J. Chem. Soc. Nigeria, Vol. 42, No.1, pp 7-11 [2017]

DETERMINATION OF OPTIMUM BURNING TIME AND SILICA COMPOSITION OF RICE (*Oryza sativa*) HUSK ASH AND GUINEA-CORN (*Sorghum bicolor*) HUSK ASH FOR POZZOLANA PRODUCTION

[1] A. A. Jimoh, [2]* O. M. Ameen and [3]J. A Atolagbe
[1]Department of Civil Engineering, University of Ilorin
[2]Department of Chemistry, University of Ilorin
[3]Department of Industrial Chemistry, University of Ilorin, Ilorin
Accepted:17/02/2017
*Corresponding Author: moameen@unilorin.edu.ng

Abstract
The quest for production of cheap construction cementing material from agricultural wastes, particularly the ones with high silica content, formed the basis of this study. The process of determining the silicious material and silica content was carried out on two agricultural waste husk types, the Rice (Oryza sativa)husk and Guinea corn(Sorghum bicolor) husk to produce rice husk ash (RHA) and guinea corn husk ash (GCHA) respectively. Both husks were burnt separately in a furnace at 650 ^oC to produce the ash. The ash physical, chemical and cementitious properties were investigated. The particle size range of the ash was between 0.063 and 0.25mm while the silica content were 70.6 % and 79.1 % for both the RHA and GCHA respectively, at burning time of 6 hours. Their respective ash cementing property was tested by mixing their ashes with varying proportion of lime and water to form paste. The hardened paste 28th day compressive strength was 3.0 N/mm^2. In comparison with common mortar, the ashes can be applied for plastering activities and block beddings.

Keywords: *SEM, EDX, pozzolans, cementitious, ash, tensile strength.*

Introduction

The presence of silica oxide (SiO_2) in an agricultural waste is an indication of its cementitious and pozzolana potential [1, 2]. According to Gupta and Amit[1] and Neville [2], a pozzolana is a silicious or silicious (SiO_2) and aluminous material which itself possesses little or no cementitous properties, but in finely divided form and in the presence of moisture (H_2O), it chemically reacts with calcium hydroxide ($Ca(OH)_2$) at ordinary temperatures to form compounds possessing cementitious properties. Silica in the pozzolana mixed with cement forms a calcium silicate hydrate (CSH) gel around the cement particles in the paste and makes the paste highly dense and less porous, and may increase the strength of concrete against cracking [3 – 5]. A pozzolana can be sourced from agricultural waste or from mineral origin for example rocks.

According to Ashworth and Azevedo [6], agricultural waste, which includes both natural (organic) and non-natural wastes, is a general term used to describe waste produced on a farm through various farming activities. These activities included but are not limited to dairy farming, horticulture, seed growing, livestock breeding, grazing land, market gardens, nursery plots, and even woodlands.

Agricultural and food industry residues refuse and wastes constitute a significant proportion of worldwide agricultural productivity and it has variously been estimated that these wastes can account for over 30% of this productivity [6]. The focus of this study is to find a way of utilizing the wastes from agricultural activities for producing cementitious material (pozzolana) as mortar in plastering.

Cement generally is an essential ingredient in concrete production because it acts as the vital binding agent. Cement used in construction is characterized as hydraulic or non-hydraulic. An example of hydraulic cement is the Portland cement, which hardens because of hydration. Hydration is a chemical reaction and occurs when water is mixed or placed underwater or when constantly exposed to wet weather. Non-hydraulic cements are lime and gypsum plaster and must be kept dry in order to retain their strength [7]. Lime, gypsum, lime plus pozzolana, and lime plus clay are early types of cement [8]. The technical knowledge of making hydraulic cement was later formalized by French and British engineers in the 18th century [8, 9]. History has it that pozzolanic materials have been used for construction for many years; however, their use has reduced drastically since the discovery of Portland cement. Ordinary Portland cement (OPC) is the most

ISSN 0795-2066

common type of cement produced and used throughout the world for building construction and ready mix concrete industries [8, 9]. However, pozzolana can find use and application in low strength concrete structures and where the raw materials are in abundance. In Nigeria and other parts of the world, agricultural waste can be continually reproduced and increased due to the nation's call for more participation in agriculture. Most agricultural wastes like rice husk and guinea corn husk are burnt off, producing emissions which are thrown into the atmosphere. However, utilization of these waste materials will lead to reduction in pollution in the environment and also will greatly improve the economy of the nation.

