

# Evaluation of Rheological Properties of Local Metakaolin Based Geopolymer Concrete

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*Abstract- Studies on properties of local metakaolin geopolymer concrete are limited to strength and its workability. The durability properties of this novelty concrete through water penetration properties have been evaluated and presented as the findings of this investigation work. In this work, geopolymer concrete is made by mixing the Alkaleri metakaolin and the alkaline solution made of 10M sodium hydroxide solution and sodium silicate solution of 98% purity all from Bauchi chemical and equipment store. The alkaline solution was prepared from sodium silicate to sodium hydroxide ratio of 1.5. Five mix proportions were designed by central composite design of response surface methodology, which was used to produce the 100mm X 100mm X 100mm geopolymer concrete cubes with the preheated binder for 45minutes at 60°C and cured for 24hrs at the laboratory ambient temperature. The influence of the water absorption, apparent volume of permeable voids, sorptivity and permeability on the geopolymer concrete cubes were evaluated. From the results obtained, it is found that the geopolymer concrete is a very good water penetrating resistance material according to the water permeability values recorded. Also, this is evident by the low values of water absorption, apparent volume of permeable voids and sorptivity recorded by this study. It was found that use of low water to binder ratio say 0.23 could help much in achieving low water penetrability value of geopolymer concrete and enhance compressive strength at the same time. Also the metakaolin content positively influences the water penetrating properties of geopolymer concrete.*

*Key Words: Water Absorption, Apparent volume of permeable voids, Sorptivity, Permeability.*

## I. INTRODUCTION

Properties of water penetration such as absorption, permeability and sorptivity are some crucial concrete durability measurements. Penetration of liquid into the concrete involves movement of the liquid or fluid through a porous medium, diffusion and absorption. In this case, pores in concrete have an important role to allow the liquid/fluid move through the concrete.

However, the tendency of concrete to absorb and transmit water by capillary action are not only depend on the porosity but also on its pore diameter, distribution, tortuosity and continuity. Geopolymer concrete is a new type of concrete that can be made from fly ash/metakaolin/slag and mixed with alkaline solutions. Many studies confirmed that fly ash/metakaolin geopolymer concrete has good engineering properties (Hardjito, 2005; Fernandez-Jimenez, et al. 2006; Sofi, et al. 2007). Other studies also show that low calcium fly ash geopolymer concrete has good durability on acid and sulphate condition (Wallah, 2005), alkali-aggregate reaction (Guneyisi, et al. 2008) and fire resistance (Kong, et al, 2007). Despite the superior resistance of the low calcium fly ash geopolymer concrete in various severe environments, according to Kong, et al. (2007) and Sabir (2001), the fly ash geopolymer paste contains higher proportion of pores in the mesopores size. This condition may lead water to penetrate easily and will affect the durability of the material. Previous studies made on geopolymer concrete show that metakaolin geopolymer concrete has permeability of 10-11 m/s (Davidovits, 1994a), while Shi (1996) and Zain (2000) found that permeability of slag geopolymer concrete is more than 10-12 m/s. Since the water penetration properties of local metakaolin geopolymer concrete is rarely reported, hence the aim of this investigation is to determine water penetration properties of Alkaleri metakaolin geopolymer concrete, namely water sorptivity, permeability, water absorption and Apparent volume of permeable void.

## II. MATERIALS AND GEOPOLYMER CONCRETE PREPARATIONS

Geopolymer concrete in this study was made from local Alkaleri Metakaolin (AMK) mix with a combination of sodium hydroxide solution (NaOH)

and sodium silicate solution (Na<sub>2</sub>SiO<sub>3</sub>). The chemical composition of the AMK from Alkaleri Local Government Area in Bauchi State, Nigeria is shown in table 1.

Table 1. AMK Chemical Analysis

Oxides	%
SiO <sub>3</sub>	53.30
Al <sub>2</sub> O <sub>3</sub>	31.54
Fe <sub>2</sub> O <sub>3</sub>	10.20
CaO	2.27
K <sub>2</sub> O	0.55
MgO	1.44
Na <sub>2</sub> O	0.36
P <sub>2</sub> O <sub>2</sub>	1.58
SO <sub>3</sub>	0.32
LOI	0.54

