio of water and cement. Operability of concrete mix is improved by an improvement in the w / c ratio, but performance is inversely impacted. It is very difficult to obtain a low water to cement ratio for the sake of improving the strength characteristics of concrete and to retain high workability unless water reduction (superplasticizer) is added into the mix. For example, the use of Nano-particle materials

increases the absolute volume of cemented materials relative to non-Nano-particle concrete; thus, the volume of the paste is increased, resulting in decreased aggregate particle interference and enhanced concrete workability. (Liu et al., 2012)

2.2.3.4 Bleeding

Bleeding is a form of separation also recognized as ‘’water gain’’, where certain mixed water tends to upsurge to a freshly placed surface of the mortar. Key factors causing mortar bleeding are the inability of the mix’s strong constituents to retain all mixing water when they settle down, as Neville and Brooks (2010) argue that it is primarily found in a highly humid mixture, poorly proportioned and insufficiently blended concrete or mortar.

2.2.3.5 Segregation

Separation can be characterized as separation during handling and placement of the constituent concrete materials. However, Shetty (2015) described segregation as isolation of mortar / concrete essential materials and their distribution is therefore no longer compatible. Successful mortar / concrete are where all the materials are used to spread evenly and create a homogeneous mix. It is a concrete condition in which the constituents are isolated and thus the purpose will not be accomplished.

2.2.3.6 Strength of cement

Strength is an essential property of cement and many factors depend on this during its application, such as the water to cement ratio, cement strength and control. Standards such as European Standard BS EN 196-1:2016 recommend a check compressive strength on specimen mortar. The samples are supposed to be measured as 40 mm cubes equivalent; from 40 to 40 to 160 mm prisms, measured first in bending, they may break down to half or otherwise divided into half. Therefore an optional bending core test is possible over a distance of 100 mm. The code suggests a fixed

composition mortar test using a 'CEN norm powder.' (CEN is the French acronym for the European Standardization Committee) The natural sand CEN, silica and circular sand, can be collected from different sources. The cement / sand ratio (s / c) of 1:3 is defined as the blend ratio specified by BS EN 196-1:2016 for the concrete strength test. It has a size not standardized but ranges from 80 μm to 1.6 mm. Neville, 2012 recommends that the mortar be mixed in a pastry blender and compacted with a 15 mm drop shaker; a table can be used to vibrate as long as the compaction results are equal.

2.2.3.7 Cement Hydration

Chemical reactions between cement and water are termed cement hydration. A schematic presentation of hydration of cement appears in Table 2.2. Concrete chemistry is simply chemical reaction chemistry amongst water and cement. These products are formed because of the hydration. These are vital products since they are of cemented or adhesive nature. Hydration products are important in quality, quantity, consistency, stability and rate of development. Anhydrous cement compounds react when combined with water, forming hydrogen compounds of low solubility

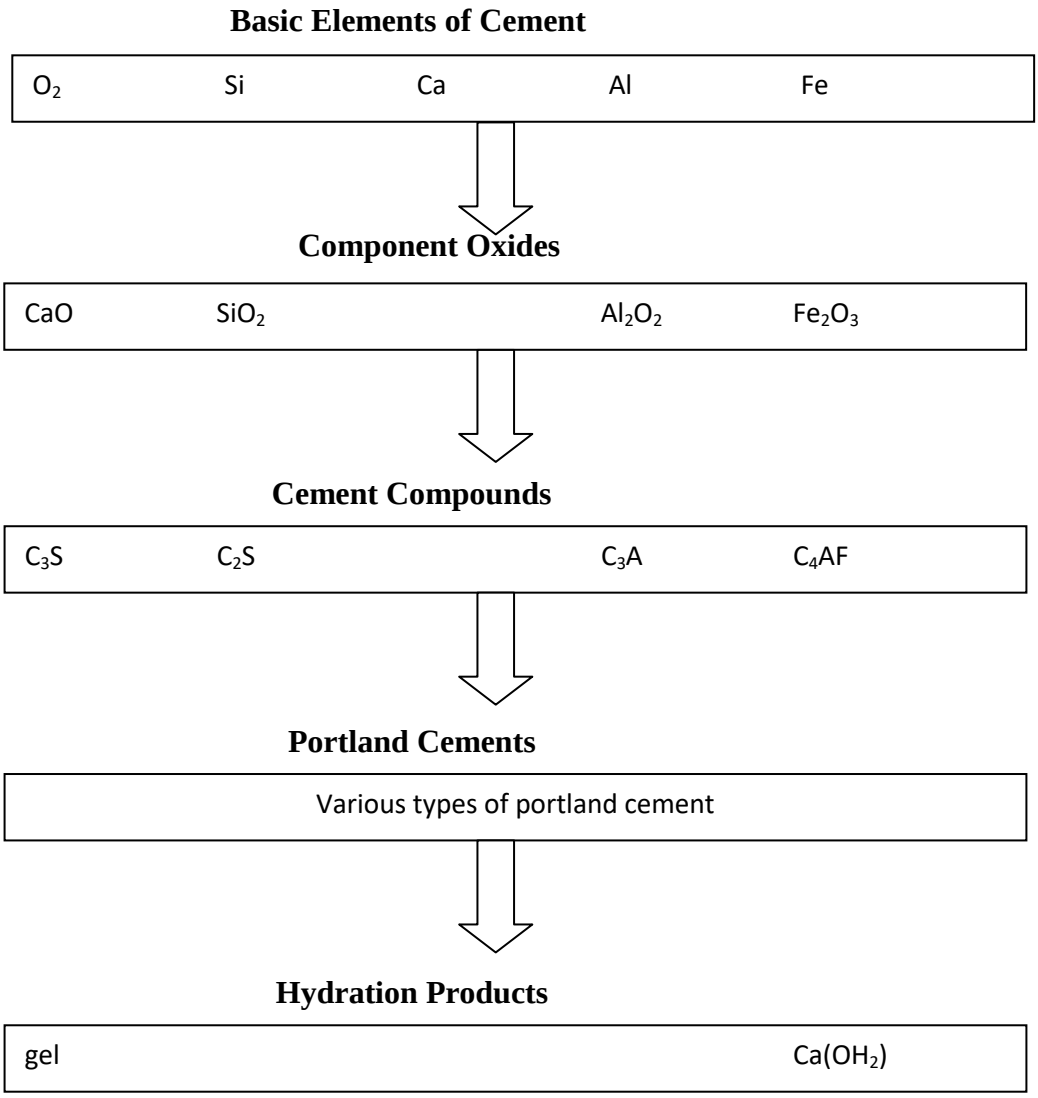

Fig 2.1: Schematic representation of the formation and hydration of Portland cement

2.3 Heat of Hydration

Cement and water react in an exothermic way, a cycle of reaction during which heat is released in material. This heat released is termed hydration heat. When a newly mixed concrete is put in a vacuum flask and the weight temperature read at intervals, it becomes obvious. In construction, concrete dams and other mass concrete constructions, the analysis and mechanism of the hydration heat becomes important. It has been found that at the time of placement, in broad mass cement, the temperature is above 50 ° C initial concrete weight temperature and remains at

elevated temperature for an extended amount of time (Shetty, 2015).

When cement is combined with water a rapid heat can build for a short time. This production of heat is possibly due to reactions from the aluminium and sulphate solution. This initial heat evolution ends swiftly when gypsum decreases the solubility of aluminates. The C-S - H gel reaction is responsible for the next heat growth formed by di-calcium silicate (C3S) and tri-calcium silicate (C2S) (Hewlett, 2006; Mehta & Monteiro, 2014)

Specific compounds hydrate at differing levels, releasing various heat amounts. Since retarders are added to track tri-calcium aluminate (C3A) flash setting properties, in fact early hydration heat mainly contributes from C3S hydration. Standard cement normally produces between 89 and 90 cal / g in 7 days and between 90 and 100 cal / g within 28 days (Hewlett, 2006)

2.4 Mortar

Mortar is a combination of water, sand, and cement used to bind together and fill the gaps between building materials such as blocks, stone, and brick. Mortar gets tough as it sets, leading to a solid aggregate framework (Shan, 2001). It is called lime mortar when made with lime, sand and water however, the addition of cement is called cement-lime mortar or simply cement-mortar (Shan, 2001)

Mortar has numerous applications which include:

i. It bonds the individual masonry units together
ii. It serves as a seating material for masonry units
iii. It allows for levelling of masonry units and seals irregularities
iv. It is used as a plaster to walls
v. It gives aesthetic quality.

The mixtures used in mortar moves through a sieve size of 4.75 mm which is held on

600 um and are called fine aggregates. These aggregates produce a hard mass known as mortar, once combined with lime, cement and water.

2.4.1 Lime Mortar

Lime, water, and fine aggregates are called lime mortars since their strength is poor compared to that of cement mortar; lime mortar is used for temporary masonry construction.

Lime can be classified as hydraulic and non-hydraulic depending on its contact with water. As with the PC, hydraulic lime sets in the presence of water. Before PC was introduced (Givi, 2011), this was used as a preliminary binder until the mid1800s. Non-hydraulic carbonation lime sets require air (Yates & Ferguson, 2008).

2.4.2 Cement Mortar

Cement mortar is a mixture of fine aggregates, water, and PC. Figure 2.1 demonstrates cement mortar used to create masonry.

Plate 1: Cement mortar used in masonry work

Source: Bricklaying Workshop, Federal Polytechnic, Bida

2.5 Pozzolanic Materials

2.5.1 Pozzolans

Pozzolans are a broad variety of silicon and aluminium materials with little to no cement value, but which react in a fine divided form and form chemical compounds with calcium hydroxide when in the presence of water (ASTM C 125 2015). It is critical, according to John and Ding (2007), Pozzolan is thinly divided, since SiO_2 can react to $Ca(OH)_2$ (produced by hydrating PC) only in the presence of water to form stable, cemented calcium silicates (Neville, 2012). Moreover, the silica must be amorphous, that is, shiny as it is extremely poor in reactivity. In addition, Duggal (2008) claimed that the word Pozzolan derives from Pozzouli, a town on the Bay of Naples near Mount Vesuvius in Italy. When mixed with hydrated lime the sand (volcanic dust) around this town was found to possess hydraulic properties Pozzolan was used with lime in those days before the arrival of cement to make concrete, but it is often used to substitute a proportion of cement in concrete construction. Pozzolan also has two distinct significances (Hewlett, 2006) The first one shows the essentially glassy and sometimes zeolitised pyroclastic rocks that occur either in the Pozzuoli neighbourhood (the ancient Roman-era Puteoli) or around Rome. All natural and artificial pozzolans rich in SiO_2 and Al_2O_3, only comprise a insignificant amount of alkali (Hewlett, 2006; Duggal, 2008) Examples of pozzolanic materials include volcanic ash, pumice, shales of clay, brick dust, marble dust, and fly ash. To be reactive the SiO_2 in a pozzolanic material must be amorphous, or glassy.

Pozzolan materials can be divided into two groups,:

i. Natural Pozzolans
ii. Artificial Pozzolans

2.5.1.1 Natural Pozzolans

Natural Pozzolans have a magnetic base and are commonly available throughout the world. They are also of volcanic origin with volcanic ash called Pozzolan origin (Neville 2012). According to Parhizkar *et al.* (2010), Natural Pozzolans can also be extracted from natural materials containing reactive SiO_2 or Al_2O_3, which alone have small to no binding features, but which are fixed and hardened like cement when mixed with PC and lime in the presence of water. Natural Pozzolans are classified into four classes based on Ramezanianpour (2014) current principal reactive lime constituent. It consists of tuff and pumicitis, volcanic rock, calcined clay or shale, and coarse opaline silica or calcinated silica.

2.5.1.2 Artificial Pozzolans

Artificial Pozzolans are mainly natural materials produced through thermal processing as well as low Pozzolan activity materials that require more treatment in order to achieve pozzolanic activity; they are the result of chemical or structural modifications of materials which originally had no or poor pozzolan properties (Ramezanianpour, 2014). Examples of artificial Pozzolans as set out in (Shetty, 2015) are: Fly ash, burning slag, Silica smoke, Rice husk ash, Guinea maize ash and Metacaolin

2.5.1.3 Specifications of Pozzolanic Materials

Pozzolan materials are categorized in three classes according to ASTM C 618 (2012).

i. Class N: Natural carbonized pozzolans which fulfill the related class criteria set herein, such as certain diatomacea, shales, opaline cherry tuffs and volcanic ashes or pumicites and various materials which need calcination to induce appropriate properties such as certain clays and shales

ii. Class F: Usually extracted from burning anthracite or bituminous coal, the fly ash meets the applicable criteria for the class as laid down in this document. This form of fly-ash has pozzolanic characteristics.

iii. Class C: Normally generated lignite- or sub-bituminous coal fly ash, which meets the applicable class requirements, as laid down in this text. In addition to its pozzolan properties this type of fly ash also has some cement properties.

iv. For a material to be a pozzolanic material, ASTM C618 (2012). must be satisfied

Standard chemical and physical requirements as seen in the below

Table 2.2: ASTM C618 (2012) Specifications of Pozzolanic Materials

Material contents	**Mineral Admixture Class**		
	N	F	C
$\Sigma SiO_2 + Al_2O_3 + Fe_2O_3$ min, %	70.0	70.0	50.0
SO_3 max, %	4.0	5.0	5.0
Moisture content	3.0	3.0	3.0
Loss on ignition, max, %	6.0	6.0	6.0

Source (ASTM 618 (2012)

2.5.2 Pozzolanic Reactions

Pozzolan activity is measured by the degree of reaction in water over time or the response rate from Pozzolan and Ca^{2+} or Calcium hydroxide (Ca(OH)2). According to Duggal (2008), however, in combination with ordinary Portland cement, Pozzolan silica is mixed with lime that is formed during the period of cement humidity and is called the action of pozzolan. Pozzolana activity is caused by fine glass silica and lime moisture, which create a calcium silicate similar to that formed by Portland cement hydration. In Pozzolan, SiO2 react to lime produced by the cement moisture in Portland and helps to create strength. Slowly and slowly, a supplementary hydrate of calcium silicate

forms a binder and fills room, providing permeability, resilience, and increased power.

Hydration of Portland cement may be expressed as given in Equation 2.1

$$C_3S + H_2O \longrightarrow C\text{-}S\text{-}H + Ca(OH)_2 \quad (2.1)$$

(Calcium silicate hydrate) (Lime)

$Ca(OH)_2$ produced from equation 2.1 combines with SiO_2 of Pozzolan to produce C-S-H as shown in equation 2.2

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

(Silica) (Lime)

Amorphous SiO_2 formed readily reacts with $Ca(OH)_2$ this distinction lies between pozzolanic and similarly chemical composing materials with low pozzolanic activity than those of crystalline form. Since pozzolanic effect can only be produced in the presence of water, adequate moisture is required to complete the pozzolanic action for a long time. Ca $(OH)_2$- SiO_2 is often assumed to be the main or only reaction, but recent data show that, when present, Al_2O_3 and Fe_2O_3 both take part in the chemical reaction. The best Pozzolan quantity, as a substitute for cement, is estimated to be 10-30 per cent (Duggal, 2008) recent studies have shown that replacement can go beyond 30 percent for a good quality pozzolanic material. This is thought to be a substitute for large quantities, e.g. fly ashes (Atadero & Namagga .2007)

2.5.3 Supplementary Cementitious Materials (SCM)

2.5.3.1 Rice Husk Ash (RHA)

Rice husk is a farm waste that contains a high level of SiO_2, distributed in large amounts worldwide. Rice production in 2018 was approximately 475 million tonnes, according to the FAO report (2015) The annual rice husk generation

(RH) is estimated at around 1/5th annual gross rice production worldwide During milling process the RH is converted to RHA which is used to generate electricity at mills.

