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Evaluation of Mechanical Properties And Sorptivity Of Cementitious Composites Produced With Dosages Of Nano-Silica

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Abstract: *The durability of cementitious composites continues to be an issue that receives the attention of practitioners and researchers around the world, which has led to the design of high-performance cementitious composites such as engineered cementitious composites (ECC). ECCs have high ductility capacity that makes them more durable, however, further, improvement isdesirable.This paper, therefore,sought to evaluate the mechanical properties and sorptivity capabilities of ECCs produced with dosages of nano-silica.The 28th -day test results showed improvements in the compressive strength of the specimens with increasing dosage of nano-silica. Nonetheless, the 28th -day flexural strength of the specimens slightly reduced with an increasing amount of nano-silica dosage. Concerning the 28th-day sorptivity measurements,it was observed that the sorptivity coefficients reducedwith increased dosages of nano-silica. There were significant reductions in the sorptivity values of the various specimens after they had been subjected to 7 and 28 days of continuous water. It can be concluded from the results of the study that nano-silica can help improve the compressive strength and sorptivity properties of cementitious composites.*

Keywords: *Cementitious composites; Compressive strength; Flexural strength; Nano-silica, PVA fibers, Self-healing, Sorptivity*

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I. Introduction

The durability of cementitious products continues to be a major concern to practitioners and as a subject that attracts research attention.One of the causes of deterioration of concrete is when there is the formation of cracks at the macro and micro levels and subsequent ingress of dissolved particles in fluids, unwanted acidic gasses, and water [1]. These substances then attack the reinforcements in the concrete, thereby compromising its durability and sustainability. This demandsasignificant amount of money for repair, maintenance, and replacement work for proper functioning [2]. Therefore, efforts are being made in diverse ways to improve the performance of concrete such as toughness and strength. Such efforts are evident by the usage of several materials including fibers, blast furnace slag, fly ash, graphene, and silica fume to improve the performance of concrete [3-5]. The materials improve the properties of concrete at various degrees depending on the proportion they constitute in the concrete mix. For instance, research has shown that the application of fly ash in concrete production improves its strength capacity as it reduces the water/cement ratio required and improves resistance against chemical attack since it reduces matrix permeability [6].High-performance fiber reinforced cement composites (HPFRCC) have also been developed to improve other properties such as ductility.Engineered cementitious composite (ECC) is a special type ofHPFRCC with significantly improved tensile strain hardening behavior and multiple microcracking [5, 7-10]. ECC has therefore gained immense popularity in research, where it is considered as a viable approach to improve the ductility of concrete. Especially, it allows systematic microstructure tailoring of ECC as well as materials optimization. ECCs have high tensile ductility with moderate fiber content, typically 2% by volume [11]. Its design is predicated on the principles of micromechanics and fracture mechanics theory [9, 12]. Li [9] posits that ECC design relates macroscopic properties to microstructures of a composite, which forms the backbone of materials design theory.ECC strain-hardens after first crack formation, which is contrary to ordinary concrete and other fiber-reinforced concretes.One interesting characteristic of ECC is its capacity to deform to high strains under load, typically between 3-5% while maintaining a tight crack width such as 60μm up to failure [11, 12].

The crack width of ECC is an inherent material property, which is independent of structural size, steel reinforcement, or load applied to the built structure [12]. The crack width of ECC is a result of its capacity to undergo flat crack propagation, where much of the crack flank maintains constant crack width as the crack

length increases [11]. Research shows that ECCs with tight crack widths exhibit high permeability resistance similar to uncracked concrete, which mitigates durability issues [10].These special characteristics of ECC enable it to meet strict requirements for applications such as bridge deck link slab and coupling beams in high rise buildings [12]. Whiles ECCs possess these superior characteristics, further, improvement is still desirable.

