EARLY-AGE PROPERTIES OF SORGHUM HUSK ASH AND CALCIUM CARBIDE WASTE BINDER IN MORTAR

C. I. Egwuda, I. O. Hassan, B. J. Olawuyi, and M. O. Enejiyon
Department of Building, School of Environmental Technology, Federal University of Technology, Minna, Nigeria

Portland cement (PC) is a binder that is most commonly used as construction material in the production of mortar in masonry and concrete. The manufacturing process of PC during clicker production is however noted to contribute to CO2 emission which makes it a non-eco-friendly material. Notwithstanding, reports on total replacement of PC are scarce in literature. Sorghum husk ash (SHA), which is an incinerated ash from agricultural by-product consisting majorly of amorphous silica (SiO2), when combined with calcium carbide waste (CCW) an industrial by-product generated from an acetylene gas production process with major component of lime (CaO) in the presence of water forms compounds possessing cementitious properties. This paper reports on the early-age properties of SHA (as SiO2 source) and CCW (as CaO source) binder in mortar. Paste from different binder combinations of SHA/CCW were studied for setting time while the mortar samples were used to study the rate of hydration and strength development. The study revealed the SHA sample to be of high SiO2 (84%) and CCW is majorly CaO (66% content). The results obtained showed improvement in the performance of binders with superplasticizer formulated from 70/30, 60/40 and 50/50 SHA/CCW respectively, having 28days compressive strength of 7.6 N/mm2 [MPa], 7.0 N/mm2[MPa] and 5.7N/mm2[MPa] representing 36%, 34% and 28% of cement type I (CEM I) strength. The study showed that addition of superplasticizer reduced the water demand and improved the rate of hydration. The binder combinations of 70/30, 60/40 and 50/50 SHA/CCW with water-reducing admixture can be adopted for use in masonry works as it conforms to type N of ASTM C270 mortar.

Keywords: Binder; Calcium carbide waste (CCW); Mortar; Sorghum husk ash (SHA); Superplasticizer.

INTRODUCTION

The process of concrete and mortar production uses Portland Cement (PC) as binder for strength development and other desired properties and this has been noted to be the prominent global practice (Mehta and Monteiro, 2014). The manufacturing process of PC is however noted to contribute around 5% of global CO_2 emission resulting from clinker production and the fossil fuel used for pyro-processing (Rubenstein, 2012). Clinker production involves heating calcium carbonate ($CaCO_3$) in the kiln at temperatures of above 900ºC resulting in lime (CaO) and CO_2 as shown in equation 1.

$$CaCO_3 \xrightarrow{heat\ (>900\ oC)} CaO + CO_2 \quad (1)$$

The quick lime CaO is further made to react with materials containing silica (SiO_2), alumina (Al_2O_3) and iron (Fe_2O_3) at higher temperatures of about 1450ºC. This is then removed from the kiln, allowed to cool, ground to fine powder and mixed with about 5% gypsum to control the setting process (Neville, 2012; Mehta and Monteiro, 2014). The major components of PC is stated as CaO, SiO_2, Al_2O_3 and Fe_2O_3 with strength determinant being the CaO in combination with SiO_2 which forms hydrated lime – $Ca(OH)_2$ in the presence of water resulting in formation of $CaO\text{-}SiO_2\text{-}H_2O$ – Calcium Silicate Hydrate (C-S-H) which is the final product for strength development as cement hydration progresses after water contact.

egwudaclement@gmail.com
i.hassan@futminna.edu.ng
babatunde@futminna.edu.ng
enejiyonmuhammed@gmail.com

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Research and development of alternative binders to Portland Cement (PC) is continuously in the forefront in recent years due to the increased awareness on climate change attributable to global warming. Stratospheric ozone depletion and climate change resulting from emission of greenhouse gases (GHG) due to human and industrial activities with chlorofluorocarbons (CFC) and non-CFC gases such as carbon (IV) oxide (CO_2), adjudged the primary gas emitted (Waterloo News, May, 2013; US National Climate Assessment (NCA), 2014; US Environmental Protection Agency, 2016). This coupled with constant excavation and depletion of lime stone ($CaCO_3$) from their natural sources has resulted in research for alternative materials with focus on re-use and recycling of the abundant agricultural and industrial waste materials.

