EFFECT OF SILICA NANOPARTICLES ON THE PERFORMANCE OF SORGHUM HUSK ASH AND CALCIUM CARBIDE WASTE BINDER BASED MORTAR

 \mathbf{BY}

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ABSTRACT

Research trends in the construction industry is toward the application of nano materials in mortar/concrete which could act as set accelerator due to the ultrafine nature of silica nano_particles (SNPs). The study is aimed at investigating the effect of SNPs on the performance of Sorghum Husk Ash (SHA) and Calcium Carbide Waste (CCW) binder based mortar with a view to establishing its suitability. SHA was an incinerated ash from agro-by-product of sorghum husk with major component of amorphous silica (SiO₂) when combined with calcium carbide waste (CCW) an industrial by-product from an acetylene gas production process with major component of lime (CaO) in the presence of water to forms compounds possessing cementitious properties. Assessment of mortar in fresh state, early-age strength development and hardened properties were carried out. Pastes from different binder 70/30 (SHA_CCW) containing 0.5% to 5% at 0.5 step interval of SNPs. The water-to-binder ratio (w/b) and High Range Water Reducer Admixture (HRWRA) was optimized at 0.5 and 1.5% respectively by mass of binder. With a normal mix of 1:3 binder/sand, samples were studied for flowbility, air content, setting times, degree of hydration, water absorption, abrasion resistance and compressive strength was also determined at ages (3, 7, 14, 28, 56 and 90 days). Mortar samples with 3.0% (SNPs), exhibited better performance at 28 days with compressive strength value of 7.45 N/mm² representing 137% of 70/30 (SHA_CCW) strength and 90 days with compressive strength value of 9.01 N/mm² representing 134% of 70/30 (SHA CCW) strength. SNPs (3.0%) and SHA_CCW (70/30) in 1:3 binder/sand mortar at 0.5 w/b with 1.5% water-reducing admixture is recommended for use in masonry work as it conforms to class N mortar of ASTM C270.

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ABREVIATIONS AND SYMBOLS

ACI American Concrete Institute

ASTM America Society for Testing and Materials

BS British Standard

C₃S Tricalcium Silicate

CCA Corn-Cob Ash

CCR-FA Calcium Carbide Residue-Fly Ash

CCW Calcium Carbide Waste

CH Calcium Hydroxide

CO₂ Carbon Dioxide

C-S-H Calcium Silicate Hydrate

EDXRF Energy Dispersive X-Ray Fluorescence test

H₂0 Water

HRWRA High Range Water Reducing Admixture

IS Indian Standard

LOI Loss on Ignition

MHA Millet Husk Ash

OPC Ordinary Portland Cement

PC Portland Cement

PSA Particle Size Analysis

RHA Rice Husk Ash

SF Silica Fume

SHA Sorghum Husk Ash

XRD X-ray Diffraction

XRF X-ray Fluorescence

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background of the Study

Nano-technology according to Khasn (2011) and Birgisson *et al.* (2010), was interpreted as the science of altering, monitoring the exhibition, performance of materials at the nanoscale Khan (2011) went on to describe nanoscale materials as a set of parts, all of which are interconnected whereby one point is actually less than 100 nanometers. A nanometer is one millionth of a millimeter - nearly 100,000 times tinier than the width of the hair of a human. Bakhoum *et al.* (2017), concurred that it is the process of making an appliance or material with key components at the atomic and molecular scale.

Bakhoum *et al.* (2017) went on to say that nanotechnology is a current promising area in terms of enhancing environmental improvement such as energy conservation and non-renewable resource management, as well as reducing waste, hazardous substances, and carbon emissions. Ganesh (2012) stated that nanotechnology would contribute to the creation of objects with specific features, which could provide interesting answers for achieving sustainable housing project and introducing innovations for cost-effective spaces inside a facility. Nanotechnology is a branch of research and innovation that focuses on both knowing and managing issues at the subatomic level and as a result, affecting the material's properties (Grove, 2010 and Vishwakarma *et al.*, 2016).

According to Abbas (2011), Jonbi (2013), and Khanzadi (2010), nano silica (NS) has a great pozzolanic reactivity, which has important implications for the behaviour of cement-based matrix composites in both the freshly and set states. Sadrmomtazi (2012) and Abbas (2011) pointed out that NS reduces set times, increases hydration temperature escape, and alters the rheological characteristics of solid pastes and mortars in the fresh form. Sadrmomtazi (2012), Abbas (2011), and Jonbi (2013) have

demonstrated that NS improves crushing performance, reduces porosity, and improves a few aspects of resilience in the hardened state. SNPs have been discovered to cause significant mineralogical changes, especially in C-S-H and portlandite, according to a few studies (CH). In terms of C-S-H, NS floods the movement, the models blended with NS have a greater C-S-H material, and it provides a strategy of lasting C-S-H as compared to the control samples (Abbas, 2011; Jonbi, 2013 and Khanzadi, 2010). The need to use wastes from manufacturing or unindustrialized processes in the construction industry stems from the need to include construction constituents that can be maintained (Hardjito *et al.*, 2012). According to Olumuyiwa *et al.* (2018) and Sunusi (2015), this can be accomplished by looking for and incorporating new eco-friendly resources and products, as well as leading to a reduction in carbon dioxide emissions into the atmosphere.

Sorghum belongs to the family of cereal crop also known as grain sorghum which has a place with the general name of sorghum, (Chukwu, et.al., 2011 and Ndububa *et al.*, 2015). Tahomah *et al.* (2017) revealed that it is one of the most well-known seed grain delivered in Africa. Sorghum/sorghum grain is for the most part developed in the northern region of Nigeria in states like Kaduna, Bauchi and Plateau. When collected, it is ordinarily handled precisely by the utilization of consolidated reapers or physically by sifting with sticks leaving a huge amount of buildup (husk) establishing ecological waste yearly.

The benefits of using agro waste to replace cement will lower cost per ton as compared to Portland Cement (PC), reduced waste management, waste movement control at a low cost, and improved economy base/gross domestic product of local farmers when such waste is exchanged, enabling more production conservation of

limestone deposits (Ndububa *et al.*, 2015; Mahmoud *et al.*, 2012 and Manasseh., 2010).

The examination of the Sorghum Husk Ash (SHA) indicated that a blend of its substance constituents qualified it as a pozzollana (ASTM C618-92a). Pozzolan as uncovered by Ndububa *et al.* (2015) and portrayed by ASTM (C618) are "siliceous, aluminous substance which in themselves have no/little cementitous properties yet in unbelievably confined structure and with water they can react with calcium hydroxide (CaO) which is dissipated during the hydration of PC at room temperarure to form blends having cementitous characteristics". Calcium_Carbide_Waste (CCW) is a waste material produced using oxy-acetylene gas utilized in welding works (Chukwudebelu *et al.*, 2013). CCW is abundant in our condition and explores have been composed to pick its use as an assistant structure progression material. Calcium Carbide Waste (CCW) as uncovered by Makaratat *et al.* (2011) is side-effect gotten from the acetylene-gas (C2H2) produce process as appeared in Equation (1.1).

$$CaC_2 + 2H_2O \rightarrow C_2H_2 + Ca (OH)_2$$

$$(1.1)$$

Research discoveries by Makaratat *et al.* (2011); Olumuyiwa *et al.* (2018) have indicated that CCW, when gotten together with certain pozzolan, for instance, FlyAsh (FA), Silica Fume (SF), containing high silicon dioxide (SiO₂) and aluminum oxide (Al₂O₃) could yield last things that resemble those acquired from Portland Cement (PC) hydration process. More looks at are up 'til now being coordinated on usage of CCW being developed. Sun (2015), further observed that CCW contains some blend sythesis.

1.2 Statement of the Research Problem

The inclusion of wastes from industrial or Agro processes in the construction industry according to Hardjito *et al.* (2012) and Abdurra'uf *et al.* (2017) is accepted out of the necessity to provide sustainable materials for building. This is achieved either by incorporating existing or incorporating new materials and waste products that are more eco-accommodating as well as contributing towards the lessening of carbon dioxide (CO₂) discharged into the atmosphere. Previous study on the use of sorghum husk ash (SHA) and calcium carbide waste (CCW) for the production of pozzalanic binder was found to exhibit positive hydration characteristics. Notwithstanding, the SHA-CCW combination was reported to exhibit slow hydration process with an accompanying lower strength characteristic, particularly at early age (Egwuda, 2017). This study is an attempt to improve on the early and latter properties of SHA-CCW binder as continuation of previous attempt at development of an alternative binder by Egwuda (2017). It is in this attempt that this current research focused on the use of silica nano particles (SNP) with a view to improving the strength development characteristics of the developed binder.

1.3 Aim and Objectives

This thesis is aimed at assessing the pozzolanic behaviour of SNPs and the impact it has on the mechanical, transport properties of SHA and CCW based mortar with the end goal of establishing its sustainability.

The objectives are to:

- 1. Determine the physio-chemical properties of the SHA, CCW and SNPs.
- 2. Determine reasonable quantity of the SNPs as constituent material and its impact on the early properties of SHA_CCW based mortar.
- 3. Examine the preparation and characterization of SHA for the manufacture of nano-silica.

4. Examine the mechanical and long-term toughness of mortars made with SNPs as an admixture in the SHA CCW binder.

1.4 Scope of Work

This exploration was aimed at examining the usage of the SHA and CCW as whole substitution of cement in mortar products and choosing the physio-engineered characteristics of the fundamental materials, examining the physical properties tests, for instance, (sieve analysis, specific-gravity, moisture content, and bulk density), and the mixture structure of the important components. Regardless of how previous SHA and CCW examinations have been performed as a distinct experiment in Egwuda (2017), there is an evaluation gap in the study of blending mixed SNPs in SHA and CCW on the properties of mortar. This evaluation will look at the new properties of mortar made with SNPs as an admixture in a 70/30 SHA CCW ratio, for example.

1.5 Need of the Study

For several years, the high cost of cement is a major impediment to stable and long-term accommodation. Nigeria's government however announced the launch of a mortgage refinance scheme that would offer 10,000 dwellings to the country's people (Andrews, 2014). One answer to the housing affordability and sustainability crisis, according to Ndububa *et al.* (2015), is to substitute a portion of cement in concrete and mortar with economical and possible pozzolanic assets from agro and mechanical litters.

Additionally, in land filling using waste materials produced from most industries, for example, marble, stone, flexible, plastic, ended, material, calcium carbide and so on; as indicated by Bakhoum *et al.*, (2017) is inciting waste ejection issues. Such sorts of

litters as detailed by Vishwakarma *et al.*, (2016) have pozzolanic activity which are progressively utilized in the improvement business for concrete so as to diminish the Co₂ spread from the industrial activities.

Regardless Bakhoum *et al.* (2017) concurred that during the modification of structures, Engineered Nano-Materials (ENMs) contained in past developed facility are reused or become improvement fillers. Nattapong *et al.* (2010). Reviewed the impacts of Calcium Carbide ResidueFly-Ash (CCR-FA) binder on mechanical performance of concrete and saw that the solidified cement passed on from CCR-FA blends had mechanical properties like those from typical PC concrete.

A few researches had been made of late to profit the utilization of nano- applications and sciences in the field of cement binder-based materials age as a substitute for cement or fine sand with varying degrees. By a wide edge a massive bit of this evaluation depended in the wake of utilizing improvised materials (for example Silica particles) in a nano-scale to strengthen the properties of concrete based materials unequivocally concrete and mortar, (Bakhoum *et al.*, (2017). This investigation investigate the fruitful regions of utilizing SNPs in improving the performance and qualities of cement-based materials and upgrading the idea of practical development, from this time forward, the imperative to check for the impact of quantity of SNPs as set quickening agent in SHA_CCW fastener.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Nano-particles and their Origins

Nano-particles as coined by Nicolae *et al.* (2018) and Aitken and Creely (2012) was characterized as particulate matter with in any one measurement that is under 100 nm. This declaration looked at reusing cost of waste materials produced using most industrial waste, for example, marble, stone, flexible, plastic, ended, material, calcium carbide, and so on; as indicated by Bakhoum *et al.*, (2017). Such sorts of present-day waste as detailed by Vishwakarma *et al.*, (2016) have pozzolanic content which are progressively utilized in the improvement facility as a halfway trade for concrete so as to diminish the Co₂ spread from the cement products.

