

Effect of Calcination Temperatures of Kaolin on Compressive and Flexural Strengths of Metakaolin-Concrete

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ABSTRACT: The incorporation of pozzolanic materials in concrete construction is progressively increasing. This is due to technological advancement and climate change problems associated with carbon emissions resulting from the large-scale manufacturing of cement and its usage for concrete production. In this study, metakaolin obtained was used to partially substitute cement in metakaolin-concrete. Calcination temperatures of kaolin were varied from 500°C to 800°C at an interval of 100°C for 60 minutes. The metakaolin obtained was used to partially replace cement at 0, 5, 10, 15, 20 and 25 % by weight using a mix ratio of 1:2:4 and 0.4 water-cement ratio. Compressive strength test was carried out at curing ages of 7, 28 and 90 days, while the flexural strength test was performed at curing ages of 28 and 90 days. For both compressive and flexural strengths, 15 % by weight replacement with metakaolin gave the best strength values at all temperatures. An Increase in temperature led to a significant increase in the strength of metakaolin-concrete. ANOVA showed all factors significantly affected the flexural strength ($P < 0.1$), whilst the calcination temperature was significant ($P < 0.1$) to the compressive strength. This study showed that metakaolin is a supplementary cementitious material (SCM) and is a potential alternative to cement and can be used in the construction industry. Also, the calcination temperature of kaolin has a significant effect on the properties of the resulting metakaolin-concrete produced from it.

KEYWORDS: kaolin, metakaolin concrete, ANOVA, compressive, flexural strengths.

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I. INTRODUCTION

One of the world's leading industries is the cement manufacturing sector. The history of human dependence on cement is as old as civilization. In 2015, three billion metric tons of cement was manufactured worldwide and this accounted for a 6.3 percent growth rate per annum (Tamanna *et al*, 2020). During cement manufacture, approximately 800 to 1000 kg of CO₂ emissions occur per ton of cement. It is estimated that 5 to 8 percent of global man-made CO₂ emissions are produced through cement manufacture (Tosti *et al*, 2018; Teixeira *et al*, 2019) and has contributed to global climate change. Seasons of drought and excessive flooding brought on by climate change globally have hampered agricultural activity and resulted in a loss of shelter. In Nigeria, the National Emergency Management Agency (NEMA) reported the devastating aftermath of the flood to lives and livelihoods in the country (Davies, 2022). One major use of cement in structural works is the production of concrete for use in the housing and other infrastructure sectors.

II. BACKGROUND OF STUDY

A vital basic necessity for every person is housing, much like food and clothing (Fasakin *et al*, 2019). It is essential for human comfort, survival, and health (Okafor *et al*, 2019). As a result, one of the greatest ways to determine someone's level of living and status in society is through their house. Shelter, according to Kehinde (2010), is essential to human survival. Housing entails having access to the property, a place to live, and the facilities needed to make that place safe, sanitary, convenient, and visually beautiful. Therefore, filthy, unhealthy, hazardous, and insufficient housing can impact a person's privacy, security, and physical health. The housing industry's performance is frequently used as one of the benchmarks to gauge a country's overall health (Olawale *et al*, 2015; Charles, 2003). The housing shortage in Nigeria is a result of factors, such as poverty, a rapid urbanization rate, expensive building material costs, and outdated construction technologies (Kabir, 2004). In achieving innovative infrastructure, the improvement and use of new materials, consciously designed with affordability as a principal goal, will help to improve structural decay, mitigate environmental risks, and boost the economy of every developing country like Nigeria (Mukherjee and Vesmawala, 2013).

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Furthermore, pressure to minimize cement usage through the use of supplemental materials has been generated by environmental concerns resulting from the high energy cost and CO₂ emission connected with cement manufacturing. (Sabir *et al*, 2001; Mindess *et al*, 2003). Potential supplementary materials, mostly pozzolans include fly ash, metakaolin, silica fume, and rice husk ash. Pozzolans can be of natural or industrial origin (Batis *et al*, 2005). Metakaolin, one of the supplementary cementitious materials obtained from the dehydroxylation of kaolin and commercially available in Nigeria was employed for use in this study. Khatib *et al* (2012) researched high-volume metakaolin as cement replacement in mortar. It was shown that using metakaolin in place of cement at a rate of 20% produced the greatest improvements in the pore refinement of pastes, whereas using metakaolin at a rate of 30% or more lowered compressive strength. Sabria *et al* (2010) aimed at improving the rheological behaviour of cement paste with metakaolin and found that replacement at 10% by weight and 15% by weight of metakaolin showed better viscosity, shear stress, and improved cement paste flow-ability. According to Kannan and Ganesan (2012), who studied the strength and absorption characteristics of ternary blended cement mortar using rice husk ash and metakaolin, an increase in compressive strength of 20.9% was observed at 15% by weight replacement of rice husk ash, 17.42% at 25 % by weight replacement of metakaolin and 24.61% at 30% by weight replacement of the two materials combined in equal ratios (1:1). This study aims at investigating the mechanical properties and suitability of kaolin calcined at varying temperatures used in concrete production. This has helped to tap into the available mineral resources for the development of effective and environmentally friendly alternative building materials for construction purposes and the encouragement of investors in the area.

III. MATERIALS AND METHODS

A. Materials

Kaolin was obtained from Imeko (Lat. 7° 46' 56" N, Long. 2° 84' 78" E), located at Imeko-Afon Local Government Area in Ogun State, Nigeria. Its geological characteristics include accessible and well-connected roads and footpaths. Imeko township is located in southwest Nigeria's basement complex terrain. The thickness of the sedimentary rock formation is noticeable. The most common rock types are a range of hard to extremely hard rocks that have undergone varying degrees of metamorphism into migmatites and gneisses and have been invaded by granites (Olabode and Mohammed, 2016).

B. Methods

1) Calcination and Chemical Composition Test

The kaolin was subjected to varying temperatures from 500°C to 800°C at an interval of 100°C for 60 minutes. After heating, the samples were cooled to room temperature at ambient conditions to avoid crystallization of amorphous metakaolin. Chemical analysis through Atomic Absorption Spectroscopy was carried out on the raw kaolin and each of

the calcined samples to determine the percentages of the chemical compounds present.

