



PERFORMANCE OF CONCRETE USING METAKAOLIN AS CEMENT PARTIAL REPLACEMENT MATERIAL

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ABSTRACT

Concrete, due to its versatility and ready availability, continues to be of great importance around the world, and with the increasing need for shelter, the force pulled by construction activity brought about the high demand for construction materials and a devastating effect on the environment due to the activities from the raw materials needed in the production of cement and concrete. This research investigates the effect of metakaolin as a partial replacement of cement for concrete production. Cement was partially replaced by metakaolin at 5%, 10%, 15%, 20%, 25% and 30%. A constant water/binder ratio of 0.50 was used. The parameters investigated include the chemical composition of metakaolin, setting time, workability, density, compressive strength, split-tensile strength, and flexural strength. The results showed that metakaolin is a good pozzolan with combined SiO_2 , Al_2O_3 and Fe_2O_3 to equal 92.89%. The results from the test show that the inclusion of metakaolin increased the setting time of cement-metakaolin paste and resulted in the workability reduction of concrete as partial cement replacement with metakaolin increases. This research reveals that partial cement replacement with 0% and 10% MTK in concrete both gave an average compressive strength of 28.9 N/mm², at 28 days. Meanwhile, the highest value for compressive strength obtained at 90 days is 34.8 N/mm², which was at 10% replacement with MTK. The result shows that metakaolin (MTK) has a pozzolanic effect, considering the strength activity index and improved compressive strength, split tensile strength and flexural strength of concrete. The optimum cement replacement was 10% metakaolin, which performed better than the control concrete. At later stages of curing, 15% also shows promising results. When metakaolin is calcined, it is expected to undergo pozzolanic reactions with cement hydrates, forming secondary Calcium Silicate Hydrate (C-S-H), which aids strength development. It can be concluded that metakaolin is a suitable material for partial replacement of cement up to 10%. Thus, using MTK as a partial replacement for cement in concrete at a lower replacement volume will reduce cement usage, production costs, and environmental pollution from cement production.

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1.0 Introduction

Concrete, due to its versatility and ready availability, continues to be of great importance around the world, and with the increasing need for shelter, the force pulled by construction activity brought about the high demand for construction materials and a devastating effect on the environment due to the activities from the raw materials needed in the production of cement and concrete. According to Harley (2007), the construction industry uses more than twice the amount of concrete compared to all other building materials combined, such as wood, steel, plastic, and aluminium. This is because of the availability of its constituents, as well as its adaptability, flexibility, strength, durability, impermeability, Etc. It is a composite material used to construct buildings, industries and infrastructures. Concrete is composed of cement, aggregate, and water, and the

addition of admixture and additives to alter and modify its properties. Concrete is second to water as the most utilized material and substance worldwide. According to Shoubi *et al.* (2013), it was estimated that the global concrete industries produce about 12 billion tons of concrete annually and use about 1.6 billion tons of Portland cement. The high demand for concrete requires a large amount of cement to develop its strength. Thus, approximately 1 ton of concrete is produced yearly for every human globally. There is a need for a more concerted approach to producing concrete and an understanding of the need to improve its properties. The amount of CO₂ produced in concrete is directly proportional to the cement used in the concrete mix. 900 kg of CO₂ is produced to manufacture each cement ton (Gao *et al.*, 2015). OPC is currently being discussed not only due to its cost but also because of its environmental impact during manufacturing. The current trend is to reduce the amount of cement in concrete by using pozzolana and other supplementary cementitious materials to enhance concrete performance. The American Society for Testing and Materials (ASTM) defined pozzolan as a siliceous or siliceous-aluminous material. This material does not have much cementitious value on its own, but in the presence of moisture, when finely divided, it reacts with calcium hydroxide at ordinary temperatures to form compounds with cementitious properties. Shetty (2009) classified pozzolans as either natural or artificial pozzolan. Natural pozzolans include materials such as clay, shales, opaline cherts, diatomaceous earth, volcanic ash, volcanic tuffs, and pumicites; artificial pozzolans consist of silica fume, fly ash, rice husk ash, and metakaolin (Agboola *et al.*, 2020a). Metakaolin, a kaolin product, is one of the classifications of artificial pozzolans and is an area of study for this research. It is available in abundance, economical and easily accessible. The use of metakaolin is one of the potential means of generating affordable binders for concrete production. It effectively converts naturally occurring material that has not been exploited for decades to wealth. Metakaolin is one type of calcined clay from the calcination of kaolin clay. Metakaolin differs from other artificial pozzolans in that it is not a waste product resulting from an industrial byproduct, nor is it completely natural. It originated from kaolinite clay mineral and is processed for different applications, including cementitious material products. Metakaolin is mainly produced by a heat treatment called kaolin clay calcination within 600^o to 800^oC. The treatment process of calcination is essential for the production of highly reactive pozzolanic material, as the water is driven off from the kaolinite clay, and the material structure collapses, resulting in an amorphous alumino-silicate form referred to as metakaolin or metakaolinite. According to Agboola *et al.*, (2024), using pozzolan to replace OPC in concrete lowers heat development during hardening and improves the durability of the final concrete structures. It also improves the performance of concrete mix and reduces the cost of concrete production when introduced into concrete. However, the most important single property of concrete is strength because the primary aim of structural design is that the structural elements must resist or carry the load imposed on them. Strength is an essential property for consideration because it is related to several other properties that are more difficult to measure directly. A simple strength test can indicate such properties as durability. However, this study tends to assess the strength property of concrete produced with metakaolin.

