

Potassium Oxide Analysis in Rice Husk Ash at Various Combustion Conditions using Proton-Induced X-ray Emission (PIXE) Spectrometric Technique

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Abstract

Rice husk ash (RHA) has been found to be a useful bio resource for various industrial applications due to its high silica content. Although, RHA is composed predominantly of silica (SiO_2), it also contains small quantities of metallic oxides with potassium oxide (K_2O) being among the highest in concentration. The presence of potassium is of significant importance in rice husks because it has been found to be responsible for the formation of grey-coloured ash instead of white, particularly at high combustion temperatures. This study investigated the effect of various combustion temperatures and times on the potassium oxide concentration of RHA and also examined the effects of heating rice husks at 900°C and 1000°C , at high and low heating rates, on the colour of the ash. Rice husks obtained from a rice mill in Abakaliki, Nigeria were first washed to remove contaminants, dried in an oven, then subjected to controlled combustion in a muffle furnace at varied temperatures in the range of $400\text{-}1000^\circ\text{C}$ and duration of 1-6hrs. The concentrations of potassium oxide at the various combustion conditions were analysed using the high-tech and highly sensitive Proton-induced X-ray emission (PIXE) spectrometric technique. The results of the PIXE analysis showed that highest K_2O levels (0.2-0.37%) were obtained at $400\text{-}800^\circ\text{C}$ while the lowest values (0.06-0.20) were obtained at $900\text{-}1000^\circ\text{C}$. The combustion duration had little or no effect on the K_2O concentration of the ash as the values obtained at 1hr combustion time were close to those obtained after six hours. The ash samples obtained at 900 and 1000°C at low heating rates of 15.52°C and $9.8^\circ\text{C}/\text{min}$ respectively, were milky-white in colour while those obtained at high heating rates of 32°C and $27^\circ\text{C}/\text{min}$ respectively were greyish.

Key words: potassium oxide, PIXE spectrometry, analysis, rice husk ash, combustion

1. INTRODUCTION

Rice husks generated as agro-waste product during the rice-milling process has been a material of interest to researchers due to its abundance, little or no cost and 'renewability'. Rice husk ash (RHA) obtained by burning rice husks has a high silica content in the range of 67-97% [1-3]. This high level of silica in RHA has stimulated several research investigations on its use as an alternative to natural and synthetic silica in various industrial applications which include coatings formulations [4-7], adhesives [8-9] and concrete mixes [10-11]. Although predominantly silica (SiO_2), in composition, RHA also contains small quantities of oxides with potassium oxide (K_2O) being among the highest of the elemental oxides in concentration.

The chemical composition of rice husks varies as it is dependent on several factors and since RHA is obtained from the husks, the composition of the ash and thus concentration of K_2O in the ash is affected by these same factors which are soil chemistry climatic conditions, paddy variety [12, 13], use of fertilizer, type of fertilizer [14, 15], year of harvest, sample preparation and methods of analyses [16]. The levels of potassium oxide in RHA have been found to vary widely from location to location due to the said factors. The values of K_2O from different countries that have been quoted in literature include Brazil, 1.06 [17], Indonesia 1.38 [18], India, 0.20 [19], Nigeria, 0.55, 1.90, [20], 1.33 [21] and 0.23-0.28 [22]

'White' RHA is desirable and has been used for most research investigations [4-9], however the presence of potassium in rice husks has been found to affect the colour of rice husk ash obtained at high combustion temperatures if the rice husks is untreated. Muthadhi [23] reported that at high temperatures, there's strong interaction between potassium and silica ions which causes formation of potassium silicate combined with carbon, resulting in grey-coloured ash. If however, the husk is pretreated with acid, a major portion of the potassium will be removed and the ash will not attain grey colour [12]. In furtherance of this postulate on the chemical interaction between potassium and silica, Muthadhi explained [23] that low-melting oxides (like K_2O) fuse with silica on the surface of the rice husk char and form glassy or amorphous phases, preventing the conversion of carbon. Singh et al, [24] in a related study, observed that there was decreasing concentration of potassium with increasing temperature and suggested that K_2O could be present in some form of hydrated salt which sublimates with increasing temperature resulting in the observed drop in K_2O level.

