

Comparative Elemental Analysis of Rice Husk Ash Calcined at Different Temperatures Using X-ray Fluorescence (XRF) Technique

Taku, J. K^{1,*}, Amartey, Y. D², Kassar T¹

¹Department of Civil Engineering University of Agriculture, Makurdi, Nigeria

²Department of Civil Engineering Ahmadu Bello University, Zaria, Nigeria

*Corresponding author: kumataku@yahoo.com

Abstract In this study, the effect of the calcination temperature of rice husk on the pozzolanic properties of the resulting rice husk ash (RHA) especially its silica content was investigated. Rice husk was collected from a rice milling plant and washed to remove sand and other impurities, beneficiated using the water beneficiation method and calcined at temperatures of 400, 500, 600, 700 and 800°C, respectively for three hours. Samples were taken for XRF analysis, setting time determination and specific gravity test. The result of XRF analysis revealed that RHA calcined at temperatures between 400°C and 800°C contains more than 70% silica as stipulated by ASTM C618 for pozzolanas. The silica content though varies slightly with different calcination temperature of the rice husk ash. Also, calcination removed impurities present in the rice husk. Besides that, the specific gravity of RHA decreases with increasing calcination temperature from 2.00 at 400°C to 1.05 at 800°C. Setting times of RHA mortars at 15% replacement of OPC with RHA shows no definite pattern with increasing temperature. However, the initial and final setting times of OPC-RHA mortars at all calcination temperatures were higher than that of OPC mortar. As a whole, calcination improves the silica content of rice husk ash for use as a pozzolana as well as removes mineral impurities that may affect the pozzolanic properties of the rice husk ash.

Keywords: rice husk ash, calcination temperature, oxide composition, setting time, specific gravity

Cite This Article: Taku, J. K, Amartey, Y. D, and Kassar T, "Comparative Elemental Analysis of Rice Husk Ash Calcined at Different Temperatures Using X-ray Fluorescence (XRF) Technique." *World Journal of Agricultural Research*, vol. 4, no. 1 (2016): 28-31. doi: 10.12691/ajcea-4-1-4.

1. Introduction

Rice husk is an agricultural waste product obtained from the milling of rice and it contributes to about 20% of the weight of rice [1]. The major compounds from rice husk are silica and cellulose which yields carbon when thermally decomposed [2]. In the majority of rice producing countries much of the husks produced from the processing of rice is either burnt or dumped as a waste, which constitute an environmental challenge.

Starting from 1973 however, various researches have been carried out, not just to control the disposal and pollution problems associated with rice husk but also to find a value addition to this waste product by using it as a secondary resource material. In certain regions, the husk is used as fuel for parboiling paddy in the rice mills [3]. The use of rice husk in the power production has been reported by Reddy and Alvarez [4] and several researchers have investigated on the use of rice husk ash (RHA) as a pozzolanic material which can be used in partial replacement of cement [5-11]. However, a comparison of a typical pozzolanic reaction, $(CH + S + aq \rightarrow C-S-H)$ and Portland cement hydration, $(C_3S + aq \rightarrow C-S-H + CH)$, shows that it is the silica component of the RHA that

contributes to strength development when used as a pozzolana. For RHA to be used as a pozzolanic material in concrete or cement production, ASTM C618 specifies that the silica content of the ash $+Al_2O_3+Fe_2O_3$ should be 70% minimum. Thus, it is imperative that the silica content of RHA meet this specification to be used as a cement replacement material.

A research indicates that RHA from the same source calcined at the same temperature but using different methods has different silica content levels [12]. The silica content of RHA also depends on the location from which the rice husk is obtained as well as the variety of rice used [13].

Varying the calcination temperature of rice husk as well as the calcination time will to a large extent determine whether the silica content of the husk will remain amorphous or produce silica in the crystalline phase [3]. In this research work, the effect of the calcination temperature of rice husk ash by burning in a programmable furnace and at a constant calcination time, on the silica content of the rice husk ash is investigated.

2. Experimental Program

The experimental program for this research work includes the treatment of rice husk and the testing was carried out on rice husk ash.

2.1. Materials and Methods

The materials used for this research work include:

Rice husk- It was collected from rice milling plant dump in Gboko, Benue state, Nigeria.

Dangote cement -a brand of Ordinary Portland cement produced locally in Nigeria-was used in the course of the research. Tests were carried out in accordance with BS EN 196 to determine the physical and chemical properties of the cement used.