The AMK geopolymer concrete mixes were designed according to the central composite design (CCD) technique in response surface methodology (RSM) and displayed in table 2. It can be observed that the mix design parameters are percentages of the precursor which is the AMK content. The mixes are primarily used to produce geopolymer concrete with properties and performance that is equivalent to OPC concrete of grade 30N/mm<sup>2</sup>. Five different mixes for the Alkaleri Metakaolin based geopolymer concrete were made by varying the metakaolin content (350-450grams) and water/binder (W/B) ratio (0.21-0.25). The optimal dosage of Na<sub>2</sub>SiO<sub>3</sub>/NaOH ratio is set at 1.5, using a 10M NaOH concentration solution. The sodium hydroxide solution was obtained by dissolving the measured required quantity of the sodium hydroxide pellet in water and the solution was left to stay for 24 hours. The sodium silicate solution used was supplied by Bauchi Chemicals Nigeria Limited. The extra water was included in the mixes to improving the workability. Then, the metakaolin powder was mixed with the sodium hydroxide solution and stirred for 1 minute. The sodium silicate solution was added, and the mixture was heated for 45 minutes at interval of 15 minutes stirring at temperature of 60<sup>o</sup>c until it was well mixed. Plates 1 and 2 depicted the preparation procedure for the alkaline solution. The measured aggregates (sand & gravel) quantities were added to the heated mixture and thoroughly mixed for 5 minutes. After the mixing procedure is done, the fresh geopolymer concrete was

cast into 100 mm X 100 mm X 100 mm cube mold. The concrete cube specimens were obtained after compacting and pounding. In this work the concrete works carried out include preparations of sodium hydroxide solution and alkaline solution, dry and wet mixing of the geopolymer concrete ingredients, compaction and placing of the prepared concrete into the cube moulds and ambient curing of the geopolymer concrete specimens. Compressive strength test on the geopolymer concrete cube specimens at 3<sup>rd</sup>, 7<sup>th</sup> and 28<sup>th</sup> days of curing was also conducted.

Table 2: Design of Mixes by Central Composite Design

Mix ID	Metakaolin (Kg/M <sup>3</sup> )	Alkaline Solution	Sand	Gravel	W/B	Extra Water
M1	350	0.35	0.6	0.4	1	0.05
M2	375	0.35	0.6	0.4	2	0.05
M3	400	0.35	0.6	0.4	3	0.05
M4	425	0.35	0.6	0.4	4	0.05
M5	450	0.35	0.6	0.4	5	0.05

### III. DURABILITY STUDY OF THE GEOPOLYMER CONCRETE

#### 3.1 Water Absorption Test

The water absorption test was performed in accordance to ASTM C 642 (ASTM 2006a) at 28 days of water curing, on the cube samples of 100 × 100 × 100 mm geopolymer concrete. These concrete cubes were oven dried at 110<sup>o</sup>C for 24 h until the mass became constant and were then weighed as W1. The specimens were then immersed in water for 24 h and weighed as W2. The percentages of water absorption were calculated as:

$$Wab (\%) = (W2 - W1) / W1 \times 100 \text{ ----- (1)}$$

The absorption test was carried out on two 100 mm cubes as per ASTM C 642 (ASTM 2006a) at 28 days of water curing. Saturated surface dry cubes were kept in a hot air oven at 100–110 °C till a constant weight was attained. These are then immersed in water and the weight gain was measured at regular

intervals until a constant weight is reached. The absorption at 30 min (initial surface absorption) and final absorption (at a point when the difference between two consecutive weights at 12 h interval was almost negligible) is reported to assess the concrete quality. The final absorption in all cases is observed to be at 72 h.

### 3.2 Volume of Permeable Void Test

The volume of permeable voids test for the geopolymer concrete sample was carried out according to test procedures spelled out in ASTM C 642. The concrete cubes were oven dry, saturated by immersion in water, boiled for at least 5 hours and cool for about 14 hours, weighing them in water, then measuring the percentage of boiled specimens with dried mass and mass in the water. The Apparent Porosity (Pa) (the percentage of the volume of permeable voids) is calculated using the following formula, based on the mass method (Archimedes' principle).

$$Pa = (Msat - Mdry) / (Msat - Msusp) \times 100\% \text{ ---- (2)}$$

Where:

Mdry = Mass of the dry sample

Msat = Mass of the sample saturated with a liquid (water)

Msusp = Mass of the saturated sample suspended in the liquid (water)

### 3.3 Sorptivity Test

Sorptivity test was carried out according to ASTM C1585-13 and Hall (1989). In this test, three specimens were dried in the oven at 105°C to constant mass. After cooling, the bottom surface of the specimen was cut and this cut surface was made to contact with water. The test was carried out by measuring the weight gain of the specimen at the set time intervals of 5 min, 10 min, 30 min, 1 h, 2 h, 3 h and 4 h. Then uptake of water per unit area of concrete surface I (g/mm) followed a linear relationship with the square root of time for the suction periods (t), hence

$$I = C + ST^{0.5} \text{----- (3)}$$

Where S, the sorptivity, is the slope of I vs. T<sup>0.5</sup> plot and can be obtained by linear regression.

