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ORIGINAL RESEARCH ARTICLE

MECHANICAL AND MICROSTRUCTURAL CHARACTERIZATION OF ALKALI-ACTIVATED COCONUT SHELL ASH MORTAR

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ABSTRACT

In this paper, potential of alkaline activation of coconut shell ash (CSA) as binder in mortar was investigated. Coconut shells were collected and calcined at temperature ranges between 500 and 900°C at interval of 100 for 30, 60 and 90 minutes to determine optimum burning condition that produces ash of higher silica content. Chemical composition of the ashes was then determined using X-ray Florescence (XRF) technique. Sodium hydroxide (NaOH) of different concentrations of 10, 12, 14 and 16 Molar (M) was used as alkaline activator, while mortar of mix ratio 1:3 was produced, while ratio of the alkali to CSA was of 0.5. Mortar prisms of size 40 mm × 40 mm × 160 mm were cast and cured in an oven at a temperature of 80 ℃ for 7, 28, 56, 90 and 120 days. Thereafter, flexural and compressive strengths were determined at the end of each curing ages, following standard procedures. Scanning Electron micrographs of the hydrated mortar at 120 days were obtained using scanning electron microscopy (SEM) The results showed the oxide composition did not follow any pattern with increase in temperature but CSA produced at 800 ℃ for 1h gave the highest combined silica, alumina and ferric oxides above 70%, which is accepted as minimum value for pozzolanic materials. Both compressive and flexural strengths of the activated mortar samples were found to increase with increase in activator concentration up to 14M and decline thereafter. At 120 days, the compressive strength was 13.9 N/mm^2 while the flexural strength was 6.88 N/mm^2. These values were higher than the strengths recommended by Nigerian Industrial Standard (NIS) for load bearing blocks. It was concluded that activation of CSA with NaOH could be used as binder for non-structural use. The SEM results indicated that mortar made from 14 M had structure that was densely packed compared to other mortar samples produced from other concentrations

1.0 Introduction

The production of modern cement (ordinary Portland cement) consumes huge energy and has been implicated as the major contributor to greenhouse gas emission next to the automobile industry. It is estimated that the cement industry contributes between 6 and 10% of the total annual CO_2 emitted (Metz, 2007, and Carstensen and Rapf, 2008); while about 5.5 million BTU of energy is required to produce just one tonne of cement (Naik, 2008).

Unfortunately, the demand for ordinary Portland cement (OPC) is ever increasing, especially in developing countries, like Nigeria, where there is thirst for infrastructure development. Oluwakiyesi (2011) reported that Nigeria has an estimated deficit of 16 to 18 million housing units and that in 2009, the Presidential Committee on Implementation of Affordable Housing estimated about N60 trillion to finance the deficit. This would require about 112 million metric tonnes (mmt) of cement. Globally, the demand for cement in the year 2015 was 4.6 Gt/a (CEMBUREAU, 2015) and could reach 13.5 Gt/a by 2050 (Edwards, 2016). If this trend continues unabated, then the yearning for clean and sustainable environment would be a mirage in the face of meeting infrastructure deficit.

In responding to this challenge, several efforts are being made to curtail the amount of OPC consumed locally for global objective. One of such emerging and attractive approaches is alkali-activation of waste materials. Large amount of industrial and agricultural waste materials is produced, effective disposal of which remains a challenge with adverse effect on the ecosystem at large. The volume of these wastes generated in the world is increasing over the years due to increase in population, socio-economic activities and social development.

Alkali activation as defined by Martinez and Palomo (2001) is a chemical process where amorphous structure is transformed into a skeletal structure that exhibits cementitious properties. Once a material contains reactive silica or alumina, it could be considered for activation using appropriate alkali solution (equations 1a, 1b and 1c). Several materials and alkali have been studied with positive results.

$$\mathrm{Ca} + 2(\mathrm{OH}) + \mathrm{SiO_2} \rightarrow \mathrm{C-S-H(Gel)\ as\ in\ OPC} \quad (1a)$$

$$\mathrm{Na} + 2(\mathrm{OH}) + \mathrm{SiO_2} \rightarrow \mathrm{N-S-H(Gel)\ as\ in\ geopolymer} \quad (1b)$$

$$\mathrm{Na} + 2(\mathrm{OH}) + \mathrm{Al_3O_2} \rightarrow \mathrm{N-A-H(Gel)\ as\ in\ geopolymer} \quad (1c)$$

Where -: $\mathrm{C-S-H}$ and $\mathrm{N-S-H}$ (Gel) are Calcium-Silicate-Hydrate and Sodium-Silicate-Hydrate Gels, respectively.