Bakhoum *et al.* (2017) however concurred that during the demolition of structures, Engineered Nano-Materials (ENMs) contained some materials which are reused or become improvement waste. Nattapong *et al.* (2010). Looked into the impact of Residue of Calcium Carbide-Fly Ash (CCR-FA) on mechanical performance of concrete and saw that the solidified cement passed on from CCR-FA blends had mechanical properties like those from typical PC concrete.

Efforts have been made two to three decades ago to benefit from nano science and applications in the area of cement matrix-based materials particulate as a replacement for cement or sand to varying degrees. By a wide edge an enormous bit

of this evaluation depended in the wake of utilizing "colloidal" cost muddled materials (for example Silica-particles) within a nano-scale to sustain the properties of concrete based materials unequivocally concrete and mortar, formed by structures composition for a long time, or of anthropogenic beginning. Barometrical colloids might be made by warming up system philosophy, discharged as results in

mechanical method, by incidental and bombardment discharge in any case will once settle and change and maritime condition. Nevertheless, colloids may be moved over huge divisions in the earth and even regularly occured structures like carbon nanotubes have been found in countless years old models from ice-focuses of the cold (Heera and Shanmugam, 2015; Esquivel and Murr, 2012). Dependent upon their beginning stage natural maritime colloids can be sub-confined into four social occasions. Furthermore, the course of action made by various experts like (Heera and Shanmugam, 2015; Nowak and Bucheli, 2017) isolated anthropogenic from manufactured nano-particles as demonstrated close by impact from mechanical technique and afterward again coming about in light of targeted-arranged generation.

Additionally, there have been a current drive to depict much more energetically nano-particles, particularly collected nano-particles (ENPs) (Nicolae *et al.*, 2018). There have been a few reports on nano-particles and nano-movement (The Royal Academy of Engineering 2004 and The Royal Society and NIOSH 2014) respectively. It is evenly seen as perceived that while the vitality of other little particles dependably uses a chief scale to delineate size, by greatness of nano-particles, such a twisted definition, excuses their erratic and different properties. This has started now and again to the use of the properties of the materials as a key

depicting factor regarding the material, which is at present being endorsed (Heera and Shanmugam, 2015)

2.2 Types of nano-particles

2.2.1 Inorganic nano-particles:

In the domain of contemporary material science, dormant nanoparticle has progressed and the activity dependent on their specific physical properties and fantastically in bio_technology (Ladj *et al.*, 2013). Centred upon these two issues of lethargic nano-particles they have certain properties that generally include size penniless, engaging, visual, reactant and electric properties. Bio related presentation are made for the investigation of these captivating nano-particles like iron oxides, silver, gold, silica, quantum spots, (Ladj *et al.*, 2013 and Mark *et al.*, 2011).

2.2.2 Polymeric nano-particles

Polymeric nano-particles as demonstrated by Abhilash (2010), is in like manner a kind of nano-particles. In the present years' polymeric nano-particles has an enhanced improvement in the field of research. The spreading of polymers preformed and the polymerizing of monomers are two in number systems from a general point of view included for organizing, 10-1000nm is the mix of size conflicted with strong - particles (Nagavarma *et al.*, 2012).

2.2.3 Solid lipid nano-particles

Heera and Shanmugam (2015) and Abhilash (2010), highlighted that strong lipid nano-particles was utilized for managing the medication move in 1990s and anticipated an especially fundamental action. There are sure substitute transporter

plans to liposomes, emulsions, and polymeric nano-particles as colloidal Carrier plot. (Abhilash, 2010).

2.2.4 Nanocrystal

A nanocrystal as founded by Amudha *et al.* (2014) is a sort developed upon quantifiable particles taking in at least one estimation lesser than 100 nanometres and customarily made out of particles in either a specific or poly-crystalline system. Nanocrystals are totals of around hundreds or thousands of particles that chain in a crystalline structure, orchestrated of unadulterated drug with only a slim covering including surfactant or mix of surfactants.

2.2.5 Dendrimers

Heera and Shanmugam (2015), explained that Dendrimers emanates from two_Greek words; Dendron denotating tree and Meros connotating part. Dendrimers has a well-defined structure, degree, and depicted atomic mass that are more effective. Dendrimers are hyperbranched, mono_disperse, three dimensional nanoscales fabricatedPolymers. Sub-nuclear participation and polymer science both showed very much described features of Dendrites (Anirudha *et al.*, 2012).

2.2.6 Nanotube

A nano_tube is nanometer scale tube like procedure (Bououdina *et al.*, 2013). We arrange Nanotubes as associates of the fullerene key party. Their name is gotten from their wide, hollow structure with the dividers encompassed by one-atom thick sheets of carbon called grapheme (Heera and Shanmugam. 2015). These bits are passed at express and unequivocal points of view, as well as a combination of the observing perspective and distinct angles of nanotube properties, such as when the actual

nanotube shell is a metal or a semi-conductor. Single-walled nanotubes (SWNTs) and multi-walled nanotubes (MWNTs) are two types of nanotubes (Bououdina *et al.*, 2013).

2.3 Evaluation/Characterization of Nano-particles

Nano-particles as explained by Heera and Shanmugam. (2015), are totally clasified by their size, surface charge and morphology utilizing such techniques as (transmission electron microscopy (TEM), scanning electron microscopy (SEM) and atomic force microscopy (AFM). The mean particle broadness, their size course and charge impact the solid security and the vivo scattering of the nanoparticles (Sovan *et al.* 2011). Electron microscopy techniques are critical for determining the general state of polymeric nano-particles, and can help determine their stinging propensity. The physical silt content and redispersibility of the polymer scattering are also affected by the surface charge of the nano-particles, as is the in vivo execution. (Kavitha *et al.* 2013).

2.3.1 Zeta potential

The Zeta capacity of a nanoparticle as composed by Aarti *et al.* (2014) is commonly used to portray the surface charge state of nano-particles. It shows the electrical capacity of tests and is affected by the structure of the grain and the medium where it is scattered. Nano-particles with a zeta potential above (±) 30 mV have shown to be tenacious in suspension, as the surface charge forestalls grouping of the particles. (Kavitha *et al.* 2013).

2.3.2 Shape of Particle

SEM describes the nano suspension before taking for assessment, the nano suspension is lyophilized to create strong particles. The strong particles are

covered with platinum compound utilizing a sputter coater (Nagavarma *et al.*, 2012).

2.3.3 Size of Particle

The most important parameters of nanoparticle characterization, according to Jitendra *et al.* (2014), are particle size distribution and morphology. Morphology and size are assessed using electron microscopy. Nano-particles often need to find a balance between small size and maximum stability (Nagavarma *et al.*, 2012).

Molecule size dispersing and morphology as demonstrated by Jitendra *et al.* (2014) are the most basic parameters of delineation of a nano-particles. Morphology and size are assessed by electron microscopy. Also, there is a tradeoff between to some degree size and most over the top power nano-particles (Nagavarma *et al.*, 2012). Polyme emissions can be impacted by molecule size; for example, in vitro, the degradation rate of poly (lactic-co-glycolic hazardous) was found to increase as molecule size was increased (Bououdina *et al.*, 2013).

2.4 Applications of Nano-particles

Nano-particles have been seen as valuable in regions of biomedical applications, for instance, medicine and quality movement, demonstrative gadgets and threatening development treatment, sustenance, it has been comprehensively analyzed all through the earlier decade and moreover nanoparticle made a significant energy on account of their outstandingly little measure and discoveries (Jitendra *et al.*, 2014).

Nevertheless, not quite long nano-particles are utilized in new applications in regions like information and correspondence advancement, mechanical structure, power planning, compound industry, normal planning, solution, in pharmaceuticals and enhancing operators, vehicle guards), (Mann, 2016).

While others that are as of late found are utilized as sunscreens and beauty care

products, coatings, materials, sports merchandise, charges, explosives, and fireworks or their applications are by and by being worked on (for example in batteries, sunbased cells, light sources, energy units, electronic stockpiling media, show innovations, bioanalysis and bio locators, medicate conveyance frameworks, therapeutic inserts and new organs) (Heera and Shanmugam, 2015). Use of nano-innovation in the distinctive field is abridged in the table beneath.

Table 2.1: Fields of Application of nano-technology.

| Applied field | Application |
|-----------------------------|---|
| Nanomedicines | Medical devices, Nano drugs, Tissue Engineering |
| Cosmetics and Chemical | Nanoscale compounds, chemicals and paints, |
| | Coatings. |
| Energy & Military | Weapons, biosensors, sensory enhancement |
| Electronics | Semiconductors chipps, memory storage, photoneca, optuelectronics |
| Materials | biopolymers, points and coatings nano-particles and carbon nanotubes, |
| Agriculture | microscopic and scanning, Atomic- force, Tunneling microscope. |
| Food Sciences and nutrition | Processing, nutra-cetical food, nano-capsules. |
| Environmental Energy | Waterand air purification filters, fuel cells, |
| ScientificTools | Microscopic, scanning and Atomic force, |
| | tunneling microscope |

Source: Sovan et al. 2011

2.5 Measures Adopted in Synthesizing Nano-particles

Generally nano-particles were masterminded particularly by physical and chemical methodologies (Anto *et al.*, 2015). A segment of the generally used physical blendstrategies are solvothermal amalgamation, atom faltering, and sol gel system. Essentially there are two strategies for nanoparticle blend as appeared by Prathna *et al.* (2010), to be unequivocal the Bottom up approach and the Top down methodology. In the Top down frame work, prosattempt to figure nano-particles utilizing relentlessly basic ones to channel through their gathering whethe Bottom up approach is a framework that pushes toward consistently clear and solidly complex structures by beginning at the sub-atomic level and keeping up control of Molecular structure. (Prathna *et al.*, 2010). In Top_down process very large material is chaged to silt particles, in Bottom_up procedure atom is reverted to nuclei and finally to nano-particles, these are the procedures used for the preparation of nano-particles.

2.6 Nanotechnologies in Construction Industry

Nano-advancement can convey things with various momentous features that can improve the present advancement materials, lighter, more grounded and all around improved fundamental composites, better cementitious materials, low upkeep coatings, lower heat move, pace of fire retardant and security, better reflectivity of glass and better strong osmosis of acoustic_absorbers(Lee *et al.*, 2010).

2.7 Nano-Materials in Building Products

Present studies inquire about endeavors expected to evaluate the job of nanoadvancement for the improvement applications in the zone of construction and building materials. adding nano-particles in these items reestablished its properties, quatity and quality. Furthermore, the shear, quality, malleable and flexural nature of cement-based materials were improved. Bhuvaneshwari *et al.* (2011); Bakhoum *et al.*, (2017) and Mann (2016) indicated the utilization of nanodevelopment on a couple of construction materials among which are:

Nano glass: Glass can be made to make them clean by itself, and cleansing of foul properties by utilizing TiO₂ in nano structure as a glass coat.

Nano solid: The size of the cement atoms can be modified to be in nano size, for example, by adding nano-tubes and receptive nano size silica particles. It has been discovered that using nano cement in a solid blend would take precedence over using carbon strands or nano carbon tubing in concrete.

Nano steel: Addition of copper nano-particles modifies the surface divergence of steel which additionally obliged the development in steel pressure and as such exhaustion breaking. In any case, vanadium and molybdenum nano-particles improves the split issues happening by virtue of incredible stuns. Scientists proclaimed that refreshed steel associations can be passed on by refining the cementite season of steel to a nano size.

Nano-coat for solid: To protect rigid surfaces from abrasive spots and synthetic attack, nanometer thick coatings that are stable and have the potential to make them more self-cleaning can be used. Nano-particles, such as nitrides, phosphors, and nano Al₂O₃, may reveal intriguing surface properties, such as the ability to adsorb charged organisms.

Nano_Sensors for Concrete Structures: Smart nano dust (hard and fast) detectors can be used as far-reaching sensors in concrete, sprayed ostensibly on the structure, or mixed in with the overall blend. By using data on improvements in thickness and

hardness, shrinkage, dampness, and chlorine get together of cement, nano-studies based sensors can be used in solid structures for quality management and flourishing testing.

2.8 Nano materials in concrete

Nano SiO2, Al2O3, TiO2, quartz can be utilized to improve the solid properties and produce invigorated quality, overwhelming and self-compacting concrete.

2.9 Nontechniques for Concrete Mixtures

The expansion of nano-silica (SiO₂) to set-based products will check the defiling of the calcium-silicate-hydrate reaction, which is achieved by calcium pushing down in water, blocking water component, and inciting invigorates in consistency (Bakhoum *et al.*, 2017).