2) Compressive and Flexural Strength Tests

The calcined kaolin (metakaolin) was used to replace cement at 0 % (control), 5%, 10%, 15%, and 20% by weight of the binder, which is ordinary Portland cement or simply cement. A mix ratio of 1:2:4 (cement: sand: coarse aggregate) and a water-cement ratio of 0.4 was employed in all the mixes. For the Compressive strength test, two hundred and sixty-two cubes of size 150 mm × 150 mm × 150 mm were cast and cured in water for 7, 28 and 90 days. The compressive strength test procedure was in accordance with BS EN 12390-3 (2019). The compressive strength was estimated using Eqn. (1):

$$P = F/A$$

(1)

Where; P is the compressive strength (N/mm²); F is the applied load (N); A is the area of the concrete cube (mm²).

To determine the flexural strength, two hundred and sixty-two beams of size 150 mm × 150 mm × 750 mm were cast and also cured in water for 28 and 90 days. The flexural strength test procedure was in accordance with BS EN 12390-5 (2019). At each data point, three repeat samples were cast and tested throughout the experiment. The maximum tensile stress, which is the modulus of rupture attained at the outermost fibre of the beam under tension was estimated using Eqn. (2):

$$F = PL/bd^2$$

(2)

where; F is the flexural strength of modulus of rupture (N/mm²); P is the applied load (N); L is the length of the beam sample (mm); b is the breadth of the beam sample (mm); d is the depth of the beam sample (mm).

3) Statistical Analysis

Regression analysis was carried out to determine the effect of the percentage replacement of the metakaolin, calcining temperature, and the curing time on the compressive strength of cast cubes and flexural strength of cast beams. The regression analysis considered linear and interaction terms and utilized a backward elimination to remove terms that affect the prediction of the properties. The regression analysis was carried out at a level of confidence of 90%. Analysis of Variance (ANOVA) was carried out to determine the factors that were statistically significant to the mechanical properties (Compressive strength and flexural strength) of metakaolin concrete.

The significance of the interactions of the factors was also determined using ANOVA. Main effects plots were used to determine the effects of the factors on the compressive and flexural strengths of metakaolin concrete. Regression models were developed to predict the compressive and flexural strength of metakaolin concrete in terms of the percentage replacement of cement with metakaolin, calcining temperature, the curing time and interactions between these factors.

4) *Microstructure of Metakaolin-Concrete*

A scanning electron microscope (Phenom ProX Desktop SEM) operating at 10 kV was utilized in the study to examine the surface morphology of metakaolin-concrete samples cured at 28 days. Metakaolin samples calcined at 500, 600, 700 and 800C for 60mins were used to produce the metakaolin-concrete samples. Scanning Electron Microscope (SEM) images with 100× magnification level were used to study the surface morphology of the metakaolin-concrete samples.

IV. RESULTS AND DISCUSSION

A. *Results*

1) *Physical Properties and Chemical Composition of Metakaolin*

The physical properties are presented in Table 1. The chemical composition test on the raw kaolin (untreated) showed that the percentages of SiO₂, Al₂O₃ and Fe₂O₃ were 48.50, 32.75 and 4.28 respectively as reported by Abiodun *et al.* (2019). The result of the chemical composition of calcined kaolin (Metakaolin) at different temperatures and a duration of 60 minutes is presented in Table 1.

Table 1: Physical Properties and Percentage Chemical Composition of Metakaolin

Physical properties of Metakaolin	
Appearance	Off white powder
Texture	Fine
Odour	Odourless
Percentage Chemical Composition of Metakaolin	
Oxides (%)	Temperature at 60 minutes
	500°C 600°C 700°C 800°C
SiO ₂	48.62 48.47 50.46 53.49
Al ₂ O ₃	31.45 33.67 35.56 39.90
Fe ₂ O ₃	4.08 2.97 2.27 0.52
CaO	2.24 2.32 1.63 0.12
MgO	0.86 1.46 2.42 0.21
Na ₂ O	0.28 0.18 0.28 0.11
K ₂ O	1.21 0.74 0.74 0.53
TiO ₂	1.04 1.21 0.99 0.54
SO ₃	2.26 2.02 1.49 0.01
LOI	11.55 11.04 9.38 4.51

2) *Compressive strength*

The effect of calcining temperatures and curing days on the compressive strengths of metakaolin-concrete at different curing days is shown in Figure 1.

Different statistical plots were used to check that the assumptions of ANOVA are not violated as shown (Figure 2).

Figure 2(a) is the normal probability plot, Figure 2(b) shows the residuals versus fits and Figure 2(c) shows that the independence assumption for ANOVA is not violated. The ANOVA table for the Compressive strength of metakaolin concrete is presented in Table 2. The main effects plot showing the relationship between the percentage of metakaolin replacement, the curing days, and the calcining temperature on the compressive strength of metakaolin-concrete is presented (Figure 3).

The Pareto chart for standardized impacts on metakaolin-concrete's compressive strength is shown (Figure 4). The regression equation generated for the compressive strength in terms of the percentage replacement of metakaolin (*M*), curing time in days (*D*) and the calcining temperature (*T*) using the terms in the ANOVA in Table 2 is presented in Eqn. (3) with a coefficient of determination (*R*²) of 41.84%.

The predictions from the equation are compared with experimental data as presented (Figure 5). Due to the low value of *R*², the model predicts the trend of the experimental data but there is a large deviation from the experimental values.

$$Compressive\ Strength = 11.15 - 0.0272M + 0.0095D + 0.01704T + 0.00197MD \quad (3)$$

3) *Flexural strength*

The result of the flexural strength test is presented (Figure 6) and the failure mode of the unreinforced metakaolin-concrete beams subjected to the flexural strength test is shown (Figure 7).

The assumptions for ANOVA were checked using the plots (Figure 8). The ANOVA for the flexural strength is presented in Table 3.

The main effects plot showing the relationship between the percentage of metakaolin replacement, the curing days, and the calcining temperature on the flexural strength of metakaolin-concrete is presented (Figure 9). The Pareto chart for standardized impacts on metakaolin-concrete's flexural strength is shown (Figure 10).

