



## Characterization and Effects of Nanosilica on Consistency and Setting Times of Metakaolin Blended Cement Mortar

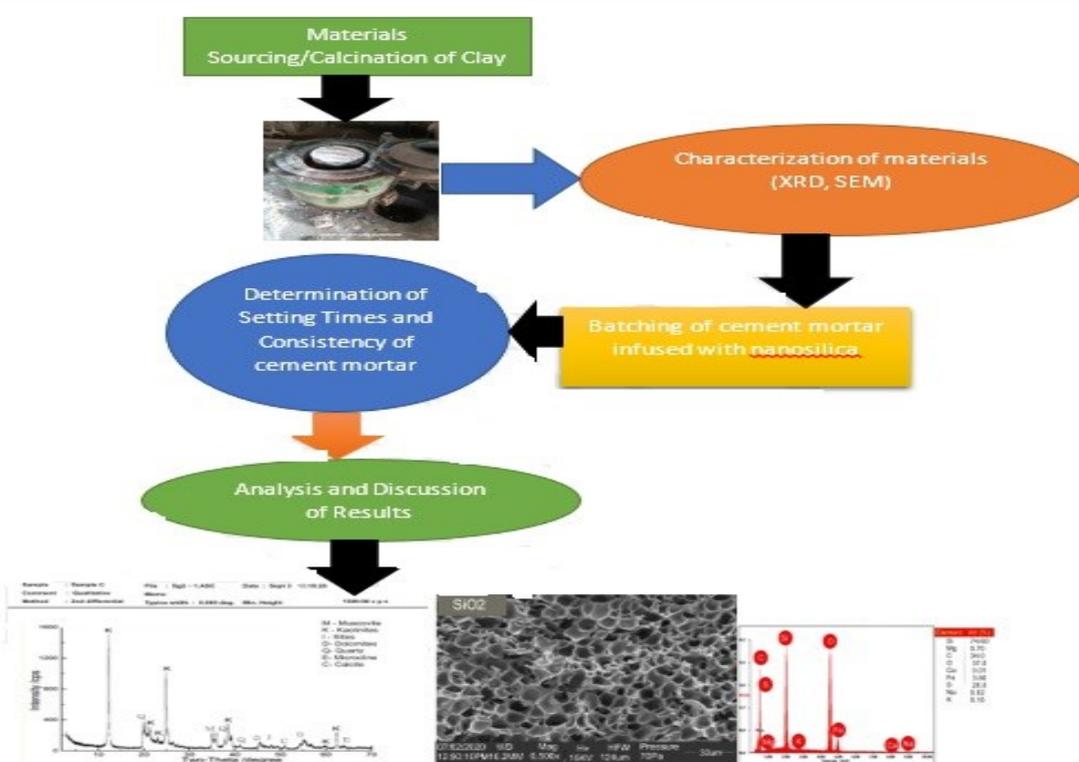
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### Highlights

- Factory blended cement as compared to previous work where the metakaolin are mixed on site
- Kaolin sourced from three (3) different locations as compared to previous researches where kaolin was obtained from a location
- Nanosilica was synthesized from extract of kola pod via green chemistry
- Consistency and setting times of mortar were determined

### Graphical Abstract



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**Abstract**

The idea of sourcing for innovative construction Supplementary Cementitious Materials (SCM) in concrete or mortar cannot be overemphasized. Metakaolin has always been used to replace cement in concrete or mortar batching on site. This research investigated the properties of factory produced blended cement using metakaolin obtained from three different sources and consider effects of nanosilica on blended cement mortar. The kaolin clay sourced from Ijero, Ikere, and Isan in Ekiti states were calcined at 700 °C for 1 hour and thereafter, characterized using X-Ray Fluorescence Analyser (XRF) and X-Ray Diffraction Analysis (XRD). Ordinary Portland Cement (OPC) clinker and 10% metakaolin were used to produce blended cement. Liquid nanosilica was synthesized from cola pod extract and silica precursor in ratio 1:5. A cement-sand ratio of 1:3 and water/binder ratio of 0.5 were used to produce metakaolin blended cement mortar with addition of nanosilica in varying percentages of 1, 2, 3, 4 and 5% by weight of the binder. The setting times and water demand of the blended cement mortar were determined. The results revealed that the setting times and water demand were observed to reduce as the percentage of nanosilica was increased to a level of 4%. In addition, results of the XRF classified metakaolin as class N pozzolans with the addition of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> greater than 70%. Also, the SO<sub>3</sub> for all the clay was found to be less than 4% and the peak intensities as reflected on the XRD showing the amorphous nature of the silica.