Carbon nanotubes fortifies the crushing performance of strong mortar models and alter their electrical properties which can be applied in succeeding checking and further studies. The addition of 1% carbon nanotubes will increase the material properties of a PC and water blend starter (Bakhoum *et al.*, 2017). Oxidized multiwalled nanotubes displayed stronger updates in both flexural and crushing efficiency, although the reference models remained unchanged (Lee *et al.*, 2010).

Nano-sensors have an unending potential to be applied in solid structures for quality control and quality checking. (To overview solid thickness and consistency, to check shrinkage or temperature and to screen solid restoring and moistness, chlorine fixation, pH, stresses, carbon dioxide, fortress use or vibration), (Lee *et al.*, 2010 and Bakhoum *et al.*, 2017).

2.10 Nano-Technology in Sustainable Construction

Eco-advancement in nano-development is as yet focusing more on possibilities instead of genuine turns of events. As asserted by Allam *et al.* (2014) the primary zone of centre in the development business is to receive new advances planned for diminishing energy and CO₂ discharges during the development procedure just as while the structure is been used. This energy decrease as well as safeguarding of common assets can be accomplished through making new nano-organized materials with expanded toughness.

2.11 Pozzolanas

Pozzolanas, as instituted by Ndububa *et al.* (2015) and ASTM, C618 are " siliceous or siliceous and aluminous material which without any other individual have basically little or zero cementitous properties yet in finely confined structure and presence of liquid can respond with calcium hydroxide (CaO) which is freed upon the hydration of OPC at standard temperatures to contribute to cementitous properties". Hashem *et al.* (2013), further detailed that: "Pozzolana often have the qualities of getting along with the free lime freed during the hydration strategy for OPC to pass on steady, insoluble calcium silicates accordingly lessening the arrangement of mortar and solid assaults from sulfates, salts and chlorides". Ndububa *et al.* (2015) communicated that pozzolans can be isolated into two social occasions:"normal Pozzolana, for example, volcanic debris and diatomic and counterfeit Pozzolana, for example, calcite earth, pounded fuel debris and debris from consumed agrarian squanders, for example, rice husk, sorghum husk and grasshopper bean husk". The expansion of Pozzolana in either a lime or OPC based item has various points of interest, for example, the expenses are generally low and

unquestionably well underneath that of lime or OPC. Additionally, "Pozzolanas like fly debris, rice husk debris and ground granulated impact grounded slag have been found to add to its enhancement (for instance; high quality, high sturdiness and decrease of heat-of-hydration) just as decrease of energy consumed and carbon dioxide created in the creation of concrete (Manasseh, 2010).

2.11.1 Types of Pozzolanas

Manasseh (2010) recognized that pozzolanic materials can be segregated into two: normal pozzolana and artificial pozzolana.

2.11.2 Common pozzolanas:

Common pozzolanas are basically volcanic remains from freshly volcanic exercises. Regular Pozzolans incorporates: Mud and Shales, Diatomoceous Earth, Volcanic Turffs, Opaelinc Cherts, and Pumicites.

2.11.3 Artificial pozzolanas

Made up pozzolanas result from various present day and agro / industrial systems, generally as by-products. Made up Pozzolans include: Fly waste, Blast Furnace Slag, Rice Husk Ash, Silica Fume, Metakaoline, Surkhi, Burnt Clay, and Groundnut Shell Ash.

2.11.4 Pozzolana Classification

The ASTM C 618 grouped normal pozzolana into Class F, Class N, and Class C.

"Class N— Raw or calcined materials of pozzolans that fit in with the reasonable characteristics instance, some diatomaceous earths; opalinecherts and shales; turffs

and volcanic remains or pumicites, calcined or uncalcined.

"Class F—Fly ash ordinarily gotten from heated anthracite or bituminous coal that meets the material necessities for this class as given hence. This class often possess pozzolanic properties".

"Class C—Fly trash normally passed on from lignite or sub bituminous coal that meets the requirements for this class as given in like manner. This class of material, seen to have little pozzolanic properties, additionally has some cementitious characteristics".

2.11.5 Chemical and Physical Requirements of Pozzolan

As stated by ASTM C 618-5 the chemical characteristics of Pozzolan as to different classes of N, C, and F are as tabulated below;

Table 2.2: Chemical Requirements of Pozzolan

| CLASS | N | C | F |
|--|------|------|------|
| Moisture content, max, % | 3.00 | 3.00 | 3.00 |
| Aluminum oxide (Al2O3) + Silicon dioxide (SiO2) + iron oxide (Fe2O3), min, % | 70.0 | 50.0 | 70.0 |
| Sulfur trioxide (SO ₃) % | 4.00 | 5.00 | 5.00 |

Source: ASTM C 618-5

2.11.6 Mineralogical of Pozzolan

As stated in ASTM standards, "material which must have Silicon dioxide (SiO₂),

aluminum oxide (Al₂O₃), iron oxide (Fe₂O₃) must have a consolidated level of 70%"

as expressed in C618-05, dampness content limit of 3%, and LOI start up to 10%, but

the use of common Pozzolan containing up to 12.0% LOI might be endorsed by the

researcher if either adequate execution records or lab test results are made accessible.

These properties change significantly as expressed by (ASTM 618-05), "contingent

upon their starting point, due to the variable extents of the constituents and varied

mineralogical and physical qualities of the dynamic materials. Pozzolanic action

cannot be resolved just by measuring the nearness of silica, alumina and iron. The

measure of formless material as a rule decides the reactivity of common pozzolana".

The constituents of a characteristic Pozzolan can start in different structures, running

from shapeless receptive materials to crystalline items that will respond either

gradually or not in the slightest degree. Since the measure of material cannot be

dictated by standard systems however it is imperative to assess every regular

Pozzolan to affirm its level of pozzolanic movement. By and large, undefined

silica responds with calcium hydroxide than that silica in the crystalline structure.

2.12 X-Ray Florescence (XRF) Method of Detecting Materials

The American Society for Testing and Materials (ASTM 618-05) defines XRF as the

"transmission of a characteristic (fluorescent) x-ray beam from a substance that has

20

been bombarded with high importance x-rays or gamma rays." The immediate association of particles as they come into contact with X-radiation causes an evaluation of major follow sections of earth materials by XRF. A XRF spectrometer goes after the ground that a model is enlightened by an astounding X bar, known as the incident beam zone of the centrality is dissipated, in any case some is additionally heated up inside the model in a way that relies upon its science. The scene X-fragment beam is usually made using Rh centre, regard less of the way that W, Mo, Cr and others can in like way be utilized, subordinate upon the application. Unequivocally when this head X-ray section bar enlightens the model, it is supposed to be fortified. The empowered test in this way conveys X-column along an extent of frequency characteristics of the sorts of particles present in the model".

2.13 Sorghum Husk

"Sorghum is a significant nourishment crop delivered in enormous amount in the savannah belt of West Africa and Nigeria. It positions among the three significant grain crops developing especially in the northern conditions of Nigeria. Sorghum is for the most part reaped and handled physically for nourishment, leaving the huge volume of buildup comprising waste in the homestead, a large portion of which are flared off in anticipation of consequent cultivating season" Ndububa *et al.* (2015).

Roughly, twenty kilogram of sorghum debris are gotten from hundred kilogram of sorghum husk. Sorghum husk contains 80% common substances and 20% inorganic material (Tahomah *et al.*, 2017). The outcome made from sorghum preparing is known as sorghum husk that envelops the grain. During the milling of the sorghum pad, about 78% of the weight is dealt with as sorghum, fragmented_sorghum and wheat, and the rest 22% of the development is the husk. It has been communicated

that sorghum preparing produces around one ton of sorghum husk produce gotten from five tons of sorghum husk, and it's been foreseen that around 120 million tons of sorghum husk can be gained every year on a general scale for pozzolanic age, as the sorghum husk waste constituent by weight is about 20%, there are extremely 24 million tremendous measures of the SHA obtained as a pozzolana (Chukwu *et al.*, 2011).

Table 2.3: Physical Characteristics of Sorghum Husk

| Structure | Nature / value |
|----------------------|--------------------|
| Size | 4.5mm (average) |
| Hardness | 6 Moh's Scale |
| Bulk Density | 96-100 kg/m3 |
| Fuel Worth | 2800 - 3700 cal/kg |
| Angle of Repose | 35° (ungrounded) |
| Thermal Conductivity | 3.3 K cal-cm/°C |
| Color | Golden/Red |

Source: ASTM C618 (1981)

2.14 Sorghum Husk Ash (SHA)

Manasseh (2010) noted that SHA as reported by Elinwa & Awari (2011), was acquired from consuming of dried sorghum husk, at a temperature of between 600°C - 700°C, after which the consumed husk will be grounded into extremely

fine powder utilizing pestle and mortar (without ball factory) and made to go through 212 microns BS strainer. The husk will as a rule be acquired from sorghum a significant nourishment crop in the tropics; developed in the majority of the focal and northern conditions of Nigeria. Manasseh likewise noticed that SHA is a pozzolanic_material with a particular gravity of 2.41 with its oxide sythesis as displayed by Ndububa *et al.* (2015).

2.15 Mortar

Mortar according to Emmitt and Gorse (2014) is one of the constituents of the composite anisotropic stone work material. The commitment in regards to making a uniform weight transport, changing the irregularities of squares and satisfying mutilations related with warm expansion and shrinkage is furnished on the Mortar (Vladimir *et al.*, 2011). The data about the cemented what's more, new quality of mortar is fundamental to guarantee a regular presentation of square work dividers (Vladimir *et al.*, 2011). Emmitt and Gorse (2014) depicts strong mortar as the blend of concrete and sand at a blend degree and water substance to layout a glue.

2.15.1 Types of Mortar

Mortar have been basically divided into four huge classes, to be explicit; S, M, N and O (ASTM C 270, 2002). In any case, Types M, S, and N are truly required by development standards. Development benchmarks in like manner restricted the usage of specific mortars for explicit applications, for example, exploratory arrangement of foundation construction requires Type M or S mortar and glass unit stone work requires Type N or S mortar. In seismic structure orders, D, E, and F Portland solid/lime or mortar solid mortar Types S or M are required.

Table 2.4: ASTM C270-02 Property Specification Requirements

| Type | Mean strength at 28 days |
|------|--------------------------|
| | min. psi (Mpa) |
| M | 2500 (17.2) |
| O | 350 (2.4) |
| S | 1800 (12.4) |
| N | 750 (5.2) |
| | M O S |

Source (ASTM C270-02)

2.15.2 Properties of Fresh and Hardened Mortar

2.15.2.1 Workability

Workable mortar as indicated by Neville and streams (2010) characterized a new mortar as the measure of valuable internal work (physical property of cement/mortar and work or effort required to conquer the interior contact between the individual particles in the solid) important to deliver full compaction/combination. Workable mortar is the capacity of the mortar/solid mix to be set inside the formwork, and to be adequately compacted/vibrated by hand or mechanical means to clear got air pockets (Lyon, 2012). Factors affecting Workable mortar are; water solid ingredients, mix degrees, size of aggregates, condition of and usage of admixtures as recorded by (Shetty, 2012).

2.15.2.2 Bleeding

Neville and creeks (2010) clarifies that bleeding is a sort of separation where an aspect of the water in the blend will move to the outside of late put mortar surface. The basic issue that caused spilling of mortar is frustration of the strong constituents of the blend to hold the whole of the blending water when they settle downwards.

2.15.2.3 **Density**

Mortar density /concrete density was described by Kazjonovs *et al.*, (2010) as mass per unit volume. BS EN 12390-7:2009 can be used to measure density, also known as unit mass or unit weight in air. Density is calculated by dividing the total mass of all materials in a batch of mortar/concrete by the volume packed by the mortar. According to the standard, mortar/concrete specimens with a density greater than 2000 kg/m³ are classified as normal weight mortar/concrete (ASTM C 140, 2003).

2.15.2.4 Segregation

Shetty (2009) explained that segregation is the unit of the constituent materials of scattering in the mix. A better mortar/concrete is one which has everything on the inside properly dispersed to make homogeneous mix. It is a state of mix of concrete wherein the constituents will be secluded from one another and the goal will not be achieved.

2.16 Compressive Strength

Shetty (2009) portrayed that the quality of mortar/concrete is it's resistance from breaking that might be estimated in number of ways, for example, quality in pressure, in strain, in shear or in flexure. Compressive quality of mortar/concrete is a significant property that is legitimately identified with numerous different properties, in this way is the most generally utilized in concrete practice (Garba, 2010). Quality of samples is likewise decided somewhat by the general properties of the concrete, coarse and total compactions. Components that impacted compressive quality of cement are; amount, type, and nature of concrete, level of compaction, age, water/concrete proportion, relieving conditions. The compressive quality of cement is normally evaluated by the cube test. This is completed on a block tests which is

squashed in an electrical or manual compressive testing machine (Neville, 2012).

