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MULTISCALE CHARACTERIZATION OF NEEM SEED HUSK ASH CONCRETE

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Abstract: Current research in concrete Science and Technology is modelled towards the multiscale characterization of concrete produced from binary blended cement paste with a view of optimizing concrete performance using minimal cement content by blending the cement with a secondary cementitious material. The use of multiscale characterization has revealed interesting properties of concrete that can form the basis for the re-engineering of concrete to maximize its benefits and use. This current paper studied the properties of Neem seed Husk Ash (NSHA) at the macro, micro and nano scales by investigating the physico-chemical, micro-structural and nanomechanical properties using compressive strength test, XRF and SEM analysis as well as a nonaindentation technique, which was used to characterize the hardness, modulus of elasticity, C-S-H content and porosity of the cement paste. It has been concluded from the study that the effect of the addition of 10% NSHA to cement paste on the properties of concrete cured for 28 days at the macro, micro and nano scales correlate well. It is recommended that multiscale characterization of cementitious materials provides the best insight into pozzolanic concrete behaviour and should be adopted for designing of pozzolanic concrete for optimal performance.
Keywords: multiscale characterization, concrete, Neem seed husk ash, nanoindentation, compressive strength, microstructural analysis

1. INTRODUCTION

Sustainable development of any nation hinges on the provision of affordable housing and road infrastructure for the benefit of the majority of the populace (Adesanya & Raheem 2009). In pursuant to this, developing countries like Nigeria spend billions of dollars annually in an attempt to bridge this infrastructural gap. The use of conventional construction materials like cement has inherent disadvantages of high cost as well as negative environmental effects (Taku et al 2015). This environmental hazards, coupled with the high cost of construction occasioned by the use of cements have driven a lot of research in the use of secondary cementitious materials that can be used wholly or partially in replacement of cement (Taku & Amartey, 2016, Utsev & Taku 2012, Saalu & Olonade 2011). Materials that meet this specification are often pozzolanic in nature, and according to ASTM C618, should possess 50% (Class C) or 70% (Class F and N) of Fe_2O_3+SiO_2+ Al_2O_3.

The production and use of binary cements have become global practice and is fast gaining acceptability in the developing world. Most of the secondary cementitious materials in use in Nigeria are agro-based. While some of them meet the specifications of ASTM C618, most of the researches show some contradiction between the performance of these materials in concrete and the specification of the codes. Naturally it is expected that pozzolans having high pozzolanic activity index and whose chemical content meet up with the code specification should improve the strength, durability and other properties of concrete, but this is not always the case.

One of the materials investigated for use in partial replacement of cement in Nigeria is Neem Seed Husk Ash (NSHA). In all the cases investigated, the strength decreases with increase in ash content. However, all the researchers agree that 5 to 10% of the ash can be used in partial replacement of cement in concrete and mortar (Ejeh et al 2014, Ibiwoye&Naalla 2017, Ramsharath et al 2017). All the investigations are however limited to the physico-chemical, and micro-structural characterization of the material in concrete, which gives nothing as to what is happening at the nano level. No effort has been made to use nanotechnology to investigate the nanobased properties of NSHA blended cement mortars and concrete.

The advantage of using Nanoscience and technology in the nano-characterization of concrete cannot be overemphasized as it holds the key to this understanding of the properties of the products of pozzolanic cements hydration (Constantinides et al. 2003). Recent progress made in the areas of theoretical and experimental nanomechanics has opened up new frontiers in materials sciences such that cement based composites can be nanoengineered and nanoindentation techniques adapted to understanding the nature and form of C-S-H gels and other hydration products. The possibility of using nanotechnology to improve concrete performance to the next level and also develop cements based composites that will save both the cost of construction and construction safety is enough motivation to tailor research in this wise.