Previous studies on the search for alternative binders centred on utilisation of natural Pozzolan such as volcanic ash (Hossain 2003 & 2005; Hassan, 2006; Olawuyi, 2011) or ashes from agricultural wastes (agro-wastes) such as rice husk ash [RHA] (Okpala, 1987; Chaowat, 2001; Abalaka & Okoli, 2013), corn-cob ash [CCA] (Raheem, 2010), sawdust ash [SDA] (Elinwa & Mahmood, 2002), millet husk ash [MHA] (Jimoh *et al*, 2013;) Palm Oil Fuel Ash [POFA] (Hassan et al., 2013) and palm kernel nut ash [PKNA] (Joshua *et al,* 2015) amongst others as partial PC replacement in mortar or concrete.

Attempt on total cement replacement in concrete brought about studies into geo-polymer concrete which involve alkali activation of Pozzolanic materials with the use of chemical based hydroxide [NaOH] at elevated temperatures or ambient temperature (Ul. Haq*et. al,* 2014; Turner and Collins, 2013). Some studies on total cement replacement with Pozzolan in combination with alternative CaO source (calcium carbide waste [CCW]) include the works of Rattanashotinunt *et. al*., (2013) – baggase ash combined with CCW; Makaratat *et. al*., (2010) – combination of fly ash (FA) and CCW. Joshua et al (2016), combination of pulverized calcined clay (PCC) with CCW, both sourced within Nigeria and reported a hydration reaction with a 28 day strength of 11 MPa without any treatment to the CCW.

Incinerated ashes from agro-wastes at controlled temperature have been found to be pozzolanic with major components been amorphous silica which combines with lime in the presence of water to give cementitious properties. Pozzolan by definition is siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties (ACI Terminology of Concrete, 2013 in ACI Manual of Concrete, 2016; Neville, 2012).

The concept of pozzolanic reaction according to Mehta and Monteiro (2014) is based on the fact that Portland cement react using Tricalcium Silicate (C_3S) with water to give Calcium-Silicate-Hydrate (C-S-H) and Calcium Hydroxide (CH)

$$C_3S + H \rightarrow C - S - H + CH \quad (2)$$

and the Portland-Pozzolan cement reaction follows as

$$Pozzolan + CH + H \rightarrow C - S - H \quad (3)$$

Where C = CaO, S = SiO_2 and H = $(OH)^-$

The reaction in Equation 2 is known to be fast and lime producing while the reaction in Equation 3 is rather slow or latent depending on the properties of the pozzolanic material. The pozzolanic reaction in (Equation 3) is basically lime-consuming and does not necessarily require presence of cement but an active source of lime, hence the thought for alternative source of lime to enhance pozzolanic reaction with an agricultural waste ash as Silica source. Sorghum husk ash (SHA) is the focus of the present study as silica source (SiO_2). The CaO source in this study is an industrial waste material (calcium carbide waste (CCW)).

Calcium carbide waste (CCW) is a by-product of acetylene gas generated from calcium carbide used in the production of Polyvinyl Chloride (PVC) and in welding steels especially in the auto industry. CCW in Nigeria is reported to be 70-80% calcium hydroxide ($Ca(OH)_2$) with the impurities in it listed as copper, lead, iron, manganese, nickel and zinc (Chukwudebelu *et al*., 2013). SHA is an ash gotten from open-air burning of the husk of sorghum, which popularly serves as major staple food in the northern part of Nigeria. The drive towards food security and sustainability by the Nigeria Government with cereals grains like sorghum, maize and millet being the central focus and Niger State known as a major contributor to cereals production in Nigeria is an indication that the husk of the crops will ever be in abundant supply. Thus, utilization of these agricultural and industrial waste materials in concrete and mortar production should be seen as a welcome development. Previous attempt reported by Olawuyi *et al*., (2017) on SHA – CCW binder showed slow hydration process, high water demand, slow setting time and strength development. A research into early-age properties of SHA – CCW binder in mortar will surely offer contribution towards improving knowledge in concrete technology and infrastructural development.