2.17 Abrasion Tolerance

Abrasion is characterized by Lamond & Pielart (2016) as damage or tear caused by hard particles or hard protrusions being pressed against and sliding along a solid surface. Abrasion resistance refers to a surface's ability to resist being worn away by rubbing and friction. The following are the different types of concrete wear:

- Wear on sliding surfaced structures, for example, spillways, dams, connect wharfs, because of the activity of rough activities conveyed by streaming water (disintegration).
- ii. Wear on solid floors.
- iii. Wear on solid street surfaces because of overwhelming shipping, and autos, with and without studded snow tires or chains (whittling down, in addition to scratching and percussion).
- iv. Scratched spot restriction is the limit of a surface to withstand being disintegrated by using, scouring and pounding.
- v. Wear of strong surface can be recorded as follows: Wear on concrete tunnels, dams, spillways, and other water- carrying systems where high speed and negative pressure are present. This is generally regarded as cavitation erosion (cavitation) (Castro *et al.*, 2011)

To withstand abrasion from scratching, impact, scouring, slipping, scraping, grinding, gouging, scraping, or slicing from mechanical or hydraulic forces, the following factors must be involved in the analysis and development of concrete surfaces. The

failure of concrete surfaces to resist abrasion can be attributed to a number of factors, including insufficient compressive strength, soft aggregate, excessive curing or finishing, or overhandling during the finishing process (Rajput, 2016).

2.18 Water ingestion

Ingestion (as a proportion of penetrability) is depicted as a method through which liquid invades into and fill porous medium inside a solid body, for instance, paste, mortar or concrete (ASTM C125, 2015). Castro *et al.* (2011) depicted that water ingestion is a huge perspective for estimating the strength of cementitious structures. Water maintenance routinely depends on the unique volume of pores, filler type, thickness and intrusion structures of the strong as delineated by (Rajput, 2016); Nambiar and Ramamurthy, 2017). Water absorption, as described by Castro *et al.*, (2011), is the potential of concrete to absorb water through capillary suction. Concrete with a lower water absorption rate can protect the reinforcement design inside it better (Nwaubani & Poutos, 2013).

According to Williams *et al.*, (2014), the total absorption of concrete test specimens should not exceed 5%, and individual units should not exceed 7%. Saraswathy *et al.* (2012) tested the water absorption of concrete containing up to 30% RHA according to ASTM C642-06. When concrete was replaced with RHA in all proportions, the coefficient of moisture absorption decreased, according to the author.

2.19 Summary

Current research focuses on projects that aim to assess the role of nanotechnology in improving construction and building materials. By incorporating nanoparticles into these things, their qualities, quantity, and quality were restored. Cement-based materials' shear, quality, malleability, and flexural properties were also enhanced.

Nano-development has been used on a few of building materials, according to Bhuvaneshwari et al. (2011), Bakhoum et al. (2017), and Mann (2016).

NS has been found to favourably alter the characteristics of cementitious materials under typical mixing and curing temperatures (22 2 C) in several investigations (Abbas, 2011; Jonbi, 2013; Khanzadi, 2010; Senff et al.2016 and Sonebiet al.2012). By speeding hydration processes and improving the pore structure, they increase the compressive and flexural strengths of mortar mixes while also shortening the setting time. As a result, the major purpose for this study, which examines the influence of NSPs on the hydration of masonry mortar joints in early fall periods (temperature threshold of 51°C), is to solve some of the concerns associated with masonry construction's delayed setting time.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

The materials utilized for this examination work include: Silica Nano-particles (SNPs), Calcium Carbide Waste (CCW), Sorghum Husk Ash (SHA), sharp sand and water, High range water reducer admixture (HRWRA).

3.1.1 Calcium Carbide Wastages

Calcium Carbide Waste (CCW) is a by-effect of acetylene gas products and were gathered from the roundabout region of the vehicle welder's workshop in the town of Bida Local Government Area of Niger State. The CCW was sun-dried for 4 days to reduce the water, after which it was crushed in a nearby plant at Bida milling plant and sieved with 75 µm apparatus.

3.1.2 Sorghum husk ash

Sorghum husks ash (SHA) is an eventual outcome of the devouring improvement of sorghum husk after obtain of the sorghum grains. The sorghum husk (SH) was gathered from Bida Local Government Area, Niger State and was sun-dried for 4days before devoured in the heater. The ensuing powder were ground in a preparing machine at National Cereal Research Institute (NCRI) Bida and sieved with 75 μ m apparatus. The ground remains were kept freely in a desiccator for 2 days foreseeing other depiction tests.

3.1.3 Extraction of Silica Nano-Particles

The STEP-B centre for Genetic Engineering & Bio-information at the Federal University of Technology Minna prepared the silica nano-particles. Nano Silica was obtained from SHA samples using an acid pretreatment process in which 1200 g of SHA was weighed and immersed in an acidic medium (hydrochloric acid) within a concentration of 1N acid solution for 2.5 hours at temperature with constant stirring. After draining the acidic solution, SHA was later rinsed with deionized water until the pH reached 7.0, it however filtered and dried in oven for about 24 hours at 500°C. In addition, the Los Angeles abrasion system was used for additional milling.

3.1.4 Mixing water

The water utilized for the production and restoring of mortar preliminary of this examination work is clean drinkable water stored at Building Laboratory of the Federal polytechnic Bida.

3.1.5 Fine aggregate

The sand utilized for this appraisal work is the reflected reference sand (size range 1.18 mm to 75 µm sieved out from the open ordinary sand picked up from Bida Local

Government Area, Niger State in consonance with BS EN 197:2003) reference sand answer for quality test on binder.

3.1.6 High Range Water Reducer Admixture (HRWRA)

The superplasticizer utilized for this examination work is Conplast SP430 a High-Performance Super Plasticising Admixture (HRWRA), lively improvement for the hydration of substances and, along these lines, higher attributes at early age. The ideal impact is gotten by circuit of Conplast SP430 in the mortar after the expansion of blending water in the blender. It is utilized from 0.3 to 2.0 liters per 100kg of the matrix binder. Conplast SP430 meets the necessities Type A. E. and F. of BS EN934-2:2009 and (ASTM C494, 2015).

3.2 Research Work plan

i. Work plan one

Work plan one was intended to accomplish objective number one which focused on the

extraction and charaterization of sorghum husk debris for nano-silica potentials

- a) Burning husk to obtain ash
- b) Acid leaching to extract the silica
- c) Milling using the los Angeles abrasion machine

ii. Work plan two

Work plan one was proposed to achieve target number two which concentrated on looking into physio-mixture properties of the individual compounds (SHA,

CCW and SNPs). Trial of the materials were dissected in the laboratory office for require gravity, bulk mass, and other physical properties.

- a) X-ray diffraction (XRD)
- b) Quantitative synthetic examination by X-ray fluorescence (XRF)
- c) Scanning Electron Microscopic Analysis
- d) Multi plot BET
- i. particle size
- ii. surface coverage
- iii. pore volume
- iv. Pore curvature

iii. Work plan three

Work plan three was intended to accomplish target number three which manages the improvement of reasonable blends of the SNPs materials for appropriate binding properties.

This includes making the essential blend of the materials SNPs/SHA_CCW at different rate of SNPs (0.5; 1.0; 1.5; 2.0; 2.5; 3.0; 3.5; 4.0; 4.5 and 5.0) of the mixes to be examined while the SHA_CCW (70/30) was utilized to fill in as control.

Different tests on binder (consistency, setting time, soundness, pH test and fineness test) were done on the mix combinations of SHA_CCW (70/30) at 1:3 binder: sand mix in consistence with the BS measures for quality test on cement and affirmation of the degree of hydration.

iv. Work plan four

Work plan four were proposed to achieve target number four which oversees taking a gander at the effect of mechanical and durability properties of mortar made from the clasp blend containing SNPs blended in with SHA_CCW. The properties that were investigated here were dissected using the going with tests and models sizes:

Abrasion resilience – 50 mm mortar cube at 28, 56 age.

moisture – 50 mm mortar cube shapes at 28 and 56 day curing.

Compressive and flexural strength – 50 mm mortar cubes at 3, 7, 14, 28, 56 and 90day curing age.

3.3 Methods

3.3.1 Mortar mix details

The mortar specimens were prepared using 50mm cube size, a binder combination of SHA_CCW (70/30) which has been established by previous research (Egwuda 2017) to give optimum strength was used, moreover optimization was carried out for water proportion at 0.35, 0.40, 0.45. 0.50, 0.60 and super plasticizer at 0%, 0.5%, 1.0%, 1.5%, 2.0%, and SNPs at 0.5% to 5.0% at 0.5 advance increment. Every mix proportion yielded twenty-one (21) cubes, for a total of 241 cube specimens that were cast and cured for 3, 7, 14, 28, 56, and 90 days period, separately. The mortar ingredients were batched and mixed at a ratio of 1:3 (c/s) by weight. Moreover, 0.5 water/cement (w/c) ratio was chosen, which is in line with BS EN 196-1:2016. For the alternate binders of various ratio combinations of SNPs, SHA CCW (70/30) was used as a sensor, and dry room temperature was measured. Table 3.3 shows the mixture design proportions for making around 30 kg of mortar.

Table 3.1: Mix design to optimize water-cement ratio

| | | FLOW mm | | |
|---------|-------------------------|--|--|--|
| SHA 70% | CCW 30% | Sand | Water | |
| | | | | |
| 163.3 | 70 | 699.7 | 81.65 | 0 |
| 163.3 | 70 | 699.7 | 93.32 | 0 |
| 163.3 | 70 | 699.7 | 104.98 | 0 |
| 163.3 | 70 | 699.7 | 116.65 | 85 |
| 163.3 | 70 | 699.7 | 139.98 | 140 |
| | 163.3 163.3 163.3 | SHA 70% CCW 30% 163.3 70 163.3 70 163.3 70 163.3 70 | 163.3 70 699.7 163.3 70 699.7 163.3 70 699.7 163.3 70 699.7 163.3 70 699.7 | SHA 70% CCW 30% Sand Water 163.3 70 699.7 81.65 163.3 70 699.7 93.32 163.3 70 699.7 104.98 163.3 70 699.7 116.65 |

Table 3.2: Mix design to optimize HRWRA

| HRWRA | | Mass of materials (g) | | | | |
|-------|----------|-----------------------|---------|----------|-------|-----|
| | w/c(0.5) | SHA70% | CCW 30% | Sand | Water | mm |
| 0% | 350 | 490 | 210 | 2,099.25 | 350 | 80 |
| 0.5% | 350 | 490 | 210 | 2,099.25 | 350 | 95 |
| 1% | 350 | 490 | 210 | 2,099.25 | 350 | 110 |
| 1.5% | 350 | 490 | 210 | 2,099.25 | 350 | 135 |
| 2% | 350 | 490 | 210 | 2,099.25 | 350 | 135 |

Table 3.3: Mix design of Silica-Nano Particles

| SNPs | Mass of materials (g) | | | | | |
|------|-----------------------|---------|---------|----------|-------|--|
| | HRWRA | SHA 70% | CCW 30% | Sand | Water | |
| 0.5% | 10.5 | 490 | 210 | 2,099.25 | 350 | |
| 1.0% | 10.5 | 490 | 210 | 2,099.25 | 350 | |
| 1.5% | 10.5 | 490 | 210 | 2,099.25 | 350 | |
| 2.0% | 10.5 | 490 | 210 | 2,099.25 | 350 | |
| 2.5% | 10.5 | 490 | 210 | 2,099.25 | 350 | |
| 3.0% | 10.5 | 490 | 210 | 2,099.25 | 350 | |
| 3.5% | 10.5 | 490 | 210 | 2,099.25 | 350 | |
| 4.0% | 10.5 | 490 | 210 | 2,099.25 | 350 | |
| 4.5% | 10.5 | 490 | 210 | 2,099.25 | 350 | |

5.0% 10.5 490 210 2,099.25 350

3.3.2 Experimental Procedures

The experimental processes utilized in this research are classified into four groups (preliminary tests, early-age strength developments, mechanical properties) with the discussion giving the necessary attention to important experiments.

3.3.3 Preliminary-tests

The starter research facility trial of materials that were completed to get the properties of the constituent materials were; chemical examination of different cementitious materials, the molecule size circulation, moisture quantity and gravity.

