

Experimental Investigation of the Strength Properties of Corn Cob Ash Blended with Metakaolin Geopolymer Concrete

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Abstract

The concrete industry has a considerable impact on the environment especially in cement production. The negative environmental impact and high cost of cement are reasons for research efforts into discovering alternatives to concrete that are environmentally friendly such as geopolymer concrete. Also, in an attempt to reduce the accumulation of waste in the environment corn cob ash was introduced in the metakaolin-based geopolymer. This research investigates the strength properties of corn cob ash blended metakaolin geopolymer concrete. Cube mold of size 100 mm x 100 mm x 100 mm and cylinder mold of size 200mm x 100mm were used to cast concrete samples containing 0, 10, 20, 30, and 40 % CCA. GPC was produced and cured at a temperature of 60°C in the oven for 24 hours, after which they were cooled at room temperature in the laboratory and their properties were evaluated after 7, 14, 28, and 56 days of curing periods. The average compressive strength obtained at 28 days with 0, 10, 20, 30, and 40 % CCA is 28.4N/mm², 28.6N/mm², 28.4N/mm², 27.9N/mm², 27.40N/mm² respectively. Concrete produced with 10%CCA has high compressive strength as compared to the control concrete. Concrete produced with 20%CCA has high splitting strength as compared to the control concrete. This study concluded that hardened GPC specimens with various replacements of CCA show comparable strength to that of the control specimens. This research recommends that metakaolin-based geo-polymer concrete containing CCA of 40 % CCA will lead to strong and durable concrete and could be used for structural concrete.

Keywords: Compressive Strength, Corn Cob Ash, Geopolymer Concrete, Metakaolin, Split Strength.

Introduction

The construction industry is one of the major consumers of materials in large quantities and at the same time produces large amounts of waste [1, 2]. The use of naturally occurring materials and waste materials as partial cement replacement material in concrete production which leads to low cement consumption enables economic and environmental benefits to the construction industry [3]. A considerable amount of work has been reported in the literature on how to use agricultural waste products as supplementary cementitious materials [4]. Because of their cementitious or pozzolanic properties, these materials can serve as partial cement replacements. Ideally, the development of such materials serves three separate purposes simultaneously. Cement as an important constituent of concrete is becoming gradually expensive compared to other ingredients of concrete. The mining of its raw materials leads to the depletion of natural resources and the degradation of the

environment [5]. Its production pollutes the environment due to the emission of CO₂. The emission of CO₂ is such that for every ton of cement produced almost a ton of CO₂ is emitted [6].

As the demands for concrete continue to increase due to rapid infrastructural development, so does the demand for Ordinary Portland Cement. Liew, et al. [7], explain the growing concerns on environmental impact caused by the extraction of raw materials for cement and CO₂ emission during cement manufacturing. Given this, the concept of geopolymer concrete which is environmentally friendly was developed to be used as an alternative to OPC in concrete production. Geopolymers are inorganic aluminosilicate materials synthesized at room temperature or a temperature slightly above room temperature through alkali activation of reactive aluminosilicate [8]. These silicon and aluminum are dissolved in an alkaline activating solution and subsequently polymerize into a molecular chain that works as calcium silicon hydrate to bind the aggregate materials [9, 10]. The reaction of the materials having aluminosilicate with others having alkalis produces a synthetic alkali silicate material called geopolymer [11]. The geo-polymerization process involves the dissolution of the geopolymer reactants in a strong alkali solution yielding polymeric Si – O – Al – O bonds in amorphous form [12]. Geopolymer was first introduced by Davidovits in 1978 to describe a family of mineral binders that could cure in alkaline solution as an alternative to conventional Portland cement concrete. Tavor, et al. [13], defined geopolymer as an amorphous polymer formed through the ionic bonding reaction between an aluminosilicate (Al-Si) material and a strong alkaline solution. It results in polycondensation of silica and alumina from source material rich in silica (Si) and alumina (Al) like fly ash, metakaolin, silica, fume, and other pozzolana to attain structural strength instead of forming calcium-silicate-hydrates (CSH) as in the case of OPC [14]. This pozzolanic binder shows a promising application for use in concrete production [15], production. According to Priya and Partheeban [16], pozzolanic application in concrete production could reduce CO₂ emissions caused by the cement industry by 80%.

