EFFECT OF SUPERPLASTICIZER ON SETTING TIME AND STRENGTH OF MORTAR MADE USING RICE HUSK ASH (RHA) AND CALCIUM CARBIDE WASTE (CCW) AS BINDER

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Consequent to the challenge of global warming partly associated to high CO_2 emission and energy consumption from cement production processes, research focus has shifted to sourcing alternatives to Portland cement in concrete and mortar production. Incinerated ashes from agro-wastes such as Rice Husk Ash (RHA) at controlled temperature has been found to be pozzolanic with major components been amorphous silica which combines with lime in the presence of water to give cementitious properties. RHA – CCW binder has however been reported to be of high water demand, slow setting rate and strength development. This study incorporates a superplasticizer (Master Glenium ACE 456) – a water reducing agent and set accelerator as an attempt at overcoming the limitations of this new binder. Pastes of 1:3 binder: sand (b:s) and 0.5 water cement (w/c) ratio in accordance to BS EN 197: 2000 made from the different proportion combinations of RHA – CCW were examined for influence of superplasticizer on setting times, degree of hydration and strength development. The study showed that the superplasticizer enhanced early setting and strength development of the mortar. Mortar samples from 60/40 RHA – CCW binder showed superior performance over the other percentage blends with respect to 28 days compressive strength.

Keyword: Calcium Carbide Waste; Mortar; Rice Husk Ash; Setting Time; Strength Development; Superplasticizer

INTRODUCTION

Portland cement (PC) – the major known binder in concrete and mortar production is adjudged non-environmental friendly due to its carbon dioxide (CO_2) and energy consumption with resultant global warming effect from its production processes. The PC production processes contributes about 5% to global anthropogenic CO_2 emission making the cement industry an important sector of CO_2 emission mitigation strategies (Rubenstein, 2012).

Most research efforts have been on the partial replacement of PC while little has been reported on total cement replacement in concrete/ mortar. Incinerated ashes from agro-wastes such as rice husk ash (RHA) 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 (Habeeb & Mahmud, 2010). RHA, known to be of high silica content (Parande, *et al,* 2011) was combined at varied proportions in this study with oven-dried calcium carbide waste (CCW) – CaC_2 , which is known to be mainly calcium oxide (CaO). Yunusa (2015) reported CCW to react with water yielding calcium hydroxide as a by-product as shown in equation 1 below.

$$\mathrm{Ca}C_2 + 2H_2\mathrm{O} \rightarrow C_2H_2 + \mathrm{Ca(OH)_2} \qquad (1)$$

The concept of pozzolanic reaction is based on the fact that PC react using tricalcium silicate (C_3S) with water to give calcium-silicate-hydrate (C-S-H) and calcium hydroxide (CH) (Mehta & Monteiro, 2014)

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

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While 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 SiO_2 source is the focus of the present study.

RHA and CCW have been used in most research as partial replacement of cement in the production of mortar and concrete (Abalaka, 2012; Rao *et al.,* 2014; Gupta & Wayal, 2015 and Yunusa, 2015).

Nigeria's drive for Agriculture with rice production at the centre stage and Niger State being a major rice producing State in Nigeria is a motivation for this study. This study is an attempt to explore the possible utilization of RHA and CCW as an alternative binder in construction, which will not only provide low cost cement and concrete but also help to decrease environmental hazard posed by CO_2 gas emission during cement production. The use of these materials in mortar as a binder may help to achieve energy conservation with economic, ecological and technical benefits. A recent report on RHA – CCW binder (Olawuyi, *et al*., 2017) reports high water demand, slow setting and strength development. This paper reports attempt at enhancing better performance of this new binder by adding a superplasticizer in the mixture.

LITERATURE REVIEW

Pozzolan is defined as a siliceous or siliceous and aluminous material which itself has little or no cementitious value but will in finely divided form and in the presence of moisture, chemically reacts with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties (ASTM C125; Neville, 2012). RHA is classified as Pozzolan due to its high silica content (say about 90%) (Kartini, 2011; Thirougnaname & Sundararajan, 2013)., Roa *et al.,* (2104) reported that RHA which took about 48 hours to burn under uncontrolled combustion process with temperature ranges between 600 – 850°C, grinded with ball mill has about 85%-90% amorphous silica. Rice husk is an agricultural residue obtained from the outer covering of rice grains during milling process. It constitutes 20% of the 500 million tons of paddy produced in the world. When the husk was converted to ash by uncontrolled burning above 500°C, the ignition would not be completed and leads to considerable amount of un-burnt carbon being found in the resulting ash (Givi. *et al.*, 2010).