3.3.4 Chemical organization and portrayal of materials

samples of the SNPs was in like manner sent to STEP-B place for Genetic Engineering and Bio- information at the Federal University of Technology Minna for Particle Size Distribution (PSD) examination and the Brunauer–Emmett–Teller (BET) express surface by Nitrogen (N2) adsorption.

The materials (SHA, CCW and SNPs) were packaged and sent to Ewekoro Works Department of Lafarge Cement Lagos to establish X-ray Fluorescent (XRF) and X-ray Diffraction (XRD) examination for information on the microstructure, oxide association and the level crystalline nature of the materials.

3.3.5 Bulk density of constituents

The density of sample was carried out according to BS 812-2 (1995). The apparatus are wooden cube, trowel, rammer and checking balance.

$$D = \frac{M}{V} \tag{3.1}$$

Where:

D = Density of the samples in kg/m³

M =weight of the samples in kg

V = Volume of the samples in m³

Additionally, the mass of the totals test was controlled by subtracting the mass of void jar from the mass of jar in addition to substances example utilizing equaution 3.1.

M = B - A

Where:

M = mass of the samples in kg

A = mass of the unfilled jar in kg

B = mass of jar plus substance in kg.

3.3.6 Sieve examination of materials

Particle size analysis of the fine sand was done using the dry-sieve method in agreement with BS EN 196-1:2016 for accurate analysis of the fine sand. The equipment utilized are the sieve of varied sizes (pan, 75μmm, 150μmm,300μmm, 600μmm,1.18mm, 2.36mm, 4.75mm,), scoop, sieve brush, weighing balance, electronic balance, sieve shaker and clock. The reference sand needed for mortar production in strength determination analysis specified in the requirement (BS EN 196-1:2016) were then produced using an adjustment of sieve size 1.18 mm and 75 μm. The particles going through the 1.18 mm sieve but trapped on the 75 μm sieve was adopted for the mortar mixture for the performance test. Because the 1.6 mm sieve was not available in the laboratory, the 1.18 mm sieve was chosen as the higher upper limit for the simulated reference sand instead of the 1.6 mm sieve specified by BS EN 196-1:2016.

The sieve analysis results from the test were sketched on a semi-log graph with the particles size or sieve size as the X-axis, the logarithmic axis as the logarithmic axis, and the percent passing as the Y-axis, as shown in figure 4.1. D60 represents the

grain size passing at 60%, D30 represents the grain size passing at 30%, and D10 represents the grain size at passing 10%.

Consistency Coefficient (Cu) =
$$\frac{D60}{D10}$$
 (3.2)

Coefficient of Curvature (Cc) =
$$\frac{D30^2}{D10*D60}$$
 (3.3)

All around reviewed necessities were consequently exhibited as: $Cu \ge 4$ for rock; $Cu \ge 6$ for sand and Cc = 1 to 3 for all kind of soil. Soils having a Cu < 2 are delegated consistently reviewed. It was expressed that both Cu and Cc demonstrates the dirt arrangement, Vladimir *et al* (2011).

3.3.7 Moisture content of materials

This involves oven-drying known weights of the aggregate sample for 24hours at a temperature above 105°C. The weights will be taken after drying to determine the weight of water evaporated and that of the dry sample (Neville, 2012).

The moisture content was be calculated as follows;

Moisture content =
$$\frac{\text{Initial weight of sample} - \text{Dry weight of sample}}{\text{Dry weight of sample}}$$
(3.4)

3.3.8 Materials Specific Gravity

The specific gravity of the test ingredients was settled as showed by the British Standard, the framework was driven according to BS EN 1097: 2003. The gadget used for the test consolidate thickness container and attachment, spatula, funnel and checking balance.

The specific gravity (Gs) of the materials was resolved using equation 3.5

specific gravity Gs =
$$\frac{(W2-W1)}{(W4-W1)-(W3-W2)}$$
 (3.5)

 w_1 = the mass of container

w₂ =the mass of bottle plus Dry soil

 w_3 = the weight of bottle plus Soil plus Water

 w_4 = the weight of bottle plus Water.

3.3.9 Flow test

Flowability of the fresh mortars was carried out in conformance with the test method described in CSA A3004-C1-08 (CSA 2008c) by using a standard flow table. For this practical, the cone mold was put at the middle of the flow table and filled with mortar mixture to a thickness of about 25 mm which was tamped 20 times with a tamping rod. The cone was then completely filled with the mortar mixture and tamped 20 times more. After that, the mold was rapidly removed, and the flow table handle was rotated 25 times in 15 seconds to drop mortar 25 times. Finally, the diameter growth was measured on the flow table at 45° along the prescribed lines, the flow value was calculated by averaging the readings.

3.3.10 The air content test

The air content of fresh mortar mixtures was determined according to CSA A3004-C4-08 (CSA 2008d), test method based on fresh mortar with a volume of 400 cm³. For calculation of the air content, mass and density of the nano-particles as well as the HRWRA were taken into consideration according to the following equation:

$$D = \frac{\text{cement mass+sand mass+water mass+HWR mass+Nanoparticles mass}}{\frac{\text{cement mass}}{\text{DC}} + \frac{\text{sand mass}}{\text{DS}} + \frac{\text{water mass}}{\text{Dw}} + \frac{\text{HWR}}{\text{Dh}} + \frac{\text{nanoparticle mass}}{\text{Dn}}}{\text{Dn}}}$$
(3.4)

Where:

D = density of the air-free mortar

Dc = density of cement,

Ds = density of sand, g/cm^3

 $Dw = density of water, g/cm^3$

Dh = density of HRWRA, g/cm³

 $Dn = density of nanoparticle, g/cm^3$

Equation (3.5) below was used to calculate the air content

$$A = 100 - \frac{M}{4D} \tag{3.5}$$

Where:

A = percentage of air content

M = mass of mortar, g

D = density of the air-free mortar

3.3.11 Consistency -test

This is completed to decide the level of water for typical consistency for a given example of concrete. The equipments to be utilized are vicat's device with the plunger device of 10mm measurement, gauging balance, cylinder, trowel and weight box. This involves deciding the consistency of substance. Take 250g of binder and weigh it carefully before adding 20% water. Mixing time should not be shorter than 3 minutes, and gauging should begin when the water is added. Fill the mold with the paste. Trim any extra paste and give it a good shake to get rid of any air bubbles. Fix the 10mm plunger in the moving rod and lower it till it touches the paste. Repeat the operation until the plunger penetrates 33-34mm from the top, then record the water percentage.

3.3.12 Setting time test

The setting time test is done to decide the underlying and last setting time of a given example of concrete. The mechanical assembly to be utilized are vicat's device form and needle, gauging, non-permeable plate, estimating chamber, balance, weight box, and stop watch. The underlying and last setting occasions tests for the binder matrix (CEM I 42.5N; SHA_CCW and the different extent mixes of SNPs) were resolved utilizing perfect glues of standard consistency in understanding to BS EN 196 3:2005. This indicates deciding the dampness substance of the mortar glue which will convey the perfect standard consistency (Neville, 2012).

3.3.13 Soundness test

The aim of the soundness test was to evaluate how the cement paste behaved after being immersed in liquid for about 24 hours and 4 hours, as well as the extent of soundness after the specimen had been boiling for about 3 hours. The Le-cheitelier mould and wash, as well as a plate, spatula, bottle, and vernier calliper, would be included. The soundness of the binders was also tested using a Le-Chatelier device Model No EL 38 – 3400 by ELE in accordance with BS EN 196-3-2005. The binder mix was placed in a lecheiterlier mold, which then was submerged in a lecheiterlier bath filled with clean drinking water and the period of submerged was registered until the mold was tested after 24 hours. A vernier calliper was used to document the increase. The mold with the binder paste then was placed in a bath boiling water and heated for 3 hours. After 3 hours, the mold was removed and examined to see if it had expanded much further.

3.4 The strength and degree of hydration.

As specified in Section 3.3.1, 50 mm mortar cubes have been used to assess the strength and extent of hydration of the binders. Measuring the constituent materials and checking that the SHA / CCW was adequately combined with the SNPs in a container until it was placed on the estimated quantity of the simulated reference sand previously placed onto the steel mixed frame is all part of the mortar sample manufacturing process. After that, the sand and binder were thoroughly combined before adding the mixing water and mixing until a smooth blend was obtained before casting into the 50 mm cubes molds. Until casting into the molds, the control mortar sample has the SHA CCW mixed as mentioned above with the simulated reference sands and necessary quantities of mixing water. The specimens were filled with jute sacks and cured by liquid splash until 72 hours before demoulding and liquid curing by soaking was made to proceed before testing age, based on the findings of the setting times tests stated in Section 3.3.12. As a result, the strength test and extent of hydration assessment procedures are identical to those described in (Lam *et al.*, 2010; Dillshad, 2011; Olawuyi *et al.*, 2017). The following is a summary of the process:

The procedure for the strength test and degree of hydration determination thereby adopt a similar approach as reported in (Lam *et al.*, 2010; Dillshad, 2011; Olawuyi *et al.*, 2017). The procedure is as highlighted below:

- a) To measure the strength development, the mortar cubes were cast and crushed in the Digital Universal Testing Machine (DUTM 20) at various curing ages (immediately after demoulding at 3, 7, 14, 21, 28, and 56 days)...
- b) The remnant of the example in (a) above were then processed appropriately utilizing the 150 mm x 150 mm Θ round and hollow molds accessible in the lab and 25 mm width bar as mortar and pestle. The proceed example was moreover vacuum-dried for 1 hour in other to stop further hydration.

- c) The vacuum-dried example had a known mass of around 25g from the molecule passing 75 m normal strainer, which was measured and dried for 24 hours at 105° C before being gauged again to assess the evaporable water, such as slender water + gel water.
- d) This test was then set in the heater [Model No SNOL 8, 2/1100 1LZ] set to 900 ° C. At one hour time after the heater temperature peruses 900° C, the heater was turned off, permitted to cool and the example weighed for assurance of the measure of synthetically bound water, that is, the non-evaporable water.

LOI of the samples (SNPs, SHA_CCW) and hydrated pastes to be determined by LOI (%) = 100 x (as received weight – ignited weight)/ received mass (3.6)

The content of (non-evaporable water) in the hydrated mortar pastes was measured to determine the degree of hydration as defined in the literature (Lam *et al.*, 2010; Neville, 2012). The disparity in mass calculation of the crushed paste at 900°C and 105° C is used to determine the degree of hydration (α) on the assumption that 1g of anhydrous cement contains 0.23g of w_n , so the w_n is determined using the formula below.

$$W_n\% = \frac{100 \, x \, (dried \, weight \, of \, paste - ignited \, weight \, of \, paste)}{(Ignited \, weight \, of \, paste - loss \, on \, ignition \, of \, cement)} \tag{3.7}$$

The degree of hydration (α) is then:

$$\alpha = 100 x \frac{Wn}{0.23} \tag{3.8}$$

However, the hydration degree in the SHA CCW SNPs binders at the different mixes was determined using the SCM's LOI and their proportion was changed to account for their w n percent as desired.

3.5 Mechanical Properties Tests

It involves determining the mortar's mechanical behaviour, which is the researchers key trial job.

3.5.1 Density test

The mortar samples were ousted from the curing tank and put outside to surface dry, the mass of the specimen is noted using measuring balance in consent to BS EN 12390-7 (2009). The density of mortar samples was determined, utilizing equation 3.9.

Thickness D =
$$\frac{M}{V}$$
 (3.9)

Where the following parameters can be defined as follows:

D = Thickness of the mortar example in kg/m³

M = Mass of mortar example in kg

V = Vol. of mortar example in m^3

3.5.2 Compressive quality

Crushing strength is a significant characteristic of hardened mortar, and many of its behaviour are linked to (Neville, 2012). The mortar specimens (cubes) were taken out of the curing jar and left to dry on the surface before being weighed and placed in the centre of the compression machine to be crushed. The mortar cube specimens were loaded for evaluation, and the crushing machine adapted force to the specimen until it failed to check its behaviour under load. A compressive strength test on mortar specimens is required by European Standard BS EN 196-1:2016.

Compressive strength
$$F = \frac{P}{A}$$
 (3.10)

Where:

 $F = crushing strength in kN/mm^2$

P = Ultimate load at fracture, in kN

A = Sectional area of the specimen on which the compressive force acts.