Rice husk ash is a very reactive material; temperature regulated rice husk pozzolanic material. Amorphous SiO_2 is produced, which is highly reactive in nature, depending on the time and the rate of burning. When over-burning occurs (above 700 ° C), the amorphous SiO_2 will transform to cristoballite, quartz, and tridymite, and when burned at low temperatures (below 500 ° C), there will be a great deal of carbon in the product; in any case, the pozzolanic activity of RHA will be greatly reduced. The bulk concrete densities are recorded to decrease as the percentage of RHA content rises (Obilade, 2012). RHA is of low specific gravity and can be used to produce lightweight construction materials (Sadrmotezi, 2017). RHA in cement is optimally replaced by nearly 10 percent in terms of workability and strength (Dhinakaran *et al*, 2014). This offers many advantages, such as improved resistance and resilience, environmental advantages of waste recycling and lower CO2 emissions (Ferraro *et al*, 2017).

2.5.3.2 Calcium Carbide Waste

Calcium carbide is a by-product of calcium carbide-generated acetylene gas used in processing, and for welding of polyvinyl chloride (PVC), particularly in the automotive industry. The main part of CCW is $Ca(OH)_2$, which can respond to a product close to that obtained from the cement hydration process by pozzolana reactions with siliceous materials (Sun *et al.*, 2015). Traces of aluminum oxide, magnesium oxide and sulphate oxide are believed in this material (Ndububa & Omeiza 2016). The cement replacement standard is found to be excellent at 5-10%.

CCW concrete's insulation properties also enhance setting times with CCW percentage content increase. Due to its higher setting time, CCW will therefore perform better in mass concreting and in the hot climate (Reddy & Vardhan, 2017). CA(OH)2 with copper, plum, iron, manganese , nickel, and zinc impurities are estimated to be 70%-80% of calcium carbide waste (CCW) in Nigeria (Memon *et al.*, 2010).

2.5.3.3 Nanoparticles

Nanomaterials are Nanometer particle scale Nanometers of very thin materials. Such materials are very efficient in modifying the properties of concrete at an extremely low level with the benefit of their very small scale. The small particle dimension also translates into a broader region (Givi, 2010) the pozzolanic particle reaction rates are proportional to the available surface area and allow for quicker responses. Generally only a small proportion of cement can produce the results you want. Through pore filling and minute vacuums in the cement paste microstructure phase, Nanomaterials boost the strength and non-permeability of concrete and mortar. The results of increasing tensile and bending strength of concrete and mortar have been demonstrated in the use of Silica-Nano particles in mortar and concrete mixtures. It moisturizes early and therefore normally involves mixing when the material is mixed, cement that contains Nano-silica mixtures that create C-S - H gel hydrating Nano-crystals. Such Nano-crystals absorb cement in the micro pores and thus improve the mortar ability, density and strength of the concrete.

While Narender & Meena (2017) expressed several benefits when applying NS, it was not determined if its chemical response to the solution indicated that the cement in NS was higher. Although their research pretends that the mechanical conduct of concrete materials affects more effectively the strength of structural elements and

phénomènes at the micro and Nano stage.

2.5.3.4 Fly Ash (FA)

Fly Ash is a finely fragmented mineral remains produced by combustion in thermal plants of soil or powdered coal (Rohini & Arularasi, 2016) It is composed of the coal component of non-combustible minerals. FA particles are finer than cement particles in glassy, spherical 'ball bearings' (Pitroda *et al*, 2012). All fly ash has a particle size range of under 150μm. The properties of fineness and particle size are of great importance, as they affect the air content and water demand of concrete (Namagga & Atadero, 2009). It is a pozzolanic material that react to the Ca-S-H, the strongest and most durability portion of the concrete paste, if water is containing free $Ca(OH)_2$. This substance is a useful concrete mineral admixture that affects many concrete and mortar materials, both in fresh and hardened condition.

Moreover, the use of waste materials in the cement and concrete industries removes the environmental problems of power plants and lowers energy production costs for cement production. Cement containing fly ash increases concrete permeability and results in thick C – S – H (Pitroda *et al*, 2012). Fly-ash increases concrete strength. It also increases alkaline aggregate resistance, slows down moisture, oxygen, chloride, carbon dioxide and aggressive chemicals and prevents corrosion (Memon *et al*, 2010). Class F (low calcium) fly ash is preferred because it has been readily available as a low-cost by-product from the coal power plants and traditionally reduces the likelihood of alkali-silica reactivity as compared to class C fly ash (Lahri, and Dixit, 2015).

Low calcium (Class F) fly ash was chosen because it is easy to obtain from coal power plants as a low cost byproduct and traditionally reduces the likelihood of alkali-silica reactivity as compared to class C fly ash (Ferraro *et al*, 2017)

The optimum content of fly ash will vary on a case-by - case basis. Content from fly ash up to 50 per cent can be ideal for most elements given that the project's early-age strength criteria can be met and sufficient moist-curing can be assured. For flat work, the finishing requirements can determine the level. If adequate healing cannot be provided or the amount of fly ash should be restricted (for example, 25 percent) (Obilade. 2017) if concrete is exposed to de-icing salts, freezing and tailing is required.

2.6 Nano-Silica Cement Replacement Material in Mortar and Concrete

Several researchers have studied the impact of Nano-silica on pastes, mortar and concrete. Nano-silica-integrated cement pastes was analysed to consider the hydration cycle and the microstructure evaluation. This technique is psychologically healthy and is used to research the essential science behind cement hydration. The properties of fresh and hardened states are being studied by mortar tests.

2.6.1 Fresh Properties:

Before they are begin used to set, the term fresh property means the wet mix of concrete and mortar ingredients, these properties include time, temperature, workability, and consistency.

2.6.1.1 Setting Time

The effect of adding Nano-silica to cement paste was investigated by Senff *et al.* (2009), and the initial and final setting cycle decline was observed due to the Silica-Nano coating surface Qing *et al.* (2007) made the same statement in (2007). The first and final classification span of concrete was continuously decreased, including some Nano-silica percentages. In addition, the findings of the Nano silica (NF) and Nano-silica (NS) analysis have been stated by Qing *et al.* (2007), showing that the time and strength of NF and NS integrated into concrete or moulds vary, with the NS

thickening the cement pulp and speeding up the cement hydrate compared to the SF mortar and concrete.

However, Jemimah *et al.* (2012) shared a contrary view where they studied the influence of Nano-silica materials (Nano-silica, Nano-silica fume and Nano-fly ash) on consistency and setting time and noted that consistency is not affected by the presence of Nano-substances. They further said setting times (initial and final setting time) are to a greater extent influenced by the presence of Nano material.

2.6.1.2 Workability:

Workability defines the ease with which concrete or cement mortar can flow naturally, the volume of water in the mix often influences that. It was widely agreed that the more water being applied to the concrete or cement mortar paste, the weaker the end product during the hardening process. However, the incorporation of pozzolanic materials like Silica-Nano Particle demonstrated a tremendous effect on the workability of cement material without necessarily increasing the amount of water in the mix. The effect of 2% Silica-Nano Particles on concrete has a high impact on the workability and concrete setting time (Givi & Rashid, 2011). reported high workability of cement mortar with a higher percentage of Nano-silicate and a decreased proportion of water binder ratio (W / B), the authors suggested that 7 percent addition of Nano-silicate would be of great benefit to cement mortar mixture.

2.6.1.3 Heat of Hydration of Paste and Concrete Containing SNP

Hydration is a chemical reaction between water and cement. This process is usually exothermic in nature, in which heat is released into the environment known as hydration heat. This becomes evident if newly mixed cement put in a vacuum bottle and mass temperature is read out at intervals. In concrete mortary studies, hydration

heat and control is critical.

The hydration process of three-calcium silicate (C_3S) cements and other pozzolanic materials was explained by Bjornstrom *et al.* (2004) and colloidal Nano-silica (CNS) acceleration effects and water hydration function were identified. During the analysis the breakup of the C_3S phase was accelerated by CNS and thus the swift formation of the C-S - H phase was observed. If Silica-Nano particles are mixed with cement-based materials, the new materials can have some exceptional properties. The NS is capable of reacting in the interfacial transition zone (ITZ) with C-S - H-gel Calcium Hydroxides crystals arranged between hardened cement pastes. Thus the CH crystals are considerably reduced in dimension, volume, strength and the hardened cement paste is strengthened in early ages.

Likewise, Ye *et al.*, (2007) analysis showed that concrete integration times and consistency were different for SF and NS but NS densified cement paste and accelerated cement hydration compared to NS, showed improved compressive force and bonding strength. Furthermore, Jo *et al.* (2007) also observed that hydration heat showing the amount of CH created through the addition of NS could boost the heat generated during cement setting and hardening..

The combined effect has been stated by Nazari et al. (2010) of silica-Nano particles and GGBF, and the result shows that Nano particles decrease the amount of concrete water absorption, thereby making hydrated product formation faster.

2.7 Harden Properties

2.7.1 Density

Morsy *et al.*, (2010) describes the density of the material as the mass by volume of the object. Experimentally, air weight or unit weight can be determined by BS EN 12390-7:2009. The density represents the mass total of all the ingredients, divided by filled

mortaring or concrete volume. According to ASTM C 140, 2003, the normal weight of a mortar / ceiling sample of more than 2000 kg / m^3 has been identified. The so called lightweight mortar or concrete are those of less than 2000 kg / m^3.

2.7.2 Compressive Strength

Compressive concrete and mortar is the strength of the hardened concrete measured in the compressive test. The ability to withstand loads to compact concrete is also assessed. However, Shetty (2015) said that mortar / correct strength is the resistor to rupture that can be calculated in various ways, such as strength of compression, stress, shear and bending. Has suggested that compressive strength of mortar / concrete is a big feature because it directly relates to many other features. Concrete strength is also partly determined by the relative proportions of cement, fine and coarse components. Compressive strength factors affecting concrete include: shape, quantity and quality of cement, the admixture material, compaction rate, water-cement ratio, age, curing conditions. The compressive strength of concrete is usually calculated by the cube test. This is achieved on a cube that is divided into an electrical or manual control unit (Neville, 2012).

Li *et al.* studies conducted (2004) the mechanical properties of Nano-Fe_2O_3 and Nano-SiO_2 cement mortars were investigated and the strength of 7 and 28 days was found to have been much higher than the strength of single concrete. Jo and Al (2007). The characteristics of Nano SiO_2 cement mortar were studied also and higher intensity was found on the blended mortar between 7 and 28 days. Nill *et al.* (2009) similarly combined effect on concrete, micro silica and colloidal Nano silica and found out it comprises 6 percent micro silica and 1.5 percent Nano silica, to reach optimum pressure strength. Moreover, Givi *et.al.* (2010) Silica-Nano particulate effects on scale have been investigated. Cement was replaced by 15 nm

Nano silica and 80 nm 0-5, 1, 1, 1, 5 & 2 percent with cement weight (b_{wc}). A cumulative compressive strength of 1.5 percent b_{woc} was observed to increase compressive strength. The maximum resistance of the 80 NM particles to 15 NM particles was seen in a comparison between particle sizes and a significant improvement in the Nano SiO_2 bending and split tensile power.

Likewise the effect of Polypropyrene (PP) fibre is studied by Byung, *et al.* (2007) along with Nano SiO_2 particles. Up to 7% was substituted for the Nano SiO_2, this raised cement mortar's compressive strength by 6. 49%. 49% PP fibre quantities above 0.3 percent are less compressive but bending power is increased below 0.3% dose of PP fibre, which indicates that Nano SiO_2 particles are less effective. Diminishing mortar water consumption, suggesting pore refinement, up to 0,5% of the PP fibres.

However, Nazari *et al.* (2010) studied Nano SiO_2 and GGBFS's combined effect on concrete properties. Nano silica with b_{woc} substitution of 3% and GGBFS b_{woc} of 45% indicating an improvement in strength of the split tensile, there has been an increase in the porous silica particle SCC structure.

The author investigated the impact on SCC concrete by ZnO_2 Nano particles with a constant ratio of 0.4 w / c. The findings have shown that a super plasticizer's flexural strength is decreased as its content is increased. Up to 4% of b_{woc}. ZnO_2 was observed as an improvement in the bending strength of SCC. Furthermore, Nazari *et al.* (2010) explored Al_2O_3 Nano particles' effect on concrete properties in another experiment. The results show that up to 2% cement can be replaced to improve mechanical characteristics of concrete, but the proportion of concrete water absorption was decreased by Al_2O_3 Nano bits. Sample XRD research revealed that the hydrated material grows faster.

2.7.3 Durability

The quality of concrete subjected to harsh conditions is primarily dependent on the properties of transport, which are determined by the pore mechanism. Durability under water absorption and resistance to abrasion is discussed in this study.

2.7.3.1 Water absorption

As a test for permeability, ASTM C642 (2013) defines absorption as a mechanism in which liquid or fluid passes into a solid body and fills the porous medium, such as paste, mortar or cement. Water absorption depends primarily on the total volume of pore (Rajput, 2006; Namibia and Ramamurthy, 2007) of the filler type, concrete density and permeation systems defined. The water absorption was defined by Castro *et al* . (2011) as a capillary suction capability in water for cement samples. Low water absorption concrete provides better protection for the construction of the reinforcement. Pitroda and Shah (2014) reported a minimum of 5% on the average absorption of concrete tests and a maximum of 7% on the individual specimens. Tao (2005) water absorption experiments have been performed in compliance with ASTM C 642-13 with concrete containing RHA up to 30 percent. The author has claimed that the water absorption coefficient decreased when concrete containing RHA is replaced by all percentages. Castro et al. (2011) have suggested that the absorption of water is an significant factor in the quantification of longevity of cement systems. Specifications are used to include a parameter in scientific studies that can define an element of concrete drug intake resistance.

2.7.3.2 Abrasion Resistance:

Concrete and mortar abrasion resistance is directly related to strength, while rising resistance is mainly due to an upsurge and decline in cement content w / c ratios. However, Lahri & Dixit (2015) proposed that abrasion refers to wear or tear because

of small objects or small outbursts that are forced to shift toward a solid surface. It can thus be regarded as the ability of the surface to resist wear, rub and friction.

Wear of concrete surface can be classified as follows:

i. Wear on concrete floors.
ii. Wear on concrete road surfaces due to heavy trucking, and automobiles, with and without studded snow tires or chains (attrition, plus scraping and percussion).
iii. Wear on hydraulic structures such as dams, spillways, bridge piers, and abutments due to the action of abrasive materials carried by flowing water (erosion).
iv. Wear on concrete dams, spillways, tunnels, and other water-carrying systems where high velocities and negative pressure are present. This is generally known as cavitation erosion (cavitation) (Castro *et al.*, 2011).

The factors to avoid abrasion due to scratching, scouring, gliding, impact, grinding, erosion, perk, gouging, or mechanical or hydraulic forces must be taken into consideration when designing and constructing concrete surface. Abrasion-resistant failure can be due to long term effects such as soft aggregates, poor compressive power, improper cure, finishing and manipulation in concrete surfaces (Damtoft et al., 2008)

2.8 Microstructure:

The microstructure of cement matrix is a function of cement mortar composition and a measure of resistance properties without actually destroying the specimen Cement mortar micro-structure analysis is performed using XRD, SEM, and EDX. SEM micrographs are characterized in terms of the homogeneity of the cement matrix, the voids and the presence of flaky crystals, while EDX analyzes show elementary matrix

compositions used to determine the hydrate content. Whereas XRD peaks and their intensity give insight into the different interactions and compound formations The SEM-EDX and XRD analysis is performed and then corresponds with the specific mechanical strength and durability tests to study the effect of adding or substituting a binder material with SCMs.