### 3.4 Permeability Test

Permeability coefficients in the range from 10<sup>-4</sup> to 10<sup>-10</sup> m/s for construction materials are measured by the procedures spelled out in the falling head method. Hence this study adopted this method. The geopolymer concrete cubes coefficients of permeability were calculated using equation 4 below.

$$Kt = 3.84aL/At \text{ Log}_{10} (h_1/h_2) \times 10^{-5} \text{ m/s ----- (4)}$$

Where a = cross sectional area of tube

L = length of the permeable cell or sample

A = cross sectional area of sample

t = time elapse in seconds or minutes

## IV. RESULTS AND DISCUSSIONS

### 4.1 Fresh and hardened properties of the geopolymer concrete

Table 3 presents the workability through slump measurement and compressive strength of the geopolymer concrete cubes. The slump measured for the five geopolymer concrete mixes appeared to have high values, probably due to the addition of extra water. Even though the slump values are within the range of 100mm to 135mm and fall within the acceptable limit of true slump, yet, the mixes are workable. Increase in metakaolin content and W/GS ratio, is followed by increase compressive strength values up to M3 and decreases followed from M4 to M5. This suggests that M3 is the optimum mix for the mixes. It shows that compressive strength is enhanced when there is increase in metakaolin content and W/GS ratio up to a certain limit. It also shows that geopolymer concrete of grade 30 and above could have high slump value. According to Hardjito, et al. (2004), when water content is drastically increased, it will tend to produce bigger crystals of geopolymer mix and decrease the specific surface area of the concrete. This will eventually lead to a decrease in strength. M3 which has the highest compressive strength of 34.94N/mm<sup>2</sup> at 28 day curing, is higher than M2 and M4 by 7.0% and 10.0% respectively. This confirmed the optimality of M3 mix. This is evident that metakaolin content significantly influence compressive strength development of geopolymer concrete, while water/binder ratio influences more as depicted in figure 1.

Table 3. Measured Slump and Compressive Strength of the Geopolymer Concrete

Mix I.D	Slump (mm)	3dComp. Str. (N/mm <sup>2</sup> )	7day Comp. Str. (N/mm <sup>2</sup> )	28d Comp. Str. (N/mm <sup>2</sup> )
M1	101	26.26	27.95	30.58
M2	108	28.14	29.95	32.76
M3	112	30.02	31.95	34.94
M4	123	27.17	28.91	31.62
M5	135	26.05	28.54	31.44

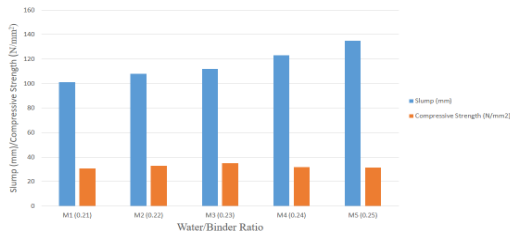


Fig.1: Variation of Water/Binder Ratio versus Slump and Compressive Strength.

#### 4.2 Water Absorption and Apparent Volume of Permeable Voids

The results for the water absorption and the Apparent Volume of Permeable Voids (AVPV) at different water/binder ratio levels for the five geopolymer concrete mixes are presented in Figure 2. The AVPV decreases with an increase in the metakaolin content, this could be due to the reduction of the open pore spaces (porosity) among the metakaolin particles as the metakaolin content increases. Both the water absorption and AVPV increase with an increase in water content of the mixes. This indicated that the water absorption and AVPV were much affected by the addition of the extra water to the mixes since it increases capillary porosity of concrete. This is evident in figure 2. The water absorption and AVPV for mix M5 with water/binder ratio of 0.25 has higher values than the other mixes. This suggest that geopolymer concrete requires a very small amount of water during mixing. The additional extra water is meant to achieving a particular strength and workability. However, a relatively higher water/binder ratio produces a weaker and pervious concrete matrix, leading to higher capillary porosity, in turn responsible for the increase in water absorption and AVPV of geopolymer mixes.

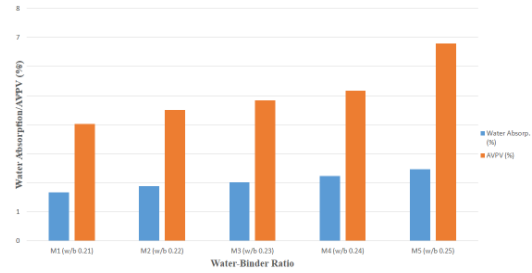


Fig 2: Variation of Water/Binder Ratio versus Water Absorption and Apparent Volume Permeable Void.