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**1. Introduction**

Blended cements are hydraulic cements consisting essentially of an intimate and uniform blend of Portland cement and one or more inorganic materials which could either be agricultural wastes or industrial by-products like fly ash (FA), rice husk ash, silica fume (SF), calcined clay, ground granulated blast furnace slag, metakaolin, etc [1]. They are obtained by intergrinding ordinary Portland cement clinker with supplementary cementitious materials (SCM). SCMs are by-products of other processes or natural materials. Blended cements have been shown to have both technical and environmental benefits. They influence the characteristics of hardened concrete via hydraulic or pozzolanic activity. SCMs are either used as admixture or additive either partly or fully in the production of high strength and impermeable concrete or mortar [2].

Metakaolin is a compound of majorly aluminosilicate obtained as a result of roasting of kaolin clay at temperature range of 700-850 °C [3]. This calcination temperature is lower than that of cement production (1450 °C) and therefore, reduces the amount of CO<sub>2</sub> emission and energy consumption [4]. A very reactive MK with high pozzolanic properties could be ob-

tained at a calcination temperature of 700 °C for 1 hour [5-7]. MK has been discovered to be a pozzolanic material of good prospect in the construction industry due to its high silica content thereby classifying it to be an SCM with tendency of producing high performance concrete or mortar [8]. Metakaolin shows high reactivity when used in combined for with cement with corresponding enhancement in strength and durability properties of the composite. This makes metakaolin advantageous over other pozzolans [9]. In addition, the replacement of cement with metakaolin had reduced the application of limestone in the production of cement and consequently, reduced the emission of greenhouse gases that could pollute the environment [10]. The uniqueness of kaolin as compared to other clay minerals is as a result of high abundance of quartz, pyrite, siderite, feldspar, rutile [11]. The benefits of metakaolin as SCM which other pozzolans lack include: dilution effect, filling ability and enhancement in the rate of hydration [12].

Nanoparticles are small particles with a size under 10<sup>-9</sup> m produced from the agglomeration of atoms and molecules to produce large-scale materials. Nanoparticles have large surface area/volume ratio thereby aiding their reactivity

[13]. According to Kanad [14], nanoparticles can be broadly classified into (natural and artificial nanoparticles). Natural nanoparticles are materials possessing biological systems like substances in bone matrix and viruses. The artificial nanomaterials are synthesized from different experiments and can be classified into four (4) different categories: carbon based, metal based, dendrimers and composites [14]. The metal nanoparticles are synthesized using some of the following precursors: oxides of metals like silicon dioxide, titanium oxide, aluminium oxide, zinc oxide etc depending on the type of nanoparticle to be produced [15].

Naresh [16] opined that the mechanical properties of concrete were greatly enhanced at 10% metakaolin replacement in the properties of concrete containing both metakaolin and silica fume. In addition, Regina [9] worked on the effects of incinerator residual ash-metakaolin blended cement on the hydration and properties of cement. It was observed that the optimum percentage replacement of metakaolin was 10%. However, the durability and compressive strength were found to be high in the case of production of Hybrid Cementitious Composite (HCC) containing 10% metakaolin, 1% for both nanosilica and epoxy. The incorporation of nanosilica enhanced the refinement of pores thereby reducing the porosity and permeability, hence better strength [17].

