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Characteristics of Wood Ash Cement Mortar Incorporating Green-Synthesized Nano-TiO_2

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Abstract

This paper presents the findings of an investigation into the influence of green-synthesized nano-TiO_2 on the characteristics of wood ash (WA) cement mortar. Mortar specimens were prepared by partial replacement of cement with WA (10% by weight) and addition of 1, 2 and 3% nano-TiO_2 by weight of binder; using constant water-to-binder ratio (w/b) for all mixtures. The properties evaluated are setting time of the binder and flexural and compressive strength with water absorption of the mortar. The results indicated that addition of 1 and 2% nano-TiO_2 reduced setting times of WA cement paste. Also, the flexural and compressive strength of WA cement mortar were higher with the incorporation of up to 2% nano-TiO_2. The water absorption of WA cement mortar was reduced when nano-TiO_2 was added with 2% incorporation having the best result. The incorporation of NT in WA cement mortar improved its workability and strength characteristics.

Keywords: nano-TiO_2, wood ash, setting times, flexural strength, compressive strength, water absorption

1 Introduction

Wood wastes are commonly used as fuel to generate heat energy needed for various purposes such as cooking, drying and other heat-related processes. Wood wastes are preferred as fuel compared to other agricultural wastes because their incineration produces comparably less ash than other residual materials (Chowdhury 2015a). The use of wood waste as fuel gave rise to the problem of disposal of the ash produced after its combustion. The common method of disposal is land filling or use as soil supplement (Campbell 1990). This could, however, lead to environmental pollution or result to the contamination of groundwater due to the leaching of toxic elements into the water. Thus, a sustainable ash management that will integrate it within natural cycles was proposed (Obernberger 1997). This can be achieved by using wood ash (WA) as partial replacement for cement in concrete and mortar.

Several research efforts were carried out on the use of WA in construction materials to advance a sustainable way of its disposal. Chowdhury et al. (2015b) replaced cement partially with wood waste to produce structural grade concrete. The characteristics of blended cements produced by incorporation of three different types of hardwood were investigated by Raheem and Orogbade (2018). Raheem and Ige (2019) examined the chemical composition and physicomechanical properties of sawdust ash blended cement. Wood ash from bread bakery was used by Raheem and Adenuga (2013) to replace cement partially for concrete production. A study by Raheem and Sulaiman (2013) reported the use of sawdust ash as partial substitution for cement in sandcrete block production. In all these studies, WA behaved excellently well in enhancing the properties of construction materials. WA concrete and mortar exhibited high compressive strength at later ages of curing ($\geq$ 28 days). A study by Cheah and Ramli (2011) indicated that while strength at early age prior to 28 days is sacrificed, the later strength is superior. Thus, low workability and reduced early strength development of WA concrete and mortar pose a problem, especially with high WA content ($>$ 10%). The use of nanoparticles in construction materials

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has brought about improvement in their properties. As reported by Stenfanidou and Papayianni (2012), nanoparticles act as fillers in empty spaces and as crystallization centres of hydrated products, thereby increasing the rate of hydration and strength development. Nanomaterials that are commonly used in concrete and mortar are nano-SiO_2, nano-TiO_2, nano-Al_2O_3, nano-Fe_2O_3, nano-ZnO and carbon nano-tubes (Mohamed 2016; Heikal 2013; Sharobim and Mohammedin 2013; Ren 2018; Raheem and Ikotun 2019). Several of the available studies had focused on the effect of nano-SiO_2 on properties of cement pastes, mortar and concrete. Limited studies are available on the influence of nano-TiO_2 on the characteristics of concrete and mortar.

Nano-TiO_2 not only act as a filler, but also as a reactive element in concrete and mortar. According to Chen et al. (2012), addition of nano-TiO_2 powder significantly accelerated the hydration rate of cementitious materials. Similarly, Nazari and Riahi (2010) affirm that nano-TiO_2 accelerate C–H–S gel formation in concrete. Jalal et al. (2013) examined the effects of fly ash and nano-TiO_2 on rheological, mechanical, microstructural and thermal properties of high-strength self-compaction concrete. An experimental study by Nazari and Riahi (2010) showed improved water infiltration resistance by adding up to 4% by mass of nano-TiO_2. The influence of nano-TiO_2 on physical and hydration properties of fly ash–cement was assessed by Ma et al. (2016) while Praveenkumar et al. (2019) evaluated the mechanical and durability behaviour of blended cement concrete incorporating nano-TiO_2. The present study investigated the properties of WA cement mortar incorporating green-synthesized nano-TiO_2. This is with a view to address the drawback of WA cement mortar by enhancing its workability and structural characteristics.

