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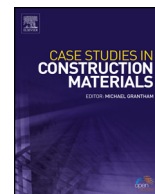
Short communication

The Influence of some calcined clays from Nigeria as clinker substitute in cementitious systems

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A B S T R A C T

This paper examines the possibility of the use of some clays calcined from Nigeria as partial substitutes for clinker in the production of Limestone Calcined Clay Cement (LC^3). The clays were calcined in the oven at a temperature of 800 °C and for one hour. Dehydroxylation of raw and calcined clays with TGA were carried out between 400 °C and 650 °C after which the materials were again characterized with XRD. The R^3 pozzolanic test was then carried out to evaluate the reactivity of calcined clays based on the heat release at 24 hours of a model system of portlandite, calcined clay and limestone with adjusted sulfate and alkali content. After the characterization, two clay samples were deemed fit for use as partial replacement of cement because they contain more than 30% kaolinite content. Concrete cubes prepared with the cementitious blends (PC, LC^3-50A, LC^3-50D and LC^3-35D) were cast for mechanical and durability tests, while mercury intrusion porositometry (MIP) was used to determine the porosity of the cementitious blends. The results showed that LC^3-50D with about 50% kaolinite content gave the best mechanical and durability results followed by LC^3-50A and whereas, LC^3-35D containing only 35% cement can still be used for good construction which is a demonstration of the advantage offered with the use of LC^3 in cementitious systems.

1. Introduction

The use of cement-based materials to produce building materials have increased enormously over the last 50 years, this rate of growth surpassed other building materials. Contemporary societies have witnessed incredible built environment of different shapes and sizes of construction with cement-based materials. The improved standard of living in most countries have led to the increase in per-capita consumption of cement [1]. Mehta and Monteiro [2] noted that about 18 billion tonnes of concrete would be required globally for construction by the year 2050. The massive concrete requirement for the construction industry globally shows there will be increase in greenhouse gases emission such as carbon dioxide with serious associated economic and environmental issues worldwide due to cement production [3]. It was reported further by UNEP [1] that carbon dioxide (CO_2) generation from the production of cement comes from two sources: combustion of fossil fuel during clinker production which account for less than 40% of total CO_2 generated and the decomposition of limestone ($CaCO_3$) during calcination. The total CO_2 generated in the process is put at 842 kg CO_2/tonne.

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The rise in the demand for cement because of growth in infrastructural and other needs in the developing countries indicate that anthropogenic greenhouse (GHG) gases such as CO_2 will continue to rise. This is one of the key reasons why limestone use must be reduced in cement production to minimize CO_2 released because over 60% of CO_2 emitted is in the process of calcination [4]. The rise in infrastructural development in the developing countries have positively impacted the built environment and led to the rise in cement consumption with attendant rise in CO_2 emission that may continue to rise to an unprecedented level by the year 2050 according to UNEP [1] and Nigeria is not an exception. Cement will continue to be dominant construction material for a long time to come. Therefore, efforts are being made to reduce the CO_2 emission from cement production with the use of supplementary cementitious materials (SCM) such as fly ash (FA), ground granulated blast-furnace slag (GGBS), natural pozzolans and limestones among others. Averagely about 20% of SCMs are incorporated as substitutes for clinkers in cements produced nowadays. However, good quality FA and GGBS availability are limited to about 25% worldwide according to UNEP [1]. Other agro-based materials such as rice husk have been investigated for use in concreting by Akindahunsi and Alade [5], while Akindahunsi and Schmidt [6] and Akindahunsi [7] examined the influence of starch on shrinkage creep of concrete with very promising results. SCMs are however, limited by production capacities and availability in various parts of the world.

This shows there is the need to explore others SCMs for probable use as substitute in cement production to reduce CO_2 emission, since cement will continue to be in great demand in developing countries of the world especially. A recently developed tenary cement named Limestone calcined clay cement (LC^3) that can attain comparable strength to Ordinary Portland Cement (OPC) with clinker content as low as between 40% - 50% was originally developed by Scrivener et al. [8]. In this tenary cement, the remaining quantities of the cementitious system is completed with a blend of crushed limestone, calcined clay, and gypsum. The use of LC^3 in concrete production reduces CO_2 emission, energy requirement for clay calcination is about half the required amount for clinker calcination. This will bring down the cost of production and gives credence to environmental sustainability. Limestone is available in substantial quantities in most parts of the world and clays are in abundant globally. Clays required for use in LC^3 in blending are not as substantial so clays can still be available for other needs. It has been noted that the clays required are not supposed to be clays with very high content of kaolinite as this may not be readily available.