Nanosilica has been shown to increase the rate of cement hydration with consequent enhancement pore reduction [18]. Nanosilica has proven to be an effective mineral addition for Portland cement with mechanical performance [19], [20] and high pozzolanic activity, which enables the nanosilica to have a high lime-fixation capacity [20]. The problem of inadequate homogeneity of onsite mixing of metakaolin and cement in the production of concrete has been a major concern of the construction industries. Also, the delay in early strength development attributed to pozzolans when used as SCM in composite require adequate attention. This research involves

incorporation of metakaolin obtained from different sources into OPC clinker at the factory during cement production as well as addition of nanosilica into the blended cement mortar to improve its properties.

## 2 Materials and Methods

### 2.1 Materials sourcing

The kaolin clay was sourced from three (3) different locations: Ijero (Lat: 7.829°N, long: 5.056°E, elevation: 466 m) represented as sample A; Ikere (Lat: 7.4997°N, long: 5.2445°E, elevation: 378.8 m) as sample B; and Isan (Lat: 7.946°N, 5.325°E, elevation: 561.8m) as sample C all in Ekiti State, South West Nigeria. The clinker and Gypsum were obtained from Lafarge, West African Portland Cement (WAPCO) factory, Sagamu, Ogun State. Cement mortar was produced from a CEN standard sand in accordance with BS EN 196-1[21], obtained from Lafarge, Sagamu, Ogun State, Nigeria. The nanosilica was biosynthesized from silica dioxide precursor using extract of kola nut pod at the Laboratory of Industrial Microbiology and Nanobiotechnology, Ladoke Akintola University of Technology, Ogbomoso, Nigeria.

### 2.2 Methodology

The kaolin clay was calcined at 700 °C for one hour in a muffle furnace equipped with temperature control device (Figure 1) at the Department of Mechanical Engineering



Fig. 1: Calcination of kaolin in muffle furnace

Workshop, Kwara State Polytechnic, Ilorin, Nigeria. The calcined clay was allowed to cool and sieved to 45  $\mu\text{m}$  particle size. The characterization of the metakaolin was done in the Laboratory of National Geosciences Research, Kaduna for XRF (EDX3600B X-

ment, an indication of synthesis of nanosilica. Three sets of specimens were produced; 100% cement mortar, metakaolin blended cement mortar and metakaolin blended cement mortar with nanosilica. Binder was mixed with sand at a ratio of 1:3 while water/

**Table 1: Selected bands in DNA and PPy/DNA spectra and their assignments**

Chemical Compounds	Percentage Composition (%)		
	Sample A (Ijero)	Sample B (Ikere)	Sample C (Isan)
SiO <sub>2</sub>	65.10	59.90	66.40
Al <sub>2</sub> O <sub>3</sub>	21.43	24.54	20.01
Fe <sub>2</sub> O <sub>3</sub>	0.18	0.59	1.01
CaO	0.00	0.066	0.090
MgO	0.00	0.00	0.00
SO <sub>3</sub>	1.15	1.44	1.30
Na <sub>2</sub> O	8.00	0.00	0.021
K <sub>2</sub> O	0.00	0.00	0.33
LOI	2.00	12.74	11.02
$\Sigma$ (SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> )	86.71	85.03	87.42

Ray Fluorescence) to determine the chemical composition and in the Department of Geology, University of Ibadan, Nigeria for both XRD (EMPYREAN-0000000011078671) analysis and Scanning electron microscope (SEM) analysis. The production of the MK blended cement was carried out at Lafarge, (WAPCO) Sagamu Plant, Ogun State, Nigeria. The metakaolin blended cement was produced by intergrinding 10% metakaolin being optimum as reported in previous studies [16, 22, 23] with Portland cement clinker during the cement manufacturing process. The kola nut pod extract was made by dissolving 1 g of powdered pod in 100 ml of distilled water. The solution was kept in water bath at 60 °C for 1 h and thereafter, filtered and centrifuged for 20 min at a revolution of 4000 rpm. The clear supernatant decanted to give the extract. Thereafter, 1 ml of the extract was allowed to react with 5 ml of prepared silicon dioxide solution and placed under ambient temperature for a duration of 3 h for colour develop-

ment, an indication of synthesis of nanosilica. Three sets of specimens were produced; 100% cement mortar, metakaolin blended cement mortar and metakaolin blended cement mortar with nanosilica. Binder was mixed with sand at a ratio of 1:3 while water/ cement ratio of 0.5 was selected in accordance with established standard [24-28]. This mixture was used for the preparation of mortar with addition of 1, 2, 3, 4 and 5% by weight of the binder nanosilica. The consistency and setting times of the mortar were determined using vicat apparatus.