2.9 Summary

Previous work on silica Nano particle in mortar and concrete is centred on cement addition or partial replacement. Among the recent study, the mechanical properties of cement mortar Nano-$Fe2O_3$ and Nano-SiO_2 have been investigated by Li *et al.* (2004), and the strength of 7 and 28 days was found to be substantial greater than that of single cement. The microstructure analysis reveals, because of the pozzolanic reaction, that the pores filled with silica Nano particles and the reduced $Ca(OH)_2$ material. Similarly, the impact of silica Nano particles on water permeability and concrete micro-structures was experimentally studied by Tao (2005). The results show that the inclusion of silica-Nano particles will increase the water-penetration resistance in concrete and that the microstructure becomes stronger denser than ordinary concrete.

Alirza *et.al*. (2010) therefore, analysed the effect on their size of silica Nano particles. They substituted cement with 15 nm of SNP by a weight of cement (bwoc) with 80 nm by 0-5, 1, 1, 5 and 2%. The compressive strength increased with a maximum compressive strength of 1.5% bwoc was observed. Compared to 15 nm particles, the maximum resistance of 80 nm particles and major increases in flexural and separate traction resistance of SNP mixed concrete were also seen in a comparison between particle sizes.

Besides this, Morsy *et al.* (2010) along with SNP, the effect of fibre was also studied.

The SNP has been replaced by 7%, which increased compressive strength of cement mortar by 6.49%. Fibre volumes decreased by 0.3%, but fibre dose increased by over 0.4% and revealed the effectiveness of Silica-Nano-Particulates. Fibres also decrease up to 0.5% in water absorption in mortar, indicating pore refining.

Next, Nazari *et.al*. (2010) investigated the combined effect on concrete properties of silica Nano particulate and of GGBFS. The SNP replacement of GGBFS was used to show increased force in divided tensile structures by 3 percent b_{woc}. and 45 percent b_{woc}) There has been an increase in the porous structure (SCC) with silica Nano particles. Givi *et al.* (2011) are examining the effect of silica Nanomaterial on the compressive, splitting and bending characteristics of concrete (water permeability, workability and set-up) of Nano SNP mixed concrete which has significantly more compressive properties up to 2 percent split strength and flexural strength against standard splitting strength and strength are considered to be significantly greater. Another result was that partial substitution of silica Nano parts limits the working potential and the setting of fresh concrete times for lime-solvent samples. Likewise, said *et.al*. (2012) by combining silica Nano particle with fly ashes groups, the effect of silica Nano particles on concrete and by adding variable amount of SNP, the effect of silica Nano ash was investigated and significantly improved. The combination of 30% FA and 6% SNP dramatically improves power. SNP-containing mixtures had slightly less porosity and average pore diameter. The RCPT test indicates that passing charges and physical penetration have been greatly enhanced. Likewise Dhinakaran *et al.* (2014) analysed silica Nano particulate concrete microstructure and Resistance properties. In the planetary ball mill the silica was soiled until Nano size was reached and mixed with concrete of 5 %, 10% and 15% b_{woc}. Experimental findings showed an increase in compressive force with maximum strength at a

substitution of 10%. The importance of this area of study can be seen by analysing a variety of literatures. Results show that a substantial amount of Nano materials including SiO_2, TiO_2, Al_2O_3, colloidal Nano silica, Metakaolin and others are possible to be applied in order to enhance concrete features. In terms of fresh and hard properties the findings of various authors indicate an improvement of the characteristics of the mixed concrete. Furthermore, features such as the permeability of the specimen can be enhanced by adding a small proportion of the Nano material. The improved microstructure with decreased pores, such as SEM, XRD, and other tests. This study focuses on the addition of RHA-developed Nano-SiO_2.

CHAPTET THREE

3.0 MATERIALS AND METHODS

3.1 Materials

The materials used for this study includes

a. Calcium Carbide Waste (CCW) was source from local automobile mechanic site located at Bida opposite Minibell Guest House
b. Rice husk ash (RHA) was source from Nigeria Cereal Research Institute (NCRI) Baddegi.
c. Silica Nano particle (SNP) was exacted from RHA.
d. Fine aggregate was obtained from borrow pit located in Bida,
e. Superplasticizer composing of poly-carboxylate ether (PCE) was used as water reducing agent

3.1.1 Calcium Carbide Waste

Slurry form calcium carbide waste (CCW) was collected in the waste area of the local car welder's workshop opposite the Mini Bell Guest House in the Bida Local Government Area. CCW was collected in an acetylene gas development by way of a by-product.

The CCW has high moisture content because of its exposure to the natural whether condition (approximately 52 %), it was sun-dried for approximately 3 days to reduce the moisture content. After which, it was ball milled using Los Angeles Abrasion Machine located at Civil Engineering Laboratory of The Federal Polytechnic, Bida, and the grounded materials were sieved with 75μm sieve to obtain fine particle comparable to cement texture.

3.1.2 Fine Aggregate

The fine aggregate for this study is the simulated sand of reference size, which was selected from the actual natural sand obtained from a borrowed pit in Kuso Tachin in the Bida Local Government, Niger state, in accordance to the BS EN 196-1:2016 bonding intensity test referencing sand prescription (BS EN196-1:2016)

3.1.3 Rice husk Ash (RHA)

Rice husk (See Plate 1, Appendix C) was collected from National Cereals Research Institute (NCRI), Badeggi located in Bida, Niger State. The material collected was washed to remove impurities and was sun-dried to remove the moisture to enhance proper combustion using the furnace available at NCRI, Badeggi (Plate 3, Appendix C) to produce the rice husk ash (Plate 2 Appendix C).

3.1.4 Water

The water used for the production and curing of mortar samples of this research work is clean potable water available at Building Laboratory of the Federal Polytechnic, Bida.

3.1.5 Admixture

This research has used a high-end water-reducing mixture (HRWA), a super plasticising agent with the brand name MasterGlenium ACE 456. By allowing an expanded area of the cement grains to be reacted with water, MasterGlenium ACE 456 accelerates cement hydration. In this way, the heat of the water is produced earlier, the hydration products can be quickly developed and thus the qualities can be enhanced at an early age. After the addition of 80-90 percent of the mixing water into the blender, MasterGlenium ACE 456 is applied to the mortar as an optimum effect. It is used for every 100 kg of a binder, from 0.3 to 2.0 litres. Type A, E & F of the BS

EN 933-2:2009 and (ASTM C494, 2015) are fulfilled by the MasterGlenium ACE 456.

3.1.6 Silica Nanoparticle production

Acid Leaching method was used to remove silica Nanoparticles from rice husk ash (RHA). In a concentration of 300 N for 2.5 hours, the ash was immersed in the hydrochloric acid solution at constant string at room temperature. The acid solution was subsequently drained out. The RHA was purified with water and then filtered up to 7 pH and dry air for further use in the desiccator.

3.2 Experimental Plan

The experimental plan is designed as follows to be able to achieve the stated objectives.

3.2.1 Work Plan One

Work plan one is designed to achieve objective one which is study of the material characteristics of RHA, silica-Nano-particle (SNP) and CCW. The specimens (RHA, SNP and CCW) were packaged and sent to Ewekoro Works Department of Lafarge Cement Laboratory to carry out X-ray Fluorescent (XRF). However, particle size analysis (PSD), on silica-Nano particle (SNP) and Brunauer-Emmett-Teller (BET) specific surface area, were conducted at Centre for Genetic Engineering and Bio-informatics Technology of Federal University of Technology Minna.

3.2.2 Work Plan Two

Work plan two was designed to achieve objective two which deals with determination of appropriate proportions of the constituent materials for mortar and appraise the fresh properties of mortar made with RHA-CCW and SNP

i. This involves making the necessary combination of the materials of RHA and CCW at 60:40; i.e. (RHA: CCW) with varied content of SNP (0.5 % to 5 % at 0.5 % steps). Ten SNP contents were studied with RHA-CCW binder. While RHA-CCW without SNP was also examined as the control.
ii. Various tests on cement (consistency, setting time, soundness, pH test and fineness test) were carried out on the binder combination types and the control.
iii. Workability, consistency, setting time, and fineness and soundness was tested at various mix proportion.

3.2.3 Work Plan Three

Work plan three was designed to achieve objective three which deal with examining the effect of SNP on the mechanical and durability properties of mortar produced from the binder combination. The properties investigated here were examined using the following tests and specimen's sizes:

i. Compressive strength – 50 mm cubes at 3, 7, 14, 28, 56 and 90 days curing ages.
ii. Water absorption – 50 mm cubes at 28, 56 and 90-day curing age.
iii. Abrasion resistance – 50 mm cubes at 28, 56 and 90-day curing age.

3.2.4 Work Plan Four

Work plan four was designed to accomplish objective four which deal with examination of the microstructure of the mix proportion studied. Specimen obtained for 0 and 3% SNP at 90 days curing were used for this examination and the test carried out was SEM-EDX of the hydrated (hardened) mortars obtained from the crushed compressive strength specimen.

3.3 Method

This describes the methods employed to conduct the experiment in accordance to relevant standards.

3.3.1 Preparation of Test Specimen

The mixed proportion of the materials used for the experiment is shown in Table 3.1. The quantity of all components mixtures is also provided for total percentage of SNP contents of 33 numbers of 50 mm x 50 mm cube (Plate 5, Appendix C). For compression force checks of 3, 7, 14, 28, 56 and 90 days, three cubes were cast and cured by immersion into water (Plat 6, Annex C) from each combination, totalling 198 mortar cubes. For alternative binders of varying proportions RHA / CCW and SNP combinations, batching or mixing the samples of mortar were performed with a constant 0.5 W / B and an HRWR of 1.5% defined in the BS EN 196-1:2016.

Table 3.1: Mix details for cube samples with HRWR

	Mass of Materials/Cube (g)					
SNP %	SNP	RHA	CCW	Sand	Water	SP
0	0	1399.5	933.0	6997.3	1141.13	35.9
0.5	11.7	1392.9	928.3	6997.5	1141.13	35.9
1.0	23.3	1385.5	923.7	6997.5	1141.13	35.9
1.5	34.9	1378.6	919.0	6997.5	1141.13	35.9
2.0	46.7	1371.5	914.3	6997.5	1141.13	35.9
2.5	58.3	1364.5	909.7	6997.5	1141.13	35.9
3.0	69.9	1357.6	905.0	6997.5	1141.13	35.9
3.5	81.6	1350.5	900.4	6997.5	1141.13	35.9
4.0	93.3	1343.5	895.7	6997.5	1141.13	35.9
4.5	104.9	1336.6	891.0	6997.5	1141.13	35.9
5.0	116.6	1329.9	886.4	6997.5	1141.13	35.9

3.3.2 Experimental Procedures

Test procedures included in this analysis are divided into four categories, with

sufficient attention paid to particular experiments (materiel characterization, early-age abilities, and mechanical properties).

3.3.2.1 Production of RHA

Rice husk (RH) was collected at NCRI Badeggi Bida Niger State's local rice processing facility, while at room temperature 25^{o}C, air was dried for 24 hours. In an electronic fireplace, the air-dried material was then calcinated for around two hours at 700^{o}C. Before the sample was removed, the stove was automatically reset to room temperature. The ash was then pulverized by Los Angeles Abrasion Machine for 90 minutes. For further material characterisation tests the base material was placed in a desiccator separately.

3.3.2.2 Silica Nanoparticle Production (SNP)

Silica has been removed by leaching process (water and acid) from rice husk ash. The ash was immersed at 300 N at 2, 5 h with constant string at room temperature in the solution of hydrochloric acid. Thereafter the acidic solution was drained off. The RHA was rinsed with distilled water until the pH value rose to 7 then filtered and air dried and kept in desiccator for further use. Similarly, the rice husk ash sample was leached using water. The sample was soaked in boiling water at 100 ^{o}C for 10 hrs. And they filtered and air dried at room temperature

3.3.2.3 Characterization

RHA silica Nano particle chemical analysis was conducted using the Philips PW 1480 spectrometer X-ray fluorescence (XRF). RHA silica crystalline structures were tested using GBC-Difftech X-ray (XRD) diffraction spreading MMA. The angle and strength of diffraction were tested from 20 to 90 (2Ø). A BET method and the average particle size were used to calculate the real surface area of the RHA SNP.

3.3.2.4 Sieve Analysis of Materials

The natural sand available was distributed by particle size using a dry-sieve system to better characterize the natural Sand according to BS EN 196-1:2016. Different size sieves (1,18 mm, 2.36 mm 4.75 mm, 75µmm, 150µm, 300µm, 600µm, scooping, stitching brush, weighing balance, mechanical shaker, weighing balance and stop watch were the instruments used. In the strength test, laid down by BS EN 196-1:2016, the appropriate reference sand was subsequently extracted by using a set of seven sizes 1, 18 mm and 75 µm. In order to measure the strength of particles through a 1, 18 mm sieve on the 75 µm sieve, the mortar mixture was used. The 1.18 mm sieve was used as its top limit for the simulated reference-sand instead of the 1.6 mm sheet as specified by the BS EN 196-1:2016 because the 1.6 mm sieve was not available in the lab. The results from a semi-log graph of the particle diameter or size were drawn in the form of the x-axis with log axis and the rate that passed as the y-axis gives a good idea as shown in Figure 4, 1 of the particle size distribution. D60 is 60 percent grain diameter, D30 is 30 percent grain diameter and D10 is 10 percent grain diameter.

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

$$\text{Coefficient of Curvature } (Cc) = \frac{D30^2}{D10 \; x \; D60} \tag{3.2}$$

Thus, a highly graded demand was made, such as Cu ≥ 4 for gravel; Cu ≥ 6 for sand; and Cc= 1-3 for all soil types. Soils with Cu < 2 are uniformly graded. The soil classification Vandevelde (2008) has been confirmed to be both Cu and Cc.

The following limitations for the Fineness Modulus (F.M.) of sand are provided by Shetty (2015) for concrete works.

Fine Sand, F.M.: 2.2 – 2.6

Medium Sand F.M.: 2.6 – 2.9

Coarse Sand F.M.: 2.9 – 3.2

Sand with F.M > 3.2 is not fit to make strong concrete.

3.3.3 Fresh Properties of RHA/CCW and SNP

The consistency test, setting time and soundness were used for testing fresh properties.

3.3.3.1 Consistency Test

The goal is to calculate the percentage of water in a given sample of specimen combination for normal consistency. Vicat devices were used with 10 mm diameter plungers, measurement plug, weighting balance, weight box and trowel. The amount of water in the paste providing the desired consistency was determined (Neville 2012). ELE was employed for consistency measurement according to the procedures defined in the standard (BS EN 196-3:2005) by Vicat apparatus MODEL No EL 38-2010. 250 g of binder was taken and weighted, before adding 20% water. The mixing period was made not to exceed 3 minutes and time reading taken from the period water has been applied. The paste was then filled into the pot and excess paste used to decrease and vibrate to eliminate air bubbles. The dia was then set to 10 mm and the moveable rod (Plunge) positioned to allow placing the paste in contact with it. The process was repeated until the water percentage penetrates between 33 and 34 mm from the surface.

