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PHYSICAL PRODUCTION OF NIGERIAN RICE HUSK ACTIVATED CARBON: A CASE OF WASTE TO WEALTH

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Abstract
The study involves production of activated carbon from Nigerian rice husk waste by physical method. The samples were obtained from milled rice husk dump in Omor, Anambra State. Three treatment stages were carried out involving carbonization of the rice husk, leaching/separation of SiO_2, and activation of the resulting carbonaceous material. Carbonization was done in a Flow Reactor of 5000cm^3 capacity at 600^{o}C. Leaching of carbonized rice husk was done using NaOH followed by precipitation of silica and filtration. Obtained carbonaceous material was activated by physical activation method using water vapor as well as carbon dioxide in a Reactor with external electric heating without access of air. After carbonization, material containing 43.11% C and 25.01 % silicon with a specific surface area of 51.6 m^2/g was obtained from material originally containing 39.21% C, 7.01% silica, and specific surface area of about 0.158m^2/g . The specific surface area and carbon content of the material increased to 204.5 m2/g and 70.75% by weight respectively while the silicon content decreased to 1.24% by weight after leaching. Activation of carbon material using water vapor at the flow rate of 0.09dm^3 /min per 500g of material and at the temperature of 850 °C yields 1346m^2/g specific surface area - the best result. Therefore, activated carbon can be effectively produced from Nigerian rice husk by physical method using water vapor which is economical and environmentally friendly method.

Keywords: Rice-husk, carbonization, leaching, activation, carbon, and area.

Introduction
Nigeria with vast area of arable fertile land and abundant mineral resources such as coal, natural gas, and crude oil is numbered amongst the growing economies faced with the challenges of utilization and management of waste from these resources namely farm produce and petroleum. Among the great challenges facing the government and people in developing country such as ours are management and utilization of agricultural wastes. On top of the list of these agricultural wastes which occupies the limited arable lands as well as causes environmental pollution is rice husk waste from rice mills due to the enormous quantity in which they are produced (Hieu, Korobochkin and Tu, 2015). Omor, a town in Anambra State that houses one of the biggest federal government agricultural projects in Nigeria - the Anambra and Imo River basin Development Authority (AIRBDA), is faced with the challenges of rice husk waste disposal.

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Rice husks (RH) also known as rice hulls are the hard-outer part that cover and protect the grains or the rice seed which mostly cannot be digested by humans (Rice Knowledge Bank, 2018). One of the ways in which this rice husk waste can be converted into useful product (wealth) is by carbonization of the rice husk. Carbon that has been processed to become extremely porous and to have a very large surface area available for adsorption of oil in water, chemicals, heavy metals, separation of gases, removal of organic pollutants, etc., is called activated carbon (AC), activate charcoal or activated coal (Hariprasad, Rajeshwari, and Aniz, 2016). In addition to the use of rice husk as adsorbent as in activated carbon, it can also be used as building material, fertilizer, insulation material, or fuel. High porosity and absorption capacity are known characteristics of good activated carbon (Hariprasad et al., 2016).

Usually, the rice husk waste is burn in open air releasing CO_2 into the atmosphere and causing other environmental pollutions. Management and utilization of these waste is a huge concern to the people and government (Haibin et al., 2012). However, Rice husk and rice husk charcoal have carbon and silica as major components; therefore, they are suitable for precursor materials used for production of activated carbon commonly used as adsorbent (Arunrat and Sukjit, 2014). Activated carbon production from rice husk has been the subject of many studies which show that obtaining activated carbon with high specific surface area from rice husk is very possible (Korobochkin, Tu, and Hieu, 2016). Three steps involved in rice husks processing technology are: Carbonization of rice husks in air tight reactor (reactor with limited supply of air to prevent emission of CO_2) with an optimum temperature to remove moisture and volatile organic substances, and for the prior formation of porous structure; leaching of carbonized rice husk using NaOH followed by precipitation of silica and filtration; and activation of carbon material (Korobochkin et al., 2016; Khu and Thu, 2014; Ghosh and Bhattacherjee, 2013).

Some work has been reported on activation of carbonized rice husk with solutions such as biocompatible cationic polymer and strong H_3PO_4 acid (Arunrat and Sukjit, 2014; Kun-Yi and Shen-Yi, 2015). However, these add to the cost of produced activated carbon - hence, activation of the carbonized rice husk (CRH) with water vapor or CO_2. The effect of temperature, water vapor (gas) flow rate, and the activation time on the value of the specific surface area has been investigated during experiments. In this study therefore, an efficient, inexpensive, and environmental friendly conversion of rice husk waste to wealth is proposed as the main objective, while specific objectives are: to carry out cost effective process of activation of carbon residue separately with water vapor as well as with CO_2, to evaluate the effect of temperature, water vapor (gas) flow rate, and the activation time on the value of the specific surface area – hence, determining the optimum conditions.