2.2. Treatment of Rice Husk

The rice husk was washed with water to remove impurities and dried through direct sunlight. Then, the husk was burnt in the open air (i.e., carbonation under uncontrolled conditions) to reduce the carbon content of the rice husk ash, after which it was loaded into a furnace and fired at a steady temperature of 400°C for two hours. The RHA was left in the furnace to cool for 48 hours. After that, a beneficiation of the RHA was carried out using the water beneficiation method. In this method, the carbonated rice husk was washed with water to remove sand and unburnt carbon (charcoal) material present. It was then washed using a BS (British Standard) sieve (150µm aperture). The ash was then soaked in a dilute solution of 2M nitric acid for about two hours. The aim of the soaking is to remove carbonized material and any other acid soluble material. The acid was washed off and the ash dried in the open air for 48 hours. Subsequently, the sample was divided into five equal parts and burnt at temperatures of 400, 500, 600, 700 and 800°C, respectively for three hours using a programmable furnace of 1300°C capacity. The burning time was chosen following Salas et al (2009).

2.3. Mix Design and Mixing Process

The aim of mix design is to determine the proportion of each of the constituents of the cement mortar in the mix. The absolute volume method of mix design was used to

determine the quantities of cement and water required. Water to cement ratio of 0.5 were used. The OPC was replaced with 15% RHA. The quantities of the various materials used are presented in the Table 1

Table 1. Mix Design Quantities

Material	Quantity for control	Quantity per RHA-OPC mix
Cement	0.61Kg	0.524Kg
Water	0.308Kg	0.308Kg
RHA	-	0.092Kg

2.1.4. Tests on Rice Husk Ash

Samples were taken from the rice husk, beneficiated rice husk ash and rice husk ash calcined at 400°C, 500°C, 600°C, 700°C and 800°C respectively for oxide composition analysis. This analysis was carried out using XRF technique at the center for energy research of the Ahmadu Bello University Zaria, Nigeria.

Other tests carried out include specific gravity test on RHA calcined at temperatures of 400°C, 500°C, 600°C, 700°C and 800°C respectively. The specific gravity was determined as per BS 4550-3: 1978. Initial and final setting time tests were also carried out on OPC-RHA mixes at 15% replacement levels for RHA calcined at 400, 500, 600, 700 and 800°C, respectively, using Vicat's apparatus. The test was carried out in line with the provisions of BS EN196-3:1995.

3. Results and Discussion

3.1. Oxide Composition Analysis

Table 2 shows the result of XRF analysis carried out on the RHA to determine the oxide composition at the various calcination temperatures. The rationale behind conducting XRF analysis was to determine if change in calcination temperature will have any effect on the elemental composition of the RHA especially its silica content.

Table 2. XRF analysis for rice husk and rice husk ash

Compound	Percentage composition						
	Rice husk	Ben. RH	RHA@400	RHA@500	RHA@600	RHA@700	RHA@800
Al ₂ O ₃	5.0	1.4	2.8	3.48	2.0	3.22	3.50
SiO ₂	48.8	69.0	71.4	72.5	73.1	73.2	72.7
Fe ₂ O ₃	3.23	2.57	2.60	1.78	1.17	1.77	1.92
Pb ₂ O ₃	-	8.67	8.97	9.82	7.17	8.75	9.60
SO ₃	8.75	1.2	0.24	0.21	0.34	0.15	0.17
K ₂ O	10.2	7.33	7.30	6.02	3.45	5.67	5.98
CaO	3.23	4.41	4.51	4.63	2.75	4.38	4.61
TiO ₂	0.38	0.37	0.37	0.23	0.20	0.24	0.24
Cr ₂ O ₃	0.17	0.088	0.09	0.16	0.03	0.003	0.003
MnO	0.679	0.669	0.70	0.70	0.41	0.67	0.69
NIo	0.18	0.12	0.10	Nd	0.03	0.06	0.005
CuO	0.12	0.089	0.09	0.12	0.02	0.02	0.03
ZnO	0.10	0.22	0.22	0.13	0.09	0.17	0.17
Cl	1.34	-	Nd	Nd	0.02	Nd	Nd
BaO	0.43	0.28	0.28	0.007	0.10	0.12	0.11
Re ₂ O ₃	0.99	0.17	0.17	0.15	0.14	0.12	0.13
Na	-	5.17	Nd	Nd	Nd	Nd	Nd
V ₂ O ₅	-	0.02					
Cr ₂ O ₃	0.17						
IrO ₂	0.74						
PtO	3.23						
PbO	1.3						
OsO ₄	0.57						

Nd=not detected.

