Influence of Synthesized Nanosilica on Properties of Wood Ash Cement Mortar

B. D. Ikotun and A. A. Raheem

Abstract The effect of incorporating synthesised nanosilica (NS) on selected properties of wood ash (WA) cement mortar was experimentally studied. The mortar was prepared by adding 1, 2, and 3% NS by weight of binder. The binder in this case is referred to as cement alone and cement replaced with 10% WA. A constant water-binder ratio of approximately 0.4 was maintained for cement pastes samples and 0.5 for mortar samples. Control sample was maintained as sample without the addition of WA nor NS. The tests performed are, setting times, flexural strength and compressive strength. The results showed reduction in setting times of WA cement mortar with 1 and 2% NS. Increased in flexural and compressive strength compared to the control sample were observed with WA cement mortar samples with 2% NS.

Keywords Nanosilica · Wood ash · Mortar · Setting times · Flexural strength · Compressive strength

1 Introduction

Since the invention of Portland cement by Joseph Aspdin through a patent in 1842, there has been extensive research on the material to further improve its development. The use of cement-based materials such as concrete and mortar has contributed tremendously to modern building and civil engineering construction. Cement producing industries consume a lot of energy and have a high rate of carbon dioxide (CO_2) emissions. It was reported by Flower and Sanjayan [1] that production of 1.0 tonne of Portland cement emits 0.8 tonne of CO_2 into the environment. This greenhouse gases emission leads to increase in temperature and causes global warming [2]. Several attempts have been made to reduce energy consumption and

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CO_2 emissions in cement industry. One way that is gaining ground now is partial replacement of Portland cement with supplementary cementitious materials (SCM), some SCM that had been studied are fly ash [3, 4]; rice husk ash [5, 6]; corn cob ash [7]; rice straw ash [8] and wood ash [9, 10]. Wood ash (WA) is obtained by incinerating wood or timber at temperature of 650—700 ºC in about 8 h. Several researchers had worked on the incorporation of WA in concrete and mortar [10–12]. Findings from these studies indicated that use of WA as partial substitution for cement in concrete and mortar has adverse effects on workability and early strength development of the materials, especially at high percentage replacement level (>10%). Dawood et al. [13], alluded to the fact that wood ash can be used in the same manner as fly ash and silica fume in concrete. Hence, there will be needed to improve its effect on concrete's workability and early strength.

The advent of nanotechnology and use of nanoparticles in cement-based materials has played a major role in improving the performance of concrete and mortar incorporating pozzolanic materials like fly ash, rice husk ash and WA. This is attributed to the nano-scale size of the materials, which makes them highly reactive and act as fillers in the pores of concrete and mortar thereby refining their structural characteristics. More than a few nanomaterials had been used in cement-based products amongst which are nanosilica, nano-TiO_2, nano-Al_2O_3, nano-Fe_2O_3 and carbon nanotubes [14–16]. It was reported however that nanosilica is the most widely used nanoparticles in concrete and mortar due to its high pozzolanic activity with calcium hydroxide released during cement hydration [17].

Nanosilica had been shown to improve the properties of concrete and mortar in the fresh and hardened state as well as in short and long term [18–20]. According to Laím et al. [21], nanosilica enhances the microstructure of cement, which improves the pozzolanic reaction. However, the availability and cost of commercially produced nanosilica and other nanomaterials is a source of concern in Africa. This challenge is being addressed through green synthesis of nanoparticles. The biogenic synthesis of silver nanoparticles using pod extract of cola nitida and its application as an additive in paint was studied by Lateef et al. [22]. Also, Emeka et al. [23] evaluated the antibacterial activities of synthesized silver nanoparticles using pineapple. Literature is however scarce on the application of synthesized nanosilica in concrete and mortar. Therefore, this study investigated the characteristics of WA cement mortar containing synthesized nanosilica.)".