The need to seek alternative replacement for cement due to consistent rise in cost of the material in Nigeria with no sign of the problem abating anytime soon resulted in investigating the use of Nigerian clays for LC^3. Gushit et al. [9] pointed out that kaolinite clay deposit in Nigeria is more than three billion metric tonnes. GTI Research [10] puts Nigeria's estimated spending on infrastructural needs by 2025 at $77 billion, based on the annual population growth rate of 3.0% as determined by the Nigerian Bureau of Statistics (NBS) [11,12]. Cement and concrete play pivotal roles in the building of the physical infrastructure. The cement industries in Nigeria now have a combined capacity to produce over 30 million metric tonnes of cement per annum. This, however, has not reduced the cost of cement in the country which is much higher than in many other countries. The high cost of cement, therefore, is a major hindrance to infrastructural development both for government and private developers. The environmental impact of cement production is also a major issue because it contributes a substantial amount of CO_2 to the environment. Climate change is a major challenge in the world today and efforts are being made to reduce the emission of greenhouse gases into the atmosphere. Hence, this study investigates the suitability of clays from Nigeria for LC^3.

2. MATERIALS AND METHODS

2.1. Materials

The five clays containing varying amounts of kaolinite content ($Al_2Si_2O_5(OH)_4$) investigated were mined from five different locations in Nigeria are as shown in Fig. 1 and were designated in the samples as A, B, C, D and E in this study with appearances as indicated in Fig. 2. Portland-limestone cement (CEM II/B-L 42.5 N) from one of the local cement manufacturers in Nigeria was used for the investigation. Additional limestone from Durcal 5 from Omya Company Germany and gypsum were also introduced into the mix proportions.

2.2. Methods

2.2.1. Materials Characterization
The study of the effect of selected clays from Nigeria on LC^3 systems was carried out at EPFL laboratory in Switzerland. The five different clay materials were well spread on trays and labelled appropriately and placed in oven set at $100\,°C$ to dry for 24 hours, after which they were allowed to cool down to laboratory room temperature. The clays were placed and ground in batches in a 10 kg capacity ball mill for 3 hours. Physico-chemical properties of the cement and the five different clays such as particle size distribution was carried out using Malvern particle size analyzer. Surface area of the five samples were measured with the BET method by gas adsorption where surface area is typically calculated from nitrogen adsorption measurements and composition of the samples were determined using X-ray fluorescence (XRF). The clays were calcined in a high-temperature furnace (Borel FP1100) that was set at $800\,°C$ and the calcination process carried out for one hour. Alumina crucibles were used as the containers for putting the clays inside the furnace. Kaolinite content for the raw clays were determined by Thermogravimetric Analysis (TGA) using equation 2 from loss of weight as a result of kaolinite
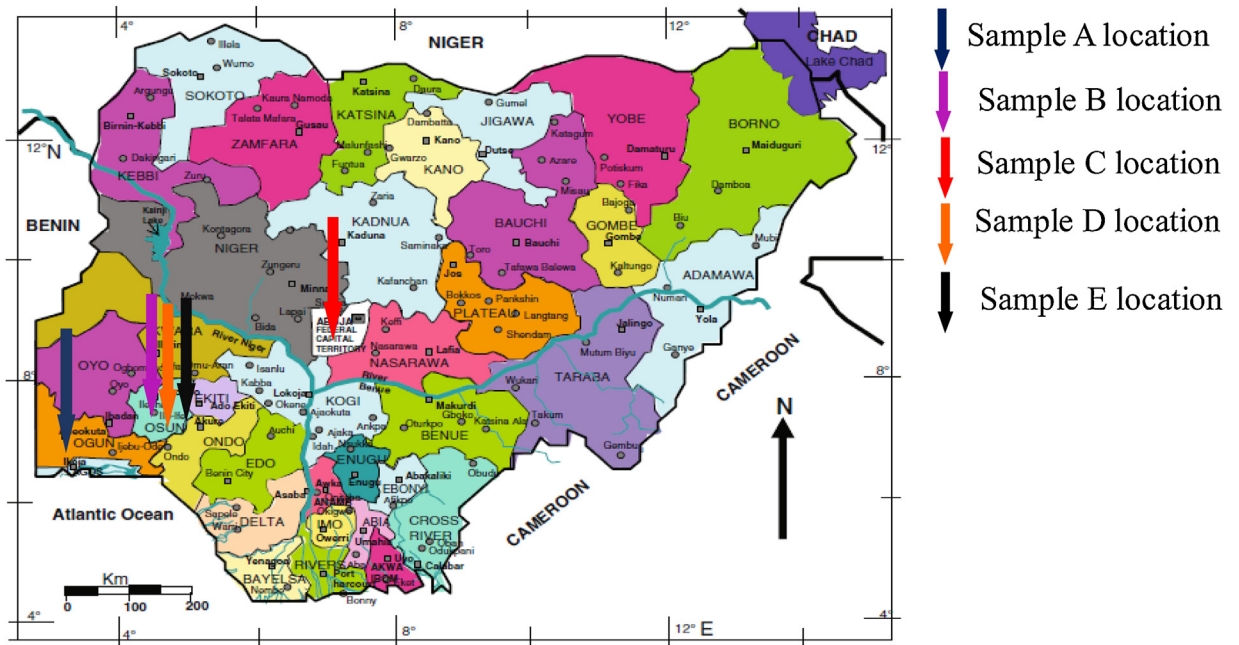
Fig. 1. Map of Nigeria showing clay locations.

