Engineering Properties of Agro Waste Ash-Cement Mortar Incorporating 0.5% Nano Silica

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Abstract- Urbanization in Nigeria requires a huge quantity of cement. The atrocious impact of cement on the environment necessitates novel and greener locally developed *materials*. *Supplementary* Cementitious Materials (SCMs) offer possible solution. This study investigated some engineering properties of cement mortars containing Nano silica (NS), Rice Husk Ash (RHA) and Groundnut Shell Ash (GSA). Rice husk was burned to ash at 700°C while groundnut shells were slightly milled and burned to ashes at 600°C in a furnace to obtain the RHA and GSA respectively. NS was synthesized from RHA. Mortar mixing ratio 1:3(binder/aggregate) and 0.5 (water/binder) were adopted. Cement was partially replaced with NS (0, 0.5%), GSA (0, 7.5, 10, and 12.5%) and RHA (0, 5%) to produce 8 mixes named M0-M7. The particle size, specific surface areas and chemical compositions of the SCM were determined. Fresh mortar was tested for workability and cast mortar cubes were tested for compressive strength, abrasion resistance and water absorption. Results indicated that NS had the least particle size (46 nm), highest specific surface area (72 m2/g) and the sum of the three pozzolanic oxides was 89% while RHA and GSA were 78 nm, 42 m2/g, 81% and 94 nm, 43 m2/g, 27%, respectively. Workability reduced with rise in SCM content. Compressive strength improved with the addition of the combined SCMs. Inclusion of NS improved both the abrasion resistance and the water absorption. Conclusively, 0.5% NS enhanced the engineering properties of the mortar.

Indexed Terms- Impact of cement, Engineering properties, Supplementary Cementitious Materials

I. INTRODUCTION

The construction industry utilizes a high volume of cement as binding material. The over dependence on this incredible material has resulted into serious

degradation of natural resources, high construction cost due to outrageous cement prices and release of greenhouse gases in heavy dose into the atmosphere [1], [2]. These have led to the search for sustainable alternative materials. Agriculture is a renowned business in Nigeria which generates a lot of wastes. Most times, the wastes are dumped in landfill and burnt for no advantage except to provide chance for successive wastes generated. Otherwise, they are allowed to decay, generating notorious pollution in the environment [3]. Ashes of some agricultural wastes such as rice husk, groundnut shells, cassava peel, sugarcane bagasse and coconut fiber are recently being processed for use as SCM. Effectively using agro waste materials in building will reduce environmental pollution, raw material shortages, energy crises, and construction overhead expenses [1], [14] The characteristics of concrete, mortar, and other composites can be enhanced as well. Nigeria produces rice, an indispensable meal. Rice husks are dumped as rubbish. The fine, powdery residue left after burning rice husks under controlled conditions is called RHA, which possesses high silica concentration for pozzolanic reaction and could replace cement in concrete. The use of RHA as SCM was first patented in 1924 [4]. . Many projects have used it since.

[5] investigated the effects of RHA and superabsorbent polymer (SAP) on the characteristics of cement-based composites by the individual or combined incorporation of varying amounts of RHA and SAP. They found that 0.15% SAP combined with 5% RHA markedly enhanced the strength of concrete and refined its microstructure. The diminutive particle size of RHA occupied the voids created by SAP during water release, hence improving the microstructure of the concrete by filler effect. [6] asserted that RHA comprises amorphous silica, which can serve as a pozzolan in the production of concrete and mortar, provided the optimal proportion is not exceeded. [7] documented a positive correlation between a 15% substitution of RHA and an approximate 20% enhancement in compressive strength over the control mix. The optimal strength and durability features were achieved with an addition of up to 20%, beyond which there was a modest reduction in strength parameters.

Groundnut is a renowned cash crop. The shells are the outer envelope encasing the seed. GSA is obtainable by burning the shells. [8] reported that 10% GSA replacement of cement gave 2.95% and 1.21% improvement on compressive strength at 7 and 28 days curing respectively when compared to control concrete. Optimum of 10% GSA gave lower water absorption, had better resistance to acid and chloride attack compared with control concrete.

Many domains of application have focused on nanoparticles to create novel useful materials. Ultrafine particles, when mixed with Portland cement paste, mortar, or concrete, produce unique materials [11]. The effectiveness of cementitious materials depends on nano-sized solid particles. O wing to its ultra-fine particle size and excellent pozzolanic activity, NS is increasingly employed as a SCM mortar and concrete. If applied in the optimal dosage, it may drastically alter the mortar's fresh and hardened qualities [10], [[9].