Therefore, it is imperative to obtain green concrete as an innovative solution to the aforementioned challenge. One such solution is geopolymer concrete manufacture which is also known as zero-cement concrete [17]. Geopolymer concrete has increasingly attracted attention due to its increasingly environmentally beneficial applications as the process of alkali activation will utilize industrial refuse [18]. Geopolymers can be produced by the reaction between aluminosilicate materials such as metakaolin, fly ash, granulated blast furnaces, and alkali activators [9]. Research has utilized different materials in the production of geopolymer concrete ranging from Fly ash [19, 20, 21], Metakaolin [20] Slag [22]; but no research has been carried out producing concrete using blended corn cob ash and metakaolin geopolymer.

Corncob Ash (CCA) is one of the pozzolanic materials, alongside others such as Pulverized Fuel Ash (PFA), Fly Ash (FA), Metakaolin (MTK), Ground Granulated Blast Furnace Slag (GGBS) and Rice Husk Ash (RHA). Corncob was defined by Adesanya & Raheem, [23] as the

agricultural waste that is obtained after the harvest of corn. Corn cobs and CCA are thrown away in a landfill, causing an environmental nuisance; and this could be mitigated by using it in concrete as a pozzolanic material. Corn or Maize, is a common name for the cereal grass widely grown for food and livestock fodder. Adesanya and Raheem [24] extensively studied the pozzolanic properties of CCA. The sum of SiO_2 , Al_2O_3 , and Fe_2O_3 in the chemical composition of CCA satisfies the requirements of ASTM C618, suggesting its pozzolanic nature. The strength properties of concrete and the insulation properties are improved by blending the CCA in concrete [24]. Metakaolin is obtained by heat-treating of kaolin, a natural, finely divided, alumina siliceous mineral and it is abundant in Nigeria. Metakaolin is also a valuable admixture with many excellent advantages including porosity, high specific area, good absorbability, and strong coordinated bonds. Metakaolin is produced by heating kaolin at a temperature of $650 - 900^\circ\text{C}$ [25]. During the heating process, the structure of kaolinite mineral transforms from crystalline to amorphous which makes it a highly reactive pozzolan. This makes metakaolin suitable for the production of metakaolin-based geopolymer concrete. According to Getso [26], Kaolin can be obtained in abundance from the following states in Nigeria: Katsina, Plateau, Ogun, Imo, Rivers, Bauchi, Anambra, Kebbi, Ekiti, Kogi, and Akwa Ibom. Metakaolin is produced under controlled temperature to refine its color and remove inert impurities so that a much higher degree of purity and pozzolanic reactivity can be obtained [27]. Therefore, this paper tends to experimentally investigate the strength properties of corn cob ash blended metakaolin geopolymer concrete.

Literature Review

Geo-Polymer Concrete

The name geopolymer was formed by French Professor Joseph Davidovits in 1978 to represent a broad range of materials characterized by networks of inorganic molecules [28]. The geopolymers depend on thermally activated natural materials like Meta kaolinite or industrial byproducts like fly ash or slag to provide a source of silicon (Si) and aluminum (Al). These Silicon and Aluminum are dissolved in an alkaline activating solution and subsequently polymerize into molecular chains and become the binder. Polymers are either organic materials, i.e. carbon-based, or inorganic polymers, for example, silicon-based. Organic polymers comprise the classes of natural polymers (rubber, cellulose), synthetic organic polymers (textile fibers, plastics, films, elastomers, etc.), and natural biopolymers (biology, medicine, pharmacy). Raw materials used in the synthesis of silicon-based polymers are mainly rock-forming minerals of geological origin, hence the name: Geopolymer. Joseph Davidovits after coining the term in 1978 created the non-profit French scientific institution (Association Loi 1901) Institut Géopolymère (Geopolymer Institute). Tavor, et al. [13], defined geopolymer as an amorphous polymer formed through the ionic bonding reaction between an aluminosilicate (Al-Si) material and a strong alkaline solution. It results in polycondensation of silica and alumina from a source material rich in silica (Si)

and alumina (Al) like fly ash, metakaolin, silica fume, and other pozzolana to attain structural strength instead of forming calcium-silicate-hydrates (CSH) as in the case of OPC [14].