2 Experimental Investigation

2.1 Materials

For the purpose of this research, the following materials were used: Portland Cement (CEM 1, Grade 52.5); locally produced silica sand, wood ash (WA), green-synthesized nano-TiO_2 and distilled water. Cement was collected from Pretoria Portland Cement (PPC) company, South Africa. The properties of the cement as obtained from the product datasheet (PPC 2018) are given in Table 1. The locally produced silica sand is South Africa silica sand (SASS) prepared from mixture of silica sand of sizes 0.8–1.8 mm, 0.4–0.85 mm, and 600 µm in the proportion 14:15:8, respectively. This was used in place of European silica standard sand (ESSS). Figure 1 shows the grading analysis of SASS and ESSS as obtained from Ikotun and Ekolu (2010). Both SASS and ESSS compared favourably well with fineness modulus (FM) of 3.5 and 3.45, respectively (see Fig. 1). WA was obtained from Mr. Thulani and Sheila Mahlanga compound, 10,372 Utlhanong drive, Kagiso, Riverside, South Africa. The chemical composition of the WA is presented in Table 2. The WA has combined $SiO_2 + Al_2O_3 + Fe_2O_3$ of 72.58% (> 70%), thus it is

Table 1 Typical properties of the Portland cement used (CEM 1 52.5 N).

Chemical composition	Typical results	SANS 50197-1 requirements
Insoluble residue: % by mass	2.0	5.0 maximum
Sulphur trioxide: % by mass	2.0	4.0 maximum
Loss on ignition: % by mass	2.5	5.0 maximum
Chlorides: % by mass	< 0.01	0.1 maximum
physical properties	**Typical results**	**SANS 50197-1 requirements**
Setting times:		
Initial: minutes	125	45 minimum
Final: hours	2.5	No requirement
Specific area (Blaine): m^2/kg	400	No requirement
Compressive strength (mortar prism EN 196-1):		
At 2 days (MPa)	28	≥ 20.0
At 28 days (MPa)	± 58	≥ 52.5 no maximum
Soundness: Le Chatelier Expansion (mm)	1	10 maximum
Densities:		
Relative density	± 3.14	
Bulk density, aerated, kg/m^3	1100–1300	
Bulk density, as packed, kg/m^3	± 1500	
Approximate volume: 50 kg bag, ℓ	± 33	

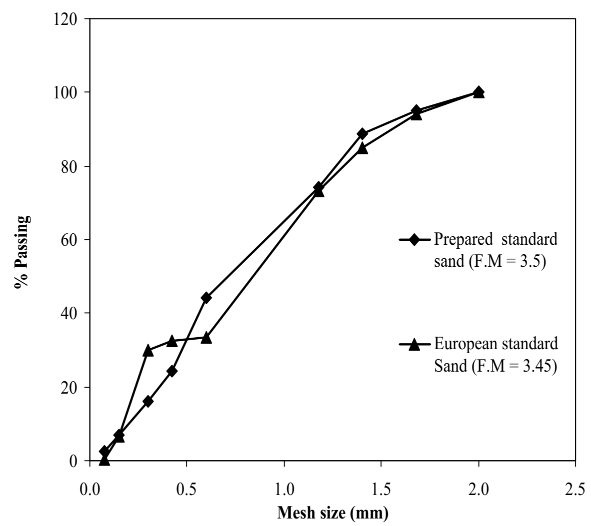


Fig. 1 Grading curves of SASS and ESSS (Ikotun and Ekolu 2010).

Table 2 Chemical composition of WA.

Chemical constituents	Percentage composition (%)
SiO_2	65.10
Al_2O_3	5.32
Fe_2O_3	7.25
CaO	10.56
MgO	2.75
SO_3	1.62
Na_2O	1.04
K_2O	3.36
$CaCO_3$	4.37
LOI	5.18
LSF	1.23
SR	4.43
AR	6.96
Total $SiO_2 + Al_2O_3 + Fe_2O_3$	77.67

a good pozzolan. The production of green-synthesized nano-TiO_2 was carried out at nanotechnology research group (NANO+) laboratory, Ladoke Akintola University of Technology (LAUTECH), Ogbomoso, Nigeria. Type 1 distil water, produced at the water resources section of Civil Engineering Laboratory, University of South Africa (UNISA) was used.

