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Mineralogical Properties of Kaolin and Metakaolin from Selected Areas in Nigeria and Its Application to Concrete Production

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Abstract: *Thermal treatment was applied on same mass of kaolin samples selected from Edo, Ogun, Ekiti and Ondo states of Nigeria at varying temperature of 500, 600, 700, 800, 900 and 1,000°C for 30, 60, 120 and 180 minutes to obtain metakaolin, which is a complementary cementitious material in concrete production or as a geopolymer in Metakaolin based concrete. However, not all kaolin deposits can offer these characteristics. Virtual comprehensive dehydroxylation of the material was achieved at optimal calcination parameters of 800°C/60 min. The thermal, chemical, differential thermal (DTA), X-ray Diffraction Spectroscopy (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) characterisations of raw kaolin samples were carried out. FTIR and XRD investigations revealed the presence of kaolinite in the raw samples. Metakaolin powder was used to substitute cement portion in the designed mix of 1:1.1:2.6 and prescribed mixes of 1:1.5:3 and 1:2:4 at 0, 5, 10, 15, 20 and 25% using varying water-cement ratios ranging from 0.4 to 0.6. Compressive strength of the cubes was determined at the curing days of 7, 28 and 90. Flexural strength of beams was also determined at curing days of 28, 90 and 180. Results from the DTA showed that kaolin can be calcined to get metakaolin at temperature ranging from 700°C - 850°C. The transition of the kaolinite to metakaolinite was established in all the samples after thermal treatment (calcination) by the FTIR. Results showed that the yield increased as the calcination temperature increased. For compressive strength, results showed that the highest strength was obtained at 15% Metakaolin replacement and 0.4 water-cement ratio. At this, Metakaolin concrete(Mk-C) gained strength rapidly, exhibiting approximately 43.6%, 41.8% and 41.9% increase over 7-day Mk-C strength by 28-days for 1:1.5:3, 1:2:4 and 1:1.1:2.6 mix ratios.*

Keywords: *Calcination, Compressive strength, Fourier Transform Infra-red Spectroscopy, Flexural strength, Scanning Electron microscopy, X-ray diffraction spectroscopy*

1. Introduction

The Improvement of construction materials, which offers practical and eco-friendly benefits, is the focus of this innovative era. Kaolinite is a solid homogeneous inorganic substance with chemical composition of $Al_2Si_2O_5(OH)_4$, which means each particle has one tetrahedral silica layer and one octahedral alumina layer. Individual particles of kaolinite form stacks with hydrogen bonds and van der Waals forces holding together successive particles. The strength of these bonds prevents water from entering the interlayer spaces and causing swelling (Mitchell and Soga, 2005). Metakaolin (MK), one of such eco-friendly materials is categorised as an innovative complementary cementitious material. When metakaolin is used in cement-based systems, practical and significant environmental benefits (eco-friendly) are obtained (Ilic *et al*., 2010). Metakaolin is a dehydroxylated form of the clay mineral kaolinite (see Equation 1)

$$Al_2Si_2O_5(OH)_4 \rightarrow Al_2O_3.2SiO_2 + 2H_2O\uparrow \qquad (1)$$

The transformation of kaolin to metakaolin through calcination depends on the mineralogical states of the kaolin deposit. The thermal transformation of kaolinite has shown that factors such as temperature, heating rate and time, cooling rate and ambient conditions, meaningfully has effect on the dehydroxylation process (Kakali et al., 2001). Most of kaolinites adsorbed water is lost between 100-200°C. Kaolinites become calcined and by losing water through dehydroxilisation in the range of 500-800°C. An endothermic process occurred during the dehydroxilisation of kaolinite to metakaolin. This could be attributed to the great volume of energy required to remove the chemically bonded hydroxyl ions. The hydroxyl ions are responsible for the breakdown of the crystal structure producing a transition phase with high surface area. Metakaolin is a highly pozzolanic and reactive material and burning at higher temperature will cause recrystallisation into quartz and mullite (Salvador, 1995). Metakaolin (MK), a complementary cementitious material, adapts to ASTM C 618, Class N pozzolan specifications (ASTM 1994).

