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An Investigation of Producing Activated Carbon from *Moringa Oleifera* Seeds Husk

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Abstract

Activated carbon is an important adsorbent in many industries. The cost of many products is increasing because of the high price of activated carbon. Therefore, to find an alternative is of great importance. The alternatives can be produced from many plant wastes one of which is *Moringa oleifera* seeds husks. Three samples were used (mechanically removed husks, manually removed husks, and the fine size of manually removed husks). The seeds husks were carbonized with one stage steam pyrolysis, because the process requires less energy compared to two heating stages. The three samples were heated in a cylindrical pyrolysis reactor which is heated by electrical heater to the required temperature of 600, 700, and 800°C, and the samples were kept at each temperature for an hour. The produced activated carbon was characterized by measuring surface area and adsorption capacity by Langmuir adsorption isotherm and Iodine test. The results showed that three samples are having good properties as activated carbon compared to commercial one. As a conclusion, the *Moringa oleifera* seeds husks can be considered for more research work to be a good alternative for commercial activated carbon.

Keyword: Activated carbon, *Moringa oleifera*, Seeds husks.

1. Introduction

Activated carbons are carbonaceous materials that can be distinguished from elemental carbon by the oxidation of the carbon atoms found on the outer and inner surfaces of carbon materials [1]. The characteristics of activated carbons are large surface areas, well-developed porosity and tuneable surface-containing functional groups [2, 3]. Other than that, they have good kinetic properties and high adsorption capacities. For these reasons, activated carbons are widely used as adsorbents for the removal of organic chemicals and metal ions of environmental or economic concern from air, gases, potable water and wastewater [4].

Nowadays, activated carbons from many sources have been used to remove dissolved metallic salts from aqueous solutions [5, 6]. Adsorption on activated carbons also can reduce

COD [7, 8]. Due to high price of commercially produced activated carbons [9], many substances have been screened as an alternatives to the conventional raw materials for the production of activated carbons such as pine sawdust, rose seed, and cornel seed [10]. Other renewable sources are coconut shells, and *Moringa oleifera* seeds husks [11], rubber wood sawdust [12], and rice husk [13].

Moringa oleifera is the best native to North India, it is known as multipurpose tree as its part are useful for many applications. This tree grows fast and develops to a full tree within one year of its plantation. The dried pods and husks can produce activated carbon by single – step steam pyrolysis [14, 15].

2. Methods and Materials

2.1 Moringa Oeifera

Moringa oleifera seeds husks were collected from Kuantan, Pahang, Malaysia. Three samples were used (sample 1 represents mechanically removed husks which is collected from Mitomasa Sdn. Bhd as by-product, sample 2 is manually removed husks, and sample 3 represents the fine size of manually removed husks which is grinded with size of 70 μ m).

2.2 Preparation of Activated Carbon

The seeds husks were carbonized with one stage steam pyrolysis (where no chemicals are required), and the process requires less energy compared to two heating stages. The three samples were heated in a cylindrical pyrolysis reactor which is heated by electrical heater to the required temperature of 600, 700, and 800°C, and the samples were kept at each temperature for an hour. To remove liquid products, the water – cooled condenser, ice traps and a column with cotton and phosphorus pentoxide are used as drying agent with gas meter. The reactor tube was heated by an electrical heater. The sample was placed in the removable inner cylinder with a net in the bottom. The outer cylinder is connected to a metal tube with a screw holder which makes it easy to clean. The water was pumped to the steam generator by a peristaltic pump. The steam temperature is around 105°C, and volatiles were cooled down in a water – cooled condenser. The gas was led through a column with cotton and phosphorus pentoxide as drying agent, and then collected in a bag.

The processes that are involved in steam pyrolysis are thermal decomposition, multi – component steam distillation, fast escape and stabilization of the volatiles, mild oxidation, and activation of the solid residues and formation of the activated carbon. The multi – component distillation which accompanies the pyrolysis in a stream of water vapour will give a mild process and efficient escape of the volatiles from the solid material. The quick removal of the volatiles and stabilization of the radicals in the presence of the steam results higher

yields of volatiles and formation of solid products with a highly developed active surface free from organic compounds.

The reactor was heated with heating rate of 25°C/min up to the required temperature of 600, 700, and 800°C, and the samples were kept at each temperature for an hour [15]. The surface area of produced activated carbon was measured, and the adsorption properties were tested by Langmuir Adsorption Isotherm and Iodine number.

2.3 Surface Area

The internal surface area of a carbon is usually determined by the BET method (Brunauer, Emmerr and Teller). This method utilizes the low – pressure range of the adsorption isotherm of a molecule of known dimensions (usually nitrogen). This region of the isotherm is generally attributed to monolayer adsorption. Thus, by assuming the species is adsorbed only one molecule deep on the carbon's surface, the surface area can be measured. The surface area was measured using Thermo Scientific (SURFER).

2.4 Langmuir Adsorption Isotherm

The Langmuir adsorption isotherm relates the coverage or adsorption of molecules on a solid surface to concentration of a medium above the solid surface at a fixed temperature. During the test, a sample of activated carbon is contacted for some time with a test substance. The graph of $\log x/m$ versus $\log c$ are plotted where x is amount of impurities absorbed at equilibrium, m is mass of the activated carbon and c is the concentration of impurities remaining in liquid. From the data collected from the experiments, it was shown that the three different sizes of husks have good absorbance properties.

