# RICE HUSK ASH (RHA) AND PALM OIL FUEL ASH (POFA) AND SOAKING TIMES: ANALYSIS OF COMPRESSIVE STRENGTH OF PORCELAIN CERAMICS

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## ABSTRACT

Rice husk ash (RHA) and palm oil fuel ash (POFA) is a byproduct from agricultural waste produced thousand tonnes every year. This paper presents the use of RHA and POFA as a substitute material for quartz in fabricating an improved porcelain ceramic. The RH was thoroughly washed with distilled water in order to remove adhering soil and dust. After that it was dried in an oven at 100 °C for 24 hours. Then the dried husk was subjected to the chemical treatment; 2M HCL, 5% solid at 25 °C before calcinations to increase silica content. Untreated POFA was dried in an oven at 100 °C for 24 h. It was ground in a ball mill for 1.5 h with the revolution rate of 200 rev/min to reduce the particle size. Untreated POFA was sieved to remove the particles coarser than 50 µm. The POFA was treated by heating it at a temperature of 600 °C for 1.5 h. The mixed powder was then pressed into pellets at mould pressure (MP) 91 MPa. All the pellets were sintered at the temperature of 1100 °C for 1 h hour, 2 h hour s and 3 h hours soaking times. It was found that the highest compressive strength occurred at 20 wt% RHA and POFA and a soaking time of 2 h. The increment in the strength could be attributed to the changes in the increase in mullite and critobalite.

Keywords: Compressive strength, Porcelain, Ceramics, XRF, XRD

# INTRODUCTION

Rice husk is an agricultural residue which accounts for 20% of the million tons of rice produced annually worldwide (Tchakouté et al., 2016; Noh et al., 2016; Shata et al., 2016). The RH produced is partially burnt husk from the milling plants when used as a fuel also contributes to pollution (Hassan et al., 2016; Jamo et al., 2014; Jamo et al., 2016). The chemical composition of rice husk is found to vary from one sample to another due to the differences in the type of paddy, crop year, climate and geographical conditions. Due to growing environmental concerns and the need to conserve energy and resources, efforts have been made to burn the husks at a controlled temperature and atmosphere, and to utilize the ash so produced as a supplementary cementing material. Many researchers (Zhang et al., 1996; Basha et al., 2005; Ganesan et al., 2008b; Chindaprasirt et al., 2008) found that RHA has pozzolanic properties and could be used as a replacement of cement in concrete. The test results on the performance of RHA revealed that it has a good potential in reducing the expansion due to alkali-silica reaction (Chindaprasirt et al., 2008a; Chindaprasirt et al., 2007). The RHA has low pozzolanic reaction due to its large particle size and porous structure. However, this is not enough, more alternative ways of utilising RHA has to be looked upon. Another ash that causing similar problems is POFA.

In Nigeria, palm oil production has had a dramatic increase over the past 3 years, becoming one of most important crop since 2015; the main use of palm oil residues are burned daily as fuel in the paddy milling process. The use of this fuel generates a huge volume of ash. The POFA has no useful application, is usually dumped into water streams and causes pollution and contamination of springs (Chindaprasirt et al., 2008a). To solve this potential environmental problem, many researchers have studied the uses of POFA in blended cement (Chindaprasirt et al., 2007). In order to improve the pozzolanic reactivity of POFA, the ground POFA (GPOFA) was therefore investigated. GPOFA is a good pozzolanic material and can be used as a replacement in Portland cement up to levels of 20 % and 30 % for medium particle size and small particle size, respectively (Chandara et al., 2012). Despite this RHA and POFA still remain a problem to the environment, more efficient ways of utilizing RHA and POFA has to be sourced. As a result, the RHA and POFA has aroused great interest in Nigeria. Therefore, RHA and POFA are minerals replacement for substitution of quartz in porcelain production. Hence, this research wishes to investigate the influence of combined effects of RHA and POFA on the compressive strength of porcelain body at different soaking times.

# MATERIALS AND METHODS

## Experimental

The RH was thoroughly washed with distilled water in order to remove adhering soil and dust. After that it was dried in an oven at 100°C for 24 hours. Then the dried husk was subjected to the chemical treatment; 2M HCL, 5% solid at 25 °C before calcinations to increase silica content. After the leaching process, the treated husk was washed with distil water and then dried again. The treated husk was then subjected to calcinations at 700°C for six (6) hours, after which it was subjected to the XRF analysis. The machine used for the analysis was XRF Bruker S4 Pioneer which was operated at 60 KV

The POFA was dried in an oven at 100 °C for 24 hours. After that it was be grounded in a ball mill to reduce the needed particle size to improve reactivity. The milling time was approximately 90 minutes at 200 rpm. Afterwards, the materials were subjected to a set of sieves less than 50  $\mu$ m in order to remove the particles coarser than 50  $\mu$ m. The untreated POFA was heated at a temperature of 600 °C for 1.5 hours in an electric furnace to remove excess carbon.