Another researcher [25] linked the formation of grey ash at high temperatures to high heating rates with the explanation that the potassium in rice husks does not volatilize, at high heating rates, but reacts with silica, forming potassium silicate combined with carbon. Therefore, rapid burning of rice husks causes high residual carbon in the ash. Igwebike-Ossi [26], in another study, also reported the formation of grey ash at 900 and 1000^oC at high heating rates. Similarly, Krishnarao et al [27] observed that the tendency to form black particles (unburnt carbon) increased with increase in the heating rate and the temperature of calcination of untreated rice husks. Chandresekhar and co-researchers [28], based on their findings from the investigation of the effects of heating rate on the properties of rice husk ash, concluded that a high potassium content in the husk inhibited the carbon removal during ashing which affected the colour as well as reactivity of the ash.

Different analytical techniques have been used to determine the potassium oxide composition of RHA. X-ray Fluorescence (XRF) spectrometry has been one of the most commonly used techniques for this purpose [20, 29]. However, XRF has some inherent design limitations which has reduced its sensitivity to some elements [30]. The XRF technique is known to be unable to detect elements with atomic weights lower than that of sodium [20]. Other techniques that have been used for compositional analysis of RHA are Atomic Absorption Spectrometry (AAS) [21, 31] and thermogravimetric methods [17, 31]. The use of the high-tech, uncommon and highly sensitive Proton-induced X-ray Emission (PIXE) spectrometric technique for the analysis of potassium oxide in RHA has introduced novelty to this investigation. 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 [32]. 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 [33]. The PIXE technique is expected to give more accurate results than other techniques previously used [17, 20, 29, 31] due to its greater sensitivity.

The objective of this study was to determine the K_2O concentration in RHA using the high-tech PIXE spectrometric technique; determine the effects of combustion temperature and duration on the concentration of potassium oxide on RHA by varying the temperatures from 400 to 1000⁰C and time from 1 to 6hrs for each temperature under investigation; determine the colour of the ash obtained at high combustion temperatures of 900 and 1000⁰C at slow and high heating rates and consequently establish the relationship between the potassium oxide concentration and ash colour at high temperatures. The systematically varied combustion temperature, combustion time of rice husks will reveal how the incineration conditions affect the concentration of K_2O in the ash.

2. 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 30mm² 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°C to a constant weight in an electric oven and then stored in a dessicator.

Combustion of Rice Husks in a Muffle Furnace

Exactly 2.00g of the washed and dried rice husks were weighed into three different medium-size porcelain crucibles, which were then placed inside the muffle furnace without the covers. The furnace was then switched on, set to the required heating rate and left to attain the desired combustion temperature. Once this temperature was attained, it was maintained until the required combustion duration for the ash sample was exhausted. The ash samples were then withdrawn from the furnace, allowed to cool in a dessicator. The heating rates were calculated using the expression:

$$\text{Heating rate (}^{\circ}\text{C/min)} = \frac{\text{Temperature attained (}^{\circ}\text{C)}}{\text{Time taken (min)}}$$

Determination of the Potassium Oxide Concentrations in RHA using PIXE Spectrometry

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 using the PIXE technique.

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 potassium expressed in ppm (e.g 2100.6ppm) to potassium oxide (K₂O) expressed in percentage concentration (% conc):

$$\text{K}_2\text{O \% Conc} = \frac{2100.6 \text{ ppm}}{0.6031} \times \frac{1}{10,000}$$

$$= \frac{3483.00 \text{ ppm}}{10,000} = 0.3483\% \text{ of K}_2\text{O}$$

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 K₂O, the conversion factor is calculated as follows:

$$\text{K} = 39.098 \quad \text{O} = 15.999$$

$$\text{K}_2\text{O} = [(39.098 \times 2) + 15.99] = 94.186$$

$$\text{Ratio of K in K}_2\text{O} = \frac{39.098}{94.186} = 0.4151 \text{ (conversion factor for K to K}_2\text{O)}$$

3. RESULTS AND DISCUSSION

Potassium Oxide Concentration of RHA at Different Combustion Conditions

The potassium oxide concentrations in RHA samples obtained at different combustion temperatures and times are given in Table 1 while plots of K₂O concentration as a function of temperature and time are shown in Figs. 1 and 2, respectively.

