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Characterization of Rice Husk via Atomic Absorption Spectrophotometer for Optimal Silica Production

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ABSTRACT

Rice husk is one of the main agricultural wastes in milling processes that is abundantly available in Nigeria. The aim of this research work is characterization of local rice husk and its objective is determination of its chemical constituents using Atomic Absorption Spectrophotometer. Rice husk from two methods were subjected to calcination for temperature range of 50-750^{0}C to determine its characterisation temperature. Characterisation temperature was determined via the temperature at which the highest Specific Surface Area (SSA) and highest amount of silica were observed which was 700^{0}C. Parameters characterized are:Na_2O, K_2O,Fe_2O_3,MnO,CaO,LOI and Specific Surface Area. The results showed that the optimal temperature and maximum amount of silica was 700^{0}C with highest specific surface area. It was also noted that at 750^{0}C, there was decrease in the amount of silica obtained. It was concluded that optimal silica was produced at characterisation temperature of 700^{0}C. This will also reduce the environmental pollution caused by opening burning of rice husk

Keywords: *Rice Husk Ash, AAS, Specific Surface Area, Silica*

1. INTRODUCTION

Rice husk is an agro-waste which is produced in about 100 million of tons. About 10^8 tons of rice husks are generated annually in the world. In Nigeria about 2.0 million tons of rice is produced annually (Oyetola and Abdullahi, 2006). Approximately, 20kg of rice husk are obtained from 100kg of rice. It burning generates rice husk ash which is rich in silica and can be an economically valuable raw material for production of natural silica (Kalapathy et al. 2000). Industrial importance of rice husk (RH) is due to the presence of silica in hydrated amorphous form. The rice husk contains 80 per cent organic volatile materials and remaining 20 per cent silica (James and Subbarao, 1996). The analysis of raw rice husk obtained from Erin-Oke (Nigeria) is shown in table 1.0.The chemical composition of the rice husk ash varies from Rice husk to Rice husk which may be due to geographical and climatic conditions, type of rice and the quantity of fertilizer used (Govindarao, 1980). In addition to protecting rice during the growing season, rice hulls can be put to use as building material and also be used as fertilizer (wiki rice, 2007). The disposal of rice hulls is a substantial problem for rice growers, since the hulls are not suitable for use as fertilizers and until now have to be disposed of either by open burning or burying. When burnt in an uncontrolled manner, the ash which is essentially silica is converted into crystalline forms and becomes less reactive (Oyekan and Kamiyo, 2008).Burning of rice hulls also releases Carbon-monoxide to the atmosphere (which is poisonous).Space for burning is frequently not available in heavily populated areas and burning creates undesirable atmospheric pollution. However; there is high amount of biogenetic silica content in rice hulls which has great potential economic advantage for developing countries that have large quantities of rice hulls readily available and virtually free of cost. Many methods have been developed to produce silica from rice husk ash in low cost. It has been reported that purity of silica is highly affected from chemical treatment (Banerjee et al. 1982) than thermal treatment (Ikram and Akhter,1988).The silica (SiO_2) in the rice husk exists in the hydrated amorphous form like silica gel. Thermal degradation and pyrolysis of rice husk (Krishnarao et al.1991) followed by combustion of the char, percentage of unburnt carbon(Kapur,1985).Combusted at moderate temperature, the ash obtained from rice husk contains approximately 92-97% of amorphous silica(Mishra,1985) and some amount of metallic impurities that can be further removed by a simple acid leaching treatment. In this study it was reported that acid leaching helps to remove most of the metallic present and helps to obtain ash with a high specific surface area. Leaching is an extraction of certain materials from a carrier into a liquid or removes the impurities of the materials by dissolving them from the solids. The chemical process industries often used organic solvents for leaching process (Maroti et al. 2009).

2. ATOMIC ABSORPTION SPECTROPHOTOMETER

In this study atomic absorption spectrophotometer will be used to characterize the chemical constituents of rice husk ash. The potential application of atomic absorption principle is to determine metallic constituents of silicate materials. The material being determined through this technique is reduced to elemental state and vapourised. It is usually accomplished by converting solution of the sample into fine mist and then drawing it to a suitable flame .Heat of the flame excites outer most electrons to higher orbital which after short intervals of time, return to ground state and a quantum of radiation is emitted. Each element in vapour phase absorbs at a limited number of wavelengths and therefore its absorption spectrum

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consists of a series of well–defined narrow lines due to the said electronic transactions (Muhammed,1993). The analysis of raw rice husk is as shown in Table 1.