The pozzolanic activity of metakaolin is its distinctive characteristic for use in cement based systems. Metakaolin reacts chemically with hydrating cement to form an improved paste microstructure. Complementary cementitious materials could increase the workability, mechanical properties, and durability of concrete besides their positive environmental impact. They also possess latent hydraulic reactivity or pozzolanic or a permutation of these qualities (Khatib and Hibbert, 2005). The term pozzolan can be defined as a siliceous material, which, in finely divided form and in the presence of water, reacts chemically with calcium hydroxide to form cementitious compounds (Batis *et al.*, 2005). Pozzolans, like metakaolin helps to reduce bleeding of concrete and also improves the interfacial zone of the concrete. This is done by enhancing the hydration process of portland cement due to the presence of calcium silicate hydrate (CSH), which acts as a strength enhancer in the concrete containing pozzolan (Mehta and Monteiro, 1995).

In Nigeria, there are vast deposits of kaolin in the South-West, South-East, South-South and North-West regions of the country and an estimate on kaolin mineral deposit reserve in the country amounts to about 2 billion metric tonnes. Presently, kaolin deposits are extracted for commercial use in the manufacturing of ceramics and porcelain wares, production of drugs and for cosmetics purposes. Hence, it is important to determine the suitability of the metakaolin that could be obtained from Nigeria kaolin deposits for good assessment of the high cost of building materials in a growing economy like Nigeria. This would help to sustain rapid infrastructural development with affordable shelter and constructional materials.

In this study, methods such as differential thermal analysis (DTA), X-ray diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) are employed. This study is focused on the investigation of the mineralogical properties of selected Nigeria kaolin deposits and to determine the cementitious characteristics of metakaolin from these kaolin deposits in concrete production. The pozzolanic ability of metakaolin was also tested for, by casting concrete samples with metakaolin to determine their mechanical responses through compressive strength and flexural strength tests. The objectives are to determine the optimal calcination parameters for obtaining metakaolin from kaolin clay, characterisation and analysis of both the raw kaolin and the calcined kaolin (Metakaolin); and to determine metakaolin cementitious properties in concrete production.

2. Materials and Methods

2.1 Materials

Kaolin was obtained through mining operations at a depth of (1.5-2 metres) from the top soil and it is commercially available in large quantities in the study states. Four locations in Nigeria were selected for this study; Ogun (Imeko), Edo (Okpela), Ondo (Ifon) and Ekiti (Isan-Ekiti) states (see Figure 1).

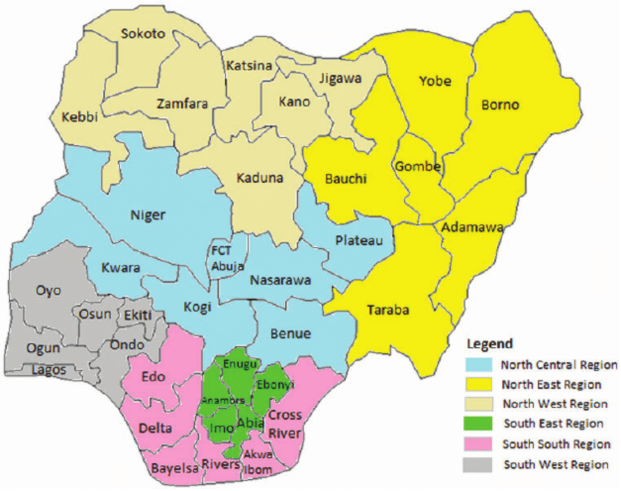

Figure 1. Map of Nigeria showing the study locations

2.2 Methods

2.2.1 Thermal Treatment

For the dehydroxylation process, representative 1.5 kg samples of kaolin were taken from the original materials using the quartering method. Thermal treatment was applied to the kaolin samples in the laboratory furnace at different temperatures (500, 600, 700, 800, 900 and 1,000°C) and at holding times of 30, 60, 120 and 180 mins. After heating, samples were cooled to room temperature (24.5°C) to prevent the crystallisation of amorphous (unstructured) metakaolin then the percentage mass loss was determined. This thermal treatment process of kaolin samples was carried out at the Department of Metallurgical Engineering, Yaba College of Technology, Lagos.

2.2.2 Mineralogical procedures

Characterisations of both the raw kaolin and thermally treated kaolin (Metakaolin) were carried out. Differential thermal analyses (DTA) of the samples were carried out at the Centre for Energy Research and Development, Obafemi Awolowo University, Ile-Ife using an INETZSCH DTA 404 PC. 5mg of each sample mass was subjected to heat using DTA/TG Al_2O_3 crucible and heated from 20 to 1,000°C at a constant rate of 10 °C/min in air.

