International Journal of Advances in Engineering and Management (IJAEM)
Volume 6, Issue 12 Dec. 2024, pp: 34-45 www.ijaem.net ISSN: 2395-5252

Alkaleri Metakaolin Powder Performance Evaluation by Response Surface Method.

Usman Abubakar[1], Danladi Slim Matawal[1] Abbagana Mohammed[1] and S. A. Yero[1]
Department of Civil Engineering, Abubakar Tafawa Balewa University, Bauchi

Date of Submission: 01-12-2024 Date of Acceptance: 10-12-2024

ABSTRACT
Alkaleri metakaolin (AMK) based geopolymer binder has been identified as one of the alternatives to the Ordinary Portland Cement (OPC), which qualifies the criteria for green construction material. In this work, 20No of experimental mix proportions (RUNs) were designed by Central Composite Design (CDD) of Response Surface Method (RSM). Mixture of AMK and Alkaline Solution was heated for 45mins at 60^{oc} with stirring interval of 15mins, after which aggregates were added to the hot mix. 60No of 100X100X100mm cubes were cast. Samples were tested at the 28th day of ambient curing. The reported Compressive Strengths are the average of the 60 sample tested with 38.6N/mm^2 as the maximum, hence established the experimental optimum mix proportion (EOMP). The ANOVA model developed statistically validated by having F-value of 15.54 and P-value of less than 0.0001 indicating that the model is statistically significant. Also, the adequate precision of 10.7850 which is greater than 4.00 indicates adequate signal and desirable. Furthermore, the difference between predicted R^2 and adjusted R^2 is less than 0.2 testifying that they are rationally in good agreement. Effects of three principal variables namely: molarity of sodium hydroxide, sodium silicate to sodium hydroxide ratio, and fineness (particle size) on the properties of the fresh and hardened AMK geopolymer concrete (normal consistency, soundness, setting times, slump, dry density, water absorption, compressive strength, flexural strength, split tensile strength, sorptivity and acid attack) were investigated. All the results for the properties investigated were found to have fallen within the ranges of the international standard codes such as ASTM and British Standard, hence AMK is qualify to be used as Metakaolin based geopolymer binder.

I. INTRODUCTION

Geopolymer is a class of inorganic polymers formed by reacting silica-rich and alumina-rich solids with a high alkaline solution, which combines the properties of polymers, ceramics and cements. To fulfil the commitment of sustainable development, the concrete of tomorrow should not be restricted to strength and durability. Along these lines, geopolymer concrete is gaining popularity in the construction industry and considered an appropriate alternative building material which has been used in different field applications like precast squares, asphalt, blocks, and water tanks (Sabnaet al., 2014). Geopolymers have remarkable properties like low consumption of raw resources, little CO2 emission, low production cost, less energy consumption and rapid setting.

NaOH and KOH have been used as the alkalis for metakaolin geopolymer production. The concentration of these alkalis significantly influences the physical and mechanical properties of metakaolin-based geopolymer. It is among the most important factors for geopolymer synthesis. It determines the number of ions and controls the metakaolin aluminosilicate dissolution process (Xiao et al., 2009). Studies have shown that increasing the concentration of NaOH in metakaolin geopolymer in the range 4-12M increases the strength of geopolymer

(Ming et al., 2016). For geopolymer synthesized with low sodium content, OH-ion will be insufficient to completely cause the dissolution of aluminosilicate to Si4+and Al3+ions. This insufficient Na+ or k +ion cause incomplete geopolymerization.

To produce geopolymer, the proportion of Na_2SiO_3 to NaOH in the alkaline solution is paramount. It determines the rate and extent of geopolymerization reaction (Rahier et al., 2007).NaOH acts as an aluminosilicate dissolvent

International Journal of Advances in Engineering and Management (IJAEM)
Volume 6, Issue 12 Dec. 2024, pp: 34-45 www.ijaem.net ISSN: 2395-5252

while Na_2SiO_3 acts as a binder in the geopolymerization process. Previous studies have shown wide range use of different Na_2SiO_3/NaOH ratios in the study of metakaolin-based geopolymer. Na_2SiO_3/NaOH ratio of 0.24 was recommended by Wang et al. (2005) as the optimum for metakaolin-based geopolymer. The ultimate compressive strength achieved using this ratio was 59 MPa. On the other hand, Pelisser et al. (2013) in their quest to contribute to the development of geopolymer characterized the mechanical and micro and mechanical properties of metakaolin-based geopolymer through evaluation of the effect of the Na_2OSiO_2/NaOH molar ratios. 1.0, 1.6 and 2.2 Na_2SiO_3/NaOH ratios representing a higher range of alkaline reactant ratios were used in their study.

Solid and liquid content in geopolymer formation represents the aluminosilicate and alkali reactant respectively. The importance of solid/Liquid (S/L) ratio cannot be overemphasized as it governs the solid-liquid mass needed for homogeneous mixing which in turn could affect the geopolymer formation process, and the ultimate strength of the final geopolymer product(Ming et al., 2016). According to research conducted by Xiao et al. (2009) on geopolymerization process of alkali-metakaolin, S/L ratios above 2.0 were observed to result in metakaolin geopolymer with low workability while at low values of S/L ratio, geopolymerization reaction seemed to be delayed. An S/L ratio of 0.8 was recommended by Kong et al. (2007) as the optimum for metakaolin-based geopolymer. Kang et al. (2013), on the other hand, studied the effects of solid-to-liquid (S/L) ratios in the range of 0.97 to 1.19 and nano-SiO_2percentages (0%to 3%) on some properties of metakaolin-based geopolymer. In their work, the setting time and compressive strength were investigated. High compressive strength was obtained when the S/L ratio was 1.03. Early strength developed rapidly, and later strength developed steadily. When the amounts of 0%, 1%, 2%, and 3% nano-SiO_2were added, the compressive strength of geopolymer was 43.7 MPa, 68.4 MPa, 59.5 MPa, and 49.2 MPa at 60 days respectively.