EXPERIMENTATIONS

Materials

The materials used for this study are Sorghum husk for production of SHA as sources of SiO_2, CCW (an Industrial waste from automobile oxy-acetylene welding) as CaO source at varied combinations (70/30, 60/40, 50/50, 40/60 and 30/70 of SHA/CCW respectively) which formed the alternative binder; CEM I 42.5N (Dangote 3X) from Obajana factory of Dangote Cement Company served as the binder for the control mortar mix and Master Gleniun ACE 456 as superplasticizer. The fine aggregate used is the simulated reference sand (size range 1.18 mm [Sieve No. 16] to 75 µm [Sieve No. 200]) sieved out from the available natural sand in consonance with BS EN 196-1:2016 reference sand prescription for strength test on cement (binder), while potable water available at the Building Laboratory of the Federal University of Technology, Minna was used for mixing.

Sorghum husk were collected from Bosso Local Government Area, in Niger State, Nigeria. The husks were burnt in open air with a locally fabricated incinerator presented earlier in Abalaka & Okoli (2013). This was ground to finer particles in a local mill at Gidan-Mongoro Village of Minna and sieved with a 75 µm sieve and the particles passing were used as the SHA for the experiment. The CCW on the other hand was obtained from a local automobile Welder's (i.e. "Panel-beater" using oxy-acetylene gas) workshop in Minna as sludge. It was sun-dried and sieved with 75 µm sieve and the particles passing were used as the CCW sample in this study.

Methods

The study involved the evaluation of the physical and chemical properties of the constituent materials for proper characterisation of the materials used. Also determined were the fresh properties of the binder pastes and mortar before an examination of the strength properties and degree of hydration of the hardened mortar samples.

Mortar samples of 1:3 (c/s) and 0.5 water/cement (w/c) ratio specified by BS EN 196-1:2016 were used as control and for the alternative binders of varying proportion combinations of SHA/CCW respectively as stated in Section 3.1 were prepared and tested for strength and degree of hydration at different curing ages (3, 7, 14 and 28 days).

Physical and chemical Properties

Particle size distribution of the available natural sand was conducted using the dry-sieve approach in accordance with BS EN 933-1:2012 specifications for proper classification of the available natural sand. The reference sand required for mortar production in strength determination test specified in the standard (BS EN 196-1:2016) was then extracted using an arrangement of sieve size 1.18 mm and 75 µm. The particles passing the 1.18 mm sieve but retained on the 75 µm sieve was used for the mortar mixture for the strength test. The 1.18 mm sieve was adopted as the upper limit value for the simulated reference sand instead of the 1.6 mm sieve specified by BS EN 196-1:2016 because of non-availability of the 1.6 mm sieve in the laboratory. Figure 1 of Section 3.1 present the particle size distribution of both the natural sand and the simulated reference sand.

The physical properties determined for all the materials used for this experiment is the specific gravity test carried out in accordance with BS EN 1097: 1998 while fineness test was also conducted on the CEM I 42.5N and the varied combination of SHA/CCW via dry-sieving method as prescribed by BS EN 196-6:2010 using a 75 µm sieve available in the Laboratory.

X-Ray Fluorescence (XRF) analysis for determination of the oxide composition was conducted on the cementitious materials (CEM I 42.5, SHA and CCW) at Ewekoro Works Department of Lafarge Cement using XRF Analyser connected to a computer system for data acquisition.

Setting time and soundness of cement and the SHA/CCW binders

The initial and final setting times and the Le-Chatelier soundness tests for the binders (CEM I 42.5N and the various proportion combinations of SHA/CCW) were determined using neat pastes of standard consistency in accordance to BS EN 196-3:2016. This involved determining the water content of the paste which produced the desired standard consistency (Neville, 2012). Vicat apparatus Model No EL 38 - 2010 by ELE was used for measurement of the consistency and both the initial and final setting times following the procedures as outlined in the standard (BS EN 196-3:2016). The soundness test was also carried out on the respective binders using a Le Chatelier apparatus Model No EL 38 – 3400 by ELE.

