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[1.]Yetunde O. ABIODUN, [2.] Joseph Ifeolu ORISALEYE, [3,4.]Samson O. ADEOSUN

IMPACT OF KAOLIN CALCINATION TEMPERATURES ON THE COMPRESSIVE AND FLEXURAL STRENGTHS OF METAKAOLIN–CONCRETE

[1.]Department of Civil and Environmental Engineering, University of Lagos, Akoka, NIGERIA
[2.]Department of Mechanical Engineering, University of Lagos, Akoka, NIGERIA
[3.]Department of Metallurgical and Materials Engineering, University of Lagos, Akoka, NIGERIA
[4.]Industrial Engineering Department, Durban University of Technology, Durban, SOUTH AFRICA

Abstract: The exploitation of pozzolanic materials in concrete construction is progressively increasing. This is due to technological advancement and climate change problems associated with carbon emission resulting from the large–scale manufacturing of cement and its usage for concrete production. In this study, metakaolin 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. Metakaolin was used to replace cement at 0, 5, 10, 15, 20 and 25 wt. % using mix ratio 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 flexural strength test was performed at curing ages of 28 and 90 days. For both compressive strength and flexural strength, 15 wt. % replacement with metakaolin gave the best strength values at all temperatures. Increase in temperature led to significant increase in 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 potential alternative to cement and can be used in the construction industry and that calcination temperature of kaolin has significant effect on the properties of metakaolin–concrete produced.

Keywords: analysis of variance; compressive strength; flexural strength; metakaolin; temperature

1. 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 6.3 percent growth rate per annum (Tamanna et al., 2020). During cement manufacture, approximately 800 to 1000 kg of CO_2 emissions occur per ton of cement. It is estimated that 5 to 8 percent of global man–made CO_2 emissions are produced through cement manufacture (Tosti et al., 2018; Teixeira et al., 2019). One major use of cement in structural works is the production of concrete for use in the housing sector. Housing is a crucial basic need of every human being just as food and clothing (Fasakin et al., 2019). It is very fundamental to the welfare, survival and health of man (Okafor et al., 2019). Hence, housing is one of the best indicators of a person's standard of living and his place in the society. Kehinde (2010) noted that shelter is central to the existence of man and housing involves access to land, shelter and the necessary amenities to make the shelter functional, convenient, aesthetically pleasing, safe and hygienic. Hence, unsanitary, unhygienic, unsafe and inadequate housing can affect the security, physical health and privacy of man. Invariably, the performance of the housing sector is one of the yardsticks by which the health of a nation is measured (Olawale et al., 2015; Charles, 2003). The reasons for shortage of housing in Nigeria include poverty, high rate of urbanization, high cost of building materials, as well as rudimentary technology of building (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). Furthermore, environmental concerns stemming from the high energy expense and CO_2 emission associated with cement manufacture have brought about pressures to reduce cement consumption through the use of supplementary materials (Sabir et al., 2001; Mindess et al., 2003). Potential supplementary materials, mostly pozzolans include fly–ash, metakaolin, silicafume 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 on high volume metakaolin as cement replacement in mortar. It was observed that cement replacement with metakaolin, 20 wt. % gave maximum enhancement in pore refinement of pastes and compressive strength reduced when metakaolin addition was above 30 % as cement replacement. Sabria et al. (2010) aimed at improving the rheological behaviour of cement paste with metakaolin and found that replacement at 10 wt. % and 15 wt. % metakaolin shows better viscosity and shear stress and improved the cement paste flowability. Kannan and Ganesan (2012) studied the strength and absorption properties of ternary blended cement mortar using rice husk ash and metakaolin and reported that enhancement in compressive strength of 20.9 % was observed at 15 wt. % replacement of rice husk ash, 17.42 % at 25 wt. % replacement of metakaolin and 24.61 % at 30 wt. % replacement of equal ratios (1:1) of rice husk ash and metakaolin combination. This study aims to investigate the mechanical properties and the suitability of kaolin calcined at varying temperatures in concrete production. This has helped to tap into the available mineral resources for the development of effective and environmentally friendly alternative building material for construction purposes.

2. MATERIALS AND METHODS

— Calcination and Chemical Composition Test

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 an accessible and well–connected roads and foot paths. Imeko town falls within the basement complex terrain of Southwestern Nigeria. It has an appreciable thickness of sedimentary rock formation. The dominant rock types include a variety of hard to very hard rocks which have metamorphosed in varying degrees to migmatites and gneisses and intruded by granites (Olabode and Mohammed, 2016). 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 the Atomic Absorption Spectroscopy was carried out on each of the calcined samples to determine the percentages of the chemical compounds present.

