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Characterization of Local Metakaolin Powder for Application as Geopolymer Precursor for Concrete Binder

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ABSTRACT
It is known that metakaolin powder has proved to be a promising alternative concrete binder to Ordinary Portland cement (OPC). Portland cement production contributes about 8% to the global CO_2 emission, which is detrimental to the environment and a major contributor to global warming. It is therefore pertinent to develop alternative cost-effective and environmentally friendly cement materials. This study examines the performance of a Nigerian metakaolin-based powder as a sustainable material alternative to Portland cement. The Gwaram-Alkaleri Kaolin clay from Bauchi State, Nigeria, was calcined at 800°C for 3 hours as step prior to metakaolin formation. The powder specimens were characterized using XRF, moisture content, specific gravity, density, particle size distribution analysis and specific surface area techniques. Parametric analyses of the values of the specific gravity, density, particle size distribution and specific surface area were undertaken and comparisons and deductions made. The results revealed that Gwaram-Alkaleri metakaolin-powder can serve as a potential sustainable construction material for the Nigerian construction industry because of its availability, affordability, physical and chemical properties values, and significant reduction in CO_2 emission, taking cognizance that the results positively indicate that it can be used as an alternative binding powder to cement in concrete and mortar production.

I. INTRODUCTION

Geopolymer is a sustainable alternative to Portland cement that can be used as a binder (Duxson et al., 2007; Provis and Bernal, 2014). Geopolymers are synthesized from an aluminosilicate precursor and an activating medium such as alkali (Giogetti et al., 2021; Liu et al., 2020). Extensive studies on the development and applications have been carried out all over the world. However, the majority of such geopolymers are synthesized using fly ash and/or slag~~as~~ aluminosilicate precursors (Das et al., 2020; Zakka et al., 2021).

With the limited availability of these conventional precursors all over the world, it is essential to find alternative locally available materials that can be used in the synthesis of geopolymers. In certain parts of the world such as Nigeria where clayey materials are available in large quantities, such materials can be sourced and utilized in the synthesis of geopolymer in order to promote sustainable construction. Clayey materials such as kaolin and halloysite are some of the locally available materials that can be calcined to obtain metakaolin (MK) and meta-halloysite (MH), respectively, and then used in the production of geopolymers. Current studies on the use of clayey materials as a precursor in geopolymers are mostly focused on the tropical laterites (Kaze et al., 2018, 2017). However, there is a need to explore the use of calcined clayey materials such as kaolin and halloysite in the synthesis of geopolymers. Limited studies exist currently on the use of MK and MH as precursors in geopolymers (Bouna et al., 2020; Nana et al., 2020; Sellami et al., 2019). The majority of the existing studies only focus on the influence of the precursors on hardened properties such as compressive strength. However, understanding the characteristics of these precursors (i.e. MK and MH) and their corresponding influence on the reactivity and fresh properties of geopolymers is limited, especially the internal mechanism of their conversion to binders.

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Tchakoute et al. (2020) focused their study on the mechanical and microstructural properties of the end products made using MK and MH as the precursor. Findings from the studies showed that a compressive strength up to 88 MPa can be achieved with the use of MH as the pre-cursor, which is very attractive because cement concrete are far lower. The higher compressive strength of geopolymer binders made with MH compared to MK was associated with the finer nature of the MH that resulted in higher dissolution and a corresponding higher polycondensation that favoured the formation of dense and strong matrix. However, there is limited or no information on the reactivity and the influence on the fresh properties of the geopolymers. The study by Zhang et al. (2012) demonstrated that the presence of halloysite (31%) as a secondary mineral in kaolin improved the reactivity of MK. The isothermal calorimetry conductivity (ICC) assessment carried out in the study showed that the presence of halloysite as a secondary Mineral contained in kaolin favoured a high degree of geopolymerization of metakaolin as recorded by the heat released.

Despite these existing studies on the use of MH or MK as precursors in the synthesis of geopolymers, there is no study where a comparative evaluation of both MH and MK has been carried out. Moreover, there is limited knowledge on the comparative study based on fresh properties of MH and MK used in the synthesis of geopolymers especially in terms of setting time, rheology and kinetic reaction from metakaolin and meta-halloysite based geopolymers. Besides, most of the existing studies only focus on the influence of these precursors (MH and MK) on the properties of geopolymers without an in-depth study on the properties of the precursors themselves and how they influence the corresponding performance of the geopolymers. Therefore, this study aims to characterize the Gwaram-Alkaleri Metakaolin and use it as precursor in the synthesis of geopolymers.

