Compressive Strength and Thermogravimetric Analysis of Metakaolin Blended Cement

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ORIGINAL RESEARCH ARTICLE

Abstract- Cement concrete is the most extensively used construction materials across the world. The environmental unfriendliness attached to the production of cement which is one of the major constituents of concrete demands persistent attention and the wide use of pozzolanic material as partial replacement of cement in concrete looks to be a promising supplement for cement in concrete production. Based on past research works, metakaolin was selected and its compressive strength and thermal resistance as well as other properties were determined. The replacement percentage of metakaolin employed was 5%, 10%, 15% and 20% by weight of cement with a mix ratio of cement to sand to granite of 1:2:4 cured at 7 days, 14 days, 28 days and 56 days. The output of this research work shows that with respect to compressive strength and thermal resistance, the metakaolin blended cement examined here is not suitable as supplementary materials for cement in construction industry.

Keywords- Cementitious materials, compressive strength, metakaolin, thermal resistance, thermogravimetric

1 INTRODUCTION

One of the most universally used and acceptable building and construction materials on earth is concrete. The strength, durability and other properties of concrete depend on the mix proportions of the constituents of materials, quality of the constituent materials, method of compaction and type of curing process. Review of past works expresses that raw materials (especially the local content) for the concrete differs from country to country which is responsible for the variation in the performance of concrete produced in different countries (Yaun, Liu, Zheng and Ma., 2021). Cement, an expensive and vital component of concrete possess a threat to the environment through a high percentage of CO_2 emissions during its manufacture. Therefore, the need for its replacement either partially or totally. Among the different ways proposed for reducing CO_2 emission into the atmosphere, the most effective way is to minimize the use of cement clinker by substituting it partially with supplementary cementitious materials.

Consequently, researches are been geared towards the use of supplementary materials one of which are pozzolans (Sabir, Wild and Bai 2001, Ikumapayi, Arum and Alaneme, 2020, Aminu *et al*, 2020). The term "pozzolan" represents all the materials which react with lime and water giving calcium silicate and aluminate hydrates such as Slag, Metakaolin, Fly-ash, Rice ash husk and bamboo leaf ash among others. Specifications for pozzolans are available in the standard (Bensted and Barnes, 2002; ASTM 618, 2012).

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These pozzolans can replace cement to some certain degrees which make their behaviour and properties to be of paramount importance. Their behaviour and properties as blended cement and in the concrete at large needs to be established. Some of the paramount properties of blended cement are consistency of fresh concrete, fineness of cement, strength, soundness, setting time, heat of hydration, bulk density and loss of ignition. While some of their behaviour and properties in concrete that should be investigated include compressive strength, tensile strength, corrosion resistance, sulphate resistance, fire/ thermal resistance, abrasion resistance, water absorption, porosity and density.

Pozzolanic activity can be directly determined by measuring the amount of reacted lime, using the technique of thermogravimetry analysis and differential thermal analysis (DTA) and their thermal resistance and behaviour can also be determined using same methods. Thermogravimetric analysis (TGA) is one of the major thermal analysis techniques. This technique monitors the behaviour of the mass of a substance under a controlled temperature in relation to time. It measures parameters such as mass loss, time, and temperature as the sample weight is being heated and cooled in a furnace (Haines, 1995**).** Heating rate is an important parameter in TGA (Soriano *et.al.* 2013). Kaolin is also one of the natural raw materials that can be found worldwide in countries like Georgia in United States, Nigeria, China, South Africa, Algeria, Tanzania, Cameroon, Egypt, Burundi, Botswana, Zimbabwe among others (Ekosse, 2010; Raw Materials Research and Development Council (RMRDC), 2008). Metakaolin (MK) is one of the most abundant natural minerals and is a synthetic product that is formed by calcining kaolin; it is a very fine material with high pozzolanic activity, as reported by some past researchers (Sabir, Wild and Bai, 2001). This research aims to study the behaviour in term of compressive strength and thermal resistance of metakaolin pozzolanic concrete under different temperatures and to obtained the most effective percentage for replacement.