3.3.3.2 Setting Time Test

The goal is to determine the beginning and end time of the concrete sample. Vicat instruments are mould and non-porous plates, needles, balance of weight, weight box, cylindrical measurement and stopping watch. The initial and final binding times tests were performed with the use of straight standard consistency pastes in compliance with BS EN 196 3:2005 at various proportional RHA / CCW / SNP combinations.

The water content of the paste producing the necessary quality consistency was determined (Neville, 2012) Vicat system Model No EL 38-2010 ELE used consistency measurements and the start and finish times as defined in the standard (BS EN 196-3:2005) to calculate consistency and time. In order to render a regular paste consistency, water, super plasticiser and SNP were applied to around 300 g binders.

3.3.3.3 Soundness Test

Soundness, test in accordance to BS EN 196-3-2005 was carried out. Behaviour of cement paste in the time of expansion and contraction is to be determined. Le-Chatelier mould and bath, glass tile, spa and vernier calliper are the appliances used. The test was performed by Le Chatelier Model No. EL 38 – 3400 from ELE on the respective binder. The binding paste was placed in Le Chaterlier, and the mold was submerged in a water-filled Le-Chaterlier bath and the immersion times were recorded and the mould tested 24 hours later. The expansion reading was done with the Vernier calliper and registered. The sample was taken off the mould and returned to a bath full of water and heated for approximately three hours, during which calliper was used to test the quality of the sample.

3.3.4 Hardened Properties Tests

The mechanical properties of the mortar cubes was determined. These entails testing triplicate cube samples for respective specimen at the specified curing ages. The cubes was measured before being subjected to test determine the weight of the spacemen:

3.3.4.1 Density Test

The specimens of mortar were extracted from the treatment container and placed outside on a dry surface. They were measured using the measurement balance

according to BS EN 12390-7 (2009). The density was calculated of single specimens, using equation 3.70.

$$\text{Density D} = \frac{M}{V} \quad (3.7)$$

Where D is the density of the mortar specimen in kg/m^3

M is the mass of the mortar specimen in kg

V is the volume of the mortar specimen in m^3

3.3.4.2 Compressive Strength

The compressive force is a key aspect of the mortar that has many features connected to it (Neville, 2012). The mortar samples (cubes) were taken from the healing tub, cleaned, weighed and placed outside the surface at the middle of the hydraulic manual crushing unit and the power of the exhibition was applied by swinging the handle of the crushing machine until its action was not tested under load (BS EN 196-1:2016)

The RHA-CCW binder based-mortar strength determination was carried out using 50 mm mortar cubes as defined in section 3.2.3. The manufacture of the mortar samples involved the weighing of the required material and making sure that the RHA was properly mixed in a headpan to the CCW, prior to being poured into the steel mixing platform on the specified volume of the simulated reference sand. Before weighting water was applied and blended, the sand and binder were thoroughly mixed until homogenous mix was reached and casted into the 50 mm cube moulds, which had already used mould-oil. Following an observation of set-time tests in Section 4.2.2, the specimen was left covered with jute bags and sprayed with water up to 24 hours before deforestation and immersion cure until the test period was completed.

A strength test protocol included the submission by a automated universal testing system of the handled mortar cubes to a crushing force at various curing ages (3, 7, 14, 21 and 28 days) (see Plate 7, Appendix C).

Compressive strength – 50mm cubes

Compressive strength F = $\frac{P}{A}$ (3.8)

Where; F is the compressive strength in kN/mm^2

P is the maximum load at failure, in k N

A is the cross-sectional area of the specimen on which the compressive force acts.

3.3.4.3 Water Absorption Test

The cubes were then removed and dried from the treatment tank and dried onto an electronic oven for 72 hours, dried at 105^{o}C. Then the samples of the mortar were placed in the oven. The samples were taken from the oven and cooled at room temperature. They were measured and reported as w1 for the initial weight determination. After 30 minutes of immersion in the healing medium, the final weight was determined, and the value was then dressed, dried and recoded and registered as w2. The results were measured and reported to determine the absorption rate of the mortar samples in compliance with BS 1881-122 (2011). The values are estimated.

Water Absorption = $\frac{(w2-w1)}{w1}\ x\ 100$ (3.9)

Where w1 is weight of the sample before absorption and w2 is weight of the sample after absorption

3.3.4.4 Abrasion Resistance Test

During removal from the container, the mortar samples (cubes) were dried and washed and dried on the surface, at 105 ° C to 110 ° C, and values were calculated under w1. Then the sample of the mortar was placed with the necessary steel balls in the Los Angles abrasion test machine and the steel cylinders were rotated at speeds of 30–33 turns / minute for 500–1000 revolutions and a sample was taken and put into

1.7 mm seven and the material that passed through 1.7 mm was weighed with a value of w2. The findings were determined to test the resistance to abrasion of the mortar specimens according to ASTM – C131 (2014)

$$\text{Abrasion resistance test} = \frac{w2}{w1}\ x\ 100\% \quad (3.10)$$

$$\text{or Abrasion resistance test} = 100 - \frac{(w1-w2)}{w1}\ x\ 100 \quad (3.11)$$

Where;

w_1 is the mass weight of the sample

w_2 is the mass weight of fraction passing 1.70 mm B.S sieve

3.3.4.5 Scanning Electron Microscopy

Relevant information on the grade of concrete, degree of cement hydration, composition, and distribution of hydration products, adhesion and homogeneity of cement pastes can be obtained from the Electron Scanning Microscopy (SEM). According to Winter (2012). In this study, the concern is to examine the microstructure and assessing influence of SNP inclusion on the products of hydration of the eco-friendly mortar.

The procedure adopted for the test was as explained by Winter (2012) which entails a cut-off of the crushed compressive strength (of 3% SNP and the control (0% SNP)) specimen tested after 90 days of curing was packaged and sent to the Scanning Electron Microscopy Laboratory of the University of Ahmadu Bello Chemical Engineering Department, Zaria, Nigeria. The specimen was coated at the Laboratory with 5mm coat of gold using sputter coater made by Quorum, United Kingdom before its being subjected to SEM-EDX test using Phenom ProX manufactured by Phenoworld Eindhoven, Netherlands. Results from the test (SEM image, EDX spectra and the weight compositions) are as discussed in Section 4.4.

3.4 Method of Data Analysis

For the graphical and the general model – multivariate analysis by means of the Statistical Package for social sciences (SPSS) – the outcomes of various tests conducted during this research work were analysed.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Material Characterization

4.1.1 Sieve Analysis of Fine Aggregate

The particle size distribution (PSD) plot of real sand and the simulated reference sand is shown in Figure 4.1 while the PSD plot is summarized in Table 4.1. The findings show the uniformity coefficient (Cu) of the increase from 1.82 (for natural sand) to 2.36 (for natural sand simulated while the same gradation coefficient (Cc = 1.1) and fineness module (FM = 1.5) still remain. This implies that the simulated reference sand used for the study is a BS EN 933-1(2012) fine-sand classification.

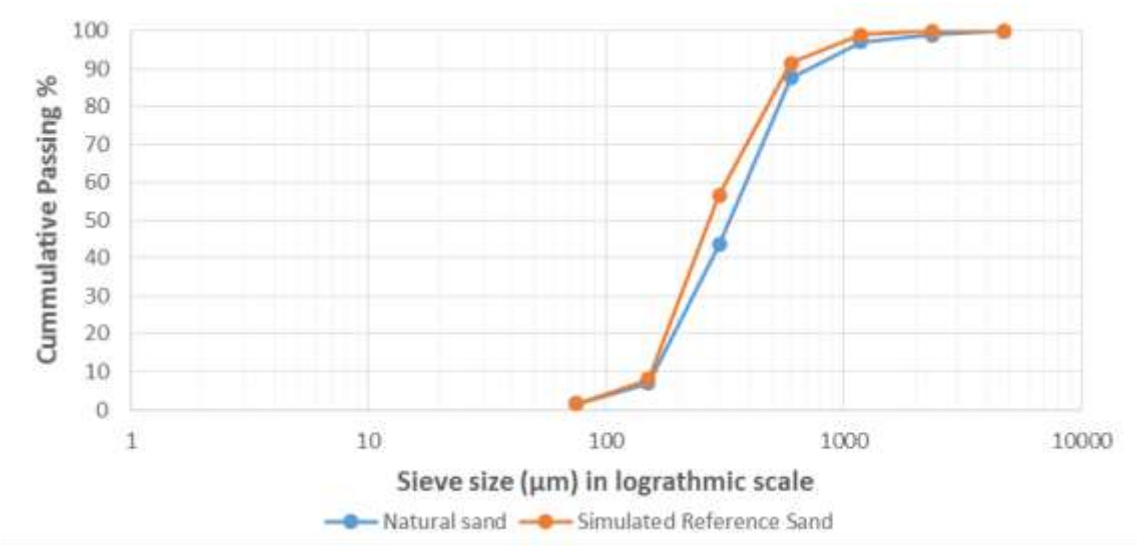

Figure 4.1: Particle Size Distribution of Fine Aggregate

Table 4.1: Summary of Particle Size Distribution of Sand

	D60	D30	D10	Cu	Cc	FM
Natural	**310**	**240**	**170**	**1.82**	**1.09**	**1.48**
Simulated Reference	**400**	**270**	**170**	**2.35**	**1.07**	**1.45**

An examination of the simulated reference sand compared to the CEN sand reference provided for in BS EN 196:1-2016 for PC strength (Table 4.2) revealed the simulated reference sand fits within three of the six particle size ranges specified for the CEN

reference sand. Nonetheless, the properties observed for the simulated reference sand sample are identical to those reported for the sand used in Enejiyon's research (2018), thus the findings for the current study can be related effectively to those reported therein.

Table 4.2: PSD of reference Sand (Simulated as compared to CEN)

Sieve opening (mm)	CEN Reference Sand (%)	Simulated Reference Sand (%)	Remark
2	0	0	√
1.6	7 ± 5	0	
1	33 ± 5	1	
0.5	67 ± 5	9	
0.16	87 ± 5	92	√
0.08	99 ± 1	98	√

4.1.2 Chemical composition of RHA, SNP and CCW

The chemical composition of RHA, SNP and CCW It is presented in Table 4.3 for comparison purposes.

Table 4.3: Oxide composition (%) of RHA, SNP and CCW

Oxide composition	RHA	SNP	CCW
SiO_2	83.79	93.40	8.6
Al_2O_2	0.54	ND	1.10
K_2O	1.15	0.02	0.10
CaO	1.26	ND	65.59
T_1O_2	0.26	0.01	0.12
MgO	1.55	0.28	0.05
Fe_2O_3	1.38	ND	0.30
LOI	2.93	2.27	21.70
SiO_2+Fe_2O_3+Al_2O_3	85.71	93.4	10.00

It can be seen from Table 4.3 that both RHA and SNP have identical chemical properties because of the same substance. The SiO_2 content increased from 84.8% (for RHA) to 93.4% for SNP, while the ignition loss (LOI) also dropped from 2.93% to 2.27%. The CCW sample in SiO_2 is obviously small but mainly CaO (65.6%) and a

LOI of 21.7%. SNP's higher SiO_2 content and lower LOI value may be attributed to chemical leaching, where heavy metals have been de-ionized in the specimen. Farshid *et al.* (2015) published a comparable result. The higher silica content is expected to influence the pozzolanic reaction using CCW's free CaO and thus create additional C-S - H gels that are beneficial for the development of strengths in mortar or concrete. Low Al_2O_3 and Fe_2O_3 content in the SNP specimen was seized to exist in the RHA sample. It can be determined that the acid used for chemical leaching reacted with these oxides to form another compound. The sum of useful oxides (SiO_2 + Fe_2O_3 + Al_2O_3) in both the RHA and SNP samples is well above the minimum specification of 70 percent for ASTM C618 (2015), which makes this pozzolanic material qualified for code classification as Class F Pozzolan.

The content of CaO stands to be very low in RHA (1.26 percent) and seizes for SNP to exist. With the CCW, a value similar to PC CaO content, the CaO content (65.6 percent) stands to be a benefit obtained. Enejiyon (2018) reported a similar finding, in which CCW CaO content was reported as 65.8 percent. The presence of these Oxides provides advantage when the constituent materials are mixed together for early resistance benefit. In other words the free CaO in CCW reacts with the SiO_2, Al_2O_3 and Fe_2O_3 forming a substance like a jelly.

The RHA and SNP LOI obtained are both below the specification code; however, CCW's LOI was higher than the minimum requirement. Other compounds, including trace elements such as Potassium oxide (K_2O), MgO and titanium oxide (TiO_2), as found in RHA and SNP, are lower than expected and comply well with the standard specifications, while LOI values were also found to be lower than the maximum values of 7% and 12% in BS EN 196 (2016) and ASTM C618 (2015) for both Class C and Class C.

4.1.3 Structural Features of Silica Nano Particle

Table 4.4 explains the structural properties of the silica Nano sample in terms of average particulate size, BET surface area, total pore ability and average pore length (Platte 3, Appendix C). The table shows that, when the liquid treatment is made of silica Nano particles (snails), it greatly impacts the surface area and the RHA sample porous thickness. The average diameter of the pore is 2,647 nm, which means that it is primarily mesoporous. The SNP pore volume is 0.255 cc / g suggesting that the treatment does not adversely affect the volume of the pore as stated by Xu *et al.* (2018) Low calcium (class F) is preferred ash as a low-cost by-product from coal-fired power stations and traditionally reduces the likelihood of alkali-silica reactivity as compared to class C fly ash. (Farshid *et al*, 2015)

The hydrolysis of lignin and cellulose into lesser materials, and the dissolution after leaching of alkaline metals, which helps to volatilize fixed carbon of RHA during combustion, contributes to this structure. Furthermore, the elimination of organic carbohydrates causes the opening of the internal pores of RHA to a less porous RHA framework.

Table 4.1: Structural Properties of Silica Nano Particles

RHA Sample	Average Particle size (nm)	BET surface area (m^2/g)	Total pore volume (cc/j)	Average pore width (nm)	Microspore value (cc/j)
Chemical leaching	65.31	574.505	0.255	2.647	0.291

4.2 Fresh properties

4.2.1 Standard consistency

Figure 4.2 shows the effect of normal consistency with various proportions with SNP content in RHA-CCW. It can be seen from the figure that water and super plasticizer required remained constant, with varying proportion of SNP. Given the increase in

SNP content, the plunger penetration value was observed to be within the 5-7 mm range defined by ASTM C191 (2013). It can therefore be deduced that the addition of SNP has a negative impact on the paste's consistency.

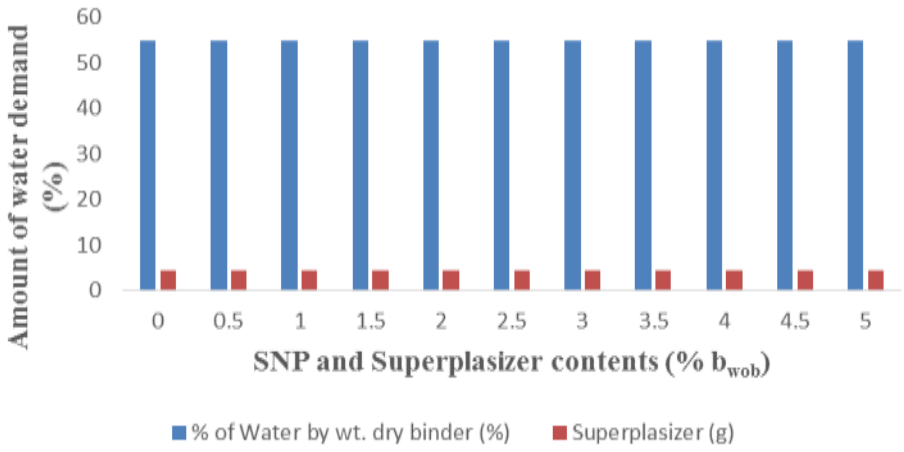

Figure 4.2: Standard Consistency of the Binders

4.2.2 Setting Time of the Binder

SNP is well known to influence the set retardation action of pozzolanic or cemented materials. The setting time behaviour of RHA-CCW paste containing SNP is shown in Figure 4.3. The test time was measured with a needle penetration of 5 mm and 7 mm into the paste respectively for initial and final setting time. As expected, the greater setting times for mixtures with lower SNP content were obtained. The initial setting time for 0 per cent SNP paste (as control) for example, was 195 minutes while the actual setting time was 360 minutes.