2. Materials and Methods

2.1 Materials

The materials for the laboratory experiment included coarse aggregate, fine aggregate, cement, metakaolin and water. Coarse aggregate was received from a quarry site within the Bauchi metropolis. The fine aggregate was obtained from the Yelwa River flow in Bauchi state. The Ordinary Portland cement is the Dangote of Grade 42.5 brand, which was procured from vendors within the Bauchi metropolis and conforms to BS EN 196-1 (2016). Kaolin used for this research was obtained in sufficient quantity from the Alkaleri local government area of Bauchi state. The particle sizes of fine aggregate were those passing through sieves with an aperture size of 2.36 mm but retained on sieves of 150 μ m. It was confirmed to be free from dust and harmful substances and conform to BS 882 (1992). The coarse aggregates used in this study were granite with a particle size range between 5 mm and 20 mm, which conforms to 882 (1992). The kaolin was ground using a stone crusher and mortar, while the pestle was ground into fine powder. MTK was then fired (calcined) in the laboratory at a controlled temperature of 700^oC in a kiln furnace; the resulting metakaolin was allowed to cool at room temperature. After cooling, it was sieved through a 15 μ m sieve. These experiments used portable water that was clean, colourless, odourless, and free of organic matter. This investigation used a mix ratio of 1:2:4 by weight of cement, sand, and gravel and a water-cement ratio of 0.50. The cement in the mix was partially replaced with metakaolin at intervals of 5% to 30%. The concrete with 0% metakaolin served as the control.

2.2 Methods

2.2.1 Chemical Composition of Metakaolin

To determine the chemical constituents of Metakaolin (MTK), samples were taken and subjected to analysis at Sodexmines Nigeria Limited Plateau State, Nigeria, using the EDXRF method. The machine used to carry out this test was Minipal 4 Energy Dispersive X-Ray Fluorescence. Significant, minor oxides and losses on ignition (Lol) were measured and recorded.

2.2.2 Setting Times Tests

The setting times (initial and final) were carried out per *BS EN 196-3 (1995)* using the Vicat probe and the Vicat needle apparatus. The paste's cement content was partially replaced with metakaolin (MTK) at different cement replacement levels of 5 - 30% at an interval of 5%. The cement pastes without metakaolin served as the control. To achieve consistency, 300g of cement was weighed, and 25% of the weight of the cement sample, which is 75 g, was measured for water. To achieve consistency, 1% of the measured water was continuously added until consistency was achieved. Consistency was determined using Equation 1.

$$\text{Consistency} = \frac{\text{water consumed}}{\text{weight of cement cement sample}} \times 100\% \quad (1)$$

2.2.3 Strength Activity Index of Cement

The strength activity indices of cement and metakaolin were evaluated according to ASTM C311. The control mortars were prepared according to the recipe provided in ASTM C311. For the mixture with the metakaolin as partial cement replacement, a test mixture was prepared with constant water and sand volume fraction and a 20% replacement by volume of cement with metakaolin. The compressive strength test was carried out per ASTM C109. A 1:3 cement sand mix with water cement ratio of 0.5 was used to prepare 50 x 50 x 50mm prism specimens to determine the compressive strength of the cement mortar, cured at 7 and 28 days.