As earlier noted, NSHA has been used successfully to replace concrete at up to 10% replacement, with the physico-chemical and strength properties investigated (Nuruddeeen&Ejeh 2012, Uche&Abubakar 2009). Little is however still known about what happens at the micro and nano level during the hydration of the NSHA blended cements. Nanoindentation techniques provide an insight into the reactions that take place during the hydration of cement and into the various phases of the cement microstructure (Mondal et al 2015, Constantinides et al. 2006)

This research carries out multiscalecharacrerization of NHSA blended cement mortar. The principle of nanoindentation was used to study the effect of the partial replacement of OPC with 10% of NSHA on the Nanomechanical properties of the C-S-H gel and other phases of the hydrated cement gel.

2. METHODOLOGY AND RESULT DISCUSSION

The methodology for this research work is divided into two parts, viz; Materials characterization, strength, morphological properties and nano-mechanical characterization.

— Materials Characterization

A type I Portland cement (Dangote grade 43.5, 3X cement) manufactured by Dangote cement industries Gboko plant was used in this research. The fine aggregate used was river sand obtained from river Benue at Makurdi, while the NSH was obtained from a Neem seed fertilizer blending plant in Katsina state and calcined for 2 hours at 600°C to obtain the NSHA. Table 1 and 2 give the properties of the cement, NSHA and fine aggregates while figures 1 and 2 show the characterization of the fine and coarse aggregates respectively using sieve analysis.

Table 1: Chemical Characterization of OPC and CSA

Oxide	CaO	Fe2O3	Al2O3	SiO2	MgO	K2O	Na2O	SO3	P2O5	Mn2O3	LOI
NSHA	32.90	8.600	2.94	25.40	0.54	14.20	0.15	4.42	1.01	0.63	9.75
OPC	65.6	6.83	5.60	16.20	2.30	0.48	0.78	2.15	Nd	Nd	

Table 2: Physical characterization of CSA, OPC and Sand

S/No.	Property tested	Material tested			
		OPC	Sand	Gravel	NSHA
1	Specific gravity	3.19	2.01	2.60	2.32
2	Setting times (min): Initial (Final)	90(148)	-	-	-
3	Moisture content (%)	-	1.03	-	-
4	Standard consistency (%)	28	-	-	-
5	Soundness (mm)	1.0	-	-	-
6	Clay & silt content (%)	-	2.1	-	-
7	Bulking	-	2.6	-	-
8	Fineness	0.04	-	-	-
9	Aggregate impact value	-	-	24.5%	-

