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SUITABILITY STUDY OF SOYBEANS HUSK ASH AS A MIXING MATERIAL TO OPC: EFFECT OF CALCINATION TIME-PRELIMINARY INVESTIGATION

[1]Taku K. J.[*], [2]Amartey Y. D.

[1]Department of Civil Engineering, University of Agriculture, Makurdi, Nigeria
[2] Department of Civil Engineering, Ahmadu Bello University, Zaria, Nigeria

*Corresponding author's e-mail: kumataku@yahoo.com
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ABSTRACT

Soybeans husk was collected from a dump site, dried de-carbonated, separated into six samples and calcined at a temperature of 600 ^{0}C, for 1, 2, 3, 4, 5 and 6 hours respectively. Samples were taken for X- ray Florescence (XRF) analysis, Standard Electron Microscopy (SEM) imaging, setting time, standard water of consistency determination and specific gravity test. The result of XRF analysis revealed that the SHA contain less than 70 % $SiO_2+Al_2O_3+Fe_2O_3$ stipulated by ASTM C618 for pozzolanas, but has very high CaO content. The SiO_2 $+Al_2O_3+Fe_2O_3$ and CaO content though varies slightly with the calcination time of soybean husk. The specific gravity of SHA varies with calcination time of SH and ranges between 2.3 and 2.7 and is less than that of OPC irrespective of the calcination time. Setting times of OPC/SHA pastes at 10 % replacement of OPC with SHA increases with calcination time and is greater than that for OPC paste. The SEM analysis shows that the crystal structure of the SHA changes with calcination temperature. The compressive strength of mortar using OPC/SHA as a binder was determined after 7, 14 and 28 days curing and was found to be less than that for OPC mortars at all ages. However, the percentage reduction in strength decreased with curing age, with SHA calcined at 4 hours having the greater strength (i.e. 87.4 % of OPC strength at 28 days). The percentage gain in strength was more pronounced from 7 to 14 days. In all, SH calcined at 600 ^{0}C for 4 hours optimizes the pozzolanic potential of SHA**.**

Keywords: Soybeans husk ash, Calcination time, Oxide composition, Setting time, SEM imaging, Compressive strength

INTRODUCTION

Sustainable development of the construction industry requires the use of cheap and easily available materials for use in construction. The construction industry as we have it today is not sustainable due to the high cost of construction materials especially steel and cement [1]. The desire by researchers to ensure sustainability has led to a lot of research aimed at finding alternative (cheaply available) cementitious materials that can replace cement wholly or partially, since the cost of cement contributes greatly to the rising cost of construction. These materials are known as pozzolana -which are siliceous or siliceous and aluminous nature- are in themselves not cementitious but will in the presence of moisture react with lime to give products that are similar to those obtained by the hydration of Cement. Various researches have been carried out on agricultural waste products like fly ash, Rice husk ash, groundnut husk ash, Coconut shell ash, Locust Beans Ash etc., which are siliceous in nature and are found to be suitable as partial replacements of OPC in Mortar, concrete and sandcrete blocks production [2–8]. These materials can only be used when they are burnt and are in ash form, and the ash is amorphous rather than crystalline in nature [9]. This in turn is a function

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of the calcination parameters like calcination temperature and time [6, 9-10]. For a material to be pozzolanic, ASTM C618 stipulates that it must possess a minimum of 70% and 50% of SiO_2 +Al_2O_3- +Fe_2O_3 for a class C and F pozzolana respectively.

Nigeria is among the top producers of soybeans in the world and thus the husk is readily available. Burning of the husk produces SHA. This research investigates the suitability or otherwise of SHA as a pozzolana for partial replacement of OPC as well as the effect of calcination time of SHA on the properties of cement mortar.

MATERIALS AND METHODS

The materials used for the research included the following:

1) **Soybeans Husk:** This was collected at Tiortyu in Tarka LGA Benue state Nigeria and the calcination was carried out at a programmable electric furnace at the laboratory of the department of Physics, University of Agriculture Makurdi Benue state Nigeria. X-ray Florescence (XRF) analysis on the calcined SHA at the different calcination times was carried out at the National Geological Research Institute Kaduna Nigeria, while Standard Electron Microscopy (SEM) analysis was carried out at the laboratory of the Department of Chemical Engineering, Ahmadu Bello University Zaria.
2) **Cement:** The cement used for this research work was a 42.5 grade OPC manufacture by Dangote Cement Industry Plc., Gboko plant and was obtain from a local retailer in Makurdi Benue state.
3) **Aggregate:** The fine aggregate used was river sand obtained at the bank of the river Benue in Makurdi, Nigeria. Tests on aggregates were carried out at the concrete laboratory of the Department of Civil Engineering, University of Agriculture Makurdi.