RHA is reported by Ramezanianpour *et al.,* (2009) to be a carbon neutral green product and known to be a good super-Pozzolan. It produce a high amount of silicon dioxide (SiO_2), with silica content of above 89%, in very small particle size of less than 35 microns and can be used in High Performance Concrete. Oyekan & Kamiyo (2011) reported that an increase in RHA content up to 30% in sandcrete block resulted to decrease in compressive strength and density of the blocks. Sandcrete blocks permeability increases with RHA content and ambient temperature with decrease in thermal conductivity.

RHA replacement up to 25% in concretes resulted to improved compressive strength, flexural strength and increase in bond strength with reduction in density compared with conventional concrete (Nair *et al.*, 2013).

Thirougnaname & Sundararajan (2013) examined 1:4 cement mortar with RHA added as an admixture from 7.50% to 17.50% at 2.5% variation steps and reported that the compressive strength of mortar cube samples increased up to 10% RHA content.

In the work of Srinivas & Kishore (2015), RHA replacement of PC gave early strength value slightly less than the conventional concrete. There is an increase of around 23.1% and 33.9% in compressive strength for 20% RHA blended cement concrete at 28 and 56 days age of normal water curing.

Geetha & Kumar (2015) as reported by Kumar (2010) in their research got the compressive strength values of RHA concrete to be in the range of 70-80% of conventional concrete for a replacement of cement up to 20%. Early-age strength of RHA concrete was reported to be lower with strength increases recorded with age.

CCW is a CaO source and a by-product from acetylene gas production process. It is generated when calcium carbide reacts with water to produce highly flammable acetylene gas. It is majorly sourced from auto factory (Panel-beaters and Welders) as the a result of calcium carbide use by them (EMI, 2017). 10% replacement of PC with CCW was reported by Yunusa (2015) to give a satisfactory result with increase in water absorption as the RHA content in the mix increases.

MATERIALS AND METHODS

The material constituents, their mix, presence of admixtures and manufacturing process are important factors that determine the properties of the mortar cubes. The materials used and method of manufacture employed in this investigation are thus presented.

Materials

The materials for this study are Portland cement (PC), rice husk ash (RHA), calcium carbide waste (CCW), sand, superplasticizer and water.

PC

The Dangote (3X) brand of Portland cement (CEM 42.5N) from a dealer at Gidan Kwano, Minna, Niger State was be used in this study. Various physical tests were conducted on the materials for their characterization and assessment of conformity with BS standard (BS EN 197-1:2011) for Portland cement. Result of the chemical and physical properties of the RHA, CCW and the PC are given in Table 1.

RHA

The Rice Husk was collected from a milling site at Garatu Village, Minna, Niger- State. The husk was burnt to ash using the locally fabricated incinerator available at Building Department Laboratory of Federal University of Technology, Minna by open air method for about 48hours. It was allowed to cool before harvesting and milled with ball milling machine at Federal Polytechnic, Bida so as to achieve the required fineness. The fineness test was conducted to ascertain the fineness as required and stored in an air tight polythene bag. Samples of the resulting ash and powders were then packaged and sent to Ewekoro Works Department of Lafarge Cement for chemical analysis.

CCW

CCW was collected from the Mechanic Village at Keteren – Gwari as the by-product of oxy-acetylene gas welding. It was first Sun-dried and later Oven-dried at 105^{o}C in the Building Department Laboratory. The dried CCW was ground with ball mill at Federal Polytechnic, Bida. Fineness test by wet sieving method was conducted and the final powdered material kept in air tight polythene bag.

Fine Aggregate

Natural sand available in Minna served as the fine aggregate used in this study. Materials retained within 1.18 and 75μm (simulated reference sand) was adopted for this experiments. The sand has a specific gravity of 2.51 and an average moisture content of 1.20%. The coefficient of uniformity of the sand is 2.95.

Water

Portable water from the tap behind the Convocational Square of the Federal University of Technology, Minna used for the experiments was noted to be colourless, odourless and tasteless and free from organic matter of any kind.

Superplasticizer

High range water-reducing admixture MasterGleniun ACE 456 produced by BASF served as the Superplasticizer used in this study, as an attempt to combat the high water demand of RHA: CCW as observed in earlier study (Olawuyi, *et al*., 2017).