### 3. Results and Discussion

#### 3.1 XRF analysis of Metakaolin

The results of XRF of metakaolin obtained from three different sources are presented in Table 1. The results showed that the predominant compound in the samples is SiO<sub>2</sub> with corresponding value of 65.10, 59.90, and 66.40 % for samples A, B, and C respectively. The value for samples A and C were greater than that obtained elsewhere [5-6]. The sum of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub> for A, B, and C are: 86.71, 85.03, and 87.42 %, respectively. This implies that metakaolin obtained from sample C is the most reactive based on the highest value of 87.42% fol-

lowed by sample A and B. However, the high content of sodium oxide could inhibit the reactivity of sample A when in contact with cement. These were greater than 70%, thus placing the material in Class N pozzolan [29]. The values were less than that obtained by

the metakaolin as Class N Pozzolans [29].

**3.2 XRD analysis of Metakaolin**

The results of XRD of the three samples, shown in Figure 2 for samples A, B and C, respectively were obtained in the range of 10°

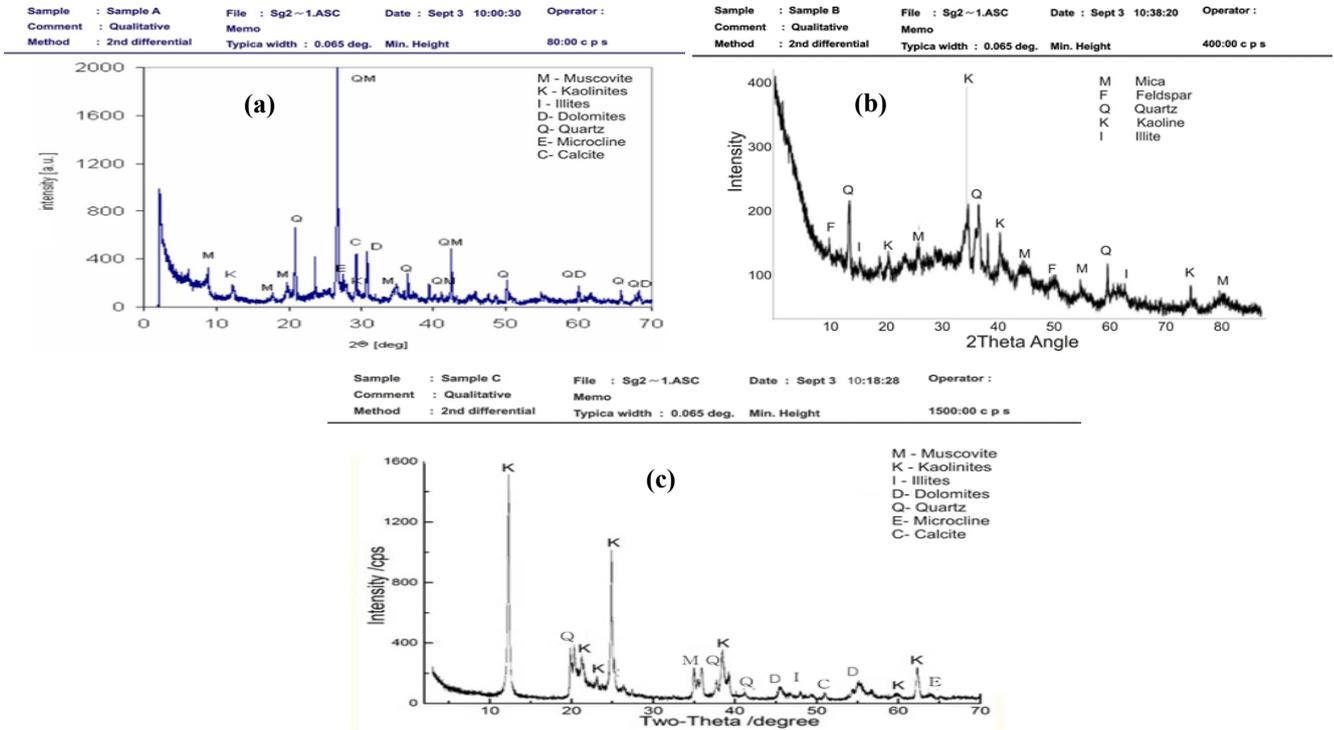


Fig. 2: XRD of Metakaolin (a) A (b) B and (c) C