With the increasing growth in knowledge of nanotechnology, researchers have sought to improve the performance of cementitious composites by incorporating nanoparticles. Researchers have used a variety of materials including nanoparticles such as montmorillonite, carborundum, and silica [13]. Nanoparticles are able to speed up cement hydration, act as nano-fillers, compacting the microstructure, and therefore reduce porosity a [14].In Biricik and Sarier [15], Khater [16], and [17] for instance, the addition of nano-silica (NS), a nanomaterial, in the mixtureofcementitious material, yielded a significant improvement in the compressive and flexural strengths of the cementitious composites. The improvement in the properties of the cementitious composites can be attributed to the nano-sized particles and the extensive surface area of nano-silica [15, 18]. With nano-sized particles, nano-silica possesses significant surface energy and atoms at high level of reactivity, which causes atoms to react with other external atoms, thereby increasing pozzolanic reactions at early ages [19]. Owing to these properties of NS, there has been a growing interest in the possible usage of NS in cement pastes and concrete. This has brought out useful findings relating to workability and rheological, transport and mechanical properties; and microstructural and C-H-S gel formation. This paper, therefore, sought to ascertain the effect of nano-silica on cementitious composites in terms of compressive and flexural strengths, and water sorptivity measurements.The paper also reports the effect of healing on the various specimens in terms of water sorptivity. The rest of the paper respectively, presents the experimental program of the study, test results and discussion. This is then followed by the concluding section of the paper.

II. Experimental Program

Materials and mix proportions

The materials that were used to prepare the ECC mixtures are Type I cement (42.5 R); fly ash (FA); Nano-silica (NS); river sand (maximum aggregate size 600µm), polyvinyl alcohol (PVA) (fibers of 2% byvolume), water and high range water reducer.

Table *1*presents the mix proportions of the various materials, while Table 2, Table *3*, and

Table *4*presentthe physical properties of PVA, nano-silica, and cement and fly ash respectively.

Table 1: Mix Proportions of Materials

W/B	WATER (kg/m^3)	NS%	NS (kg/m^3)	FA (kg/m^3)	PC (kg/m^3)	SAND (kg/m^3)	HRWR* (kg/m^3)	PVA Fiber (kg/m^3)
0.27	226	0.0	-	457	381	302	11	26
0.27	226	0.5	4.2	457	381	302	11	26
0.27	226	1.0	8.4	457	381	302	11	26
0.27	226	1.5	12.6	457	381	302	11	26

*High Range Water Reducer

Table 2: Physical/Mechanical Properties of PVA

Length (mm)	Diameter (µm)	Young's Modulus (GPa)	Elongation (%)	Tensile strength (MPa)	Density (g/cm^3)
6	15.6	42	7	1804	1.3

(a) (b)

Figure 1: (a) PVA fibers; (b) Nano-silica

Table 3: Properties of Nano-Silica

Features	%
Heat reduction (%) (105 °C 2 h) ≤	3
Loss of ignition (%) (950 °C 2 h) ≤	6
SiO_2 content (dry base) (%) ≥	92
SiO_2 content (%) (950 °C 2 h) ≥	99.5
Carbon content (%) ≥	0.3
Specific surface area (m^2/g) (BET law)	220 ± 30
pH value	5-7
Surface density (g/ml) ≤	0.15
Dispensability (%) (CCl_4) ≥	80
Oil-absorbed value (ml/100 g) ≥	250
Average particle size (nm)	10-25

Table 4: Properties of Ordinary Portland Cement and Fly Ash

Chemical composition	Cement (%)	Fly ash (%)
CaO	61.8	4.012
SiO_2	19.40	53.938
Al_2O_3	5.30	31.148
Fe_2O_3	2.30	4.160
MgO	0.95	1.011
SO_3	3.80	0.727
K_2O	1.10	2.035
Na_2O	0.20	0.888
Loss on ignition	2.10	0.21
Physical properties		
Specific gravity	3.15	2.18
% Retained on 45 µm (0.002 in.)	12.9	9.6
Water requirement, %	-	93.4

Specimens preparation

The mix proportions of the materials as presented in

Table *1* wasemployed for the preparation of the PVA-ECC mortar. This mix design adapted from extant literature [5, 20, 21]. The fly ash to cement ratio is 1.2 by mass, while water to binder ratio was set as 0.27. Percentage additions of NS were 0%, 0.5%, 1.0%, and 1.5% by mass.