The experimental program for the research work is divided into three parts, in the first part soybeans husk was collected and dried for 48 hours after which it was de-carbonated in open air for 30 minutes and allowed to cool. The SHA was then divided into six samples. Each sample placed in a crucible and calcined in a programmable electric furnace at 600 ^{0}C for 1, 2, 3, 4, 5 and 6 hours, respectively.

In the second part, samples of SHA (about 20 g each) was taken for each calcination time to determine the oxide composition (using XRF analysis), and the crystal structure investigated using SEM microscopy; while the third part of the experimental program involves testing for compressive strength of mortar, using OPC-SHA as a binder at 10 % replacement of OPC with SHA for each calcination time. In all, 54 numbers of 50 mm x50 mm x 50 mm mortar cubes were casted. Three cubes were tested for each point at 7, 14 and 28 days for mortar strength making 18 cubes per point.

RESULTS AND DISCUSSION

1. Preliminary Test on OPC and Aggregate

The preliminary test carried out on OPC included setting times, specific gravity, standard water of consistency, soundness, fineness and oxide composition. The tests were carried out in accordance with the provision of ASTM C4O3, ASTM C127, ASTM C191, ASTM C151, and ASTM C184, respectively. The result is presented in Table 1. From the Table, we can conclude that all properties of the cement used meet up with the various standards specification for class II Portland cement.

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Table 1. Characterization of the used cement

Test	Result	Code Specification	Code
Fineness	0.043	0.01-0.06	BS 12: 1990
Initial Setting Time	83 minutes	≥45 minutes	BS 12: 1990
Final Setting Time	254 minutes	≤ 10hours	BS 12: 1990
Consistency	38%	26-30%	BS 12: 1990
Specific Gravity	2.85	2.3-2.9	BS 12: 1990
Soundness	3.5mm	≤10mm	BS 12: 1990

The test carried out on the fine aggregates included moisture content, specific gravity and particle size determination. These tests were carried out in accordance with the standard set out in ASTM C70, ASTM C128, ASTM C136 and ASTM C117, respectively and the results are represented in table 2 and Figure 1.

Table 2. Properties of the used fine aggregates (Sand)

Test	Result	Code Specification	Code
Moisture content	5.73(%)	0 – 10%	ACI E1 – 07
Specific Gravity	2.65	2.3-2.9	BS 12: 1990
Fineness Modulus	2.95	2.3- 3.1 (2.0-3.1)	ASTM C33 (ACI)

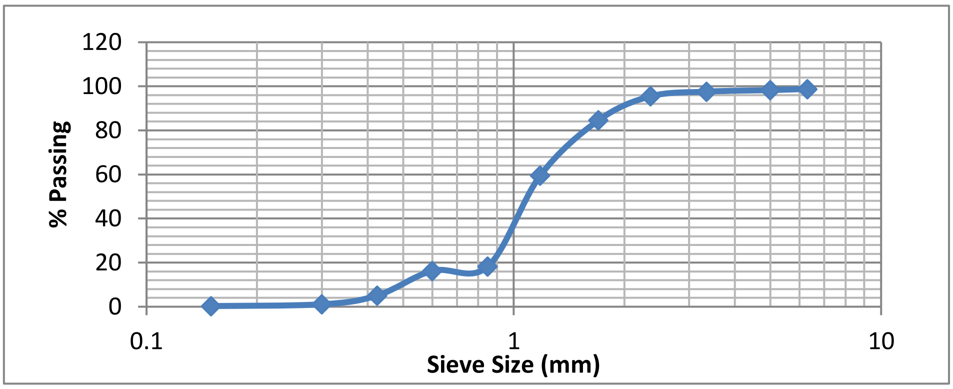

Figure 1. Sieve analysis of the used fine aggregates

The results indicated that all properties of the tested fine aggregates fall within limits specified by the respective codes.