2.2.4 Workability Test

Slump test was carried out per *BS EN 12350: Part 2 (2000)*. The replacement was done at 5% up to 30% cement with metakaolin. The slurry sample without MTK serves as the control. The compacting factor test was conducted per (*BS 1881-102, 1983*).

2.2.5 Density Test

This was carried out prior to the crushing of the concrete specimen. At the end of each curing period, the concrete specimens were weighed using an electric weighing machine balance. Density is calculated as the mass of the concrete specimen in (kg) divided by the volume of the concrete cube (m³) and expressed in kg/m³. Density tests were conducted on 100x100x100mm cube specimens at 7, 14, 28, 56 and 90 days per *BS EN 12390-7 (2000)*.

2.2.6 Compressive Strength Test of Concrete

The compressive strength tests were conducted per *BS EN 12390-3 (2009)*. Cube specimens of 100 x 100 x 100mm were prepared. All the cubes' specimens were cured by total immersion in water and were removed from the moulds after 24 hours of casting until the testing age. They were allowed to drip off and be at a saturated surface dry condition before being tested for strength. Concrete specimens were tested at curing ages of 7, 14, 28, 56 and 90 days. The cement was replaced with metakaolin at 5% to 30% at 5% intervals.

2.2.7 Split Tensile Strength Test

In assessing the split tensile strength (Fct) of concrete samples with metakaolin (MTK) as a partial replacement of cement, the splitting tensile strength was conducted on 100 x 300mm concrete cylinder specimens per the provision of *BS EN 12390-6 (2009)*. The splitting strengths were determined at a 120 kN/min loading rate until failure on a 600 kN Avery Denison Universal Testing Machine (AVDTM). The splitting test was calculated using Equation 2.

$$F_{ct} = \frac{2F}{\pi \times L \times d} \quad (2)$$

where; F is the maximum load in (kN), L is the average measured length in (mm)

d is the average measured diameter in (mm)

The tensile splitting strength is expressed to the nearest 0.05 MPa.

2.2.8 Flexural Strength

The flexural strength of concrete specimens was determined by a point load test on beam specimens of size 100 mm x 100 mm x 500 mm, and in accordance with BS 1881:116 (1983), Three numbers of specimens for each set were tested at 7, 14, 28, 56 and 90 days. These specimens were tested in a Universal Testing Machine (UTM) of capacity 1000kN. Flexural strength was computed using equation 3.

$$\text{Flexural Strength} = F_s = \frac{PL}{bd^2} \quad (3)$$

where, b = measured width in “mm” of the specimen

d = measured depth in “mm” of the specimen

L = length in “mm” of the span on which the specimen was supported

P = maximum load in “kg” applied to the specimen

2.3 Quantities of Concrete Constituents

Table I presents the quantities of concrete constituent used.

Table I: Quantities of concrete constituents at various Replacement of Cement with MTK

Sample (%)	Cement (kg/m ³)	MTK (kg/m ³)	CA (kg/m ³)	FA (kg/m ³)	Water (kg/m ³)
Control (0%)	420	0.0	1079	661	210
5%	399	21	1079	661	210
10%	378	42	1079	661	210
15%	357	63	1079	661	210
20%	336	84	1079	661	210
25%	315	105	1079	661	210
30%	294	126	1079	661	210

3. Results and Discussions

3.1 Chemical Composition of Metakaolin

Table 2 shows the chemical composition of metakaolin determined using an XRF technique. The result obtained in this study is consistent and comparable with the ASTM C618 requirement, which shows that the (SiO₂+Al₂O₃ + Fe₂O₃) minimum requirement for a standard pozzolana is 70%. According to ASTM standards, the maximum limit for SO₃, Loss on Ignition (Lol), and Moisture content are 4%, 10% and 3%, respectively. The SO₃, Lol and moisture content of metakaolin were 0.02%, 1.62% and 0.11%, respectively, below the acceptable maximum limit. Therefore, the metakaolin sample is expected to show pozzolanic behaviour in the

cementitious systems. The consumption of calcium and hydrate (CH) in the process caused the formation of calcium silicate hydrate (CSH) and stratlingite (C_2ASH_8). Minor compounds such as TiO_2 , MgO , MnO , SO_3 , Na_2O , and CaO are also found in metakaolin samples under consideration. However, the amounts of individual components of the minor compounds were not more than 0.7%.