II. MATERIALS AND EXPERIMENTS

The materials that were characterized are Alkaleri Kaolin clay and its metakaolin by product. The kaolin clay used for this study is a natural occurring material that is whitish in colour. The kaolin was in pellets form, purchased from local miners at Gwaram village of Alkaleri district in Alkaleri Local Government Area of Bauchi State, Nigeria.

The Calcined kaolin clay (metakaolin) is the powder material that passed sieve 75μm. Some quantity of the metakaolin sample was transported to the Nigerian Geological Survey Agency Kaduna for its chemical composition analysis, by way of X-ray fluorescence analysis, with the method outlined by Usman et al. (2020) being adopted.

For the purpose of quality control evaluation, the Alkaleri Metakaolin (AMK) physical properties were determined through laboratory experiments conducted at the department of Civil Engineering laboratories, ATBU, Bauchi. These experiments included: specific gravity, density, particle size distribution and specific surface, which are briefly explained hereunder.

(i) Thermal Treatment of the Kaolin Clay: The kaolin sample was calcined at the Department of Industrial Design, Abubakar Tafawa Balewa University, Bauchi in a locally designed kiln, at a temperature of 800°C for 3hours to form metakaolin. The burning temperature was carefully monitored using a thermocouple. Samples of about 200 g each were heat treated in the laboratory furnace at temperatures of 200, 400, 600, 800 and 1000 °C and at heating times of 30, 60, 90, 120 and 180 minutes, respectively. After heating, the samples were "quenched" to room temperature at ambient conditions to avoid crystallization of the amorphous metakaolin. The weight of the samples before and after the thermal treatment was measured and recorded in order to determine the weight loss. The metakaolin sample was then sieved through 75um sieve to obtain the cement like powder.

(ii) Moisture Content of the AMK: Metakaolin exposed to the atmosphere becomes associated with same amount of moisture, which is both physically and chemically bound. It is customary to differentiate between external and inherent moisture. When metakaolin is exposed to conducive drying situation, the external moisture evaporates, but the apparently dry metakaolin still contains some moisture, which can be removed only on heating above 100^{oC}. External moisture is also called accidental or free moisture whereas inherent moisture is termed as equilibrium or air dried or hygroscopic moisture. The quantity of external moisture depends mainly on the mode of occurrence and handling of powder, but the air-dried moisture is related to the inherent hygroscopic nature of the fly ash. In the test procedure, about 1 gram of finely powdered passing (212 micron) air-dried metakaolin sample is weighed in a silica crucible and then placed

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inside an electric hot air oven, maintained at 1080±20C. The crucible with the sample is allowed to remain in the oven for 1.5 hours and is then taken out with a pair of tongs, cooled in desiccators for about 15 minutes and then weighed. The loss in weight is reported as moisture (on % basis).

(iii) Specific Gravity of the AMK: Specific gravity is the ratio of the mass of unit volume of soil at a stated temperature to the mass of the same volume of gas-free distilled water at a stated temperature. The specific gravity of a metakaolin specimen is used in the phase relationship of air, water and solids in a given volume of the powder. The specific gravity is obtained with a pycnometer bottle using 10grams of dry fly ash sample passing the No.10 sieve and distilled water, ensuring that the sample is soaked for 10 minutes, prior to filing with the distilled water.

(iv) True Density of the AMK: True density of metakaolin is the weight per unit volume of very finely powdered sample. Therefore, the volume of pores spaces and the interspaces is not included here. To determine the true density, fly ash sample is dispersed in water. The amount of water dispersed per gram of the powder gives the true density of the sample. The density of the metakaolin, which ranges from 2-2.8g/cm^3, determines the volume it will occupy for a given mass. Density changes may indicate a different kaolin source. For this purpose, the procedure is to take a measuring jar graduated in ml. clean it thoroughly with pure water. Take water into the flask up to certain level and note down its level (initial reading). Drop slowly 20 grams of the supplied metakaolin sample into the jar. Shake the jar for some time. Now note down the level of water in the jar (final reading). This procedure was repeated for 4 samples and the average result was tabulated as the true density of the AMK.