Determination of strength and degree of hydration of the binders

Determination of strength and degree of hydration of the binders was conducted using 50 mm [2 in.] mortar cubes as mentioned in Section 3.2. Production of the mortar samples involved weighing out the appropriate constituent materials and ensuring that the SHA was thoroughly mixed with CCW in an head-pan before it is poured on the measured quantity of the simulated reference sand already spread into the steel mixing platform. The sand and binder was then mixed thoroughly before the

weighed mixing water was added and mixing continued until a uniform mix was achieved before casting into the 50 mm [2 in.] cubes moulds. The control mortar sample on the other hand, has the CEM I 42.5N mixed as described above with the simulated reference sand and requisite quantity of mixing water before casting into the cube moulds. Based on observation from the setting times tests as reported in Section 3.2, the samples were left covered with jute bags and cured by water sprinkling until 72 hours before demoulding and water curing by immersion made to continue until testing age.

The procedure for the strength test and degree of hydration determination thereby adopt similar approach reported in Hasholt et al. (2010) as cited in Olawuyi (2016). The procedure is as highlighted below:

i. The mortar cubes were cast and crushed at the different curing ages (immediately after demoulding – 3, 7, 14, and 28 days) in the Digital Universal Testing Machine (DUTM – 20) to assess the strength development.
ii. The remains of the sample in (i) above was then milled properly using the 150 mm [6 in.] x 150 mm Θ [6 in.] cylindrical moulds available in the lab and 25 mm [1 in.] Θ bar as mortar and pestle. The milled sample was then vacuum-dried for 1 hour to stop further hydration.
iii. A known weight of the vacuum-dried sample, about 25 g from the particle passing 75 µm standard sieve [Sieve #200] was measured and oven dried for 24 hours at 105°C [221°F] and weighed again (to determine the evaporable water i.e. the capillary water + gel water)
iv. This sample was then placed in the furnace [Model No SNOL 8,2 /1100 – 1LZ] set to 900°C [1652°F]. At one hour time after the furnace temperature reads 900°C [1652°F], the furnace was switched off, allowed to cool and the sample weighed (to determination of the amount chemically bound water i.e. the non-evaporable water).

All calculations were then based on ignited weight basis to give the following:

Loss on ignition (LOI) of the binders (CEM I 42.5 N, SHA and CCW) and hydrated mortar pastes calculated by

LOI (%) = 100 x (as received weight – ignited weight)/as received weight (4)

Non-evaporable water content w_n of the hydrated mortar pastes were determined to evaluate the degree of hydration as provided for in literature (Lam *et al*., 2000; Neville, 2012; Olawuyi *et al*., 2017). This is the difference in mass measurement of the crushed paste at 900°C [1652°F] and 105°C [22°F], to calculate the degree of hydration (α) on the basis that 1g of anhydrous cement produces 0.23g ofw_n, hence thew_n is calculated by using the following formula

$$w_n\% = \frac{100\,x\,(dried\ \ weight\ of\ paste - ignited\ weight\ of\ paste)}{(Ignited\ weigth\ of\ paste - loss\ on\ ignition\ of\ cement)} \quad (5)$$

The degree of hydration (α) is then:

$$\alpha = 100\,x\frac{Wn}{0.23} \quad (6)$$

The degree of hydration in the SHA/CCW binders at the various combinations (70/30, 60/40, 50/50, 40/60 and 30/70 of SHA/CCW respectively) were however calculated with consideration for the LOI of the SCM and their proportion made to adjust for their w_n% as appropriate.

RESULTS AND DISCUSSION

Characterisation of the Constituent Materials

Figure 1 presents the particle size distribution (PSD) of the available natural sand and the simulated reference sand used for the experiment. The PSD revealed the simulated reference sand to have a C_u and C_c values of 2.06 and 0.86 respectively and a Fineness Modulus (FM) of 2.56 indicating a fine sand classification of Shetty (2004) and the result is similar to that observed in earlier study (Olawuyi*et al*., 2017).

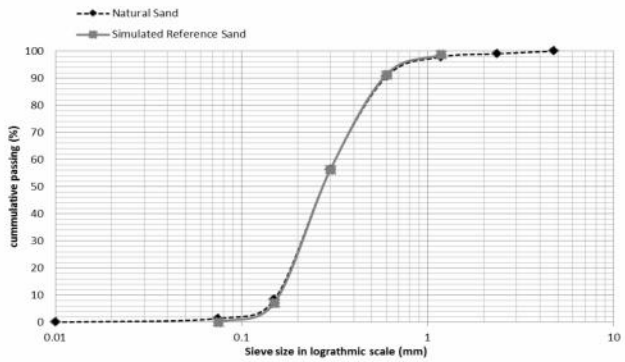

Figure 9: Particle Size Distribution of Fine Aggregate

Table 1 however present PSD of the CEN reference sand for determination of strength of cement as compared to the simulated reference sand used. It was observed that the simulated reference sand was compliant to three of the six size requirements of the CEN reference sand as prescribed in BS EN 196-1:2016.