Table ***4***presents the various NS dosages and their respective specimen designations. The sand to binder ratio was set to 0.36. The mixture was made using a mortar mixer rotating blade, where the constituents of the mixture: river sand, fly ash, Ordinary Portland cement, and Nano-Silica were thoroughly mixed for 2 minutes in the machine. Subsequently, the HRWR was added to water to form a liquid solution, which was then added to the mixture in bits. After obtaining a uniform mixture, the PVA-fibers (2.0% by volume fraction) were added gradually and manually to obtain uniform dispersion.The various mixes were cast into cube (40mm), prism (160x40x40mm), and cylindrical (100Ø50mm) specimens, after the mixture was thoroughly mixed for 5–10 minutes until uniformity was achieved. These were cast in a single layer without compaction and all specimens were de-molded after 24hours. After which, they were stored in plastic bags at 23±2° C and 95±5% RH until the predetermined testing ages after 24hours curing in the molds at 23±2 °C and 50±5% RH.

Table 5: Percentage of NS Dosages and Specimen Designation

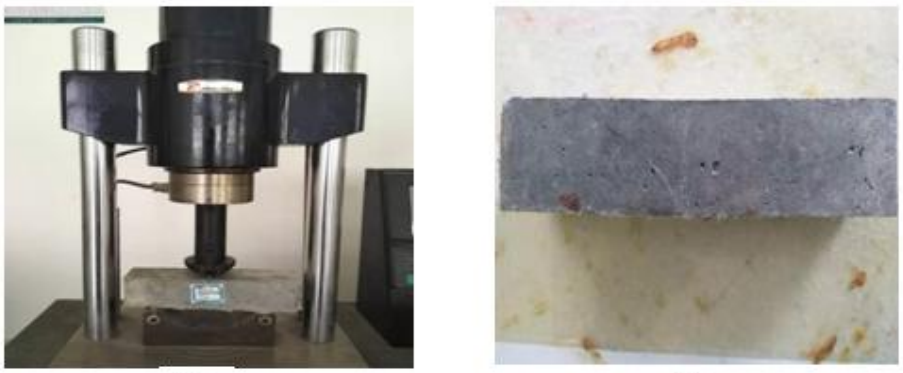
% of NS	Designation
0.0	D01
0.5	D02
1.0	D03
1.5	D04

Testing procedure

Mechanical properties

The mechanical properties were measured on the 28th day. The compressive strength and flexural strength properties were measured using five (5) specimens of each group with 40mm cubic, and 160x40x40mm prisms respectively. The universal testing machine was used for the measurement of both the compressive,

whiles three-point flexural testing machine was used to gauge the flexural strength properties of the specimens. In Figure ***2***, the setup for the flexural test (a) and a snapshot of a deformed specimen under a three-point load are depicted.



(a) (b)

Figure 2: flexural strength test setup (a); Snapshot of the deformed specimen(b)

Water sorptivity test

The water sorptivity test was used to determine the water absorption rate of the various ECC specimens. Three (3) pre-loaded specimens from each mixture group were tested with the other two (2) sound (without preloading) specimens for comparison. Firstly, the three (3) of the specimens were preloaded with their split tensile deformation capacities to induce microcrack formations after attaining their testing age (28 curing days). The preloading was done by deliberately controlling the testing machine to ensure that only microcracks were formed.Before the start of the sorptivity measurements, specimens were left to dry in an oven at 50±5 °C until a constant weight was achieved, adopted from similar studies such asSahmaran and Li [22]. Figure 4shows setups for preloading (a) and oven-drying of specimens (b). After all, specimens were completely dry, they were immersed in water by 3-5 mm, as evinced in Figure 3, where specimens were made to rest on supports to allow free access of water to the inflow surface. To ensure one-directional flow through the specimens, the circumference of the samples was coated with epoxy resin. Changes in mass of the ECC specimens that were left to absorb water through capillary suction, one of the configurations for sorptivity testing espoused by Hall [23], were recorded at time intervals of 1, 2, 3, 4, 6, 8, 12, 16, 20, 25, 36, 49, 64, 81, 120 and 360 minutes.Each time the specimens were removed from the water container, damped tissue was used to mop off the excess surface water and weighed on a digital balance.The process was completed within 30 seconds as adopted by Chen, Zheng [24]. The changes in the crack widths over the specimens for the sorptivity test were observed with anopticalmicroscope at the end of each pre-determined testing period.