2. Tests on Soybeans Husk Ash

The tests carried out on Soybeans husk ash included specific gravity, standard water of consistency and setting times for cement-SHA mix at 10 % replacement of cement with SHA, and Oxide Composition for SHA.

Specific Gravity

The result of Specific gravity (SG) test is presented in Figure 2.

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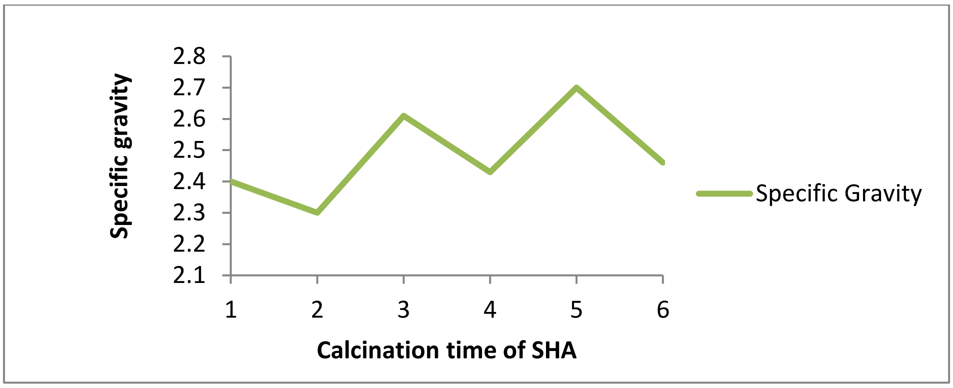

Figure 2. Result of Specific Gravity test for SHA at different calcination times

The result showed that SHA calcined at 600 ^{0}C for a time between 1 and 6 hours have a specific gravity of more than 2.20, with the highest value at 5 hours of calcination. Even though the calcination time has a profound effect on the SG of SHA, there is however no specific pattern of change in SG with calcination time as can be seen in Fig. 2. This also shows that the SG of SHA ash is lower than that of OPC irrespective of the calcinating conditions. This result agrees with Johnson 2013 [20].

Setting times

The setting times of SHA/OPC mix for SHA calcined at 600 ^{0}C for 1, 2, 3, 4, 5 and 6 hours is presented in Figure 3.

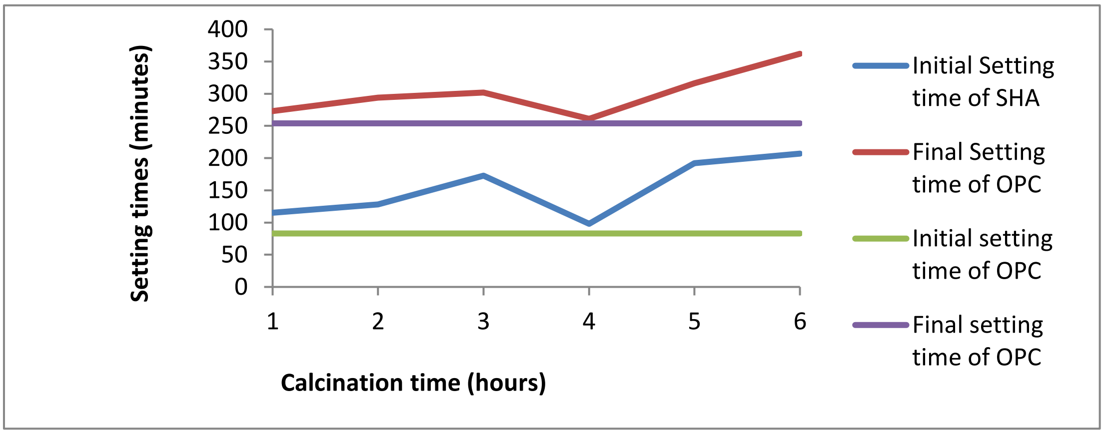

Figure 3. Setting times of SHA and OPC

It can be seen that for all cases the setting times of OPC/SHA mixes were higher than that of OPC, for both the initial and final setting times. This may be due to the reduced water adsorption ability of the binder paste as a result of the addition of the SHA. In all cases, the setting times fell within the range recommended in ASTM C191 (12). The setting times increases with calcination time except for 4 hours calcination. The lowest and highest setting times were for 4 and 6 hours calcinations, respectively for both initial and final set. Thus, it can be concluded that SHA/OPC mix for SHA calcined for 4 hours have setting properties similar to that of OPC paste.