#### 4.3 Geopolymer Concrete Sorptivity

Figure 3 depicts the sorptivity values of the geopolymer concrete at various water/binder ratio. As can be seen, the results revealed that the sorptivity is significantly influence by the water/binder ratio. This evident by the increase in the two (water/binder ratio and sorptivity) is proportionate for all the mixes. This suggests that the increase in the amount of water resulted in high porosity concrete, hence increases capillary suction. The sorptivity values recorded by this study are in close agreement with some previous studies such as Olivia et al., (2008). According to Bai et al. (2002), the decrease in indirect measure of permeability of concrete (sorptivity) is due to the influence of particle packing on the capillary pore structure wherein a wide distribution of MK particle sizes exists resulting in a denser packing than the mixtures with cement only, thus reducing the sorptivity. Also, according to Monta Olivia et al., (2008), for all geopolymer mixes, the sorptivity reduces with a decrease of water content. They also reported that the relative sorptivity values clearly reflected the strength values whereby the lowest sorptivity values had the highest strength. This shows that the water/binder ratio has significant influence on sorptivity, strength, water absorption and AVPV of geopolymer concrete mixes.

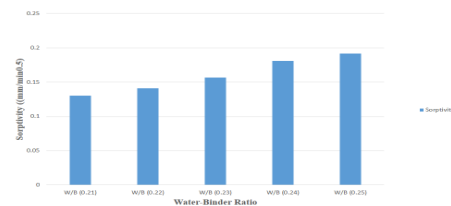


Fig. 3: Variation of Sorptivity versus Water/Binder Ratio.

M4	425	0.017
M5	450	0.015

#### 4.4 Geopolymer Concrete Water Permeability

The speed of water penetrating the different concrete mixes were measured for evaluating the permeability coefficients of this study concretes. The water permeability coefficients recorded is presented in table 4, while the variation of permeability with MK content is depicted in Fig. 4. All the permeability values fall within the range of 0.023 to 0.015 x 10<sup>-11</sup> m/s. According to Concrete Society Technical Report 54 (Rendell, et al. 2002) the MK concretes are in the range of “Good” concrete quality. Suggesting that the permeability was decreasing with increase in metakaolin content. This is because the pore sizes decreased with time either by reduction in the voids or by disconnecting the interconnected voids with hydration products or the metakaolin particles. In the present study, it was observed that M5 exhibited the lowest coefficient of permeability. This could be due to the fact that the pores were filled by hydration products, which would result in pore reduction leading to improved performance of the concrete (Jawed et al. 2021). Also, the permeability of the geopolymer concrete has a tendency to increase with an increase in water/binder ratio. It is obvious that concrete mixes with high water/binder ratio will have high permeability coefficient value. This can be attributed to the presence of large capillary pores that allows water to flow easily in much quantity within a small span of time. Thus, the open pores space in the concrete and the rate of the water flow within the concrete are proportionate. Thus, the ability to fill the pore structure depends mainly on the curing process. The increase of strength, eventually, is only a continuing effect of geopolymerization without any hydration. This shows that variation in water/binder ratio significantly influences geopolymer concrete permeability.

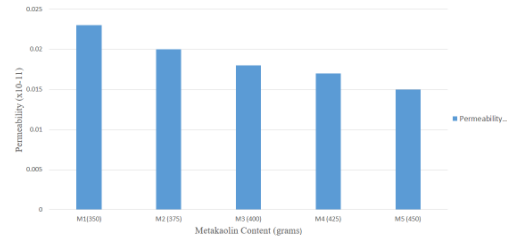


Fig. 4: Variation of Permeability versus Metakaolin Content.

## V. CONCLUSIONS

Based on the discussions on the test results obtained, the following conclusions can be drawn as:

1. Local (Alkalari) metakaolin geopolymer concrete exhibits high slump value up to 135mm when there is addition of extra water. Even though the concrete is workable, eventually, this may lead to decrease in the compressive strength.
2. The third mix (M3) appeared to be the optimal mix among the five mixes designed for this study with compressive strength of 34.9N/mm<sup>2</sup> at 28 day curing. This value is higher than M2 and M4 values by 7% and 10% respectively.
3. Alkalari metakaolin geopolymer concrete exhibits highest water permeability value of 0.023X10<sup>-11</sup>. According to Concrete Society Technical Report 54 (Rendell, et al. 2002), the metakaolin concretes are in the range of “Good” concrete quality.
4. According to the water permeability coefficient values recorded by this study, it can be inferred that water/binder ratio of 0.23 is adequate to be used, which in turn reduces capillary, porosity and the overall porosity of geopolymer concrete.
5. The results of this study revealed that increase in water/binder ratio is proportionate to water penetrability properties such as water absorption, sorptivity and water permeability. Also, observed that water/binder ratio is an important parameter that influences the water penetrability properties of local metakaolin based geopolymer concrete

Table 4. Water permeability coefficients of geopolymer concrete with different mixes

Mix ID	Metakaolin Content (g)	Water permeability coefficient (x10 <sup>-11</sup> m/s)
M1	350	0.023
M2	375	0.020
M3	400	0.018

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