Therefore, this work wish to investigate the pozzolana properties of rice husk ash (RHA) and guinea corn husk ash (GCHA) and their uses as cementitious material as an alternative to Portland cement.

Materials and Methods

The accomplishment of the study involved collection, treatment and burning of the rice and guinea corn husk to produce ash.

Sample collection and treatment

The materials were rice and guinea corn husks. Both were obtained from the rice millers and farmers respectively in Ilorin, Nigeria. The husks were pretreated by removal of remains of the plant stalk and non – husk materials by hand picking, so as to make them clean, and then sun-dried. Other materials used were the lime, cement and tap water.

Ash preparation

Husk weight of 300g was poured into each of clean crucible of known weight. They were then put into a muffle furnace, which later was switched on and set to a temperature of 650^{o}C.The maximum temperature of 650^{o}C was maintained so as to get amorphous compound which makes the silica highly reactive as reported by Sarangi and Beher [12]. As burning continued, three crucibles each were removed at an interval of preset time of two hours, that is, 2,4,6,8 and 10 hours, respectively. The crucibles with burnt ash were cooled in a desiccator to room temperature, re-weighed, and the percentage of ash content was determined using equation 1.

$$\%Ash = \frac{(weight\ of\ ash + crucible\ ) - weight\ of\ crucible}{weight\ of\ husk} \ X\ 100 \qquad (1)$$

The ash was then ground manually using mortar and pestle, to achieve the minimum surface area required of pozzolanic materials.

Chemical tests of ash

Seven oxides (SiO_2, Al_2O_3, CaO, Fe_2O_3, MgO, Na_2O_3 and K_2O) were determined. Test for Silica (SiO_2) was particularly important as its percentage is a criterion for a good pozzolana. The test was undertaken according to the ASTM C618 [13] for Gravimetric Analysis standard procedures. Silica content was tested by using 0.2 g of the ground ash on a filter paper and adding calcium hydroxide solution. The residue from the filter paper was heated at about 900^{o}C, cooled and re-weighed. A drop of sulphuric acid was added followed by treatment with hydrofluoric acid in order to expel SiO_2 present. The residue was then fumed, dried, cooled and weighed. The difference between the weight of the residue and the initial weight of the ash (0.2g) gave the weight of SiO_2content, which was then expressed as a percentage of the original samples.

Other Oxide composition

These were determined using Energy Dispersive X-ray Spectrometry (EDX) method. Elements were detected at high sensitivity and with a spatial resolution on the order of a few hundred nanometers. The detection of a line showed the presence of a particular oxide.

Test for ash particle shape and size

Test for particle shape was carried out using Scanning Electron Microscope (SEM) to show the sample particle image and texture. This was carried out at Kwara State University, Malete, Nigeria. A thin film of the ash specimen coating was used, with appropriate setting of microscope apertures and charging of the samples at 15 kV to display the sample image. The ash grain size was undertaken at the Civil Engineering and Geology Departments, University of Ilorin. Particle size distribution of RHA and GCHA was determined by sieve analysis using sieve aperture ranging from 0.6to 0.063mm, arranged in descending order. After pouring 5 g of the ground ash sample into the top sieve, they were shaken manually for particles to pass through. The particles passed through 0.6 mm but retained on 0.06 mm, indicating that the sizes ranged between 0.6 and 0.06 mm.