Mechanical activation (MA) by milling enhanced the reactivity due to the combined effect of lower particle size, angular shape, increase in surface area and change in bulk and surface reactivity. Microstructural features such as increase in compactness, formation of more reaction product and changes in nature of gel are also associated with mechanical activation (Sanjay and Rakesh, 2010). 80% increase in compressive strength of the geopolymer was observed by Temuujin et al. (2009) which attributes to reduction of particle size and change in morphology allowing a higher dissolution rate of fly ash particles. Kiatsuda Somna et al. (2011) in their study obtained reasonable strength for geopolymer made from ground fine fly ash (median particle size 10.5 μm) at ambient temperature curing. The higher reactivity of the raw material through MA has been again confirmed by Sanjay Kumar et al. (2013) during the development of geopolymer from Silico Manganese (Si Mn) slag. Bajare et al. (2011) also agreed with the above findings in their geopolymer study with ground glass waste. According to VanchaiSata et al. (2012), fine bottom ash was more reactive than those of the coarser fly ashes, which exhibited higher compressive strength. As a result of the investigation done by

Gabor Mucsi et al. (2014), it was found that the geopolymer strength increased as function of fly ash fineness. Differences in the network modifying element content, the amorphous phase content, and the particle size lead to large differences in compressive strength (Jae Etim et al., 2014).

Despite geopolymer concrete being a promising alternative to portland/lime cement concrete, it has no code or mix specification to guide the choice of material mix proportion for specific grades of concrete; there is therefore the need to produce a mix design (material proportioning) that will achieve a specified strength. This study examines the properties of the source material (Alkaleri Metakaolin processed from Alkaleri Kaolin, Bauchi state) using X-Ray Florescence (XRF), particle size and pozzolanic reactivity analyses. A central composite design of experiment of response surface method in Design Expert 7.0 Statistical software was used to produce mix designs for Alkaleri Metakaolin-based geopolymer concrete that will achieve a characteristic compressive strength of 25 N/mm^2 within 28 day with ambient curing. This is necessary because geopolymer concrete is a new research area in Nigeria, not much study has been done to investigate the properties of the concrete despite the fact that Nigeria is blessed with abundant Kaolin clay deposits, for example, the Alkaleri, Kankara Kaolin clay deposits in Northern Nigeria and the Ajebo kaolin clay deposit in the South-west, Nigeria.

Matawal (2023) asserted that there may be environmental and economic advantages of utilizing refuse-derived fuels (RDF) sewage sludge (SS) in cement kilns to decrease CO_2 emissions in clinkers in order to reducing the carbon foot print of cement production, a major source of green house gas emissions. He asserted that alternative materials and innovative mix designs need to be explored but also specifically noted that supplementary cementing materials, SCMs, concrete and replacements, especially polymer cement that can lead to concrete in excess of 170 N/mm^2.

This study focuses on evaluating the performance of the best mix proportion design by central composite design in Response Surface Method of Design Expert 7.0 Statistical software for Alkaleri Metakaolin based geopolymer concrete. A first principled mix design using manual computation was used to cross-checked the software design.

II. MATERIALS AND EXPERIMENTS

For the purpose of quality control evaluation, the study materials physical properties were determined through laboratory experiments conducted at the department of Civil Engineering laboratories, ATBU, Bauchi. These included:

Fine Aggregate (FA)

Sharp sand devoid of silt and clay from Bayara village stream, in Bauchi Local Government Council of Bauchi State was used. The fine aggregate quality was evaluated by conducting the following laboratory tests: specific gravity, bulk density, particle size distribution and silt content. All the tests were conducted in accordance with specifications outline in their relevant standard codes test procedures.

Coarse Aggregate (CA)

Machine crushed stone of igneous rock origin, with maximum nominal size of 19mm from Triacta Nigeria Limited quarry, in Bauchi Local Government Council of Bauchi State was used as the coarse aggregate material. The coarse aggregate quality was evaluated by conducting the following laboratory tests: particle size distribution, aggregate crushing value and aggregate impact value. All the tests were conducted in accordance with their relevant standard codes test procedures.

Alkaleri Metakaolin

Kaolin clay from one of the local vendors in Alkaleri town, the Headquarter of Alkaleri local Government Council in Bauchi State, Nigeria was purchased. Kaolin clay was crushed to particle size lower than 10 mm and fired for 3 hour at 850 °C in a local furnace of the department of Industrial design, ATBU, Bauchi, in order to get the reactive phase of metakaolin (MK-850).

Sodium Hydroxide (NaOH)

Commercial grade Sodium Hydroxide in flakes form (99% purity) was purchased from a reputable chemical dealer in Abuja, Nigeria.

Sodium Silicate (Na_2SiO_3)

Commercial grade Sodium Silicate powder having specific gravity of 1.52, 15% Na_2O, density of $1.35 g/cm^3$ and 30% SiO_2, water of 55% was used in this study.

Water

The water used for this study is portable tap water fit for drinking, obtained from Yelwa Campus ATBU, Bauchi. Therefore, no test was conducted on the water.

Preparation of Alkaleri Metakaolin Powder

Metakaolin fineness (particle size) is one of the three response surface method boundary factors. The reactive phase of the metakaolin obtained as the by-product of the dehydroxylation process was sieved through sieve sizes 150, 75 and 63μm designed for the experiments by the response surface method.