Table 1. Mix -Proportioning and their corresponding batches for the concrete

Sections	Cement in %	Metakaolin in %	Cement: Sand: Granite in (Kg).
Batch 1 (MK0)	100	0	13.8 : 34 : 60
Batch 2 (MK10)	90	10	12.42 : 34 : 60
Batch 3 (MK15)	85	15	11.73 : 34: 60
Batch 4 (MK20)	80	20	11.04 : 34: 60

2 Materials and Methods

2.1 Properties of the Research Materials

The major materials explored for this work are metakaolin, cement, fine aggregate, coarse aggregate, and water. The two major tests carried out are the compressive strength test and the thermogravimetric test. Some materials tests were also carried out to ascertain their suitability. Material tests carried out for the aggregates and their corresponding standard are sieve analysis ASTM C136/C136M-14), specific gravity (ASTM C127,2016), moisture content (ASTM D854-02, 2017), aggregate impact value (AIV) test (IS: 2386 (Part IV) – 1963) and aggregate crushing value (ACV) test (IS: 2386 (Part IV) – 1963). These materials tests were carried out at the structural laboratory of Federal University of Technology, Akure, Nigeria.

The raw powdered kaolin material for this study was sourced from Zaria, Northern Nigeria in a milled processed form. Metakaolin was obtained from calcination of kaolin clay at a temperature of 700°C based on the temperature adopted by Naghizadehi *et al*. (2015).

2.2 Mix Proportioning, Preparation and Casting of Concrete Specimen

The mix proportioning for this research is as shown in Table 1 and the cement: sand: granite mix ratio is 1:2.5: 4.35.

2.3 Preparation of Concrete Samples and Testing

Dry mixing of the sand, coarse aggregate, and metakaolin blended cement was done. Water was added and further mixing was done until an even mix was obtained. Workability tests in term of slump and the compacting factor tests were done on the fresh concrete. The concrete was cast having a dimension of 150 x150 x 150mm³. The concrete samples were demoulded after 24hrs, cured by water immersion at 7, 14, 28, and 56 days as specified in BS 1881: part 108 (1983), weighed and then the compressive strength test was carried out and calculated as shown in Equation 1.

$$f_c = \frac{P}{A_e} \qquad (1)$$

Where f_c *is the* Compressive strength,
P *is the crushing load* (N)
A_e *is the effective area* (mm^2)

Thermogravimetric test was carried out on the concrete samples of MK0 and MK15 using a TGA machine and the samples were subjected to heat with the initial temperature as 30°C and the final temperature as 950°C. The result was automatically generated by the machine.

3 Results and Discussion

The grading test using sieve analysis was done and the result is as shown in Fig. 1. The coefficient of curvature (fineness modulus) was determined from the curve (Figure 1) using Equation 2:

$$C_c = \frac{{D_{30}}^2}{D_{60} \ X \ D_{10}} = \frac{0.35^2}{0.75 \ X \ 0.15} = 1.09 \qquad (2)$$

According to the result in Fig. 1, Cc is approximately equal to 1 and it shows that the sand is well graded since the fineness modulus must fall between the ranges of 1-3. (BS EN 12620: 2002). The results of the various material tests and their significant are shown in Table 2. The results show the materials are suitable according to their different relevant codes.

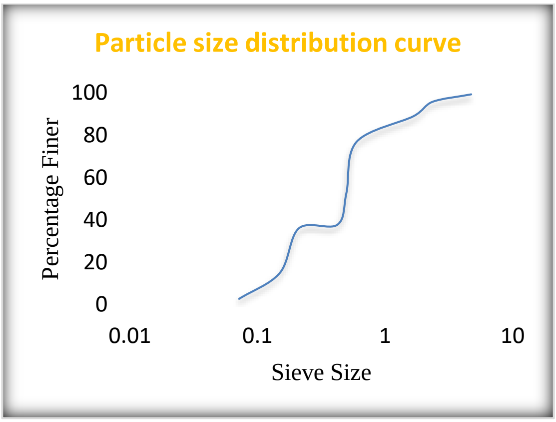

Fig. 1: Particle size distribution

The result of the oxide composition of metakaolin at 700°C are shown in Table 3. This result shows the sum of $SiO_2 + Fe_2O_3 + Al_2O_3$ is far below the minimum of 50% specified by ASTM 618 (2012) as requirement for pozzolans.

3.1 Results of Test on Fresh Concrete

The slump test of the concrete mix was carried out immediately after mixing the materials in the right proportions. The result of the slump test is as shown in Fig. 2 and it indicates that the addition of metakaolin reduced the workability of concrete considerably; hence, it requires more water to increase the workability.

The result of the compaction test for the fresh concrete is also shown in Table 4. This test shows the ability of the prepared concrete to be well compacted. This is measured using compaction factor as indicator. The compaction factor is the ratio of weights of partially compacted to fully compacted concrete. It is also a means of determining the workability of the concrete. According to standards (BS EN 12350-4:2009), this ratio ranges from 0.7 to 0.95.