Numerous studies have explored the influence of NS content, with a consensus emerging around optimal dosage ranges and potential adverse effects of overdosing. [11] reported the optimal NS content for mortar and concrete typically lies between 1% and 3% by weight of cement, stating that this range balances the benefits of enhanced properties with rheology and economy. [12] reported a linear reduction in slump with increasing NS content, particularly beyond 3% by weight of cement. NS inclusion gave 20-30% improvement of the compressive strength compared with control mortar. At early age 7 days, a 23.3% increase in tensile strength over conventional concrete was recorded [13]. Improvement in the mechanical properties was linked with pozzolanic reaction forming additional C-S-H gel.

[14] stated that Nano silica enhanced the durability of mortar by refining the pore structure and caused a significant reduction of 30% in the water absorption with a 2% NS content. The inclusion of Nano silica can significantly improve mortar properties when used in appropriate dosages. It enhances strength, durability, and microstructure, though it reduces workability and may increase shrinkage. The effectiveness of Nano silica is highly dosagedependent, and proper dispersion techniques and admixture use are critical to maximize its benefits [11], [15]. On the general note, SCMs can lead to substantial decrease of the carbon footprint of concrete, and therefore, are vital to achieve sustainability in the construction industry [16].

II. METHODS

The materials used in preparing the mortars investigated in this work were river sand, cement, rice husk ash, groundnut shell ash, Nano silica particles, and drinkable water. River sand used was dry fine grained well graded sand. The sand was suitable for mortar production. Portland limestone cement (32.5N) produced in Nigeria was used. All the cement used for this study was acquired in one batch from the local construction market in Ado Ekiti. It was ensured that the cement was far from the expiry date as shown on the packaging bag.

RHA was produced by calcining rice husks at 700°C in a furnace at the Department of Science Technology, the Federal Polytechnic, Ado-Ekiti. The ash was further grinded and sieved. Also, GSA was obtained by burning dry groundnut husks to ash at a temperature of 600°C in a furnace. Also, the ash was grinded and sieved to get finer ash required for the experiments. The Nano silica particle was obtained from RHA through precipitation process and sol-gel technique. RHA was mixed with sodium hydroxide solution, heated and filtered. Hydrochloric acid was gradually added to the filtrate, sol-gel was formed and repeatedly washed with distil water to remove sodium chloride from the solution, filtered and the residue was finally dried to obtain the white fine Nano silica particles. Particular safety precautions were taken during the production and application of the NS. These included wearing of nose masks, hand gloves and ensured cross ventilation in the laboratory.

Mixes were prepared as designed using the Department of Environment (DoE) method. Water to

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binder ratio was 0.5 and binder to sand ratio was 1:3. The preliminary tests performed on the materials were: particle size distributions, specific surface areas and chemical composition. The tests were done at the National Steel Raw Materials Explanation Agency Kaduna, Kaduna state. Cement was partially substituted with GSA, RHA and NS to produce 8 mixes namely; M0, M1, M2, M3, M4, M5, M6, M7. The detail content and description is represented in Table 1.

The mortar was prepared manually in the laboratory. The river sand was initially poured on a clean flat solid surface; the binder components (cement, NS, GSA, and RHA) were batched according to the mix proportion calculated, these were thoroughly dry mixed for few minutes. The water was gradually poured while mixing continued till the water was fully poured and the mixing was thorough. Conventional manual mixing method was used for the work. Workability of fresh mortars was determined using the slump cone. Freshly mixed mortar was scooped and compacted in two layers into the moulds that had been oiled to avoid sticking of materials to the surface. After compaction, the surface of the mortar-filled mould was smoothed and scraped off. The mortar-filled moulds were covered with polythene sheet for 24 hours and left intact in the laboratory. Cast mortar cubes were cured by full immersion in water and tested for compressive strength at 7, 28 and 56 days, abrasion resistance and water absorption.

0

M6 (G10R5N0.5)	6.5 0	1.86	0.22	0.11	0.01	1.10
M7 (G12.5R5N0.5)	6.5 0	1.80	0.28	0.11	0.01	1.10

III. RESULTS AND DISCUSSION

A. Physical characterization

The constituent materials utilized for this study were tested in accordance with ASTM standards. The results of the characterization are presented in Table 2. The fine aggregate used was continuously graded fine river sand with a specific gravity of 2.74 and has a fineness modulus (FM) of 2.5, indicating that the aggregate is fine grained. According to BS 882 (1992), the sand was appropriate for mortar production.

The cement used for this work was Portland limestone cement CEMII B-L 32.5R produced in Nigeria. It was a gray color powder whose specific gravity was 3.12. The GSA had a particle size of 94 nm, specific surface area 43 m²/g and a specific gravity of 1.81. GSA was a brownish gray powder. RHA was a light gray smooth powder with a particle size of 78 nm, specific surface area of 42 m²/g and a specific gravity of 2.20. The NS was pure white powder synthesised from the light gray RHA. It had a particle size of 46 nm, specific surface area of 72 m²/g and a specific gravity of 2.06. The high surface area of NS may enhance its reactivity over the other additives.