Table 1: Analysis of Raw Rice Husk

Component	Amount (%)
Moisture Content	9.38
Bulk Density	0.72
Ash	11.34
Volatile Matter	6.74
Nitrogen	1.15
Carbon	20.63
Sulphur	1.31

From the analysis, it was noted that the raw rice husk contained large amount of carbon. Heating of rice husk at higher temperatures, the unburnt carbon can be removed from the ashes (Krishnarao et al. 2001), but this leads to the crystallization of the ash from amorphous silica into crystalline or tridymite.(Haslinawati et al.2009).At lower temperature, amorphous nature of rice husk ash silica will occur.(Adil and Farook,2007)

Factors influencing the Ash Properties

It is understood that rice husk ash produced in uncontrolled conditions may not be useful for any effective applications. The factors influencing the ash properties are: Incinerating Conditions, Temperature and Duration, Rate of Heating, Geographical Location, Fineness Colour and Crop Variety.

This present work involves the following studies:

1. Characterization of raw rice husk
2. Comparative study of raw rice husk and leached rice husk
3. Characterisation temperature for highest amount of silica with highest specific surface area

3. EXPERIMENTAL PROCEDURE

Equipment

Equipment used for the processing of silica from rice husk obtained from (Erin-Oke town) Osun state in Western part of Nigeria. One was from the raw RH and the other was from the oxalic acid treated RH. They are labelled as RHA and *L*HA, respectively

Equipment used are: Muffle furnace and Digital weighing Balance.

Sample Preparation

Firstly, raw rice husk was collected from a milling town in Osun State; which was grounded, sieved and used and secondly, raw rice husk was treated with Oxalic acid (acid leaching) and then calcinated at temperature ranges from 50-750^{0}C.

4. METHODS OF PYROLYSING RICE HUSK

1. Raw Rice Husk as Collected from the Milling Was Subjected to Burning

The raw rice husk was grounded and sieved to remove particles.20g of rice husk was put inside a weighed clay pot and was placed inside the muffle furnace for burning.400-750˚C was used for burning the rice husk. Time for combustion was 6 minutes. When the temperature of the furnace reached the desired temperature, rice husk was left inside for 6 minutes to burn. The rice husk ash was then left inside the muffle to cool. The combustion was then ascertained visually by opening the muffle furnace and it was recorded.

The procedure was repeated for the temperature ranges from 400-750˚C.According to Krishnarao et al.(1992), Crystallization occurred at 800˚C during firing (burning) of samples of rice husk, hence burning at 750˚C or above can result into environmental pollution.

2. Digestion of Rice Husk in Oxalic Acid for Silica Content Analysis

According to Krishnarao et al (2005) 10g of the Washed rice husk will be added to 100ml Oxalic acid and boiled for 90 minutes with continuous stirring and it will then be left for 20 hours to cool. For this experiment 1Mole of Oxalic acid was used for the leaching of the rice husk

The supernatant liquid was decanted and then washed thoroughly with rain water for 5 times so as to remove the oxalic acid completely. It was dried and then burned to ash in a muffle furnace for 6 minutes and the samples were visualised at frequent intervals and the observations recorded.

The different temperatures considered were from 50-750°C.

5. RESULTS AND DISCUSSION

The ash samples prepared at different conditions and temperatures were examined. Standard wet chemical methods with instrumental techniques were used to determine the chemical constituents of the rice husk ash, calcinated at 50-750^{0}C.Silica was estimated gravimetrically by HF evaporation method,Na_2O and K_2O by flame photometer,$Fe_2O_3$by Colorimetric using UV-Visible Spectrophotometer and other trace elements by atomic absorption spectrophotometer. The surface area was measured by BET nitrogen adsorption method and also according to ASTM, 626(1997).

The process route with the highest Specific Surface Area is the best route as far as high purity silica is concerned and has been discussed in earlier publications (Real et al (1996) and de Souza and Baptista,(1999).