2.2 Synthesis of Nano-TiO_2

The nano-TiO_2 used in the study was biogenically synthesized using a pod extract of *Cola nitida* as described by Lateef et al. (2016). To obtain the extract, 1.5 g of milled pod was mixed with 150 ml of distilled water and heated in a water bath at 60 °C for 1 h. Whatman No. 1 filter paper was used to filter the extract and centrifuged at 4000 pm for 20 min. The nano-TiO_2 was synthesized by adding 120 ml of the cola pod extract (KP) to 600 ml of TiO_2 solution prepared by adding 0.5 g of TiO_2 to 0.6 ml of water. The mixture was carried out under room temperature of 30 ± 2 °C and allowed to stay for about 2 h. The formation of nano-TiO_2 (NT) was observed as a change in colour of the solution monitored visually until it stabilizes. The morphologies of the green-synthesized NT are shown in Fig. 2. As observed from Fig. 2a, the scanning electron microscope (SEM) indicated that NT particles are spherical in shape while the transmission electron microscope (TEM) as seen in Fig. 2b gave the size of NT as ~ 38 nm.

2.3 Preparation of Specimens

The preparation of specimens for testing setting times of cement, flexural strength, compressive strength and water absorption of mortar was done by partial substitution of cement with 10% by weight of WA. The 10% WA replacement was used as it had been established that it was the optimum content for structural purposes (Raheem and Adenuga 2013). WA cement mortar was incorporated with 1, 2 and 3% by weight of NT. The samples produced with only CEM1 serves as the control. Since the nano-TiO_2 is in liquid form, it was added as percentage substitution for mixing water so as not to increase the water cement ratio of the mix as practised by Berra et al. 2012 and Horszczaruk et al. 2017.

Samples for setting time test were cement pastes prepared in accordance with the provision in SANS (196-31: 2006) (EN 196-3: 2006). Table 3 indicates the mix proportion for the samples.

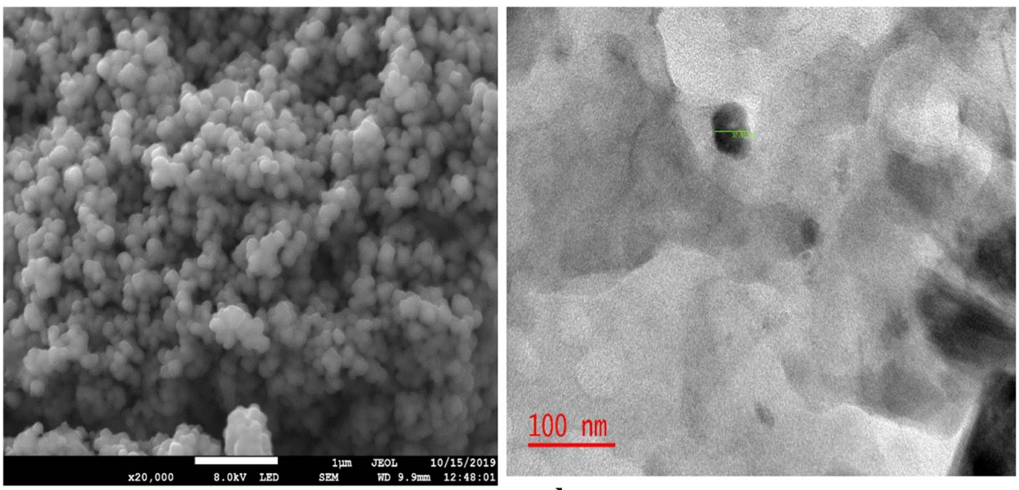
The required quantity of the materials was measured. Aqueous NT was pre-mixed in the mixing water by stirring for about 1 min, to produce a homogenous solution before adding to the binder. An automatic cement mixer with Serial No. 15/002436, capacity of 5 l and manufactured in 2015 was employed in mixing the paste. The cement paste was immediately transferred to a lightly oiled mould (diameter 70/80 mm) placed on lightly oiled base-plate, filled and compacted in line with provision in SANS 196-3: 2006 (EN 196-3: 2005). The mould containing the cement paste was placed inside the automatic setting times equipment to determine both initial and final setting times.