To Chemist, Geopolymers consist of a polymeric Si–O–Al framework, similar to zeolites. The main difference to zeolite is geopolymers are amorphous instead of crystalline. The microstructure of geopolymers on a nanometer scale observed by TEM comprises small aluminosilicate clusters with pores dispersed within a highly porous network. The cluster sizes are between 5 and 10 nanometers. For Geopolymer Material, the reaction produces SiO_4 and AlO_4 , tetrahedral frameworks linked by shared oxygens as poly(sialates) or poly(sialate–siloxo) or poly (sialate–disiloxo) depending on the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio in the system. The connection of the tetrahedral frameworks occurred via long-range covalent bonds. Thus, the geopolymer structure is perceived as a dense amorphous phase consisting of a semi-crystalline 3D alumino-silicate microstructure. Geo-polymerization occurs at ambient or slightly elevated temperatures, where the leaching of solid aluminosilicate raw materials in alkaline solutions leads to the transfer of leached species from the solid surfaces into a growing gel phase, followed by nucleation and condensation of the gel phase to form a solid binder. Geopolymer concrete is an innovative and eco-friendly construction material and an alternative to Portland cement concrete. The use of geopolymer reduces the demand for Portland cement which is responsible for high CO_2 emission.

Classification of Geopolymers

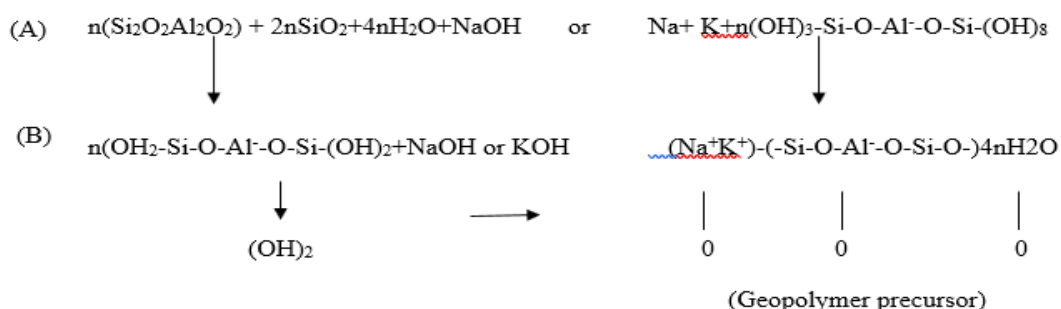
According to T.F. Yen et al. [29], geopolymers can be classified into two major groups:

1. pure inorganic geopolymers and
2. organic containing geopolymers,

Synthetic analogs of naturally occurring macromolecules. A geopolymer is essentially a mineral chemical compound or mixture of compounds consisting of repeating units, for example silico-oxide (–Si–O–Si–O–), silico-aluminate (–Si–O–Al–O–), ferro-silico-aluminate (–Fe–O–Si–O–Al–O–) or alumino-phosphate (–Al–O–P–O–), created through a process of geopolymerization. This mineral synthesis (geo-synthesis) was first presented at an IUPAC symposium in 1976. The microstructure of geopolymers is essentially temperature dependent: It is X-ray amorphous at room temperature but evolves into a crystalline matrix at temperatures above 500 °C. One can distinguish between two synthesis routes: In an alkaline medium (Na^+ , K^+ , Li^+ , Ca^{2+} , Cs^+ , and the like); In an acidic medium with phosphoric acid, organic carboxylic acids from plant extracts (acetic, citric, oxalic, and humic acids). The alkaline route is the most important in terms of research and development and commercial applications and is described below. In the 1950s, Viktor Glukovsky, of Kyiv, USSR, developed concrete materials originally known under the names "soil silicate concretes" and "soil cement", but since the introduction of the geopolymer concept by Joseph Davidovits, the terminology, and definitions of 'geopolymer' have become more diverse

Sahel and Vijaya [30], stated that “the polymerization process involves a substantially fast chemical reaction under alkaline conditions on silicon-aluminum minerals that results in a three-dimensional polymeric chain and ring structure”. The ultimate structure of the geopolymer depends largely on the ratio of Si to Al (Si: Al), with the materials most often considered for use in transportation infrastructure typically having a Si: Al between 2 and 3.5. The reaction of Fly Ash with an aqueous solution containing Sodium Hydroxide and Sodium Silicate in their mass ratio results in a material with a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds.

