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Effect of Activating Agents on the Production of Activated Carbon from Rice Husk

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Abstract— The used of activated carbon cannot be over emphasize; this is why production from locally agricultural biomass attract much attention in the field of material science and technology as this also contribute in reducing agricultural wastes from the environment. The aim of this project is to compare the effectiveness of different activating agents in the production of activated carbon (an adsorbent) from rice husk. Three different reagents were used in the preparation of the adsorbent, Zinc chloride, Sulphuric acid and phosphoric acid. The surface area of the adsorbent prepared using zinc chloride as an activating agent was found to be 551 m2/g, its ash content was 20%, pH 8.04, moisture content 36.00%, volatile matter 16.67% and fixed carbon 27.33%. While surface area of the adsorbent prepared using Sulphuric acid as an activating agent was found to be 647 m2/g its ash content was 16.06%, pH 5.17, moisture content 37.00%, volatile matter 28.17% and fixed carbon 18.77%. However, the surface area of adsorbent prepared using phosphoric acid as an activating agent was found to be 743 m2/g, ash content 11%, pH 5.32, moisture content 38.71%, volatile matter 32.68% and fixed carbon 17.61%. Thus, among the three different activating agents, phosphoric acid was found to give adsorbent with the largest specific surface area of 734 m2/g, indicating that, it is the most effective among the three activating agents in producing highly porous activated carbon.

Keywords: Activated carbon, Adsorbent, Rice husk

I. INTRODUCTION

Rice is one of the major agricultural products cultivated widely in Africa and Asian countries. Rice milling industries generate a lot of by-product which include rice husk. So far, rice husk has been successfully used as fuel in many industrial factories and used as biomass fuel in electricity generating because of its low humidity and simple operation [1]. The used of rice husk as an alternative sources of energy and as adsorbent from activated carbon help in reducing the agricultural wastes in our environment. However, reports from literatures reveal that many agricultural wastes such as *Jatropha curcas* fruit shell [2], coconut shell [3], corn cob [4], *Dendrocalamus asper Baker* and *Dendrocalamus Latiflorus* [5], mangosteen shell [6], oil plam shell [7] and jackfruit shell [8] have been studied for preparation of activated carbon because of their proper properties. Thus, a possible solving of rice husk is converting it into value-added activated carbon used as adsorbents. In other words, the expensive commercial activated carbon will be reduced.

Activated carbon, also widely known as activated charcoal or activated coal is a form of carbon which has been processed to make it extremely porous and have a very large surface area available for adsorption or chemical reactions [9]. Activated carbon has a range of applications that include gas purification, gold purification, metal extraction, water purification, medicine, sewage treatment, air filters in gas masks and respirators, filters in compressed air and many other uses. But its major application is in water treatment. The first documented use of activated carbon in a large scale water treatment application was in 19th century, where it was used to remove undesirable odours and tastes from drinking water. In recent years, the use of activated carbon for the removal of priority organic pollutants has become very common [10]. Today, hundreds of brands of activated carbon are manufactured for a large variety of purposes. The largest market for activated carbon is currently in the municipal water purification industry, where charcoal beds have been used for the dual purpose of physical filtration and sorption. In fact, activated carbon filters are used today in drinking water treatment to remove the natural organic compounds that produce carcinogenic chlorinated by-products during chlorine disinfection of water.

Activated carbon is obtained when a char is subjected to controlled gasification by oxidizing gases or when a raw material impregnated with dehydrating agents, carbonization occurs. Generally, the raw materials for the production of activated are those with high carbon (organic materials) but low inorganic contents such as rice husk, wood, lignite, nutshells, pitches, coke, peat and coal [11].

Manufacturing processes are divided into two broad categories; thermal activation and mechanical activation. The effective porosity of activated carbon produced by thermal activation is as a result of gasification of the carbon at relatively high temperatures. The porosity of chemically activated carbon is as a result of chemical dehydration reaction occurring at significantly lower temperature. Some of the activating agents used for the production of activated carbon are: carbon dioxide (CO_2), steam (H_2O), oxygen gas (O_2), sodium hydroxide (NaOH), Calcium Chloride ($CaCl_2$), Zinc Chloride ($ZnCl_2$) and Phosphoric Acid (H_3PO_4). The factors affecting the production of activated carbon are; type of a raw material, activation time and temperature. There are three types of activated carbon based on physical state which include; powdered, granular and shaped activated carbon [12].