The result of XRF for untreated rice husk, beneficiated rice husk ash and RHA calcined at 400, 500, 600, 700 and 800°C, respectively, indicates that the SiO₂ content of the ash changes slightly with temperature. Untreated rice husk has a SiO₂ content of 48.8%. Meanwhile, the calcined RHA at all temperatures has silica + Al₂O₃+Fe₂O₃ content above 70%, which falls within the limit specified by ASTM C618 for a class C pozzolan. Materials that are undesirable in a pozzolana such as Cr₂O₃, IrO, PtO, PbO and OsO₄ are removed following beneficiation and subsequent calcination.

The SO₃ content was greatly reduced from 8.75 in the untreated rice husk to tolerable limits (ASTM C618 specifies 5% max.) in the calcined product. Generally, Cl, TiO₂, Cr₂O₃, MnO, CuO, ZnO, BaO, and Re₂O₃ which were a part of the untreated rice husk eliminated or reduced to trace quantities during the calcination process. At a whole, the calcination improved the pozzolanic characteristics of RHA.

3.2. Specific Gravity of Calcined RHA

The specific gravity for RHA calcined at 400, 500, 600, 700 and 800°C were 2.00, 1.82, 1.71, 1.50 and 1.05, respectively. The result is plotted in Figure 1. It can be seen that the specific gravity of RHA decreases with increasing temperature at all calcination temperatures. This is because the higher the calcination temperature, the more the amount of volatile materials expelled from the sample, and hence, the lower the weight of the material compared to the weight of an equal volume of water. Thus, it can be concluded that the weight of concrete incorporating RHA will decrease with increasing calcination temperature of RHA.

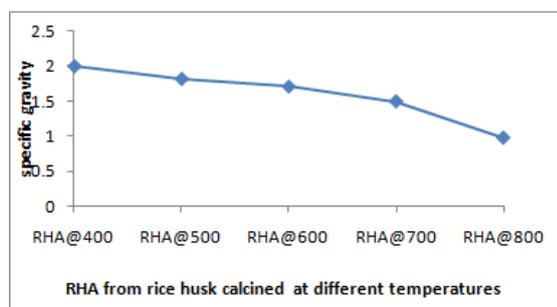


Figure 1. Specific gravity of RHA from rice husk calcined at different temperatures.

3.3. Setting Times of OPC-RHA Mortar

The result of initial and final setting times of OPC and OPC-RHA mixes is plotted in Figure 2.

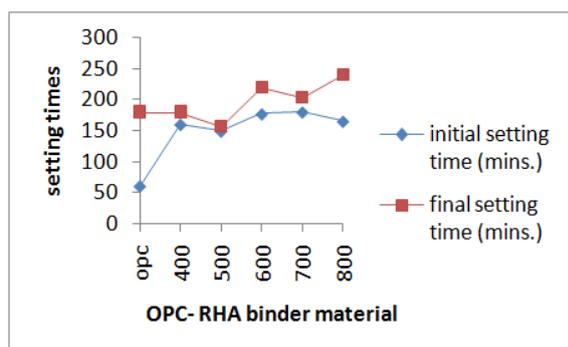


Figure 2. Variation of setting time with binder material

From the results, it can be observed that the OPC-RHA mix for RHA from rice husk calcined at 700°C has the highest initial setting time (180 minutes), while that at 400°C has the lowest initial setting time (149 minutes). The final setting times were obtained as 180, 157, 220, 204 and 240 minutes for OPC-RHA mix for RHA calcined at 400, 500, 600, 700 and 800°C, respectively. OPC-RHA mix for RHA calcined at 800 and 500°C having the highest and lowest final setting times, respectively. Figure 2 also displays that both the initial and final setting times of OPC-RHA mortars show no definite pattern with different calcination temperatures. However, the setting times of OPC-RHA cements for all calcination temperatures are higher than those of OPC mortars except for RHA calcined at 500°C, which has a lower final setting time. The setting times of RHA-OPC mortars are higher than for OPC mortars due to the slower pace of heat induced evaporation of water from the OPC-RHA paste [14].