The schematic formation of geopolymer material can be shown as described by Equations (A) and (B).



Water is not involved in the chemical reaction of Geopolymer concrete and instead water is expelled during curing and subsequent drying. This is in contrast to the hydration reactions that occur when Portland cement is mixed with water, which produces the primary hydration products calcium silicate hydrate and calcium hydroxide. This difference has a significant impact on the mechanical and chemical properties of the resulting geopolymer concrete and also renders it more resistant to heat, water ingress, alkali-aggregate reactivity, and other types of a chemical attack.

In the case of geopolymers made from fly ash, the role of calcium in these systems is very important, because its presence can result in flash setting and therefore must be carefully controlled. The source material is mixed with an activating solution that provides the alkalinity (sodium hydroxide or potassium hydroxide are often used) needed to liberate the Si and Al and possibly with an additional source of silica (sodium silicate is most commonly used).

The temperature during curing is very important, and depending upon the source materials and activating solution, heat often must be applied to facilitate polymerization, although some systems have been developed that are designed to be cured at room temperature.

Geo-Polymerization

Davidovits [31] Geo-polymerization is the process of combining many small molecules known as oligomers into a covalently bonded network. The geochemical syntheses are carried out through oligomers (dimer, trimer, tetramer, pentamer) which provide Five isolated oligomers of the Kpoly (sialate)/poly(sialate-silxo) species the actual unit structures

of the three-dimensional macromolecular edifice. In 2000, T.W. Swaddle and his team proved the existence of soluble isolated alumino-silicate molecules in solution in relatively high concentrations and high pH. One major improvement in their research was that their study was carried out at very low temperatures, as low as -9°C . Indeed, it was discovered that the polymerization at room temperature of oligo-sialates was taking place on a timescale of around 100 milliseconds, i.e., 100 to 1000 times faster than the polymerization of ortho-silicate, oligo-siloxo units. At room temperature or higher, the reaction is so fast that it cannot be detected with conventional analytical equipment. The image shows 5 soluble oligomers of the K-poly(sialate)/poly(sialate-siloxo) species, which are the actual starting units of potassium-based alumino-silicate geo-polymerization.

Example of (-Si-O-Al-O-) geo-polymerization with metakaolin MK- 750 in an alkaline medium

It involves four main phases comprising seven chemical reaction steps: Alkaline depolymerization of the poly (siloxo) layer of kaolinite; Formation of monomeric and oligomeric species, including the "ortho-sialate" $(\text{OH})_3\text{-Si-O-Al-(OH)}_3$ molecule; In the presence of water glass (soluble Kpolysiloxonate), one gets the creation of ortho-sialate-disiloxo cyclic structure (e.g. #5 in the figure), whereby the hydroxide is liberated by condensation reactions and can react again; Geo-polymerization (polycondensation) into higher oligomers and polymeric 3D networks. The geo-polymerization kinetics for Napoly (sialate-siloxo) and K-poly(sialates-iloxo) are slightly different respectively. This is probably due to the different dimensions of the Na^+ and K^+ cations, K^+ being bigger than Na^+ .

Example of zeolitic (Si-O-Al-O-) geo-polymerization with fly ash in an alkaline medium

It involves 5 main phases Nucleation stage in which the aluminosilicates from the fly ash particle dissolve in the alkaline medium (Na^+), releasing aluminates and silicates, probably as monomers. These monomers inter-react to form dimers, which in turn react with other monomers to form trimers, tetramers, and so on. When the solution reaches saturation, an aluminum-rich gel (denominated Gel 1) precipitate. As the reaction progresses, more Si-O groups from the initial solid source dissolve, increasing the silicon concentration in the medium and gradually raising the proportion of silicon in the zeolite precursor gel (Gel2). Polycondensation into zeolite-like 3D frameworks.