The simulated reference sand was used for the study despite the shortcomings of not meeting the other three requirements since the study is basically a comparative study on strength development of the alternative binder developed and the CEM I, but not product validation and certification of the cement. The strength of the mortar samples from CEM I used in this study serve purely as a reference to which the strength of the alternative SHA/CCW waste binder was compared.

The specific gravity for the constituent materials is presented in Table 2. The result shows the values is similar to that observedearlier in the study as reported by Olawuyi*et al.,*(2017). The pH value for the constituent materials is also presented in table 3. The result shows that the values are alkaline (Fereshte et al., 2015).

Table 1: Particle Size Distribution of Fine Aggregate

Sieve opening (mm)	CEN Reference Sand (%)	Simulated Reference Sand (%)	Remark
2.00	0	0	√
1.60	7 ± 5	0	
1.00	33 ± 5	3	
0.50	67 ± 5	16	
0.16	87 ± 5	92	√
0.08	99 ± 1	99	√

Table 2: Specific Gravity of Constituent Materials (kg/m^3)

Materials	Specific gravity
CEM 1	3.15
SHA	2.32
CCW	2.29
Sand	2.58
Superplasticizer(Master Glenium)	1.06

Table 3: pH value of Constituent materials

Materials	pH value
CEM I	10.8
SHA	10.29
CCW	11.9

The oxide composition of the various cementitious materials obtained through XRF conducted at Lafarge Cement in Ewekoro is as presented in Table 3. The SHA samples are majorly silica having 83% SiO_2 contents respectively. The Table reveal the SHA as Class N Pozzolan with total SiO_2+Al_2O_3+Fe_2O_3 above 70%, SO_3 below 4% and loss on ignition (LOI) of less than 10%. The

CCW was observed to contain 66% CaO, a similar value to the CaO content (64%) of the CEM I sample. The CCW was however noted to be of lower SiO_2 and Al_2O_3 when compared to the PC sample. The LOI of CCW was noted to be above the specified 10% maximum, an indication that some heat treatment might be required for more effective performance of the material.

Table 4: Result of XRF Analysis for Oxide Composition of Cementitious Materials

SAMPLE	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	TiO_2	P_2O_5	Mn_2O_3	Cr_2O_3	AR	SR	LOI	$SiO_2+Al_2O_3+Fe_2O_3$
SHA	83.0	2.9	2.7	1.3	0.8	0.0	0.2	2.8	0.2	0.5	0.0	0.0	1.1	14.7	5.6	88.6
CCW	3.6	1.6	1.3	65.8	0.2	0.0	0.1	1.0	0.1	0.0	0.0	0.0	1.2	1.2	26.4	6.5
CEM	21.5	5.2	1.2	64.0	2.9	4.5	0.6	0.0	0.1	0.2	0.0	0.0	4.5	3.4	0.0	27.8

Setting Times and Soundness of Binders

Table 4 presents the result of the consistency and soundness test conducted on the binder combinations with superplasticizer and the control (CEM I). The result shows that the water demand of the SHA/CCW binder was about twice that of the CEM I as against the triple value reported in Olawuyi*et al.,* (2017). The SHA/CCW binders reflect a high water demand trend for similar penetration values. The higher the SHA content, the higher the water demand and this was accounted for in the mortar production process for strength test of the binders.

The result of soundness test presented in Table 4 is similar to that observed earlier by Olawuyi*et al*., (2017). The resultrevealed that all the binder combinations conform to the 10 mm maximum expansion specified by BS EN 197-1:2011. Figures 2 and 3 present the plot of the setting times (initial and final) for the SHA/CCW binder combinations respectively.