Table 2: Chemical Composition of Metakaolin

Constituent	MTK (%)
Silicon Oxide (SiO_2)	48.92
Aluminium Oxide (Al_2O_3)	43.46
Iron Oxide (Fe_2O_3)	0.51
Calcium Oxide (CaO)	0.62
Potassium Oxide (K_2O)	0.16
Sodium Oxide (Na_2O)	0.21
Sulphur trioxide (SO_3)	0.02
Magnesium Oxide (MgO)	0.11
Manganese Oxide (MnO)	0.03
Titanium oxide (TiO_2)	0.56
Loss of Ignition (Loi)	1.62

3.2 Setting Time Test

Figures 1 and 2 show the setting time and consistency of Portland cement and metakaolin paste. It was discovered that the higher the amount of MTK in the binary paste, the higher the time required for the PC/MTK paste to be set. The increase in setting times may be due to the high content of K_2O in the ash, which may have reacted with water to produce caustic potassium hydroxide that absorbs the water available for the hydration of cement, as reported by Kosmatka *et al.*, (2002). However, the setting times for all percentage replacement of cement with metakaolin are within specification for Portland Cement, which is a minimum of 45 minutes for the initial setting time and 375 minutes for the final setting time (ASTM C150-15, 2015). The increased setting times of metakaolin paste means that metakaolin can be used as a set retarder in concrete to be transported over a long distance. The consistency of the paste increased because of the particle interference effect, so a large amount of water is needed to properly wet a higher percentage of metakaolin in the mix to produce cement gel, as the cement is more soluble than the metakaolin. The observed setting time and consistency behaviour correspond and are consistent with the findings of Agboola *et al.* (2020b), who observed that setting time increases with the addition of pozzolana.

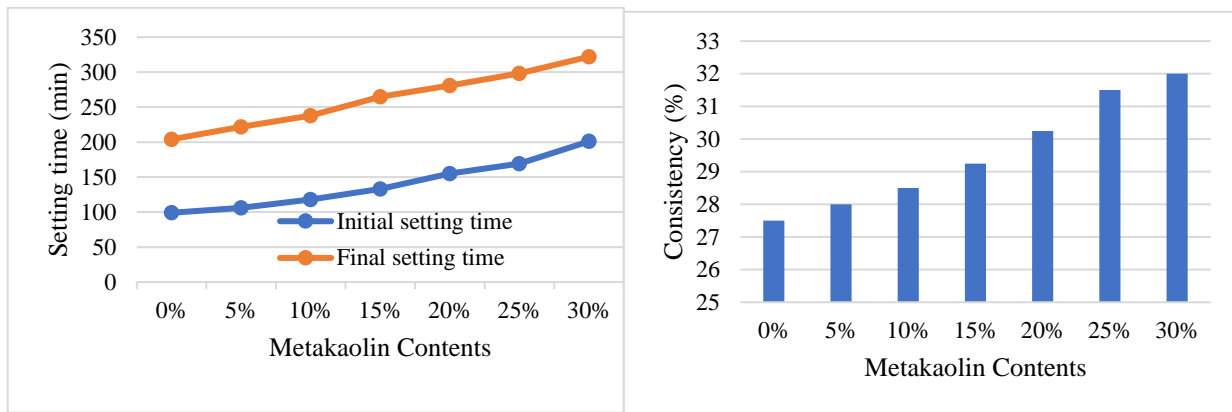


Figure 1: Setting Time of MTK-Cement Paste **Figure 2:** Consistency of MTK-Cement Paste

3.3 Strength Activity Index

The result for the strength activity index of cement mortar is presented in Table 3. The result shows an increasing strength trend at 7 and 28 days, respectively, for both control mortar and mortar produced with metakaolin as partial cement replacement. The result also shows that, at both 7- and 28-day curing age, the strength activity index of 20% MTK-cement mortar meets the minimum requirement specified by ASTM C311, that the strength activity index of pozzolana must meet 75% of the control strength at 7 and 28 days. This indicates that metakaolin is a good pozzolana and can replace cement in concrete production.