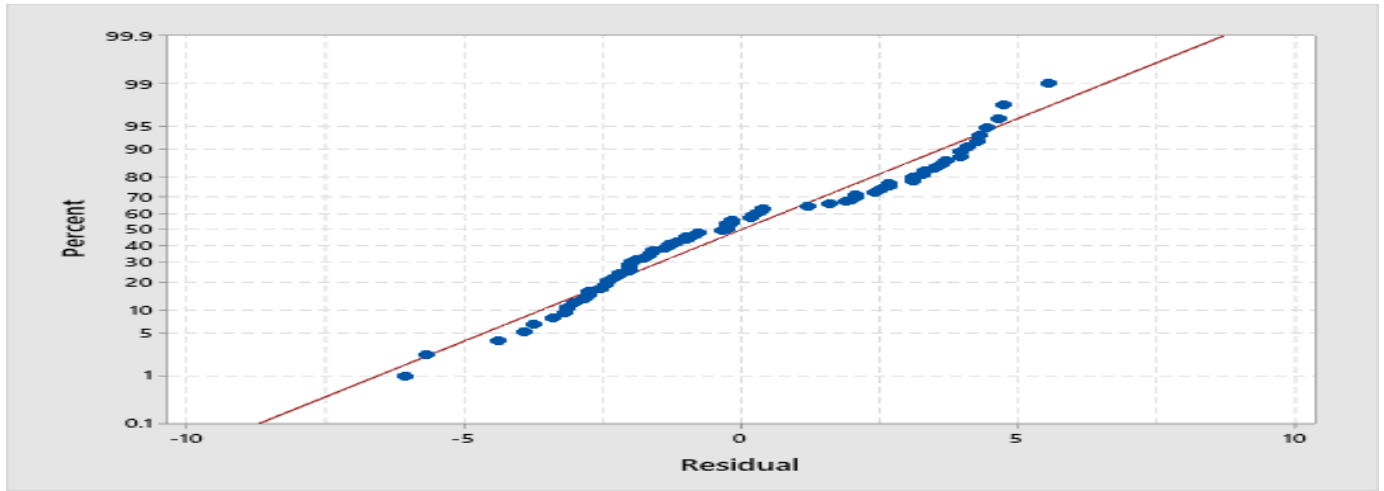
The regression equation which describes the relationship between the flexural strength and the factors identified in the ANOVA in Table 3 is shown in Eqn (4). The regression equation has *R*² of 83.73%.

The comparison of the predicted values for the flexural strength compared with the experimental data obtained is presented (Figure 11).

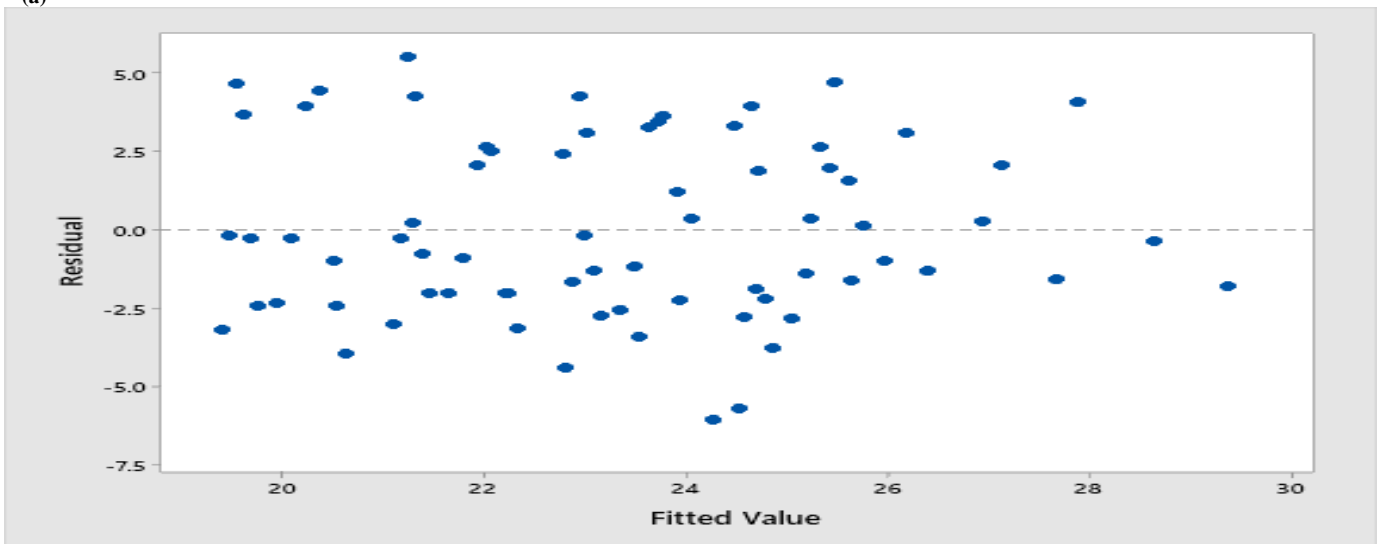
$$\text{Flexural Strength} = 0.951 - 0.1229M + 0.01452D + 0.00521T + 0.000266MT \quad (4)$$

4) *Microstructure of Metakaolin-Concrete*

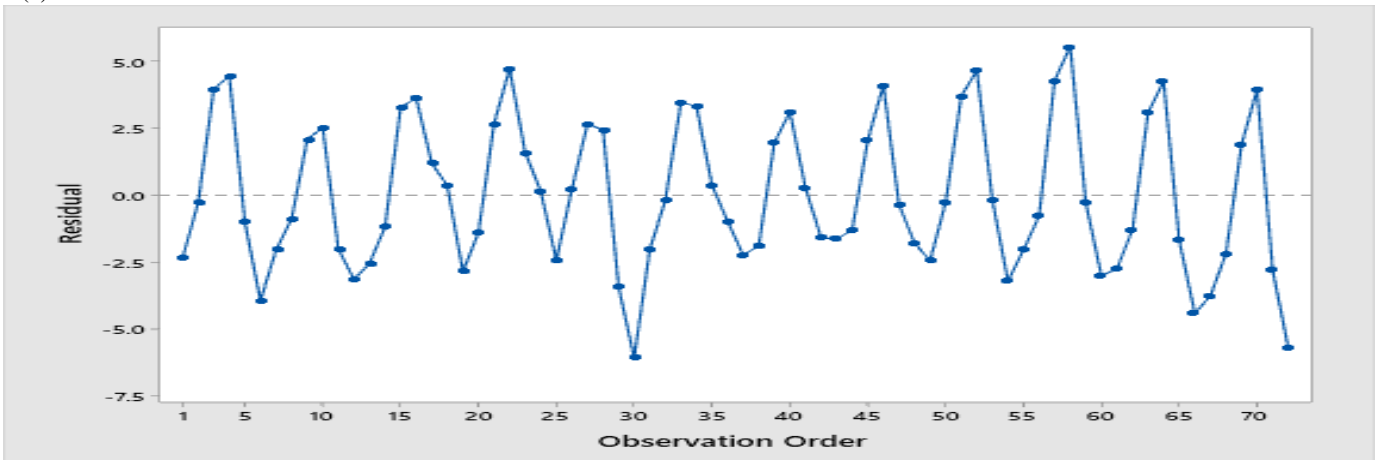
The microstructures of the metakaolin-concrete produced from metakaolin calcined at 500°C -800°C and cured at 28 days are presented (Figure 12). The micrographs were obtained using scanning electron microscopy (SEM).