Bakharev (2006) produced concrete using alkali activated fly ash with NaOH and achieved a 2-day compressive strength of 10 N/mm^2 while the 28-day compressive strength was 60 N/mm^2. The Author also used sodium silicate (Na_2SiO_3) to chemically activate fly ash to achieve a 2-day compressive strength of 2 N/mm^2 and a 28-day compressive strength of 45 N/mm^2. Similarly, Morenoa et al. (2008) also activated class F fly ash with NaOH from different sources and achieved a 28-day compressive strength of between 29 and 66 N/mm^2. Another study by Sofia et al. (2007) used class F fly ash with one or more of the following component; Sodium Carbonate (Na_2CO_3), Sodium Silicate (Na_2SiO_3) and Sodium Hydroxide (NaOH), to achieve 7-day compressive strengths in the range of 35 to 44 N/mm^2 and a 28-day compressive strength in the range of 47 to 57 N/mm^2.

Rajesh et al. (2013) studied mechanical properties of alkali activated slag with different alkalis. They reported that a significant compressive strength was achieved especially with sodium silicate and sodium hydroxide solutions, which has one-day strength of 12.46 N/mm^2 and 12.04 N/mm^2 respectively compared with 9.5 N/mm^2 for the control mix and 5.73 N/mm^2 for the Sodium Carbonate mix. It was concluded that NaOH was alkali for activating slag for higher strength. Qureshi and Somnath (2013) used potassium oxide (K_2O) and Sodium oxide (Na_2O) to activate blast furnace slag in order to investigate the relationship between alkali content and compressive strength of concrete. Durability of alkali activated cement-based products was equally studied (Bernal et al., 2011 and Atahan and Dikme 2011). Agricultural wastes are equally being used, though sparsely. Palm Oil Boiler Ash (POFA) with sodium hydroxide and sodium-silicate, using different concentrations of sodium hydroxide alkaline activator was studied (Zarina

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et. al., 2015). Based on authors' knowledge, there is no reported work on the alkali activation of coconut shell ash as binder; hence, this study was carried out to fill the research gap.

Coconut is a fruit from the coconut palm tree. It is grown in large quantity according to Food and Agriculture Organization. About 0.3 mmt of coconut was grown in 2014 as against about 0.1 mmt and 0.12 mmt produced in the 60's and 80's respectively (Figure 1). It is equally widely grown in Nigeria especially in the riverine areas of the country. During processing of coconut for oil production, large quantity of coconut shells is produced, disposal of which remains a challenge. Most often than not, they are piled up and burnt to give room for new set of coconut shells produced. This study therefore investigated suitability of coconut shell ash (CSA) as potential precursor for alkali activation.

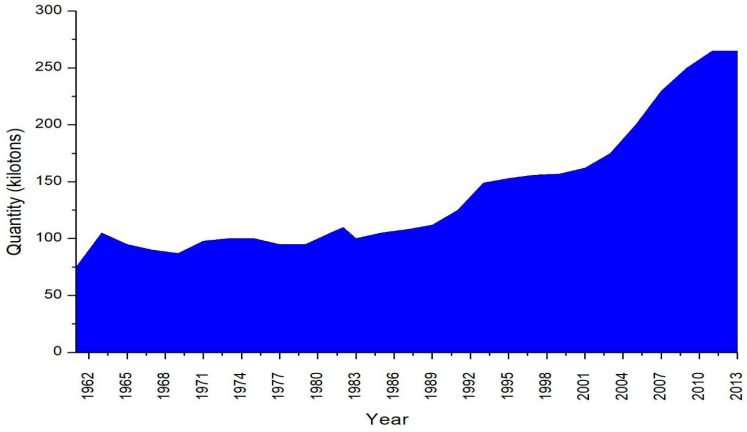

Figure 1: Coconut production quantity in tons between 1961 and 2013 (FAOSTAT, 2016)

2.0 Materials and Methods

2.1 Materials

Coconut shells were obtained from coconut shell dumpsite in Badagry area of Lagos State, Nigeria (Figure 2). River sand of maximum nominal particle size of 3.18 mm was used as the fine aggregate. The sand was washed to remove dirt and deleterious substances. Specific gravity of the sand was determined as well as sieve analysis was conducted on the sand to determine its particle size distribution following the procedures specified in BS EN 1097-3 (1998). Sodium hydroxide (NaOH) was used as alkali activator, while ordinary Portland cement (OPC) of grade 42.5 was used as binder for control sample.