Table 3: Strength Activity of Cement-MTK

% MTK	Compressive Strength (N/mm ²)		Strength Activity Index	
	7 days	28 days		
0% Control	24.0	33.5	100	100
20% MTK	19.9	29.2	82.9	87.2

As per BS EN 196-1 (EN, 2005), a constant water-to-binder ratio (0.5) was maintained to prepare a concrete mix for the workability test. 0% metakaolin concrete has higher workability compared to other percentage replacement levels of cement, as shown in Figure 3. From the values for the seven mixes, on the degree of workability for the slump test, 0% to 20% falls within high slump (50 -100), which is a category of high degree workability, 25% and 30% percentage cement replacement with metakaolin falls with medium slump (25–50) which is a low degree workability according to Neville and Brooks (2010). The slump value decreases with the increase in the metakaolin in the concrete mix. Concrete produced with metakaolin has less slump value, which may be due to the nature of the surface texture, the chemical composition of the pozzolana and the absorption capacity of metakaolin. For the compacting factor test, the degree of workability ranges from low to medium, which falls within the range specified by BS 812 and Neville and Brooks (2010). Mixes with 0% - 25%, with compacting values of 0.94, 0.94, 0.93, 0.92, 0.92 and 0.91, respectively, shows medium workability, while 30% with 0.90 shows low workability, as presented in Figure 4.

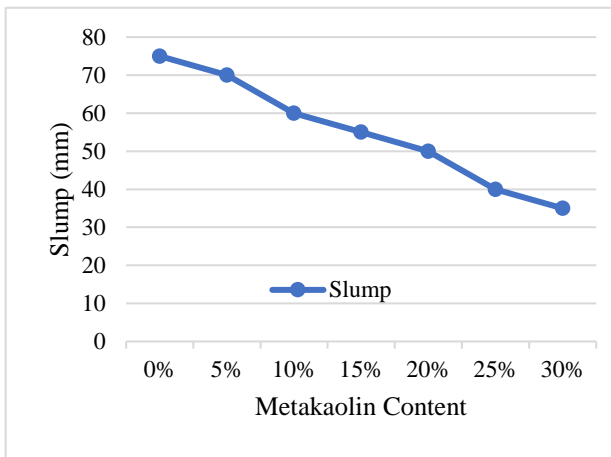


Figure 3: Slump Test of MTK Concrete

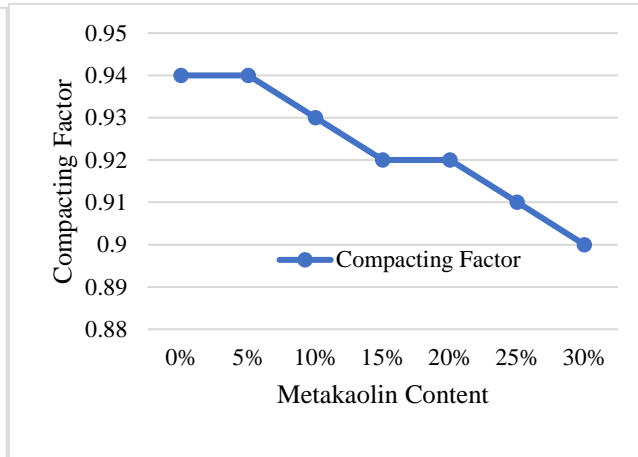


Figure 4: Compacting Factor for MTK Concrete

3.5 Density of Concrete

Figure 5 presents the density of concrete produced with metakaolin as a partial replacement of cement and replaced at 5% to 30% by weight of cement. The result shows the effect at various curing ages from 7 to 90 days. The density of metakaolin-cement concrete and control concrete ranges from 2430 to 2560 kg/m³. At early stages, say 7 to 28 days, concrete produced with metakaolin has reduced density as compared to the control concrete, while beyond 28 days, concrete produced with 5% and 10% percentage replacement of cement with metakaolin has higher density as compared with the control concrete. Considering the lowest value of density reported in this study, it is higher than the density of conventional concrete of 2400 kg/m³, according to (Kazjonovs *et al.*, 2010) and comparable to the reported value of earlier investigators (Agboola *et al.*, 2020), for pozzolanic concretes.