— Compressive and Flexural Strengths

The calcined kaolin (metakaolin) was used to replace cement at 0 % (control), 5%, 10%, 15%, and 20% by weight of binder. Mix ratio of 1:2:4 (cement: sand: coarse aggregate) and water–cement ratio of 0.4 was employed in all the mixes. For Compressive strength test, two hundred and sixty–two cubes of size 150 mm × 150 mm × 150 mm were cast as represented in Figure 1 and cured in water as shown in Figure 2 for 7, 28 and 90 days. The compressive strength test procedure was in accordance to BS EN 12390–3 (2009). The compressive strength was estimated using Equation 1.

$$P = F/A \tag{1}$$

where P is the compressive strength (N/mm^2), F is the applied load (N) and A is the area of concrete cube (mm^2).

To determine 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. Flexural strength test procedure was in accordance to BS EN 12390–5 (2019). At each data point, three repeat samples were cast and tested for 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 Equation 2.

Figure 1. Casting of cubes for compressive strength

Figure 2. Curing of test samples

$$F = PL/bd^2 \tag{2}$$

where F is the flexural strength of modulus of rupture (N/mm^2), P is the applied load (N), L is the length of the beam sample (mm), b is the breadth of the beam sample (mm) and d is the depth of the beam sample (mm).

— 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 which 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 the interactions between these factors.

3. RESULTS AND DISCUSSIONS

The result of the chemical composition of calcined kaolin (Metakaolin) at different temperatures and duration of 60 minutes is presented in Table 1.The results infer that temperature has significant effect on the chemical composition of the metakaolin. Also, it was noted that while the Loss on Ignition (LOI) at temperatures 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, 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 metakaolin samples obtained after calcination can be classified as class N pozzolans (ASTM C618–12a, 2012).

Table 1. 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_2O_3	31.45	33.67	35.56	39.90
Fe_2O_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_2O	0.28	0.18	0.28	0.11
K_2O	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

— Compressive strength

The compressive strength increased with increase in calcining temperature. It was observed in Figure 3 that the strength also increased as the percentage replacement with metakaolin increased up to 15 wt. % replacement because of the existence of high amount of calcium silicate hydrates (CSH), a strength–enhancer. Beyond 15 wt. % metakaolin replacement, compressive strength decreased.

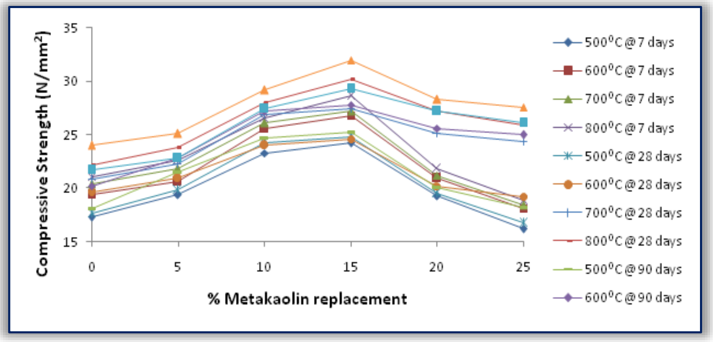
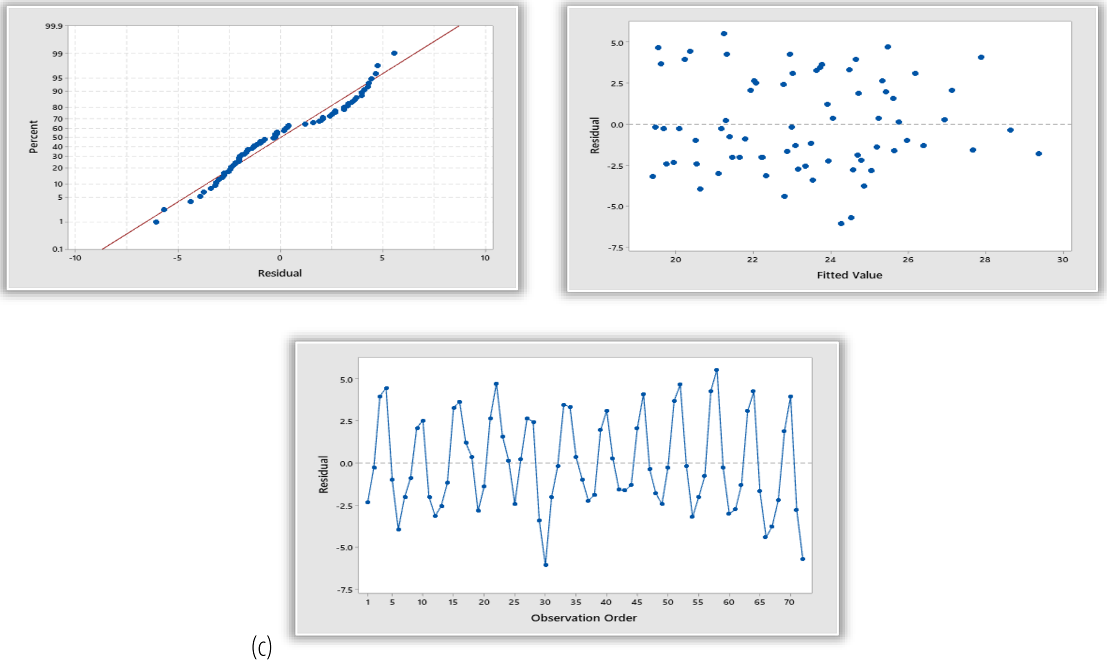