The setting time was reduced at SNP inclusion. The setting time also decreases with rising SNP material. Although, with the inclusion of SNP, the initial and final setting time decreases, the results obtained fall within the time range defined by BS EN 196-3 (2016). The level of super plasticizer and the pozzolanic binder (RHA-CCW) and SNP inclusion could have influenced this performance of SNP. Senft *et al.*, (2009) observed a similar reduction in Initial time setup (IST) and Final time setup (FST) for

paste setup, concluding that adding SNP influenced the paste behaviour. Lilifi *et al.*, (2011) and Ye *et al.*, (2007) also shared the same view, reporting that as the SNP increases so the IST and FST also decreases.

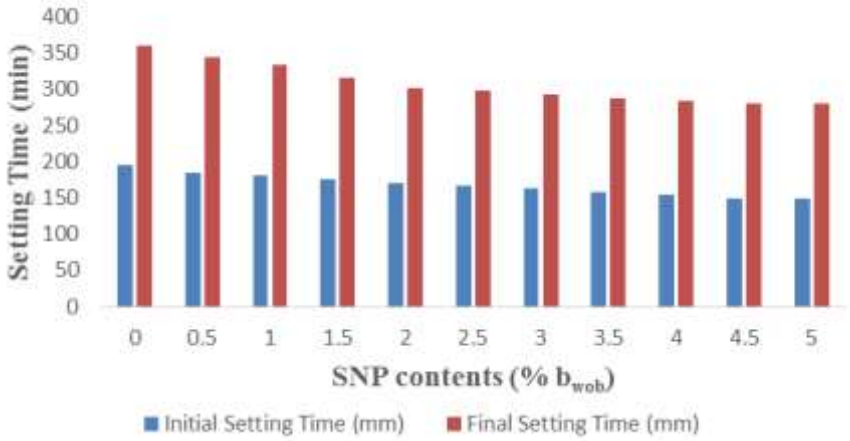

Figure 4.3: Influence of SNP contents on Setting Time of RHA-CCW Binder

4.2.3 Soundness of the Binder

To determine the soundness of mortar made from RHA-CCW binder incorporating SNP, the Le-Chatelier method described in the previous chapter was utilized. The results obtained showed less of an expansion effect on the RHA-CCW paste. The average expansion obtained with SNP included as shown in Figure 4.4 was 1 mm. The results for all the SNP contents examined fall within the BS EN 196 (2016) standard specified maximum expansion of 10 mm.

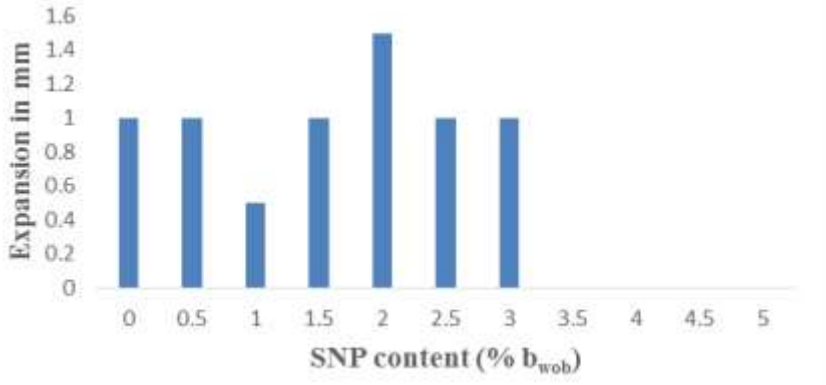

Figure 4.4: Soundness of the Binder

4.3 Hardened State Properties

Results of hardened mortar properties made from different SNP contents in the RHA-CCW binder are discussed as follows;

4.3.1 Density of RHA-CCW and SNP Mortar

In Figure 4.5 the effect of varied SNP content on average density of RHA-CCW binder-based mortar cured in water and weighted at 3, 7, 14, 28, 56, and 90 days is shown. The density of mortar cube spacer's ranges from 2072 kg / m^3 to 2231 kg / m^3, and increases with healing ages. According to ASTM C 140 (2003) the regular weight mortar is called mortar samples with density exceeding 2200 kg / m^3. Likewise, those with a density below 2000 kg / m^3 can be classified as low density. The higher the content of SNP, the higher the mortar sample density. However, the increase in density was minor up to the 2 percent SNP content while the 2.5 percent SNP resulted in a sudden density jump (from 2125 kg / m^3 to 2225 kg / m^3) when it

Became relatively steady again up to the 5 percent SNP and also studied for all the healing ages.

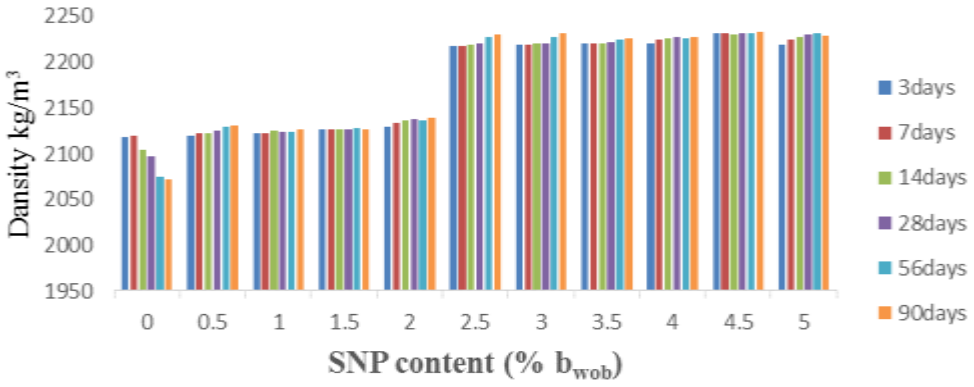

Figure 4.5: Influence of SNP content on Density of RHA-CCW Binder based-mortar

4.3.2 Compressive Strength

The effect of SNP on the production of RHA-CCW binder compressive strength in mortar was investigated and compared with specimen that does not contain SNP. The

compressive strength gain with varying SNP percentage is shown in Figure 4.6, whereas details of the strength data are shown in Appendix A.

As can be seen, the development of strength of the specimen without SNP shows somewhat lower strength compared to those with some proportion of SNP. It can be seen that strength has increased as the percentage of SNP inclusion increases by up to 3%. However, when the SNP content gets higher (i.e. replacement of RHA-CCW with SNP) the strength decreases sharply. It is imperative to note that strength development is of advantage at lower SNP content in the RHA-CCW based mortar. For eg, the strengths obtained in RHA-CCW binder-based mortar are 6.6, 6.75, 6.93, 7.0, 7.50 and 8.50 N / mm2 at .0.5, 1.0, 1.5, 2.0, 2.5 and 3 percent SNP material. These strengths are higher than the SNP value of 0 per cent. Kamar & Singh (2018) reported a similar finding, in which inclusion of SNP increased the strength of PC mortar. These works by Rao & Navaneethawma (2016) also confirm this outcome. The rising strength of the compression is attributable to the SNP filling in the microstructure of pores, which prevents the rise in $Ca(OH)_2$. The SNP also reacts with crystal $Ca(OH)_2$ that converts them to C-S – H gel. Furthermore, the SNP acted not only as a microstructure strengthener, but also as a pozzolanic-reaction activator. (Byung *et al.*, 2007)

The intensity obtained at higher SNP contents (i.e. 3.5, 4.0, 4.5 and 5.0 percent SNP) of RHA-CCW binder based mortar is 7.41, 6.41, 6.21, and 4.99 N/mm^2, respectively. The decrease in strength can be due to the increased SNP level which resulted in lower packing efficiency and thus lower compressive strength.

Figure 4.5: Influence of SNP on compressive strength of RHA-CCW binder-based mortar

4.3.3 Abrasion Resistance Test

The purpose of the test is to identify surface resistance against wear and tear of the mortar made with RHA-CCW binder with or without SNP. This test is particularly relevant in roads and transport where moving forces (load) are responsible for the surface wearing of pavements and floors. The studies were performed in compliance with the Protocol laid down in Chapter 3. Figure 4.7 indicates the amount of surface worn out of cubes in Los-Angeles system as a result of impact load (Plate 8, Appendix C).

The loss of abrasion decreases or the resistance to abrasion increased with increased SNP content and healing time as shown in the Tables. Hui et al. (2006) published a similar result, in which it was published that Nano particle abrasion resistance is better than Nano tube. It can also be deduced from Figure 4.7 that the resistance to abrasion increased as the curing age grew. This can be inferred from the increased SNP content as an effect of the parking performance.

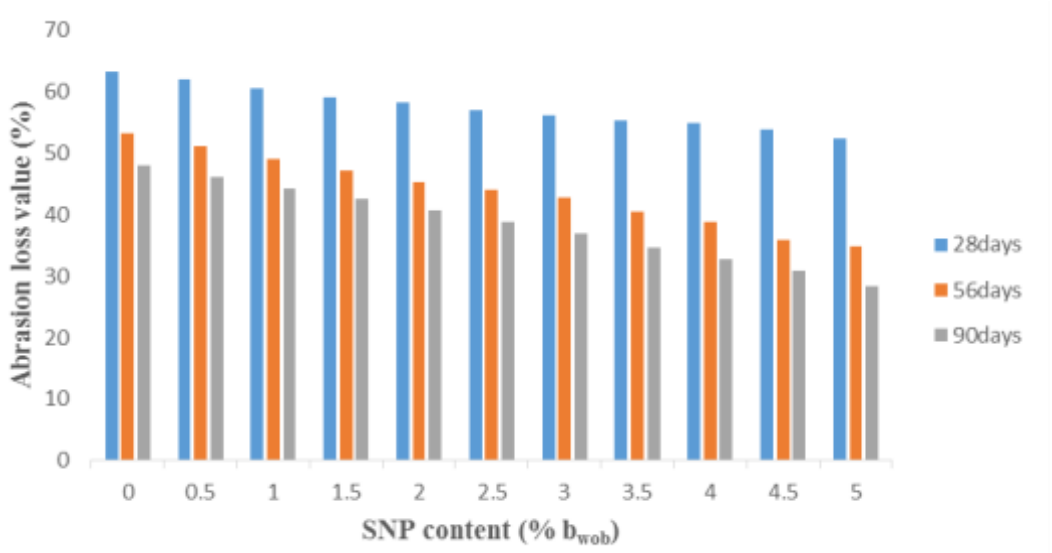

Figure 4.6: Influence on SNP on Abrasion resistance of RHA-CCW binder based-mortar

4.3.4 Water Absorption Test

Figure 4.8 shows results of effects on water absorption of mortar made with a curing age at RHA-CCW binder (28, 56 and 90 days). In this case, the SNP inclusion is used. The mortar quality declines with age as expected. From the Figure, it can be observed that the absorption rate remains nearly the same at 0.5 per cent SNP with that of the specimen without SNP control regardless of the cube age. As the percentage of SNP content increases, however, the rate of absorption increases at the early ages. But as the cube's healing age increases its absorption rate decreased.

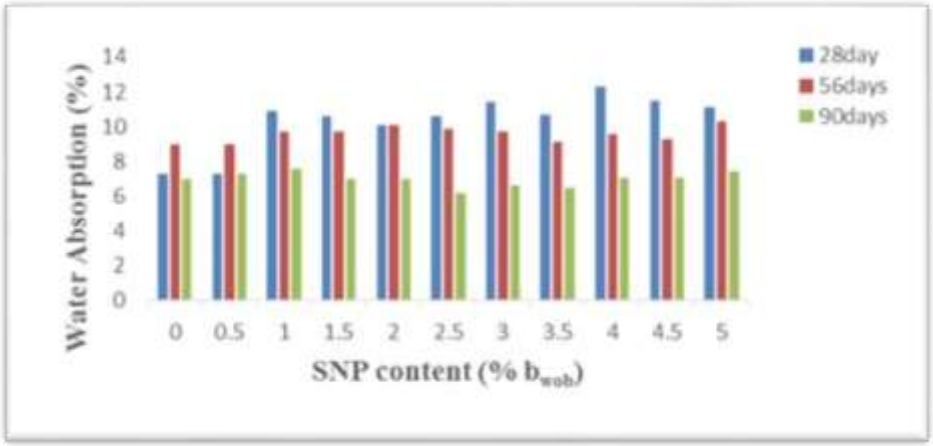

Figure 4.8: Influence of SNP on Water Absorption Value

At 1.5 per cent SNP content, for example, the absorption rate is 10.6, 9.7 and 7 per cent respectively at 28, 56 and 90days. Whereas, on the other hand, 10.1, 10.1 and 7 percent rate absorptions or 28, 56 and 90 days gave SNP content at 2 per cent.

Reducing permeability due to increased SNP content and curing ages may be due to pore refining and densification which is characteristic of SNP inclusion. Gaitero *et al.* (2008) revealed that SNP addition in pozzolanic binder increases cube densification, Pozzolanic reaction and change internal C-S-H gel structures in a binder Transformation of Portlandite into C-S-H. Therefore, Aggarwal *et al.* (2010) observed unrestrained shrinking of cement mortar cubes and weight loss with increased SNP content. From the 90-day study, it is clear that the water flow decreased as the SNP percentage increased.

4.4 SEM-EDX of RHA-CCW Mortar

Figures 4.9 to 4.12 show the SEM images and EDX spectra of the two RHA-CCW mortar (with 3 per cent SNP and control (no SNP)) specimens studied for this experiment after 90 days of curing. The back distributed microstructure image (BSD) obtained at 15 kV (Figures 4.9 and 4.11) showed that the individual grains of the constituent materials for the control specimen could still be identified, while that of the hardened RHA-CCW containing 3% SNP had already been fused into homogeneous material with no separately defined individual component shown. Spectrum EDX (Figures 4.10 and 4.12) showed that the control mortar had a strong Calcium (Ca) peak at 7.066 in 30 seconds as the highest, while the Silica (Si) element peak was found to be fairly similar to the peak of other test elements. On the other hand, the EDX spectrum for hardened RHA-CCW mortar containing 3 per cent SNP (Figure 4.12) with 13,988 counts in 30 seconds revealed two peaks of similar heights (Si and Ca) well above the peaks of the other elements. This is an indication that the

introduction of SNP in this mixture greatly improves the hydration rate and has resulted in a faster production of Calcium-Silicate-Hydrate (C-S - H), The effects of Calcium hydroxide absorption in CCW ($Ca[OH]_2$) by the silica released from the RHA and SNP.

The quantitative analysis of the elements in the two hardened RHA-CCW mortar specimens examined as shown in Table 4.5 revealed that the addition of 3 percent SNP in the RHA-CCW mortar significantly increased the content of C-S-H. The silica weight content of the RHA-CCW mortar, which contains 3% SNP after 90 days of recovery, is found to be above double the value of the control specimen. It was found that the silica to calcium (Si / Ca) ratio of the hardened RHA CCW mortar containing 3 per cent SNP was closed to triple the value obtained for the control specimen.