Test for cementitious properties of the ash

The ash was mixed with lime. This is to effect practically the pozzolanic definition in which the silica from the ash reacts with the calcium hydroxide from the lime, to form the cementitious paste. In this test, the lime was a waste obtained from automobile panel welders. Three ash-lime mix samples were studied: 0% ash + 100 % lime ; 25% ash + 75 % lime and 75% ash + 25 % lime , respectively, mixed with 0.5 water to composite weight ratio to form paste. The paste was cast in steel cylinder moulds for tension test and in 50 mm cube mould for compression test. After 24 hours,

ISSN 0795-2066

removed and cured by covering with polythene sheet for 28 days. The cast sample weight and dimension were determined and tested for tensile strength. Test method was carried out in a universal testing machine following the ASTM procedure as given in [1, 2]. The loading rate of 0.015 N/mm^2/sec was used based on the specification for tensile splitting strength. For tension, failure occurred by a split or cracks of the cylinders along the vertical plane and separated into two neat halves. The compression samples failed by crushing, while density is calculated as mass of mortar cylinder divided by its volume, and expressed in kg/m^3.

Results and Discussion

Results for the chemical properties are presented in Table 1.From the table, as the time of burning increases the oxide composition percentage increases, reaching an optimum and then decreases. This trend is presented in Fig.1, where the silica (SiO_2) optimum percentage and the corresponding burning time are 70.61 % and 6 hours for RHA and 79.17 % and also6 hours for GCHA, respectively. Similarly, the optimum percentage and the corresponding burning time for other oxides are different from one oxide to another. The results in the table also indicate that the husks have high silica content while the cap shape of the curve in Fig. 1 indicates that burning makes the oxides to form and beyond optimum, decomposed. Thus in order to optimize silica content in the ash, the corresponding burning time need be adhered to. Further, the table also shows that the silica oxide composition in cement (OPC) is lower than that obtained for the ash with that of cement values varying from 17 to 25 %. While the oxides of cement are artificially infused, that of the ash is naturally infused. As the pozzolanic activities of the husk ash depends on its silica content, and then the time of burning and the temperature require purposeful control. In addition, CaO is low in the ashes but high in cement which may make the ash not producing ash of high comparable strength.

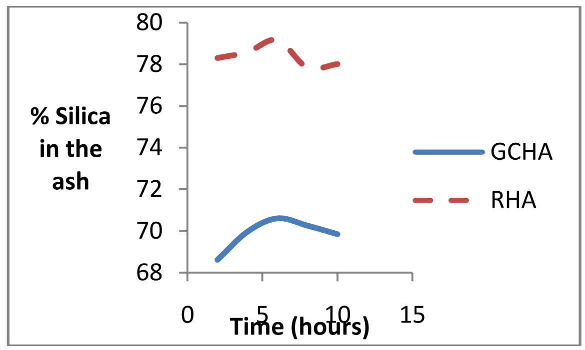


Fig 1. Trend to show time of burning and silica percentage in the ash for RHA and GCHA

Table 1: Percentage Chemical Composition of RHA and GCHA at different burning times

Percentage Chemical Composition of the ash at different burning times											OPC
Time	**2 hrs**		**4 hrs**		**6 hrs**		**8 hrs**		**10 hrs**		
OC	**R**	**G**	**R**	**G**	**R**	**G**	**R**	**G**	**R**	**G**	
SiO_2	68.62	78.31	69.97	78.59	70.61	79.17	70.26	77.85	69.85	78.02	17-25
Al_2O_3	0.56	1.33	0.70	1.46	0.84	1.52	0.47	1.43	0.47	1.40	3-8
CaO	0.58	3.61	0.59	3.74	0.61	3.12	0.60	3.28	0.54	2.99	60-67
Fe_2O_3	0.63	0.84	0.63	0.80	0.62	0.62	0.63	0.73	0.51	0.71	0.5-6.0
MgO	0.91	3.76	1.00	3.50	0.87	4.17	1.03	3.87	0.82	3.82	0.5-4.0
Na_2O_3	0.92	2.92	0.91	2.87	0.83	1.99	0.89	2.56	0.77	2.77	0.3-1.2
K_2O	0.60	7.67	0.63	7.55	0.69	7.43	0.62	8.12	0.59	7.91	-
ACR (g)	5.42	5.07	5.56	5.52	5.61	5.71	5.39	4.97	5.05	4.85	