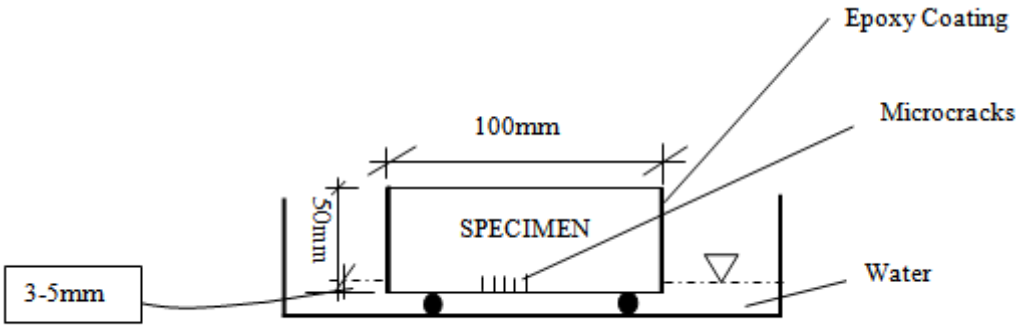


Figure 3: Setup for water sorptivity measurement

(a) (b)

Figure 4: (a) Setup for pre-loading of specimens; (b) Setup for oven-drying of specimens

III. Test Results AndDiscussion

Compressive strength

The average compressive strength values of five (5) specimens (achieved on the 28th day of curing) for each group have been presented in Table ***6***. The specimens D01, D02, D03, and D04 attained an average compressive strength value of 58MPa, which makes them suitable for practical applications[20].There were corresponding increases in the compressive strength values, with increasing dosages of nano-silica, albeit these increases in strength were marginal (<1.1%). The addition of 0.5% (D02) of NS to the mixture yielded a marginal increase in the compressive strength value as compared to the control specimen (D01). Moreover, the increase in percentage dosage of NS from 0.5% to 1.0% resulted in a marginal increase of compressive strength value as compared to the value of D01 and D02. A similar observation was made, whenthe NS dosage was increased to 1.5%, with the compressive strength of D03 being marginally higher than D04. InTable ***6***, the compressive strength values with their respective dosages of NS (0.0%, 0.5%, 1.0%, and 1.5%), which are represented by their respective designations, D01, D02, D03, and D04 as shown in Table***5.***The increase in the compressive strength of the specimens is attributable to a higher amount of calcium-silicate-hydrate(C-S-H)generated due to the addition of NS, resulting inhigherdensification of the matrix to improve its strength [18, 21]. A raft of studies suggests that the increase in a calcium-silicate-hydrate gel is the reason foranincrease in the compressive strength of the specimens [25]. Over longer ages, continues pozzolanic reaction results in increased compressive strength values of the specimens [14].The observed increase in the compressive strength, with the addition of NS, is supported in extant literaturesuch asMohamed [17]. However, there have been reports of decreases in compressive strength values after the NS dosages had reached their optimumpercentages[14, 17].

Table 6: Compressive and flexural strength of specimens at 28-day old

Designation	Compressive Strength (MPa)	Flexural Strength (MPa)
D01	58.05	16.24
D02	58.08	16.06
D03	58.13	15.50
D04	58.67	15.56

Flexural strength

A three-point bending test was used to assess the flexural strength and the ductility of the various specimens. The average flexural strength values of five (5) specimens from each of the four (4) groupsof specimens have captured in Table ***6***.