2 Material and Methods

2.1 Materials

The materials used in this study were: Suretech Premium Specialist Cement (CEM 1, Grade 52.5); the silica sand used are locally produced, wood ash (WA), synthesized nanosilica and distilled water. Pretoria Portland Cement (PPC) company, South

Table 1 Chemical composition of WA used

Chemical constituents	Percentage composition (%)
SiO_2	61.18
Al_2O_3	7.52
Fe_2O_3	3.19
CaO	11.77
MgO	2.48
SO_3	1.82
Na_2O	1.09
K_2O	3.81
$CaCO_3$	6.22
LOI	3.05
LSF	1.28
SR	4.32
AR	7.55
Total $SiO_2 + Al_2O_3 + Fe_2O_3$	72.58

Africa supplied the cement. The properties of the cement as stated in the product database [24] was presented by Ikotun and Raheem [25]. Silica sand of sizes 0.8–1.8, 0.4–0.85, and 600 μm were used to produce the Local South Africa silica sand (SASS) in proportion of 14:15:8, respectively. SASS was certified as appropriate for the test based on the published grading analysis done by Ikotun and Ekolu [26]. The WA used was collected from Ladoke Akintola University of Technology (LAUTECH) bread bakery in Ogbomoso, Nigeria. The chemical composition of the WA as obtained from Raheem and Adenuga [27] is shown in Table 1. With combined $SiO_2 + Al_2O_3 + Fe_2O_3$ of 72.58% (>70%), the WA is a good pozzolan. The synthesized nanosilica was produced at nanotechnology research group (NANO+) laboratory in LAUTECH. The type 1 distil water used was produced at the water resources section of Civil Engineering Laboratory, University of South Africa (UNISA).

2.2 Synthesis of Nanosilica

The biogenic synthesis of the nanosilica was carried out using a pod extract of Cola nitida as described by Lateef et al. [22]. Approximately 1.5 g of milled pod was suspended in 150 ml of distilled water and heated in a water bath at 60 ℃ for 1 h to obtain the extract. The extract was then filtered using Whatman No. 1 filter paper and centrifuged at 4000 pm for 20 min. The pure liquid extract was then used to synthesize nanosilica. Solution of silicon dioxide (SiO_2) was prepared by adding 0.4 g of SiO2 to 0.6 ml of water, after which the120 ml of the cola pod extract (KP) was added to the solution to synthesise nanosilica. The mixture was carried

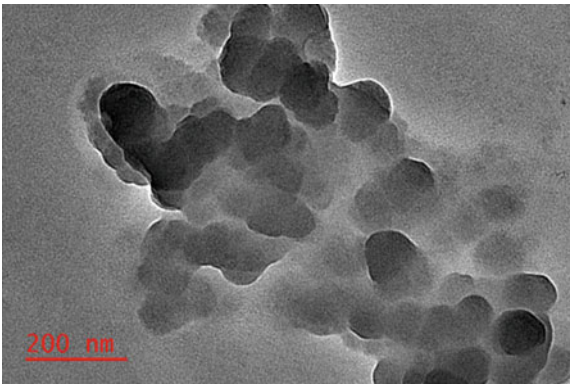


Fig. 1 Morphology of the nanosilica

out under room temperature of 30 ± 2 ºC and allowed to stay for about 2 h. The formation of nanosilica (NS) was observed as a change in colour of the solution monitored visually until it stabilizes. The Nanosilica sample used was observed under the Scanning electron microscope (SEM) to visualize its sizes. The Silica used is confirmed to be of nano size and less than 200 nm as shown in Fig. 1

2.3 Preparation of Specimens

Cement Paste Samples Were Prepared for Testing Setting Times of Cement While Mortar Samples Were Prepared to Investigate the Flexural and Compressive Strength of Mortar. The samples were prepared with 10% by weight of cement WA replacement for cement and 1, 2, and 3% by weight of binder addition of NS. The sample with only CEM 1 and without WA and NS serves as the control. Based on the previous study [26], where is has been reported that 10% WA replacement is the optimum cement replacement for structural purposes, the percentage of WA used in this study was maintained at 10%. In order to maintain the water binder ratio (w/b), NS was added as a percentage substitution for mixing water. This practice was also reported by Berra et al. [28] and Horszczaruk et al. [29]. Cement paste specimen for setting times test were mixed in accordance with the provision in SANS 196–3:2006 [30]. The mix proportion for the samples is presented in Table 2.