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Porcelain powder was grounded separately in a ball mill. The powder was sieved using sieve shaker and dried in an oven. The RHA and POFA was gradually incorporated into the body of porcelain powder from 5 %wt, to 25 %wt (Table 1). The composition was mixed using a ball mill for one and half hours. The mixed powder was pressed into pellets at mould pressure of 91 MPa. All the pellets were sintered at a temperature of 1100 °C for the soaking times of 1 hour, 2 h hours and 3 h hours, at a heating rate of 5 °C per minute. The compressive strength was determined. The chemical composition of the RHA and POFA was studied using X-Ray Fluorescence (XRF) machine while the amorphous structure of RHA and POFA was identified through XRD

**Table 1**: The composition with the substitution of quartz byRHA and POFA (wt %)

Sample name	Kaolin	Feldspar	Quartz	RHA	POFA 0	
AP1	50	25	25	0		
AP2	50	25	20	3	2	
AP3	50	25	15	6	4	
AP4	50	25	10	9		
AP5	AP5 50		5	12	8	
AP6 50		25	0	15	10	

## **RESULTS AND DISCUSSIONS**

X-ray fluorescence (XRF) analysis was used for the chemical analysis. Hence the amount of chemical elements can be observed (Table 2). The presence of various elements within the raw materials can be seen from the table. This table shows the result of XRF analysis of kaolin, feldspar, quartz, RHA and POFA. It is evident that SiO<sub>2</sub> is the major composition in all the raw materials they are: kaolin, feldspar, quartz, RHA and POFA with 69.3 wt%, 72.7 wt%, 99.4 wt%, 93.7 wt% and 66.9 wt% and then followed by alumina with 24.3 wt%, 16.4 wt%, 0.2 wt%, 2.1 wt% and 6.4 wt% respectively.

Table 2: X-Ray Fluorescence (XRF) Analysis

Sample					Cont	ent						
Oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	K20	P <sub>2</sub> O <sub>5</sub>	CaO	MgO	CO <sub>2</sub>	SO3	FeO3	Na <sub>2</sub> O	TiO <sub>2</sub>	LOI
RHA	93.70	2.11	1.18	0.96	0.81	0.53	010	0.45	-	-	-	0.16
POFA	66.91	6.44	5.20	3.72	5.56	3.13	-	0.33	5.72	0.19	-	2.30
Kaolin	69.30	24.30	2.44	-	-	-	0.10	-	0.27	-	0.27	0.36
Feldspar	72.70	16.40	0.50	2.42	-	-	-	6.87	0.40	0.29	-	0.10
Quartz	99.40	0.22	-	-	-	-	0.10	-	-	-	-	0.28

Figure 1 shows the volume shrinkage of the sintered samples containing RHA and POFA. Lower soaking time (1 h hour) favours lower volume shrinkage values. The volume shrinkage increases with the increase in replacement until complete replacement of quartz by RHA and POFA. The maximum volume shrinkage was achieved within this range of soaking time with approximate values of 17% on complete replacement of quartz by RHA and POFA. While for the soaking time of 2 h hour s the maximum of 20% value of volume shrinkage was recorded. At the soaking time of 3 h hour s the maximum volume shrinkage was recorded with a value of 21% on complete replacement of quartz by RHA and POFA. The wetting liquids act on solid particle to eliminate

porosity and reduce interfacial energy, since higher energy solid vapour interfaces gradually and replaced by lower energy solidsolid interfaces with a total decrease in free energy occurring on sintering.

Considering the soaking time, the volume shrinkage increases with the increase in temperature and the replacement of quartz by RHA and POFA. The volume shrinkage increases as the soaking time increases from 1 hours to 2 hours. The increase in the shrinkage may be attributed to the presence of fluxing alkaline and other oxides in the ashes RHA and POFA, which enter the liquid phase. Furthermore, previous reports (Prasad et al., 2001; Esposito et al., 2005; Pérez et al., 2014; Jamo et al., 2017b; Jamo et al., 2017c), show a similar trend where the results of shrinkage with different quartz, mullite and kvanite content sintered at different temperature is in the range of 4% to 8%. In another development the shrinkage of the fired samples is associated to other physical properties such as porosity and water absorption (Pérez et al., 2014; Jaya et al., 2016; Kongnoo et al., 2017; Mohammad Jaya et al., 2016; Noh et al., 2014). The shrinkage properties as function of firing temperature at the 1200 °C - 1300 °C range. Shrinkage, which initially increases, reaches a maximum value and above 1280 °C decreases due to increase in close porosity. The maximum values recoded in this study was at the temperature of 1200 °C.