Table 1: Potassium oxide Concentrations (%) of RHA at Different Combustion Temperatures and Duration of 1-6hrs

Temp °C	Potassium oxide (K ₂ O) concentration of RHA at 1 to 6hr Combustion times					
	1	2	3	4	5	6
400	0.141±0.005	0.155±0.005	0.295±0.006	0.355±0.0054	0.265±0.004	0.156±0.005
500	0.270±0.0054	0.285±0.0048	0.253±0.0054	0.271±0.006	0.287±0.006	0.304±0.0057
600	0.286±0.005	0.230±0.005	0.210±0.004	0.253±0.005	0.242±0.005	0.275±0.006
700	0.124±0.004	0.266±0.005	0.266±0.006	0.297±0.0053	0.284±0.002	0.300±0.006
800	0.293±0.006	0.133±0.004	0.259±0.006	0.238±0.005	0.369±0.006	0.241±0.005
900	0.137±0.004	0.250±0.005	0.220±0.005	0.223±0.005	0.257.006±	0.109±0.006
1000	0.175±0.005	0.064±0.004	0.076±0.004	0.222±1.105	0.083±0.004	0.092±0.004

The concentrations of potassium oxide in the RHA sample obtained at the various combustion conditions were found to be in the range of 0.06 to 0.37% as is evident in Table 1. These values are relatively low compared to values quoted in literature [17, 18, 20, 21] in which values as high as 1.3-1.9 have been reported. This could be due

to any one or combination of the several factors that affect the chemical composition of rice husk ash such as soil chemistry climatic conditions, paddy variety [12, 13], use of fertilizer, type of fertilizer [14,15] year of harvest, sample preparation and methods of analyses [16].

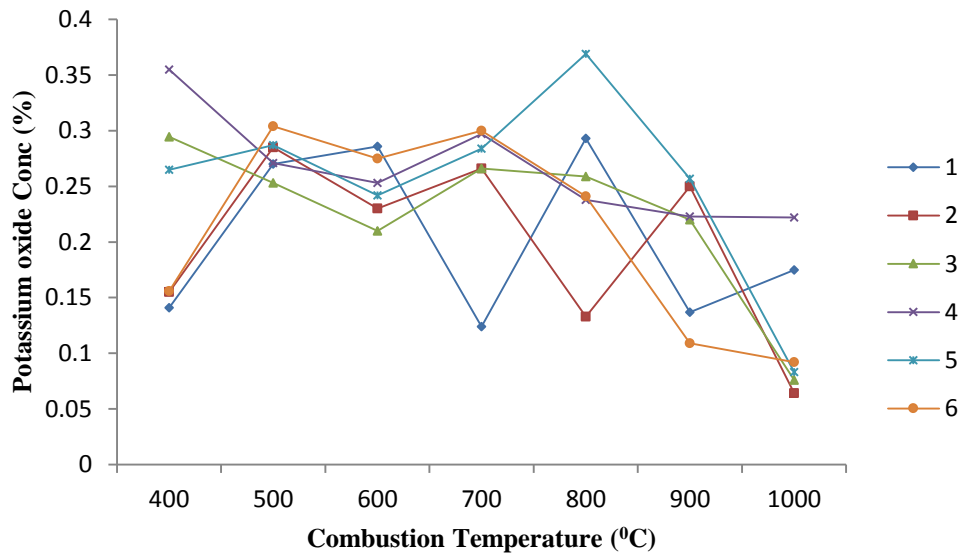


Figure 1: Plots of Potassium Oxide (K₂O) Concentration in RHA Versus Combustion Temperature at Combustion Times of 1 to 6hrs