Key: OPC = Cement (OPC) oxide composition at 4000 ^oC (After Neville [2]); R = Rice Husk Ash; G = Guinea Corn Husk Ash; ACR = Ash Content Residue; OC = Oxide Composition; Time = Burning Time

Ash-lime composite

The outcomes of the test on ash-lime composite gave densities varying from 2130 to2810 kg/m^3. When this is compared with density of normal weight concrete of 2200 to2600 kg/m^3[12], it can be deduced that the ashcan produce normal weight mortar. On the compressive and tensile strength shown in Table 2, for75 to 25 % ash-lime mixture produced, the highest strength for both RHA and GCHA, is 3.0 N/mm^2 while the tensile strength is 0.2 N/mm^2, respectively. As the ash percentage is increased the strength increases, showing that the ash is pozzolanic. This value, though low, indicates that the material is usable as plastering and bedding purpose [1]. The composite develops strength at slow rate when compared with cement mortar. The strength of composite is about 1/5th of that of cement mortar.

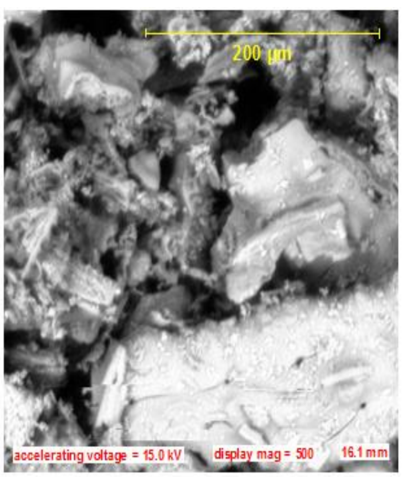
Grain size and shape

The ash grain size in this study for the RHA that was between 0.063 and 0.112 mm, with that of GCHA being between 0.25 and 0.6 mm, is courser than for OPC. According to Neville [2, 13], 95 % of a given quantity of OPC should possess size within 3 to 30 µm, with corresponding fewer very fine and fewer very coarse particles. The reason for ash grain being courser than that of OPC was that the ash was grinded manually in a crucible whereas OPC grinding is usually carried out mechanically and industrially in a ball mill [2]. Cement particles are not spherical [2], while also the shape of the ash is not spherical but angular as given in Plate I.

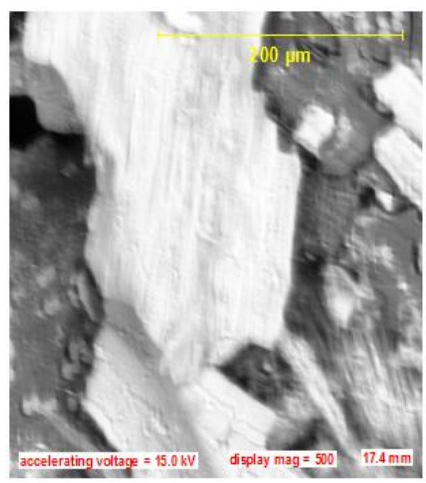
Table 2: Densities and strength of RHA-Lime and GCHA-Lime composites

