

## Elemental Analysis of Rice Husk Ash Using Proton-Induced X-Ray Emission (Pixe) Spectrometry

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### Abstract

The high silica content of rice husk ash (RHA), obtained by burning rice husks, has stimulated several research investigations on its applicability in various industrial products. In this study, the elemental composition of RHA was determined using the high-tech and highly sensitive proton-induced X-ray emission (PIXE) spectrometric technique. Milled rice husks, obtained from a rice mill in Abakaliki, Nigeria were first washed to remove contaminants, then dried and subjected to controlled incineration in a muffle furnace at a temperature of 600<sup>0</sup>C and time in the range of 2-6hrs to obtain 'white' RHA samples. Elemental analysis of the ash samples was carried out using the PIXE technique. The results of the PIXE analysis revealed the presence of a total of fourteen elements, present as oxides, in the RHA samples. The oxides detected and their concentrations (in %) at 2, 4 and 6 hrs combustion time, respectively are as follows: SiO<sub>2</sub> (71.017, 74.194, 85.314), Al<sub>2</sub>O<sub>3</sub> (0.045, 0.057, 0.070), Fe<sub>2</sub>O<sub>3</sub> (0.116, 0.119, 0.204), CaO (0.136, 0.133, 0.145), MgO (0.543, 0.642, 0.724), Na<sub>2</sub>O (0.043, 0.095, 0.121), K<sub>2</sub>O (0.230, 0.253, 0.275), P<sub>2</sub>O<sub>5</sub> (2.562, 2.609, 2.796), SO<sub>3</sub> (0.081, 0.128, 0.139), ClO<sub>3</sub> (0.052, 0.066, 0.123), TiO<sub>2</sub> (0.009, 0.011, 0.013), MnO (0.039, 0.043, 0.040) ZnO (0.002, 0.002, 0.003) and Rb<sub>2</sub>O (0.003, 0.004, 0.005)

**Keywords:** PIXE spectrometry, rice husk ash, combustion, elemental oxides, analysis

### 1. INTRODUCTION

The increased global sensitivity to environment protection has triggered a massive, research-driven transition, from non-renewable (fossil-based) resources to renewable (plant-based) resources for the development of more environment-friendly fuels and

chemicals [1-3]. Rice husks (or hulls), the hard, outer protective sheath of the rice grain are generated in large quantities as agro-waste in the rice-producing areas of the world during the milling of rough rice (paddy) to obtain white rice. Thus, rice husks present a cheap, abundant, renewable source of rice husk ash (RHA), which has been found useful in many products. The quantity of ash obtained by burning rice husks is between 19-24% of the weight of husk [4, 5]. RHA has a high silica ( $\text{SiO}_2$ ) content in the range of 70-97% [6-8] and also contains several other elements (as oxides) in small quantities. The high silica content makes RHA an attractive material to gresearchers, many of whom have explored its industrial applicability. The findings from such research investigations have shown RHA to be suitable for several applications such as in coatings formulations [9-12], as filler in adhesives [13, 14] and in concrete mixes [15, 16]. This evidently presents RHA as an important industrial resource that can be used as substitute for synthetic and natural silica (quartz).

The chemical composition of rice husks varies as it is dependent on several factors and since rice husk ash is obtained from the husks, the composition of the ash is also affected by these same factors which are soil chemistry, climatic conditions, paddy variety [17, 18], use of fertilizer, type of fertilizer [19, 20], year of harvest, sample preparation and methods of analyses [21]. The elemental composition of RHA samples from different locations is presented in Table 1 in which the variation in values is evident even with samples from the same country.

**Table 1:** Elemental Composition of Rice Husk Ash from Different Countries

Element (as oxide)	Concentrations (in %) of Elements in RHA from Different locations					
	Brazil [22]	Indonesia [23]	India [24]	Nigeria [25]	Nigeria [25]	Nigeria [26]
Si as $\text{SiO}_2$	94.4	89.08	80-95	98.90	94.43	97.46
Al as $\text{Al}_2\text{O}_3$	0.61	1.75	1.0-2.5	*	*	*
Fe as $\text{Fe}_2\text{O}_3$	0.03	0.78	0.5	0.16	1.083	0.46
Ca as CaO	0.83	1.29	1.0-2.0	0.31	0.90	0.029
Mg as MgO	1.21	1.17	0.5-2.0	*	*	0.260
Na as $\text{Na}_2\text{O}$	0.77	0.85	0.2-0.5	*	*	*
K as $\text{K}_2\text{O}$	1.06	1.38	0.2	0.55	1.90	1.33
P as $\text{P}_2\text{O}_5$	*	0.61		*	*	*
S as $\text{SO}_3$	*	*		*	*	*
Cl as $\text{ClO}_3$	*	*		*	1.175	*
Ti as $\text{TiO}_2$	*	0.00	0.00	*	*	*
Mn as MnO	0.59	0.14	*	0.04	0.17	0.004
Zn as ZnO	*	*	*	0.06	0.03	0.003