Figure 3. Effect of calcining temperature and curing days on compressive strength

Different statistical plots were used to check that the assumptions of ANOVA are not violated as shown in Figure 4. Figure 4(a) is the normal probability plot which shows that population in each treatment is normally distributed since the plotted points can be fitted with a straight line. Also, Figure 4(b) shows the residuals versus fits which reveals that the constant variance assumption is met since the plotted points are scattered with a pattern. Figure 4(c) shows that the independence assumption for ANOVA is not violated since there is no defined pattern of the plot.



(a)

(b)

(c)

Figure 4. (a) Normal Probability plot (b) Residual versus Fitted values plot (c) Residual versus observation order plot for compressive strength of metakaolin–concrete

The ANOVA table for the Compressive strength of metakaolin concrete is presented in Table 2. It is shown from Table 2 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 in Figure 5 shows that the percentage metakaolin replacement, the curing days and the calcining temperature have positive influence on strength of metakaolin concrete. Increase in any of these parameters led to an increase in the compressive strength. It could be observed from the pareto chart for standardized effects for the compressive strength of metakaolin concrete in Figure 6 that the calcining temperature has the largest effect on the compressive strength of the concrete.

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) is using the terms in the ANOVA in Table 2 is presented in Equation (3) with a coefficient of determination (R^2) of 41.84%. The predictions from the equation are compared with experimental data as presented in Figure 7. 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.

$$\text{Compressive Strength} = 11.15 - 0.0272\text{M} + 0.0095\text{D} + 0.01704\text{T} + 0.00197\text{MD} \tag{3}$$

Table 2. ANOVA for Compressive strength of Metakaolin Concrete

Source	DF	Adj SS	Adj MS	F–Value	P–Value
Regression	4	406.080	101.520	12.05	0.000
%Metakaolin	1	1.620	1.620	0.19	0.662
Days	1	2.562	2.562	0.30	0.583
Calcining temp	1	261.291	261.291	31.01	0.000
%Metakaolin*Days	1	25.222	25.222	2.99	0.088
Error	67	564.520	8.426		
Total	71	970.601			