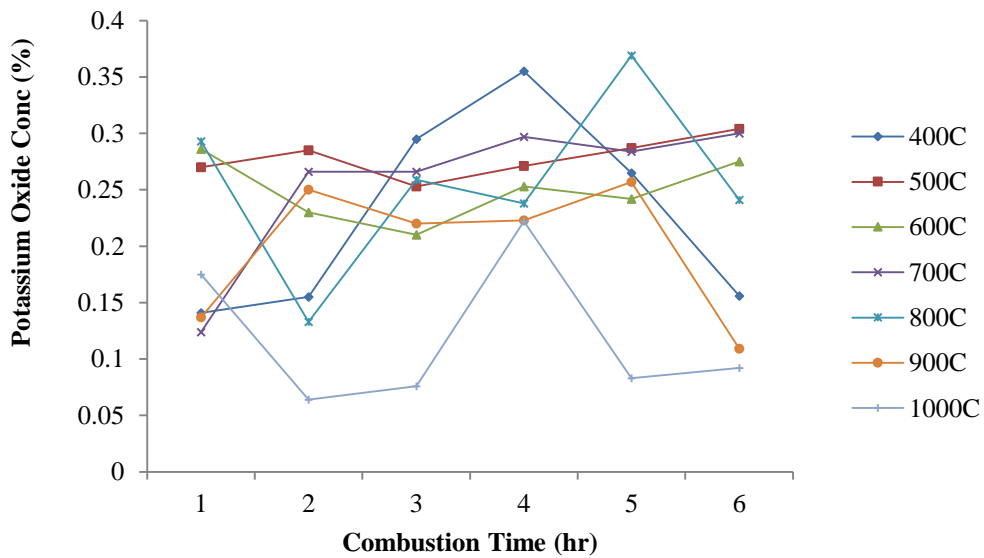


Figure 2: Plots of Potassium Oxide (K₂O) Concentration in RHA Versus Combustion Time at Various Combustion Temperatures

Plots of K_2O concentration versus temperature shown in Fig.1 reveal that the highest concentrations of K_2O (0.2-0.37%) were obtained at temperatures below $900^{\circ}C$ ($400-800^{\circ}C$), while the lowest values (0.06-0.22 %) were obtained at $900-1000^{\circ}C$. Thus, there was a slight drop in K_2O concentration from lower to higher temperatures. This is in agreement with the findings of Singh [24] who reported a drop in concentration of potassium in RHA with increasing calcination temperature. At $400-1000^{\circ}C$, interaction of potassium and silica in rice husks is likely to have occurred resulting in the probable formation of potassium polysilicate [23]. However, this interaction is probably greater at $900-1000^{\circ}C$, hence the lowest values of K_2O obtained at these temperatures. The presence of K_2O , which has a melting point of $350^{\circ}C$ [34] at all the combustion temperatures ($400-1000^{\circ}C$), suggests that it is in a form that makes it inaccessible for melting and volatilization. This therefore substantiates the likely formation of potassium silicate since at these temperatures, K_2O in RHA is expected to have volatilized and if so, will not be detected in the ash. This indicates that it did not sublime but may have fused with silica on the surface of the rice husk char [23].

Plots of K_2O concentration versus combustion time as shown in Fig. 2 reveal that the combustion duration had little effect on the concentration of potassium oxide incinerated at the different combustion temperatures. For instance the values in the range of 0.14-0.35 obtained at 1hr duration are quite close to values of 0.15-0.30 obtained after 6hrs. This implies that combustion temperature has a more pronounced effect on K_2O concentration than combustion duration and suggests that the formation of K_2O takes place rapidly, probably within the first hour, so that prolonging the period of incineration has little effect on the K_2O concentration.