(a)



(b)



(c) Figure 2: (a) Normal Probability plot (b) Residual in relation to fitted values plot (c) Residual in relation to observation order plot for compressive strength of metakaolin-concrete

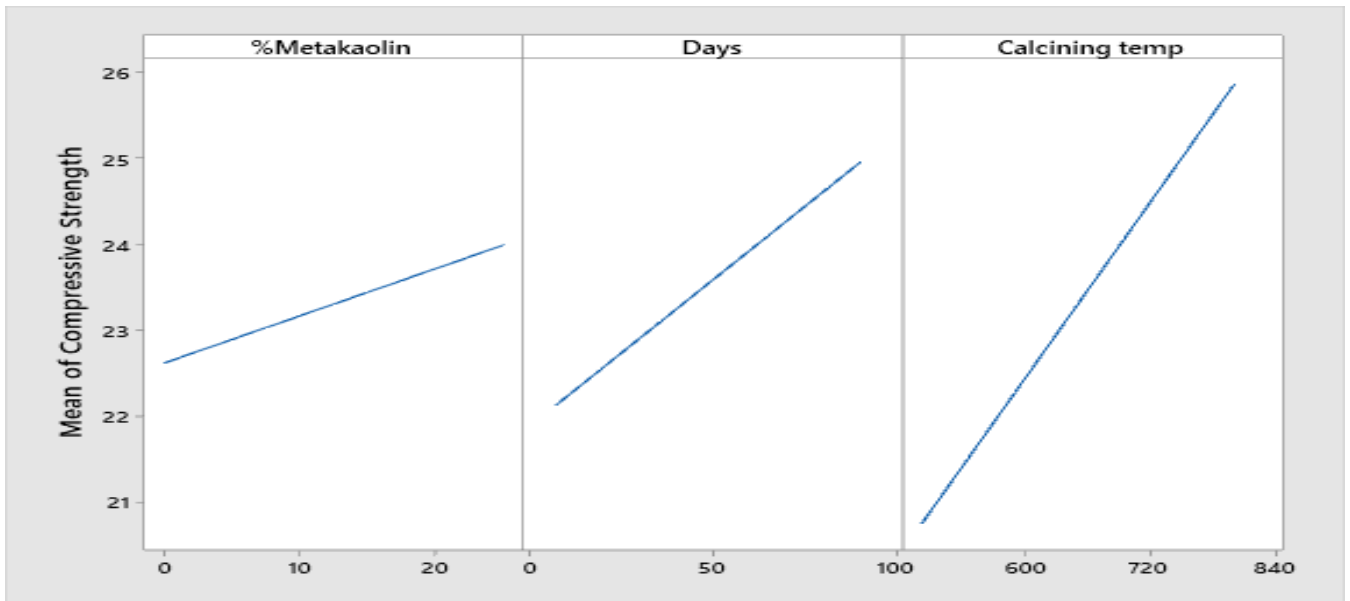


Figure 3: Relationship of main effects on the compressive strength of metakaolin concrete

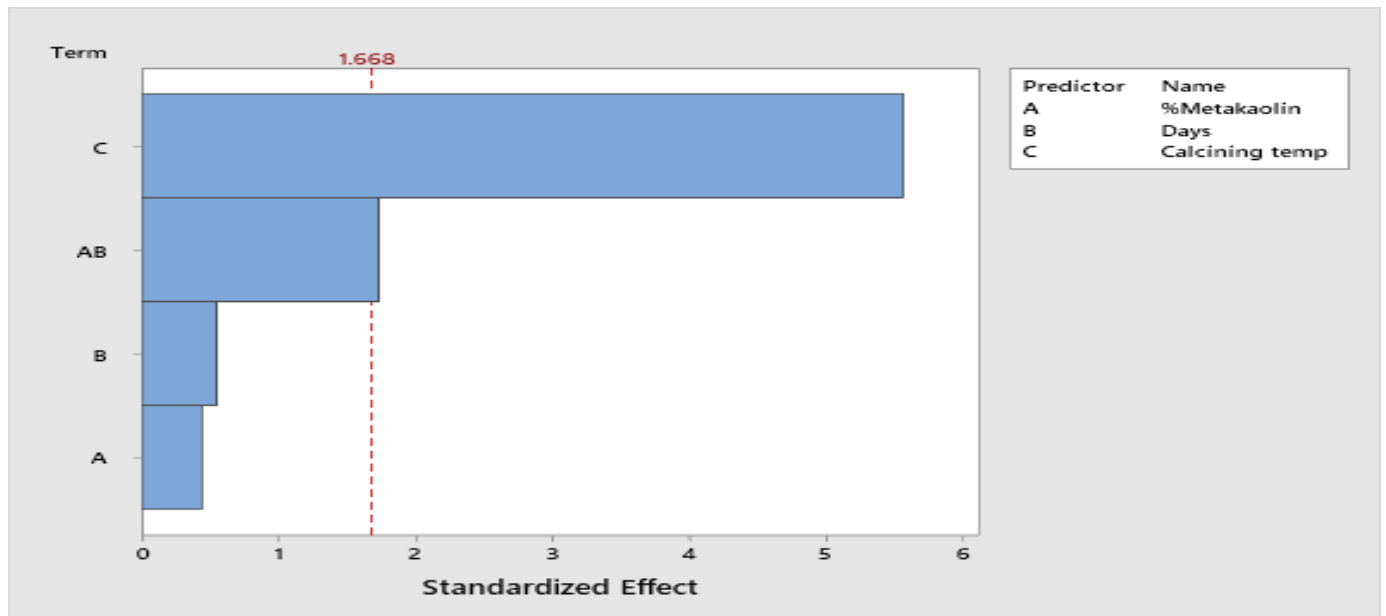


Figure 4: Pareto chart for standardized effects of compressive strength

B. Discussion

1) Physical properties and Chemical composition test of Metakaolin

The visual assessment of the metakaolin showed that it was off white in colour, platy, odourless and have a fine texture. According to Sabir *et al* (2001), the pozzolanic reactivity of kaolin is significantly influenced by the burning or calcining temperature. As presented in Table 1, it was noted that while the Loss on Ignition (LOI) at temperatures of 500°C and 600°C were higher than the maximum value (10%) recommended by ASTM C618-12a (2012), that of 800°C was far less (Shetty and Jain, 2019). The cumulative content of silica, alumina, and ferric oxides present at 500°C, 600°C, 700°C, and 800°C were 84.15%, 85.11%, 88.29%, and 93.91%, respectively.