Та	ble 1.	Mortar	mix des	sign			Pro	perties	Sand	Cement	GSA	RHA	NS
Mix ID	San	Cem	GSA	RH	NS	W	Par	ticle	Continuously		94.14 nm	78.11	46.36
	d	ent	(kg)	А	(kg)	r	size	;	graded fine			nm	nm
	(kg	(kg)		(kg)		(k	g)Spe	cific	-	-	43	42	72
)						surf	ace					
M0 (Control)	6.5	2.20	-	-	-	1.	10area	ı					
	0						(m ²	/g)					
M1 (G7.5)	6.5	2.04	0.17	-	-	1.	Spe	cific	2.74	3.12	1.81	2.20	2.06
	0						ora	vity		0.112	1101		2.00
M2 (G10)	6.5	2.00	0.22	-	-	1.	Col	or	Brown grains	Gray	Brownish	Light	White
	0							01	biown grains	Ulay	DIOWIIISII	Ligin	winte
M3(G12.5)	6.5	1.93	0.28	-	-	1.	10			powder	gray	gray	powder
	0										powder	powder	
M4 (R5N0.5)	6.5	2.08	-	0.11	0.01	1.	10						
	0							В.	The chemical co	mposition			
M5 (G7 5R5N0 5)	65	1 91	0.17	0.11	0.01	1 10 The chemical compositions of the three SCM used as			0				

Table 2. Physical characterization of materials

The chemical compositions of the three SCM used as partial substitution for cement in this investigation were determined through X-Ray Fluorescent (XRF)

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test performed at the National Steel Raw Materials Exploration Agency, Kaduna, Nigeria. The results indicated that the summation of the major oxides for pozzolanic reactions (SiO₂+Al₂O₃+ Fe₂O₃) in the materials differed. The sum of these oxides in GSA, RHA and NS were 27, 81 and 89 per cent respectively. The results depicted that GSA does not belong to the family of pozzolans. However, RHA and NS are pozzolans of high reactivity judging from their oxide composition values. ASTM C 618 (2019), stated that for a material to be pozzolanic, the sum of the oxides must be greater than or equal to 70%. [2] and [17] reported that the sum of the required oxides was very low in GSA, therefore, it is not a pozzolanic SCM. In contrast, [18] and [13] reported that NS and RHA are top rated pozzolans.

C. Workability

The workability of the mortar mixes as measured through slump is presented in Table 3. All the slumps observed were true slump. The control mortar recorded a slump of 26.5 mm. Increase in GSA content resulted in simultaneous increase in the slump values. For instance, M3 (C87.5G12.5) had a slump of 48 mm which is the highest among all the mixes. The effect of the addition of GSA is notable on the improvement of the workability of the binary mixes M1, M2 and M3 which gave higher values than the control mortar mix M0. This characteristic is attributable to the hygroscopic nature of GSA. Arivu and Selvan, (2017) concluded that GSA improved concrete workability. It is observed that all the mixes containing the combination of cement and the three additives (GSA, RHA and Nano silica) recorded zero slump. Nanosilica decreases slump due to its high specific surface area and small particle size. [2] reported a similar observation. Also, [20] reported a very low slump in their comparative study on effects of class F fly ash, Nano silica and silica fume on properties of high performance self- compacting concrete.

Mix ID	Description	Slump
	Description	(mm)
M0	C100	26.5
M1	G7.5	32
M2	G10	36
M3	G12.5	48
M4	R5N0.5	8
M5	G7.5R5N0.5	0
M6	G10R5N0.5	0
M7	G12.5R5N0.5	0

Table 3. Workability

D. Compressive strength

The compressive strength of the mortar mixes measured at 7, 28 and 56 days of curing are presented in Figure 1. At 7 days maturity, mortar mixes M1, M2 and M3 containing binary binders (cement and GSA) recorded lower compressive strength than the control mix. For instance, the mixes M1, M2 and M3 were about 30%, 12% and 18%, respectively lower than the control mix M0. This effect can be linked with the fact that GSA is not a pozzolanic SCM as indicated by its oxide composition. However, the mixes with ternary and quaternary binders (i.e. M4, M5 and M6) had higher compressive strength than the control. The mixes M4, M5, M6 had 24 %, 54 % and 73% increments respectively, in compressive strength over mix M0. The observed enhancement in compressive strength can be attributed to the effects of RHA and NS inclusion. The inclusion of 5% RHA and 0.5%NS improved the strength through the filler effect. Mix 7 had 17% reduction in compressive strength compared to the control in spite of incorporating cement, GSA, RHA and NS like M5 and M6. This performance can be connected with the dilution effect caused by 12.5% GSA in the mixture.