Figure 2: A typical coconut shell dumpsite at Badagry, Nigeria

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2.2 Preparation of specimens

2.2.1 Coconut shell ash

The shells were thoroughly washed in water to remove all the organic matters that stuck to them (Figures 3a and 3b). Thereafter, they were dried under the sun to minimize moisture and were crushed into smaller sizes manually with the aid of harmer to facilitate burning.

The crushed shells were burnt in a furnace at different temperature ranging between 500 ℃ and 900 ℃ at an incremental interval of 100 ℃ for 30, 60 and 90 min, respectively. A total of 15 samples of the ash were prepared. For instance, Sample A1 represents CSA produced by burning at 500 ℃ for 30 min (Table 1).

(a) (b)

Figure 3: Crushed coconut shell (a) before cleaning and (b) after cleaning

Table 1: Samples of ash produced at different temperatures and duration

Sample	Temperature (℃)	Duration (Min)
A1	500	30.00
A2	500	60.00
A3	500	90.00
B1	600	30.00
B2	600	60.00
B3	600	90.00
C1	700	30.00
C2	700	60.00
C3	700	90.00
D1	800	30.00
D2	800	60.00
D3	800	90.00
E1	900	30.00
E2	900	60.00
E3	900	90.00

2.2.2 Sodium hydroxide solution

Sodium Hydroxide (NaOH) in pellet form was purchased from Purechem Manufacturing Company in Lagos and was used to prepare the various concentrations for the experiment. The different concentrations prepared for this study were 10, 12, 14 and 16M. The choice of concentrations was based on findings from previous studies (Ammar et al., 2013 and Qureshi and Ghosh, 2013). Table 2 summarized the quantity used in preparing the NaOH solution.

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2.2.3 Alkali activated coconut shell ash mortar

The alkali activated coconut shell ash mortar (AACSAM) was prepared by mixing a measured amount of coconut shell ash (CSA) with the alkali activator (NaOH-solution) in a ratio of 1:2 (alkali: CSA) by weight to produce an alkali activated coconut shell ash paste (binder). The resulting binder was in turn mixed with fine aggregate to finally produce an AACSAM in a binder/sand ratio of 0.5 by weight.

Table 2: Quantity of NaOH Per Liter in each Concentration

Molarity (M) of solution	Amount of NaOH solid (g)	Quantity of water (L)
10	400	1
12	480	1
14	560	1
16	640	1

The mortar produced was cast in moulds of sizes $40 \times 40 \times 160$ mm (ASTM C78, 2018). A total of 60 short mortar beams were cast and the moulds were removed after 24 hours (Figure 4a). Thereafter, they were cured in oven at temperature of 80 ℃ for 7, 28, 56, 90 and 120 days (Figure 4b). The choice of temperature and mixing proportions were guided by principles found in the literature that the sample be cured under a temperature of 80 ℃ till the period of testing (Andrea and Herald, 2004).

(a) (b)

Figure 4: Alkali Activated Coconut Shell Ash Mortar (a) in moulds (b) stack in oven for curing

2.3 Testing procedures

2.3.1 Determination of chemical composition of CSA

Chemical composition of the 15 ash samples were determined individually, using the X-Ray Fluorescence (XRF) technique. Weltje and Tjallingii (2008) give the detail procedure of XRF technique. The test was conducted at Lafarge Cement PLC, Ewekoro plant. Statistical Package for the Social Sciences (SPSS) software was used to conduct analysis of variance (ANOVA) on the data to study the effect of the activation conditions (temperature and time) on the chemical composition of CSA. The temperature and time that gave high amorphous silica content was then used to produce the ash used for the activation.

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2.3.2 Determination of Compressive and Flexural Strengths of AACSAM

At the expiration of each curing period, the prism was weighed and the flexural strength of the mortar prism was first determined. The experimental set-up for determining the flexural force is shown in Figure 5. The amount of force that broke the sample into two halves was recorded as flexural force. Thereafter, flexural strength was calculated using Equation 2. Each halves of the prism from flexural testing was used as samples to determine the compressive strength. This is in conformity with the provisions of BS EN 196 -1 (2015).