Alkaline Solution Preparation

In this experimental work, the alkaline solution is a mixture of sodium hydroxide solution and sodium silicate solution. Firstly, sodium hydroxide solution of 8, 12 and 16 M concentrations of one litre standard solution were prepared. In each case, a known mass of sodium hydroxide pellets was dissolved in a known mass of distilled water to make the standard solution. The reaction was exothermic, so, the solution was allowed to cool down to room temperature (24 hours) after dissolution

Design of Experiments using Response Surface Method (RSM)

Response surface method (RSM) is a statistical approach uses for developing and validating the relationship between independent and dependent variables as factors and responses respectively. The whole process comprises of four steps in total, which include, the design of experiments, a collection of response by performing experiments, development of RSM numerical model and optimization and validation

International Journal of Advances in Engineering and Management (IJAEM)
Volume 6, Issue 12 Dec. 2024, pp: 34-45 www.ijaem.net ISSN: 2395-5252

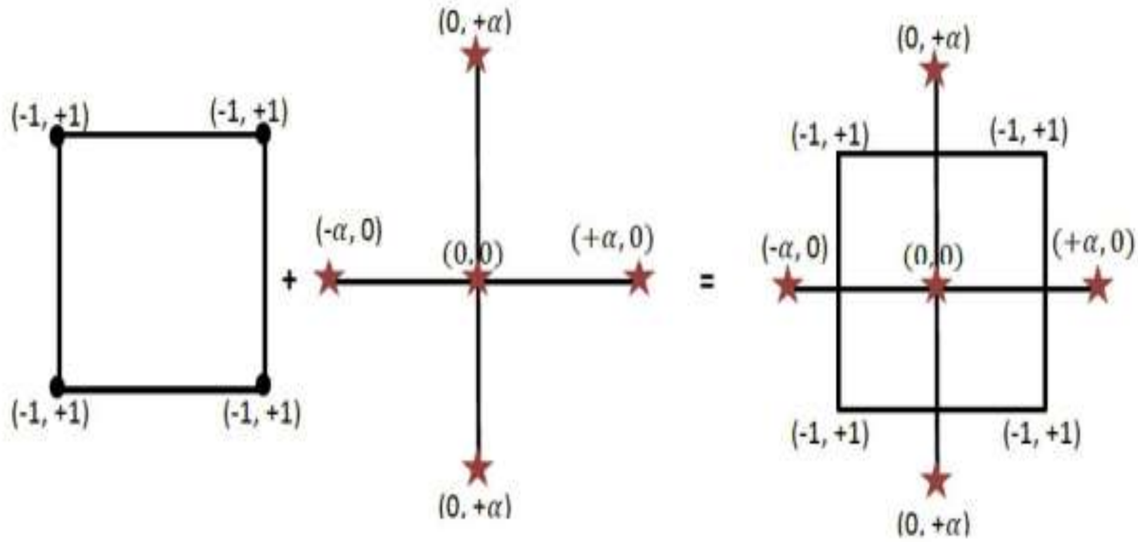
of the developed model (Ghafari et al., 2014). Central composite design (CCD) is the most practical and commonly used method for design of experiments in RSM technique (Jose et al., 2014). As shown in Fig. 1, CCD is a two-level factorial design; selection of data points include; corner points of the square for all +1 and -1 design points, a middle point between all factors, and star points having extreme +α and -α (Ferdosian and Camoes, 2017). Quadratic models are used to estimate the response (compressive strength), which is calculated according to the 2nd-degree polynomial equation (Ghafari et al., 2014).

$$y = \beta_0 + \beta_i X_i + \sum \beta_{ii} X_i 2 + \sum \beta_{ij} X_i X_j \text{ ---------------- } (1)$$

Where "y" represents the predicted response, which is a mechanical characteristic (compressive strength) of Engineered Geopolymer Composite. Furthermore, bo, bi, bii and bij are constant coefficient, linear coefficient, quadratic coefficient and interaction coefficient respectively Moreover, Xi and Xj represent independent variables. Defined factors and their corresponding code, units, and levels are shown in Table 1. The ranges of independent variables were selected based on literature review. Sodium hydroxide (NaOH) was varied from 8M to 19M whereas, Na_2SiO_3/NaOH ratio was ranged from 1 to 3.0 (Mustafa et al., 2012)., Design expert software was used to the design the experimental plan. As shown in Table 2, twenty mix designs of engineered geopolymer composite are randomly chosen according to the central composite design configurations for three factors with five centre points.

Table 1: Boundaries of Factors in RSM

Factor	Code	Unit	Level -∝	-1	0	+1	+∝
NaOH Molarity	X1	mol/litre	5.27	8	12	16	18.73
Na_2SiO_3/NaOH	X2	---	0.49	1.00	1.75	2.50	3.01
MK Fineness	X3	µm	33.34	63	75	150	179.66



a) Corner points b) Middle point and extended central-point c) CCD design
Fig. 1: Central Composite Design (CDD)

International Journal of Advances in Engineering and Management (IJAEM)
Volume 6, Issue 12 Dec. 2024, pp: 34-45 www.ijaem.net ISSN: 2395-5252

MIX PROPORTIONS OPTIMIZATION

Twenty mixes were made to study the effect of three different variables on compressive strength. These mixes were produced with alkaline solution to AMK ratio, sand to AMK ratio, gravel to AMK ratio, water to geopolymer solid ratio, extra water to AMK ratio and the AMK content. Details of these mixes are depicted in Table 2. The optimization for the mix proportions was according to the following factors:

1. Sodium Hydroxide Concentration
2. Ratio of Sodium Silicate to Sodium hydroxide of the alkaline solution. 3. Fineness (particles retained on sieve sizes 63, 75 and 150µm) of the AMK.