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Standard water of Consistency

The standard water of consistency of SHA/OPC mix for SHA calcined at 600 ^{0}C for 1, 2, 3, 4, 5 and 6 hours is presented in Figure 4. The standard water of consistency test gives an idea of the water content requirement for other tests like setting time. The standard water of consistency increased with calcination time, except for 4 hours calcination where it decreases as it is also applicable in setting times as can be seen in Figure 3. This may not be unconnected with the unique crystal structure of SHA calcined for 4 hours as shown in Figure 8.

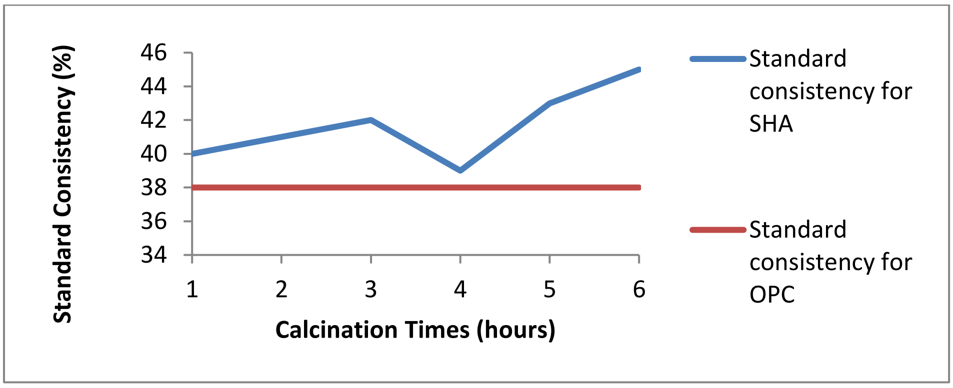

Figure 4. Standard water of consistency of SHA and OPC

Oxide composition

Result of oxide composition of SHA calcined at 600 ^{0}C for 1, 2, 3, 4, 5 and 6 hours is in Table 3.

Table 3. Oxide composition of SHA using XRF analysis

% Oxides	XRF 1HR	XRF 2HRS	XRF 3HRS	XRF 4HRS	XRF 5HRS
Al_2O_3	2.5	2.8	2.4	3.6	2.9
SiO_2	17.3	11.9	9.07	17.3	14.6
*K_2O	22.8	34.66	32.3	13.4	21.2
P_2O_5	5.84	9.87	6.03	8.2	11.3
TiO_2	0.74	0.61	0.51	0.83	0.62
CaO	45.8	39.8	47.5	53.88	47.83
V_2O_5	0.044	0.02	nd	0.041	0.03
Cr_2O_3	nd	nd	nd	nd	nd
BaO	0.6	0.35	0.58	0.61	0.5
ZnO	0.05	0.047	0.086	0.057	0.051
Fe_2O_3	3.08	1.64	2.72	3.93	2.42
Eu_2O_3	nd	0.03	0.04	nd	0.03
MnO	0.378	0.31	0.38	0.405	0.37
CuO	Nd	Nd	0.038	0.039	0.039
MoO_3	Nd	Nd	Nd	0.2	Nd
SO_3	0.82	0.71	0.68	1.1	0.94
L.O.I.	Nd	Nd	Nd	Nd	Nd

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Nd= Not determined

The results show that the SiO_2+Fe_2O_3+Al_2O_3 content of the SHA at all times of calcination did not meet up with the specifications of ASTM C610 of 50% or 70 % for pozzolanas, the highest is 24.83 % which is for 4 hours calcination. The CaO content however is very high, the highest for 4 hours calcination (53.88) and the lowest for 2 hours calcination (39.8), which are about 85 % and 63 % of the CaO content of OPC, respectively. This shows that SHA can be used in conjunction with other pozzolana in partial replacement of cement.

SEM analysis

The result of SEM analysis on SHA at 5000x magnification is presented in Figure 5 - 9.