Methods

This study centres on the characterisation (physcio-chemical properties) of the materials and the fresh hardened properties (setting times, degree of hydration and compressive strength) of the RHA-CCW pastes and binders.

50 mm mortar cubes were cast, compacted with table vibrating machine and cured by immersion in water for varied ages (3, 7, 14, 12 and 28 days) before testing. The standard mix proportion of 1:3 cement-sand ratio and 0.5 water-cement (w/c) ratio in conformity with BS EN 196-1:2016 using CEM 1 42.5N serve as control, while the alternative binders was made of varied combination proportions (70:30; 60:40; 50:50; 40:60 and 30:70) of RHA-CCW.

Physical Properties of the materials

Particle size distribution

Particle size distribution of the available natural sand was conducted using the dry-sieve approach in accordance to BS EN 933-1:1995 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 sizes 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 2 of Section 4.1 present the particle size distribution of both the natural sand and the simulated reference sand.

Specific gravity and Fineness of the Binders

The specific gravity test was conducted on the constituent materials in accordance to BS EN 1097: 2013 while fineness test was also conducted on the CEM I 42.5N and the varied combination of RHA/CCW by wet sieving method in conformity to BS EN 196-6:2016 using a 53 µm sieve available in the Laboratory.

Setting Time of the Binders

The consistency test was carried out on the paste of the cementitious materials to determine the amount of water demanded for a standard consistency (Neville, 2012). Vicat apparatus Model No EL 38 - 2010 by ELE was used for measurement of the consistency. The setting time (initial and final setting times) was carried out in conformity with the procedures as outlined in the standard (BS EN 196-3:2011). The setting time both initial and final setting times and the Le Chatelier soundness tests for the binders (CEM I 42.5N and the various proportion combinations of RHA/CCW) were determined using neat pastes of standard consistency in accordance to BS EN 196-3:2011. The soundness test was also carried out on the respective binders using a Le Chatelier apparatus Model No EL 38 – 3400 by ELE.

Chemical Properties

The chemical property of the binders used was carried out with X-Ray Fluorescence (XRF) analysis for determination of the oxide composition (CEM I 42.5, RHA, and CCW) at Ewekoro Works Department of Lafarge Cement using XRF Analyser connected to a computer system for data acquisition.

Strength Properties of the harden mortar

Determination of strength with 50 mm mortar cubes which involves weighing of various constituents materials for the production of mortar ensuring a thorough mix of the materials. The alternative binders (RHA and CCW) were measured and mixed together; the measured reference sand was poured and mixed in a mixing pan thoroughly. The weighed water and superplasticizer, mixed together was then added to the already mixed materials and thoroughly mixed until homogeneous mortar paste is arrived at. The mixed paste was then cast into the oiled 50 mm cubes moulds. The control mortar samples 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 and superplasticizer before casting into the cube moulds. Based on observation from the setting times tests as reported in Section 4.2, the samples were left covered with jute bags and cured by water sprinkling until 48 hours before demoulding and cured by immersion in water until testing age. The mortar cubes were cast and crushed at the different curing ages (3, 7, 14, and 28 days) in the Digital Universal Testing Machine (DUTM – 20) to assess the strength development.

RESULTS AND DISCUSSION

Sieve Analysis (SA)

The analysis represented in Figure 1 shows the result of both natural sand and the simulated reference sand used for the experiment. It showed the simulated reference sand to have a C_u and C_c values of 2.16 and 0.96 respectively and a Fineness Modulus (FM) of 2.67 revealing it to be medium sand classification of Shetty (2004).

Table 1 shows the summary of the particle size distribution of the CEN reference sand for determination of cement strength as compare to the simulated reference sand. It was noted that four out of six size requirement was in compliance with CEN reference sand as required in BS EN 196-1:2016.

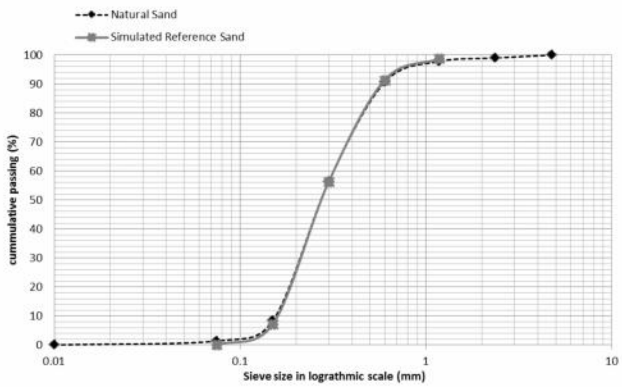

Figure 10: Sieve Analysis of Fine Aggregate

Despite the challenge observed with non-compliance of the size requirements on the simulated reference sand not correlating with the CEN reference sand as expected. The reference sand was adopted for the experiment since the study is mainly to compare the 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 agro-industrial waste binder was compared.