This shows that the degree of disorder of metakaolin is determined by the calcination temperatures, which also affects how reactive the mineral is chemically (Kakali *et al*, 2001; Abiodun *et al*, 2020). Hence, the metakaolin samples obtained after calcination are classified as Class N pozzolans according to ASTM C618-12a (2012).

2) Compressive strength

With an increase in calcining temperature, the compressive strength increased. Because metakaolin contains a significant quantity of calcium silicate hydrates (CSH), a substance that increases strength, it was found (Figure 1) that as the percentage replacement with metakaolin grew up to 15% by weight replacement, the strength likewise increased.

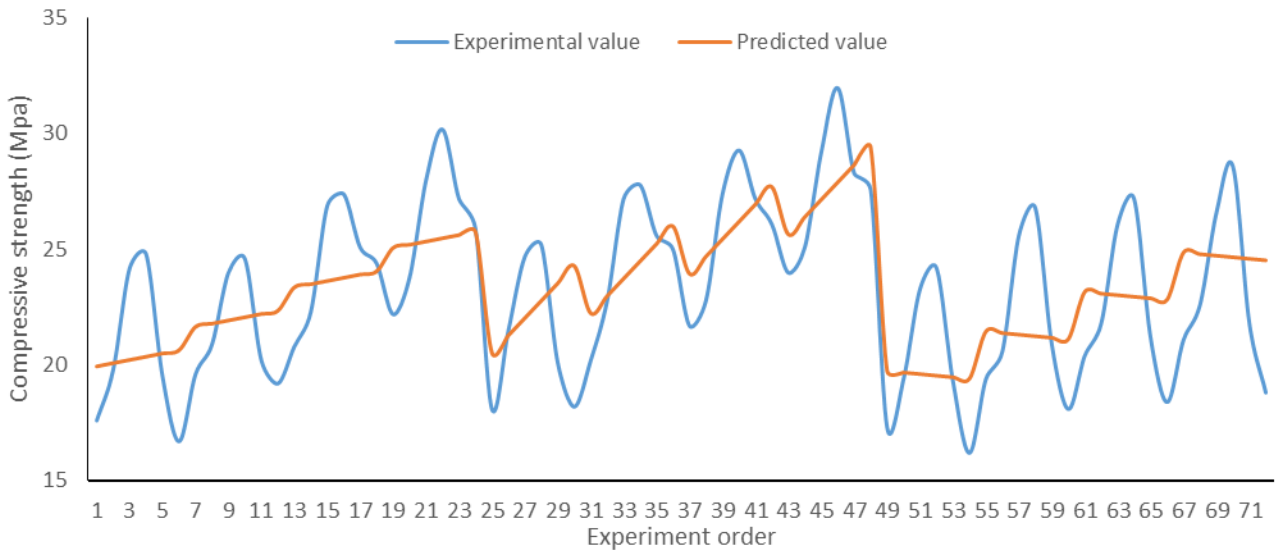


Figure 5: Comparison of predicted and experimental values for compressive strength of metakaolin concrete