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Significant effects were noted from calcination temperature of 400^{0}C.Chemical Constituents of Raw Rice Husk Ash via Atomic Absorption Spectrophotometer Analysis at 400-750^{0}C is shown in Table 2.

Table 2: Chemical Constituents of Raw Rice Husk Ash via Atomic Absorption Spectrophotometer Analysis at 400-750^{0}C

Temp (^{0}C)	SiO_2 (%)	Fe_2O_3 (%)	Zn(%)	Mn (%)	CaO (%)	MgO (%)	Na_2O (%)	K_2O (%)	LOI (%)	SSA(m^2/g)
400	97.425	0.370	0.003	0.059	0.203	0.180	0.880	0.880	1.71	150
500	97.386	0.400	0.015	0.034	0.185	0.180	0.900	0.900	0.67	75
600	97.464	0.460	0.003	0.004	0.029	0.260	0.890	0.890	1.33	125
700	97.504	0.410	0.009	0.038	0.039	0.110	0.880	1.010	2.33	216.6
750	97.397	0.390	0.007	0.038	0.038	0.290	0.920	0.920	0.92	100

Chemical Constituents of Leached Ash via Atomic Absorption Spectrophotometer Analysis at 400-750^{0}C is shown in Table 3.0.Significant effects were also noted at calcination temperature from 400-750^{0}C

Table 3: Chemical Constituents of Leached Ash via Atomic Absorption Spectrophotometer Analysis at 400-750^{0}C

Temp (^{0}C)	SiO_2 (%)	Fe_2O_3 (%)	Zn(%)	Mn (%)	CaO (%)	MgO (%)	Na_2O (%)	K_2O (%)	LOI (%)	SSA (m^2/g)
400	97.702	0.220	0.004	0.016	0.041	0.170	1.300	0.510	0.75	100
500	97.226	0.310	0.005	0.027	0.061	0.200	1.980	0.470	0.16	25
600	98.129	0.210	0.006	0.025	0.050	0.300	1.010	0.270	0.5	75
700	98.136	0.230	0.004	0.020	0.020	0.170	1.140	0.280	1.0	125
750	97.881	0.200	0.006	0.020	0.043	0.420	1.120	0.310	0.33	50

From the results, the maximum silica was produced at700^{0}C and also the highest specific surface area. It was noted that potassium and sodium compounds (K_2O and Na_2O) are the major impurities present in the ash obtained and analysed for the processes used for the calcinations. According to Mansaray and Ghaly,(1998) sodium and potassium reacts with silica in the ash to form eutectic mixtures having low melting points. The melting point of the eutectic mixtures might be as low as 600-700^{0}C at high concentration of sodium or potassium.(Armesto et al.2002).From the literature, it was stated that if the amount of potassium is$\leq$2%, then the silica is amorphous which is correlated with the result from the analysis.

AAS results showed Rice husk ash contained high % of K_2O and Na_2O. These impurities along with black particles are attributed to the carbon content of rice husk ash and also due to the dark colour of the raw rice husk. It was also noted from the analysis of raw rice husk that high amount of Carbon is present in it. Furthermore; from the Literature, it was stated that colour is one of the factors that affects the rice husk ash. Explanation for the presence of the Carbon irrespective of temperature was given by Krishnarao et al. (1992).The K_2O in the rice husk dissociates on heating at~620K to form elemental potassium which causes surface melting. The carbon gets entrapped in this melt and loses direct contact with air which prevents its oxidation on further heating. The effect of oxalic acid is very effective in reducing the Fe_2O_3 present in the ash.

6. CONCLUSION

Silica was produced at characterisation temperature of 700^{0}C for the two methods used namely: raw rice husk and leached rice husk The LOI % and amount of silica produced, when compared with the Brazilian standard corresponds. AAS analysis characterize the composition of elements contain in the rice husk ash. It was also noted that within the limit of the present research, that the results chemical constituents of rice husk determined via Atomic Adsorption Spectrophotometer showed that leaching of rice husk with oxalic acid was superior. The low impurity level of the rice husk ash showed that it is highly reached in silica. Furthermore, Specific surface area was characterized using ASTM-D626. It was observed that agricultural waste can be converted into a viable product, economically advantageous and also for future use in nanotechnology.

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