Table 2: Experimental Mix Proportions by Central Composite Design.

Run	Factor 1 Moles	Factor 2 Ratio	Factor 3 µm	MK (g)	Alk Sol (%)	Sand (%)	Gravel (%)	W/GS Ratio	Extra W (%)
1	12.00	1.75	75.00	400	0.35	0.63	0.42	0.23	0.05
2	12.00	3.01	75.00	400	0.35	0.63	0.42	0.23	0.05
3	12.00	1.75	75.00	400	0.35	0.63	0.42	0.23	0.05
4	16.00	2.50	63.00	400	0.35	0.63	0.42	0.23	0.05
5	12.00	1.75	33.34	400	0.35	0.63	0.42	0.23	0.05
6	12.00	1.75	75.00	400	0.35	0.63	0.42	0.23	0.05
7	16.00	1.00	150.00	400	0.35	0.63	0.42	0.23	0.05
8	16.00	2.50	150.00	400	0.35	0.63	0.42	0.23	0.05
9	8.00	2.50	63.00	400	0.35	0.63	0.42	0.23	0.05
10	12.00	1.75	179.66	400	0.35	0.63	0.42	0.23	0.05
11	12.00	1.75	75.00	400	0.35	0.63	0.42	0.23	0.05
12	18.72	1.75	75.00	400	0.35	0.63	0.42	0.23	0.05
13	16.00	1.00	63.00	400	0.35	0.63	0.42	0.23	0.05
14	8.00	1.00	150.00	400	0.35	0.63	0.42	0.23	0.05
15	8.00	1.00	63.00	400	0.35	0.63	0.42	0.23	0.05
16	12.00	1.75	75.00	400	0.35	0.63	0.42	0.23	0.05
17	12.00	1.75	75.00	400	0.35	0.63	0.42	0.23	0.05
18	5.30	1.75	75.00	400	0.35	0.63	0.42	0.23	0.05
19	12.00	0.49	75.00	400	0.35	0.63	0.42	0.23	0.05
20	8.00	1.00	150.00	400	0.35	0.63	0.42	0.23	0.05

Extra water is added to make W/GS ratio constant (if needed). Geopolymer Solid consists of (solids in NaOH, Na_2SiO_3 and Metakaolin).

Except Factors 1, 2 and 3, all other parameters quantities are in mass ratio of metakaolin. In all, 60No of geopolymer concrete cubes (100mm X 100mm X 100mm) were cast for the concrete laboratory experiments.

The experimental mix proportions by central composite design and experimental setup for the optimum mix design (no of specimens) are depicted in tables 2 and 3 respectively. The mixing and curing were carried out according to geopolymer concrete synthesis process depicted in figure 2.

International Journal of Advances in Engineering and Management (IJAEM)
Volume 6, Issue 12 Dec. 2024, pp: 34-45 www.ijaem.net ISSN: 2395-5252

Table 3: Experimental Setup for the Optimum Mix Design (No of Specimens)

S/no	Test/Curing Age	1	3	7	28	No of Samples
1	Consistency					
2	Paste Soundness					
3	Paste Setting Times initial					
4	Paste Setting Times final					
5	Slump of Fresh Concrete					
6	Dry Density	3	3	3	3	12
7	Water Absorption	3	3	3	3	12
8	Compressive Strength	3	3	3	3	12
9	Flexural Strength	3	3	3	3	12
10	Split Tensile Strength	3	3	3	3	12
11	Sorptivity	3	3	3	3	12
12	Acid Attack	3	3	3	3	12
	Total No. of Samples	21	21	21	21	**84**