Sample preparation for flexural and compressive strength tests involves using mortar prisms of size 40 × 40 × 160 mm made according to the provisions in SANS 196-1:2006 (EN 196-1: 2005). The binder-to-sand ratio is 1:3 and water-to-binder (w/b) ratio is 0.5. Addition of NT was done as in setting time method and the same automatic cement mixer was used for mixing the mortar but with the mode now changed as provided in

SANS 196-1:2006 (EN 196-1: 2005). Table 4 indicates the mix proportion of the materials.

After mixing, the mortar was transferred into a steel mould that contains three prism samples of size $40 \times 40 \times 160$ mm each. The mould was vibrated for 1 min on a mechanical vibrator model 5533EN with serial number 067, manufactured in 2015 by Toni Technik, Germany. The mould and its content were stored under moist air condition at a temperature of 20 ± 1 °C and relative humidity of 90% for 24 h. Thereafter, the specimens were demoulded and cured in water bath for 3, 7, 28, 56 and 90 days using a curing tank with an in-built thermostat that maintains the temperature of the water at 20 ± 2 °C.

Mortar prisms as prepared for flexural and compressive strength tests were employed in carrying out water absorption test.



a SEM Image **b** TEM Image

Fig. 2 Morphology of green-synthesized nano-TiO_2 (NT).

Table 3 Mix proportion of cement pastes.

Sample number	Sample designation	Weight of cement (g)	Weight of WA (g)	Weight of NT (g)	Weight of water (g)	Water/binder ratio
1	CEM 1 (control)	500	–	–	180	0.36
2	90CEM1 + 10WA	450	50	–	195	0.39
3	90CEM1 + 10WA + 1NT	450	50	5	187	0.37
4	90CEM1 + 10WA + 2NT	450	50	10	183	0.37
5	90CEM1 + 10WA + 3NT	450	50	15	181	0.36

Table 4 Mix proportion of mortar specimens.

Sample code	Sample designation	Weight of cement (g)	Weight of WA (g)	Weight of SASS (g)	Weight of NT (g)	Weight of water (g)
A	CEM 1 + SASS (control)	450	–	1350	–	225
B	90CEM1 + 10WA + SASS	405	45	1350	–	225
C	90CEM1 + 10WA + 1NT + SASS	405	45	1350	4.5	220.5
D	90CEM1 + 10WA + 2NT + SASS	405	45	1350	9.0	216
E	90CEM1 + 10WA + 3NT + SASS	405	45	1350	13.5	211.5

2.4 Testing of the Samples

The setting times, flexural and compressive strength as well as water absorption tests were carried out at Civil Engineering Laboratory, UNISA South Africa.

An automatic setting times equipment—ToniSET Compact, Model 7306-100/EN, manufactured in 2015, was used to determine both the initial and final setting times of the paste. The mould containing the cement paste was placed at a designated position in the equipment immediately after casting. The equipment has a Vicat needle EN (Ø 1.13 mm) that penetrates the cement paste mould at regulated intervals until final setting of the paste. Readings of the setting times were automatically recorded on a computer, which simultaneously produce a graph indicating the initial and final setting times.

A mortar press—ToniPRAX, Model 2010, Serial number 834, manufactured in 2015, was employed in evaluating the flexural and compressive strength. The specimens were removed from the curing tank on the required testing age, mopped with cloth, weighed and placed on the appropriate load frame for the test. Flexural strength was first determined by applying force on a prism specimen which breaks it into two equal halves. Each of the halve was then used to determine compressive strength. Three specimens were tested for each curing day giving three (3) results for flexural strength and six (6) for compressive strength, the average of which is taken to obtain the actual strength for the particular curing age.

Water absorption test was performed using the procedure adopted by Adesanya and Raheem (2010). The mortar prisms were cured in water for 56 days. The prisms were then removed, mopped and weighed. They were then placed in oven at temperature of 105 ± 2 °C for 24 h. The prisms were removed from oven, allowed to cool for 1 h and weighed (W1). They were then immersed in water for 24 h and thereafter removed, mopped and weighed (W2). Percentage water absorbed was calculated using Eq. 1:

$$\text{Percentage water absorbed } = \frac{W2 - W1 \times 100}{W1}. \tag{1}$$

3 Results and Discussion

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

3.1 Setting Times

The results of the setting times for all the five (5) cement mixes are presented in Fig. 3a–e. The graphs in Fig. 3 indicate the depth of penetration of the needle of the automatic setting time equipment right from the time the cement paste is introduced to the final completion of the experiment. The initial setting time (IST) and final setting time (FST) of CEM1 (control) as obtained from Fig. 3a are 299.59 and 450:38 [min:s], respectively. The incorporation of WA into the cement paste led to increase in both IST and FST to 616:19 and 835:37 [min:s], respectively (Fig. 3b). This result is consistent with the findings by Rahhal and Talero (2004) and Nocuñ-Wczelik (2001) as the partial replacement of cement with WA reduced the exothermic rate of hydration due to the lower rate of pozzolanic reaction. As observed from Fig. 3c, the addition of 1% NT caused a decrease of the FST with reference to CEM1 + WA from 835:37 [min:s] to 799:40 [min:s]. Further decrease in FST was witnessed with 2% NT addition as shown in Fig. 3d, with a value of 768:15 [min:s]. However, Fig. 3e indictes that as the NT addition increased to 3%, the FST increased to 773:51 [min:s].

The decrease in setting time with addition of 1 and 2% NT into the WA cement paste can be attributed to the high specific surface area of NT, which increased the wettable surface area and water absorption (Ma 2016). The bridging process of gaps was speeded up by the rapid consumption of free water, resulting in increase of viscosity and quick solidification (Chen et al. 2012). With 3% NT addition however, there was not enough calcium hydroxide from the binder to react with the titanium oxide from NT, thereby leading to higher setting time. This result suggests that 2% NT addition is the optimum.

3.2 Flexural Strength

The result of the influence of NT addition on the flexural strength of WA cement mortar is presented in Fig. 4. The flexural strength of CEM1 varied from 5.43 to 8.37 MPa for curing ages 3–90 days. With the incorporation of 10% WA as partial replacement for cement, the flexural strength dropped to 5.32 MPa at 3 days and 7.08 MPa at 90 days. This is expected as WA which is a pozzolan, has low rate of heat development and cement hydration, leading to low strength (Raheem et al. 2012). The addition of 1% NT to the WA concrete does not show any improvement in the flexural strength. With 2% NT addition, there was enhancement in flexural strength at 7, 28 and 90 days. However, the values obtained are lower than that of CEM1 mortar. This is because TiO_2 is not pozzolanic like some nanomaterials such as nano-SiO_2 (Ma et al. 2016). Addition of 3% NT does not help matters as it resulted in lower flexural strength compared to the 2% NT addition. The slight increase in flexural strength recorded for 2% NT addition is attributed to pozzolanic reaction of WA and densification of the mortar through physical effect of the NT (Praveenkumar 2019).