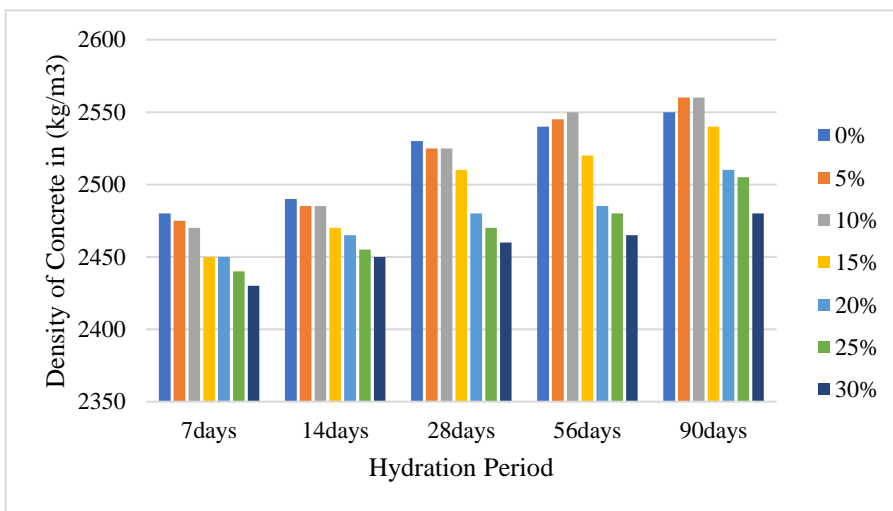


Figure 5: Density of Concrete Produced with Metakaolin

3.6 Compressive Strength of Metakaolin Concrete

Compressive strengths with percentage of metakaolin in concrete mix at different curing ages are given in Figure 6. In this study, MTK was used to partially replace cement in concretes at levels of 5%, 10%, 15%, 20%, 25%, and 30%, with water cement ratio of 0.5 at 7 days, 14 days, 28 days, 56 days and 90 days respectively. The control concrete and 10% cement have the same strength index and present high strength compared to other percentage cement replacement levels at 28 days of curing age. 10% replacement is higher than control and design strength beyond 28 days. This may be due to the hydration of cement with metakaolin. The high strength in 10% cement replacement may be due to the MTK behaving as a filler that decelerates the cement hydration process. This deceleration process of the hydration generates a large amount of calcium hydroxide at later curing ages to react with the active silica and aluminate phases of the metakaolin to produce secondary

calcium aluminosilicate hydrates, which later enhances strength performance in concrete (Mark et al., 2016). However, a further increase in metakaolin content shows a decrease in compressive strength compared to control concrete. In addition, the reduction in compressive strength of concrete with higher cement replacement levels may be due to the saturation of the cement mix with K_2O , which forms a composite that alters and limits the formation of calcium silicate hydrates from cement hydration. MTK can be used up to 10% to replace cement for concrete production since the results showed that concrete tends to gain more strength over a more extended period beyond 28 days of curing age which is in agreement (Olutoge et al., 2012).