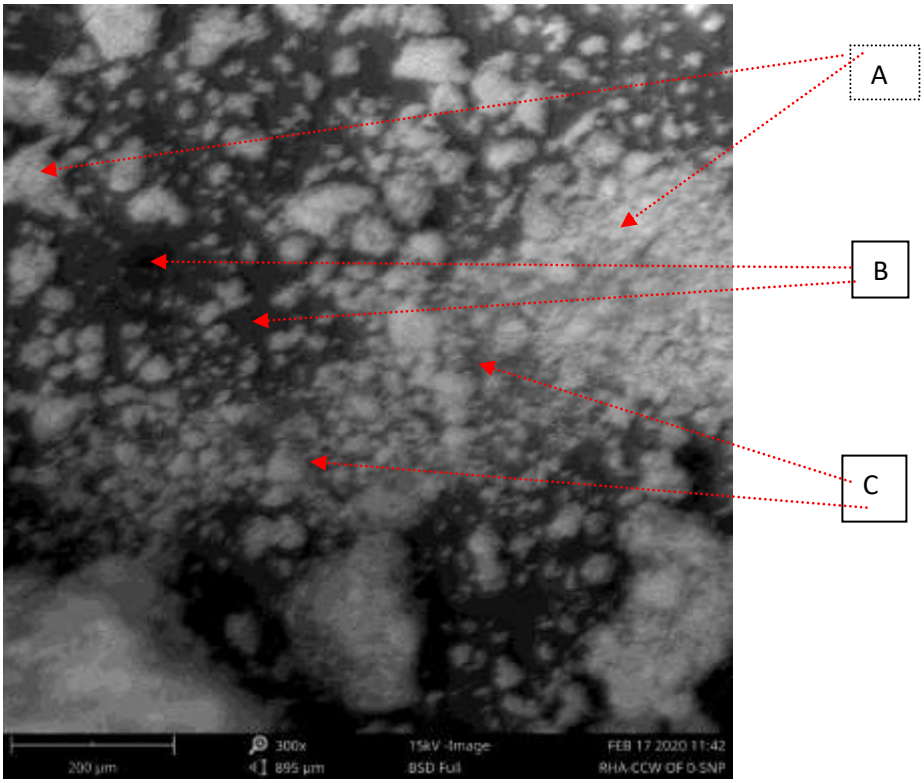

Figure 4.7: SEM Image for RHA-CCW Mortar with no SNP

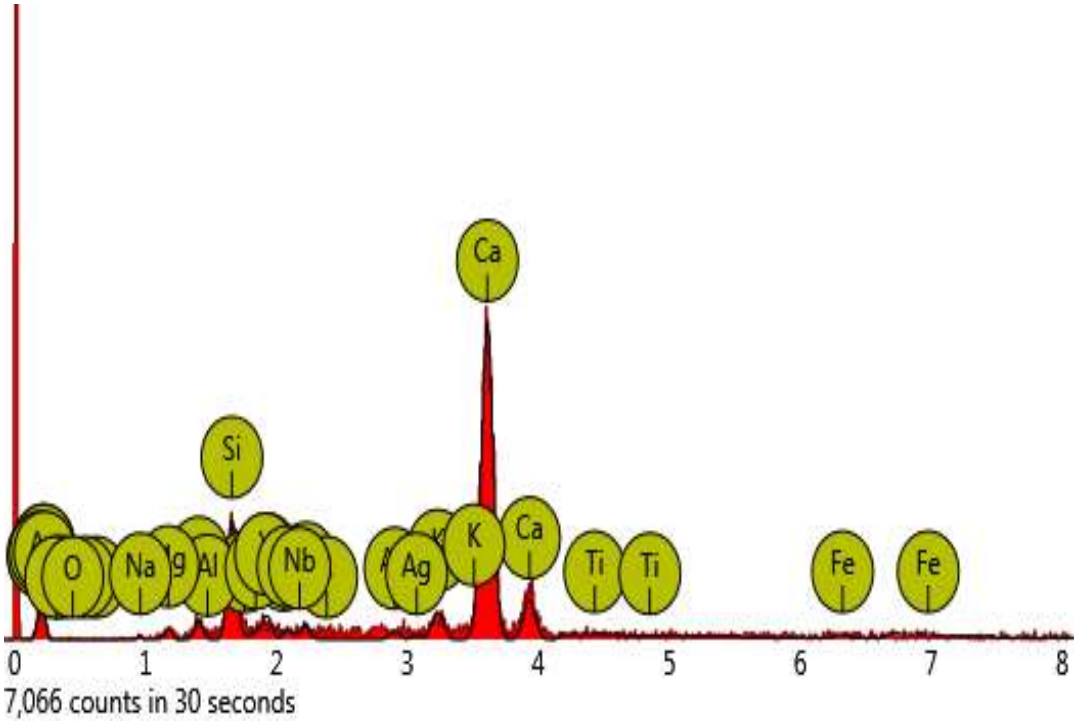

Figure 4.8: EDX Spectrum for RHA-CCW Mortar with no SNP

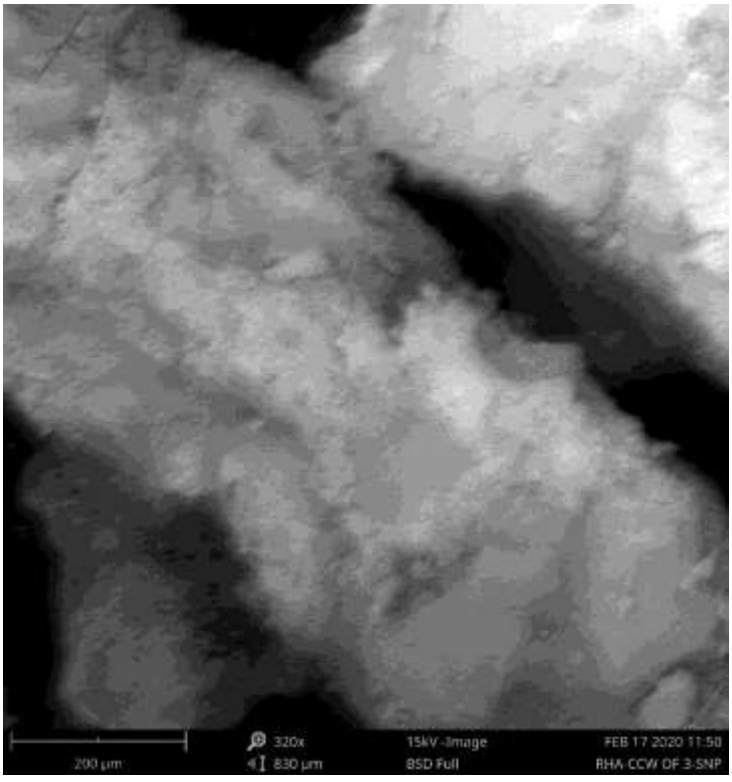

Figure 4.9: SEM Image for RHA-CCW Mortar with 3% SNP

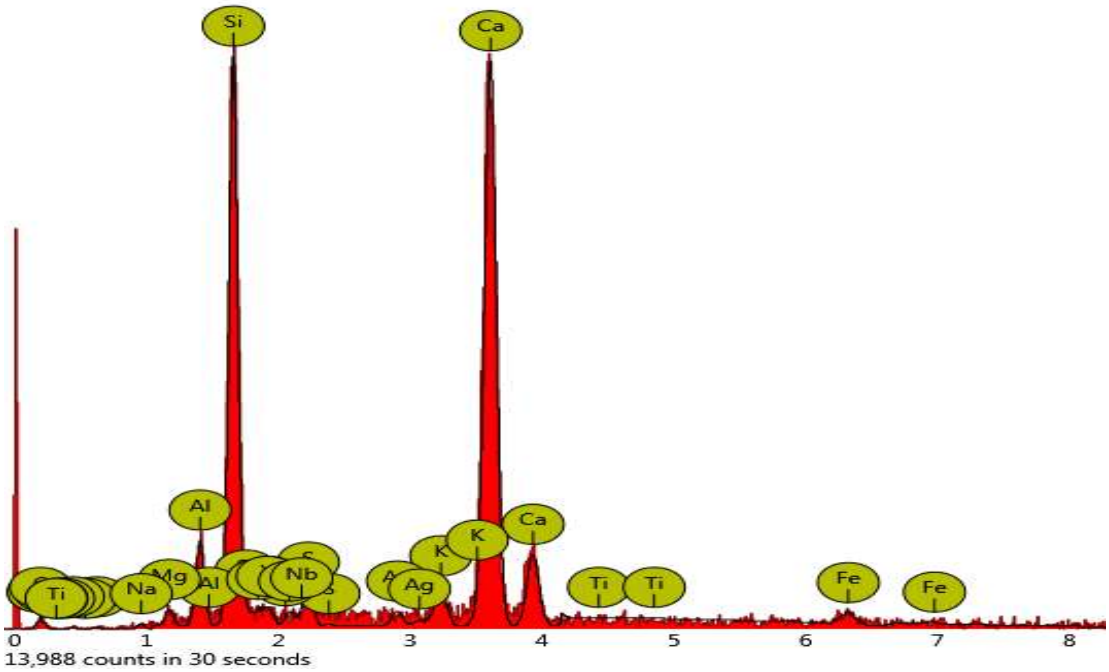

Figure 4.10: EDX Spectrum for RHA-CCW Mortar with 3% SNP

Table 4.5: Quantitative Analysis of Elements in Hardened RHA-CCW Mortar

Specimen			0% SNP		3% SNP	
Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.	Atomic Conc.	Weight Conc.
20	Ca	Calcium	58.34	62.26	47.95	53.16
14	Si	Silicon	13.91	10.40	30.20	23.46
19	K	Potassium	4.49	4.68	5.69	4.25
47	Ag	Silver	1.22	3.51	2.38	3.67
41	Nb	Niobium	1.21	3.00	2.97	3.22
39	Y	Yttrium	1.19	2.82	0.86	2.57
15	P	Phosphorus	3.01	2.49	0.90	2.22
26	Fe	Iron	1.51	2.24	0.78	2.02
13	Al	Aluminium	3.00	2.15	2.22	1.97
6	C	Carbon	5.05	1.61	1.54	1.04
12	Mg	Magnesium	2.43	1.57	0.97	0.83
16	S	Sulphur	1.83	1.57	1.72	0.76
11	Na	Sodium	1.13	0.69	0.77	0.49
8	O	Oxygen	1.33	0.57	1.04	0.35
22	Ti	Titanium	0.35	0.44	0.00	0.00
	Total		100.00	100.00	99.99	100.01
	Si/Ca		0.24	0.17	0.63	0.44

4.5 Statistical Implication of Results

Results from the experimental work have been statistically analysed, and the SNP content plots against the compressive strength values (Figures 4.13 and 4.14) reveal 0.3 percent of SNP content b_{wob} as the optimum for best performance in this study.

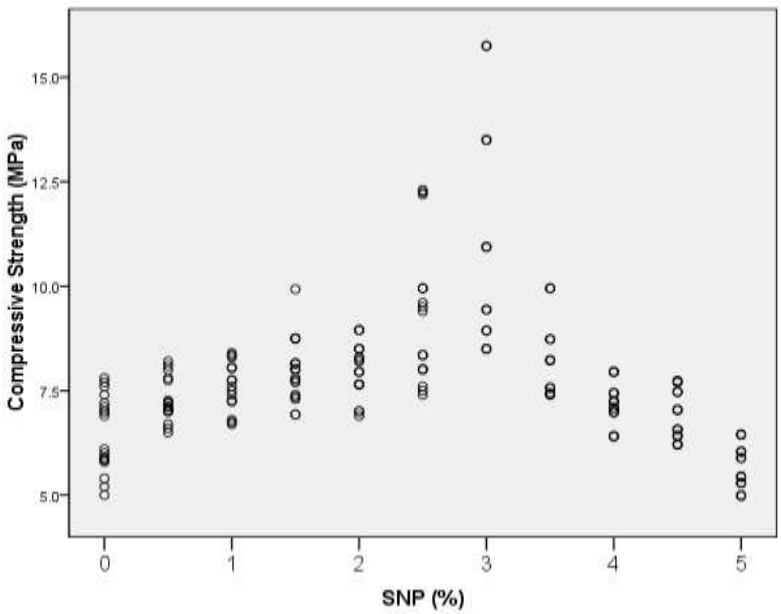

Figure 4.11: Influence of SNP content on Compressive Strength

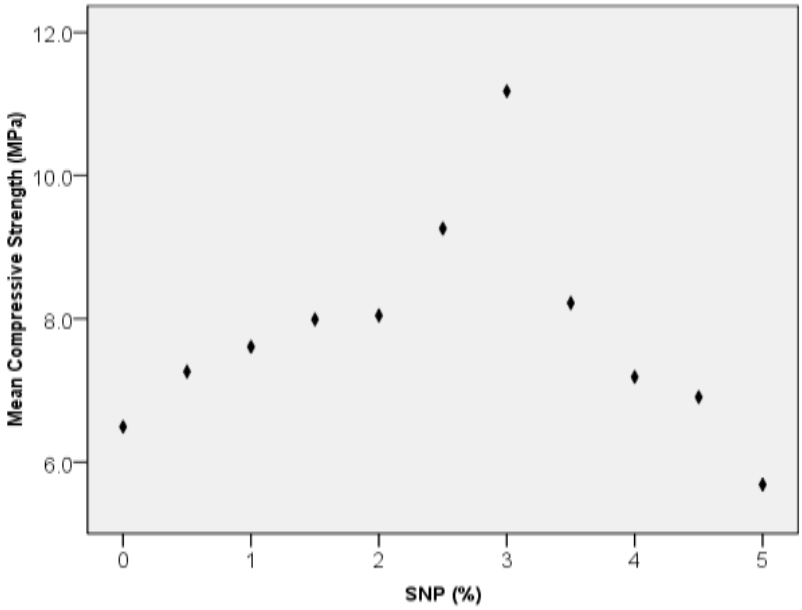

Figure 4.12: Influence of SNP content on Mean Compressive Strength

The curing ages were also noted to positively influence the production of compressive strength for all the SNP contents examined in the study (Figure 4.15 and 4.16).