X-ray Diffraction (XRD) of the samples was done to check the evaporation of kaolinite peaks after calcination at National Geosciences Research Laboratories (NGRL) Kaduna using Empyrean XRD model. Furthermore, Fourier Transform Infra-red spectroscopy test (FTIR) was carried out to confirm the presence of distinctive bands of kaolinite in raw sample and their absence in thermally treated samples at Redeemer's University, Ede, Nigeria using Shimadzu Scientific spectrophotometer model.

2.2.3 Laboratory Experimental Techniques

The Metakaolin obtained was milled to achieve a finesse of 700-900 m^2/kg and sieved using 45µm (No. 325) sieve to obtain fine texture and to remove impurities like sand from the sample. Due to the closeness of the mineralogical properties of the metakaolin from the study locations, Ogun sample was chosen to determine the mechanical properties of cement-metakaolin concrete. Optimum water cement ratio and optimum mix ratio were then determined from compressive strength test. For the designed mix ratio, a control mix (without Metakaolin) of grade M50 was designed to achieve a target compressive strength of $60N/mm^2$. Prescribed mix ratios of 1:1.5:3 and 1:2:4, and water-cement ratios of 0.4, 0.5 and 0.6 were adopted. Metakaolin (MK) was used to replace ordinary Portland cement at various levels of 0, 5, 10, 15, 20 and 25% by mass of binder content. A total of six mixes were done.

Three repeat samples were done per data point throughout the experiment. Fifty-four cubes were cast using designed mix while a total of thirty-six (36) mixes were prepared for the two prescribed mix ratios adopted (1:1.5:3 and 1:2:4) and a total of three hundred and twenty-four cubes (324) were cast. The test procedures were in accordance to BS EN 12390-3 (BSI, 2009). All cubes were of sizes 150×150×150 mm were tested for the compressive strength at 7, 28 and 90 days. Optimum values from the compressive strength test were employed to determine the flexural strength test of unreinforced beams. Figures 2 and 3 show the cast samples.

Figure 2. Cubes for compressive strength test

Figure 3. Beams for flexural strength test

A total of 30 unreinforced beams of size 150×150×750 mm were cast and cured for 28, 90 and 180 days. Curing was carried out at the laboratory environment and the temperature range of the curing water was 27±3°C. Flexural strength was carried out in accordance to BS EN 12390-5 (BSI, 2000).

3. Results and Discussion

3.1 Calcination of Kaolin Clay

As shown in Table 1, the percentage of mass loss at different temperatures and heating times were recorded. For all samples, mass loss increases up to 120 mins, while prolonged heating has a negligible effect on the mass loss. For all applied heating times at 800°C, the mass losses are similar. It is evident that at calcination temperature of 800°C and holding time of 120 mins, mass loss is almost identical with that at 800°C for 60 mins. Taking economic factors into consideration, the optimal calcination temperature and holding time are 800°C and 60 mins, respectively. This is slightly dissimilar to Elimbi *et al.* (2011) findings, where 700°C was found as the optimal temperature. This dissimilarity may be attributed to the geological structure of samples locations.

3.2 Chemical Composition of Kaolin Samples

Chemical composition of the raw kaolin was determined by silicate method in Chemistry Department of the University of Lagos and the percentage composition of the different compounds present is shown in Table 2. It is observed that Kaolin from all the locations gave above 70% of $SiO_2 + Al_2O_3 + Fe_2O_3$.

3.3 Thermal Behaviour of Kaolin Clay

Figure 4 presented the DTA curves of the kaolin samples. The first phenomenon occurred at 100°C and is related to elimination of water of hydration (i.e. the release of water absorbed in pores and on the surfaces). The second phenomenon occurred between 200°C and 500°C, and the mass loss is attributed to the pre-dehydration process that takes place because of the reorganisation in the octahedral layer.