The aqueous suspension of NS was stirred with the mixing water for about one minute to obtain uniform dispersion of the nanomaterial before adding to the binder. The cement mixer used was automated to follow the procedure described in SANS 196–3:2006 [30]. The mixing and casting were done based on the described methodology in SANS 196–3:2006 [30]. Specimens for flexural and compressive strength tests were prepared using mortar prisms of size 40 × 40 × 160 mm. The mortar prisms were made according to the SANS 196–1:2006 [31] standard. The binder

Table 2 Mix proportion of cement pastes

Sample number	Sample designation	Cement (g)	WA (g)	NS (g)	Water (g)	w/b
1	CEM 1 (control)	500	–	–	180	0.36
2	90CEM1 + 10WA	450	50	–	192	0.38
3	90CEM1 + 10WA + 1NS	450	50	5	187	0.37
4	90CEM1 + 10WA + 2NS	450	50	10	181	0.36
5	90CEM1 + 10WA + 3NS	450	50	15	177	0.35

Table 3 Mix proportion of mortar specimens

Sample code	Sample designation	Cement (g)	WA (g)	SASS (g)	NS (g)	Water (g)	w/b
A	CEM 1 + SASS (control)	450	–	1350	–	225	0.5
B	90CEM1 + 10WA + SASS	405	45	1350	–	225	0.5
C	90CEM1 + 10WA + 1NS + SASS	405	45	1350	4.5	220.5	0.5
D	90CEM1 + 10WA + 2NS + SASS	405	45	1350	9.0	216	0.5
E	90CEM1 + 10WA + 3NS + SASS	405	45	1350	13.5	211.5	0.5

to sand ratio is 1:3 and water to binder (w/b) ratio is 0.5. The setting time procedure described in SANS 196–3:2006 [30] were followed. The mix proportion of the materials is shown in Table 3.

The fresh mortar was cast in steel mould that can contain three prism samples of size 40 × 40x160 mm. A vibrating table designed for the mould was used to vibrate the sample for proper compaction after casting. After casting, the casted samples were covered with an impervious sheet and placed in a moist air-conditioned environment for 24 h. The specimen was demoulded after 24 h and placed in a curing tank, with maintained temperature of 20 ± 2 ºC.

2.4 Testing of the Samples

Setting Times. The mould made for setting times test was placed in an automatic setting times equipment—ToniSET Compact, Model 7306.100/EN. The equipment was set to capture the initial and final setting times of the cement pastes specimen. The vicat needle EN (Ø 1.13 mm) embedded in the equipment was set to penetrate the specimen at constant interval of 5 min until the final setting time reading was

recorded. The initial and final setting time results were automatically captured on the computer and graphs were drawn from the data automatically.

Flexural and Compressive Strength. At testing age, mortar specimens were removed from the curing tank. The surface water was removed by gently mop the surface with a cloth. The samples were weighed for record purpose and placed on the appropriate load frame of the mortar flexural and compressive press -ToniPRAX, Model. The results obtained for each curing age were the average of 3 prisms for flexural strength and 6 half-prisms for compressive strength.

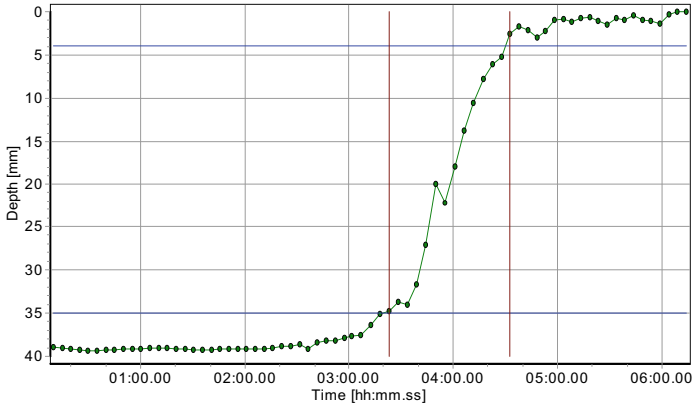
3 Results and Discussion

The results obtained from the various experiment conducted are discussed in the subsequent sections.