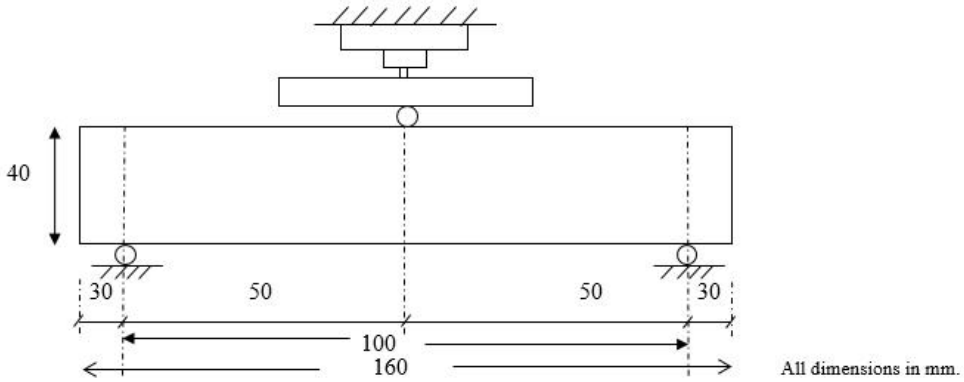

Figure 5: Experimental set-up for flexural testing

$$f_s = \frac{1.5Fl}{b^3} \qquad (2)$$

where:

f_s = flexural strength, N/mm^2

F = flexural force, N

b = side of prism square section, mm

l = span between supports, mm

3.0 Results and Discussion

3.1 Chemical properties of CSA at various burning conditions

Table 3 presents the chemical composition of the produced CSA at different temperature and duration periods. It was observed that as the time of burning increased, the percentage oxides of CSA varied, suggesting that duration of burning had effect on the chemical composition of CSA produced. For instance, the silica (SiO_2) content of the ash obtained at 500 ℃ for 30 minutes was about 75% and 65% of what was obtained when the time was increased to 60 and 90 minutes, respectively. The same trend (increased in SiO_2 content as time increased), though in different proportions, was observed for other temperatures from 600 ℃ to 900 ℃ except at 800 ℃. Looking at the effect of burning temperature, the results indicated that temperature had significant impact in determining the amount of oxides obtained. For instance, Coconut shell ash burnt for 30 min.. at 600 ℃ produced alumina content to be 5.4%, while it was 9.24% at 800 ℃ for the same duration. The thrust of these variations implied that there was individual and combined effect of temperature and duration on the chemical composition of CSA. Similar observation was reported in the literature, but for cassava peel ash and rice husk ash (Salau and Olonade, 2011 and Ramezanianpour et al., 2009).

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Table 3: Effect of Calcining Temperature and Duration on Chemical Composition of CSA

Temp. (℃)	Time (min)	Oxides (%) SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	LOI
	30	32.36	5.24	1.66	3.69	0.49	0.14	2.26	1.71	41.42
500	60	43.12	7.05	2.07	4.65	0.66	0.08	2.85	2.27	33.25
	90	49.73	8.08	2.53	5.46	0.78	0.25	3.17	2.6	25.76
	30	32.51	5.40	1.58	3.8	0.51	0.46	2.02	1.37	29.87
600	60	36.00	5.76	1.71	3.84	0.55	0.06	2.3	1.93	43.33
	90	45.01	7.25	2.25	4.82	0.15	0.15	2.83	2.26	32.74
	30	44.2	7.38	2.47	4.95	0.72	0.17	2.94	2.41	31.06
700	60	28.01	4.59	1.24	3.28	0.43	0.12	1.92	1.5	47.43
	90	30.35	4.97	1.56	3.43	0.48	0.04	1.89	1.54	51.38
	30	58.86	9.24	3.20	6.29	1.03	0.46	3.58	4.11	9.73
800	60	58.76	9.18	3.15	6.35	1.05	0.40	3.50	4.00	9.54
	90	43.38	7.03	1.95	4.72	0.68	0.11	2.53	2.07	29.94
	30	45.24	7.28	2.27	4.78	0.69	0.09	2.42	2.06	27.61
900	60	36.14	5.97	2.19	4.28	0.6	1.01	2.12	1.68	22.96
	90	49.20	7.83	2.86	5.07	0.76	0.37	2.44	2.13	26.44
OPC		19.99	5.49	3.00	63.32	1.45	0.34	0.08	0.09	3.96

Nevertheless, the focus was to obtain CSA that contained reactive and higher content of combined SiO_2, Al_2O_3 and Fe_2O_3 that was above 70% as recommended by ASTM C618 (2006). The results further suggested that burning coconut shells at 800℃ for 60 minutes could be the appropriate treatment conditions to obtain CSA that would be an acceptable precursor for alkali activation because the combined silica, alumina and ferric oxide was more than 70% minimum recommended (Figure 4).