Table 1: Mass loss (%) of kaolin for different calcination temperatures and times

		Temperature (°C)				
		Sample A (Edo)				
Heating time (min)	**500**	**600**	**700**	**800**	**900**	**1000**
30	0	2.00	10.67	13.33	14.00	14.67
60	0.67	4.00	11.33	12.00	13.33	14.00
120	2.67	4.67	10.00	12.00	12.07	12.09
180	2.00	12.00	12.18	13.40	12.12	12.27
		Sample B (Ogun)				
30	5.33	8.00	12.00	14.67	16.00	16.67
60	5.33	11.33	13.33	15.33	16.67	17.33
120	6.67	12.67	14.67	15.33	15.40	15.39
180	7.33	13.33	14.92	15.00	15.07	15.31
		Sample C (Ondo)				
30	0.67	2.00	4.67	8.00	10.67	11.33
60	0.67	3.33	4.67	9.33	11.33	13.33
120	2.00	4.00	5.27	9.33	9.46	9.50
180	2.67	6.00	6.69	9.82	10.01	9.46
		Sample D (Ekiti)				
30	2.67	3.33	4.67	8.00	10.67	13.33
60	3.33	5.33	8.67	10.00	12.67	14.67
120	6.67	8.67	9.33	10.05	10.12	9.89
180	8.00	9.09	9.86	10.00	11.33	10.37

Table 2. Chemical composition and physical characteristics of kaolin deposits from the study locations

Composition	**Content (%)**			
	Ogun	**Edo**	**Ekiti**	**Ondo**
SiO_2	48.50	47.82	42.54	39.87
Al_2O_3	32.75	31.65	29.07	30.72
Fe_2O_3	4.28	2.04	2.32	1.45
CaO	1.08	1.02	1.09	0.96
MgO	0.49	0.33	0.54	0.45
Na_2O	0.16	0.12	-	0.17
K_2O	1.48	1.54	1.03	1.26
LOI	9.26	12.98	13.46	15.52
	Physical properties			
Specific gravity (g/cm^3)	2.63	2.60	2.54	2.58
Sp. Surface area (cm^2/g)	9150	8970	8950	8970

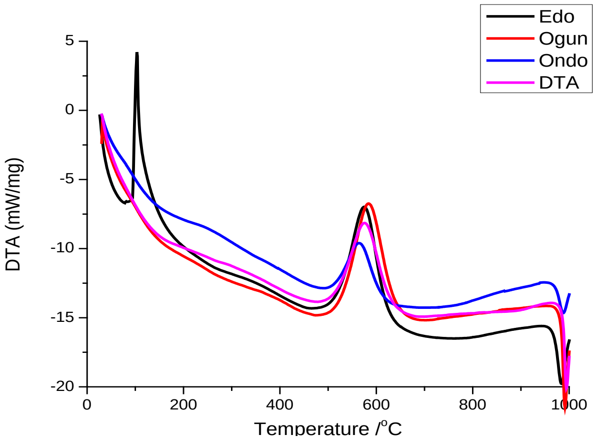

Figure 4. DTA curves of kaolin samples

The third spectacle was between 600°C and 800°C and corresponds to the dehydroxylation of kaolinite to form Metakaolin. Conversion of metakaolin to Illite started between 900°C and 1,000°C, as indicated by an exothermic peak. Less reactive material is obtained at calcination below 600°C containing more residual kaolinite. These results slightly differ from the 450°C and 550°C obtained by Kenne *et al.* (2015) in converting kaolinite to metakaolin. However, it is similar to what was reported by Bensted and Barnes (2002) where recrystallisation began and reactivity declined above 850°C, as kaolin starts its transformation to relatively inert ceramic materials, such as mullite, spinel and silica.

Figures 5-8 show the XRD test results carried out to check the evaporation of kaolinite peaks, after thermal treatment. It is evident from the diffractograms that the constituents of the kaolin samples vary. Edo sample was heavy with the existence of kaolinite only. In Ogun sample, the crystalline phases revealed by the XRD indicated mainly the presence of quartz, kaolinite and anatase. Ekiti and Ondo have similar characteristics as kaolinite was predominant with the presence of illite. These results are slightly different from what was obtained by Ilic *et al.* (2010) where the starting kaolin clay contained majorly kaolinite and quartz.