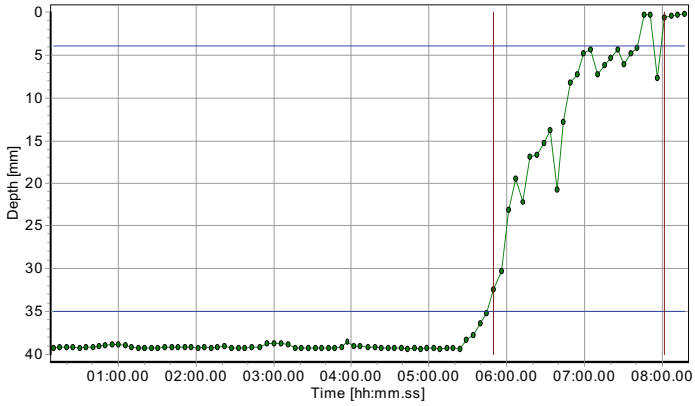
3.1 Setting Times

The effect of NS addition on the initial and final setting times of WA cement mortar in comparison to the control (CEM 1) is presented in Fig. 2(a–e). As observed from Fig. 2a, the initial and final setting times of the control (CEM1) are 203:21 and 273:06 min:sec, respectively. The introduction of 10% WA resulted to elongation of both the initial and final setting times. The initial setting time (IST) increased to 350:02 min:sec while the final setting time (FST) increased to 481:00 min:sec (Fig. 2b). This is typical of pozzolan cement mortar as they have low rate of heat of hydration especially at early ages [32].

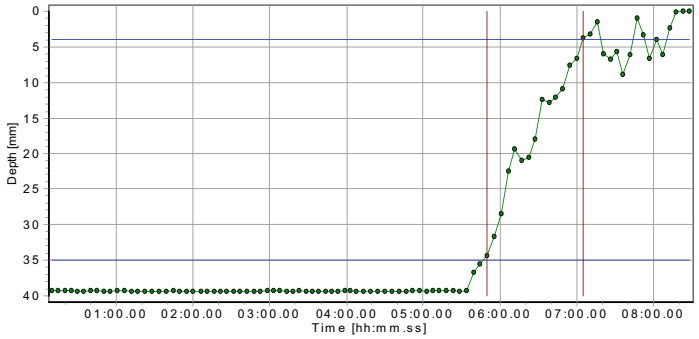
The addition of 1% NS led to decrease in setting times of the WA cement mortar as shown in Fig. 2c. The IST and FST were 349:58 and 425:10 min:sec, respectively. Further decrease in setting times was witnessed with the addition of 2% NS as indicated in Fig. 2d. The IST and FST for 2% NS addition were 342:51 and 418:08 min:sec compared to 350:02 and 481:00 min:sec, respectively for cement mortar with 10% WA substitution. The reduction in setting times is an indication that NS contributed to faster hydration reaction [18]. According to Zhang et al. [33], the reduction in setting time may also be related to the finer particle size and higher surface area of the NS in relation to that of WA. Conversely however, addition of 3% NS resulted in increase of setting times higher than that of 2% NS incorporation with IST and FST of 343:29 and 464:22 min:sec, respectively (Fig. 2e). The increase could be caused by lack of enough calcium hydroxide to react with silica oxide in NS. This increase in setting times is an indication that the 3% dosage of NS is too much and not beneficial to the mix.