Metakaolin MK-750-based geopolymer binder

chemical formula $(\text{Na-K}) - (\text{Si-O-Al-O-Si-O-})$, ratio Si: Al=2 (range 1.5 to 2.5)

Silica-based geopolymer binder

Geopolymer resins and Binders chemical formula $(\text{Na-K}) - n(\text{Si-O-}) - (\text{Si-OAl-})$, ratio Si: Al>20 (range 15 to 40).

Sol-gel-based geopolymer binder (synthetic MK-750) chemical formula $(\text{Na-K}) - (\text{Si-O-Al-O-Si-O-})$, ratio Si: Al=2 The first geopolymer resin was described in a French patent application filed by J. Davidovits in 1979. The American patent, US 4,349,386, was granted on Sept. 14, 1982, with the title *Mineral Polymers and methods of making them*. It essentially involved the geo-polymerization of alkaline soluble silicate [waterglass or (Na-K) -polysiloxonate] with calcined kaolinitic clay (later coined metakaolin MK-750 to highlight

the importance of the temperature of calcination, namely 750 °C in this case). In 1985, Kenneth Mac-Kenzie and his team from New Zealand, discovered the Al(V) coordination of calcined kaolinite (MK-750), describing a "chemical shift intermediate between tetrahedral and octahedral. "This had a great input towards a better understanding of its geo-polymeric reactivity. Since 1979, a variety of resins, binders, and grouts have been developed by the chemical industry, worldwide.

Materials and Methods

Materials

The binders used in this study were Corn Cob Ash (CCA) and Metakaolin (MTK). Corn cob was obtained from a mill in Gombe state in Gombe metropolis, and corn cob ash was produced from it. Metakaolin was obtained from Kaltungo in Gombe State and metakaolin is obtained from it at a temperature of 900°C. The aggregates used for the experiment were fine and coarse aggregates that conformed with the provisions of [32]. The fine aggregate used is sharp river sand with a maximum size of 4.75mm while the coarse aggregate used is crushed gravel with a maximum size of 20mm.

Mix Proportion

A series of trial tests were conducted to establish the optimum proportion of materials to be used as there is no specific or standard method of mix design for geopolymer concrete production. After the trial test, the mix proportions presented in Table 1 were found to be more suitable and were used for the experiment.

Table 1: Mix proportion for concrete

Sodium Silicate	Sodium Hydroxide	Extra Water	Metakaolin Aggregate	Fine Aggregate	Coarse Aggregate
212.57 kg/m ³	85.03 kg/m ³	86.4 kg/m ³	480 kg/m ³	599.86 kg/m ³	964.61 kg/m ³

Source: Laboratory Work

Preparation of the Samples

Blended CCA/MTK and aggregates were adequately mixed, after which an alkaline solution was added and mixed thoroughly. The fresh metakaolin geopolymer concrete (GPC) mix was cast into a steel mold of 100 x 100 x 100 mm in two layers and each layer was and was compacted. After 24 hours of casting, the concrete cubes were de-molded and wrapped with polythene. The specimens were then placed in an electric oven at a temperature of 60°C for 24 hours to speed up the polymerization reaction process. Thereafter, the concrete cubes were removed from the electric oven and the polythene was removed and the concrete cube specimens were stored at room temperature for different curing days of 7, 14, 28, and 56 days. After the curing days concrete cube samples were then subjected to

compressive strength and split tensile strength tests and carried out by the provisions of [33] and the equation adopted from [34] respectively.

Results and Discussions

Density of Geo-Polymer Concrete Cubes

Figure 1 presents the average density of geo-polymer concrete samples weighed at 7, 14, 28, and 56 curing days. The density of concrete cube samples varies from 2385 kg/m³ to 2435 kg/m³ and it increases with an increase in curing periods. Concrete samples with a density higher than 2600kg/m³ are called higher-density concrete samples [35]. Those greater than 2400kg/m³ but less than 2600kg/m³ are called normal-density concrete, and those less than 2400kg/m³ are low-density concrete. Therefore, geo-polymer concrete is concrete with low and normal density, though samples with 10% and 20% pozzolanic material addition were denser than the control samples at later stages.