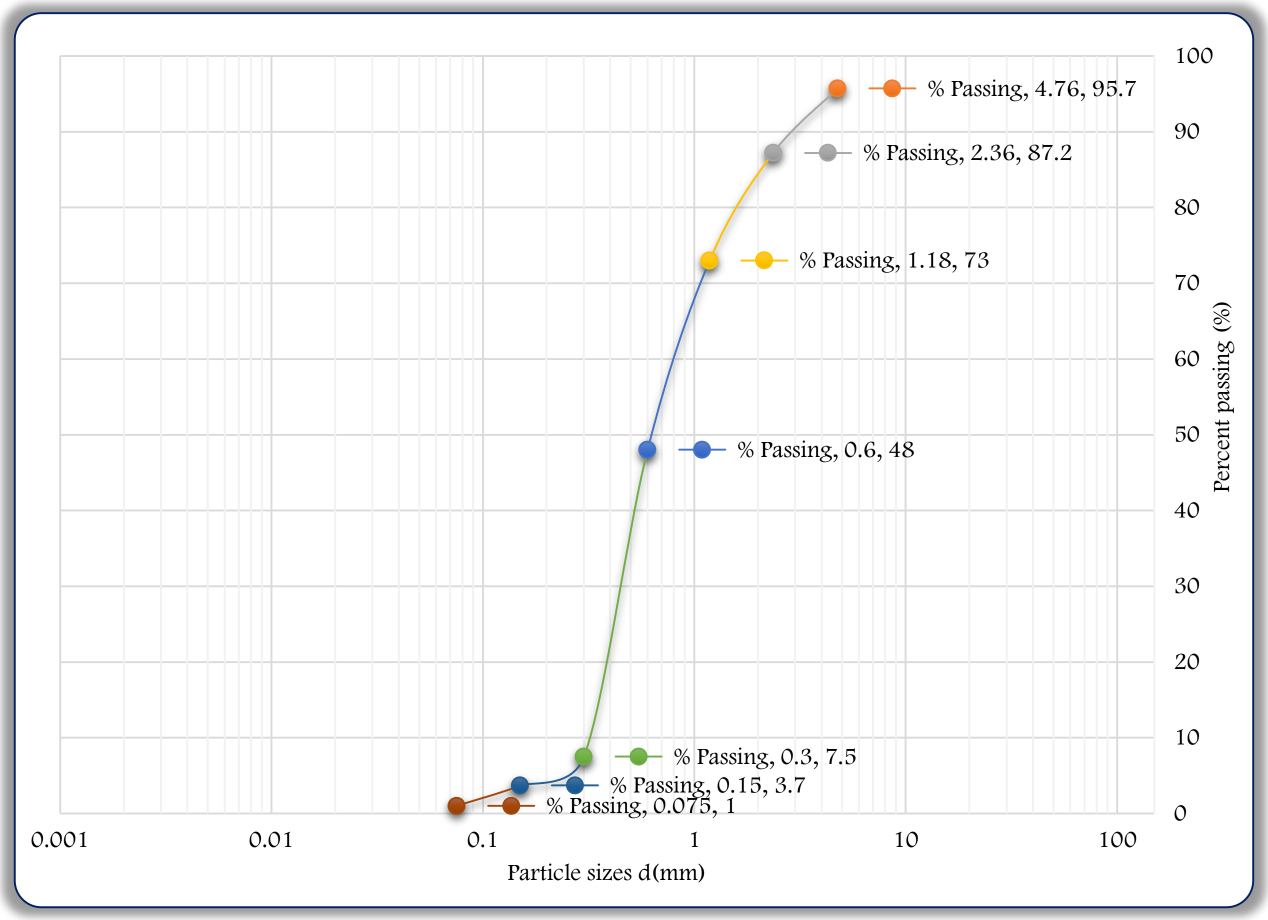

Figure 1: Particle size distribution for sand

The physico-chemical characterization of the materials meets the standards specified in ASTM C 618 and BS 12 for pozzolana, cement and aggregates.