Lime:ash mixture (%)	2hrs		4hrs		6hrs		8hrs		10hrs	
	RHA	**GCHA**	**RHA**	**GCHA**	**RHA**	**GCHA**	**RHA**	**GCHA**	**RHA**	**GCHA**
	Densities of the RHA & GCHA -Lime composite (g/cm^3)									
25:75	2.13	2.76	2.04	2.81	1.95	2.70	1.92	2.63	1.89	2.59
50:50	2.17	2.53	2.13	2.48	2.09	2.47	2.05	2.41	2.00	2.40
75:25	2.23	2.38	2.18	2.36	2.14	2.30	2.10	2.26	2.08	2.31
Compressive strength of the RHA & GCHA -Lime composite (N/mm^2)										
25:75	2.1	2.0	2.5	2.3	2.5	2.7	2.56	2.8	2.4	2.5
50:50	3.2	3.8	3.5	3.4	3.7	3.1	3.6	2.9	3.6	2.6
75:25	3.8	4.2	4.3	3.3	4.4	2.9	3.9	3.6	4.3	4.2
Tensile strength of the RHA & GCHA -Lime composite (N/mm^2)										
25:75	0.24	0.20	0.24	0.26	0.20	0.27	0.18	0.22	0.20	0.25
50:50	0.37	0.26	0.35	0.26	0.38	0.24	0.28	0.28	0.30	0.26
75:25	0.34	0.28	0.30	0.28	0.32	0.29	0.32	0.29	0.2.5	0.28

Key: RHA = Rice Husk Ash; GCHA = Guinea Corn Husk Ash



GCHA particles



RHA particles

Plate I: Image of RHA and GCHA structures in Scanning Electron Microscope

Conclusion

From the study, it can be concluded that the optimum silica is produced at 6 hours burning time for both rice husk and guinea corn husk. The Silica content of the Ash is higher than what is obtainable for Ordinary Portland Cement but has lower CaO content. Behaviour of Ash in lime forms a mortar material that is cementitious. Promotion of the use of the Ash from

ISSN 0795-2066

burnt RHA and GCHA as cement admixtures will help to reduce both agricultural wastes and environmental pollution.

References

1. B.L.Gopta, and G.AMIt (2004) Concrete Technology, 3rd Edition, Standard Publishers Distributors, Delhi, pp 50 – 53.
2. A.M. Neville (2011) Properties of Concrete, Fifth Edition,Pearson Education,England, pp1 – 83.
3. M. U.Dabai, U. B.Muhammad,A. Musa (2009). Studies on the Effect of Rice Husk Ash as Cement Admixture, Nigerian Journal of Basic and Applied Science, 17(2): 252 – 256.
4. A.M. Neville and J.J. Brooks (2010). Concrete Technology, Pearson Education Limited, England, PP 8 – 33.
5. L. D.Robert (1990). Pozzolanas for use in low-cost housing, A state of the art report prepared for: the International Development Research Centre,Ottawa, Canada, pp 78 – 80. Also available at https://idl-bnc.idrc.ca/dspace/bitstream/10625/5782/1/49685.pdf. G.S.Ashworth and P.Azevedo (2009). Agricultural Wastes, Agricultural Issues and Policies Series, 978(1): 61728 – 132.
6. S. F.Mohamad (2012). Introduction to Cement, Civil Engineering Materials SAB 2112.
7. B. O.Okoya, (2009). Investigation into the Cementitious Properties of a Mixture of Rice Husks Ash with Building Lime, A thesis submitted to the Department of Civil and Construction Engineering, University of Nairobi, Kenya, in partial fulfillment for the Degree of Master of Science in Civil Engineering, pp 1 – 5.
8. S. A. Akinloye, G.M. Bankole and A. Medubi (2014) Effect of Lime-Guinea Corn Husk Ash on the Engineering Properties of Lateritic Soil, The Electronic Journal of Geotechnical Engineering, 19: Bund. Z6, 17693 – 17699.
9. G. A.Rama Rao, R. K.Sastry, P. K.Rohatgi Bull, (1989). Nature and Reactivity of Silica available in Rice Husk and its Ashes Matter, Science Volume, 12(5): 469 – 479.
10. V. Saraswathy and S. Ha-Won (2007). Corrosion performance of rice husk ash blended concrete, Construction and Building Materials, 21(8): 1779 – 1784.
11. M. S.Sarangi and R. C.Beher, (2009). Effect of Temperature on Morphology and Phase Transformations of Nanocrystalline Silica Obtained from Rice husk. Phase Transitions: A Multinational Journal, 82(5): 377 – 386.
12. ASTM C618. (1993). Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete. American Society for Testing Material, pp 1 – 5.

ISSN 0795-2066