(v) Particle Size Analysis of the AMK: Particle size analysis was performed on the metakaolin to determine the particle size and the specific surface area. The particle size analysis was carried out by dissolving 1mg of the metakaolin powder in 10ml of distilled water and filtered through 0.2µm filter unit which was then transferred into the cuvette with the aid of stringed and analyzed using zetasizer Nano-series equipment for particle size analysis Nigerian Building and Road Research Institute Headquarters, laboratory Abuja.

(vi) Specific Surface Area of the AMK: Specific surface area (SSA) is defined as the ratio A / m (unit: m^2/g) between the absolute surface area of a solid and its mass (sample weight). The surface area includes all parts of accessible inner surfaces (mainly pore wall surfaces). It is possible to estimate the SSA using particle size distribution data based on the assumption that particles have spherical shapes.

For concrete mix design, only the outer surface in contact with water is of interest. With the consideration of the specific density, the SSA could also be expressed as area per volume (m^2/m^3). The total surface area of a set of aggregates is governed by the finer fractions of aggregates and powders according to the square-cube law. Assuming all particles were spherical in shape, the SSA, would be easy to calculate based on the particle size distribution and grading curves (McCabe et al., 1993), The square-cube law defines a mathematical principle describing the relationship between the volume and the area related to changes in size, and was first introduced by Galilei, (Galilei and Drake, 1946). According to the principle, as a shape grows in size, its volume grows faster than its surface area. Consequently, as the size decreases, its surface area reduces faster than its volume. The effect of the square- cube law becomes especially significant for calculation of the SSA of finer particles, namely powders and cement – that is, for a given mass of aggregate, the surface area increases with reducing particle size. The SSA can be calculated mathematically by the assumption of a spherical shape for the particle.

$$SSA= 6\sum_{i=1}^{n}\frac{Wi}{Di*ps} \text{-----------------} (1)$$

Where Wi is the mass of a grain fraction i, being the mass percentage of the fraction between Di and Di+1; ¯Di is the mean diameter of fraction i and i +1; and ρs_is the specific density of the particles. This law was used by this study in determining the Specific Surface Area of the AMK sample.

(vii) Determination of Chemical Composition of AMK: X-ray fluorescence was performed on the metakaolin sample to determine its chemical composition. The XRF was conducted at the Nigerian Geological Survey Agency, Kaduna, using energy dispersive x-ray fluorescence (EDXRF) spectrometer manufactured by PANalytical in Netherland of model “Minipal4’’ version PW 4030.

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III. RESULTS AND DISCUSSIONS

3.1 Dehydroxylation of the Kaolin Clay

The response of AMK to various thermal exposures is presented in the Figure 2. Studying these responses carefully using fig.1, and comparing with TGA and DTA analyses by Kakali, S., (2005) lead to other deduction. At temperatures below about 200°C release of water absorbed in pores and on the surfaces occurs. Between 200 and 450°C, mass loss attributed to the pre Dehydroxylation process takes place, as a result of the reorganization in the octahedral layer. In the temperature range 450–650°C, Dehydroxylation of kaolinite and formation of metakaolinite takes place, while at about 1000°C, mullite was formed, as indicated by an exothermic peak. The observed endothermic peak with a maximum at 562°C may be attributed to Dehydroxylation process. In order to obtain optimal calcination parameters, the AMK was subjected to thermal treatment at different heating temperatures and times. The mass loss of the AMK for the given calcination parameters is given in Table 3. As can be seen, for calcination temperatures of 500, 600 and 700°C, mass loss increases up to 90 minutes, while prolonged heating has a negligible effect on the mass loss. For all applied heating times at temperature 700°C obtained values for mass loss are nearly the same (13%). It is evident that at calcination temperature 600°C and heating time 90 min, mass loss is almost identical with the values obtained at 700°C. If we take economic factors into consideration, the optimal parameters for calcination are temperature 600 °C and heating time of 90 minutes. As can be seen, nearly complete Dehydroxylation was achieved after 90 minutes for temperature between 600 and 700 °C.