Note \*Not reported

The elemental composition of RHA has earlier been determined by researchers using different analytical techniques, which include X-ray Fluorescence (XRF), one of the most commonly used techniques found in literature [25, 27]. However, XRF has some

inherent design limitations which have reduced its sensitivity to some elements [28]. The XRF technique is known to be unable to detect elements with atomic weights lower than that of sodium [25]. Other techniques that have been used for RHA compositional analysis are Atomic Absorption Spectrometry (AAS) [29, 26] and thermogravimetric methods [29, 30]. An extensive literature search revealed that there has been no reported work on the use of the high-tech, highly sensitive and relatively uncommon, Proton-induced X-ray Emission (PIXE) spectrometry for the compositional determination of RHA, therefore the PIXE technique introduces novelty to the elemental analysis of rice husk ash.

PIXE, also known as Particle-induced X-ray Emission (PIXE) spectrometry is based on the energy spectra of characteristic X-rays emitted by the de-excitation of the atoms in the sample bombarded with high energy protons (1-3 MeV) with the aid of a suitable energy dispersive detector [31]. The use of proton beams as excitation source offers several advantages over other X-ray techniques, the major ones being its high sensitivity for trace element determination and higher rate of data accumulation across the entire spectrum, which allows for faster analysis [32]. Its greater sensitivity, is expected to give more accurate results than other techniques previously used [25, 26, 29].

Research findings [33] have revealed that there are two distinct stages in the burning of rice husks to produce rice husk ash-carbonization and decarbonization. Carbonization is the first stage during which decomposition of volatile matter in rice husks occurs at a temperature above 300<sup>0</sup>, releasing combustible gas and tar to yield rice husk char, which is black in colour. Decarbonization involves the combustion of fixed carbon in the presence of oxygen to yield rice husk ash. The conversion of raw (untreated) rice husks to rice husk ash (RHA) by incineration is accompanied by colour changes, which include brown, grey, ivory, milky-white [34]. These colour changes are associated with the completeness of the combustion process as well as the structural transformation of silica in the ash from the amorphous to the crystalline state which is dependent on temperature and duration of incineration [33, 35]. White RHA has been found most suitable for most studies since its whiteness is indicative of complete oxidation of the carbon in rice husk char and thus, availability of a large portion of silica in the ash. In several research investigations, the ash was obtained at combustion temperatures in the range of 500 to 800<sup>0</sup>C which produces amorphous silica [27, 36, 37] while a temperature greater than 800<sup>0</sup>C, gives crystalline silica. The transition temperature from amorphous to crystalline silica has been reported to be about 800<sup>0</sup>C [38]. Asavapisit and Ruengrit [39] reported that 650<sup>0</sup>C is the optimum temperature for producing reactive (amorphous) RHA while Oyawale [26] reported 700<sup>0</sup>C as the optimum temperature for obtaining the maximum amount of silica. An earlier study by Igwebike-Ossi [34] also showed that white ash is obtainable at 500-800<sup>0</sup>C and that the 'whiteness' of the ash improved with increased combustion time of 5-6hrs.

The objective of this work was to determine the elemental composition of RHA obtained at 600<sup>0</sup>C using the PIXE technique which has not been reported in literature; compare the results obtained by the PIXE technique with those obtained by other analytical techniques; examine the effect of combustion duration on the

concentrations of the elements by controlled heating of the rice husks for 2, 4 and 6 hrs at the specified temperature.

## **MATERIALS AND METHODS**

### **Equipment**

- i. Muffle Furnace ((Labline);
- ii. Laboratory electric oven;
- iii. Hydraulic Press
- iv. 1.7 MV Tandem Pelletron Accelerator, model 5SDH, built by the National Electrostatics Corporation (NEC), and acquired by the Centre for Energy Research and Development (CERD), Obafemi Awolowo university, Ile-Ife, Nigeria
- v. End station for Ion beam analysis (IBA) designed and built by the Materials Research Group (MRG) at Themba Labs, Sommerset West, South Africa
- vi. Canberra Si(Li) PIXE detector with 30mm<sup>2</sup> active area, Model ESLX30-150

## **METHODS**

### **Washing and Drying of Rice Husks**

Milled rice husks obtained from a rice mill located in Abakaliki, in Ebonyi State of Nigeria were washed several times to remove sand and stone contaminants. The washed rice husks were then spread on plastic trays and other extraneous materials like broken rice grains were removed by handpicking. The wet rice husks were dried at 100<sup>0</sup>C to a constant weight in an electric oven and then stored in a dessicator.