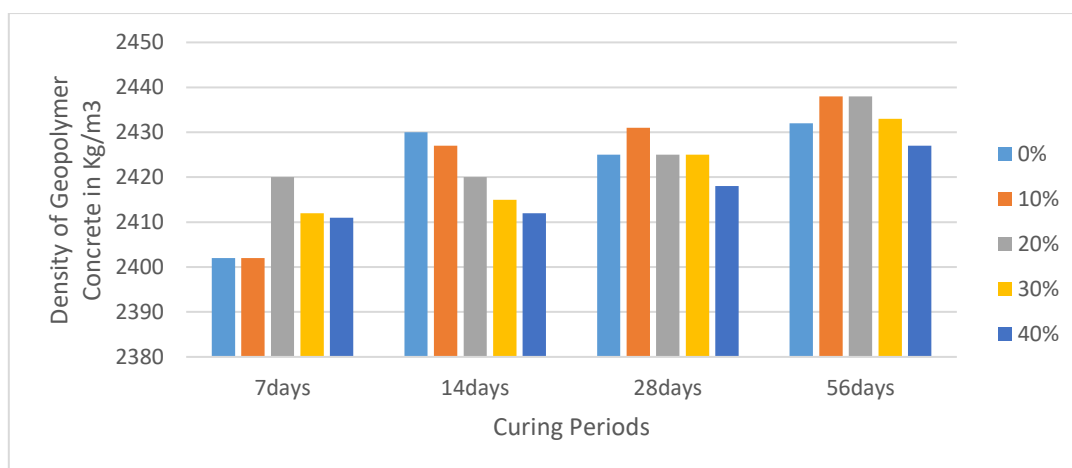


Figure 1: Density of Geo-polymer concrete

Density of Geo-Polymer Concrete Cylinder

Figure 2 presents the average density of geo-polymer concrete samples weighed at 28 and 56 curing days. The density of concrete cube samples varies from 2220 kg/m³ to 2340 kg/m³ and it increases with an increase in curing periods. The geo-polymer concrete is a concrete with low and normal density.