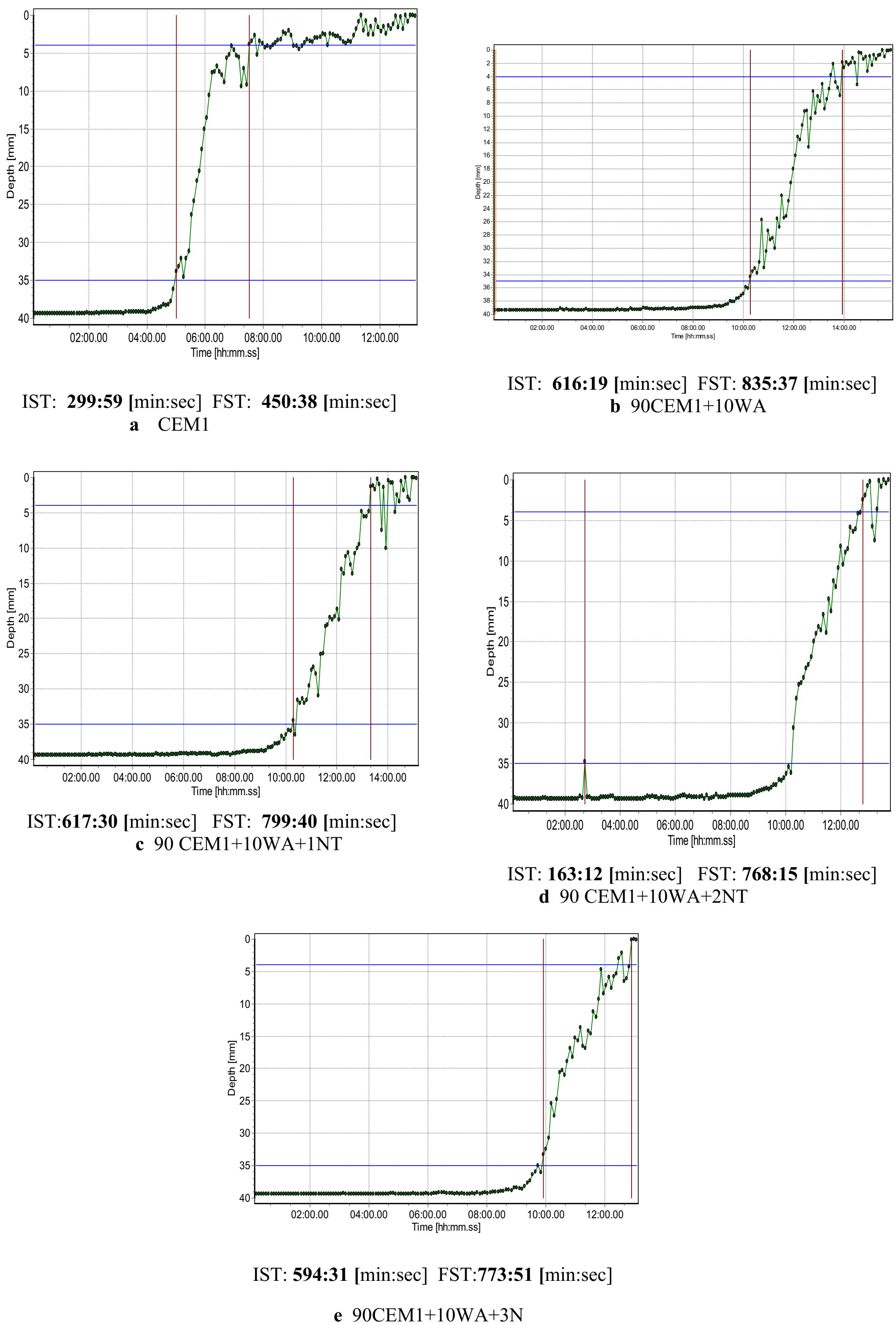


Fig. 3 Setting times of cement mortar incorporating WA and NT.

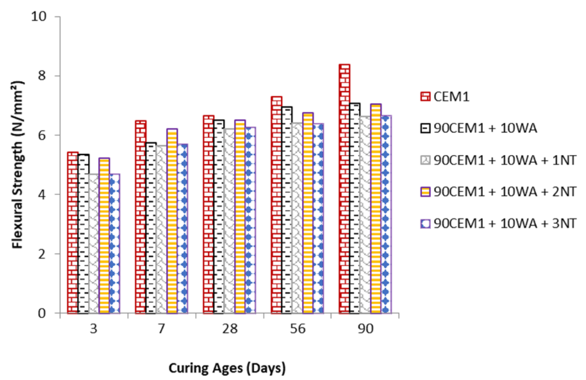


Fig. 4 Influence of NT addition on flexural strength of WA cement mortar.

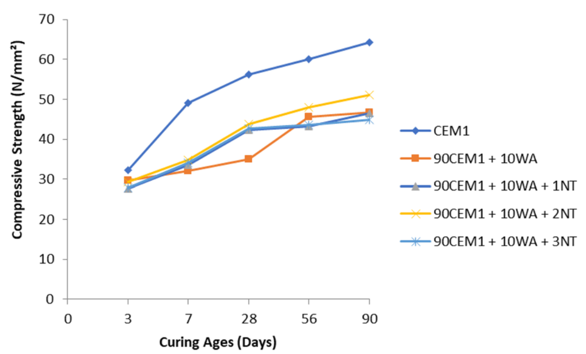


Fig. 5 Effect of NT on compressive strength of WA cement mortar.