The average flexural strength value of the specimens, D01, D02, D03, and D04 was 16MPa, which is suitable in certain structural applications.However, the flexural strength values of the specimens decreased with increasing dosages of NS, an observation which is in contrast with the compressive strength values which increased with increased dosages of NS. An increase in nano-silica from 0% to 1.5%, saw a reduction in flexural strength of the specimens decrease from 16.24MPa to 15.56MPa.This could be attributed to a higher interfacial chemical bond between the PVA fibers and matrix as nano-silica is added to the mix.Moreover, the non-oilcoated PVA fibers possess a strong bond between the surrounding hydration matrix due to the hydrophilic nature of the fiber[10]. Therefore, not coating the fibers with oil could have led to an increase in the PVA fiber-matrix bond and caused a reduction in the ductility of the specimens. There was a however contradictory

observation that was made with the flexural values of D03 and D04. As there were marginal reductions in the flexural strength values with the addition of nano-silica from D01 to D02; and D02 to D03, thevalue of D03 (15.50MPa) reduced compared to D04 (15.56MPa). In a related study thatalsousedlocal materials to develop ECC, but replaced silica-sand with local dune sand as fine aggregates, flexural strength value recorded was 9.5Mpa, which about 39% lower than those recorded in this study [7].The flexural strength of engineered cementitious composites is depended on a variety of factors, including the ultimate tensile strength and strain capacity; kind of aggregate and its grain size; and the water-to-binder ratio [7, 20, 26]. The beneficial effects of PVA fibers on the flexural strength of cementitious composites have been established by many authors, with the amount of fiber incorporated in composites corresponding to their flexural strengths [27]. Indeed, it is reasonable to assert that the combined effect of both nano-silica and fibers of the higher tensile capacity of 1804MPacontributedto the improvement of the mechanical properties of the specimens [19]. The water-binder ratio, 0.27, which was adopted and held constant for all the specimens is typical of most engineered cementitious composites.

Water sorptivity

The water sorptivity test was adopted as one of the means to measure the durability of the ECC specimenssince the sorptivity coefficient offers an indirect indication of the durability of cementitious composites [28]. Sorptivity is primarily depended on the suction rate; a function of the surface area of the specimen which is exposed to moisture for a specific duration[29]. The cumulative water absorption per unit area (*i*)increases concerning the square root of the elapsed time (*t*). The sorptivity (S) is defined as the slope of a graph of *i* against $t^{0.5}$, which is determined with regression analysis [30]. Table 7 shows the sorptivity values of the four groups of ECC specimens (averages of twoand three sound and preloaded specimens respectively) for the initial 28 daysofcuring and after the specimens had been immersed in water for 7 and 28 days. From Table 7, the sorptivity coefficients (<0.04 mm/min$^{1/2}$) of the various specimens,after the 28th day of curing were lower than those suggested in the literature for Ordinary Portland cement concrete with 0.4 to 0.5 water-cement ratio (0.230mm/min$^{1/2}$) [22, 30].The comparatively lower sorptivity coefficients of the study specimens can plausibly be attributed to the lower water-cement ratio; fly ash content;theamount of nano-silica; and absence of coarse aggregates[8, 31]. The sorptivity values of the specimens that had nano-silica incorporated weresmaller than the specimens without nano-silica (D01). Nonetheless, the sorptivity coefficients of D01for both sound and preloaded specimens were closer in value, where the sorptivity factor of the preloaded specimen was 0.56%higher.This observation was different with the sorptivity coefficients of the specimens which had nano-silica dosages, in that D02, D03 and D04 had the sorptivity figures of preloaded specimens were respectively 47.55%, 49.75%, and 23.26% higher than their sound specimen counterparts.It can also be observed from

Table ***7*** and Figure ***5***that the sorptivity coefficients of the various specimens reduce with increasing dosages of nano-silica. Nano-silica, as observed in the extant literature, acts not only as a filler to improve the microstructure but to spur a pozzolanic reaction in the matrix, which is a result of the transformation of portlandite in C-S-H and modification of the internal structure of C-S-H gel [18]. Thus, it is reasonable to ascribe the reduction in sorptivity coefficients to increased densification of the matrix of the specimens due to increased NS dosage and the combined effect of the fly ash and NS. In the following section, the effect of submerging the specimens in water for 7 and 28 days has been presented.