Experimental

Experimental Procedure

Rice husk from the farm in AIRBDA was used for this study. The rice husk was carbonized in a flow Reactor of $5000cm^3$ capacity at 600^oC. Carbonized material is exposed to leaching by sodium hydroxide. Mass of loaded material was 500g and activation was carried out in the Flow Reactor with external electric heating without access of air in order to reduce loss of the loaded carbon material. The device Scanning Electron Microscope (SEM)-JED-2300 Analysis Station (JEOL) were used to obtain elemental composition of initial raw material, products of carbonization, products after separation of silica, and products of activation. Nitrogen adsorption

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isotherms (BET method) at 77 K on the device NOVA Station, 2.11, were used to determine the characteristics of the porous structure (specific surface area, pore volume, pore size). The samples were maintained in the nitrogen environment within 17 hours at a temperature of 150°C prior to measurements.

Results and Discussion

Carbonization - The elemental composition of rice husk, products of carbonization at 600 °C and products after separation of SiO_2 were shown in Table 1. The decomposition and synthesis reaction of the organic part with recombination of removing gasses and vapors occur during carbonization stage, which as a result lead to accumulation of flat formations of hexagons – precursors of graphene. A key parameter, which affects the properties of the resulting carbons, is the carbonization temperature.

Table 1: The Elemental Composition of Rice Husk, Carbonization Products, and Products after separation of SiO_2

Materials	Contents (% wt.)								
	C	H	O	N	K	Si	Ca	Mg	Al
Rice husk	39.21	5.41	46.01	0.39	1.28	7.01	0.29	0.16	0.07
Carbonization Products	43.11	0.02	29.34	0.04	1.58	25.01	0.40	0.30	0.09
Products after separation of Silica (SiO2)	70.75	0.02	25,31	0.04	0.59	1.24	1.69	0.36	0.13

Leaching/SiO_2 Separation - In order to create favorable conditions for the formation of pores in the next stages of activation, as well as for the separation of silica dioxide, carbonized material is exposed to leaching by sodium hydroxide (Hieu et al., 2015; Korobochkin et al., 2016; Yuvakkumar, Elango, Rajendran, and Kannan, 2012). Characteristics of products of carbonization of a rice husk and products after separation of SiO_2 are provided in Table 2. From Table 2, it can be seen that the carbonized product has a small specific surface area and a low pore volume. However, after removal of SiO_2, there is significant increase in the specific surface area (up to 204.5m^2/g) and an adsorption capacity (up to 0.2249cm^3/g). Thus, the carbonaceous matrix is formed with a high content of micropores in the leaching process, which accounts for 50% of a specific surface area of material.

Table 2: Pore Structure Characterization of products of processing rice husk

Material	S_{BET},m^2/g	Pore diameter,nm
After carboni-zation at 600°C	51.6	4.9
After Separation of SiO_2	204.5	4.5

Activation - to improve on the absorptive properties, the material obtained after removal of SiO_2, was activated. According to (Gridneva, Soroka, Smirnova, Belaya, and Ryabik, 2012), physical activation and chemical activation are two major activation methods for carbon material. Physical activation method, with water vapour as well as with carbon dioxide, was used in this study. Table 3 and table 4 below show the results for activation of carbon material obtained for 1 hour and 5 hours activation respectively with water vapour as well as with CO_2.

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Table 3: Results of Activation of carbon obtained from rice husk for 1 Hour Activation Time

T°C	Flow Rate of H_2O,dm^3/min	SBET, m^2/g	T°C	Flow Rate of CO_2,dm^3/min	SBET, m^2/g
700	0.09	1114	700	0.15	892
850	0.09	1346	850	0.15	1055
700	0.17	1090	700	0.55	925
850	0.17	1240	850	0.55	1098

Table 4: Results of Activation of carbon obtained from rice husk for 5 Hour Activation Time

T°C	Flow Rate of H_2O,dm^3/min	SBET, m^2/g	T°C	Flow Rate of CO_2,dm^3/min	SBET, m^2/g
700	0.09	1093	700	0.15	781
850	0.09	1199	850	0.15	1024
700	0.17	1199	700	0.55	817
850	0.17	1103	850	0.55	1065

From table 3 and table 4, it can be seen that the maximum effect of activation is observed at a temperature of 850 °C for the same activation time and close value of water vapor (or CO_2) flow rate. The material activated with water vapor has higher specific surface area than the material activated with CO_2. Furthermore, increasing the values of water vapor (CO_2) flow rate as well as increasing the activation time up to 5 hours does not increase the specific surface area of the prepared material. From table 3 and 4, it can be observed that activation of carbon material using water vapor at the flow rate of 0.09dm^3/min and the temperature of 850 °C gives the best result.

Conclusion

Carbonization done at a temperature between 500-550°C and leaching with NaOH gives good result, while activation of carbon material done with water vapor at the flow rate of 0.08dm^3 /min per 500g of material and at the temperature of 850 °C yielded the best result. Thus, rice husk (waste from rice mill) from AIRBDA in Omor, Anambra State, Nigeria can be used as an efficient, cost effective and environmental friendly resources for production of activated carbon through 3 steps, namely: Carbonization, leaching of silica from carbonized material and depositing or separating it in the form of SiO_2, and finally activation of obtained carbonaceous material using water vapour which is economical and environmental friendly method, thereby converting this waste to wealth. More research should be encouraged towards production of AC through the studied method and its application towards oil water treatment to facilitate efficient conversion of enormous amount of RH waste generated annually into wealth.

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