Fig. 2. Clay samples.

dehydroxylation (AS_2H_2) that takes place between $400\,°C$ and $650\,°C$. The stated temperature was used to allow for full dehydroxylation of kaolinite in order to get maximum pozzolanic potential of the clay according to [13] & [14] and calcination of the clay leads to metakaolin phase (AS_2) development as given in Equation 1. XRD of the raw and calcined clays were carried out to confirm the crystalline phases in the clays, and XRD phase composition of cement carried out is presented in Table 1.

$$A_2S_2H_2 = AS_2 + 2H \tag{1}$$
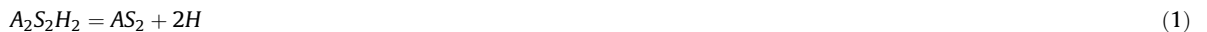
$$wt.\%_{kaolinite} = wt.\%_{kaol-OH} \times \frac{M_{kaolinite}}{2M_{water}} \tag{2}$$

Table 1
XRD phase composition of cement.

Cement	Content (%)
C_3S	45.3
C_2S	5.53
C_3A	4.09
C_4AF	7.98
Lime	0.1
Portlandite	3.07
Calcite	26.31
Quartz	0.51
Gypsum	3.3
Bassanite	2.59

Table 2
: Mix proportion for cementitious systems.

	Cement (kg)	Gypsum from cement (kg)	Limestone in cement (kg)	Calcined clay (kg)	Additional limestone (kg)	Total (kg)	Additional gypsum added (kg)	Potassium hydroxide (kg)
PC	12.24	-	-	-	-	12.24	-	-
LC^3-50	6.15	0.45	2.35	2.82	0.47	12.24	0.061	0.051
LC^3-35	4.30	0.31	1.65	3.81	2.16	12.24	0.061	0.036

Where

$wt.\%_{kaolinite}$ = Weight of kaolinite (%)

$wt.\%_{kaol-OH}$ = weight loss over the kaolinite dehydroxylation

$M_{kaolinite}$ = molecular mass of kaolinite

$2M_{water}$ = molecular mass of water

In order to affirm the accuracy of calcination carried out on the clays, TGA experiments were conducted on the calcined clays to determine the presence of kaolinite (if any) in the clays (Fig. 5). Therefore, metakaolin contents were taken as dehydroxylated kaolinite portions during the process of calcination.