4. Conclusion

The following conclusion can be drawn from the strength of this research work

1. The silica content of RHA from rice husk calcined at temperatures of 400, 500, 600, 700 and 800°C, respectively varies slightly with calcination temperature but in each case is more than the 70% minimum stipulated by ASTM-Specifications- C618 for pozzolanas.
2. Calcination time has no profound effect on the silica content of RHA calcined at temperatures between 400 and 800°C
3. The water beneficiation method of treating rice husk, coupled with thermal treatment at elevated temperatures removes undesirable elements from rice husk, thereby improving the pozzolanic properties of RHA for use as a pozzolana.
4. The Specific gravity of RHA decreases with increasing calcination temperature for RHA calcined between the temperatures of 400 and 800°C
5. OPC-RHA mixes at all temperatures show higher setting times than that of OPC pastes.

Acknowledgement

My profound gratitude is expressed to Dr. Olusegun Ajayi of the department of chemical engineering, Ahmadu Bello University (ABU) Zaria, Dr. Amana Ocholi and Dr. D. Y. Amartey, all of the department of civil engineering, ABU zaria for their contributions to the success of this research work. The Center for Energy Research, ABU Zaria is also appreciated for allowing me to use their equipment for this research work.

References

- [1] Salas, A., Ospina, M. A., Delvasto, S., Majia De Gutierrez, R.: "Study on the pozzolanic properties of silica obtained from rice husk by chemical and thermal processes". IPSS, 2007; 4(11): 4311-4318.
- [2] Adylov G, Faiziev SH, Paizullakhanon M, Mukhsimov S, Nodirmatov, E.: "Silicon Carbide materials obtained from rice husk". *Tech. Phys. Lett.* 2003; 29(3): 221-223.

- [3] Chandressekhar, S.: "Effect of organic acid treatment on the properties of rice husk silica". *Journal of Material Science*, 2005; 40(24): 6535-6544.
- [4] Reddy, D. V., Alvarez, M.: "Marine durability characteristics of rice husk ash modified reinforced concrete". *4th Latin American and Caribbean conference for Engineering and Technology. LACCET, 2006*.
- [5] Goyal, A; Kunio, H; Ogata, H; Garg, M; Anwar, A. M; Asharf, M and Mandula: "Synergy effect of wheat straw ash on strength properties of mortar". *Journal of Applied Science*, 2007; 7: 3256-3261.
- [6] Habeeb, G, Hilmi Bin Mahmud: "Study on properties of RHA and its use as cement replacement material." *Materials Research Journal*, 2010; 13(2): 185-190.
- [7] Kamal, N.L.M, Nuruddin, M.F, Shafiq, N.: "The influence of burning temperatures and percentage inclusion of microwave incinerated RHA on normal strength concrete", *ICCBT 2008 - A*-(47): 531-538.
- [8] Nehdi, M., Dequette, J., El Damatty, A.: "Performance of rice husk ash produced using a new technology as a mineral admixture in concrete". *Cement and Concrete Research Journal*, 2003; 33(8): 1203-1210.
- [9] Omotola, K. M; Onoja, A. D: "Elemental Analysis of rice husk ash using X- ray fluorescence technique", *International Journal of Physical Sciences*, 2009; 4(4): 189-193.
- [10] Oyetola, E. B., Abdullahi, M.: "The use of rice husk ash in low cost sandcrete blocks production. *Leonardo Electronic Journal of practices and Technology*, 2006; 8: 58-70.
- [11] Rasheed, A. H; Molla, A. K; Ahmed, T. U.: "Long term effect of rice husk ash on strength of mortar", *World Academy of Science, Engineering and technology*, 2010; 67: 740-743.
- [12] Salas, A; Delvasto, S; Gutierrez, R. M; Lang, D.: "Comparism of two processes for treating rice husk ash for use in high performance concrete", *Cement and Concrete Research*, 2009; 39: 773-778.
- [13] Bandara, D. H. M.: "Development of blended cement utilizing the pozzolanic amorphous silica component of rice husk ash". *Journal of National Science, Sri Lanka*, 1994; 22(2): 189-199.
- [14] Dakroury, A. E., Gasser, M. S.: "Rice husk ash as cement admixture for immobilization of liquid radioactive waste at different temperatures". *Journal of nuclear Materials*, (2008; 381(3): 271-277.
- [15] BS-EN 196-3: *Determination of setting time and soundness*, British Standard Institute, (BSI) London. 1995
- [16] BS 4550-3. *Method of testing cement- physical tests*, British Standard Institute, (BSI) London. 1978.