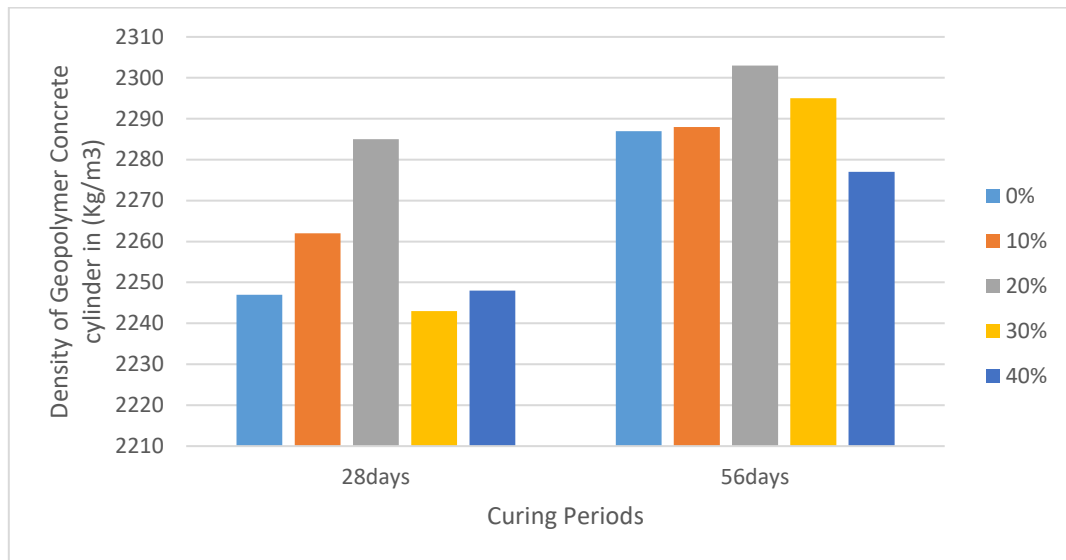


Figure 2: Density of Geo-Polymer Concrete Cylinder

Compressive Strength of Geo-Polymer Concrete

Figure 3 presents the results for the average compressive strength of metakaolin-based geo-polymer specimens containing 0%, 10%, 20%, 30%, and 40% corn cob ash and metakaolin cured at 7, 14, 28, and 56 days. From the figure, there was a general increase in compressive strength from 7 to 56 days curing period for the geo-polymer concrete specimens. The result achieved 28.6 N/mm², 28.4 N/mm², 27.9 N/mm², and 27.0 N/mm² for 10%, 20%, 30%, and 40% CCA replacement respectively while 0% replacement achieved 28.4 N/mm². This represents 0.70% decrease of control concrete as compared to 10% CCA in compressive strength at 28 days. While 10% CCA concrete shows the same strength index as compared with the control concrete. At 56 days of curing age, it was noticed that 20% CCA shows a 1.38% increase in compressive strength as compared to 0% CCA concrete. Also, it was noticed that control concrete specimens show the same strength index with 10% CCA concrete. The 20% CCA GPC specimen attained a higher compressive strength than all other concrete specimens; this could be a result of the type and amount of binder used in the production of the GPC specimen. This shows that 20% CCA is the optimum replacement in metakaolin-based geopolymer concrete. Bachhav and Dubey [36], affirm that the compressive strength of the GPC specimen increases with an increase in the molarities of the NaOH solution. The strength gained at an early age has also improved significantly up to 28 days and beyond. Nath and Sarker [37], reported that blending pozzolana in producing geopolymer concrete affects the setting time, workability, and initial strength behavior of geopolymer concrete. Nath and Sarkar [37], found that blending a small amount of OPC with pozzolana can cause quick setting time and a slight decrease in workability. Perera et al. [38], reported that curing metakaolin-based geopolymers at ambient temperature yields positive strength which is almost the same as that of oven curing but the strength of heat-cured specimens developed rapidly within a day. Yewale et

al. [39], stated that the mechanical strength result of geopolymer concrete cured at room temperature is promising compared to water curing.