2.2.2. Compressive strength tests for concrete cylinders

The mix proportions for the cementitious systems used in preparing concrete cylinders is shown in Table 2 with additional limestone used in the mix proportion indicated in the table. PC (CEM II) which is the common cement produced by manufacturers in Nigeria was the reference cementitious mix for the concrete without the addition of any calcined clay. The designations LC^3-50 and LC^3-35 indicate that the cement used in the mix were 50% and 35% respectively or stated in another way 50% and 65% replacement of clinker with calcined clay, limestone and gypsum respectively in the mixes. The mix proportions for the concretes were 390 kg/m^3, 875 kg/m^3, and 980 kg/m^3 for cementitious systems, fine and coarse aggregates, respectively. Four different concrete cylinder types were cast, and these are designated as PC, LC^3-50A, LC^3-50D and LC^3-35D. For each of the designated mix three concrete cylinders were cast for compressive strength test after curing in a fog room at 2, 7 and 28 days. Since CEM II was used for the study, thus, calcined clay ratio to limestone used for the investigation was 1:1, taking cognizance of the limestone inherent in the cement. Soluble alkali and sulphate were added to the cementitious systems in order to create an outcome similar to actual blended cement, this was done in accordance with [14], Water cement ratio used was 0.5, and polycarboxylate superplasticizer (Mapei Dynamon SP914 was used for the LC^3-35 mix. Twelve Concrete cylinders of size 110 mm x 220 mm were cast for each test regime: nine of the concrete cylinders were used for compressive strength tests for each routine type, while a set of three each were used for the modulus of elasticity test.

2.2.3. Determination of Modulus of Elasticity

Modulus of elasticity of the concretes (PC, LC^3-50A, LC^3-50D and LC^3-35D) were determined in accordance with [15]. The applied load as required should not be more than 40% of the ultimate compressive strength of concrete, and in this case 30% of the average strength of concrete obtained was applied. A compressive testing machine was used to carry out modulus of elasticity test where a set of three concrete cylinders were used for modulus of elasticity test for each type of concrete cast after curing the concretes for 28 days in a fog room.

2.2.4. Chloride migration test for concretes

The concrete disks used for the chloride migration tests were cut from a set of concrete cylinders cast and cured in fog room for 28 days. Two pieces from the central portion of the concrete cylinders were cut into an average thickness of 30 mm

Table 3
Some physico-chemical properties of calcined clays and cement.

Calcined clay/cement	A	B	C	D	E	Cement
D_{w10} (μm)	0.4	0.4	0.4	6.4	5.8	1.1
D_{w50} (μm)	13.3	18.5	12.5	28.4	25.9	8.6
D_{w90} (μm)	109.2	137.8	43.5	73.2	73.7	31.2
BET specific surface (m^2/g)	34.4	28.9	23.3	11.1	11.9	1.4
XRF compositions (%)						
SiO_2	65.8	64.5	40.3	50.5	65.9	14.8
Al_2O_3	20.5	16.3	18.2	35.2	16.6	4.1
Fe_2O_3	1.7	7.3	22.7	0.5	4.0	3.1
CaO	0.1	1.0	-	-	0.4	62.0
MgO	0.2	0.9	5.0	0.1	1.0	1.9
SO_3	0.1	0.4	0.1	0.1	1.0	0.1
Na_2O	0.3	2.6	3.9	3.8	2.4	0.2
K_2O	2.0	1.4	3.5	-	0.7	0.2
TiO_2	0.1	-	0.1	-	-	0.3
P_2O_5	-	-	-	-	-	2.0
Cr_2O_3	-	-	0.1	-	-	-
MnO	-	0.1	0.2	-	-	0.1
LOI	9.1	6.9	7.7	10.6	7.7	12.4

disks. The two surfaces of the concrete disks were ground with a grinding machine to an average thickness of between 27 – 27.5 mm. The concrete disks were placed in a 0.3 molar NaOH solution for 24 hours to saturate the samples. The circular edges of the disks are coated with epoxy after which they are allowed to dry, thereafter, they were placed in a PVC rings and placed in chloride migration cells firmly tightened. Each chloride cell containing a disk has two compartments (1. upstream that contains 0.5 M NaCl and 0.3 M NaOH, 2. downstream containing 0.3 M NaOH). The upstream compartment has negative electrode and the downstream compartment has positive electrode. The whole set-up was connected to a power source through a computer that captures the flow of current through the disk in each cell over a period of two weeks.