IST:203:21[min:sec] FST:273:06 [min:sec]
(a) CEM 1

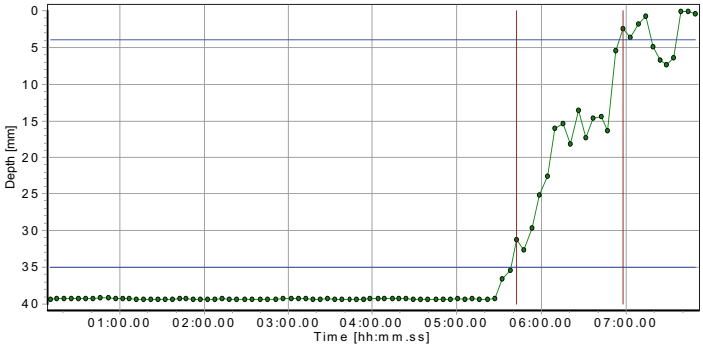


IST:350:02 [min:sec] FST:481:00 [min:sec
(b) 90CEM1+10WA

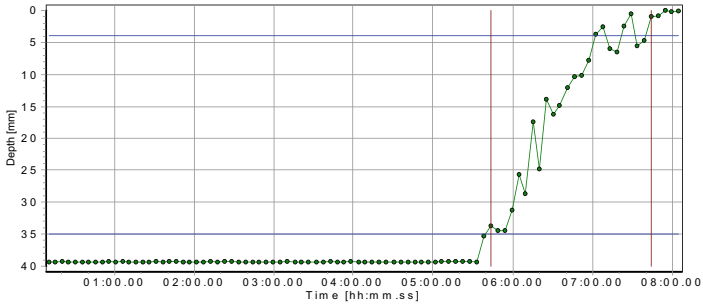


IST: 349:58[min:sec] FST:425:10[min:sec]
(c) 90CEM1+10WA+1NS

Fig. 2 Setting times of WA cement mortar



IST:342:51 [min:sec] FST:418:08 [min:sec]
(d) 90CEM1+10WA+2NS



IST: 343:29 [min:sec] FST: 464:22 [min:sec]
(e) 90CEM1+10WA+3NS

Fig. 2 (continued)

3.2 Flexural Strength

The flexural strength development of WA cement mortar incorporating NS is shown in Fig. 3. When compared with the control (CEM1), the flexural strength of mortar with 10% WA replacement for cement was lower at all curing ages. It could be observed from Fig. 3 that flexural strength of CEM1 mortar at 3, 7, 14, 28, 56 and 90 days were 6.35, 7.20, 7.38, 7.41, 8.04 and 8.11 MPa as against those of 90CEM1 + 10WA which are 3.21, 4.99, 5.56, 5.67, 5.73 and 5.90 MPa, respectively. The WA, which is a pozzolan has low rate of heat development and cement hydration hence, the lower values of flexural strength recorded. With addition of 1% NS, the flexural strength increased over those of 90CEM1 + 10WA with values ranging from 6.13 to 7.45 MPa for curing ages 3–90 days, respectively. For 2% NS addition, the flexural strength was lower than those of 1% NS at early ages but improved at later age (90 days) to surpass it. The improvement is due to continuous pozzolanic reaction of the WA at higher curing ages. The 3% NS addition resulted in lower flexural strength compared to both 1 and 2% NS with values ranging from 5.47 to 7.26 MPa for 3–90 days, respectively. The control (CEM1) has the highest flexural strength for all

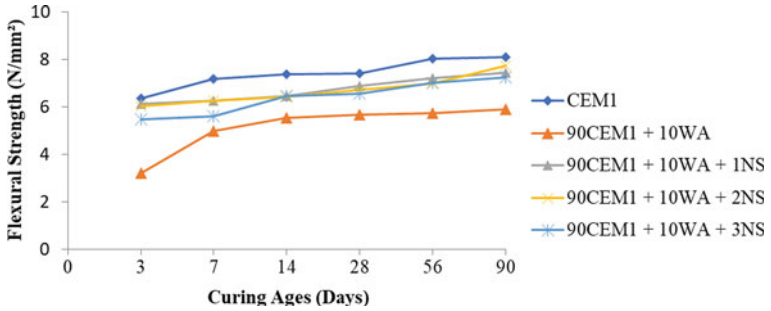


Fig. 3 Flexural strength of WA cement mortar incorporating NS

curing days irrespective of NS addition. This suggests that the effect of pozzolanic reaction of WA at later curing ages is not pronounced for flexural strength.