### **Combustion of Rice Husks in a Muffle Furnace**

The washed and dried milled rice husks (2.00g) were weighed into three different medium-size porcelain crucibles, which were then placed inside the muffle furnace without the covers. The furnace was switched on and left to attain the desired combustion temperature of 600<sup>0</sup>C. Once this temperature was attained, it was maintained until the required combustion duration (2, 4 or 6hrs) for the ash sample was exhausted. The ash samples were then withdrawn from the furnace, allowed to cool and stored in plastic containers.

### **Elemental Analysis of RHA using PIXE Technique**

#### **Sample preparation**

The RHA samples were ground in a porcelain mortar to reduce the particle size and ensure a uniform distribution of the particles. The ground ash samples were then pelletised using a hydraulic press before subjecting them to ion beam analysis (IBA) using the PIXE technique.

The ash samples obtained after combustion of rice husks at 600<sup>0</sup>C and 2, 4 and 6hrs combustion time, respectively, were coded as follows:

Combustion Temperature and Time of rice husks	Code
600 <sup>0</sup> C for 2hrs	RHA-600-2
600 <sup>0</sup> C for 4hrs	RHA-600-4
600 <sup>0</sup> C for 6hrs	RHA-600-6

### Conversion of Concentration of Elements in parts per million (ppm) to their Oxides Expressed in Percentage (%)

The PIXE analysis gives the concentration of the elements in parts per million (ppm). However, since the analyzed samples are ash samples, the elements are in the form of oxides in the ash after combustion. To convert the concentration of the elements expressed in ppm to their oxides (also in ppm), the former is divided by a conversion factor, which is obtained from a ratio of the element to its oxide. This is followed by the conversion of the oxide concentration values from ppm to percentage (%). The calculations were carried out as follows:

$$\text{Oxide of element (in ppm)} = \frac{\text{Conc. of element in ppm}}{\text{Conversion factor}}$$

To convert from ppm to percentage:

$$1 \text{ ppm} = \frac{1}{1,000,000}$$

$$\text{In \%} = \frac{1}{1,000,000} \times 100 = \frac{1}{10,000}$$

$$\text{To convert } X \text{ ppm to } X\% = \frac{X}{10,000}$$

For example to convert the value of Mg expressed in ppm (e.g 4164.5ppm) to MgO expressed in percentage concentration (% conc):

$$\begin{aligned} \text{MgO\% Conc} &= \frac{4164.5 \text{ ppm}}{0.6031} \times \frac{1}{10,000} \\ &= \frac{6905.16 \text{ ppm}}{10,000} = 0.6905\% \text{ of MgO} \end{aligned}$$

### Calculation of Conversion Factors

The conversion factors are calculated from the ratios of the atomic weights of the pure elements to their oxides. For example for SiO<sub>2</sub>, the conversion factor is calculated as follows:

$$\text{Si} = 28.086 \quad \text{O} = 15.999$$

$$\text{SiO}_2 = 28.086 + (15.99 \times 2) = 60.084$$

$$\text{Ratio of Si in SiO}_2 = \frac{28.86}{60.08} = 0.4674 \quad (\text{conversion factor for Si to SiO}_2)$$

## RESULTS AND DISCUSSION

'White' ash samples were obtained after controlled burning of rice husks at the combustion temperature of 600<sup>0</sup>C. Previous research findings have shown that white ash is obtainable from rice husks at combustion temperatures in the range of 500-900<sup>0</sup>C [34]. Thus, the temperature of 600<sup>0</sup>C selected for this study was based on energy cost considerations coupled with findings from related studies which reported 650<sup>0</sup>C [39] and 700<sup>0</sup>C [26] as the optimum temperature for obtaining the highest amount of reactive (amorphous) silica. These findings suggest that at temperatures in the neighbourhood of 600<sup>0</sup>C, the husk has been largely converted to white amorphous silica which most researchers have found suitable for various industrial applications [6-13]. Although all the RHA samples (amorphous silica) obtained at 600<sup>0</sup>C at the specified time regimen were 'white,' the ash samples obtained at 6hrs combustion time had the brightest degree of 'whiteness' which was a milky-white colour, indicating that longer combustion time improves the whiteness of amorphous silica obtained.

A total of fourteen elements (as oxides) were detected using PIXE analytical technique. The elemental oxides composition of RHA at a combustion temperature of 600<sup>0</sup>C and times of 2, 4 and 6hrs respectively are given in Table 2.