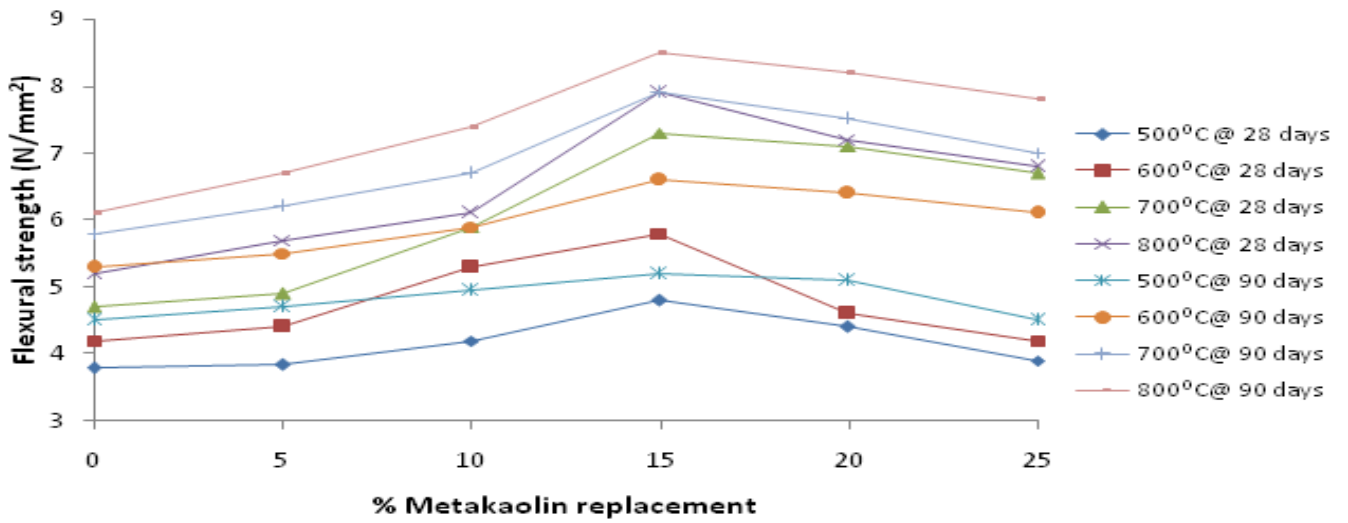


Figure 6: Effect of calcination temperature and curing days on flexural strength

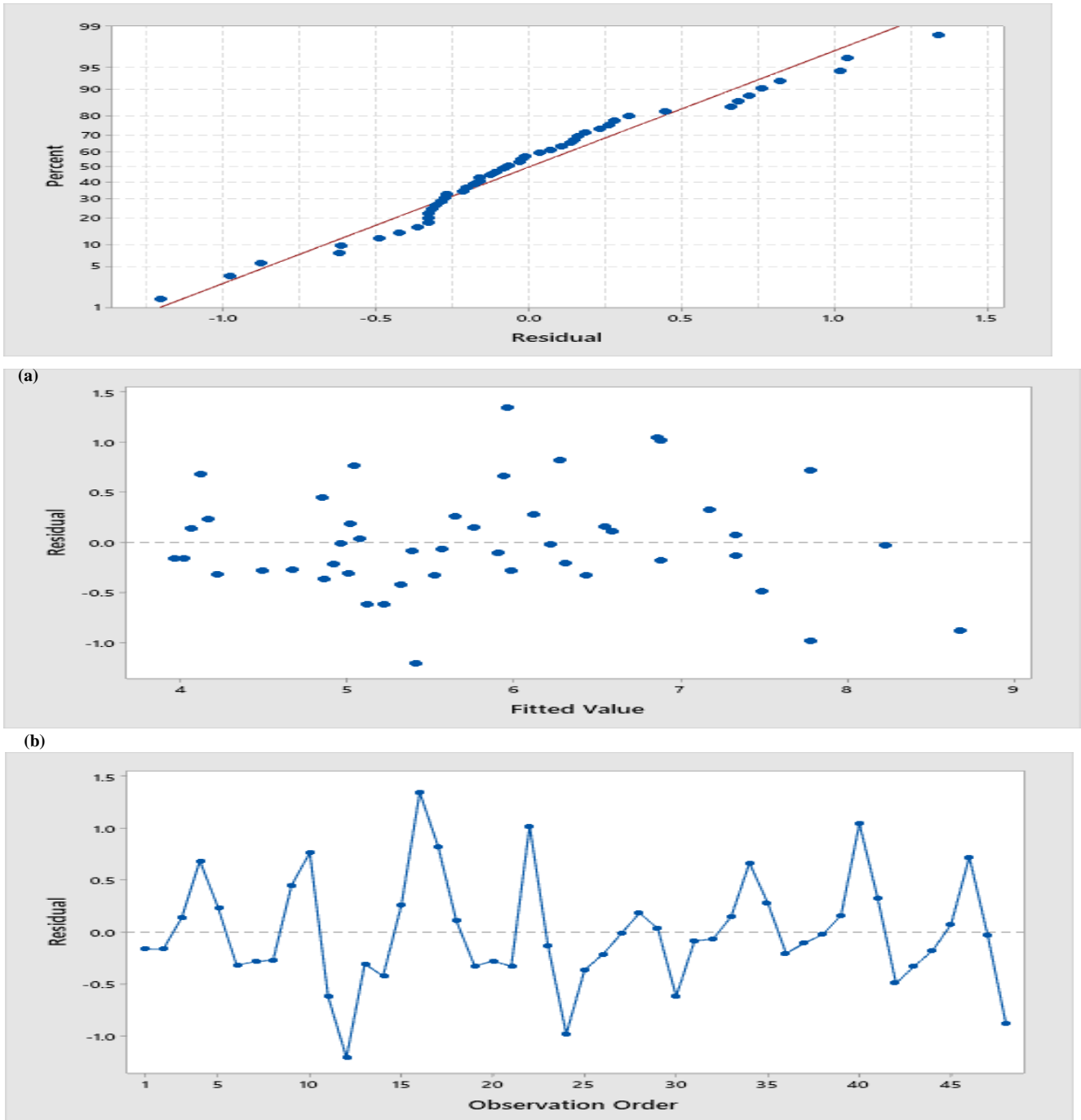


Figure 7: Failure mode of the unreinforced concrete beams containing metakaolin

Compressive strength declined above a replacement level of 15% with metakaolin.

Figure 2(a) shows that the population in each treatment is normally distributed since the plotted points can be fitted with a straight line. Also, Figure 2(b) reveals that the constant variance assumption is met since the plotted points are scattered with a pattern. Figure 2(c) shows that the independence assumption for ANOVA is not violated since there is no defined pattern in the plot.

From Table 2, it was observed that the calcining temperature was statistically significant ($P < 0.1$). The interaction between metakaolin and the curing time was also significant at a level of significance of 10%. The main effects plot (Figure 3) shows that the percentage of metakaolin replacement, the curing days, and the calcining temperature have a positive influence on strength of metakaolin concrete. The compressive strength increased with an increase in either one of these variables. The calcining temperature has the most impact on the compressive strength of the concrete, as can be seen from the Pareto chart for standardized impacts on metakaolin concrete's compressive strength (Figure 4).



(c)
Figure 8: (a) Normal Probability (b) Relationship of Residual to Fitted values (c) Relationship of Residual to observation order for flexural strength of metakaolin-concrete

3) Flexural strength

Flexural strength is enhanced by adding metakaolin to the concrete mixture. Flexural strength improves as calcination temperature and curing duration increase. As shown (Figure 6), flexural strength peaked at 15% by weight substitution of metakaolin. These findings are consistent with those made by Dubey and Banthia (1998), Qian and Li (2001), and Dinakar *et al.* (2013) who found that using metakaolin as a partial cement substitute enhanced the rupture modulus. Thus, improvements in pore structure and denser, thinner interfacial transition zones, which translate into a proportionately less weak phase, may be associated with an increase in flexural strengths in concretes containing metakaolin. All the beam samples tested, failed within the middle third length of the beam (Figure 7).

The failure was brittle and sudden.

From Figure 8, it is observed that the assumptions of normality, constant variance, and independence were met. It is shown in Table 3 that the percentage replacement with metakaolin, curing time, and calcination temperature are significant terms ($P < 0.1$) to the flexural strength of metakaolin-concrete. It is also observed from the table that the interaction between the percentage replacement with metakaolin and the calcining temperature was statistically significant ($P < 0.1$).

Table 3: ANOVA for Flexural strength of Metakaolin-Concrete

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	4	65.398	16.3496	55.31	0.000
% Metakaolin	1	1.520	1.5202	5.14	0.028
Days	1	9.720	9.7200	32.88	0.000
Calcining temp	1	5.188	5.1882	17.55	0.000
% Metakaolin*Calcining temp	1	3.102	3.1022	10.49	0.002
Error	43	12.711	0.2956		
Total	47	78.110			