3.3 Compressive Strength

The influence of NS addition on compressive strength of WA cement mortar is presented in Fig. 4. Generally, the compressive strength increased with curing age and addition of NS up to 2%, beyond this percentage, the compressive strength decreases. As shown in Fig. 4, the compressive strength of mortar made with CEM1 increases from 24.66 MPa at 3 days to 49.72 MPa at 90 days. With WA incorporation, the strength reduces to 14.85 MPa at 3 days and 44.42 MPa at 90 days. This observation is typical to pozzolans and has been linked to the reduction of the rate of heat development and cement hydration caused by pozzolans, especially at lower curing ages [34]. As observed from Fig. 4, WA cement mortar with 1% NS addition recorded increase in compressive strength over that of 90CEM1 + 10WA with values ranging from 15.59 to 49.44 MPa for curing ages 3 to 90 days, respectively. However, these values are lower than those of CEM1 for all curing ages. Addition of 2% NS led to further increase in compressive strength over those of 1% NS addition and 90CEM1 + 10WA mortar. There were 35.2, 27.1, 42.2, 41.5, 39.5 and 20.9% increase in compressive strength of WA cement mortar when 2% NS was added compared to 90CEM1 + 10WA mortar at ages 3, 7, 14, 28, 56 and 90 days, respectively. This increase in compressive strength can be attributed to two factors according to Mohamed [35]. The first is due to the packing effect of small-sizes NS that acted as fillers in the pores of microstructure of mortar to increase its density and also the strength. The second is as a result of the pozzolanic effect that combines silica elements in NS and WA with calcium hydroxide from cement to produce a strong bonding strength (C-S-H), leading to higher compressive strength of the mortar. WA cement mortar with 2% NS performed better than the control (CEM1) at later curing ages. While CEM1 has compressive strength of 45.31, 47.58 and 49.72 MPa at ages 28, 56 and 90 days; 90CEM + 10WA + 2NS recorded 48.13,

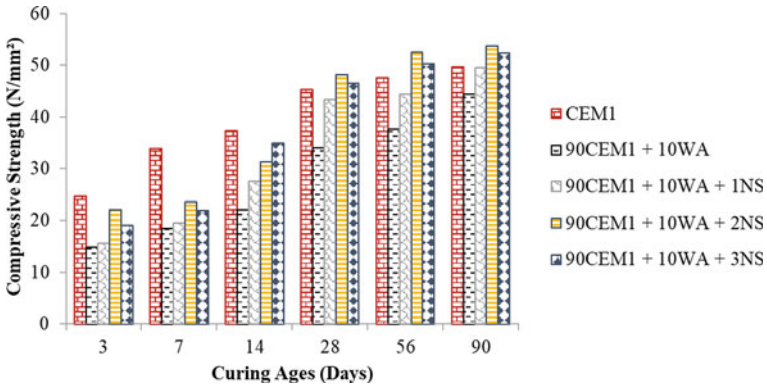


Fig. 4 Compressive strength of WA cement mortar incorporating NS

52.45, and 53.70 MPa, respectively. The increase in strength over that of CEM1 is due to continuous pozzolanic reaction of the WA at longer curing ages [10].

The incorporation of 3% NS into WA cement mortar resulted into reduction in compressive strength when compared with 2% addition, but the values are still higher than those of 1% addition and 90CEM + 10WA. This reduction in strength may be due to agglomeration of NS because they cannot be uniformly dispersed as a result of their large surface area [15]. This confirms that addition of NS beyond 2% of the weight of the binder is not beneficial to WA cement mortar.

4 Conclusion

In conclusion, based on the findings and discussion in this study, incorporation of up to 2% NS by weight of binder reduced initial and final setting times of WA cement paste. Flexural strength of WA cement mortar was increased with addition of up to 2% NS. Also, there was increase in compressive strength of WA cement mortar at various ages up to 90 days, when up to 2% NS was added. Synthesized nanosilica performed favourably in mortar at optimum percentage of 2% and may be used in lieu of commercial nanosilica upon further studies on cost analysis.

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