II. METHODOLOGY

A. Materials and Reagents

Rice Husk, Distilled water, Zinc Chloride, Phosphoric Acid, Potassium Hydroxide, Sodium Hydroxide (0.1N), Hydrochloric Acid (0.1N), Sodium Chloride

B. Sampling and Sample Preparation

The rice husk was collected from rice shellers' in Hadejia central market, Jigawa State. The sample was washed thoroughly to remove dirt and other impurities and then dry under atmospheric condition for three days to reduce part of the moisture content. And then finally further dry in an oven at temperature 100 °C for 24hrs. 500g of the sample should be weighted and crushed to 2mm particles size before carbonization.

C. Carbonization of Sample

After sieving to 2mm particle size, 200 g of the sample was obtained as powdered one. 10g of the sample was weighed into a tube and the insert it using thongs into the tubular furnace for carbonization. The furnace was set at 550 °C for 30mins with the flow of an inert gas (Nitrogen) passing through it. The procedure was repeated several times till all the powdered sample is exhausted with the 10g sample resulting to averagely 4.2g of carbonized sample and the initial total amount of the 200g became 75g. After each carbonization, the furnace was switched off and allows the sample to cool for some moment.

D. Activation of Sample

60g of the sample was measured after carbonization and divided into 3 different beakers, A, B and C. 20g of each sample was measured into the beakers. The samples were activated using three different reagents; zinc chloride, sulphuric acid and phosphoric acid. Beaker A was activated with zinc chloride, beaker B with sulphuric acid and beaker C with phosphoric acid, each of the carbonized samples was activated with the reagents in the ratio 1:2 to form a paste. The activated samples were dry in an oven for 3 hours at a temperature of 110 °C. The activated samples were washed with distilled water to remove excess acid and alkaline until a stable pH is reached. Then the samples were oven dried for 3hrs at 200 °C.

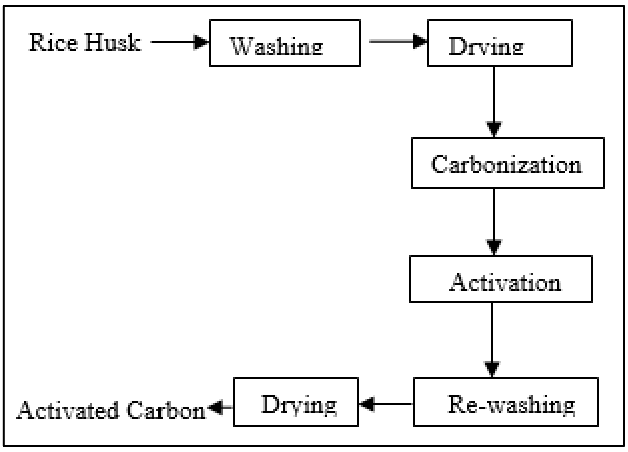

Fig. 1: Block diagram showing the procedure for carbon activation.

E. Characterization of the Activated Carbon

The produced activated carbons were characterized using the following parameters:-

1) Specific Surface Area

1.5g of the activated carbon was measured and mixed with 100ml of water and 30 g NaCl. The mixture was stirred for about 5 minutes and 0.1N HCl was added continuously till the final pH value become 4.0. The mixtures were then titrated against 0.1N NaOH, and the volume of 0.1N NaOH required to raise the pH from 4.0 to 9.0 was recorded.

The following relation is used to determine the specific surface area:-

$$A = 32V - 25$$

Where; A = Surface area of the activated carbon per gram (m^2/gm)

V = Volume of 0.1N NaOH required to raise the pH from 4.0 to 9.0

2) pH Determination

The pH of the activated carbon is measured using the standard test of the ASTMD38380. 1g of the activated carbon was heated in 10ml of distilled water to its boiling point for 15mins. The slurry was then filtered and the pH of the filtrate was measured at 50 °C.