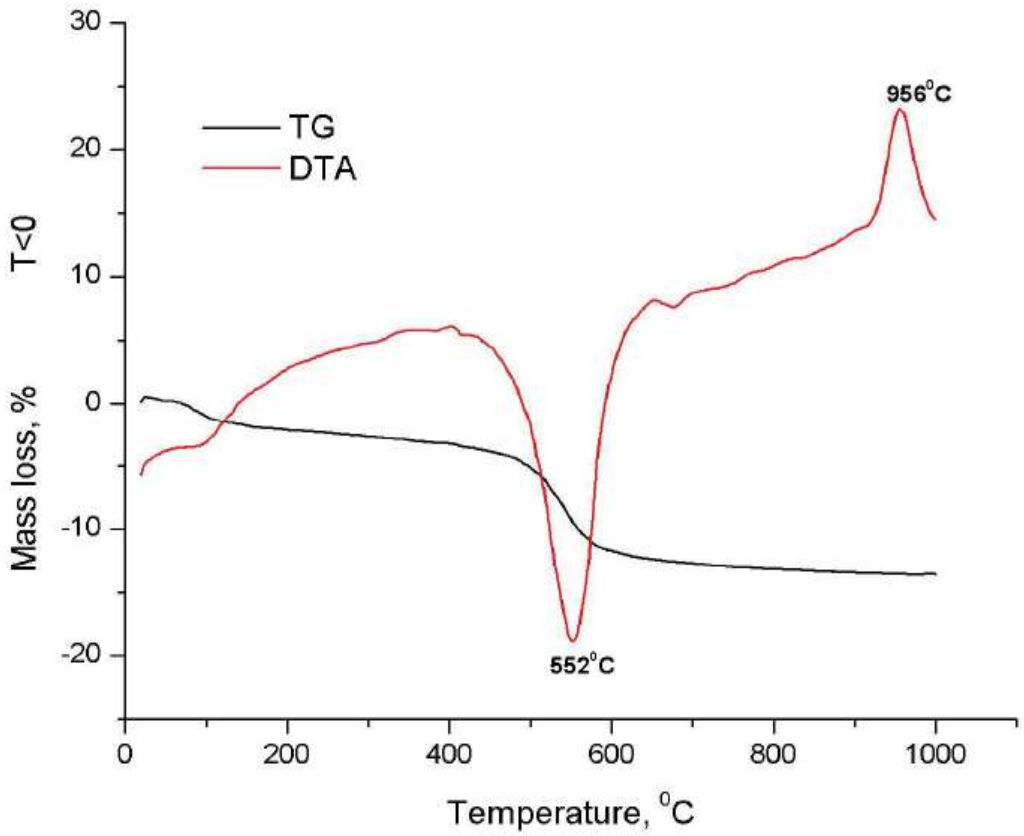

Figure1. DTA/TG curve of the starting clay.

Source: Biljana et, al., (2010)

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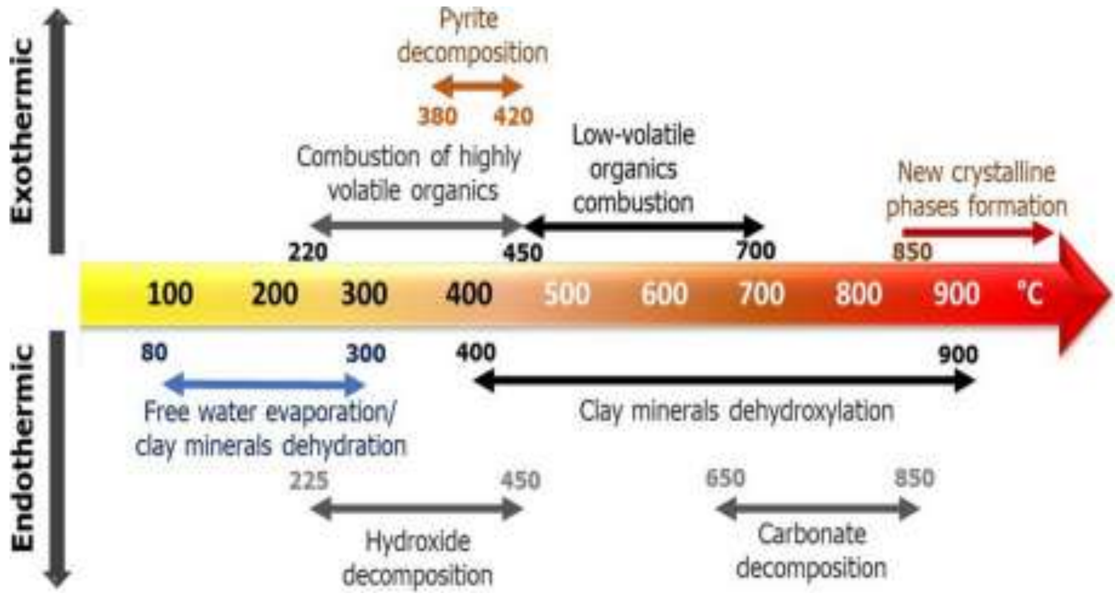