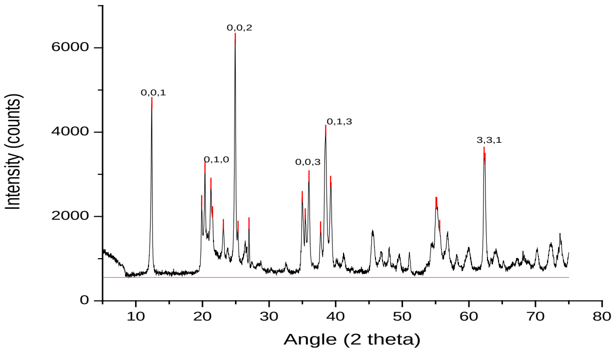

Figure 5. XRD for Ogun

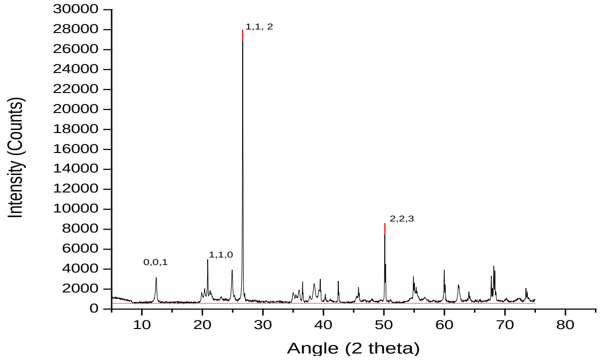

Figure 6. XRD Edo

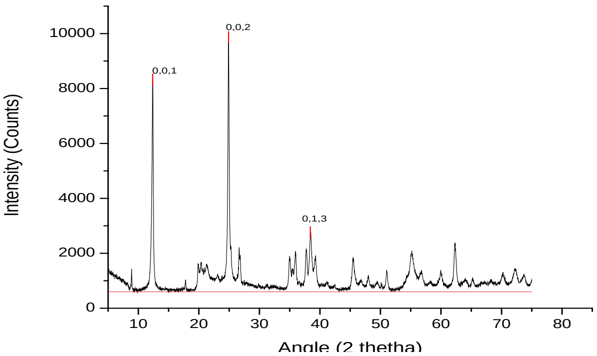

Figure 7. XRD for Ondo

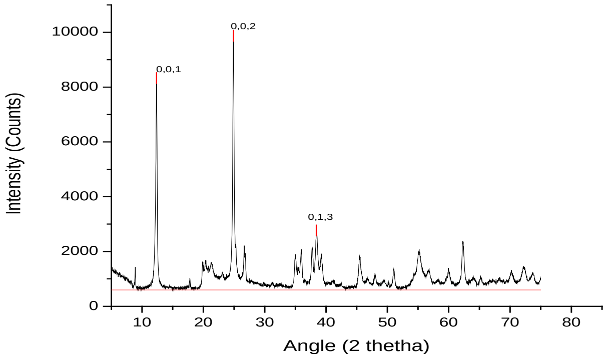

Figure 8. XRD for Ekiti

Table 3. Crystalline phases of XRD results of kaolin

Compound Name	Crystal system	Formulae	Reference code	Sample location
Kaolinite	Anorthic	$Al_2Si_2O_9H_4$	96-900-9235	Edo
Kaolinite	Anorthic	$Al_2Si_2O_9H_4$	96-900-9235	Ekiti
Illite	Monoclinic	$K_2Al_4Si_8O_{24}$	96-901-3733	
Quartz	Hexagonal	Si_3O_6	96-901-3322	Ogun
Kaolinite	Anorthic	$Al_2Si_2O_9H_4$	96-900-9235	
Anatase	Tetragonal	Ti_4O_8	96-900-8216	
Kaolinite	Anorthic	$Al_2Si_2O_9H_4$	96-900-9235	Ondo
Illite	Monoclinic	$K_2Al_4Si_8O_{24}$	96-901-3733	

The XRD diffractograms displayed the existence of different crystalline phases and are summarised in Table 3. The prominent peaks at 2θ detected are 12.3856, 21.5375, 25.3174, 38.3606, and 62.4310 for Edo; 12.3765, 25.3043, 38.4592 for Ekiti; 12.3814, 27.0007, 50.2894, 68.5130 for Ogun and 12.3560, 25.3143, 38.5582 for Ondo. The peaks at 2θ = 12.3856, 12.3765, 12.3814 and 12.3560 for Edo, Ekiti, Ogun and Ondo respectively gave the distinctive XRD form of kaolin.

The results of the FTIR tests that were used to confirm the kaolinite presence in the samples are shown in Figure 9 of kaolin clay. Samples show the distinctive bands of kaolinite: OH− in the range of 3670-3500 cm^{-1}; Al–OH vary from 914 to 912 cm^{-1}; Si–O vary from 1008.8 to 1183.5 cm^{-1} and Si–O–Al at 542.02, 542.03, 540.09 and 531.40 cm^{-1} for Edo, Ogun, Ondo and Ekiti, respectively.