3.3 Compressive Strength

The effect of NT on compressive strength of WA cement mortar is shown in Fig. 5. The compressive strength of CEM1 mortar as obtained from Fig. 5 for curing ages 3, 7, 28, 56 and 90 days are 32.24, 49.09,56.28, 59.98 and 64.30 MPa as against those of 90CEM1 + 10WA which are 29.62, 32.09, 35.01, 45.57 and 46.77 MPa, respectively. The reduction in compressive strength by partial substitution of cement with 10% WA is due to the low rate of hydration caused by the addition of WA (Raheem et al. 2012). Addition of 1% NT led to 5.9 and 20.7% increase in compressive strength at 7 and 28 days, respectively, compared to 90CEM1 + 10WA. Further increases of 8.3, 24.8, 5.4 and 9.1% were recorded at curing ages 7, 28, 56 and 90 days, respectively, with 2% NT addition. The increase in compressive strength with addition of 1 and 2% NT is credited to the filling and nucleation effects of NT (Ma et al. 2016). Also, the high surface area of NT enhances hydration rate as a result of the fast dissolution of cementitious compound (Lee et al. 2009; Lee and Kurtis 2010). However, addition of 3% NT caused a decrease in compressive strength compared to both 90CEM1 + 10WA + 2NT and 90CEM1 + 10WA mortar mixes. According to Nazari and Riahi (2010), the decrease in compressive strength with more than 2% NT addition may be because the quantity of NT in the mix is greater than the volume required to combine with the lime released during the hydration process. The excess NT leached out to cause deficiency in strength. Thus, only up to 2% NT addition is sufficient to enhance compressive strength of WA cement mortar. This result is consistent with previous findings of Praveenkumar et al. (2019) and Horszczaruk et al. (2017) that addition of nanoparticles to pozzolan mortar led to increase in their compressive strength. The addition of nano-TiO_2 to the mortar mix resulted in addressing the weakness of WA cement in terms of low workability and strength properties.

3.4 Water Absorption

Table 5 shows the result of water absorption properties of WA cement mortar incorporating NT. As indicated in the table, mortar produced with CEM1 only (control) has mean water absorption of 6.1%. With the incorporation of 10% by weight WA as partial replacement for cement, the water absorption increased to 6.8%. Similar results were obtained by Nagrockienė and Daugėla (2018) and Udoeyo et al. (2006). The increase in water absorption according to Nazari and Riahi (2010), was due to the formation of a less homogeneous and porous cementitious matrix. The water absorption of all specimens are < 10% maximum, which was prescribed for most construction materials (Chowdhury et al. 2015b).

Observation from Table 5 showed that addition of 1% NT to WA cement mortar reduced the water absorption from 6.8 to 6.6%. The reduction in water absorption is attributed to improvement in the pore structure of the WA cement mortar as NT acts as a filler to enhance the density of the mortar (Nazari and Riahi 2010). Slight reductions in water absorption of WA cement mortar were also noticed when 2% and 3% NT were added with values of 6.0 and 6.2%, respectively. All specimens recorded less than 10% maximum values recommended.

4 Conclusions

In conclusion, the following can be deduced from the findings in this study:

1. The setting time of WA cement were reduced by the incorporation of up to 2% NT by weight of binder.
2. Slight increase in flexural strength was recorded with addition of 2% NT to WA cement mortar.
3. Addition of up to 2% NT improved compressive strength of WA cement mortar.

Table 5 Water absorption characteristics of WA cement mortar incorporating NT.

Specimen identification	Weight before immersion (g)	Weight after immersion (g)	Weight of water absorbed (g)	Percentage of water absorbed (%)	Average (%)
K1	525.2	557.5	32.3	6.2	6.1
K2	526.9	558.2	31.3	5.9	
K3	526.9	559.4	32.5	6.2	
L1	515.3	551.4	35.8	6.9	6.8
L2	518.5	554.4	35.9	6.9	
L3	513.0	548.1	35.1	6.8	
M1	507.7	540.8	33.1	6.5	6.6
M2	508.1	541.7	33.6	6.6	
M3	511.4	545.3	33.9	6.6	
N1	517.7	547.8	30.1	5.8	6.0
N2	519.7	551.5	31.8	6.1	
N3	516.1	547.6	31.5	6.1	
O1	512.0	543.6	31.6	6.2	6.2
O2	520.7	553.0	32.3	6.2	
O3	518.3	551.1	32.8	6.3	

KEY: K = CEM1 L = 90CEM1 + 10WA M = 90CEM1 + 10WA + 1NT

N = 90CEM1 + 10WA + 2NT O = 90CEM1 + 10WA + 3NT

4. There was reduction in water absorption of WA cement mortar when NT was added with 1% NT incorporation having the best result.

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Authors' contributions
The design and execution of the study were equally carried out by both authors. Both authors read and approved the final manuscript.

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Availability of data and materials
The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Competing interests
The authors declare that they have no competing interests.

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