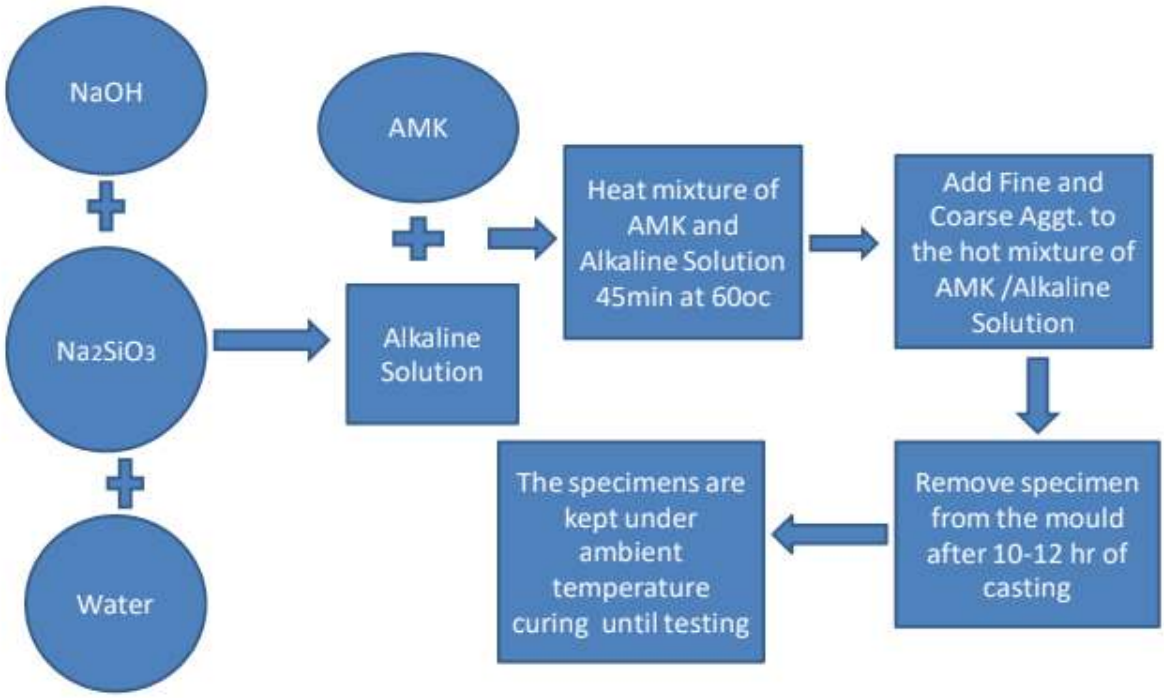


Fig.2: An adopted modified heat-cured geopolymer concrete synthesis process.
Source: Dibyendu Adak, et al., (2019).

Laboratory Evaluation of the Optimum Mix Proportion

The performance of the optimum mix proportion was evaluated by the laboratory experiments conducted on the geopolymer paste and concrete prepared as mentioned in table 3. All the tests were conducted in accordance to their relevant standard codes (ASTM/BS/EN) specifications.

International Journal of Advances in Engineering and Management (IJAEM)
Volume 6, Issue 12 Dec. 2024, pp: 34-45 www.ijaem.net ISSN: 2395-5252

III. TESTS RESULTS AND DISCUSSIONS

The influence of the three independent variables on the optimum mix proportion performance, namely sodium hydroxide concentration (Molarity), sodium silicate to sodium hydroxide ratio and fineness (particle retained after sieving the metakaolin through sieves size 63µm, 75µm and 150µm), were investigated on the fresh and mechanical properties of the Geopolymer Concrete and discussed in the following sections. The tests results are presented in figures and tables.

Optimization of Mix Proportions by Central Composite Designed

The optimization of the NaOH concentration (X_1), Na_2SiO_3/NaOH ratio (X_2) and Fineness (X_3) giving the maximum compressive is done by RSM. The details of the mixes and resulted compressive strength are presented in table 4.

Table 4: Actual Compressive Strength for the 20 Mix Proportion

Factors				Response
Run	F1 (Moles)	F2 (Ratio	F3(µm)	Compressive Strength (N/mm^2) at 28 day Age
1	12.00	1.75	75.00	38.6
2	12.00	3.01	75.00	36.8
3	12.00	1.75	75.00	38.6
4	16.00	2.50	63.00	35.8
5	12.00	1.75	33.34	37.0
6	12.00	1.75	75.00	38.6
7	16.00	1.00	150.00	36.8
8	16.00	2.50	150.00	34.4
9	8.00	2.50	63.00	35.8
10	12.00	1.75	179.66	34.7
11	12.00	1.75	75.00	38.6
12	18.72	1.75	75.00	37.4
13	16.00	1.00	63.00	36.6
14	8.00	1.00	150.00	35.4
15	8.00	1.00	63.00	34.6
16	12.00	1.75	75.00	38.6
17	12.00	1.75	75.00	38.6
18	5.30	1.75	75.00	33.6
19	12.00	0.49	75.00	37.2
20	8.00	2.50	150.00	34.8

The mix proportion with maximum compressive strength at 28 day obtained from average of 3No cubes of 100mm X 100mm X 100mm, cured at ambient temperature is recorded as the Optimum Mix Proportion (OMP). Hence RUNs 1, 3, 6, 11 16 & 17 from table 4.

Laboratory Evaluation Outcome

Table 5 presents the fresh and hardened properties of the AMK geopolymer concrete and that of Ordinary Portland Cement Concrete as control for comparism sake.

Table 5: Fresh and Hardened Properties of AMK GPC and OPC Concrete

S/No	Properties	AMK GPC	Cement Concrete
1	Paste Normal Consistency (%)	38.00	33.00
2	Paste Soundness (mm)	0.00	1.00
3	Paste Initial Setting Time (min)	Above 12 hrs	47
4	Paste Final Setting Time (min)	Exceed 24 hrs	557
5	Concrete Slump (mm)	82.00	96.00
6	Concrete Bulk Density (Kg/M^3)	2293.00	2396.00
7	Concrete Water Absorption (%)	1.8	3.2
8	Concrete Compressive Strength (N/mm^2)	38.60	25.80
9	Concrete Flexural Strength (N/mm^2)	6.20	4.90
10	Concrete Split Tensile Strength (N/mm^2)	4.40	3.80
11	Concrete Acid Attack (%)	2.7	3.20
12	Concrete Sorptivity ($mm/min^{0.5}$)	0.1262	0.2080

The Normal Consistency of the Optimum Mix

Normal consistency value of the 13.00M NaOH concentration, 1.5 Na_2SiO_3/NaOH ratios and 75µmAMK (Fineness) optimum mix is found to be 38%. This falls within the range recommended for OPC. An increase of AMK fineness in the mix, results in a reduction of the normal consistency value. The specific surface of the AMK is $14.88m^2/g$ and this material is non-plastic in nature. Owing to greater specific surface area of the AMK, a mix containing higher fraction of metakaolin, requires more amount of alkaline solution to lubricate the particle surface and it exhibits greater consistency value.

The Soundness of the Optimum Mix

The insignificant amounts of free lime and magnesia make the geopolymer binder sound, as it may not cause expansion in the hardened binder. The soundness value of the optimum mix geopolymer paste synthesized by alkalination of 75µm Alkaleri metakaolin (AMK) with 13.00M of NaOH concentration and Na_2SiO_3/NaOH ratio of 1.5 is assessed, by Le Chatelier mould technique. The geopolymer paste exhibited zero expansion value. Commonly, unsoundness of ordinary Portland cement is caused by the existence of free lime, magnesia and calcium sulphate in significant percentage. However, due to higher pozzolana content of raw materials, the available lime and magnesia get consumed in chemical reactions thus make the binder sound.