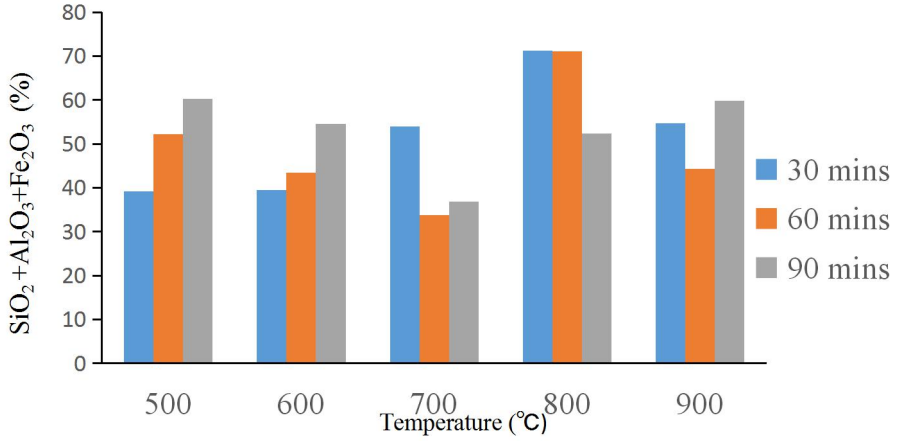

Figure 4: Combined silica, alumina and ferric at different temperatures and times

The summary of the multivariate tests conducted on the data presented in Table 3 is presented in Table 4. It is shown that there is a statistically significant effect of temperature on the combined silica, alumina and ferric oxide content of CSA ($p < 0.05$), while duration of burning did not have no significant effect statistically ($p > 0.05$)

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Table 4: ANOVA on the effect of Temperature and Time of oxide composition of CSA

Factor	SS	df	MS	F	p-value	F-crit
Temperature	108501.3	14	7750.094	1.072226	0.420296	2.063541
Time	4153178	2	2076589	287.2961	2.19E-19	3.340386
Error	202385.2	28	7228.044			
Total	4464064	44	4464064			

3.2 Physical and particle size distribution of Aggregate

Figure 2 shows the particle-size distribution curve of the sand. While less than 5% of the particle size passed through sieve size 150 μm, about 98% of its particle size passed through sieve size 2.36 mm indicating aggregate fell within the class of fine aggregate (BS EN 1097-3, 1998). The sand could be categorized as normal weight aggregate because its specific gravity was 2.67, which is within the range of 2.43 – 2.75 for normal weight aggregate (BS EN 1097-3, 1998).