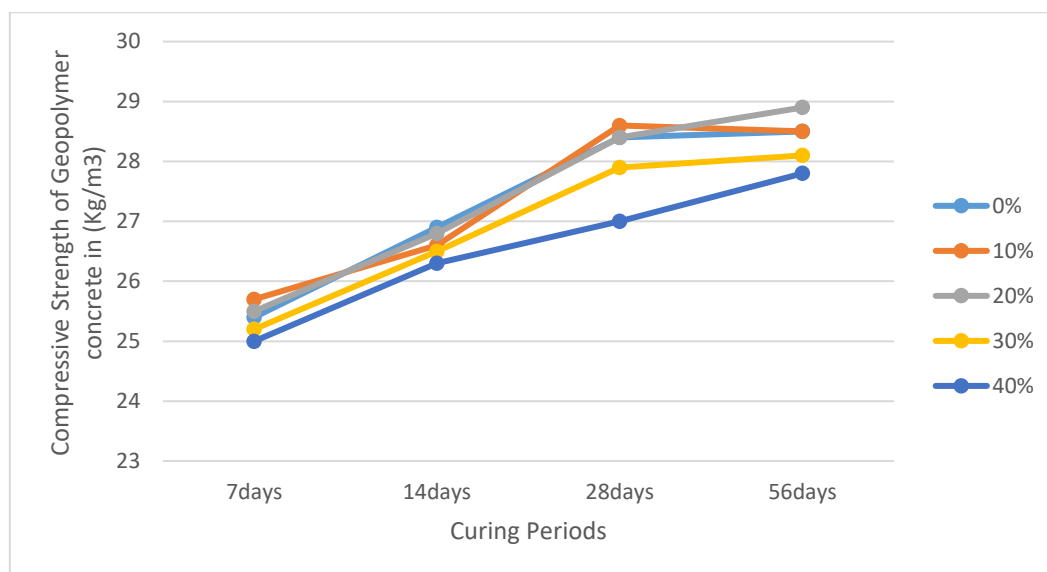


Figure 3: Compressive Strength of Geo-Polymer Concrete

Split Tensile of Geo-Polymer Concrete

Figure 4 presents the average split tensile strength of geo-polymer concrete specimens tested at 28 and 56 days. 0% control concrete achieved 2.59 N/mm², while 10%CCA, 20%CCA, 30%CCA, 40%CCA, achieved 2.60 N/mm², 2.61 N/mm², 2.54 N/mm² and 2.52 N/mm² respectively. This represents a 0.38%, 0.77%, increase of 10%CCA and 20%CCA concrete as compared to control concrete, while 0% control concrete has better strength than other replacement levels. At 56 days GPC specimen containing 0% achieved 2.63 N/mm², while 10%CCA, 20%CCA, 30%CCA and 40%CCA, achieved 2.62 N/mm², 2.65 N/mm², 2.56 N/mm², and 2.54 N/mm² respectively. This represents an increase of 0.75% of 20 %CCA, as compared to the control concrete. This result shows that at 56 days' replacement up to 20% corn cob ash in concrete gave better strength. This perhaps could be a result of the type of binder used in the production of the concrete specimen. Preethy et al., [40], discovered that the split tensile strength increases with increasing the molarity of sodium hydroxide as in the case of compressive strength. A higher concentration of NaOH solution gives higher split tensile strength in geo-polymer concrete specimens because it makes good bonding between the aggregates and paste of the concrete

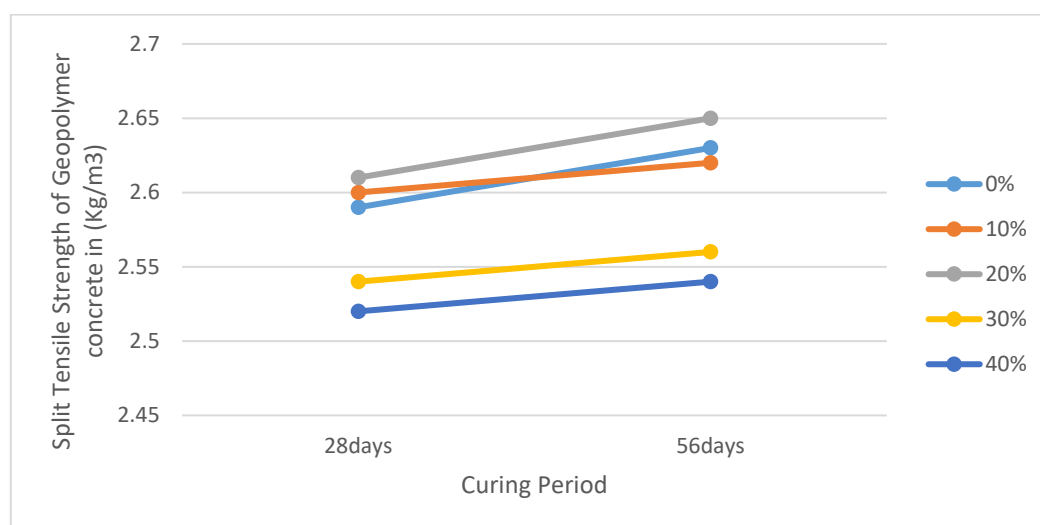


Figure 4: Split Tensile Strength of Geo-polymer Concrete

Conclusion

The density of foam concrete increases with an increase in curing age. The strength properties of the hardened concrete specimen showed that 0% control concrete has higher strength, and exhibits the same strength index with 10%CCA at 28 days, while 10%CCA and 20%CCA show higher strength above the 0% control concrete at 56 days. In addition, 0% control concrete has higher strength than other addition levels of pozzolana and exhibits the same strength index with 10%CCA, also 20%CCA/MTK shows higher strength than the control concrete at 28 days, while 10%CCA and 20%CCA show higher strength above the 0% control concrete at 56days ordinary Portland cement concrete. The research recommends the use of corn cob ash and metakaolin to replace cement in cement concrete production and the production of geo-polymer concrete due to its accessibility and availability. The use of 20% replacement of cement with corn cob ash and metakaolin is the optimum replacement level that can be used to produce durable concrete for construction purposes in both normal and chemical environments. Also, 20%CCA is the optimum addition for geo-polymer concrete, even though the addition of up to 30% shows promising results. It is also recommended that the GPC produced with RCA in this research, can be used as a standard material on Nigerian construction sites; and included in the Nigerian specification of construction materials when developed.

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