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Table 2. Results of the Material Tests

Materials	Test	Relevant Standard	Limit in the Code	Result	Significant of the result
Sand	Specific Gravity	ASTM C127,2016	Varies with weight	2.64	Suitable for concrete production
Coarse Aggregate	AIV	BS IS: 2386 (Part IV) – 1963.	0-20%	11.2%	Strong and satisfying
Coarse Aggregate	ACV	BS IS: 2386 (Part V)-1963	30%	30%	Satisfying
Sand	Cc	(BS EN 12620: 2002).	1-3	1	Well graded

AIV-Aggregates Impact values, ACV-Aggregates Crushing Values, Cc- Coefficient of curvature

Table 3. Result of the oxide composition of metakaolin at 700°C

Oxide Composition	Concentration (mg/cm²)
SiO_2	4.523
V_2O_5	0.051
Cr_2O_3	0.008
MnO	0.037
Fe_2O_3	0.557
Co_3O_4	0.015
NiO	0.015
CuO	0.064
Nb_2O_3	0.011
MoO_3	0.009
WO_3	0.012
P_2O_5	0.000
SO_3	0.000
CaO	76.591
MgO	13.232
K_2O	0.208
BaO	0.076
Al_2O_3	4.004
Ta_2O_5	0.033
TiO_2	0.020
ZnO	0.000
Ag_2O	0.000
Cl	0.504
ZrO_2	0.030

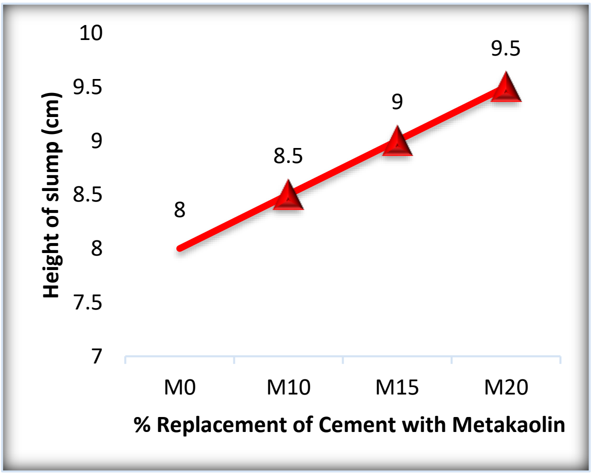

Fig. 2: Slump Test graph

Table 4. Compaction Test Result.

% Metakaolin	Partial compaction (kg)	Full Compaction (kg)	Compaction Factor
MK0	16.4	17.6	0.93
MK10	16.8	17.8	0.94
MK15	17	17.6	0.97
MK20	17.2	17.9	0.95

3.2 Average Weight and Compressive Strength of the Various Blended Concrete Samples.

The results of the average weight and average compressive strength of the various percentages blended metakaolin samples are shown in Fig 3 and Fig. 4 respectively. These results were taken after 7, 14, 28, and 56 days of curing. The average weight of various concrete cubes decreases as the curing days increases while the addition of metakaolin causes a slight increase in the weight of the samples at each of the various ages. The compressive strength of the various concrete specimens increases with curing age, there is an increase in the compressive strength from 7 day curing to 56 days curing for any of the mix proportion.

Considering the addition of metakaolin, there is a drastic reduction in the compressive strength of the blended concrete specimens as the percentage of metakaolin increases from 5% to 10% to 15% to 20% in MK5 to MK10 to MK15 to MK20 respectively. Comparing the mean of these compressive strength using one way ANOVA (Analysis of Variance) test as shown in Table 5 it is evident that there is significant change in the compressive strength of all the blended metakaolin concrete when compare to the control (MK0).

Moreover, none of the result of the compressive strength obtained for the blended metakaolin concrete meet up with the recommended compressive strength of 15N/mm² expected for grade M15 concrete (ASTMC 109/ C 109M-20, 2020). The results are in disagreement with the results of some researchers (Sims and Brown, 1998; Egwuonwu, Iboroma and Barisua, 2019; Ashik and Gomathi) This is an indication that this specific metakaolin is not a good supplementary material for cement at 5%, 10%, 15%, and 20% because there was a reduction in the compressive strength of the concrete specimens throughout. Compressive strength is one of the fundamental properties of which most other concrete properties can be measured.