Figure: 2. CLAY CALCINATION TECHNOLOGY (Temperature range of typical reaction taking place during calcination of clay)

Source: Kakali, G., et, al., 2005 and The RILEM TC 282-CCL 2021.

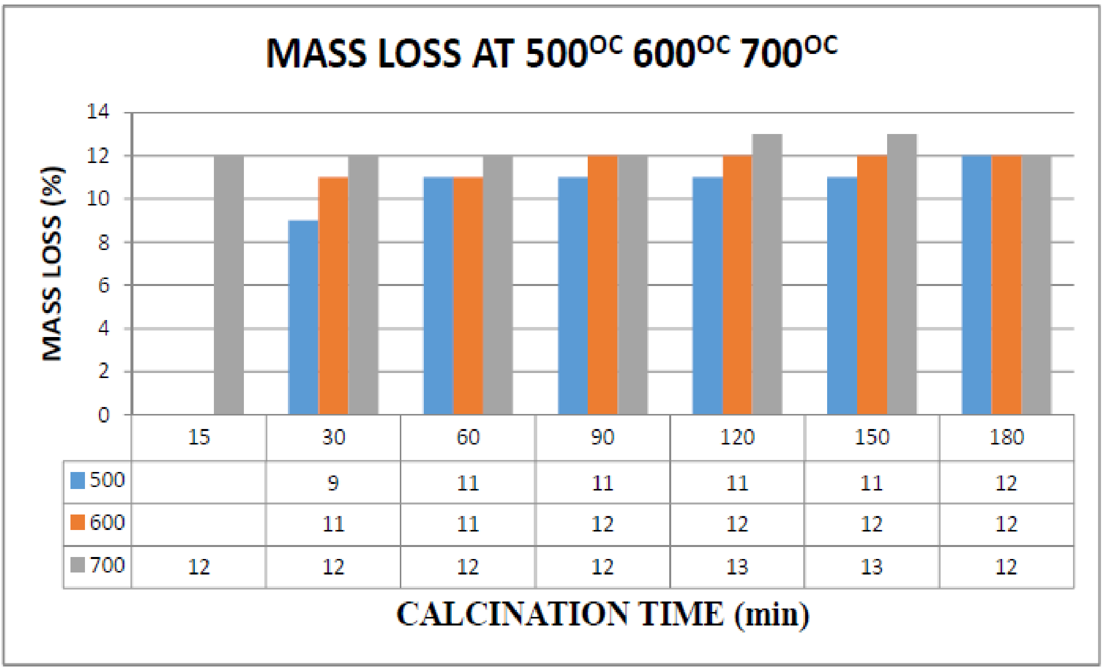

Figure 4: Variation of Mass Loss of Kaolin Clay with Calcination Time

3.2 Moisture Content

The moisture content of the AMK collected from Gwaram Village of Alkaleri District is presented in table 1 below. The average moisture content was found to be 0.177%, indicating that both the free and hygroscopic moistures have been evaporated.

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Table 1: Moisture Content of the AMK

S/No	Mass of Can (gm)	Mass of Sample (gm)	Mass of Can + Sample Before Heating (gm)	Mass of Can + Sample After Heating (gm)	Moisture Content (%)	Average Moisture Content (%)
1	15.236	1.001	16.237	16.102	0.135	
2	15.125	1.005	16.130	15.914	0.216	0.177
3	15.856	1.004	16.860	16.679	0.181	

3.3 Specific Gravity of the AMK

Table 2 presents the specific gravity of the AMK as 2.275, indicating that AMK is a pozzolan with high pozzolanic reactivity and of greater rate of hydration product formation.