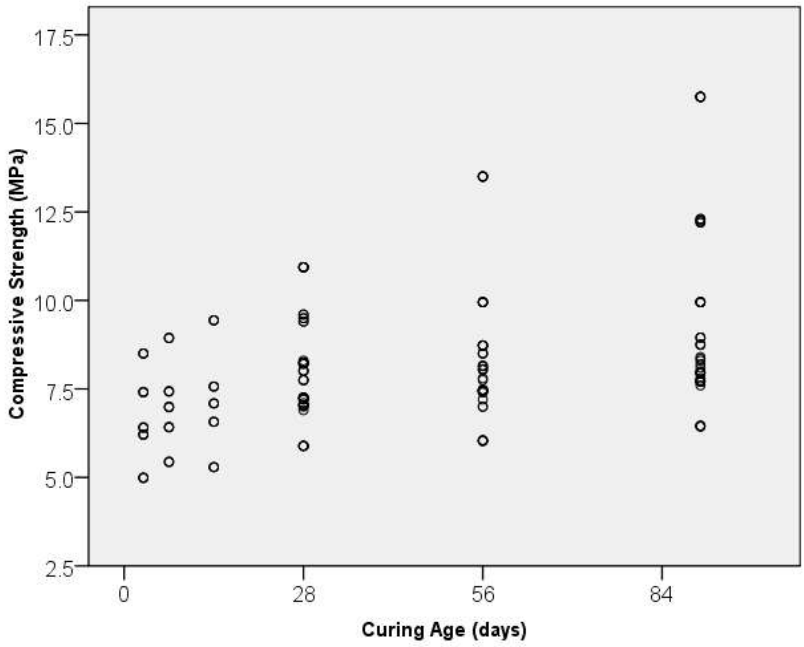

Figure 4.13: Influence of Curing Ages on Compressive Strength

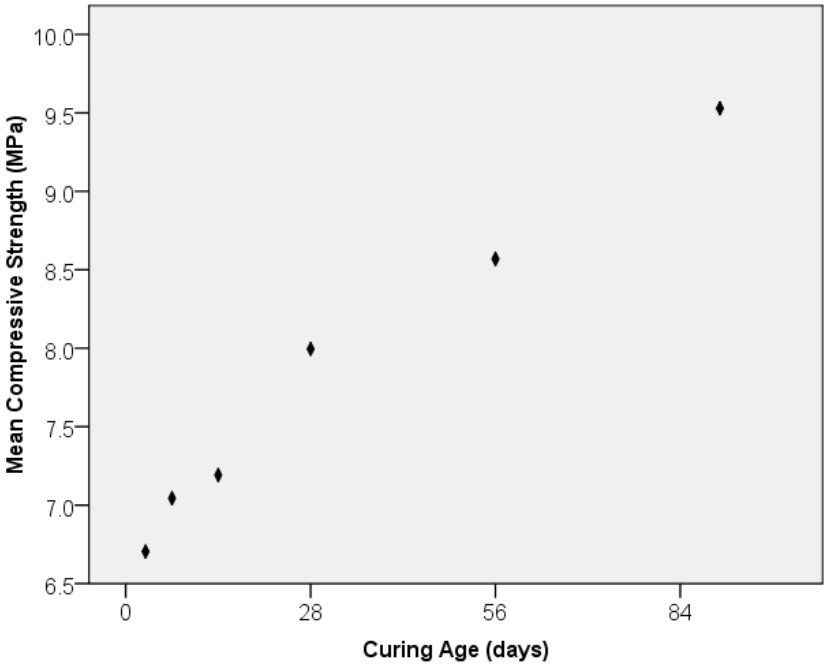

Figure 4.14: Influence of Curing Age on Mean Compressive Strength

The eco-friendly base-mortar's abrasion resistance improved with increasing curing ages, as indicated in the Abraded value decrease with continued curing (Figures 4.17 and 4.18).

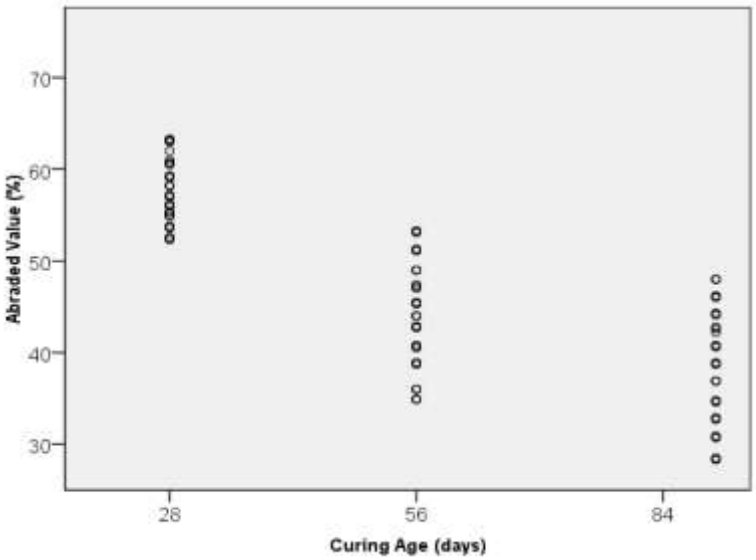

Figure 4.15: Influence of Curing Age on Abraded Value

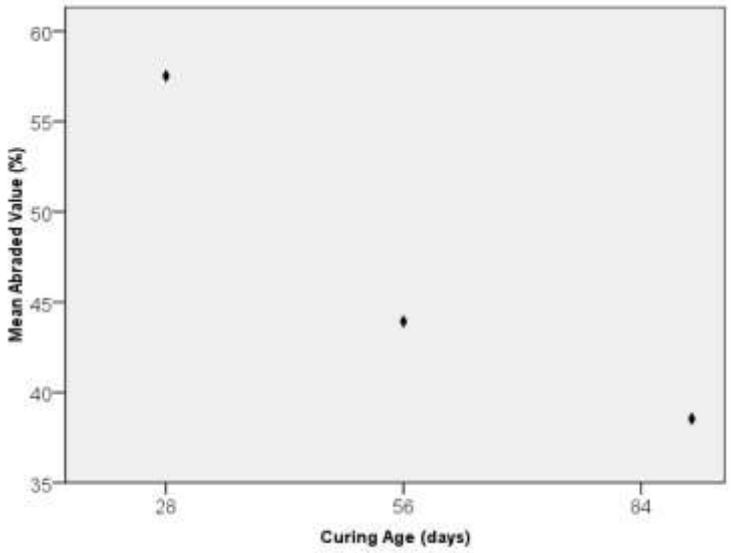

Figure 4.16: Influence of Curing Age on Mean Abraded Value

The SNP content plot against the Abraded value (Figure 4.19 and 4.20) showed that there is a close relation between the two variables. The mean abraded value shows a clear linear relation to the SNP content. In Equation (4.1) the influence of SNP content on Abrasion resistance for the different curing ages examined (Figure B1, Appendix B) is expressed at R2 values of 0.99 to 1.00, implying a very strong relationship.

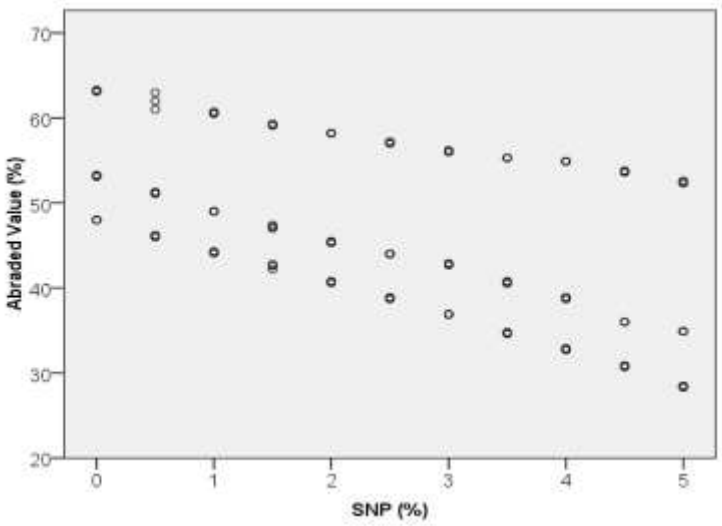

Figure 4.17: Influence of SNP content on Abraded Value (%)

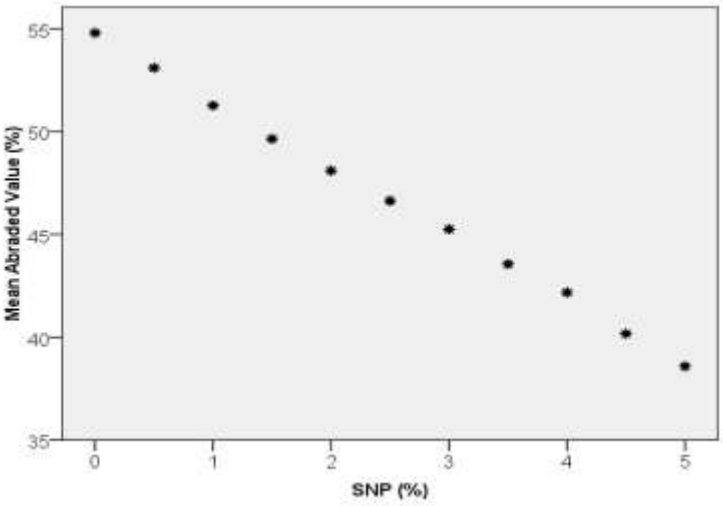

Figure 4.18: Influence of SNP content on Mean Abraded Value

$$Y = Ax + B \quad (4.1)$$

Where Y = Abrasion resistance;

A= constant of relationship (ranging between 2. 07 and 3.87)

x = SNP content (0 to 5 %) and

B = point of interception with the vertical axis (implying the minimum value of the Abrasion resistance possible for the respective curing age studied).

Influence of SNP content on mortar water absorption revealed a scattered plot with two optimum points and a minimum point for the respective ages at three distinct levels (Figures 4.21 and 4.22)

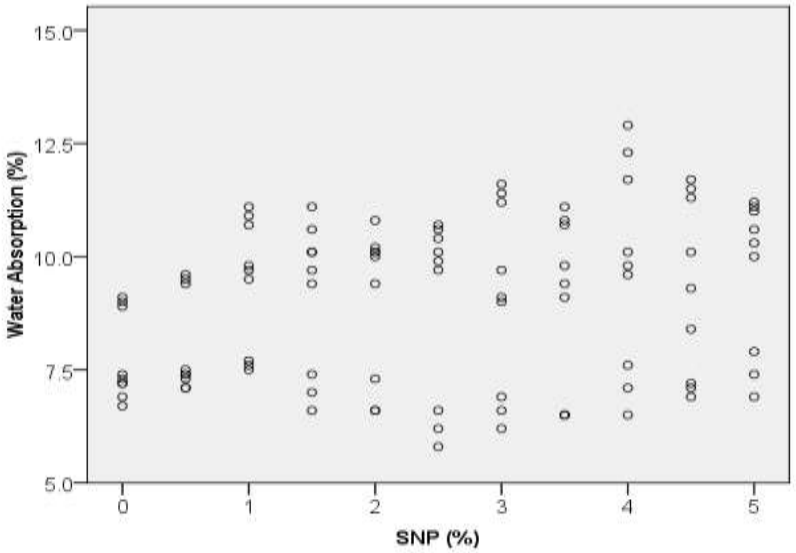

Figure 4.19: Influence of SNP content on Water Absorption

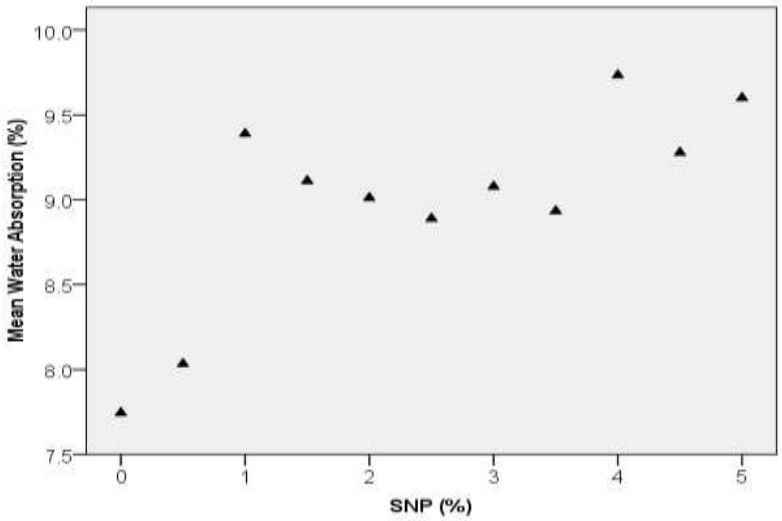

Figure 4.20: Influence of SNP content on Mean Water Absorption

Figure 4.23 Revealed decreased water absorption of the mortar specimen as the curing ages increased, an indication of improved pore refinement and, consequently, mortar durability as the curing age increased.

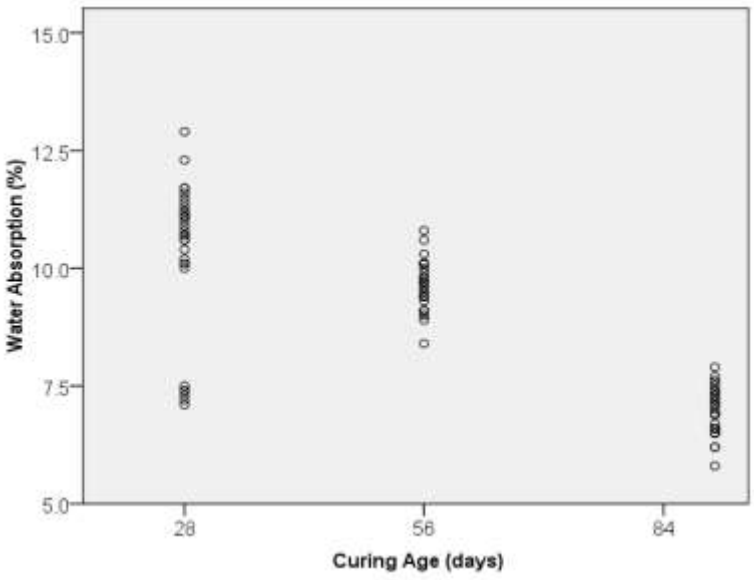

Figure 4.21: Influence of Curing Ages on Water Absorption

The effect of SNP content and curing age (i.e. independent variables) on compressive power, abraded value and water absorption (i.e. independent variables) using statistics (general linear model-multivariate) at a confidence level of 95 per cent ($\alpha = 0.05$) The Appendix B (Table B1) indicates that both independent variables have major effects on dependent variables. Test findings between topic effects (Table 4.6) further show that each dependent variable had substantial impact both as a single factor and as an analysis of combined (two) variables.

The Duncan Post hoc test provided ten homogeneous subsets for influencing SNP contents on compressive strength (Appendix B, Table B2) with 3% SNP being the subset (No.10) with the highest value of 13.97 N / mm^2; 4% and 0.5% SNP fall in the same subset (No. 4) with both 7.54 N / mm^2 and 7.61 N / mm^2 while 5% SNP has the lowest value of 6.13 N / mm^2.

The Post-hoc test further provided control to the Abraded value of eleven homogeneous subsets for SNP content (Appendix B, Table B3) with 5 percent SNP content (Sub-set No. 1) having the least abraded value whereas the highest abraded value was 0 percent SNP content (Sub-set No. 11). Post-hoc test results for effect of

SNP content on water absorption (Appendix B, Table B4) on the other gave six homogeneous subsets with 1.5, 3, and 4 percent SNP content appearing individually in three different subsets. This means that SNP has no very distinct trend.

Table 4.6: Tests of Between-Subjects Effects

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	CS	406.663[a]	32	12.708	1.441E3	.000
	AV	8989.858[b]	32	280.933	6.734E3	.000
	WA	300.687[c]	32	9.396	77.715	.000
Intercept	CS	7206.230	1	7206.230	8.171E5	.000
	AV	215562.668	1	215562.668	5.167E6	.000
	WA	7986.633	1	7986.633	6.605E4	.000
Single Factor Analysis						
SNP	CS	347.960	10	34.796	3.945E3	.000
	AV	2510.481	10	251.048	6.018E3	.000
	WA	33.018	10	3.302	27.308	.000
Cages	CS	30.724	2	15.362	1.742E3	.000
	AV	6315.472	2	3157.736	7.569E4	.000
	WA	213.310	2	106.655	882.108	.000
Single Factor Analysis						
SNP * Cages	CS	27.979	20	1.399	158.627	.000
	AV	163.905	20	8.195	196.448	.000
	WA	54.359	20	2.718	22.479	.000
Error	CS	.582	66	.009		
	AV	2.753	66	.042		
	WA	7.980	66	.121		
Total	CS	7613.475	99			
	AV	224555.280	99			
	WA	8295.300	99			
Corrected Total	CS	407.245	98			
	AV	8992.612	98			
	WA	308.667	98			

a. R Squared = .999 (Adjusted R Squared = .998)

b. R Squared = 1.000 (Adjusted R Squared = 1.000)

c. R Squared = .974 (Adjusted R Squared = .962)

*CS = Compressive strength, AV = Abraded value, WA = Water absorption, SNP = Silica Nanoparticles, CAges = Curing Ages, df = degrees of freedom, F = F-ratio, Sig. = exact significance level.