Table 2 present the specific gravity for the constituent materials. The result shows that the values are well fitted as earlier reports in literature (Neville, 2012)

Table 3: 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	28	√
0.50	67 ± 5	16	
0.16	87 ± 5	92	√
0.08	99 ± 1	99	√

Table 4: Specific Gravity of Constituent Materials (kg/m^3)

Materials	Average Gs
Cement	3.13
RHA	2.30
CCW	2.36
Sand	2.51

XRF result (Table 3) from Lafarge Cement in Ewekoro shows the oxide composition of the various binders used in this study.

Table 5: Result of XRF Analysis for Oxide Composition of Cementitious Materials

Samples	LOI	SiO_2	AlO_3	FeO_3	CaO	MgO	SO_3	NaO	K_2O	TiO_2	P_2O_5	Mn_2O_3	Cr_2O_3	AR	SR	SiO_2 + Al2O3 + Fe_2O_3
RHA	0.0	93.6	1.1	0.9	1.3	1.2	0.1	1.7	0.2	0.1	0.0	0.0	0.0	1.2	49.3	95.5
CCW	26.4	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	6.5
CEM I	0.0	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	27.8

It shows that RHA has silica content of about 94% - a good indication that the RHA is a good Pozzolan as well as a Class N Pozzolan with a total sum of the three major oxide composition (SiO_2+Al_2O_3+Fe_2O_3) of 96% far above the minimum70% as required by the standard (ASTM C618-2017). The RHA sample also meets the requirement of SO_3 below 4% and loss on ignition (LOI) of less than 10%. The CCW gave 66% CaO content, similar in value to that of the CEM I (64%). The SiO_2 and Al_2O_3 content was observed to be of lower value to that of CEM I while the LOI value is higher than the expected limit but preheating in oven at temperature 105^{0}C for 24 hour resulted in a

LOI of 2.9 as shown in Table 4 below the specified 10% maximum due the heat treatment adopted for also for effective performance.

Table 4: Result of LOI on binder materials

Binder	Cement	RHA	CCW
Wt. of Crucible (g)	52.0	48.0	60.0
Wt. of Crucible + Binder (g)	73.0	60.0	74.0
Wt. of Crucible +Ignited Binder (g)	73.0	60.0	73.6
L.O.I	0.2	0.0	2.9

Setting Times and Soundness of Binders

The consistency as well as soundness test results for the varied RHA: CCW binder combination and CEM I as control is presented in Table 5. The result revealed the water demand of RHA: CCW binder to be twice that of the CEM I pastes while Olawuyi *et al,* (2017) reported triple value of CEM I water demand. The adoption of Superplasticizer dosage administered improved setting time with a maximum twice value and one sixth of initial and final setting respectively of CEM I (Figure 2) as compare to Olawuyi, *et al.,* (2017), with one and half and triple value of CEM I initial and final setting time respectively. RHA: CCW binders reflect a trend of similar penetration values to the control. It is further noted that the higher the RHA content, the higher the quantity of superplasticizer demanded for the consistency paste. The water demand was then accounted for in the mortar production process for strength test of the binders.

Table 5: Fresh Properties of Binders

RHA/CCW (%)	Final Setting Time (min)	Initial Setting Time (min)	Depth of Penetration
CEM 1	340	138	6
70/30	369	195	5
60/40	350	152	6
50/50	412	160	6
40/60	515	257	5
30/70	570	301	6

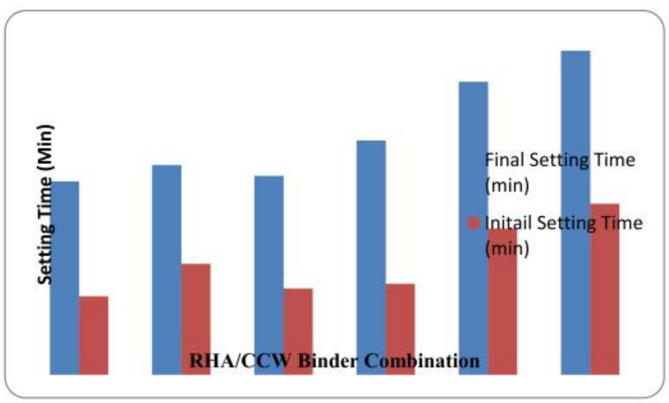

Figure 11: Setting times of the RHA/CCW Binder

Figure 3 presenting the soundness test for RHA: CCW and CEM I in accordance to BS EN 197-1:2011 revealed that all the binder combinations conform to the 10 mm maximum expansion specified by the standard.