2.2.5. Determination of porosity of sample specimen

Characterization of porosity was carried out using mercury intrusion porositometry (MIP) because MIP gives consistent information about porosity of capillary of the cementitious systems. Preparation of samples was done by exchange of solvent with isopropanol. Porosity measurement for the paste specimens after 28 days of curing were determined and specimens were vacuum dried to avoid carbonation. All the tests were conducted using Porotec 140 and 440 devices, 0.75 g per sample was used for the individual experiment.

3. RESULTS AND DISCUSSION

3.1. Characterization of materials

The results of physico-chemical properties of the five calcined clays samples are as shown in Table 3, particle size $D_{v,50}$ (μm), XRF and BET specific surface (m^2/g). Fig. 3 shows the kaolinite contents in the five (5) clays investigated in this study with samples A, B, C, D and E having 32.0%, 17.4%, 16.9%, 52.0% and 15.2% of kaolinite contents respectively. Since the kaolinite contents in clay samples B, C and E were less than 30%, there was no further investigation of their use in LC^3 systems carried out in this study. Fig. 4 shows the TGA curves of raw and calcined clays. The near flat curves of the calcined clays gave an indication that the kaolinites in the clays have been transformed into metakaolin during the process of calcination. As can be seen from the result of physico-chemical properties of clays and cement in Table 3, the $D_{v,50}$ (μm) indicates the diameter of particles that are less than 50% of mass of the sample and the surface areas of the different clays and cement are as shown in the same table. XRD analysis results of the raw and calcined clays are illustrated in Fig. 5 and the crystalline phases present in the clays is shown in Table 4. From both TGA and XRD curves, the calcined clays do no longer contain kaolinite which is an indication that the clays were well calcined.

3.2. Heat released

Isothermal calorimetry method was used to obtain the heat released for the five clays calcined per gram of solid. The total heat released were compared with heat released of other clays of known percentages of kaolinite contents. The cumulative heat evolved for the different clays per gram of solid is presented in Fig. 6. Comparing clays samples (A, B, C, D and E) with clays of different percentages of kaolinite contents in Fig. 6, the total heat released by sample D has the highest heat evolved of the fives clay samples which is a reflection of the reactivity of the metakaolin in the samples. Therefore, samples A and D were the clay samples that were further tested for other properties and suitability for use LC^3 systems.
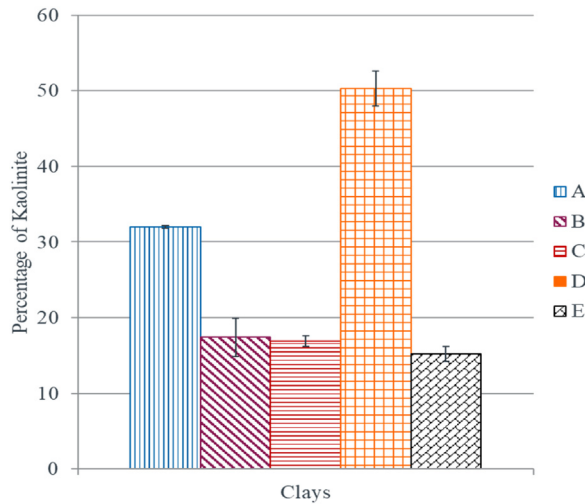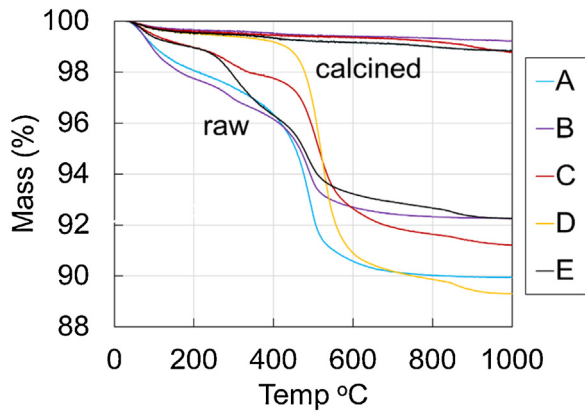
Fig. 3. Kaolinite content in clays.