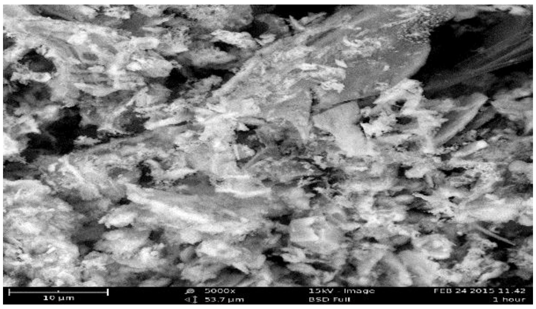

Figure 5. The SEM analysis for SHA (1 hr)

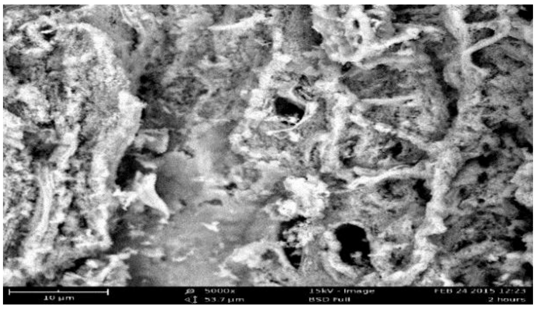

Figure 6. The SEM analysis for SHA (2 hrs.)

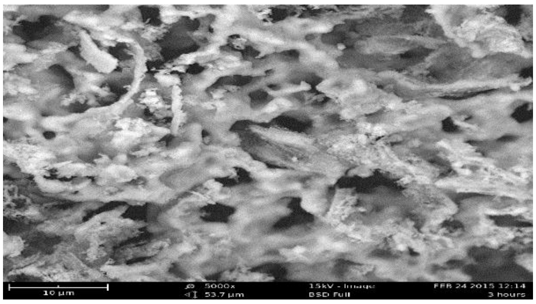

Figure 7. The SEM analysis for SHA (3 hrs)

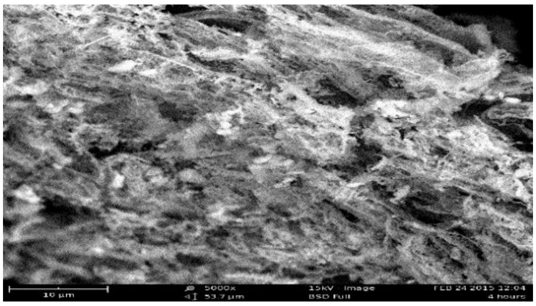

Figure 8. The SEM analysis for SHA (4 hrs)

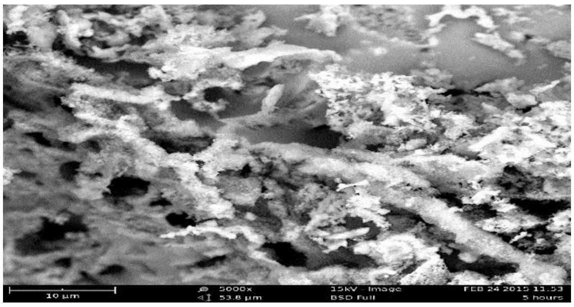

Figure 9. The SEM analysis for SHA (5 hrs. calcination)

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The results indicated that the time of calcination has a profound effect on the crystal structure of SHA.

3. The compressive strength of Mortar Specimens

Strength tests were carried out on mortar specimens to determine the effect of the addition of SHA calcined at 600 ^{0}C for 1, 2, 3, 4, 5 and 6 hours on the compressive strength of the concrete and mortar specimens. The result of compressive strength of the class M mortar at 7, 14 and 28 days curing are presented in Figure 10.