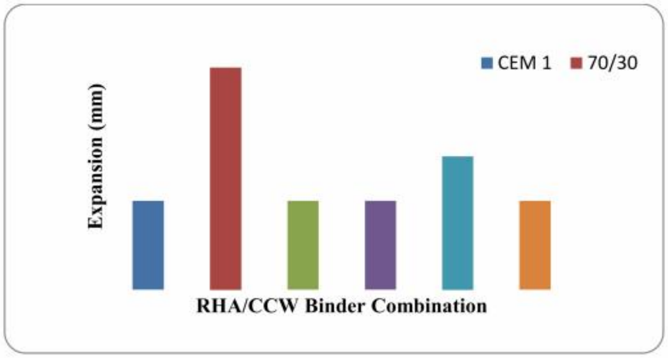

Figure 3: Soundness of the Binders

This affirms literature postulation that Pozzolan are of latent setting in nature. Addition of superplasticizer however helps in accelerating the setting times which is was confirmed by the enhancement of the strength development trends.

Strength of RHA: CCW Binders and CEM I

The plot of the compressive strength of the binders is presented in Figures 4. It follows similar trend of results with 60:40 RHA: CCW having at 28day compressive strength values of 8.78N/mm^2 [MPa] -31% of CEM I based mortar (control) strength while Olawuyi *et al,*(2017) had 5.3 N/mm^2 [MPa] – 25% of CEM I strength which indicates an improvement on strength development resulting from pre-heat treatment on the alternative binders and the adoption of the water reducing admixture (superplasticizer).There was a better strength development of the RHA: CCW observed in this study, the binding property of the materials was noted to be more effective when the samples were demoulded after 48hours. The mortar were in perfect condition when immerse in water and all through the curing period.

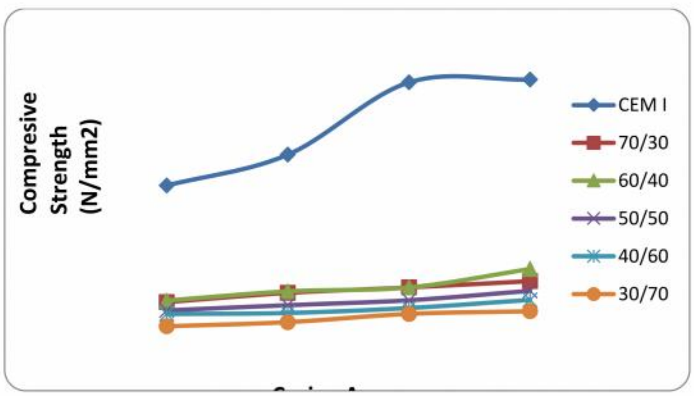

Figure 4: Compressive Strength of RHA/CCW Binder

CONCLUSION AND RECOMMENDATIONS

Conclusively, the results from the study indicates that the application of superplastizer help in reduction of water demand for the standard consistency paste which in turn improve the setting time and hence the strength development of the RHA: CCW mortar cubes as compared to the mortar cubes made without superplastizer. RHA: CCW binder at the varied combinations exhibit goo binding properties while the individual materials (RHA and CCW) met the codes requirements as a Pozzolan on basis of its physcio-chemical properties. The chemical analysis shows RHA as a Class N Pozzolan of high SiO_2 content (94%) while CCW is a good CaO source of similar percentage concentration as the CEM I used for the study. The following are thereby recommended based on the findings of this study.

i. RHA/CCW (60/40) in 1:3 binder/sand mortar at 0.5 W/B with water-reducing admixture can be adopted for use in masonry works

ii. Further studies on the RHA: CCW should focus on influence temperature above the ambient temperature on the setting time and the strength development trend.

iii. Studies the micro structure and products of hydration of the mortar specimen should be carried out to ascertain the performance of the new alternative binder.

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