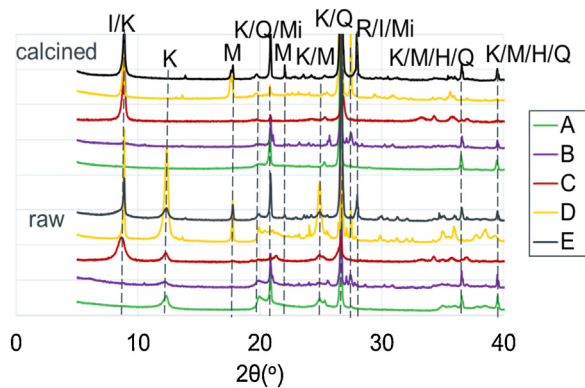**Fig. 4.** TGA curves for raw and calcined clays.


**Fig. 5.** Qualitative XRD patterns of Clays.

3.3. Compressive strengths of concretes

The results of compressive tests of concretes (PC, LC^3-50D, LC^3-50A and LC^3-35D) is presented in Fig. 7. The results after two days of curing shows the reference concrete (PC) with the highest strength when compared with other concretes (LC^3-50A, LC^3-50D and LC^3-35D). However, after seven days of curing the LC^3 concretes shows remarkable improvement in

Table 4

Phases in Clays from XRD patterns.

Constituents	A(%)	B(%)	C(%)	D(%)	E(%)
Hematite (H)	-	0.8	7.4	-	-
Illite (I)	-	6.0	-	-	-
Microcline (Mi)	-	13.6	3.4	13.3	4.6
Muscovite (M)	-	-	26.4	3.7	-
Rutile (R)	0.4	1.1	-	1.0	-
Quartz	33.3	27.4	2.7	1.5	44.4
Amorphous	65.3	51.0	60.1	80.6	40.4
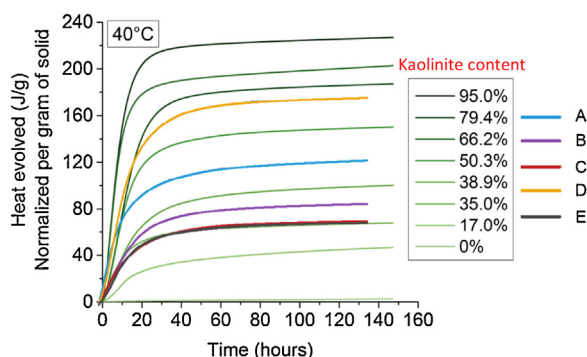
Fig. 6. Heat released for calcined clays-portlandite blends with limestone.
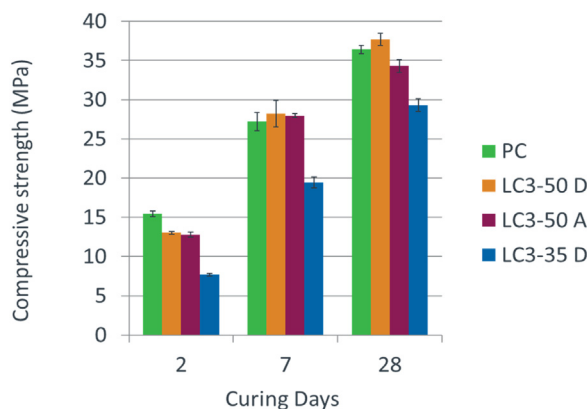
Fig. 7. Compressive strength of concrete cylinders.