Table 2: Specific Gravity of the AMK

S/No	Mass of Pycnometer (gm)	Mass of Pycnometer + dry Sample (gm)	Mass Pycnometer + dry Sample +Water (gm)	Mass of Pycnometer +Water (gm)	Specific Gravity	Average Specific Gravity
1	41.69	73.42	157.28	139.38	2.294	
2	49.93	74.69	157.29	142.40	2.254	2.275

Specific gravity plays an important role in proportioning of aggregates in concrete. The use of suitable specific gravity along with water/cement ratio in concrete mix can determine and evaluate the expected weight, quality and compressive strength of concrete mix. Hence quality of concrete mix is related to its specific gravity. The standard values for specific gravity of metakaolin material ranges from 2.00 to 2.54. The value of 2.275 for the AMK specific gravity falls within the specified limit by ASTM D854, indicating that AMK will not suffer segregation/bleeding during concrete processing and batching
.

3.4 True Density of the AMK

Based on the measured true density values presented in the Table 3, it can be seen that the AMK density is within the stipulated ASTM B527 standard values (minimum of 2.0g/cm^3), hence adequate for concreting works. It is proven that metakaolin levels affect the density of geopolymer paste or concrete. The higher the metakaolin content, the lower the density of geopolymer paste and concrete. It may happen that a geopolymer paste or concrete could have low density as well as low metakaolin content, this can be attributed to high amount of open pore in the metakaolin, suggesting that large particle size dominates the metakaolin (Palomo et. at., 2013).

Table 3: True Density of the AMK

S/No	Quantity of AMK (gm)	Initial Reading (gm)	Final Reading (gm)	Difference (cm^3)	True Density gm/cm^3	Average True Density gm/cm^3
1	20	80	89	9	2.22	
2	20	81	90	9	2.22	2.22
3	20	80	89	9	2.22	

3.5 Particle Size of the AMK

The laser particle size analyzer (Bettersize2000) was employed to determine the particle size of the samples. The Particle size significantly influences the physical and mechanical properties of geopolymer concrete. Figure.4 displays the result of the analysis of the particle size distribution for the Alkaleri metakaolin powder. It can be observed from Figure 4, that the particle size of the AMK is distributed

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across the range (23µm---9,000µm) with a D50 of 550µm. The width of the distribution is 7.13. Obviously, the particle size distribution for the AMK is multimodal, where the first mode which focused at 55µm, is regarding the smaller particle. While the mode related to the larger particles is centered at 1,500µm. From fig.4 again, it can also be observed that the mean diameter of the particles is about 400µm having 41% intensity, 750µm is the modal diameter of the particles with 84% intensity and the median diameter is 330µm with 36% intensity. In general, the particle size distribution of powder is significantly affected by the existence of agglomerations

. Meanwhile, the agglomeration content increases the percentage of larger particle sizes.