3.5.3 Water absorption test

The mortar specimen was taken out of the curing jar and left to dry on the surface before being dried further in an electric oven reading 105°C for 72 hours. The samples were taken out of the oven and allowed to cool temperature before being weighed to find the minimum weights, which were then registered as W₁. The final weights were calculated after dipping the mortar specimens in the curing medium for about 24 hours, then removing them, drying them, and weighing them again, with the value W₂ being registered. The results were determined using the values obtained to determine the rate of absorption of the mortar samples in accordance with ASTM C 1881-122. (2011).

Water Absorption =
$$\frac{(w2-w1)}{w1} \times 100$$
 (3.11)

3.5.4 Measure of abrasion resistance

The samples (cubes) were removed out from the curing basin and allowed to dry on the surface before being cleaned and oven dried from 105°C to 110°C. The samples are weighed, and the results are written down as W₁. The cube sample was then put into the Los Angeles abrasion test machine with the requisite steel balls, and the steel cylinder was rotated at a speed of 30–33 revolutions per minute for 500–1000 cycles,

after which the sample was extracted, poured into a 1.7mm sieve, and the material passing through 1.7 mm was weighed, and the quantity was registered as W₂.

Abrasion resistance test =
$$\frac{w^2}{w^1} \times 100\%$$
 (3.12)

Where:

 W_1 = weight of the sample

 W_2 = weight of fraction passing 1.70mm B.S sieve

3.6 Analytical Techniques

The data obtained for the various tests conducted in this study was analyzed using simple statistical methods (mean and percentage). According to Mudasiru (2015), the mean, also known as the arithmetic average, is the most widely used central tendency, and is defined as the sum of scores divided by the total number of points, as shown in Equation 3.13.

$$Mean X = \frac{\sum X}{N}$$
 (3.13)

Where: X (read as X-bar) is the symbol for the mean

A percent, also known as a ratio, is a ratio multiplied by 100. It was used to evaluate the effects of the abrasion tolerance test and the liquid absorbing test in this study.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Materials Characterization

Table 4.1 moreover shows PSD of the CEN reference sand for determining properties of sand and compared to the simulated reference fine sand used. The PSD

demonstrated the reproduced reference sand to have a Cu and Cc estimations of 2.06 and 0.84 separately and a Fineness Modulus (FM) of 2.52 showing a fine sand grouping of Shetty (2009).

| Sieve opening (mm) | CEN Reference Sand (%) | Simulated Reference Sand (%) | Remark |
|--------------------|---------------------------|------------------------------------|-----------|
| 2.00 | 0 | 0 | |
| 1.60 | 7± 5 | 0 | |
| 1.00 | 33 ± 5 | 3 | |
| 0.50 | 67 ± 5 | 16 | |
| 0.16 | 87 ± 5 | 92 | $\sqrt{}$ |
| 0.08 | 88 ± 1 | 99 | $\sqrt{}$ |

Table 4.1: Fine Sand Particle Size Distribution

The simulated reference sand was utilized for the examination regardless of the lack of non-complying with the other three prerequisites since the exploration is essentially a near report on quality advancement of the alternative material and SNPs, but however not approval and confirmation of the SNPs. The quality of the mortar tests from SHA_CCW (70/30) utilized right now simply as a source of perspective to which the quality of the elective fastener SNPs/SHA_CCW was analyzed. We speak to figure (4.1) as the molecule size circulation (PSD) of the accessible characteristic sand and the recreated reference sand utilized for the investigate on.

Table 4.2: Specific Gravity of Materials (kg/m³)

| Materials | Specific gravity | Bulk density (kg/m3) |
|------------------|------------------|----------------------|
| SHA | 2.60 | 2330 |
| CCW | 2.37 | 2300 |
| Sand | 2.57 | 2500 |
| Superplasticizer | 1.06 | 1070 |

The specific gravity and bulk density for the constituent materials is presented in Table 4.2. The result shows the values fit well with earlier reports in the literature (Egwuda 2017 & Neville, 2012).

Table 4.3 pH value of Constituent materials

| Materials | pH value |
|-----------|-------------|
| SHA | 10.30 |
| CCW | 11.85 |
| SNPs | 7.0 |

The materials' pH values are stated in Table 4.3. The outcome indicates that the values are alkaline (Fereshte *et al.*, 2015).

Table 4.4: XRF Analysis for Oxide Composition of Binding Materials

| Elements | % | % | % | ASTM C618 |
|----------|-------------|-------------|-------------|-------------|
| | Composition | Composition | Composition | – 05 limits |
| | SNPs | SHA | CCW | |

| Silicon Oxide (SiO ₂) | 89.30 | 76.10 | 8.69 | $\begin{bmatrix} SiO_2 + \end{bmatrix}$ |
|---|-------|-------|-------|---|
| Oxide of Aluminum (Al2O3) | 0.0 | 2.71 | 1.10 | Al2O3 + ≥ 70.0% |
| Oxide of Iron (Fe ₂ O ₃) | 0.36 | 1.23 | 0.30 | Fe ₂ O ₃ |
| Oxide of Calcium (CaO) | 1.82 | 3.32 | 65.59 | |
| Sulphur trioxide (SO ₃) | 0.03 | 0.13 | 0.10 | 4.0% max |
| Oxide of Sodium (Na ₂ O) | 0.0 | 0.07 | 0.0 | |
| Oxide Potassium (K ₂ O) | 2.30 | 3.91 | 0.10 | |
| Titanium Oxide(TiO ₂) | 0.48 | 0.0 | 0.0 | |
| Mangnesium Oxide (MgO) | 0.0 | 1.27 | 0.05 | |
| Oxide of Chromium (Cr ₂ O ₃) | 0.0 | 0.0 | 0.0 | |
| Strontium Oxide (SrO) | 0.03 | 0.0 | 0.0 | |
| Oxide of Zinc (ZnO) | 0.05 | 0.0 | 0.0 | |
| Loss On Ignition (LOI) | 2.85 | 3.0 | 21.71 | 10.0% max |

Table 4.4 shows the oxide concentrations of the major cementitious materials determined using XRF for SHA and CCW at Lafarge Cement in Ewekoro, Dangote Cement, and the SNPs at the Nigerian Geological Survey Agency. SiO2 is the most abundant component in both the SHA and SNPs samples, with mass values of 76.10 percent and 89.30 percent, respectively. In mortar containing pozzolanic material such as SHA and SNPs, this is the primary oxide component that contributes to the pozzolanic reaction and/or secondary hydration. Other important oxides, such as Oxide (Al2O3) and Fe2O3, were present in reasonable concentrations. There were other key oxides, for example, Oxide (Al2O3) and Fe2O3 in notable amounts. The Table uncover the SHA &Class F Pozzolan having all of SiO² plus Al2O3 plus Fe2O3 above 70%, SO³ beneath 4% and LOI of under 10%.

4.2 Multi Plot BET Result

Table 4.5 shows the results of BET multi plot which reveals that the normal molecule size, BET surface area, pore volume, and pore distance across of SHA tests are

77.05 nm, 723.020 m2/g, 4.448e +01 cc/g, and 2.072e+00 nm separately. The investigation's results showed that the properties of the extracted silica me*et al*l of the criteria for nano-particles as defined by Javed *et al*. (2011).

Table 4.5: Physical Properties of SNPs.

| SNPs Tests | Avg part. size | BET surf. area | pore Vol. | Avg pore dia |
|------------------|----------------|--------------------------------|-----------------|--------------|
| After extraction | 77.05 nm | $723.020 \text{ m}^2/\text{g}$ | 4.448e +01 cc/g | 2.072e+00 nm |

4.3 X-ray diffractogram (XRD)

Appendix D2 shows an X-ray diffractogram of amorphous silica isolated from SHA. The wide X-ray diffraction pattern of amorphous materials (Kamath & Proctor, 2010) confirms that the SNPs were mostly amorphous. Diffraction peak at theta = 22 degrees validates the production of amorphous silica. Diffraction wide peak at theta = 22 degrees implies amorphous silica with some crystalline silica, according to reports (Javed et al., 2011).

Table 4.6 provides the results of moisture content tests for the project's constituent components. The moisture content of fine aggregates should be within 0 to 10% according to ACI E1-99, and the moisture content of binders should be within 0 to 3% according to ASTM618-05, indicating that the fine aggregate and binder tests met the criteria.

Table 4.6: Moisture Content of the Material Sample

| Materials | Weight of material | Weight of material | Moisture content (%) |
|-----------|---------------------|--------------------|----------------------|
| | before oven dry (g) | after oven dry (g) | |
| SNPs | 500 | 498 | 0.4 |
| SHA | 500 | 499 | 0.2 |
| CCW | 500 | 490 | 2.0 |

Sand 100 100 0.0

4.4 Fresh Properties

4.4.1 Flow Test Results

To obtain the desired flowability ($110 \pm 5\%$), trial batches indicated that moderate dosages of 1.5% of HRWRA by mass of binder were needed for the mixtures containing SNPs at 0.5% to 5.0% at 0.5 step intervals. It is worth mentioning that the dosage of HRWRA was kept constant for each mixture within each group, as shown in Table 3.3

The incorporation of SNPs affected the flowability of the mortar mixtures as depicted in Figure 4.2. Flow values indicated that increasing the dosage of the nano-particles decreased the flowability of the mortar mixtures. As shown in Figure 4.2 as the dosage of SNPs increased from 2% to 4%, the flow value was reduced by 7% and 10% at 0.5 w/b ratio. Despite the optimum dosage of HRWRA (1.5%), when 5% SNPs was introduced into the mortar, the target flow of $110 \pm 5\%$ could not be achieved. In conclusion, the higher fineness of SHA_CCW provided better interaction with SNPs, which led to a drop in the flow value.

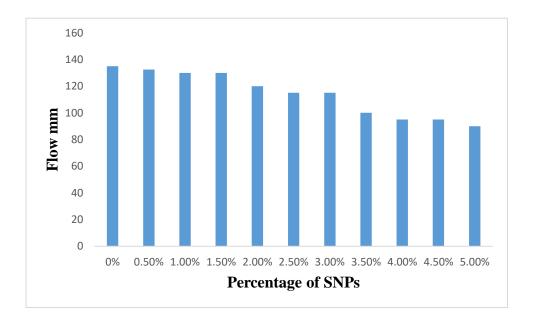


Figure 4.1 Flow results for mixtures of SNPs.

Within each mixture, higher dosage of SNPs decreased the flowability of mortar at a constant w/c and HRWRA due to increasing the cohesiveness of the mixtures. The ultrafine nature of SNPs improved the interaction between grains, and thus van derWaals based particle to particle attractions may become increasingly significant (Sonebi *et al.*, 2012).

Table 4.7: Fresh Properties of Binders with Different SNPs Content

| SNPs | Water Demand | Penetration | Soundness |
|--------------|--------------|-------------|-----------|
| content % | (%) | (mm) | Expansion |
| 001100110 70 | | | (mm |
| 0 | 43.0 | 5.0 | 0.4 |
| 1.0 | 43.2 | 7.0 | 0.3 |
| 1.5 | 43.6 | 5.0 | 0.5 |
| 2.0 | 43.8 | 5.0 | 0.5 |

| 2.5 | 44.0 | 7.0 | 0.4 |
|-----|------|-----|-----|
| 3.0 | 44.2 | 7.0 | 0.5 |
| 3.5 | 44.5 | 5.0 | 0.3 |
| 4.0 | 44.8 | 7.0 | 0.4 |
| 4.5 | 45.1 | 5.0 | 0.4 |
| 5.0 | 45.2 | 5.0 | 0.5 |
| | | | |

The soundness test result as shown in Table 4.7 revealed that all the binder combinations conform to the 10 mm maximum expansion specified by BS EN 197-1:2000.

4.4.2 Setting times of the SNPs/SHA_CCW Binder

Figure 4.3 present the plot of the setting times (starting and last) for the SHA_CCW matrix blends at different SNPs substance. The normal setting time results for blends containing differed level of SNPs diminishes in both introductory and last setting occasions as the SNPs builds contrasted with the control blends SHA_CCW (70/30). For instance, the diminishing in the underlying setting time for 3% SNPs and 5% SNPs was 152 min (43%) and 230 min (66%). The shorter setting times related with practically all the blends consolidating SNPs, could be as result the presentation of the interfacial change zone between the totals and the SNPs network as estimated by the entrance opposition.