Table 5. LSD test for Compressive Strength of Concrete Samples at 56 days

1-MKO (control);2-MK10; 3-MK15; 4-MK20;

(I) Factor	(J) Factor	Mean Difference (I-J)	P (Sig.)	Remarks
1.00	2.00	7.667*	<.001	*
	3.00	7.467*	<.001	*
	4.00	10.663*	<.001	*

*Mean Difference (MD) is significant at $\alpha_{0.05}$ i.e when $p \leq 0.05$ NS= Not Significant

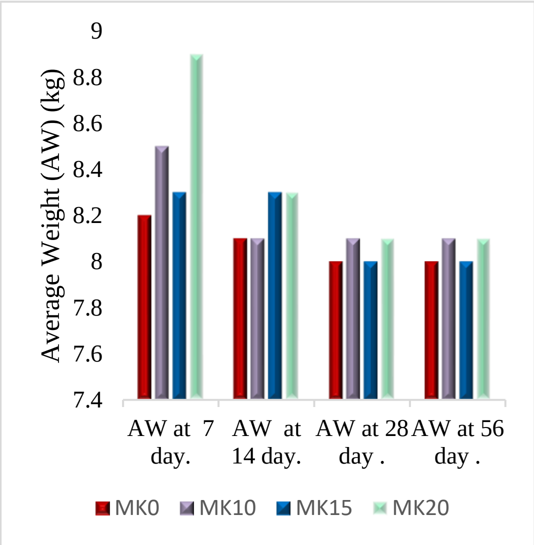

Fig. 3: Effect of Metakaolin on the Weight of the Various Blended Concrete

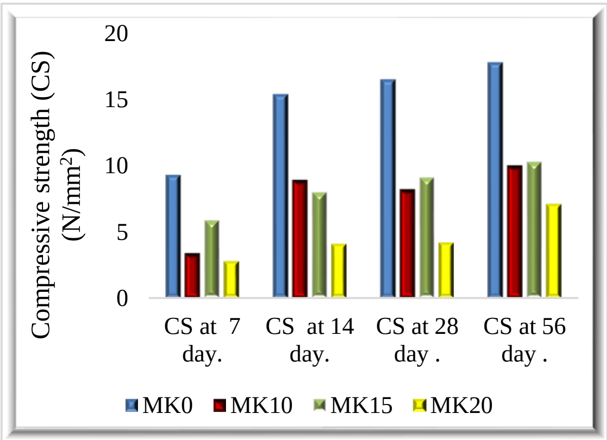

Fig. 4: Influence of Metakaolin on the Compressive Strength of the Blended Concrete

3.3 Thermogravimetric Test Analysis

One of the durability properties of concrete is the thermal resistance and this can be determined using thermogravimetric analysis of such concrete materials among other methods. The behaviour of metakaolin induced concrete at temperatures ranging from 30⁰c to 900⁰c was studied. Thermogravimetric relationship between mass and temperature of 0% (MK0) and 15% (MK15) replacement at 7, 14, 28, and 56 days is as shown in Fig. 5. The temperature produces a gradual decrease in the mass of the samples for both MK0 and MK15. The result further shows that as the temperature increased from 30⁰c to 900⁰c, the mass of MK15 decreased to 4.952%, whereas the control sample, MK0 decreased to 15.65 %. The percentage mass loss was similar from 30°C to 345°C, at that point the mass of MK15 dropped drastically, whereas mass of MK0 dropped gradually. This shows that MK15 behaves similarly to control concrete up to a certain temperature, but between 345⁰c and 500⁰c, MK0 seems to be more resistance to heat than MK15 concrete. However, this demonstrates that substituting metakaolin for cement diminishes the mass of the concrete as the temperature rises, this is an indication that the fire resistance of metakaolin concrete is lower than that of the conventional concrete.

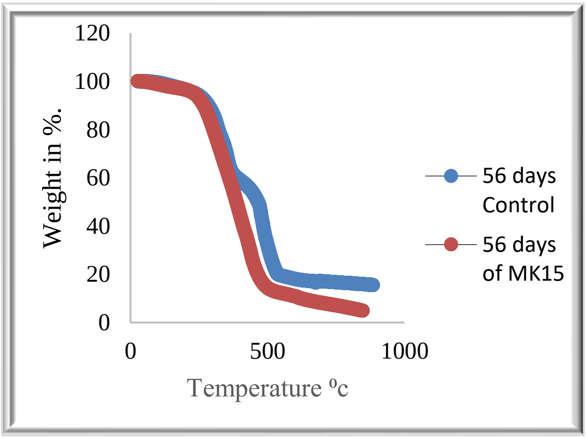

Fig. 5: Combined TGA of MK15 and MK0 (control) at 56 days.

4 Conclusion and Recommendation

It can thus be concluded that based on the experimental results of this research, cement replacement with this precise metakaolin reduces the compressive strength of concrete in relation to the control sample. Also, this metakaolin concrete has a lower thermal resistance than OPC cement. Therefore, this specific metakaolin is not a good supplementary cementitious material for ordinary Portland cement.

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