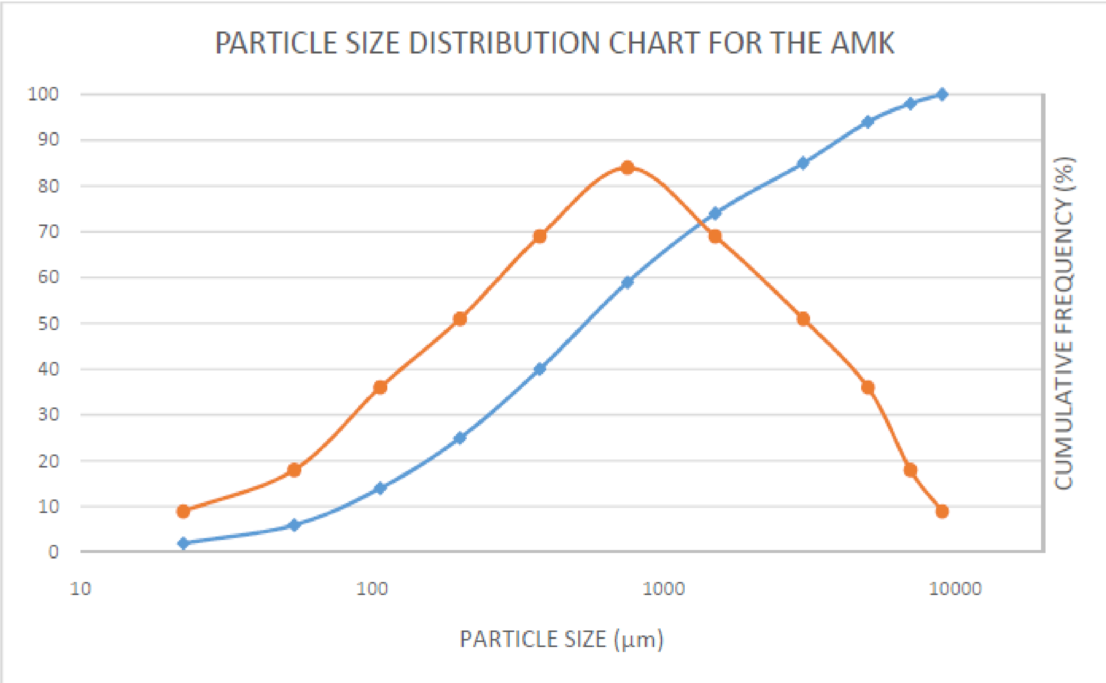

Fig. 5: Particle Size Distribution for the AMK Sample.

D10----80µm, D50---550µm, D90---4,000µm are obtained from the cumulative graph. Mean=400 µm, Mode =750 µm and Median= 330 µm are obtained from bell shape graph.

3.6 Specific Surface Area.

Based on data presented in table 4, it can be seen that the specific surface area of the Gwaram village metakaolin is about 14.88m^2/g, this is higher by 2.0% than the one reported by Salihu (2021), who worked on Pali village metakaolin, all of Alkaleri Local Government Area. Barbosa et. al., (2000) revealed that specific surface area of metakaolin increases with decreasing particle sizes, indicating a relationship that specific surface area of metakaolin is inversely proportional to particle size, hence the metakaolin particles contained pores. This relationship between the particle size and specific surface area of the AMK is depicted in figure 5. It can be observed that the higher the particle size the lower specific surface area of the metakaolin, showing that specific surface area for the AMK conformed to the Square-Cube Law.

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Table 4: Physical Properties of the AMK

Property	Value
Moisture Content %	0.17
Specific gravity	2.28
True Density g/cm^3	2.22
Specific Surface Area (fineness) m^2/g	14.88
Diameter of particle size (mean) µm	2,455.0
Physical form	Powder
Colour	Off white