Table 5: Fresh Properties of Binders with Superplasticizer

Specimen		Consistency		Soundness Expansion (mm)[0.04 in]
		SHA/CCW		
	Superplasticizer (g)	Water Demand (%)	Penetration (mm)[0.04 in]	SHA/CCW
CEM 1	0	36.8	5.0	0.0
70/30	3.71	59.2	6.0	0.0
60/40	3.71	56.8	5.0	0.5
50/50	3.71	53.2	5.0	0.5
40/60	3.71	50.4	5.0	1.0
30/70	3.71	49.6	5.0	1.0

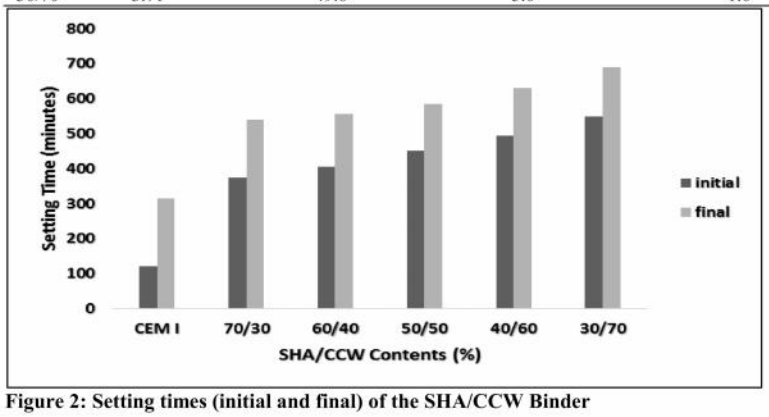

Figure 2: Setting times (initial and final) of the SHA/CCW Binder

The results revealed that the initial setting times of SHA/CCW binder combinations with superplasticizer is three multiple that of the CEM I which has similar trend to that reported by Olawuyi*et al*., (2017). The final setting times of SHA/CCW binders also show similar trend to that of the work of Olawuyi*et al*., (2017) which is also about three multiple of the final setting time for CEM I. This affirms literature postulation that Pozzolans are of latent setting in nature and improvement on the binders can be geared towards accelerating the setting times which is believed will enhance their strength development trends.

Degree of Hydration and Strength of SHA/CCW Binders

The plot of degree of hydration of the binders are presented in Figures 5, while the rate of hydration (RH_{28}) with reference to the 28 day value for the control sample (CEM I) is further shown in Table 5.

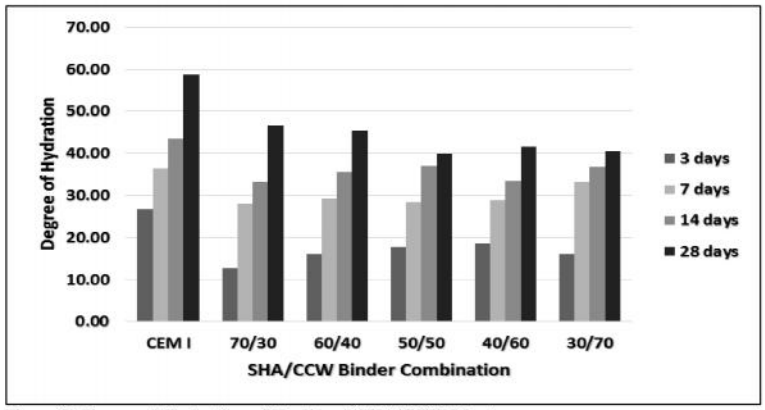

Figure 3: Degree of Hydration of Hardened SHA/CCW Mortar

The result revealed 70/30, 60/40 SHA/CCW combinations as the best of the SHA based binders with 47% and 45% levels of degree of hydration respectively by the 28 day curing age. This amount to RH_{28} values of 0.79 and 0.77 respectively with reference to the 28 days value of CEM I which is slightly higher than that of the work of Olawuyi*et al*., (2017). Hydration was observed to improve as the curing age increased and the binders are expected to show good long term strength development.