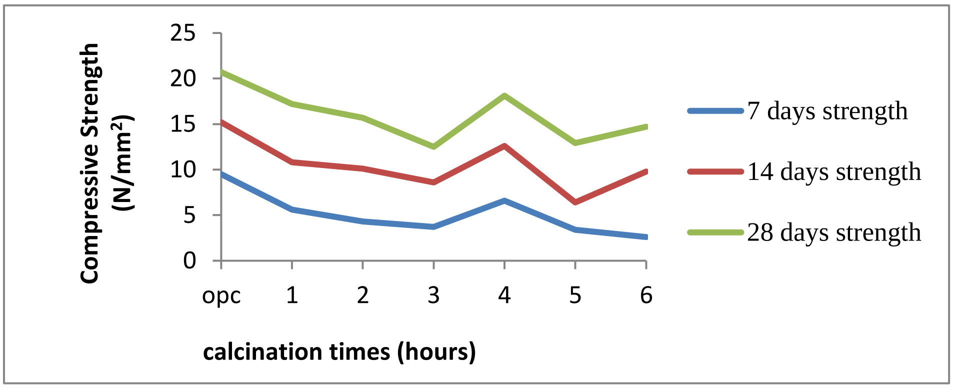

Figure 10. Mortar Compressive Strength

The result shows that the compressive strength of cement mortar at all ages is higher than that incorporating SHA, with that for OPC/SHA mortar for SHA calcined for 4 hours calcination having the greatest strength at all ages compared to other calcination times. This shows that calcination time has a profound effect on the properties of SHA. Table 4 gives the percentage decrease in strength for mortar using OPC/SHA as a binder as compared to that using OPC alone; and the percentage increase in strength from 7 - 14 days and from 14 to 28 days for OPC and OPC/SHA mortar. The result indicates that the strength of SHA mortar decreases as compared to the control, but the rate of decrease reduces with age. Also, SHA calcined for 4 hours showing the least decrease of 30.5, 17.1 and 12.6 % for 7, 14, and 28days strength, respectively.

It can also be seen that the mortar gains more strength between 7 and 14 days as compared to from 14 to 28 days curing. According to research by [21], the strength development and the ultimate strength of concrete and mortars incorporating pozzolana depend on pozzolan/lime ratio, water/binder ratio and the particle packing of the pozzolan material. Also, the time required to recover the initial strength loss caused by the replacement of pozzolan depends on the nature of pozzolans, including the content of the active phases and specific surface, the replacement level, the composition and the strength class of Portland cement. Thus, since the amount of silica is limited in SHA, it is reasonable to conclude that this behavior is due to the fact that the silica content of SHA available for the pozzolanic reaction is limited as compared to the CaO content from the ash and OPC, limiting the amount of SiO_2 available to react with the $Ca(OH)_2$.

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Table 4. Compressive Strength Characteristics of OPC/ SHA mortar

Material	Compressive Strength (N/mm^2)			%decrease in Strength compared to OPC			% increase in strength with age	
	7 days	14 days	28 days	7 days	14 days	28 days	7–14 days	14-28 days
OPC	9.5	15.2	20.7	-	-	-	37.5	26.6
SHA/OPC (1 hr)	5.6	10.8	17.2	41.1	28.9	16.9	48.1	37.2
SHA/OPC (2 hr)	4.3	10.1	15.7	49.5	33.6	24.2	57.4	35.7
SHA/OPC (3 hr)	3.7	8.6	12.5	61.1	43.4	39.6	57.0	31.2
SHA/OPC (4 hr)	6.6	12.6	18.1	30.5	17.1	12.6	47.6	30.4
SHA/OPC (5 hr)	3.4	6.4	12.9	58.9	57.9	37.7	46.9	50.4
SHA/OPC (6 hr)	2.6	9.8	14.7	72.6	35.5	29.0	73.5	50.0

CONCLUSION

The following conclusions can be drawn from this research

i. The time of calcination of Soybeans husk has profound effect on the SG of SHA calcined at 600 ^{0}C for a time period of 1 to 6 hours, and the value ranges from 2.3 to 2.7 and is lower than that of OPC irrespective of the calcination time.

ii. The initial and final setting times of SHA are affected by the calcination times of Soybeans husk as they increase with calcination time. The addition of SHA to OPC increases the setting times of the binder.

iii. The water demand of OPC/SHA paste increases with the time of calcination of Soybeans husk and is higher than that of OPC paste.

iv. The oxide composition of SHA varies with calcination time of soybeans husk but the SiO_{2-} $+Fe_2O_3+Al_2O_3$ content of the SHA at all the times of Calcination did not meet up with the specifications of ASTM C610 of 50% or 70% for pozzolanas. Since the CaO content is very high, it can be used in conjunction with other pozzolana to improve the properties of concrete and mortar.

v. The crystal structure of SHA is affected by the calcination time, as it changes with calcination time as revealed by the SEM analysis.

vi. The strength of mortar using SHA/OPC as binder is less than that of OPC. However, using SHA calcined for 4 hours gives the highest strength for the OPC/SHA mix.

vii. Calcination at 600^{0}C for 4 hours optimizes the pozzolanic potential of SHA in partial replacement of OPC.

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