Akinyele [5] and Ayininuola [6]; this may result into lower pozzolanic tendency. In addition, the percentage of SO<sub>3</sub> for all the three samples are 1.15, 1.44, and 1.3 % respectively less than 4% as required by ASTM C618 [29] which also confirms the classification of

to 80° of 2θ using copper anode material and revealed amorphous silica. The patterns revealed that the samples contain kaolinite clay and Quartz as the most abundant minerals, indicating high silica content. The result is in line with that obtained in a similar study [23]. The XRD graph for sample A pattern as pre-

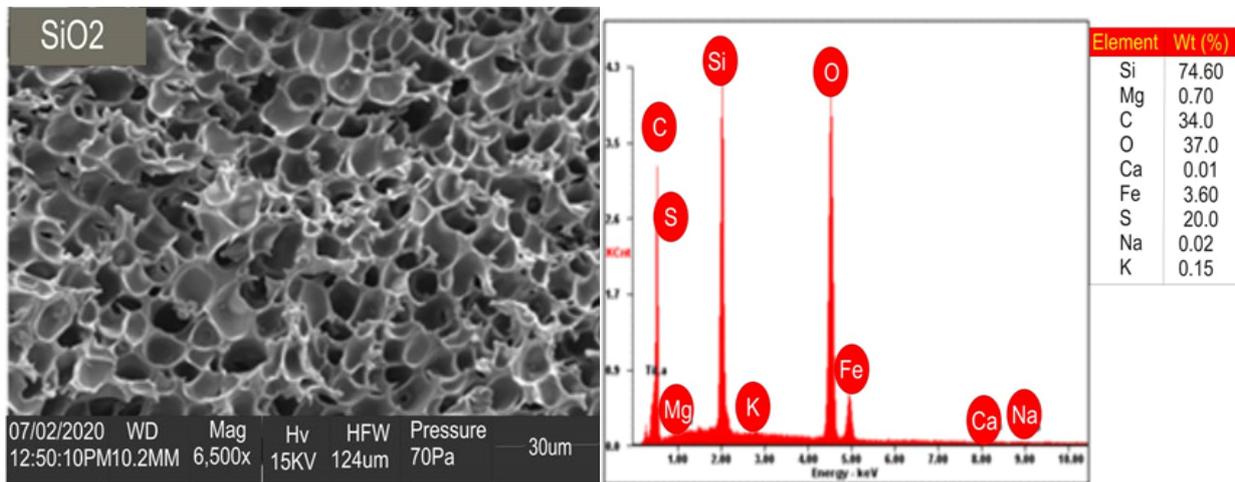


Fig. 3: (a) SEM micrograph and (b) EDX spectrum of nanosilica

sented in Figure 2(a) shows it contains both kaolinite clay and hydrated phyllosilicate; muscovite similar to Nacrite belonging to the group of kaolin clay as described by Olokede

**Table 2: Physical properties of blended cement mortar**

Sample ID	Standard Consistency		Setting Time	
	Water Demand (%)	Scale Reading (mm)	Initial Setting Time (min)	Final Sett. Time (min)
REF <sub>0</sub>	24.6	4	195	260
BCA <sub>10</sub>	25.0	4	180	240
BCB <sub>10</sub>	25.8	5	190	255
BCC <sub>10</sub>	25.4	5	180	240