4.6 Summary of Findings

The results of the study "Influence of Silica-Nano Particles on Rice Husk Ash and Calcium Carbide Waste Binder Mortar Properties" reveal some important findings that is summarized below:

i. Chemical analysis showed that RHA sample used can be classified as high total SiO2 content Type F Pozzolan (83.7 percent) while CCW is a good percentage concentration CaO source (65.6 percent) comparable to PC.

ii. The chemical structure of SNP demonstrates higher percentage of SiO_2 (93.40) when compare to the RHA (83.7 %).

iii. The physical properties of the SNP revealed it to be microscopic in nature.

iv. The studies reveal that SNP is a Sustainable Nano engineered silica source of high quality for high performance in mortar and concrete.

v. The fresh properties such has workability, setting time and soundness of SNP modified RHA-CCW based-mortar decrease as SNP content increased.

vi. The Mortar specimen made in the 60:40 RHA-CCW binder with 3 percent SNP content proved to be the best performance of the samples tested, having compressive strength values of 10.94, 13.50 and 15.75 N / mm2 of 28 , 56 and 90 days, reflecting a compressive strength increase of 40, 63 and 80 percent above the specimen without SNP.

vii. In addition to the RHA-CCW binder-based mortar, SNP has resulted in improved abrasion resistance and reduced water absorption as SNP content and curing age increases.

CHAPTER FIVE

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

The results and analysis from this study show that the chemical leaching process adopted for the production of SNP removed all impurities and improved the silica content of the RHA sample when produced in Nano form. The chemical structure shows that both the SNP and untreated Rice Husk Ash can be classified as Class F Pozzolan as defined in ASTM 618 (2015), whereas the CCW was weak in CaO but weak in LOI.

SNP's physical properties (PSD, BET surface area, total pore volume), were compatible with previously mentioned requirements. The fresh properties resulting from the analysis correspondingly show the SNP as an activator on RHA-CCW mortar with strong binding properties and early setting time and strength gain behaviour.

The study further revealed that the higher the content of SNP in mortar based on the RHA-CCW binder, the lower the permeability. Similarly, RHA CCW binder-based mortar strength and abrasion resistance increase as the SNP content increases up to 3%.

5.2 Research Contribution to Knowledge

In addition to the details in the following areas the hypothesis entitled "The effects of silica Nano particle surrounding the properties of rice husk ash-calcium carbide waste binder-based mortars";

i The production of alternative binder for rice husk ash and calcium carbide using RHA-

CCW and SNP as agro- and industrial waste

ii Production of the RHA / CCW binder and SNP hybrid mixing system for mortar.

iii To achieve good workability of RHA-CCW and SNP binder in mortar a high water enhancing admixture is required.

5.3 Recommendations

The study therefore recommends that 3 percent SNP content in RHA-CCW (60:40) binder at 1:3 binder / sand mortar, with 0.5 W / B and 1.5 percent Super plasticizer that be used for maçonnery operations as it conforms to ASTM C270 Class N mortar.

5.4 Suggestions for Further Research

Further studies are recommended in the following areas for a better understanding of the agro-industrial based binder.

i. A demonstration work on the use of RHA-CCW binder incorporating SNP in concrete production can be undertaken.

ii. A study on influence of elevated temperature on performance of RHA-CCW with SNP as an admixture in concrete/mortar should be evaluated.

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APPENDIX A: TABLES FROM EXPERIMENTAL WORKS

A1: Sieve Analysis (Fine Aggregate)

Sieve size (mm)	Weight of Sieve (g)	Weight of sample retained (g)	Percentage of sample retained	Cumulative percentage retained	Percentage passing
4.95	381	0	0	0	100
2.36	396	5	1.0	1	99
1.18	366	5	1.0	2.0	97
0.6	380	37	7.4	9.4	87.6
0.3	363	173	34.6	44	43.6
0.15	334	240	48.0	93	7
0.075	374	32	6.4	98.4	1.6
Pan	311	8	1.6	100	0

D_{60} = 0.32mm, D_{30} = 0.20mm and D_{10} = 0.15mm

Coefficient of uniformity (C_u) = $\frac{D_{60}}{D_{10}}$ = 2.13

Coefficient of curvature (C_c) = $\frac{(D_{30})^2}{D_{60}\,(D_{10})}$ = 1

A2: STANDARD CONSISTENCY OF THE ALTERNATIVE BINDER

SNP % Replacement with RHA/CCW	SNP (g)	Weight RHA (g)	Weight of CCW (g)	Weight of water (g)	% of water by weight dry binder	S/P (g)	Penetration (mm)
0	0	180	120	165	55	4.5	6
0.5	1.5	179.1	119.4	165	55	4.5	6
1.0	3	178.2	118.8	165	55	4.5	5
1.5	4.5	177.3	118.2	165	55	4.5	6
2.0	6	176.4	117.6	165	55	4.5	6
2.5	7.5	175.5	117.0	165	55	4.5	6
3.0	9	174.6	116.4	165	55	4.5	5
3.5	10.5	173.7	115.8	165	55	4.5	5
4.0	12.0	172.8	115.2	165	55	4.5	5
4.5	13.5	171.9	114.4	165	55	4.5	6
5.0	15	171.0	114.0	165	55	4.5	7

A3: Setting Time of RHA/CCW and SNP Binder Combination

SNP %	Initial (min)	Final (min)	Depth of penetration (mm)
0	195	360	7
0.5	185	345	6
1.0	181	333	6
1.5	175	315	6
2.0	170	301	5
2.5	167	298	6
3.0	163	293	5
3.5	158	287	5
4.0	155	284	6
4.5	150	280	5
5.0	149	281	5

A4: Average soundness of RHA/CCW and SNP Binder Combination

SNP %	Average Expansion (mm)
0	1
0.5	1
1	0.5
1.5	1
2	1.5
2.5	1
3	1
3.5	0
4	0
4.5	0
5	0

A5: Average Density (DS-KGM^3) of RHA/CCW AND SNP Mortar Samples

RHA/CCW WITH SNP (%)	3days	7days	14days	28days	56days	90days
0	2118	2120	2104	2097	2075	2072
0.5	2120	2123	2123	2125	2130	2131
1.0	2122	2122	2125	2124	2124	2126
1.5	2126	2127	2126	2127	2128	2127
2.0	2130	2134	2137	2138	2137	2139
2.5	2218	2218	2219	2221	2227	2230
3.0	2219	2219	2220	2221	2228	2231
3.5	2220	2220	2221	2222	2225	2226
4.0	2221	2225	2226	2227	2226	2228
4.5	2232	2231	2230	2231	2232	2233
5.0	2219	2225	2227	2230	2231	2229

A6: Average compressive strength (CS-N/mm^2) of mortar samples with HRWR

SNP %	Average Compressive Strength (N/mm^2)					
	3days	**7days**	**14days**	**28days**	**56days**	**90days**
0	5.2	5.84	6.0	6.9	7.2	7.7
0.5	6.6	7.01	7.11	7.23	7.75	8.10
1.0	6.75	7.25	7.50	7.75	8.05	8.35
1.5	6.93	7.35	7.75	8.01	8.15	8.75
2.0	7.0	7.65	7.95	8.25	8.50	8.95
2.5	7.50	8.01	8.35	9.50	9.95	12.25
3.0	8.50	8.94	9.44	10.94	13.50	15.75
3.5	7.41	7.43	7.58	8.23	8.73	9.95
4.0	6.41	6.99	7.09	7.24	7.44	7.94
4.5	6.21	6.42	6.57	7.04	7.47	7.72
5.0	4.99	5.44	5.29	5.89	6.04	6.45

A7: **Average water absorption (%) of mortar samples with HRWR**

SNP %	Average water absorption (%) of mortar samples		
	28 days	56 days	90 days
0	7.3	9	6.9
0.5	7.3	9	7.3
1	10.9	9.7	7.6
1.5	10.6	9.7	7
2	10.1	10.1	7
2.5	10.6	9.9	6.3
3	11.4	9.7	6.6
3.5	10.7	9.1	6.5
4	12.3	9.6	7.1
4.5	11.5	9.3	7.1
5	11.1	10.3	7.4

A8: Average Abrasion Resistance (%) of mortar samples with HRWR

SNP %	Average Abrasion Resistance (%) of mortar samples		
	28 days	56 days	90 days
0	51.3	49.2	46.2
0.5	64	56.5	49.8
1	63.5	59.4	50.4
1.5	60.4	55.8	49.6
2	61.7	55.2	49.8
2.5	60.0	55.2	49.8
3	59.9	55.4	48.3
3.5	74.6	63.5	59.4
4	77.9	64	60.4
4.5	79.2	74.6	63.5
5	80.7	77.2	74.6

APPENDIX B: TABLES AND CHARTS FROM ANALYSIS OF RESULTS

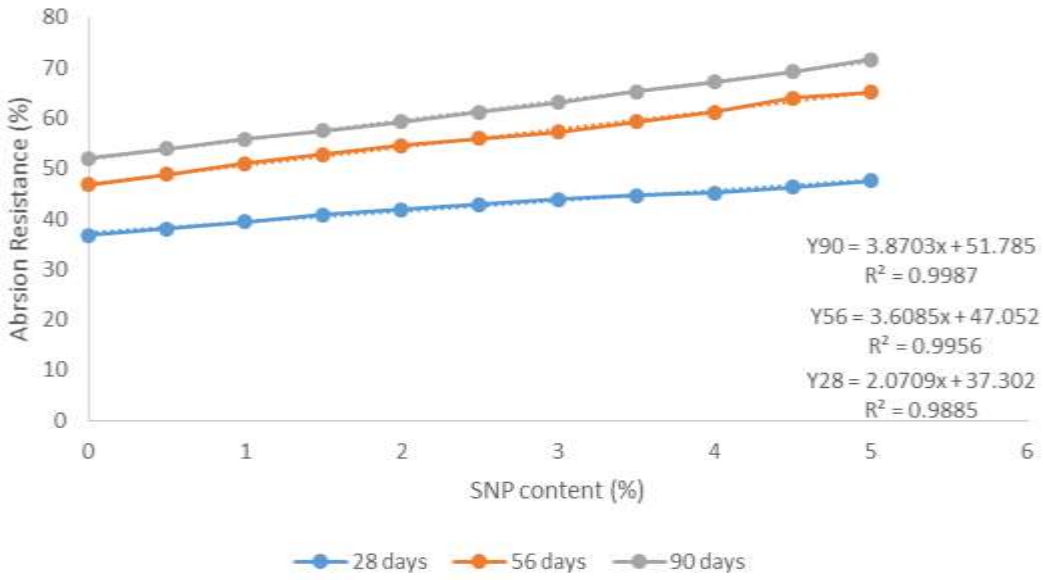

Figure B0.1: Plots of Abrasion Resistance as Influenced by SNP content

Table B1: Result of Multivariate Tests[c]

Effect		Value	F	Hypothesis df	Error df	Sig.
Intercept	Pillai's Trace	1.000	2.025E6[a]	3.000	64.000	.000
	Wilks' Lambda	.000	2.025E6[a]	3.000	64.000	.000
	Hotelling's Trace	9.492E4	2.025E6[a]	3.000	64.000	.000
	Roy's Largest Root	9.492E4	2.025E6[a]	3.000	64.000	.000
SNP	Pillai's Trace	2.638	48.167	30.000	198.000	.000
	Wilks' Lambda	.000	796.529	30.000	188.529	.000
	Hotelling's Trace	1.514E3	3.163E3	30.000	188.000	.000
	Roy's Largest Root	920.962	6.078E3[b]	10.000	66.000	.000
CAges	Pillai's Trace	1.937	664.752	6.000	130.000	.000
	Wilks' Lambda	.000	4.135E3[a]	6.000	128.000	.000
	Hotelling's Trace	2.394E3	2.513E4	6.000	126.000	.000
	Roy's Largest Root	2.379E3	5.154E4[b]	3.000	65.000	.000
SNP * CAges	Pillai's Trace	2.698	29.524	60.000	198.000	.000
	Wilks' Lambda	.000	71.908	60.000	191.776	.000
	Hotelling's Trace	116.389	121.562	60.000	188.000	.000
	Roy's Largest Root	64.760	2.137E2[b]	20.000	66.000	.000

a. Exact statistic

b. The statistic is an upper bound on F that yields a lower bound on the significance level.

c. Design: Intercept + SNP + CAges + SNP * CAges

Table B2: Duncan Post hoc Test for SNP Influence on Compressive Strength

		Subset									
SNP	N	1	2	3	4	5	6	7	8	9	10
5	9	6.1267									
0	9		7.3000								
4.5	9			7.4100							
4	9				7.5433						
0.5	9				7.6156						
1	9					8.0500					
1.5	9						8.3033				
2	9							8.5667			
3.5	9								8.9700		
2.5	9									10.5667	
3	9										13.3967
Sig.		1.000	1.000	1.000	.108	1.000	1.000	1.000	1.000	1.000	1.000

Means for groups in homogeneous subsets are displayed Based on observed means.
The error term is Mean Square (Error) = .009.

Table B3: Duncan Post hoc Test for SNP Influence on Abraded Value

		Subset										
SNP	N	1	2	3	4	5	6	7	8	9	10	11
5	9	38.5889										
4.5	9		40.1667									
4	9			42.1778								
3.5	9				43.5556							
3	9					45.2556						
2.5	9						46.6333					
2	9							48.1000				
1.5	9								49.6444			
1	9									51.2667		
0.5	9										53.1000	
0	9											54.8000
Sig.		1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000

Means for groups in homogeneous subsets are displayed Based on observed means.
The error term is Mean Square (Error) = .042.

Table B4: Duncan Post hoc Test for SNP Influence on Water Absorption

		Subset					
SNP	N	1	2	3	4	5	6
0	9	7.7444					
0.5	9	8.0333					
2.5	9		8.8889				
3.5	9		8.9333	8.9333			
2	9		9.0111	9.0111			
3	9		9.0778	9.0778	9.0778		
1.5	9		9.1111	9.1111	9.1111		
4.5	9			9.2778	9.2778	9.2778	
1	9				9.3889	9.3889	
5	9					9.6000	9.6000
4	9						9.7333
Sig.		.083	.235	.064	.087	.067	.419

Means for groups in homogeneous subsets are displayed Based on observed means.
The error term is Mean Square (Error) = .121.

APEENDIX C: PLATES OF LABORATORY ACTIVITIES

Plate 1: Unprocessed rice husk

Plate 2: Rice husk ash

Plate 3: Silica-Nano Particle

Plate 4: Furnace

Plate 5: Cast cubes

Plate 6: Cubes under curing

Plate 7: Compressive machine

Plate 8: Los Angeles Machine for Abrasion with Uncrushed Cube