The Setting Times of the Optimum Mix

The initial setting time (IST) and final setting time (FST) of the optimum mix proportion samples were assessed by Vicat Apparatus test method. The sample displayed IS time of above 12 h and the FS time exceeds 24 h. This is owing to sluggish chemical reaction of metakaolin powder with time under an ambient room temperature. It is observed that an increase of the concentration of the alkaline solution results in a decrease of setting time. However, no significant reduction in setting times is noticed beyond 13.00M concentration.

A significant decrease of setting time is noted with 45mins at 60^{oc} heat cured. This tendency clearly indicates that at an ambient temperature metakaolin slowly reacts with NaOH solution in comparison to slag.

The Workability of the Optimum Mix

The workability of the optimum mix geopolymer concrete for this study was measured by means of slump test. The slump value of 82mm obtained is attributed to the alkali content, water to geopolymer solid ratio and the extra water in the mix. This slump value indicates slump of GPC increased in an almost linear relationship with the water to solids ratio. This trend is within expectation, as in the case of conventional cement concrete. The observed relationship for MK-based GPC is also consistent with fly ash-based GPC tested by Aliabdo et al. (2015). In comparison, cement concrete sample had a zero slump value for mix with water to cement ratio of 0.28. However, the mix was very dry. On the other hand, for the same amount of water to solids ratio, metakaolin-

International Journal of Advances in Engineering and Management (IJAEM)
Volume 6, Issue 12 Dec. 2024, pp: 34-45 www.ijaem.net ISSN: 2395-5252

based GPC showed better workability properties with the slump of 82mm for the water to solids ratio of 0.28. This could be due to low viscosity of sodium hydroxide solution as compared with sodium silicate solution. It is important to highlight that GPC workability is relying on the binder proportions (i.e., quantities of MK, NaOH, and sodium silicate),

The Bulk density of the Optimum Mix

The density of the GP mix was determined after setting at 3, 7, and 28 days. The average densities for three ages are 2396, 2308 and 2293 Kg/m^3 respectively. As can be seen from the values that the density of geopolymer mixes tend to be the highest when fresh (i.e., wet conditions). Reduction in density is observed after 7 days of setting, which is 3.7% less than the fresh density. Dry density at 7 days is observed to be almost the same as the density at 3 days but reduces at the curing age of 28 days. Dry density at 28 days falls within values of 2293kg/m^3 (+/- 5), which is less than the average wet density by 4.3%. Generally, density for geopolymer mix tends to decrease with the age of curing due to excess water evaporation.

The Optimum Mix Water Absorption

Water absorption result for the optimum mix is 4.9%. The water absorption generally tends to decrease or remain almost the same with the increasing amount of sodium silicate content. Water absorption of 5.2%, 4.9% and 4.7%, was observed for mixes with sodium silicate to NaOH ratio of 1.00, 1.75 and 2.50, respectively. Husain A. (2021) showed that the adoption of very low or very high alkaline solution to MK ratio contributes to the production of concrete with high water absorption percentages. The alkaline solution to MK ratio of 0.35 for the optimum mix has water absorption percentage of 4.9%, representing the middle value within the tested range. This may be attributed to the SiO_2/Al_2O_3 molar ratio and its implication on the process of geopolymerization and associated microstructure. Low SiO_2/Al_2O_3 molar ratio could results in a less dense structure while high SiO_2/Al_2O_3 molar ratio may result in impedance of the process of geopolymerization.

The Mechanical Strength of the Optimum Mix

Table 6 presents the mechanical strengths of the optimum geopolymer concrete mixes and the control concrete mix at 3, 7, and 28 days. The strengths of GPC are higher than that of control mix at all ages. It is observed that the concentration of alkali solution played a key role in strength gain. With the increase of the concentration from 8 to 13 M, the strength increased significantly. Thereafter, the strength reduces as the concentration of NaOH solution increased further from 13 to 16 M. The improvement in strength is mostly governed by the quantity of leached alumino-silicates from the source materials. In the polymer structure, the OH− ion from the alkaline solution plays as catalyst in the reaction process and stimulates the Si4+ and Al3+ ions dissolution from the source material. The mechanical strengths of the samples tested at 28 day with sodium silicate to NaOH ratios of 1.00, 1.75 and 2.50 achieved compressive strength of 37.4, 38.6, and 37.0 N/mm^2, respectively. However, increasing the ratio to 3 resulted in a strength drop to 37.0 as shown in table 4. This is because the higher amount of the reactive silica contributed to the better density of Si-O-Si bonds. This trend has also been reported in the literature as a result of the impedance of geopolymerization caused by Al-Si precipitation in which the excess sodium silicate inhibits water evaporation and structure formation. It is observed that the compressive strength of geopolymer concrete increases with increase in fineness of metakaolin powder. This might be due to most of the particle of metakaolin powder goes under polymerisation and smaller particles fill up the voids present in fine aggregate. Also it is seen that there is no abrupt change in strength of geopolymer concrete for 7 and 28 days test after curing. It clearly shows that the fineness of metakaolin powder plays vital role in developing strength of geopolymer concrete in a short period of time.