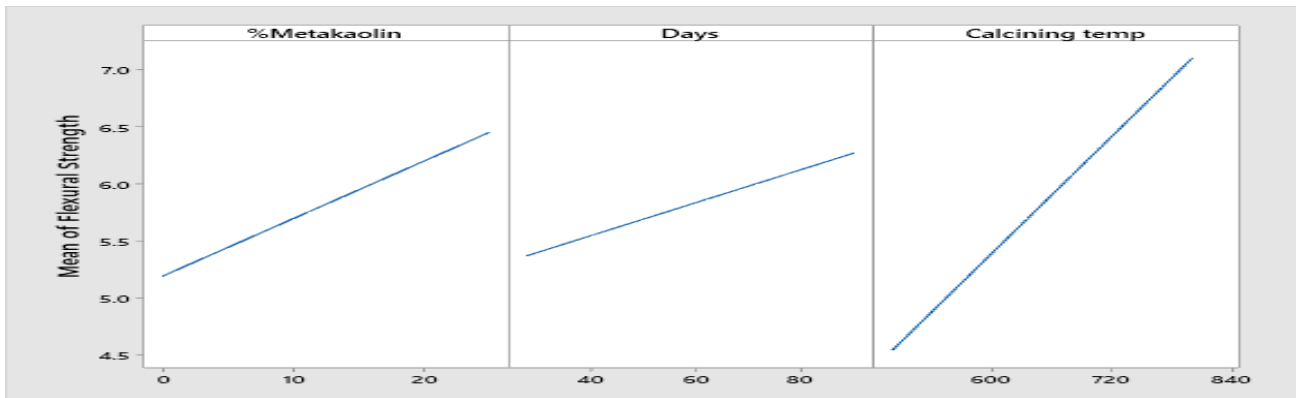


Figure 9: Relationship of main effects on flexural strength of metakaolin-concrete

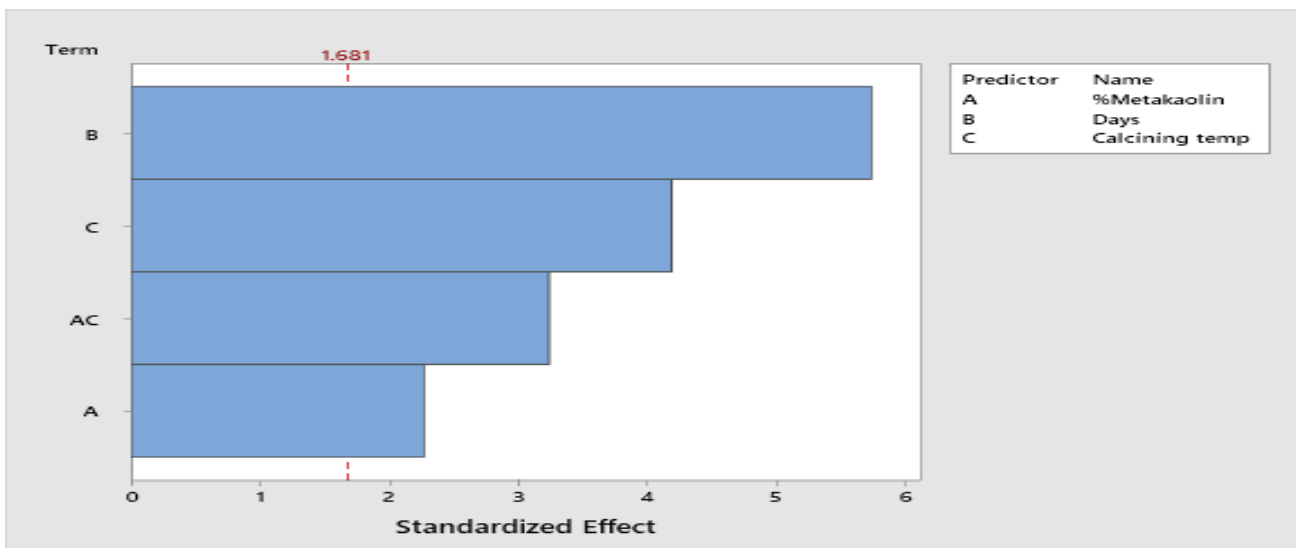


Figure 10: Pareto chart for standard effects of flexural strength of metakaolin-concrete

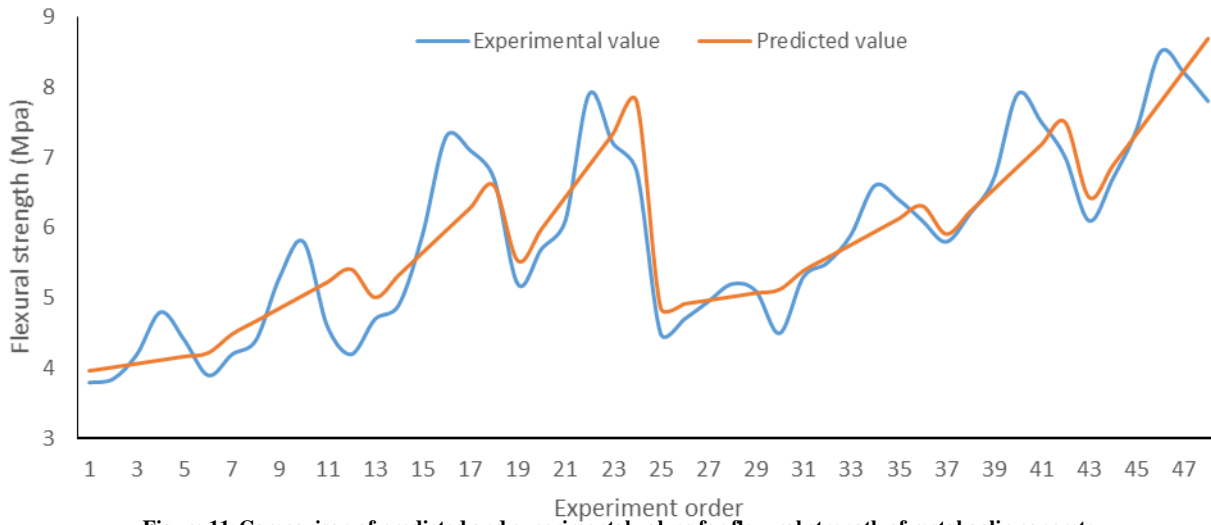


Figure 11: Comparison of predicted and experimental values for flexural strength of metakaolin concrete