strength development especially LC^3-50D and LC^3-50A when compared to the reference concrete. This shows that the pozzolanic properties of the LC^3 concretes have been activated and its reactivity lends credence to increase in strength of the concretes as seen in Fig. 7. After twenty-eight days of curing, the compressive strengths of the concretes (PC, LC^3-50D, LC^3-50A and LC^3-35D) are 36.4 MPa, 37.7 MPa, 34.3 MPa and 29.3 MPa respectively. It can be seen that LC^3-50D concrete with 50% kaolinite content had higher strength than PC and even LC^3-50A with only 32% kaolinite content gave a good result also. It should be noted that LC^3-35D having 50% kaolinite content with only 35% clinker gave a promising result of concrete strength of more than 25 MPa after 28 days of curing. It can be seen from Fig. 7 that LC3-50A developed better strength in comparison with the reference PC at seven (7) days. This may be due to the availability of aluminate from calcined clays which accelerate reaction at early ages, however, the effect of its depletion can be seen from the results of twenty eight (28) days with a drop in strength of LC3-50A in comparison to PC. It should however be noted that the kaolinite content in LC3-50A is only 32%, and for it to give comparable strength to PC shows even clays with low kaolinite content can still provide adequate properties for concretes.
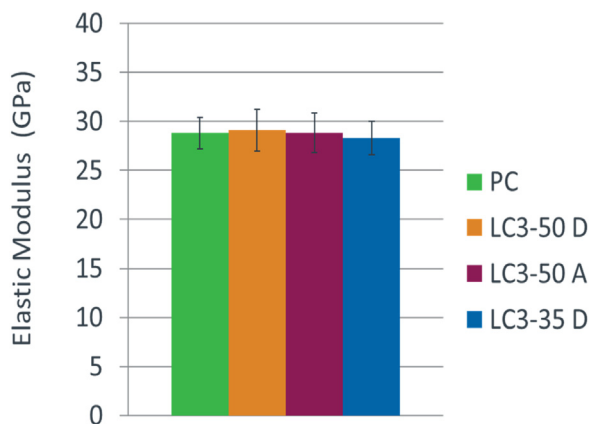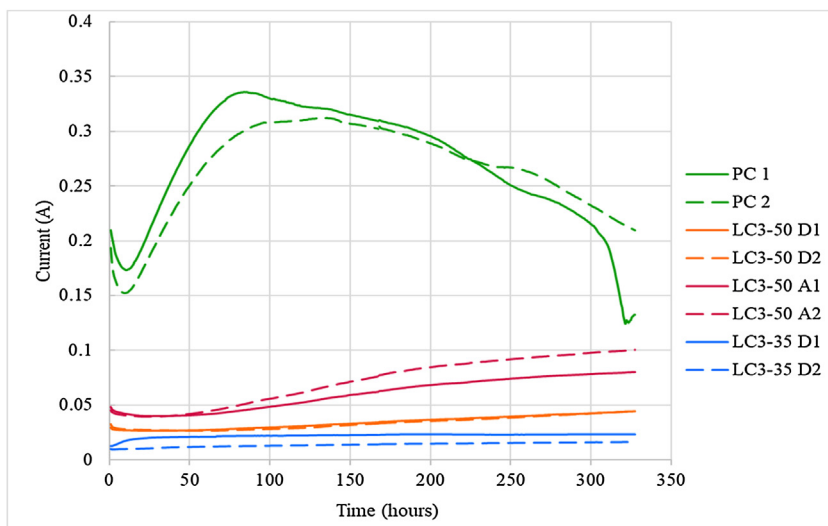
Fig. 8. Elastic moduli of concretes.

Fig. 9. Chloride migration of concretes.

3.4. Elastic moduli of concretes

The average elastic moduli of the concrete cylinders obtained from the tests carried out is shown in Fig. 8. The elastic moduli result of PC, LC³-50D, LC³-50A and LC³-35D are 28.8 GPa. 29.1 GPa, 28.8 GPa and 28.3 GPa respectively. It shows no significance differences in the elastic moduli of the concretes, with LC³-50A which has only 30% of kaolinite having the similar elastic modulus with the reference concrete. It is an indication that LC³ systems with even 30% level of kaolinite content can offer comparable results with normal conventional concretes.