Table 6: Mechanical Strength Properties of the Optimum Mix Concrete

Mix	Compressive Str. (N/mm^2)	Split Tensile Str. (N/mm^2)	Flexural Str. (N/mm^2)
GPC	38.6 (28d)	4.4 (28d)	6.2 (28)
	29.7 (7d)	3.1 (7)	4.6 (7)
	15.8 (3d)	------	-------
CC	25.8 (28)	2.1 (28d)	3.7 (28)

Durability Performance of the Optimum Mix

The results of water absorption, acid attack and sorptivity for the optimum mixes are presented in table 7.The water absorption reduces with reduction in fineness (particle size) of the metakaolin powder. These results indicated that the water absorption is much affected by the extra water added into the mixture and metakaolin fineness since they increase capillary porosity and permeable void of concrete. As shown in table 7, water absorption with Water to binder ratio of 0.28 was significantly higher than those for other geopolymer mixes. Geopolymer concrete uses a very small amount of water in the mixture. The additional extra water is useful to achieve a particular strength. However, a relatively higher water/binder ratio and larger particle size produce a weaker and pervious matrix, leading to higher capillary porosity and in turn responsible for the increase in water absorption and permeable void of geopolymer mixes. Also, high amount of sodium silicate in geopolymer mix is vulnerable since more channels in the concrete will be created during oven curing process. Table 7 presents sorptivity values for both cement and geopolymer concretes. As shown in the table, the results indicated that the sorptivity was significantly lower for all the geopolymer mix than for the control mix. For all geopolymer mixes, the sorptivity reduces with a decrease of water content, sodium silicate content and better grading distribution. It is revealed that the water/binder ratio has significantly influenced the sorptivity as per water absorption and permeable voids. This suggests that the increase of amount of water resulted in high porosity concrete, hence increases capillary suction. Geopolymer concrete exhibits superior durability in acid solution compared to cement concrete. This is evident in table 8, as can be seen; mass lost by geopolymer concrete mix is lesser than cement concrete mix by about 31% of the cement concrete lost. Table 9 presents the fresh and hardened properties of the AMK geopolymer concrete and that of Ordinary Portland Cement Concrete as control for comparism sake.

Table 7: Durability Performance of the Optimum Mix after 28thday Immersion

Mix	Water Absorption (% mass gain)	Acid Attack (% mass loss)	Sorptivity (mm/min$^{0.5}$)
Geopolymer Concrete	1.8	2.7	0.1262
Cement Concrete	3.2	3.9	0.2080

IV. CONCLUSION

Based on the data obtained from this study and relevant literatures, the following principal conclusions were drawn:

1. Response surface method (RSM) via central composite design (CCD) was used to acquire the optimum mix proportion.
2. The RSM can be used to design experiment for Engineered Geopolymer Composite (EGC) mixtures for any target value of response from the possible variable combination of factors. 3. Compressive strength of the AMK geopolymer concrete increases with increase in fineness of the metakaolin, while molarity of sodium hydroxide (NaOH) solution increases to a certain limit.
4. The workability of freshly mixed geopolymer concrete increases with increasing fineness of the AMK powder due to reduction in internal friction between aggregates. Workability can also be increased by increasing alkaline solution ratio.
5. It is also seen from the densities of the AMK geopolymer concrete that higher fineness of metakaolin powder gives more dense concrete because voids get filled by finer particles of the powder.
6. Generally, the durability properties values are of AMK geopolymer concrete are lower than Cement Concrete ones.

REFERENCES

[1]. Aaron P, W (2015). Establishing a Mix Design Procedure for Geopolymer Concrete. A dissertation submitted to the Faculty of Health, Engineering and Sciences, University of Southern Queensland.

[2]. Abdulrahman Albidah, Mohammed Alghannam, Husain Abbas, Tarek Almusallam, Yousef Al-Salloum (2020)* Characteristics of metakaolin-based

International Journal of Advances in Engineering and Management (IJAEM)
Volume 6, Issue 12 Dec. 2024, pp: 34-45 www.ijaem.net ISSN: 2395-5252

geopolymer concrete for different mix design parameters. Journal of Materials Research and Technology.

[3]. Abdel-Gawwad, H. A., & Abo-El-Enein, S. A. (2016). A novel method to produce dry geopolymer cement powder. HBRC Journal, 12(1), 13–24.

[4]. Abdul Aleem. M. I, and Arumairaj P. D. (2012). Geopolymer Concrete- A review. International Journal of Engineering Sciences & Emerging Technologies. ISSN: 2231 – 6604 Volume 1, Issue 2, pp: 118-122 ©IJESET.

[5]. ACI 211.4R-93 (1998). Guide for Selecting Proportions for High-Strength Concrete with Portland cement and Fly Ash. Report by ACI Committee 211.

[6]. Aliyu, I., Kaura, J. M., Abubakar, I. and Ocholi, A. (2019) Metakaolin Based Geopolymer Concrete Mix Design Suitable for Reinforced Concrete Use. FUDMA Journal of Sciences (FJS) Vol. 3 No. 4, December, 2019, pp 403 –411

[7]. A.M. Ibrahem and A.A. Wahab, "Effect of Temperature on the Pozzolanic Properties of Metakaolin Produced from Iraqi Kaolin Clay". AL- Fatih Journal, Diyala University, Iraq, Vol. 4, Issue 32, 2008, pp. 268- 285.

[8]. Antoni, M.; Rossen, J.; Martirena, F.; Scrivener, K. (2012). Cement substitution by a combination of metakaolin and limestone. Cement Concrete Resource 42, 1579–1589.

[9]. Aroke, U., El-Nafaty, U., & Osha, O. (2013). Properties and Characterization of Kaolin Clay from Alkaleri, International Journal of Emerging Technology and Advanced Engineering, 3(11), 387–392.