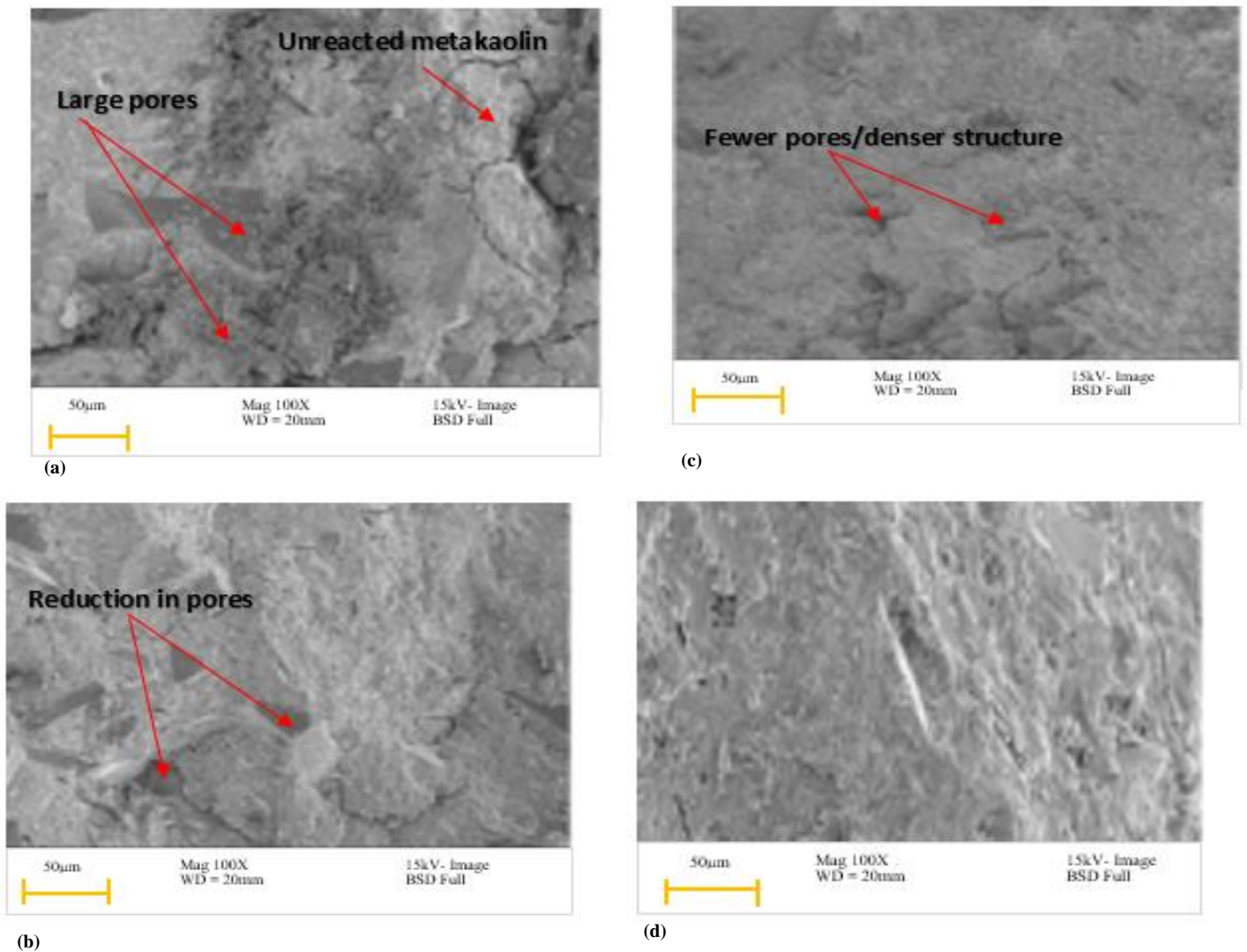


Figure 12: SEM of Metakaolin-concrete sample at (a) 500°C metakaolin calcination temperature (b) 600°C metakaolin calcination temperature (c) 700°C metakaolin calcination temperature (d) 800°C metakaolin calcination temperature

From the main effects plot (Figure 9), it is shown that all factors considered have positive effects on the flexural strength of the metakaolin-concrete when they are increased. The Pareto chart of standardized effects (Figure 10) for flexural strength shows that the calcining temperature, followed by the curing time, has the greatest impact on the flexural strength of metakaolin-concrete. The comparison of the predicted values for the flexural strength compared with the experimental data obtained presented (Figure 11) showed that the trend was well predicted with the predicted values quite close to the experimental data.

4) *Microstructure of Metakaolin-Concrete*

At 500°C, unreacted metakaolin powder can be seen in the sample (Figure 12a). As calcination temperature progressed from 600°C to 700°C, the amount of unreacted metakaolin was largely reduced (Figures 12b and 12c). The overall structure was denser and fewer pores were shown in the gel matrix. Increased calcination temperature promotes the dissolution of metakaolin and generates more gels to bind the quartz powder. When the calcination reaches 800°C, the amount of unreacted metakaolin is further reduced (Figure 12d). The SEM findings are consistent with what Mostafa *et al.* (2010) discovered in their investigation of reactive pozzolan replacement at high levels in blended cements, where the fracture surface revealed areas with large concentrations of unreacted silica fume. Ayeni also reported a related discovery (2017).

V. CONCLUSION

This study evaluated the metakaolin-concrete mechanical properties at varying calcination temperatures and various percentage replacements of cement with metakaolin. At all temperatures considered, the cumulative percentage of silica, alumina, and ferric oxide present was greater than 70% and is classified as Class N pozzolan. According to the results of the experiment, the compressive strength increased as the calcination temperature increased. This was also observed for flexural strength. Calcination temperature of 800°C and 15% by weight of metakaolin replacement gave the best strength values; 32.0 N/mm² at 90 days curing age for compressive strength and 8.5 N/mm² at 90 days curing age for flexural strength. At every calcination temperature investigated, strength was higher in all metakaolin admixed mixes than the control except in 25 % by weight of metakaolin replacement and this could be attributed to the effect of dilution. This implies that even in areas where there is no access to an electric furnace, kaolin can be calcined to metakaolin with a temperature as low as 500°C and be used in construction works.

Entrepreneurship and other associated lines of business may develop in local communities where kaolin can be processed into metakaolin due to the low calcining temperature. This will surely bring prosperity to the local communities where they are found all over the country. Results from the ANOVA showed that the percentage replacement with metakaolin, curing time and the calcining temperature all have significant effects on the flexural strength. The compressive strength was significantly affected

by the calcining temperature, which also had the most impact. The compressive and flexural strengths may be predicted using the empirical models created for the investigation.

AUTHOR CONTRIBUTIONS

Y.O. Abiodun; S.O. Adeosun and J. I. Orisaleye: Conceptualization, Software, Validation, Writing – original draft. **Y.O. Abiodun and S. O. Adeosun:** Conceptualization, Methodology, Supervision. **Y.O. Abiodun:** Writing – review & editing.

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