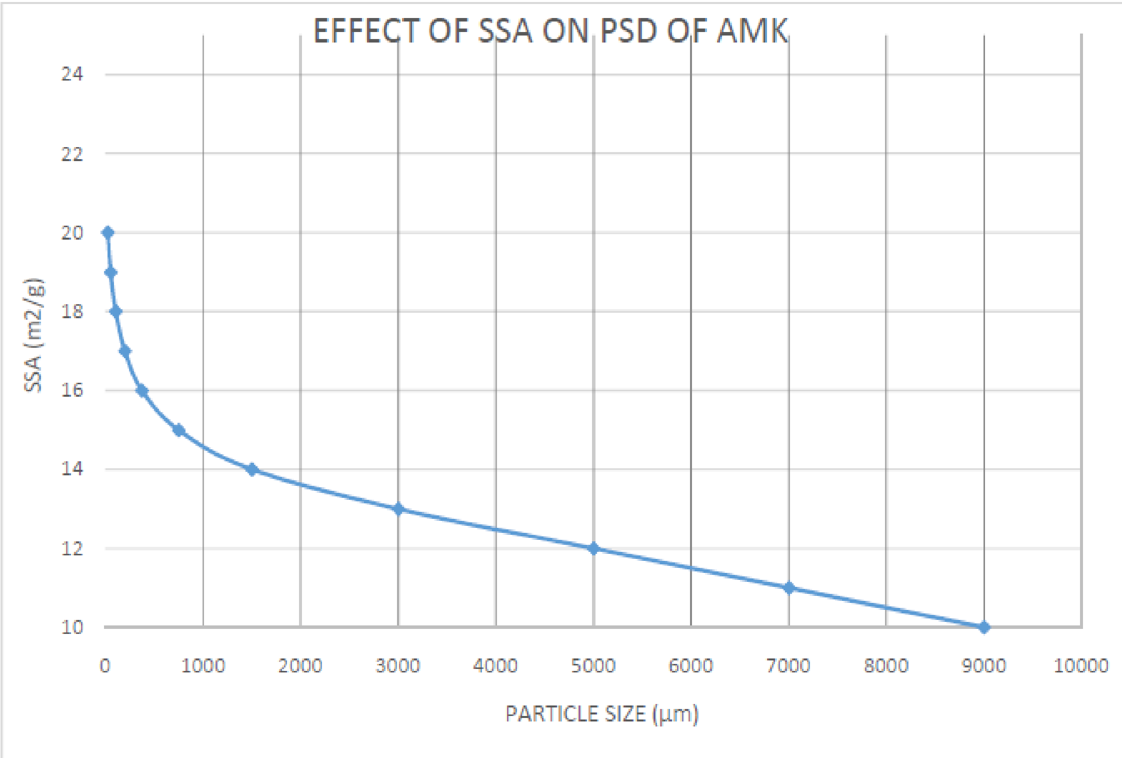

Fig. 6: Effect of Specific Surface Area on Particle Size Distribution for AMK

3.7 Chemical Composition of the AMK

The mineral oxides of the AMK determined by XRF technique is presented in Table 5. The major oxides present in the AMK are AL_2O_3, 31.60% and SiO_2, 53.70, in the ratio of 1.76. The trace amount of other mineral oxides such as Fe_2O_3, 4.89%, K_2O, 4.05%, Na_2O, 2.32%, CaO, 0.385%, SO_3, 0.06%, MgO, 0.40%, MnO, 0.11% and LOI, 1.41% is also shown in Table 8. This result has met the chemical oxides requirement defined by ASTM C618-17, 2017, and classified the AMK as class N pozzolan. Having Si/Al ratio of 1.76, there is likelihood that paste to be prepared with this metakaolin could experience slow dissolution, stiff paste and low workability as observed by Usman N.D. et. al., (2020), that a trial mix with Si/Al ratio >1.6 <2.0, the dissolution of the metakaolin was very slow and the resulting paste was stiff which will give a low workability. Salihu, C. H. et, al., (2021), collected his sample from Pali Village in the eastern part of Alkaleri town. While the sample for this study was collected from Gwaram Village in western part of Alkaleri town. The loss on ignition (LOI) of the Pali sample is 57.3% higher than that of this study, suggesting that Gwaram Kaolin for this study contains less carbon than Pali kaolin.

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Table 5: Chemical Composition of the Metakaolin

Oxides	Mass (%)
AL_2O_3	31.60
SiO_2	53.70
Fe_2O_3	4.89
Cao	0.39
SO_3	0.06
MgO	0.40
Na_2O	2.32
K_2O	4.05
MnO	0.11
L.O. I	1.41

The Oxides mass were measured at the Nigerian Geological Survey Agency, Kaduna.

IV. CONCLUSION

The physical and chemical characterization of the study materials were carried out and accordingly presented the results. From both the physical and laboratory results obtained, the following conclusions are drawn.

I. A detailed of review of literatures on starting material or precursor of geopolymer concrete has been carried out and consolidated. It was found that metakaolin is the right material for use as a geopolymer starting material.

II. The AMK produced is thermally stable at 300^{oC} as detected by Kakali et al., 2005 and can withstand temperature up to 800^{oC}. The optimal conditions of AMK thermal treatment are calcination temperature of $600^{oC}$to 700^{oC}, heating time of 90 minutes and pozzolanic activity is 780mg of $Ca(OH)_2$ per 1g of the AMK.

III. XRF results of the AMK revealed that the sum of the three oxides namely: Al_2O_3, SiO_2 and Fe_2O_3 are above 70% and met the requirement of ASTM C-618 for materials to be used as pozzolana. The tests results were found helpful in developing a better understanding of AMK behavior.

IV. The performance of the AMK produced could be predicted by the characterized physical, chemical and mechanical properties.

V. The characterization of the AMK carried out has exposed vital information such as its degree of resistance, reliability and its possible application.

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