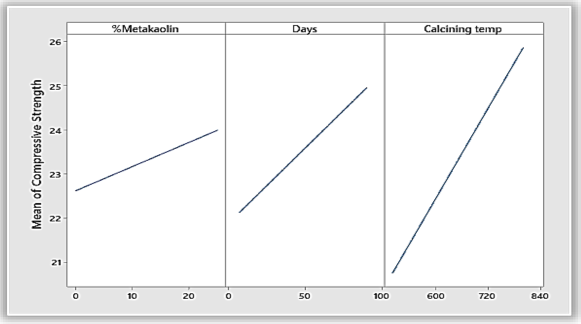


Figure 5. Main effects plot for compressive strength of metakaolin concrete

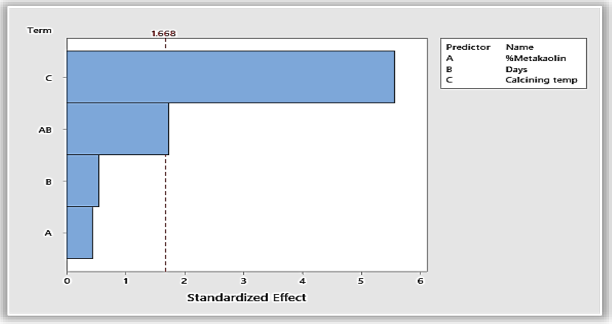


Figure 6. Pareto chart for standardized effects for compressive strength

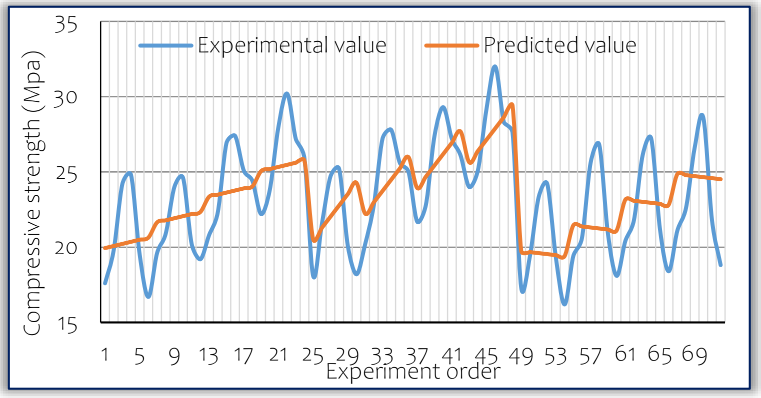


Figure 7. Comparison of predicted and experimental values for compressive strength of metakaolin concrete

— Flexural strength

The inclusion of metakaolin in the concrete mix leads to increase in flexural strength. As the calcination temperature and duration of curing increases, flexural strength increases as shown in Figure 8.

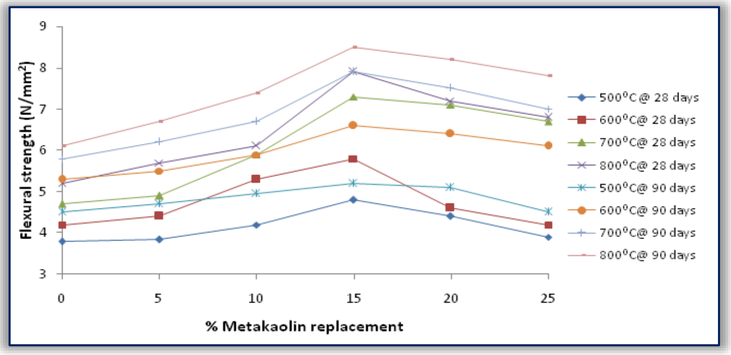
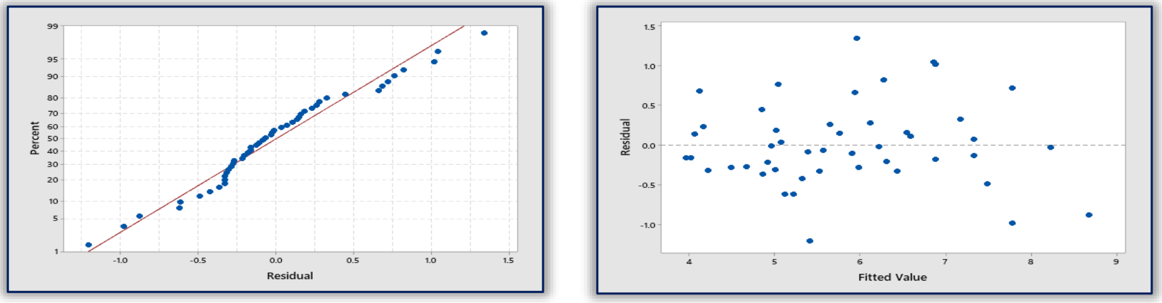


Figure 8. Effect of calcining temperature and curing days on flexural strength

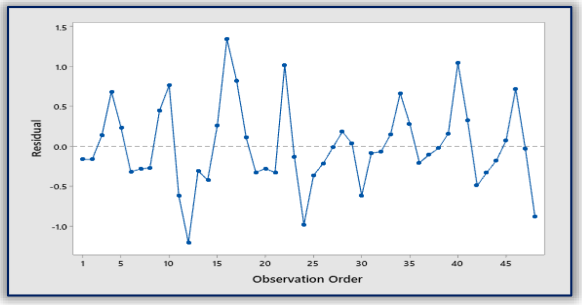
Figure 9. Failure mode of the unreinforced concrete beams containing metakaolin

The highest value of flexural strength was recorded at 15 wt. % metakaolin replacement. These results agree with those reported by Dinakar et al. (2013), Dubey and Banthia (1998) and Qian et al. (2001) where modulus of rupture increased when metakaolin was used as partial replacement for cement. Thus, increase in flexural strengths in concretes containing metakaolin may be related to refinements in pore structure and denser, thinner interfacial transition zones, which means proportionally less of weaker phase. All the beam samples tested, failed within the middle third length of the beam as presented in Figure 9. The failure was brittle and sudden.