Table 6: Degree of Hydration and RH_{28} Factor of the SHA/CCW Binders

Binder Type	Specimen	Degree of Hydration				RH28 Factor			
		3 days	7 days	14 days	28 days	3 days	7 days	14 days	28 days
Control	CEM I	26.83	36.41	43.48	58.75	0.46	0.62	0.74	1.00
SHA/CCW	70/30	12.80	28.08	33.25	46.64	0.22	0.48	0.57	0.79
	60/40	16.02	29.21	35.53	45.34	0.27	0.50	0.60	0.77
	50/50	17.77	28.45	37.05	39.88	0.30	0.48	0.63	0.68
	40/60	18.64	28.93	33.38	41.61	0.32	0.49	0.57	0.71
	30/70	16.00	33.23	36.84	40.50	0.27	0.57	0.63	0.69

The early age (3 days) hydration values for the SHA/CCW binders was observed to be about half that of the PC. The plot of the compressive strength of the binders (Figures 6) was observed to follow similar trend as the inference drawn from the degree of hydration results. SHA/CCW (70/30, 60/40 and 50/50) gave 28day compressive strength values of (7.6, 7 and 5.7) N/mm^2 [MPa] which corresponds to 36%, 34% and 28% of CEM I strength respectively. The low strength can be attributed to the setting time of the mixture and also the additional water used for the binders established from the result of the consistency test.

Despite the low strength development of the SHA/CCW binders as observed in this study, the samples were noted to bind effectively with the fine aggregates after de-moulding at 72 hours (3 days) after casting. The mortar made from the SHA based binders did not dissolve in the immersed water in the curing tank all through the curing ages in this experiment. The SHA/CCW binder when improved upon possibly through the use of water reducers, keeping the water/binder ratio same as that of the control or by using set accelerating mixtures.

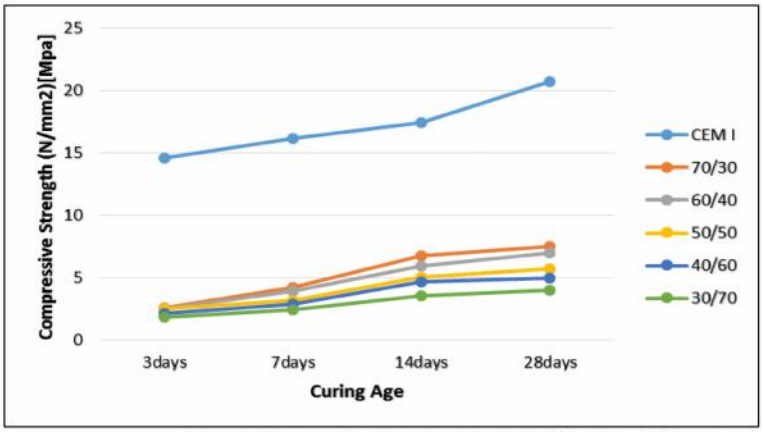

Figure 4: Compressive Strength of SHA/CCW Mortar with Superplasticizer

CONCLUSION AND RECOMMENDATIONS

The results of the study showed that the alternative binders from the SHA (an agricultural waste material) in combination with CCW (an industrial waste material) possess binding properties. The chemical analysis revealed that SHA is a Class N Pozzolan of high SiO_2 content (83%) while CCW is a good CaO source of similar percentage concentration (66%) as the CEM I used for the study. The study further revealed that the water demand was greatly reduced due to the addition of superplasticizer as well as slight improvement on the rate of hydrationof the mortar from the respective binder combinations. Further studies to be targeted at set acceleration and improved early strength development of the binder combinations through the use of set-acceleration with high range water reducer. This may hold a promise towards the desired breakthrough in early-age properties of sorghum husk ash and calcium carbide waste binder in mortar. The following are thereby recommended based on the findings of this study.

i. Further studies on the SHA/CCW should focus on set acceleration and early strength development through the use of set acceleration admixtures with high range water reducer.
ii. Further studies on heat treatment should be conducted due to high loss of ignition (LOI) of CCW.
iii. Investigation into the influence of temperatures slightly above the ambient temperature (40 – 90^{o}C) on the initial and final setting of the SHA/CCW binders should be carried out.
iv. Future studies on product of hydration should be conducted using scanning electron microscopy and X-ray diffraction analysis.
v. SHA/CCW (70/30, 60/40 and 50/50) in 1:3 binder/sand mortar at 0.5 W/B with water-reducing admixture can be adopted for use in masonry works as it conform to type N of ASTM C270 mortar.

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