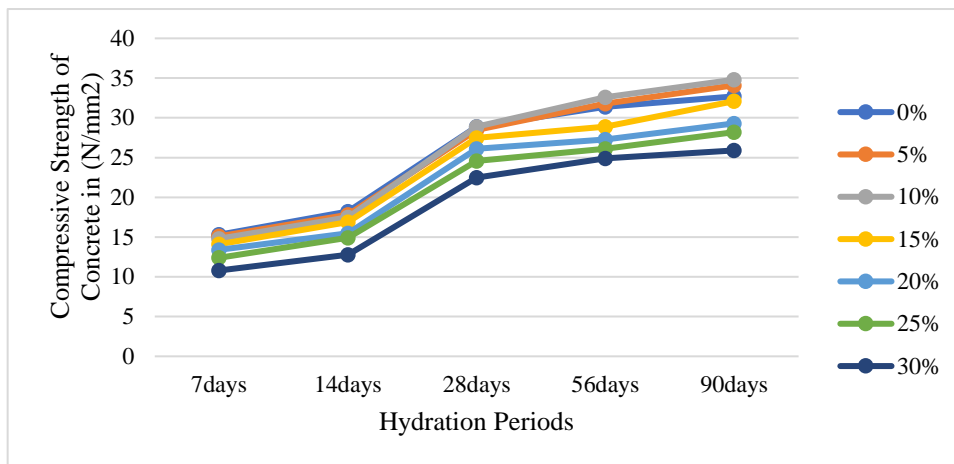


Figure 6: Compressive Strength of Concrete Produced with Metakaolin

3.7 Split Tensile Strength of Metakaolin Concrete

Figure 7 shows the split tensile strength of concrete produced with metakaolin at 5% to 30% replacement level. The split tensile strength of concrete attained is in the range of 2.08 to 2.48 MPa and 2.63 to 3.26 MPa at 7 and 28 days, respectively. The result shows that the split tensile strength of concrete increases as curing age increases. The control concrete is stronger than other percentage cement replacement levels at earlier curing age. At later curing stages beyond 28 days, 5% and 10% are stronger than the control concrete. The high strength observed in 5% and 10% cement replacement may be due to the MTK behaving as an excellent filler and makes good bonding between the aggregates and paste of the concrete. It is observed that the replacement of cement with metakaolin, up to 15%, has only a marginal reduction in splitting strength.

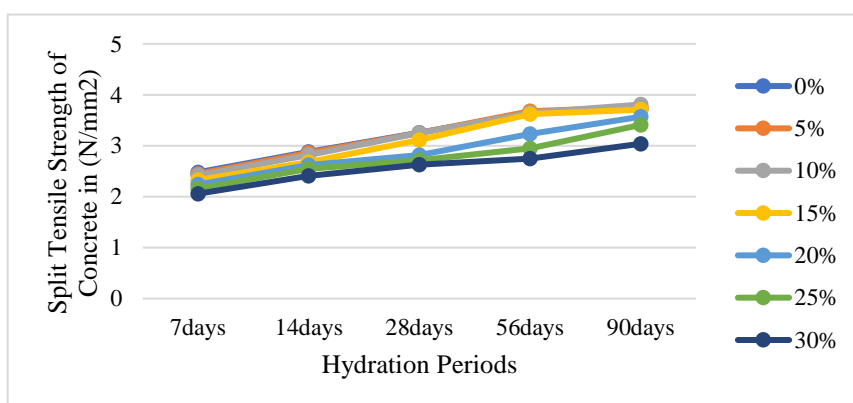


Figure 7: Split Tensile Strength of Concrete Produced with Metakaolin

3.8 Flexural Strength of Metakaolin Concrete

Figure 8 shows the flexural strength of concrete produced with metakaolin at a replacement level of 5% to 30%. The flexural strength of concrete attained ranges from 3.69 to 4.37 MPa and 3.91 to 4.65 MPa at 7 and 28 days, respectively. The result shows that the flexural strength of concrete increases as curing ages increases. The control concrete is stronger than other percentage cement replacement levels at 7 and 28 days of curing age. At later curing stages, 5% and 10% are stronger than the control concrete. The high strength observed in 5% and 10% cement replacement may be due to the MTK behaving as an excellent filler and makes good

bonding between the aggregates and paste of the concrete. It is observed that the replacement of cement with metakaolin, up to 15%, has only a marginal reduction in the flexural strength.