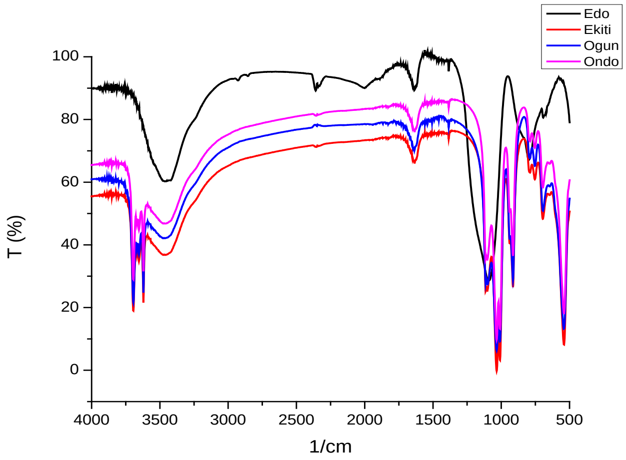

Figure 9. FTIR of Kaolin samples

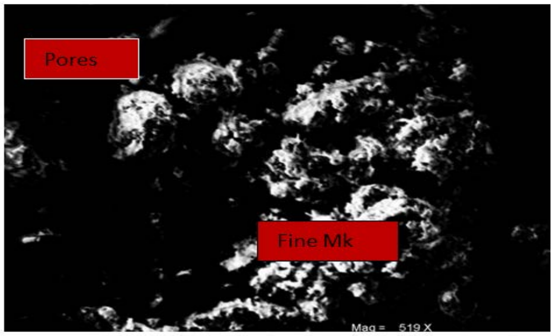

Figure 10. SEM of Edo MK at 100x

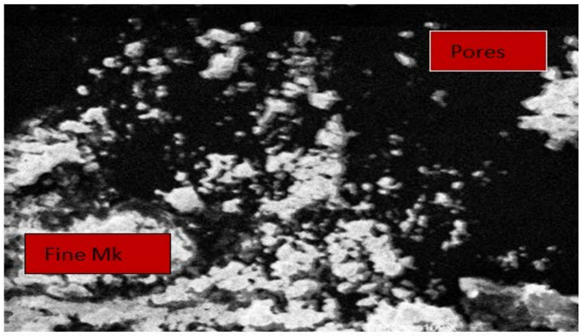

Figure 11. SEM of Ogun MK at 100x

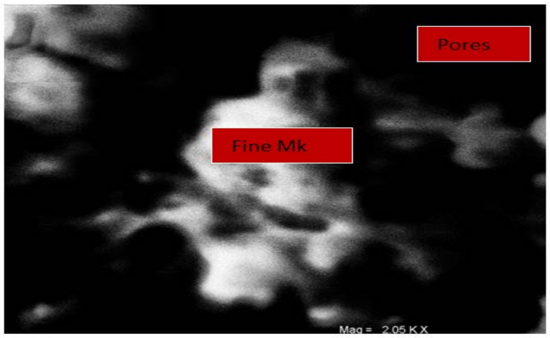

Figure 12. SEM of Ondo MK at 100x

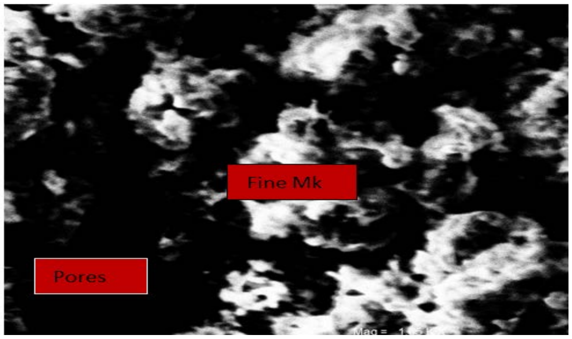

Figure 13. SEM of Ekiti MK at 100x

3.4 Thermal Behaviour of Metakaolin Samples

Figures 10-13 show the microstructure of Metakaolin from the two sample locations; Ogun and Ekiti at 200µm and accelerating voltage of 15kv. The distance between the microscope and the sample (machine microscope distance / working distance) was 15.9mm. The optimal temperature and heating time of 800°C/1hour was adopted and scanning electron microscopy examination (SEM) was carried out on the four samples. Within the metakaolin samples, more fine particles with many residual pores are distributed as presented in the SEM images. The presence of silica, an active and highly porous material with a large internal surface area was observed. The broken Kaolin during calcination exhibits a highly porous structure.