[10]. ASTM C 109, 2001. Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in or [50 mm] Cube Specimens). ASTM C 109M-99, Annual Book of ASTM Standard 04.01, pp: 83-88.

[11]. Bashar, Alengaram, Jumaat, and Islam, (2014). "The effect of variation of molarity of alkali activator and fine aggregate content on the compressive strength of the fly ash: palm oil fuel ash based geopolymer mortar," Advances in Materials Science and Engineering, vol. 2014,Article ID 245473, 13 pages,.

[12]. Basil S. Al-Shathr, Tareq S. Al-Attar, and Zaid A. Hasan (2015) Optimization of Geopolymer Concrete Based on Local Iraqi Metakaolin. The 2nd International Conference of Buildings, Construction and Environmental Engineering (BCEE2-2015)

[13]. Campbell, G.M. & Detwiler, R.J (1993). Development of mix designs for strength and durability of steam-cured concrete. Concrete International: 37-39. July 1993.

[14]. Chandra K.P and B. Sarath C.K.B (2017). An Experimental Study on Metakaolin and GGBS Based Geopolymer International Journal of Civil Engineering and Technology (IJCIET) Volume 8, Issue 1, pp. 544–557.

[15]. Das and Dimas, (2020). An Overview of Current Research Trends in Geopolymer Concrete. International Research Journal of Engineering and Technology (IRJET) 05(11): 376-381

[16]. Dibyendu Adak, Ph.D., A.M.ASCE1; and Saroj Mandal, Ph.D. (2019) Strength and Durability Performance of Fly Ash–Based Process- Modified Geopolymer Concrete © 2019

[17]. Davidovits, J. Properties of geopolymer cements (1994). 1st International Conference on Alkaline Cements and Concretes (Krivenko, ed.). Kiev, Ukraine, 1: 131-149. 1994

[18]. Davidovits, J. High alkali cements for 21st century concretes. In Concrete Technology, Past, Present and Future. Proceedings of V Mohan Malhotra Symposium. Mehta, K. (ed.). ACI SP. 1994.

[19]. Ephraim T. K (2016). Effects of different Molarities of sodium hydroxide and Curing Regimes on Metakaolin based Geopolymer Concrete. Unpublished MSc Dissertation submitted to the Department of Building, Faculty of Environmental Design, Ahmadu Bello University, Zaria.

[20]. Ferdosian, I., Cam~oes, A., (2017). Eco-efficient ultra-high performance concrete development by means of response surface methodology. Cement Concrete. Composite. 84, 146-156

[21]. Ghafari, E., Costa, H., Júlio, E., 2014. RSM-based model to predict the performance of self compacting UHPC reinforced with hybrid steel micro-fiber. Constr. Build. Mater. 66, 375- 383.

[22]. Hall, C. Water sorptivity of mortars and concretes: a review. Magazine of Concrete Research. 1989. 41: 51-61.

[23]. H. Kamarudin and A.K. Omar, "The Effect of Alkaline Activator Ratio on the Compressive Strength of Fly Ash-Based Geopolymer" Australian Journal of Basic and Applied Sciences, ISSN 1991-8178, 2011, pp. 1916-1922.

[24]. Hardjito, D. and Rangan, B.V. (2005), "Development and properties of low calcium fly ash based geopolymer concrete", Research Report GC-1, Curtin University of Technology, Perth, Australia.

[25]. Jacob E A., (2017). Evaluation of the properties of Metakaolin based Geopolymer Concrete made with Recycled concrete Aggregate. Unpublished MSc Dissertation submitted to the Department of Building, Faculty of Environmental Design, Ahmadu Bello University, Zaria

[26]. Khan, M.Z.N., Hao, Y., Hao, H., Shaikh, F.U.A., 2018. Mechanical properties of ambient cured high strength hybrid steel and synthetic fibers reinforced geopolymer composites. Cement Concr. Compos. 85, 133e152.

[27]. Matawal, D., S., Opeyemi Joshua and Olusola Kolapo. (2018), Development of a Fully Pozzolanic Binder for Sustainable Construction: Whole Cement Replacement in Concrete Applications. International Journal of Civil Engineering and Technology, February 2018.

[28]. Nematollahi, (2015). Matrix design of strain hardening fiber reinforced engineered geopolymer composite. Compos. Part B Eng. 27, 253-265

[29]. N. Martin. (2004). "Role of supplementary cementing materials and super-plasticizers in reducing greenhouse gas emissions." In Proc., ICFRC Int. Conf. on Fiber Composites, High Performance Concrete, and Smart Materials, 489–499.

[30]. Patil G.S and K. Manojkumar, "Factors Influencing Compressive Strength of Geopolymer Concrete" International Journal of Research in Engineering and Technology, 2013, pp. 372 – 375.

[31]. Rao, G. M., & Rao, T. G. (2015). Final setting time and compressive strength of fly ash and GGBS-based geopolymer paste and mortar. Arabian Journal for Science and Engineering, 40(11), 3067–3074.

[32]. Wong and Lam, (1997). The influence of different curing conditions on the pore structure and related properties of fly-ash cement pastes and mortars. Construction Building Material, 11: 383-393.

[33]. Zahid, M., Shafiq, N., Nuruddin, M.F., Nikbakht, E., Jalal, A., 2017. Effect of Partial Replacement of Fly Ash by Metakaolin on Strength Development of Fly Ash Based Geopolymer Mortar, in Key Engineering Materials. Trans Tech Publications, Switzerland, pp. 131-135

[34]. Zhang H.Y., Kodur V., Qi S.L., Cao L. and Wu B. (2014). Development of metakaolin–fly ash based geopolymers for fire resistance applications. Journal of Construction and Building Materials, 55, pp. 38–45.