Table 7: Sorptivity values for Sound and Preloaded Specimens

DESIGNATION	STATE	SORPTIVITY (mm/min$^{1/2}$)		
		28 days (initial)	+7days	+28days
D01	Sound	0.0355	0.0184	0.0081
	Pre-loaded	0.0357	0.0187	0.0082
D02	Sound	0.0204	0.0096	0.0081
	Pre-loaded	0.0301	0.0134	0.0094
D03	Sound	0.0197	0.0100	0.0106
	Pre-loaded	0.0295	0.0106	0.0115
D04	Sound	0.0172	0.0159	0.0062
	Pre-loaded	0.0212	0.0173	0.0093

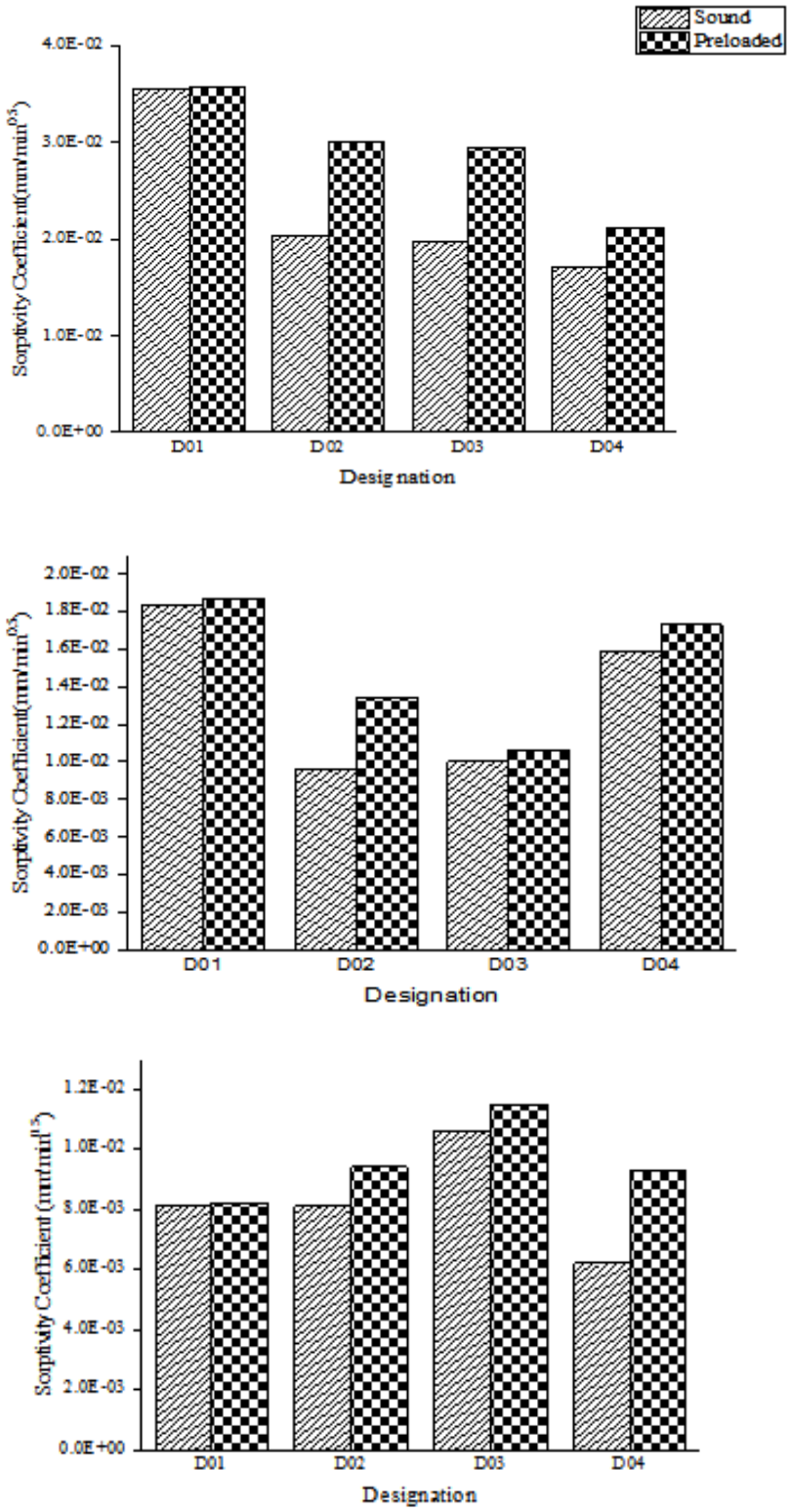


Figure 5: Sorptivity coefficients (a) After 28 days curing (b) 7 days healing (c) 28 days healing