The Fourier Transform Infra-Red Spectroscopy (FTIR) method is much less prevalent for quantifiable determination of clay and other minerals. Results of the FTIR of samples calcined at 800°C/1hour (the optimal temperature and time) are presented in Figure 14. Absence of the detectable Al–O–H bands between 914 - 912 cm^{-1}, and the doublet between 3,670 and 3,500 cm^{-1}, is evident. Absence of the band at 542.02, 542.03, 540.09 and 531.40 cm^{-1} for Edo, Ogun, Ondo and Ekiti respectively and the appearance of a new band at 800 cm^{-1} can be related to the change from octahedral coordination of Al^{3+} in kaolinite to tetrahedral coordination in metakaolinite.

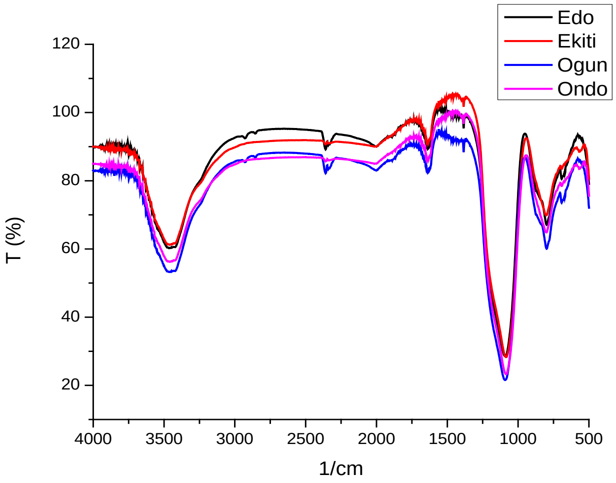

Figure 14. FTIR for Calcined kaolin at 800°C/1hour

The workability of the cement-metakaolin concrete mix as measured by the slump test is shown in the linear graph in Figure 15. It was observed that there was reduction in the concrete workability due to the presence of metakaolin in the mix and greater reduction occurred at higher replacement level. This implies that to maintain consistency and workability of concrete mix, additional water is required due to the high absorption capacity of metakaolin.

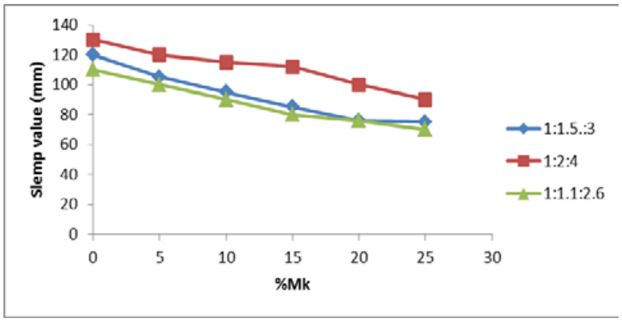

Figure 15. Linear-graph of 0.4w/c slump values

3.6 Compressive Strength on the Cement-Metakaolin Concrete

For all mixes, compressive strength increased with decreasing water-cement ratio, as expected. In prescribed mixes, the compressive strength of control specimen compared with various percentages of Metakaolin shows that the 28- day strengths varied between 19.5 and 32.6 N/mm^2 for mix ratio 1:1.5:3 and between 17.5 and 30.2 N/mm^2 for mix ratio 1:2:4 at different water-cement ratios. Furthermore, the 90- day strengths varied between 21.6 and 34.7 N/mm^2 for mix ratio 1:1.5:3 and between 18.2 and 32.0 N/mm^2 for mix ratio 1:2:4 at different water-cement ratios. The designed mix of 1:1.1:2.6, however, gave the best results with compressive strengths higher than what was obtained in prescribed mixes at all ages.

Based on the results, it is observed in Figure 16 that the Metakaolin admixed mixes attain the highest compressive strength values at 15% replacement due to the presence of higher proportion of calcium silicate hydrates (CSH), a strength-enhancer. However, at replacement level of 25%, the compressive strength of the cubes was lower than the control at all test ages.