Effects of High and Low Heating Rates on RHA Colour

The effects of high and low heating rates on the colour of rice husk ash are shown in Table 3.

It is evident from the results that high heating rates resulted in grey-coloured ash at all the combustion hours (1-6hr) while a low heating rate produced milky-white ash at the various incineration times. The production of grey ash at 900 and $1000^{\circ}C$ at high heating rates (32 and $27^{\circ}C/min$ respectively) corroborates the findings of Maeda [25] from which he postulated that the potassium in rice husks does not volatilize, at high heating rates, but reacts with silica, forming potassium silicate combined with carbon. This residual carbon is responsible for the grey colour of the ash. The formation of grey ash suggests that at high heating rates, the carbon conversion during the decarbonization process takes place with high inefficiency resulting in residual carbon which makes the ash greyish in colour. The inefficiency in carbon conversion occurs because at high heating rates, the temperature increases so rapidly that the heat does not uniformly spread out through the rice husk char during the transformation of the char (carbon) into RHA. Thus, portions of unburnt carbon or partially converted carbon remain in the ash giving it a greyish colour.

Table 3: Heating Rates, and Colours of RHA obtained at Combustion Temperatures of 900 and 1000⁰C and Time of 1 to 6hrs

Comb. Temp (⁰ C) and Colour	Heating Rate (⁰ C/min)	RHA Colour at 900 ⁰ C and 1000 ⁰ C for 1 to 6hrs incineration time at high and low heating rates					
		1	2	3	4	5	6
HIGH HEATING RATE							
900 Colour	32	Dark Grey	Grey	Light Grey	Light Grey	Light Grey	Light Grey
1000 Colour	27	Grey	Grey	Grey	Grey	Grey	Grey
LOW HEATING RATE							
900 Colour	15.52	Milky white	Milky white	Milky white	Milky white	Milky white	Milky white
1000 Colour.	9.8	Milky white	Milky white	Milky white	Milky white	Milky white	Milky white

Also, the formation of grey ash right from the first hour to the sixth hour of combustion suggests that the reaction between potassium and silica to form potassium polysilicate with the accompanying residual carbon occurs rapidly at the first hour and extension of the incineration time does not improve the efficiency of the carbon conversion (decarbonization) process that would yield white ash. This implies that once the grey ash is formed at the first hour due to high heating rate, it remains as such, so prolonging the combustion duration is ineffective in whitening the colour of the ash. Combustion temperature is therefore the dominant factor in the formation of grey ash at high heating rates.

The formation of milky-white ash at 900 and 1000⁰C at low heating rates (15.52 & 9.8⁰C/min respectively) and at all the combustion hours has proven that contrary to the report of Muthadhi [23], 'white' ash is obtainable at high temperatures but at low heating rates. A probable explanation is that the strong interaction between potassium and silica ions takes place with high carbon conversion (decarbonization) efficiency due to the slow heating rate such that there are no remnants of carbon in the ash produced. The slow rise in temperature causes an even spread of heat within the char which results in a uniform and efficient transformation of the char (carbon) to ash. The resultant effect is the formation of 'white' ash without any residual carbon that would have imparted a greyish colour to the ash.

CONCLUSION

The combustion conditions had an effect on the potassium oxide concentrations in RHA such that higher values of K₂O (0.2-0.37%) were obtained at relatively lower temperatures (400-800⁰C) while the lowest K₂O values were obtained at 900-1000⁰C. The drop in the level of potassium oxide at 1000⁰C silica suggests the formation of

potassium polysilicate (K_2SiO_3) with residual carbon which reduces the available K_2O detectable by the PIXE technique. The combustion duration had little or no effect on the K_2O content. The heating rate had an effect on both the concentration and colour of K_2O at 900 and 1000⁰C. High heating rates resulted in grey-coloured ash at all the combustion hours (1-6hr) while low heating rates produced milky-white ash at all the incineration hours. The formation of white or grey ash at 900-1000⁰C is therefore dependent on heating rates.

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