Figure 1. Compressive strength of mortar

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At 28 days, all the mixes had experienced appreciable increments in compressive strength compare to their strength at 7 days. The improvement in compressive strength at 28 days ranged from 16% to 44% over the strength at 7 days. This observation is linked with hydration of cementitious media and curing progression. The design strength obtained at the 28 days of curing ranged between 9.5 and 15.6 N/mm2 for the mixtures. Mortar mixes M4, M5 and M6 recorded higher strength than the control while the remaining mixes had lower compressive strength than the control. Dilution effect may be responsible for this behavior.

Considering the compressive strength at 56 days of maturity, all the mixtures had recorded advancement in strength compared to what was obtained at the previous ages investigated. Mixes M4, M5, M6 and M7 recorded 28%, 42%, 37% and 18% increments respectively over the control mix M0 at 56 days of curing. The enhanced performance at this later age is attributable to the pozzolanic reactivity of the NS and RHA by forming extra C-S-H gel and causing additional densification of the microstructure. The improvement in strength is also caused by accelerating the hydration process. Pehlivan, (2022) stated that Nano silica particles were also found to be efficient in enhancing the compressive strength of concrete and mortar.

It was observed generally that mixesM1, 2, 3 gave lower compressive strength than the control at all tested ages. At the 56 days maturity, the 3 mixes recorded 13%, 15% and 3% reduction in compressive strength compared with the control. This indicates that GSA can only perform as a filling SCM.

E. Abrasion Resistance

The abrasion resistance of the mortar mixes incorporating varying proportions of cement, GSA, RHA and NS are depicted in Figure 2. The results showed that all the mortar mixes had varying resistance to abrasion. The control (M1) and mix M3 gave equal abrasion resistance of 20%. The value of resistance offered by the other mixtures ranged from 32% to 100%. Mortar mixes containing GSA were more resistant to abrasion than the control mix except M3 that that had competitive resistance as the control. This proves that up to an optimum GSA content of 10% replacement, filler effect of GSA as a SCM is noticeable, while at higher composition level, (M3 containing 12.5% GSA), dilution effect has reduced its resistance capacity against abrasion.

Mortar mixtures M5, M6, and M7, which contained cement, GSA, RHA, and NS, were 4, 5, and 3 times more resistant than the control mix M0. The mixes show that RHA and NS as reactive pozzolans produce stronger products following hydration. Pillai et al. (2020) found that SCMs improve concrete durability, making them vital for sustainability. The high specific surface area of NS particles must have helped performance. This behavior is linked to microstructure densification and SCM filler effects in mixtures. Abrasion resistance was best in mix M6, which had the maximum compressive strength at all ages.



Figure 2. Abrasion

F. Water absorption

Cement mortar mixes with varied GSA, RHA, and NS concentrations absorb water as shown in Figure 3. The water absorption reduced with increase in the percentage of SCMs replacing cement in the mortar. For instance, the control mortar mix (M0- C100) gave an average water absorption of 7.5% while mix M2 (C90G10) had 4.9 % and mix M7 blending C82G12.5R5N0.5 gave 2.1 % average water absorption. This observation may be attributed to the filler effect of the SCMs added especially the Nano silica particles which filled up the voids and thereby creating a densely filled up pores.

It is noted that the average water absorption rate of binary mortar mixes incorporating only cement and GSA (M1, M2 and M3) gave values that are lower than that recorded by the control mortar. The effects of RHA and NS were more pronounced in the mixes than those incorporating only GSA as a result of their higher specific surface areas and the stronger pozzolanic reactivity. All the mixes containing Nano silica recorded massive reduction in water absorption much less than the control mortar mix M0. For instance, the values for mortars containing 0.5% NS (vis: mixes M4, M5, M6, and M7) ranged from 2.1 % to 3.1% while control had 7.5 % i.e. the control mix absorbed twice to thrice as much water as the samples with NS. This behavior can be connected with the fact that the use of NS enhances the mortar microstructure through its filler impact and pozzolanic reaction. The microstructure must have been better refined uniformly and resulting into reduced water absorption. [22] reported similar observation majorly at the ITZ of concrete containing Nano silica. Nano-silica prevented and filled harmful components' pathways through concrete matrix. In all mortar mixes, M7 absorbed the least water and M0 the most.



Figure 3. Water absorption

CONCLUSION

The Nano-silica decreased slump due to its high specific surface area and small particle size. The inclusion of 5%RHA and 0.5%NS improved the strength through the filler effect. Mortar mixes containing GSA were more resistant to abrasion than the control mix. The filler effect of the SCMs especially the Nano silica particles which filled up the voids and thereby creating a densely filled up pores recorded lower water absorption. The inclusion of SCM in cement mortar enhance the engineering properties and performance.

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