Note: REF<sub>0</sub>: Conventional OPC  
 BCA<sub>10</sub>: Blended cement containing metakaolin from Ijero Ekiti  
 BCB<sub>10</sub>: Blended cement containing metakaolin from Ikere Ekiti  
 BCC<sub>10</sub>: Blended cement containing metakaolin from Isan Ekiti

[30]. The Figure 2(b) shows the XRD result of Metakaolin B with peak intensities silica rich minerals such as kaolinite, mica, feldspar, and quartz. All these have been reported to be the constituents of metakaolin in another study [31]. The XRD for sample C Figure 2(c) contains kaolinite has the most abundant minerals with considerable amount of silica-based minerals like illites, microcline, muscovite among others. The range of intensities for all the three samples fall within  $2\theta = 14^\circ$  to  $35^\circ$  and hence, affirming the amorphous nature of the silica. This is in agreement with the findings of [32]. Although, the patterns of all the three samples are similar with different intensities at  $2\theta$ . The intensity for sample B starts from  $0^\circ$  and the other two from  $2^\circ$ . This implies that the amorphous phase of silica contained in samples A and C are rapidly attained at smaller angle. Consequently, have greater influence on their reactivity as compared to that of sample B. This is as opined in other studies [32,33].

### 3.3 Characterization of synthesized nano-silica

The SEM image and energy dispersive X-ray (EDX) spectrum of Figure 3 revealed the

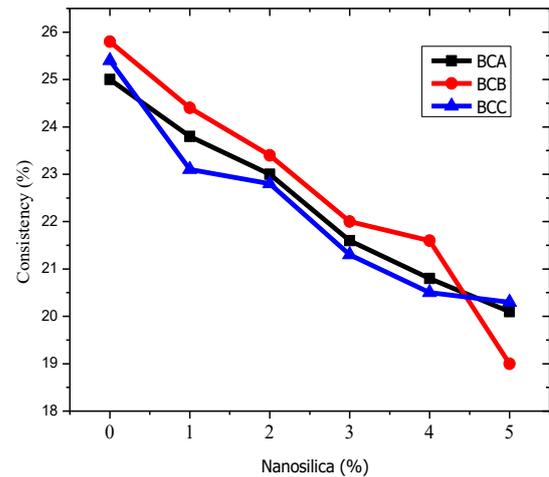


Fig. 4: Graph of cement mortar consistency infused with nanosilica

microstructure and elemental of the nanosilica. According to the SEM micrograph, the nanosilica particles are spherical in shape with  $30 \mu\text{m}$  in sizes. This is similar to the findings of Kamilah [34] where sugar cane bagasse was used for nanosilica synthesis. The EDX shows the most abundant element is silica with relative abundance value of 74.6% greater than a value of 25.74% as in the case of another researcher Yadav [35] where nanosilica was synthesized from fly ash.

### 3.4 Effect of nanosilica on consistency and setting times of blended cement mortar

Figure 4 shows the effect of addition of nanosilica on the workability of blended cement mortar. It could be inferred that as the percentage of nanosilica increases, the water demand for all the metakaolin blended cement mortar was found to reduce accordingly. An indication that minimal water is required to achieve a standard consistency, hence, enhancement in the workability of mortar. This is in agreement with the study elsewhere [36].

However, at 5% nanosilica in the case of sample C, the water demand was found to significantly lower than other sources, changing the trend of the results. This could be attributed to the source and morphological nature of sample B induced during the calcina-

tion process as compared to the other two samples which agrees with previous study [37]. Table 2 shows the consistency of the OPC with a value of 24.6 % lower than that of blended cements. The high-water demand of the blended cements may be attributed to the high surface area to volume of the metakaolin. However, the consistency for both OPC and blended cements were found to be close to 26 % having scale readings in the range of  $(6 \pm 2)$  mm specified by BS EN 196 [38].