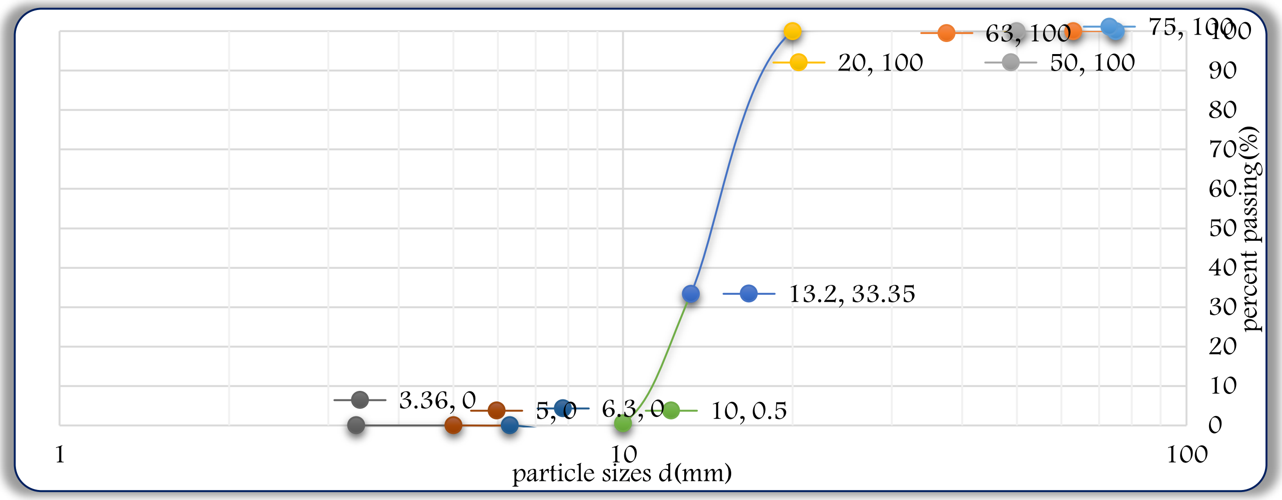

Figure 2: Particle size analysis for coarse aggregate

Macro, Micro and nanoscale analysis

» Compressive strength

The result for compressive strength of concrete cured at 7, 14 and 28 days is presented in Figure 3.

It can be observed from the result that the compressive strength decreased as the percentage of replacement increased. Worthy of note is the fact that compressive strength increases with age of concrete but decreases as percentage replacement increases. This result agrees with Ejeh et al 2014, Ramsharat et al 2017, Ibiwoye and Naala 2017 and Uche & Abubakar 2009).

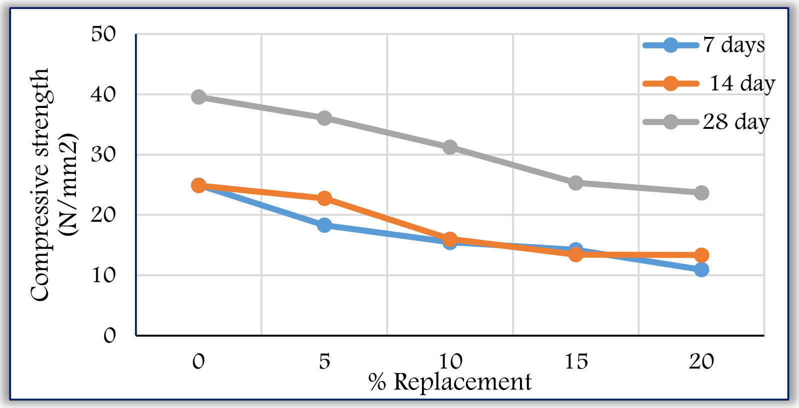

Figure 3: Compressive strength test result for 7, 14 & 28 days for varying percentage replacement

» Modulus of elasticity

The static modulus of elasticity was determined in accordance with the provisions of ASTM C469. The specimens were tested for uniaxial compressive load after 28 days of curing using universal testing machine. The stress-strain characteristics were determined and the elastic modulus measured as a tangent to the elastic range. The result shows that concrete containing 10% NSHA had an elastic modulus of 32822.79MPa while that of the control (without NSHA) has an elastic modulus of 20054.4MPa, which indicates that the elastic properties of the concrete are greatly improved by the use of 10% NSHA.

» Micro analysis

The micro analysis on the concrete was carried out using SEM and XRF. While the SEM analysis provided an insight into the morphology of the concrete with and without the NSHA, XRF analysis gave the oxide composition of the NSHA. The result of XRF is presented in table 1 while the result of SEM is presented in plate 1 and 2 and fig. 3 respectively,

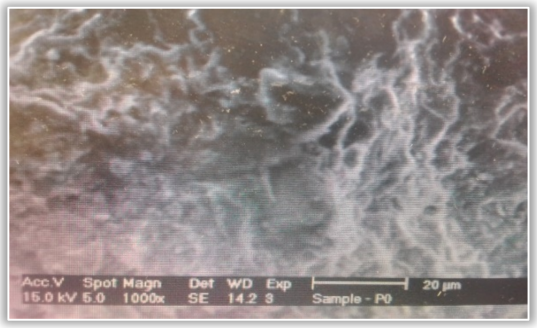

Plate 1: SEM for OPC concrete (0%NSHA)

Plate 2: SEM for concrete with 10% NSHA

A comparism of the result in plates 1 and 2 shows that concrete containing NSHA has a denser structure as compared to the control (without NSHA). This could be due to the reduced porosity of the concrete due to the filling of the pore spaces with the NSHA particles. This result agrees with what is obtainable in literature.