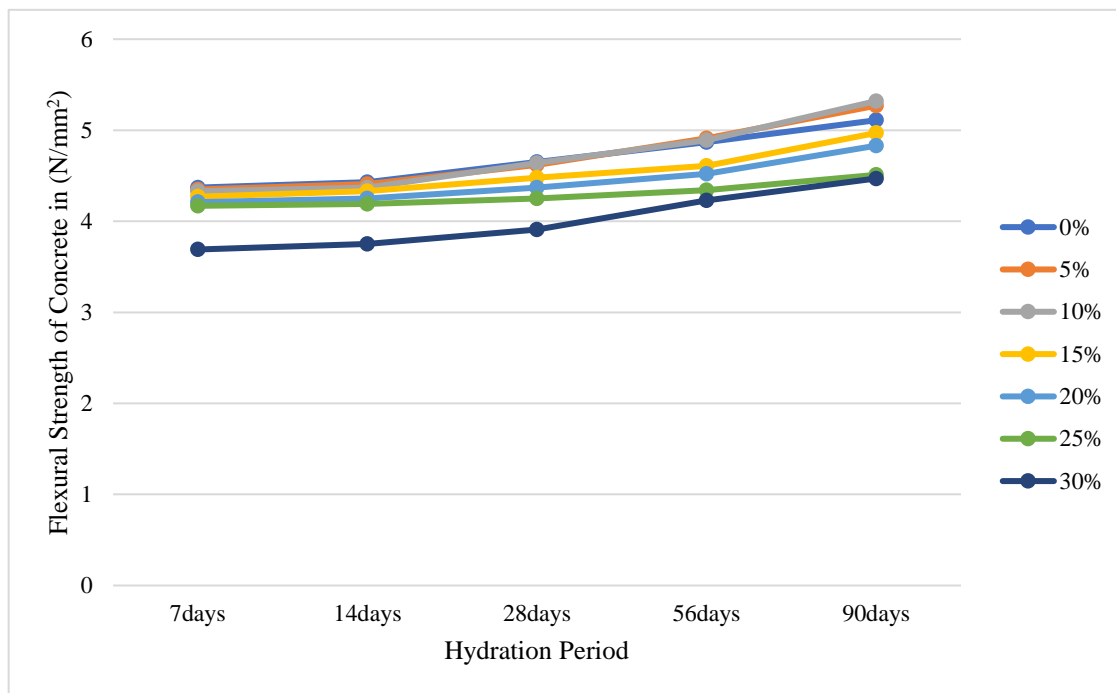


Figure 8: Flexural Strength of Concrete Produced with Metakaolin

3.9 Effect of Metakaolin as a Pozzolan on the Strength of Concrete

The study presents the percentage of cement replacement in concrete with MTK, which was replaced at 5% to 30% of cement weight. The results show the effect at various curing ages from 7 to 90 days. At early stages, up to 28 days, concrete produced with MTK has reduced density compared to the control concrete. The same trend was observed for compressive, split tensile, and flexural strength. The strength of concrete drops as the percentage of MTK increases. As the curing age increases beyond 28 days, concrete produced with 5% and 10% MTK has increase in strength beyond the control up to 90 days of curing age. This shows that the pozzolanic reaction progresses later in curing, and continued cement-MTK hydration will result in more compact concrete samples and higher strength. At earlier ages, there was little or no pozzolanic reaction as strength and density did not indicate any significant influence on the concrete samples, but at later stages, the impact of MTK as a pozzolana was observed. The effect of MTK on the density of concrete shows that as the density of concrete increases, the compressive strength of concrete also increases.

4. Conclusion

The results of the pozzolanic property and the oxide composition of metakaolin showed that it is a good pozzolana with essential constituents, which include 48.92% SiO₂, 43.46% Al₂O₃ and 0.51% Fe₂O₃ content, summing up to 92.89%. The presence of mineral constituents such as silica, alumina and ferrite oxides in MTK showed a promising potential for a pozzolana. Utilizing locally available materials is an option to reduce the cost of production of building materials. Using locally available materials as building materials will also solve the environmental impact problem. In addition, producing every ton of metakaolin concrete reduces each ton of CO₂ emission from cement production and saves the environment significantly by reducing greenhouse gas and particulate emissions. The MTK-blended cements have a higher setting time than the control; hence, they are most applicable where a low rate of heat development is required, such as in mass concreting. This indicated that MTK-blended cement is as good as low-heat cement. Concrete made using metakaolin to replace cement partially can be used for construction works involving long hauling distances due to its increased final setting time. The optimum MTK content is 10%, considering the compressive strength of concrete at 90 days. Metakaolin up to 20% can be used for mass concrete production. It is recommended that further tests be conducted to determine the permeability, shrinkage resistance, fire resistance, and durability of concrete and mortars made with metakaolin as cement replacements. Admixtures may be added to improve performance; using a different mix and altering the water-cement ratio is also recommended. The test should

also be conducted to determine the effects of metakaolin on the mechanical properties of concrete for a longer curing duration beyond 90 days.

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