Effect onhealing

Table 7 shows sorptivity values, measured after the specimens were subjected to continues water for 7 and 28 days of curing for the evaluation of self-healing in the ECCspecimens.It can be observed from

Table *7*, a general decrease in sorptivity coefficients of the various specimens after they have been subjected to 7days continuous water curing as compared to their respective initial sorptivity coefficients.Reduction of the

sorptivity coefficients for the sound (no preloading) specimens after 7 days healing ranged from 7.56 to 48.17%, while those of preloaded specimens saw reductions ranging from 18.4 to 64.07%. Significant reductions in the sorptivity coefficients were also recorded after an additional 28 days of continuous water curing. The sorptivity reductions after the additional 28 days of the sound specimens ranged from 46.19 to 77.18%, with those of the preloaded specimens ranging from 56.13 to 77.03%. The reductions in the sorptivity coefficients after the 7 and 28 days of healing can partly be attributed to the influence of the continuous water curing, which is greatly influential on improving the sorptivity values of the various specimens,irrespective of the dosage of nano-silica and the nature oftheircracks.The addition of nano-silica to cementitious materials generally enables small pores to be formed which can improve their durability properties such as water sorptivity coefficients [14]. Generally, there wasavarying rate of reduction in the sorptivity values of the various specimens after the two healing periods. For instance, the sorptivity coefficients of the sound specimens of D01 reduced by 48.17% and 77.18% after 7 and 28 days of healing respectively, whereas their preloaded counterparts reduced by 47.62% and 77.03% after 7 and 28 days of healing respectively. For specimen D04, the rate of reduction of the sorptivity values of the sound specimen was lower than the preloaded ones after a 7-day healing period, with sound specimens reducing by 7.56% and preloaded specimens reducing by 18.40%.Nonetheless,after the 28-day healing period, the reduction in the sorptivity coefficients of the sound specimens (63.95%) was higher than that of their preloaded (56.13%) counterparts. With regards to D02 and D03, there were higher reductions of sorptivity values of the sound specimens than those of their preloadedspecimens.The propensity of self-healing is also dependent on the nature of microcracks in terms of instances such as the total cracks, tortuosity of the crack paths in the specimens.A plausible explanation for the influence of the nature of microcracks on autogenous healing performance that was observed in the pre-loaded specimens is that the microcracks comparatively allow easier ingress of CO_2 dissolved in water during the continuous water curing, and offering more space for newly-formed healing products[32].Again, thechemical composition and continuous pozzolanic reactions of fly ash and nano-silica, also contribute to the self-healing performance of the various specimens.

IV. Conclusion

This study explored the effects of dosages of nano-silica on engineered cementitious composites produced with river sand and non-oil coated PVA fiber. The compressive, as well as the flexural strength characteristics of the specimens, were studied. Again, the sorptivity capacities of the various specimens were evaluated. This sorptivity evaluation was done for both sound and preloaded specimens after 28days of curing, and after specimens had been subjected 7- and 28-days healing conditioning, with specimens subjected to continuous water.Basedon the result and analysis of the study, the following conclusions have been made:The compressive strength value of the specimen increased with increasing dosages of nano-silica. The flexural strength of the specimens decreased with increasing dosages of nano-silica. However, this observation was contradicted in the case of D03 and D04, where the flexural strength of D04 increased marginally instead of a reduction. Generally, water sorptivity coefficients reduced with increasing dosages of nano-silica. For instance,theinitial sorptivity coefficient (after 28-day curing), which was recorded for D01 was about 28% higher than that of D02.Similar observations were made for the initial sorptivity coefficients of D03 and D04, whose values were lower than D01 by about 31% and 46% respectively. The sorptivity of the specimens improved further after they had been submerged in water for the two healing periods, i.e. 7 and 28-day environmental conditionings.Nonetheless, there was much improved in the sorptivity values of specimens with nano-silica additions than D01, which has no nano-silica dosage which wasanimprovement for specimens with higher dosages of nano-silica. This observation was made for the two healing periods, 7-day and 28-day healing periods.

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