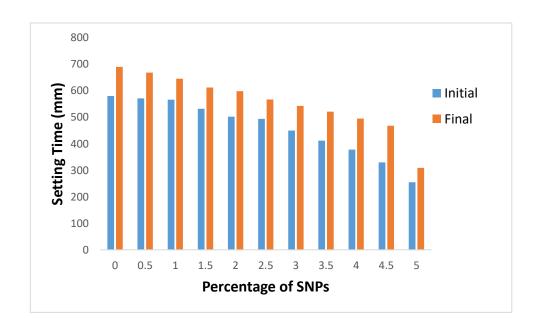


Figure 4.2: Setting times of the SNPs/SHA_CCW Binder

4.4.3 Air Content

The incorporation of SNPs into the mortar mixtures increased their fresh air content as presented in Table 4.8. For all the samples, the step up in the air content of the SNPs mixtures is in a range of about 20% of the air content of control mixtures. The lower density of fresh mortar mixtures incorporating nano-particles was an indication of the higher air content which is in understanding with other researches (Senff *et al.*, 2016).

Table 4.8: Density and air content of fresh mortar mixtures

| Samples | SNPs | Density (g/cm ³) | Air Content (%) | | |
|---------|------|------------------------------|-----------------|--|--|
| | 0% | 2180 | 15 | | |
| | 0.5% | 2177 | 15 | | |
| | 1.0% | 2175 | 16 | | |
| | 1.5% | 2175 | 16 | | |
| | 2.0% | 2165 | 16 | | |
| | | | | | |

| SHA_CCW | 2.5% | 2155 | 17 |
|---------|------|------|----|
| 70/30 | 3.0% | 2145 | 17 |
| | 3.5% | 2130 | 17 |
| | 4.0% | 2110 | 17 |
| | 4.5% | 2010 | 18 |
| | 5.0% | 1995 | 18 |

4.4.4 Extent of Hydration of Hardened SNPs/SHA_CCW Mortar

The results of degree of hydration of the SNPs/SHA_CCW binders as presented in Figures 4.3 revealed that 3 % SNPs binder combinations as the best of the binder combination with 42.90 % levels of degree of hydration by the 28 day curing age.

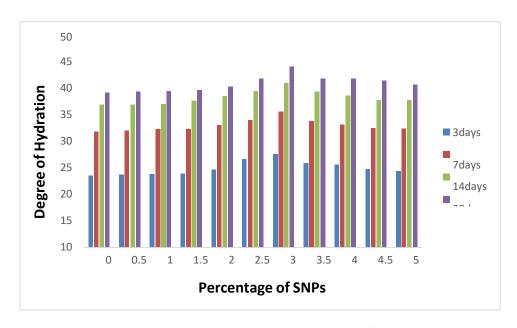


Figure 4.3: Extent of Hydration of Hardened SNPs/SHA_CCW Mortar

The result of specimen without SNPs binder was 36.72 % level of hydration with RH₂₈ value of 0.85 referenced to the 28 days value of 3 % SNPs at 28 days curing age. Hydration was observed benhance as the curing age increased and the binders were expected to show better long-term age strength performance, although a decrease was observed at 5 % SNPs binder with 38.61 % level of hydration and RH₂₈ factor of 0.9.

Table 4.9: Degree of Hydration and RH₂₈ Factor of the SNPs/SHA_CCW Binders

| Binder | SNPs | Degree of Hydration | | | RH ₂₈ Factor | | | | |
|---------|----------|---------------------|-------|--------|-------------------------|-------|-------|--------|--------|
| Type | Specimen | 3days | 7days | 14days | 28days | 3days | 7days | 14days | 28days |
| SHA_CCW | 0% | 17.01 | 27.43 | 33.79 | 36.72 | 0.39 | 0.63 | 0.78 | 0.85 |
| | 0.5 | 17.21 | 27.65 | 33.81 | 36.89 | 0.40 | 0.64 | 0.79 | 0.86 |
| | 1.0% | 17.35 | 27.97 | 33.89 | 37.01 | 0.40 | 0.65 | 0.79 | 0.86 |
| | 1.5% | 17.51 | 28.01 | 34.76 | 37.27 | 0.41 | 0.65 | 0.81 | 0.87 |
| | 2.0% | 18.47 | 28.96 | 35.82 | 38.07 | 0.43 | 0.76 | 0.83 | 0.88 |
| | 2.5% | 20.87 | 30.09 | 37.02 | 39.98 | 0.48 | 0.70 | 0.86 | 0.93 |
| | 3.0% | 22.08 | 32.18 | 38.87 | 42.90 | 0.51 | 0.75 | 0.90 | 1.0 |
| | 3.5% | 19.89 | 29.87 | 36.89 | 40.01 | 0.46 | 0.70 | 0.86 | 0.93 |
| | 4.0% | 19.67 | 29.07 | 35.91 | 39.97 | 0.45 | 0.68 | 0.83 | 0.93 |
| | 4.5% | 18.57 | 28.23 | 34.86 | 39.54 | 0.43 | 0.66 | 0.81 | 0.92 |
| | 5.0% | 18.01 | 28.09 | 34.85 | 38.61 | 0.42 | 0.65 | 0.81 | 0.90 |

4.5 Hardened Properties

4.5.1 `Compressive Strength

The type of binder and dosage of SNPs is affecting the strength of the mixtures, depending on the age of testing (Figure 4.6). Generally, all the mixtures had an increased compressive strength until 3.0% SNPs before the strength starts experiencing a decline which do not conforms to the air content results and setting times of these binders.

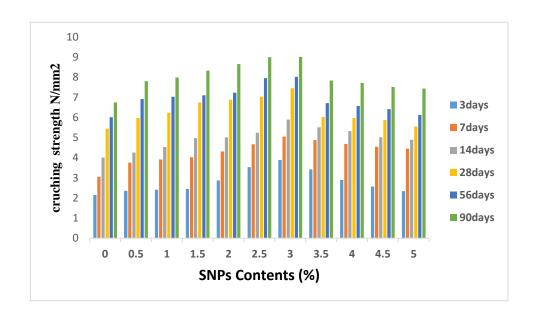


Figure 4.4: Compressive strength results of mixtures containing SNPs

The early age (3 days) hydration values for the mortar binders was observed to improve with addition of SNPs. The plot of the compressive strength of the binders (Figures 4.6) was observed befollow similar trend as the inference drawn from the degree of hydration results. 2.5% and 3.0% SNPs gave 28day compressive strength values of 7.02 N/mm² and 7.45 N/mm² which represent 98% and 92% of SHA_CCW (70/30) strength. 2.5% and 3.0% SNPs also gave 90day compressive strength values of 8.99 N/mm² and 9.01 N/mm² which represents 155% and 132% of SHA_CCW (70/30) strength. The high strength can be adduced to the high rate of hydration on the binders on basis established from the result of the setting time test.

More so, the crushing strength development at early ages can be ascribed to the well-dispersed SNPs provided nucleation sites which led to acceleration of cement hydration, which conforms to the degree of hydration values in the sense that addition of SNPs shortened the dormant period.

4.5.2 Early age crushing strength

The early age (3 and 7days) compressive quality in figure 4.5 was utilized to advance water proportion of the binder, it was seen that 0.5 w/c gives an obvious value of 2.5N/mm2 and 2.65 N/mm2 at 3days and 7days individually.

Furthermore, after demoulding 72 hours (3 days) of casting, the cases were observed to bind effectively with the fine aggregates. Throughout the curing ages of this experiment, the mortar made from SNPs/SHA CCW based binders did not dissolve in the submerged water in the curing tank..

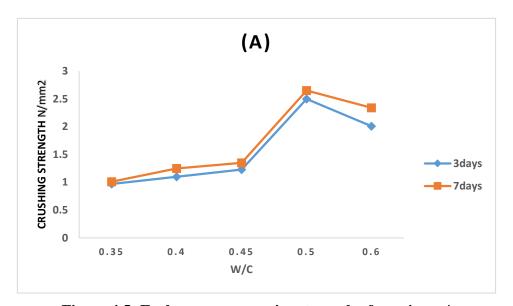
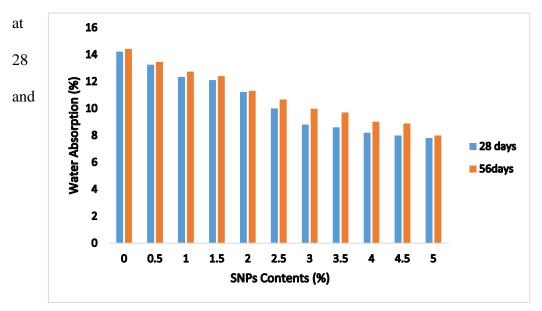


Figure 4.5: Early-age compressive strength of varying w/c

4.5.3 Water absorption of SHA_ CCW mortar with SNPs

The figure 4.7 report the result of water absorption test done out on mortar made from SHA_CCW containing SNPs binder combination specimens cured and tested



56curing days. The results revealed an average value of 14.18 % for control (SHA_CCW) while 2.5 % and 3 % SNPs absorbed 10.67 % and 9.98 % at 56 days. The results revealed that both fall within the limit of 20% by weight specified by Rajput (2016) for building work. It was however noted that the quantity of water absorbed by binder combination of control (SHA_CCW with SNPs) twice that absorbed by the specimen containing 5 % SNPs. The quantity of water absorbed by binder combination of SNP decrease as the curing age increase

Figure 4.6: Water absorption of SHA_ CCW mortar with SNPs

4.5.4 Average density of SHA_CCW and SNPs mortar

The normal thickness of SHA_CCW mortar and fluctuated SNPs matrix mix restored in ordinary water and weighed at 3, 14, 28, 56 and 90 days individually were displayed in figure 4.8. The thickness of 3D shape tests differs from 1799 kg/m3 to 2185 kg/m3 and increment with increment in SNPs yet diminishes with restoring ages. Mortar tests with higher thickness than 2000kg/m3 are known as would be expected weight mortar/concrete as indicated by ASTM C140. In this way, 5 % SNPS mortar is a mortar with higher thickness.

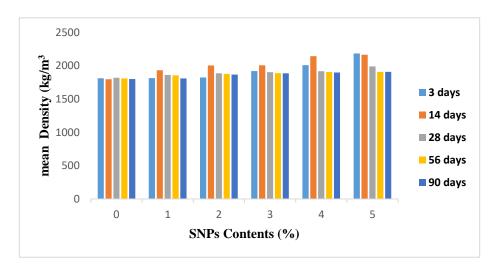


Figure 4.7: Average density of SHA_CCW mortar with SNPs

4.5.5 Abrasion resistance test results of SHA_CCW mortar with SNPs

The result in figure 4.8 results shows average durability values of 92.12 % for SHA_CCW while 3 % SNPs is 94.45 % 90 days. This implies that SNPs inclusion in SHA_CCW binder-based mortar will result in improved resistance to wear and abrasive attack as against samples with SHA_CCW binder combination. The abrasion resistance general increase as the curing age increase.