— Nanoscale analysis

Table 3 gives a summary of the results of the fraction volumes calculated using the model distribution curves in combination with the theoretical line of best fit by calculating the area under each distribution.

Table 3: Nano-mechanical properties of the phases studied

Binder	Phase studied	Mean of the modulus of Elasticity (GPa)	SD of the modulus of Elasticity (GPa)	Mean of the Hardness (GPa)	SD of the Hardness (GPa)	Fraction Volume
Control (OPC only)	Porous Phase	10.0	3.79	0.22	0.15	12
	LD C-S-H	16.5	4.26	0.43	0.13	63
	HD C-S-H	27.1	4.54	0.97	0.26	17
	CH	36.9	5.20	1.32	0.25	8
10%NSHA 90%OPC	Porous Phase	9.2	3.27	0.17	0.13	9
	LD C-S-H	17.5	2.95	0.49	0.17	60
	HD C-S-H	26.0	3.31	0.92	0.14	26
	CH	31.8	2.49	0.99	0.08	5

LD C-S-H and HD C-S-H are the products of hydration while the porous phase denotes the porosity of the sample studied. The reduction of the porous phase in cement pastes containing NSHA is due to the reduced amount of voids within the cement matrix as a result of the very fine CSA particles filling the empty voids between the hydrated cement particles. It could also be due to the filling of the voids by the additional products of hydration arising from the formation of addition C-S-H due to the pozzolanic reaction. It is therefore no wonder that there is a reduction of the porous phase with the addition of 10% NSHA by 25% after 28 days of curing.

Furthermore, the inclusion of 10% NSHA in the cement paste produces a pozzolanic reaction, turning calcium hydroxide into additional C-S-H, which has a much greater volume than the calcium hydroxide phase that it replaces. There is a decrease in the volume of CH by 37.5% with the addition of the CSA. This reduction in volume of the CH produces evidence of pozzolanic activity. This is confirmed by an increased in total volume of C-S-H by about 10%. This suggests that the pozzolanic reaction either forms more C-S-H or improved packing in the sample containing NSHA facilitates formation of more HD C-S-H thereby greatly improving the HD C-S-H to LD C-S-H ratio. Since the volume of the cement paste itself does not change, the increased volume of C-S-H gel should reduce the porosity of the cement matrix as shown by the reduced porosity reported in this work. This result agrees with what is obtainable in literature. Addition of 10% NSHA increases both the general volume of C-S-H present in the sample (7.0%) and that of HD C-S-H (34.6%).

3. CONCLUSION

The multiscale characterization of concrete containing NSHA provides a better insight into the behaviour of concrete containing NSHA in the binder matrix. The following are deductions from this investigation.

- The microscale characterization using SEM and XRF reveals that NSHA contains about 35% of SiO_2+Fe_2O_3+Al_2O_3 - which is less than 50% stipulated by ASTM C618, however, the CaO content of up to 32% means that it has cementitious properties, more so, the addition of 10% NSHA produces a denser structure in the concrete.
- The use of NSHA in concrete improves the elastic properties of the concrete but lead to a reduction of the strength of concrete at all ages.
- The nanoscale characterization using nanoindentation reveals that the use of NSHA in concrete causes a reduction in volume fraction of portlandite due to the pozzolanic reaction. There is also reduced porosity and an increase in volume fraction of HD C-S-H as well as a slight increase in the overall volume fraction of C-S-H. This could either be due to the pozzolanic reaction or improved packing.

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