Figure 1: Effect of soaking time on shrinkage of the samples with different percentage of RHA and POFA

Figure 2 clearly displays differential changes in bulk density through the soaking time. The bulk densities continued to increase, reached a maximum values of 2.33 g/cm<sup>3</sup>, 2.43 g/cm<sup>3</sup> and 2.38 g/cm<sup>3</sup> at a the soaking time of 1 hour, 2 hours and 3 hours on 20 wt% of RHA and POFA. Replacement above 20 wt% of RHA and POFA causes the values of bulk density to decrease. The increase of soaking time is as a result of increase in liquid formation from RHA, POFA and feldspar which fills up the voids thereby increasing the densification. Substitution above 20 wt% causes the porosity values to drop, due to excess glassy phase formation.



Figure 2: Effect of soaking time on the bulk density of the samples with different percentage of RHA and POFA

Considering soaking time the bulk density increases as the soaking time increases from 1 hour to 2 hours. That it is due to increased mobility of glass when the Na<sub>2</sub>O is heated (Jamo *et al.*, 2017a; Bondioli *et al.*, 2010; Abadir *et al.*, 2002; Noh *et al.*, 2018). The same phenomenon of glass mobility was also observed in case of K<sub>2</sub>O containing glazes during sintering. The Figure further indicates that vitrification took place at the soaking time of 2 hours. At a soaking time of 3 hours, the porosity tends to increase because of bloating. This results in decrease in bulk density.

The result of compressive strength versus soaking time is presented in Figure 3. The compressive strength increased with an increase in soaking time and replacement of RHA and POFA. With an approximate values of 40 MPa, 45 MPa and 43 MPa on 20 wt% of RHA and POFA the maximum compressive strength values was achieved at the soaking time of 1 hour, 2 hours and 3 hours respectively. Compressive strength was found to increase with increase in bulk density as observed by (Jamo et al., 2016; Jamo et al., 2017a; Zainudin et al., 2017), for triaxial porcelain compositions. Theoretically, compressive strength developed in a porcelain body is maximum when apparent porosity decreases to zero. Progressive inclusion of RHA and POFA causes the porosity to decrease, bulk density to increase which results in increase in the mechanical properties of the porcelain body. Substitution above 20 wt% causes the compressive strength to decrease. This is attributed to excess glassy formation.

In terms of soaking time compressive strength increases as the soaking time increases from 1 hour to 2 hours. In line with the quantitative XRD observation presented in Table 3 the compressive strength increases with increase in mullite and cristobalite. But as the soaking increases to 3 hours the compressive strength decreases due to bloating. It could be concluded that the best composition is 20 wt% of substitution of quartz by RHA and POFA.



Figure 3: Effect of soaking time on compressive of the samples with different percentage of RHA and POFA

Figure 4 and Table 3 show the XRD analysis of the sintered samples containing RHA and POFA. The major phases identified, are quartz (ICDD 046-1045), mullite (ICDD 074-4143) and cristobalite (ICDD 082-0512). The quartz decreases with increase in temperature. While the mullite and cristobalite increases between the soaking time of 1 hour and 2 hours. Between the soaking time 2 hours and 3 hours both mullite and cristobalite decreases.



Figure 4: The XRD curves of the samples containing 20 wt% of RHA and POFA

 
 Table 3: XRD quantitative analysis of the samples containing RHA and POFA sintered at different soaking times

Soaking (Hour)	Time	Quartz (%)	Mullite (%)	Cristobalite (%)	Glassy Phase (%)
1		60.7	12.8	9.7	16.8
2		57.0	25.9	12.7	4.4
3		43.2	24.0	6.5	26.3

#### Conclusion

The compressive strength of the porcelain body was found to increase with increase in soaking time and also with the substitution of quartz by RHA and POFA. The maximum compressive strength for porcelain samples containing RHA and POFA occurred at a soaking time of 2 hours with on 20 wt% substitution of quartz with RHA and POFA. The soaking time of 2 hours exhibits highest compaction and minimum porosity. The increase in the soaking time and the substantial decrease in porosity of the mixes containing RHA and POFA, are attributed to the glassy formation and densification of the individual grains during the vitrification process.

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