Table 2 shows the setting time of OPC which was observed to be greater than that of metakaolin blended cements. This is an indication of enhancement in the rate of hydration exhibited by all the blended cements as compared to the OPC. However, the setting times for OPC and blended met the recommended minimum of 60 mins as specified by BS EN 197 [39]. Figures 4 and 5 show setting times of blended cement mortar infused with nanosilica. As the percentage of nanosilica in-

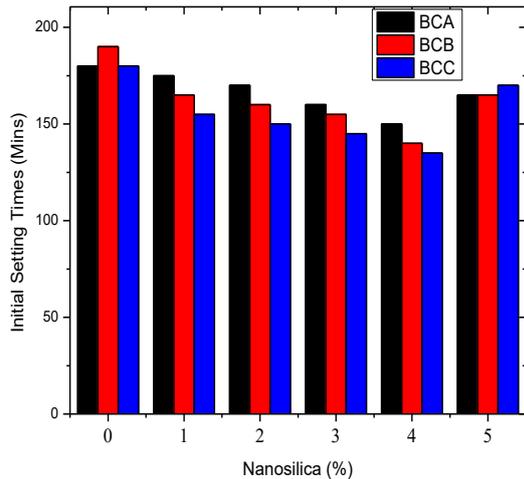


Fig . 5: Graph of initial setting time of mortar against nanosilica

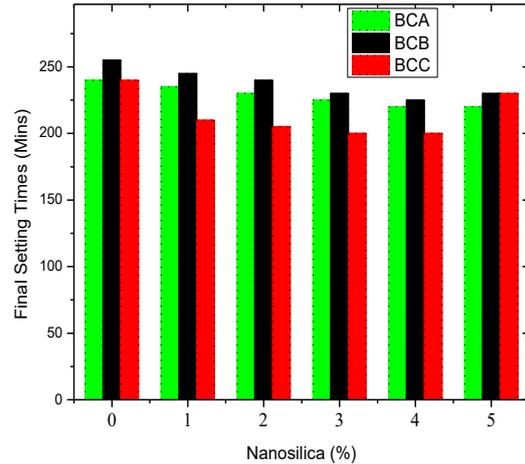


Fig . 6: Graph of final setting time of mortar against nanosilica

creases to a level of 4%, the setting times were observed to reduce with sample C having the least setting times compared to samples A and B, an indication of enhanced rate of hydration and hence, highest reactivity. This could be as a result of short dormant du-

ration and accelerated hydration reaction due to adequate nucleation site produced by the nanosilica and it is in agreement with the findings of other researchers [40,41]. Thereafter, the setting times were observed to increase at replacement greater than 4% nanosilica addition and this may be due to abundant availability of nanosilica than the formed nucleation site for hydration and hence, delay in the hydration reaction. The result is in line with the outcome of the works of some other researchers [14]. However, the result of the setting times is in contrary to the work of Heikal [42] where nanosilica was observed to increase the setting times of slag blended cement mortar. The results of setting times for the three samples of metakaolin blended cements gave satisfactory results as compared to the conventional OPC [43].

#### 4. Conclusion

In conclusion, the metakaolin obtained from the three locations exhibit pozzolanic te-

ndency and all belong to the Class N pozzolan with different reactivity tendency as presented results of characterization and influence on the blended cement mortar. The setting times and consistency were greatly influenced for all the blended cement as compared to the conventional OPC. However, the degree of influence varies according to the source of the kaolin. Sample C gave the best satisfactory results for both setting times and consistency as compared to samples A and B. This implies that the source of metakaolin has great influence on its characteristics.

Furthermore, the addition of nanosilica to a level of 4% was found to reduce the setting times and water demand of metakaolin blended cement mortar. The rate of reduction was observed to be more pronounced as the percentage of nanosilica was increased but at 5%, the setting times tend to increase. The reduction in the setting times implies faster rate of hydration of the cement and consequently, could enhance early strength development of the mortar.

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#### Conflict of Interest

No conflicts of interest amongst the authors

#### Author's contributions

**AA:** Conceptualization, Supervision, Methodology, Investigation, Writing- review & editing, Validation.

**RA:** Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Resources, Visualization, Writing- original draft,

**MK:** Formal analysis, writing- review & editing.

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