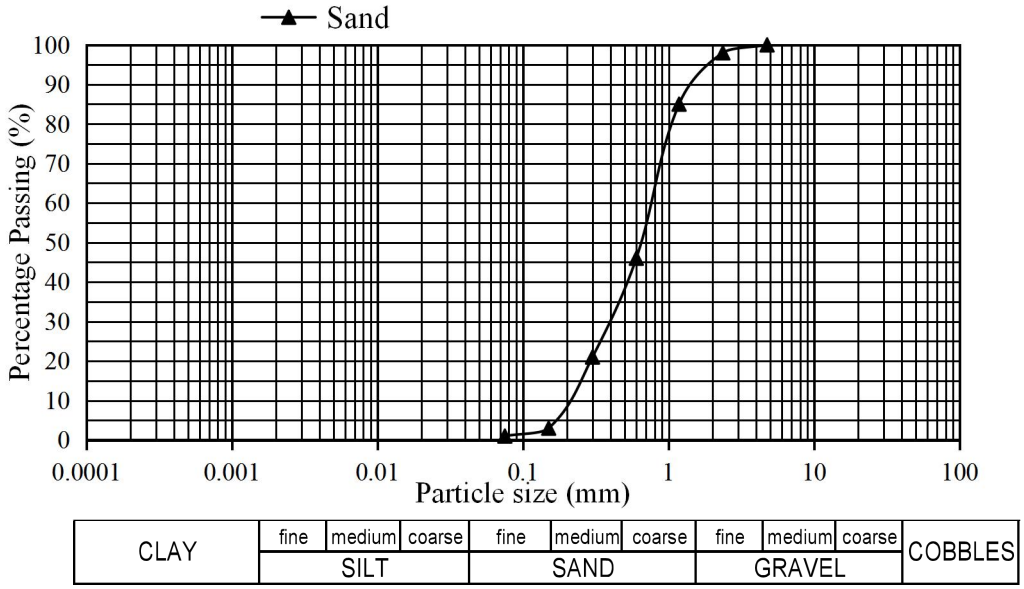

Figure 2: Particle size distribution curve of the fine aggregate

3.3 Strength characteristics of alkali activated coconut shell ash mortar

3.3.1 Compressive strength

Compressive strength of the AACSA mortar varied with concentrations of NaOH solution (Figure 5). At early age of 7 days, CSA activated with 16M of NaOH produced the highest strength (4.03 N/mm^2), which was about 102%, 116% and 125% of that of 10, 12 and 14M concentrations, respectively. However, at age 28 days, alkali activated mortar produced with 14M concentration had gain more strength, which was about double its strength at 7 days (9.9 N/mm2). Other mortar produced with 10, 12 and 16M gained marginal strength over their 7-day strength by just 4, 18 and 2%, respectively. After 28 days, the 14 M mortar sample maintained the lead up to 120 days followed by 12M mortar sample, while 16M mortar sample had the least strength. But, comparing the 28-day strength of 14M mortar sample with that of normal cement mortar, 14M mortar had strength of about 25% of that of the normal cement mortar. These results are in agreement with Smith's (1999) findings which showed

Nevertheless, the rate of strength development in AACSAM was low compared to normal mortar. It was observed that normal mortar had reached about 80% of its 120 day-strength at 28 days

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compared to what 14 M mortar reached at the same period, which was about 37%. The implication of this is that AACSAM will continue to gain more strength faster than normal mortar as shown by their strength curves (Figure 5). It is known that pozzolanic reaction is a slow process compared to hydration in OPC (Taylor, 2009). These results are comparable to what Ammar et al. (2013) reported in their study with different materials.

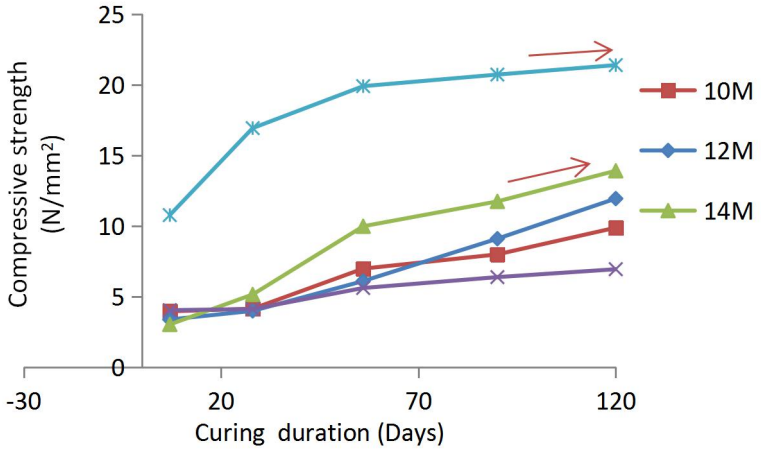

Figure 5: Compressive strength of AACSAM and control

Despite the relative low strength obtained for alkali-activated coconut shell ash mortar (AACSAM), the 28-strength of 14M mortar was still higher than the stipulated minimum strength of 3N/mm^2 for load bearing sandcrete blocks as prescribed by the Nigerian Industrial Standard, (2004). Thus, the AACSAM could be considered for use in non-structural element. In future study, use of other alkalis or combination of other alkali with NaOH would be investigated to improve the strength of AACSAM.

3.2.2 Flexural strength

Figure 6 shows the flexural strength values of both the AACSAM and the normal cement mortar (control). The strength pattern was similar to that of compressive strength. The strength increased with age for all the samples but at different rate. While 16M mortar had highest flexural strength (1.76 N/mm^2) at age 7 days, 12M mortar had least strength (1.2 N/mm^2). Strength of 10 and 14M mortar were 91 and 86% of the strength of the 16M mortar, respectively. As the age increased beyond 7 days, the strength pattern changed with 14M mortar sample gaining strength steadily from 2.37 N/mm^2 at 28 days up to 5.88 N/mm2 at 120 days. Other mortar samples followed the same trend but with relative lower strength. Surprisingly, 16M mortar sample that had initial high strength turned to give lowest after 28 days. The likely reason for this observation is that the concentration of the alkali speed up the initial reaction for the formation of geopolymer product that is responsible for increase in strength (Pacheco-Torgal, et. al., 2008). Nevertheless, there was possibility of concentrated NaOH to be absorb moisture (hygroscopic) from the atmosphere, which may lead to weaker zone in the matrix or increase the moisture content of the matrix, leading to lower strength. These results agree with what Hashim et al. (2015) reported about alkali activated slag.