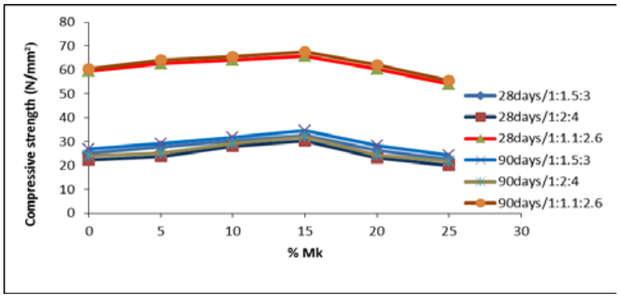

Figure 16. Compressive strength at 0.4 w/c

Furthermore, 15% MK-concretes gained strength most rapidly. It exhibited approximately 85.2, 93.6 and 57.2% increase over 7-day control strength by 28-day for 1:1.5:3, 1:2:4 and 1:1.1:2.6 mix ratios, respectively (see Figure 17). The designed mix gave higher compressive strength when compared with the prescribed mixes. This shows that this designed mix is preferable and can be used to achieve higher compressive strength.

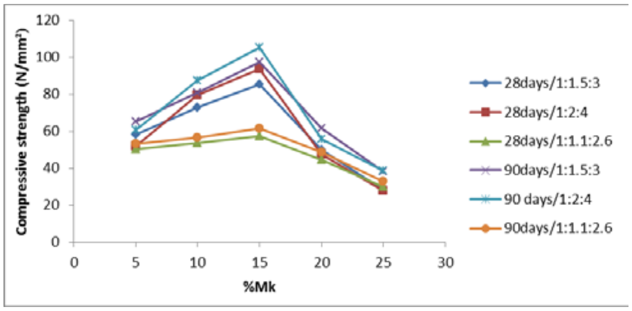

Figure 17. Rate of compressive strength development, shown as a percentage increase over 7-day control strengths for 0.4w/c

3.7 Flexural Strength of Unreinforced Beams

It is observed from the results that Metakaolin incorporation generally increases flexural strength. As the curing age increases, flexural strength increases (see Figure 18). At 90 days, Metakaolin concrete beam gained 69.1% of the 28-day control strength. These results agree with those of Dubey and Banthia (1998) and Qian *et al*. (2001), where modulus of rupture increase when MK was used as partial replacement for cement. Thus, increase in flexural strength in MK-concretes may be related to refinements in pore structure and denser, thinner interfacial transition zones, which means proportionally less of weaker phase.

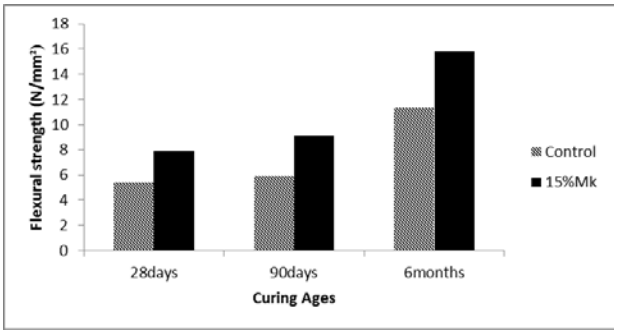

Figure 18. Average peak flexural strength (modulus of rupture) versus curing age

4. Conclusions

Metakaolin, a pozzolanic additive, may be obtained by calcination of kaolin clay. From the thermal analysis, the optimal conditions of the thermal treatment are; calcination temperature of 800°C and holding time of 60 minutes. It should be noted that the geological space where test sample is obtained could alter these conditions and the constituents of the deposit. The presence of other compounds like quartz, Illite and anatase in some kaolin samples does not affect their properties. However, anatase, a titanium oxide compound present in Ogun kaolin sample can cause mild irritation to the nose and throat if inhaled and can also cause irritation to the eyes and skin when contacted while ingestion is not harmful.

Metakaolin helps to reduce the workability of concrete especially at higher replacement levels. This implies that the water absorption capacity of metakaolin is high and additional water is required to maintain consistency and workability of concrete mix. This could be attributed to the larger surface area of metakaolin. Metakaolin admixed mixes attained higher compressive strength values than the control mix up to 20% Metakaolin replacement. However, at replacement level of 25%, the compressive strength of the cubes was lower than the control at all test ages. This is attributed to the "dilution effect". Besides, 15% replacement percentage had higher ultimate strength than ordinary portland cement concrete because of the presence of a strength-enhancer, calcium silicate hydrates.

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