**Table 2.** Elemental oxides composition of RHA at 600<sup>0</sup>C and 2, 4 and 6hrs Combustion times

Elemental Oxide	Concentration (in %) of Elemental oxides at 2, 4, 6hrs combustion times respectively		
	RHA-600-2	RHA-600-4	RHA-600-6
Si as SiO <sub>2</sub>	71.017±0.085	74.194±0.09	85.314 ± 0.09
Al as Al <sub>2</sub> O <sub>3</sub>	0.045±0.004	0.057±0.004	0.070± 0.005
Fe as Fe <sub>2</sub> O <sub>3</sub>	0.116±0.003	0.119±0.003	0.204±0.003
Ca as CaO	0.136±0.003	0.133±0.003	0.145±0.003
Mg as MgO	0.543±0.004	0.642±0.004	0.724± 0.041
Na as Na <sub>2</sub> O	0.043±0.003	0.095±0.027	0.121±0.026
K as K <sub>2</sub> O	0.230±0.005	0.253±0.253	0.275±0.006
P as P <sub>2</sub> O <sub>5</sub>	2.562±0.009	2.609±0.094	2.796±0.107
S as SO <sub>3</sub>	0.081±0.024	0.128±0.027	0.139±0.023
Cl as ClO <sub>3</sub>	0.052±0.011	0.066±0.013	0.123±0.016
Ti as TiO <sub>2</sub>	0.009±0.011	0.011±0.002	0.013±0.001
Mn as MnO	0.039±0.002	0.043±0.002	0.040±0.002
Zn as ZnO	0.002±0.000	0.002±0.001	0.003±0.001
Rb as Rb <sub>2</sub> O	0.003±0.002	0.004±0.001	0.005±0.002

Previous studies [8, 21-23] reported the presence of a preponderance (70-90%) of silica (SiO<sub>2</sub>) with small quantities of other metallic and non-metallic oxides. The oxides that have been frequently reported to be present in RHA using other analytical techniques include Fe<sub>2</sub>O<sub>3</sub>, CaO, MgO, K<sub>2</sub>O and MnO which were also detected by the

PIXE technique. The PIXE values obtained for MgO (0.543-0.724) and MnO (0.039-0.043) were comparable to those quoted in literature [22-26]. However the PIXE values obtained for CaO (0.136-0.145), Na<sub>2</sub>O (0.043-0.121) and K<sub>2</sub>O (0.230-0.275) were generally lower than literature values [22-26] obtained by other analytical techniques. The PIXE values of Fe<sub>2</sub>O<sub>3</sub> (0.12-0.20) were comparable to a few literature values [25], but differed widely with others [22-24] due to the wide variation in the concentrations of Fe<sub>2</sub>O<sub>3</sub> found in literature [22-26]. The oxide not commonly found in RHA but which was detected by the PIXE technique in trace quantities is TiO<sub>2</sub> while Rb<sub>2</sub>O has previously not been reported to be found in RHA. These variations in concentrations of the elements can be attributed to factors such as paddy species, type of climate, type of soil, use of fertilizers etc as earlier mentioned [17-21]. The detection of TiO<sub>2</sub> and Rb<sub>2</sub>O, present in trace quantities is most probably due to the high sensitivity of the PIXE technique [32].

### **Effect of Combustion Time on RHA Composition**

There was a general increase in the concentration of silica, the major component of RHA as well as the other oxides, with increase in combustion time from 2 to 6hrs. This shows that increased combustion time enhances carbon conversion efficiency and oxidation during the decarbonisation stage to give higher yields of silica. It also implies that the decarbonisation process requires some time for the conversion of rice husk char to rice husk ash to be completed. The silica content values of 71%, 77% and 85%, obtained at 2, 4 and 6hrs combustion time, respectively are comparable to values quoted in literature [8, 22-26] and corroborate the finding that prolonged combustion time generally increases the concentration of silica at all the combustion temperatures [34] and also improves the whiteness of the ash which is an indication of increased level of amorphous silica.

### **CONCLUSION**

The PIXE analytical technique detected a total of fourteen elements in RHA present in the form of oxides. These include Titanium dioxide (TiO<sub>2</sub>), which had been rarely detected in RHA while Rb<sub>2</sub>O has not been previously reported in literature using other analytical techniques like XRF and AAS. This may be attributed to the high sensitivity of PIXE spectrometric technique to trace elements. 'White' RHA (amorphous silica) was obtained at 600<sup>0</sup>C but the ash obtained at 6hrs combustion time had the brightest whiteness of the three samples. The concentration of silica (SiO<sub>2</sub>), the predominant component of RHA was found to increase markedly with increase in combustion time. The levels of the other oxides present in minute quantities were also generally found to increase slightly with increased combustion time. These findings indicate the importance of combustion duration in the efficiency of the decarbonisation stage during which the carbonised rice husk (rice husk char) is converted to rice husk ash.

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