The assumptions for ANOVA were checked using the plots in Figure 10. It is shown that the assumptions of normality, constant variance and independence were met. The ANOVA for the flexural strength is presented in Table 3. It is shown from Table 3 that the percentage replacement with metakaolin, curing time and calcining 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 were statistically significant (P<0.1).



(a) (b)



(c)

Figure 10. (a) Normal Probability plot (b) Residual versus Fitted values plot (c) Residual versus observation order plot for flexural strength of metakaolin–concrete

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			

The main effects plot is shown in Figure 11. It is shown that all factors considered have positive effects on the flexural strength on the metakaolin concrete when they are increased. It is observed from the pareto chart of standardized effects for the flexural strength that the curing time followed by the calcining temperature have the highest effect on the flexural strength of metakaolin concrete (Figure 12).

The regression equation which describes the relationship between the flexural strength and the factors identified in the ANOVA in Table 3 is shown in Equation (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 in Figure 13 and it is seen that the trend was well predicted with the predicted values quite close to the experimental data.

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

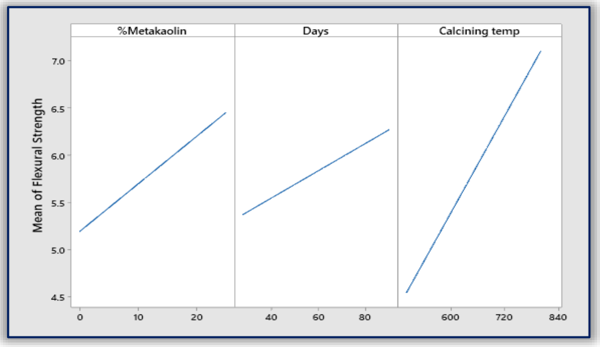


Figure 11. Main effects plot for flexural strength of metakaolin concrete

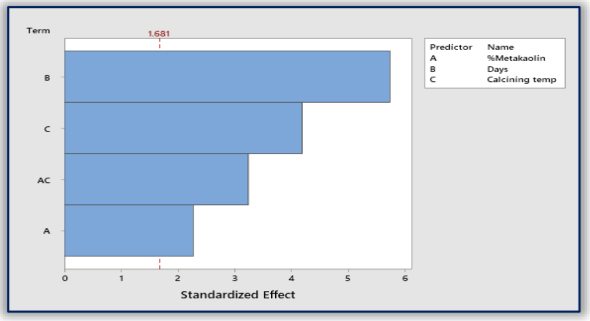


Figure 12. Pareto chart for standard effects of flexural strength of metakaolin concrete

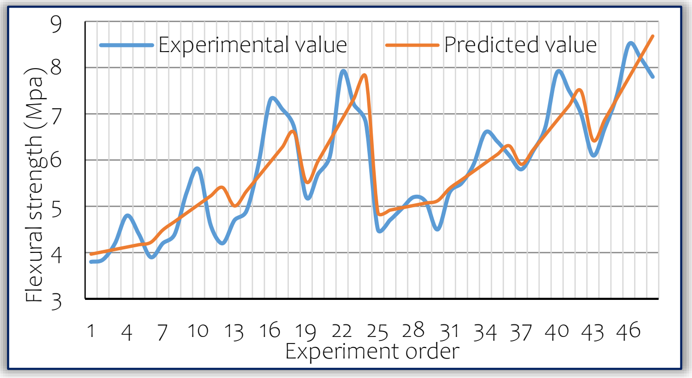


Figure 13. Comparison of predicted and experimental values for flexural strength of metakaolin concrete

4. CONCLUSIONS

This study investigated the mechanical properties of metakaolin concrete at varying calcining temperatures and different percentage replacement with metakaolin. At all temperatures considered, the cumulative percentage of silica, alumina and ferric oxide present were > 70% and could be classified as Class N pozzolan. From the experiment carried out, it was observed that the compressive strength increased as the calcining temperature increases. This was also observed for flexural strength. Calcination temperature of 800ºC and 15 wt. % metakaolin replacement gave best strength values; 32.0 N/mm^2 at 90 days curing age for compressive strength and 8.5 N/mm^2 at 90 days curing age for Flexural strength. At every calcining temperature investigated, strength was higher in all metakaolin admixed mixes than the control except in 25 wt. % metakaolin replacement and this could be attributed to the effect of dilution. This implies that even at areas where there is no access to electric furnace, kaolin can be calcined to metakaolin with temperature as low as 500ºC and be used in construction works.

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 calcining temperature was significant, and had the greatest effect, on the compressive strength. The empirical models developed in the study are useful to predict the compressive and flexural strengths.

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