3) Ash Content Determination

The ash content of the sample is determined using the ASTM procedure D2886694. The sample was heated at 650 °C for about 1hr until a constant weight is achieved. The ashes were then measured. Ash content of each activated carbon is evaluated as follows:-

$$\text{Total Ash} = \frac{(A-B)}{(C-B)} \times 100$$

Where, A= weight of the crucible plus ash containing sample

B= weight of the crucible in gram

C= weight of the crucible plus original sample weight in grams

4) Moisture Content

A given amount of each sample of activated carbon was heated in a furnace at a temperature of 105 °C for an hour. The ratio of change in weight to the original weight expressed in percentage gives the moisture content. It is given by;

$$\frac{WL}{WO} \times 100$$

Where; WL = Weight loss (Original weight – final weight)

WO = Original weight

5) Volatile Matter Content

A given amount of each sample of moisture-free activated carbon was heated in a furnace at a temperature of 600 °C for 10 mins in the absence of air. The ratio of change in weight to the original weight expressed in percentage gives the volatile matter content. It is given by;-

$$\frac{WL}{WO} \times 100$$

Where WL = Weight loss (Original weight – final weight)

WO = Original weight

6) Fixed Carbon Content

This is the residue left after the moisture, volatile and ash is given up. It is deduced by subtracting from 100, the percentage of moisture, volatile matter and ash content. The fixed carbon content (FC) is given as;-

$$FC = 100 - (\%\text{moisture} + \%\text{volatile matter} + \%\text{ash})$$

III. RESULTS

Table 1 below shows the results of the characterization of activated carbons produced using three different reagents labeled A, B and C. The activated carbons were characterized using the parameters; Specific surface area, ash content, pH value, moisture content, volatile matter content and fixed carbon content. Sample A was activated with zinc chloride, sample B was activated with sulphuric acid and sample C was activated with phosphoric acid.

Sample	Specific Surface Area (m^2/g)	Ash Content (%)	pH Value	Moisture Content (%)	Volatile Matter content (%)	Fixed Carbon (%)
A	551	20.00	8.04	36.00	16.67	27.33
B	647	16.06	5.17	37.00	28.17	18.77
C	743	11.00	5.32	38.71	32.68	17.61

Table 1: Properties of the Activated Carbon.

Fig. 2: Activated carbon produced using Phosphoric Acids

Fig. 3: Activated carbon produced using Sulphuric Acid

Fig. 4: Activated carbon produced using Zinc Chloride

IV. DISCUSSION

As presented in table 1, sample C has the largest surface area of 743 m^2/g followed by sample B with a surface area of 647 m^2/g and then sample A with a surface area of 551 m^2/g. All specific surface areas for the activated carbons produced using different activating agents lie with the limit of generally acceptable surface areas of 500-1200 m^2/ [12]. According to literature activated carbon produced using phosphoric acid is expected to have surface area higher than those produced using sulphuric acid and zinc chloride, because the adsorptive ability of activated carbons generally depends on surface area and pore volume. According to [13], activated carbon from rice husk, which is activated using H_3PO_4 at a temperature of 450 °C, has high adsorption capacity. However, from gasoline adsorption study, the optimum conditions were 0.1 g of activated carbon, 70 °C of adsorption temperature and 30 minutes of adsorption time.

After activation, all three activated carbons were dried at 200 $^\circ$C for three hours in an ovum. As it was discussed earlier ash blocks some pores of activated carbon thereby reducing its surface area.

The residue after carbonaceous material is burned off is referred to as ash. The ash exists as a result of inorganic, inert and unstable parts present in the activated carbon. Ash can lead to increased hydrophilicity and can have catalytic effects, causing restructuring process during regeneration of used activated carbon. The inorganic material contained in activated carbon is measured as ash content. From table 1, sample A, B and C have ash content values of 20%, 16.06% and 11%, respectively. Sample C has the lowest ash content of 11%; this may be due to its larger surface area than the other two. The lower the value the ash content the better the activated carbon.

For pH measurement, all the pH values obtained in table 1 show that each of them approaches neutral state. This is as a result of washing the samples with distilled water after activation. The pH values are; 8.04, 5.17 and 5.32 for sample A, B and C respectively (table 1). This occurred because sample A was activated with zinc chloride, it’s should be expected to have pH closer to that of neutral state. Sample B was activated with sulphuric acid; it should be expected to have acidic property. Sample C was activated with phosphoric acid, is should be expected to have acidic property as well.

Moreover, activated carbons of sample A, B and C have moisture content values of 36.00%, 37.00% and 38.71% respectively, with sample C having the highest percentage and sample A with lowest percentage. The volatile matter content for activated carbon of sample A, B and C have the percentage of 16.67%, 28.17% and 32.68% respectively with sample A has the lowest percentage and sample C with the highest. Then for the fixed carbon, the activated carbon for sample A, B and C have the percentage of 27.33%, 18.77% and 17.61% but this time around sample C has the lowest percentage, whereas sample A has the highest percentage. These shows that among all the activating agents used in this research, the used of phosphoric acid as an activating agent is the most effective in the production of activated carbon.

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