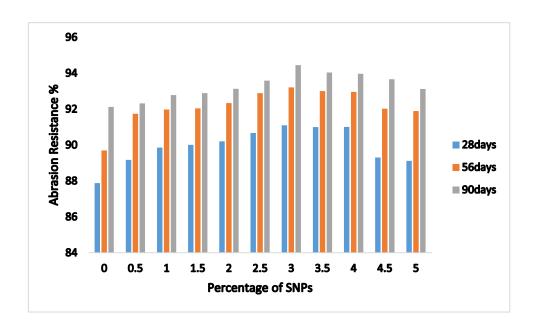


Figure 4.8: Abrasion resistance test results of SNPs/SHA_CCW mortar

4.6 Summary of Findings

The exploration work includes the assurance of physiochemical properties of constituents of materials. The level of hydration and quality properties of mortar example produced using varied SNPs content in SHA_CCW were resolved. Water ingestion and scraped spot opposition of the mortar example were likewise assessed in order to evaluate the achievability of its utilization in a situation that is presented to wind, downpour and wear impacts. The highlights of the noteworthy disclosures are according to the accompanying:

- i. The SNPs were primarily amorphous, as evidenced by their wide X-ray diffraction pattern, which is typical of amorphous materials. The development of amorphous silica is confirmed by a diffraction peak at theta = 22 degrees.
- ii. The joined oxide synthesis of Al₂O₃, SiO₂ and Fe₂O₃ substance of SHA gave 80.04 % while the oxide creation of CCW is significantly CaO with a 65.59 % content. CCW has high LOI of 21.71 % while the Loss of Ignition of SHA

- gave 3.0 %. The absolute evaporable dampness substance of SNPs, SHA, CCW are 0.4 %, 0.2 %, and 2.0 % individually.
- iii. The consequence of PSD uncovered that reference sand to have a Cu and Cc estimations of 2.06 and 0.84 individually and a FM of 2.52 which is between the scope of 2.2 2.6 for fine sand. The evaporable damp content substance of the sand was 0.0 %.
- iv. At various ages of 3, 7, 14, 21, 28, 56, and 90 days, there was no noticeable wear or tear on mortar specimens in water.
- v. SNPs, however during mixing of the mortar, the constituent tend to float, this is as theresult of the specific gravity of constituents' material.
- vi. The setting times results reveals that SNPs inclusion in SHA_CCW reduce the setting time by about half. The specimen with SHA_CCW (control) has final setting times value twice the specimen containing 5%.
- vii. The compressive strength of the binders was observed to have followed a similar trend as the inference drawn from the degree of hydration results. (Figures 4.8 and 4.9). 2.5 % and 3.0 % SNPs content in SHA_CCW binder-based mortar gave 28-day compressive strength values of 7.02 N/mm² and 7.45 N/mm² representing 129% and 137% of the control specimen without SNPs. The 90day compressive strength values of the same sample (2.5 % & 3.0 % SNPs content) are 8.99 N/mm² and 9.01 N/mm² 133 % and 134 % of specimen without SNPs. The high strength can be adduced to the high rate of hydration on the binders on basis established from the result of the setting time test.
- viii. The result revealed that SNPs (3 %) and SHA_CCW binder combinations has the best of the binder combination with 42.9% levels of degree of

- hydration by the 28-day curing age. This amount to RH_{28} value 1.0 with reference to the 28 days value of 3 % SNPs while 2.5 % and 3.5 % SNPs recorded 0.93 RH_{28} value each respectively.
- ix. The absorption test result shows average value of 14.22% and 14.42% for SHA_CCW (70/30) at 28 days and 56 days while 3 % SNPs inclusion gave 8.80 % and 9.98 % at 28 days and 56 days respectively.
- x. The abrasion test results revealed average abrasion resistance values of 93.49 % and 94.45 % for SNPs (2.5 % and 3 %) inclusion at 90days, while the specimen without SNPs has abrasion resistance of 92.12 % at 90 days. Implying SNPs inclusion in the SHA_CCW binder resulted in better performance and resistance has better performance and durable against wear effect and abrasive attack than the SHA_CCW binder without SNP
- xi. The addition of the SNPs decreased the density of fresh mortar mixtures and increased the fresh air content. This disclosed that the dispersing agent in the colloidal SNPs provided air entrainment in the cementitious system, while dry density of cube samples varies from 1799 kg/m³ to 2185 kg/m³ and increases with increase in SNPs but decreases with curing ages.

CHAPTER FIVE

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

The potential for the use of SNPs to enhance the fresh and hardened characteristics of SHA-CCW based mortar mixtures, cured at room temperature was studied in this report. Based on the weight of the binder constituents, the SNPs were applied to the cementitious binder matrix at dosages of 0.5 percent to 5% by 0.5 percent phase intervals. The W/B and HRWRA is set at 0.5 and 1.5 % respectively which gives the mortar mixtures the optimal flowability of 110 ± 5 percent. Furthermore, in order to satisfy the research goals, the hardened and fresh properties of the nano-modified masonry mortar mixtures were tested. Flowability, air content, and degree of hydration, as well as setting time, absorption, abrasion, and compressive strength, were all determined for both fresh and hardened mortar mixtures.

The current study revealed that the incorporation of SNPs resulted in the reduction of

setting time with optimum degree of hydration at 42.9% and increased compressive strength characteristics up to 137% of the control specimens. SNP is therefore considered to be very suitable for the production of SHA-CCW based mortar and concrete.

5.2 Research Contribution to Knowledge

The research topic, effect of silica nano-particles on the performance of sorghum husk ash and calcium carbide waste binder-based mortar; has added to the assemblage of information in the following areas;

- Chemical process for preparation and characterization of SNPs from SHA.
- ii. Build an acceptable combination of SNP-SHA-CCW binder.
- iii. Workable W/B and HRWRA to establish the water requirement for the SHA_CCW and SNPs binder combination for mortar.

5.3 Recommendations

The Following are hereby recommended based on the findings of this study.

- SNPs (3.0%) and SHA CCW (70/30) in a 1:3 binder/sand mortar at 0.5 W/B with 1.5 % Conplast SP430 as a water-reducing admixture should be used in masonry work since it satisfies ASTM C270 Class N.
- Conplast SP430 is recommended as a water-reducing admixture in SHA CCW and SNPs binder-based mortars.

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APPENDIX A

A1: Sieve Analysis (Fine Aggregate)

| | | | :) |
|-----------|---------------------------------------|--|--|
| Weight of | % retained | Cum. % | Cum. % passing |
| son ict. | | retained | passing |
| 0 | 0.0 | 0.0 | 100.0 |
| 80 | 16.0 | 16.0 | 84.0 |
| 185 | 37.0 | 53.0 | 47.0 |
| 173 | 34.6 | 87.6 | 12.4 |
| 21 | 4.2 | 91.8 | 8.2 |
| 35 | 7.0 | 98.8 | 1.2 |
| 0.5 | 0.1 | 98.9 | 1.1 |
| 5.5 | 1.1 | 100.0 | 0.0 |
| 500 | | | 252.46 |
| | soil ret. 0 80 185 173 21 35 0.5 5.5 | soil ret. 0 0.0 80 16.0 185 37.0 173 34.6 21 4.2 35 7.0 0.5 0.1 5.5 1.1 | soil ret. retained 0 0.0 0.0 80 16.0 16.0 185 37.0 53.0 173 34.6 87.6 21 4.2 91.8 35 7.0 98.8 0.5 0.1 98.9 5.5 1.1 100.0 |

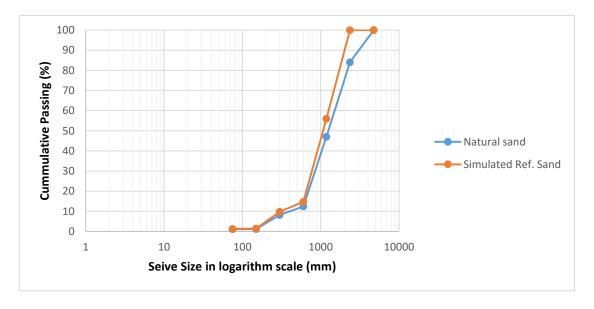


Figure A2: Graph of Sieve Analysis Fine Aggregate

Fine Aggregate

$$D60 = 0.33mm$$
, $D_{30} = 0.21mm$ and $D_{10} = 0.16mm$

Coefficient of uniformity (Cu) =
$$^{D60}/_{D10} = 2.06$$

Coefficient of curvature (Cc) =
$$\frac{(D30)2}{D60(D10)} = 0.84$$

Fineness Modulus (FM) = 2.52

APPENDIX B

B1: Setting Time of Binder Combinations

| SNPs | Setting Time (minute) | | |
|------|-----------------------|---------|-------|
| | Depth of penetration | Initial | Final |
| 0 | 5.0 | 580 | 690 |
| 0.5% | 7.0 | 571 | 668 |
| 1.0% | 5.0 | 566 | 645 |
| 1.5% | 5.0 | 532 | 612 |
| 2.0% | 7.0 | 502 | 598 |
| 2.5% | 7.0 | 494 | 567 |
| 3.0% | 5.0 | 450 | 543 |
| 3.5% | 7.0 | 412 | 521 |
| 4.0% | 6.0 | 378 | 495 |
| 4.5% | 5.0 | 330 | 468 |
| 5.0% | 5.0 | 255 | 309 |

Table B2: Average compressive strength (CS-N/mm²) of mortar samples

| SNPs | Average Abrasion Resistance (%) of mortar samples | | | | | |
|------|---|--------|---------|---------|---------|---------|
| | 3 days | 7 days | 14 days | 28 days | 56 days | 90 days |
| 0% | 2.15 | 3.05 | 4.01 | 5.44 | 6.01 | 6.74 |
| 0.5% | 2.35 | 3.75 | 4.25 | 5.97 | 6.91 | 7.80 |
| 1.0% | 2.41 | 3.91 | 4.53 | 6.23 | 7.03 | 7.98 |
| 1.5% | 2.45 | 4.02 | 4.97 | 6.74 | 7.10 | 8.32 |
| 2.0% | 2.87 | 4.31 | 5.01 | 6.88 | 7.23 | 8.65 |
| 2.5% | 3.53 | 4.67 | 5.24 | 7.02 | 7.95 | 8.99 |
| 3.0% | 3.89 | 5.05 | 5.89 | 7.45 | 8.01 | 9.01 |
| 3.5% | 3.42 | 4.87 | 5.51 | 6.02 | 6.71 | 7.83 |
| 4.0% | 2.89 | 4.68 | 5.32 | 5.97 | 6.57 | 7.71 |
| 4.5% | 2.57 | 4.54 | 5.02 | 5.87 | 6.41 | 7.51 |
| 5.0% | 2.34 | 4.45 | 4.89 | 5.55 | 6.12 | 7.43 |

B3: Average density of SHA_CCW mortar with SNPs

| SNPs | Average density (DS-Kg/m ³) of mortar samples | | | | |
|------|---|---------|---------|---------|---------|
| | 3 days | 14 days | 28 days | 56 days | 90 days |
| 0 | 1812 | 1799 | 1820 | 1811 | 1801 |
| 1 | 1814 | 1934 | 1864 | 1856 | 1811 |
| 2 | 1824 | 2004 | 1887 | 1877 | 1867 |
| 3 | 1921 | 2007 | 1905 | 1890 | 1887 |
| 4 | 2011 | 2145 | 1919 | 1906 | 1900 |
| 5 | 2185 | 2165 | 1990 | 1909 | 1908 |

APPENDIX C

Table C1: Average Abrasion Resistance (%) of mortar samples with HRWR

| SNPs | Average Abrasi | Average Abrasion Resistance (%) of mortar samples | | |
|------|----------------|---|---------|--|
| | 28 days | 56 days | 90 days | |
| 0% | 87.88 | 89.70 | 92.12 | |
| 0.5% | 89.17 | 91.74 | 92.32 | |
| 1.0% | 89.86 | 91.98 | 92.78 | |
| 1.5% | 90.01 | 92.04 | 92.89 | |
| 2.0% | 90.21 | 92.34 | 93.13 | |
| 2.5% | 90.67 | 92.89 | 93.59 | |
| 3.0% | 91.10 | 93.21 | 94.45 | |
| 3.5% | 91.00 | 93.01 | 94.04 | |
| 4.0% | 91.01 | 92.96 | 93.97 | |
| 4.5% | 89.31 | 92.03 | 93.67 | |
| 5.0% | 89.21 | 91.89 | 93.12 | |

Table C2: Average water absorption (%) of mortar samples

| Specimens | Average water absorption (%) of mortar samples | | |
|-----------|--|---------|--|
| | 28 days | 56 days | |
| 0% | 14.22 | 14.43 | |
| 0.5% | 13.25 | 13.45 | |
| 1.0% | 12.34 | 12.74 | |
| 1.5% | 12.11 | 12.41 | |
| 2.0% | 11.23 | 11.31 | |
| 2.5% | 10.53 | 10.67 | |
| 3.0% | 8.80 | 9.98 | |
| 3.5% | 8.60 | 9.71 | |
| 4.0% | 8.20 | 9.02 | |
| 4.5% | 8.00 | 8.89 | |
| 5.0% | 7.80 | 8.00 | |

APPENDIX D

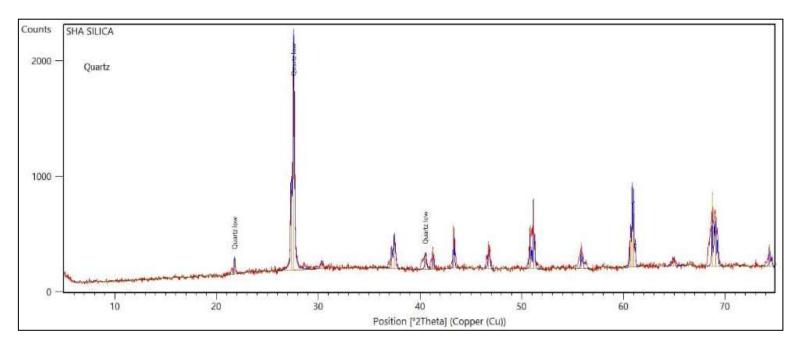


Figure D1: XRD of extracted amorphous silica

APPENDIX E



Plate I: Furnace



Plate II: Crushing machine



Plate III: Flow test machine



Plate IV: Cube specimens