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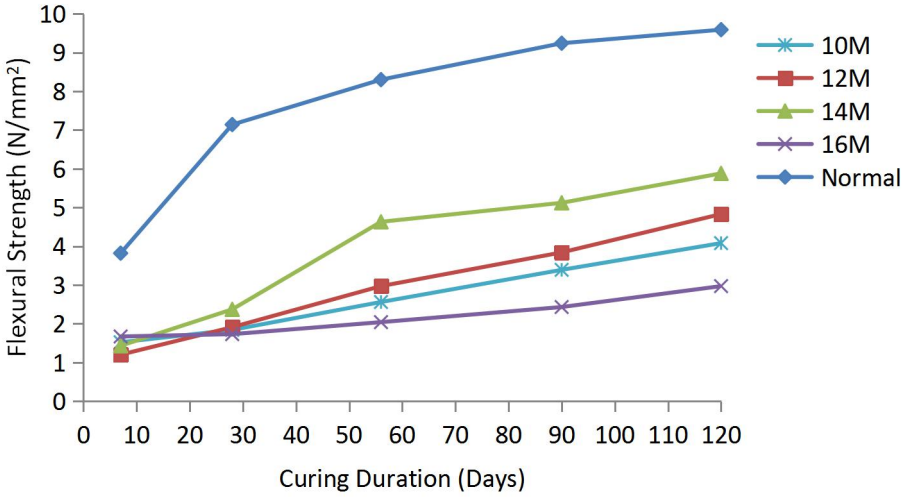

Figure 6: Flexural strength of AACSAM and control

4.3.3 Microstructure of alkali activated coconut shell ash mortar

The SEM images of CSA alkali activated mortar at ages 28 with different concentrations of NaOH are illustrated in Figure 7. At 28 days of hydration, when NaOH concentration was 10M, the microstructure was characterized by anhydrous CSA grains as most of the particles had not reacted with the NaOH (Figure 7a). Subsequently, the matrix was more porous and thus with little strength development. As the concentration of NaOH increased, some hydrated compounds started forming, which were more pronounced at 14M concentration than found in 12M that had some traces of incomplete hydration (Figures 7b and 7c). Furthermore, at higher concentration of 16M, apart from the fact that some CSA grains were hydrated, there were traces of anhydrous CSA with some dark portions and fragile zones (Figure 7d), which is suspected to be linked to hygroscopicity of high concentration of NaOH.

Figure 8 on the other hand shows microstructure of activated mortar samples cured for 120 days. It is seen that more hydrated compounds were formed even at 10M concentration (Figure 8a) due to longer period of hydration (120 days) as compared to 28 days (Figure 7a), only that hydrated compound formed was not completed. The increase in hydrated products caused the sample to be more compacted with relative low pores, leading to higher strength. The most striking observation from these microstructures is that of 14M mortar samples that that no traces of CSA grains. It seemed that all the CSA had been hydrated forming well-compacted matrix, which suspected to be responsible for the highest strength observed (Figure 8c). According to Türker et al., 2016, a complete alkali activation formed additional hydrated compound characterized by a glassy shaped similar to calcium hydrate silicate found in hydrated Portland cement; thus improve the strength properties.

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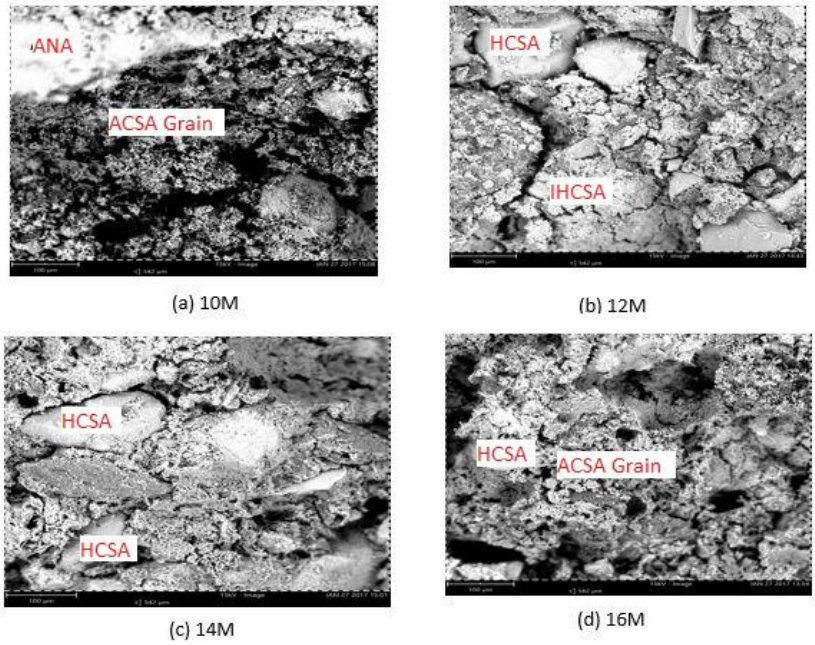