3.5. Chloride migration

The result of chloride migration test for the concretes is presented in Fig. 10. It is obvious from Fig. 9 that chloride migration in LC³ systems were much lower than that of the reference concretes which are PC 1 and 2, this is probably due to pore refinement by LC³ systems as reported by [13]. Even though LC³-35D is shown to offer better resistance to chloride migration from the obtained results this may not be a complete reflection of performance of the cementitious system. The results obtained from mercury intrusion porosimetry shown in Section 3.5 shows LC³-35D to be more porous than PC and weaker in strength (Fig. 7), hence the need for further investigation in this area of work.
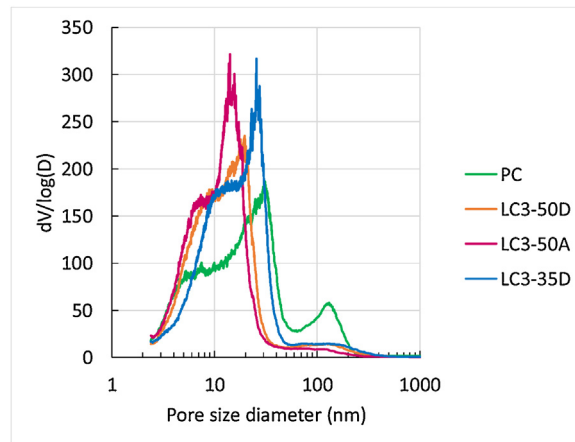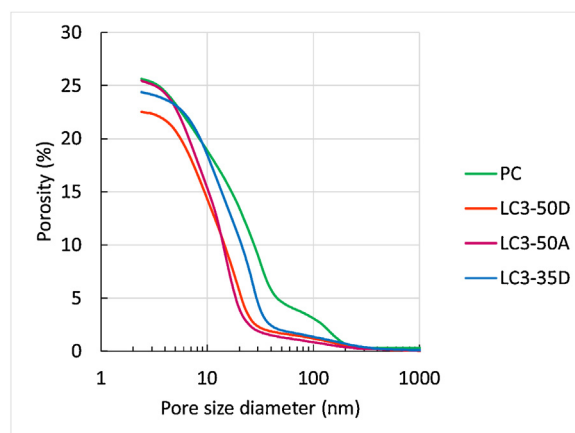
Fig. 10. Differential pore diameter distribution.

Fig. 11. Pore diameter distribution.

3.6. Mercury Intrusion Porosimetry (MIP) results

Porosity results obtained from mercury intrusion porositometry (MIP) measurements are as presented in Figs. 10 and 11. Fig. 10 shows the derivative of pore size distribution or porosity spectrum of the pastes. LC^3-50A and LC^3-35D showing higher peaks than LC^3-50D and PC with smaller peaks, respectively. The smaller peaks are an indication of smaller pores, however, the average pore size LC^3-50D, LC^3-50A and LC^3-35D are much smaller compared with PC. Pore refinement is significantly demonstrated in LC^3 blended pastes as shown in Fig. 11 and this is supported by Avet and Scrivener [14]. Entry pore radius that is critical is kink (inflexion point) of the cumulative curve (Fig. 11) or topmost crest of the derivative curve (Fig. 10). For the blends LC^3-50A and LC^3-50D, they are with the range of 10 - 20 nm, LC^3-35D is within 20 – 30 nm and PC is between 30 - 40 nm This is however, greater than the results obtained by Avet and Scrivener [14] and [16] which may be due to particle sizes of the materials used. Refinement as presented in Fig. 10 improves with LC^3 blended cementitious systems with LC^3-50A and LC^3-50D showing the best pore refinements with the least percentage of porosity shown by LC^3-50D blend which is a confirmation of the finding of Avet and Scrivener [14]. This is also a confirmation of the fineness the clay as shown in Table 3. All the LC^3 blended pastes have pore radii thresholds that are lower than that of PC. A look at LC^3-50A which contains 30% of content shows a good pore refinement to that of PC and even LC3-35D which contain only 35% of cement exhibits better pore refinement in comparison to PC with porosity percentage of about 25% as illustrated in Fig. 11, which is less than that of PC. Pore refinement is critical to durability of concrete as it will inhibit ionic movement within the pores because of the connectivity refinement and LC^3 blends offer improvement in connectivity of pores as seen in Figure 12 and as illustrated in chloride migration test result in Fig. 9.

4. Conclusions

This study shows that Nigerian clays with 50% kaolinite gaves good or in some cases better mechanical properties when compared with the reference concretes especially after 7 days of curing. Clay with kaolinite content greater than 32% (LC^3-50D and LC^3-50A) performed significantly well in the tests carried out. The outcome of the results of LC^3-50A in the tests is an evidence to the fact that clays with kaolinite content greater than 32% can be used in the cementitious blends. Pore refinement of LC^3 systems as demonstrated in the study gives confidence and credibility to the use of LC^3 systems in concrete not only for purpose of strength in concrete but also for durability. It shows Nigerian clays with kaolinite content of over 32% can be used in LC^3 system, it will reduce the greenhouse gas emission such as CO_2 and bring down the cost of cement production and ultimately cost of construction.

Declaration of Competing Interest

The authors report no declarations of interest.

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