Figure 7: SEM images of AACSAM at 28 days of hydration with different NaOH concentrations. ANA: Anhydrous NaOH, ACSA: Anhydrous CSA, HCSA: Hydrated CSA; IHCSA: Incomplete Hydrated CSA

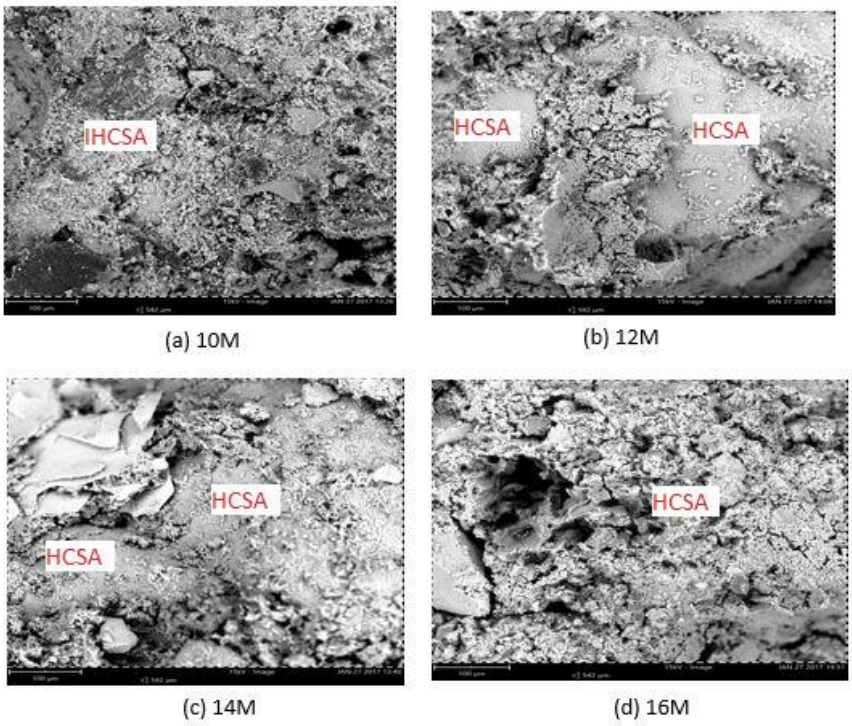

Figure 4.10: SEM images of AACSAM at 120 days of hydration with different NaOH concentrations. HCSA: Hydrated CSA; IHCSA: Incomplete Hydrated CSA.

The hydrated products formed because of alkali activation of CSA could be amorphous alkaline aluminosilicate with a 3D structure similar to a zeolitic gel type. The hydrated products caused densification of the hydrated matrix due to increase in volume and filling effect of the pores within the matrix. This action has been attributed to increase in the strength properties of cementitious matrix (Yuya et. al, 2015). Thus, it could be safely concluded that more hydrated products formed, as a result of reaction between CSA grains and 14M NaOH is responsible for the increase in compressive and flexural strength obtained (Figures 5 and 6).

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4. Conclusion

In this study, characterization of coconut shell ash was performed and mortar samples were prepared with different concentrations of NaOH. Strength of the mortar samples was equally determined. From the study the following conclusions were drawn:

Coconut shell ash produced at 800 ℃ for 60 minutes possessed highest content of combined silica, alumina and ferric for alkali activation.

Alkali activator concentration has influence on the strength development of AACSAM, as well as its microstructure.

Sodium hydroxide of 14M concentration produced highest compressive strength, higher than what was recommended for non-structural use.

There were indications that CSA could be further explored using different optimization approaches to produce higher strength for structural application, like normal cement mortar.

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