The test was performed as follows: i) Oxalic acid with 0.5 N was prepared; ii) 2 g of activated carbon was transferred into each of five bottles; iii) different volumes of oxalic acid of 50, 40, 30, 20 and 10 ml were added followed by 0, 10, 20, 30 and 40 ml of distilled water so that the total volume is 50 ml in each bottle; iv) The bottles were shaken thoroughly for an hour by mechanical shaker and left aside in a trough containing water to reach equilibrium; v) The supernatant liquid of each bottle was filtered and the filtrate was collected in properly labelled conical flasks; vi) The initial 5 ml of the filtrate was rejected, and 10 ml of the filtrate was pipette out into a clean conical flask, and titrated against standardized KMnO_4 solution until a pink colour appears; vii) The titrations was repeated to get concordant values, from the titration values, the concentration of oxalic acid remaining and the amount of oxalic acid adsorbed were calculated; viii) $\log (x/m)$ against $\log c$ are plotted, and the value of R^2 was obtained from the graph.

2.5 Iodine Adsorption

Iodine number represents the surface area contributed by the pore larger than 10Å [16, 17]. To perform this test, the following procedure was followed; i) the activated carbon produced with a mass of 2 g was transferred to a dry 250 ml Erlenmeyer flask; ii) 10 ml of 5% HCl was pipette into the flask and mixed until the activated carbon is wet; iii) The flasks are placed on a hot plate and the contents were brought to boil and allowed to boil for 30 seconds; iv) The contents are allowed to cool to room temperature; v) Then, 100 ml of 0.10 N iodine solution was added; vi) The flask was closed with a stopper immediately and shaken vigorously for 30 seconds; vii) The solution was filtered by gravity through a filter paper immediately after 30 seconds shaking period; viii) The initial 20 – 30 ml of titrate are discarded and the remainder are collected in a clean beaker; ix) The filtrate was stirred in the beaker with a glass rod and 50 ml was pipette into a 250 ml Erlenmeyer flask; x) The 50 ml sample was titrated with 0.10 N sodium thiosulphate solution until the yellow colour disappeared; xi) About 1 ml of starch solution was added and the titration was continued until the blue indicator colour disappeared, and the volume of sodium thiosulphate solution was recorded.

3. Results and Discussion

The yield of activated carbon produced from three samples was 50% for all experiment conditions applied. Other tests results are as follows:

3.1 Surface Area

The effect of temperature is the key factors during the preparation of the activated carbon in order to optimize the best condition to produce the activated carbon. The highest surface area was obtained at temperature of 600°C compared to 700°C and 800°C. This is because materials which was undergone deep carbonization accompanied by removal of large amount of volatile organic compounds are converted into solid residues with low contents of organic material and higher content of ash. From the results obtained, sample 1 has the largest surface area, although sample 2 and 3 are giving good surface area as well, as shown in Table 1. The results are compared with commercial activated carbon available in markets from Sin Guan Hup Oil and Rice Mill Sdn. Bhd., Pulau Pinang, Malaysia. The company produces rice husk activated carbon as by product from furnace and steam boiler. The comparison is shown in Table 2.

Table 1 Surface area for produced activated carbon.

TEMPERATURE(°C)	SURFACE AREA(m ² /g)		
	Sample 1	Sample 2	Sample 3
600	0.003	0.002	0.0026
700	0.0013	0.0012	0.0014
800	0.0005	0.0008	0.0007

3.2 Langmuir Adsorption Isotherm

The Langmuir model describes the relationship between the amount of oxalic acid adsorbed and its equilibrium concentration in the solution using activated carbon. The results in Figures 1, 2, and 3 showing R² of 0.9891, 0.9887, and 0.9899 for samples 1, 2, and 3, respectively. The results reflect the good adsorption property of the produced activated carbon.

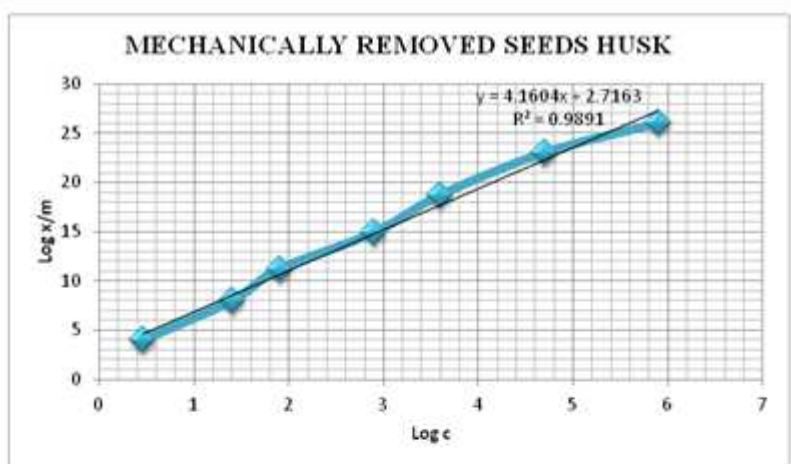


Figure 1 Langmuir Adsorption Isotherm of Sample 1.

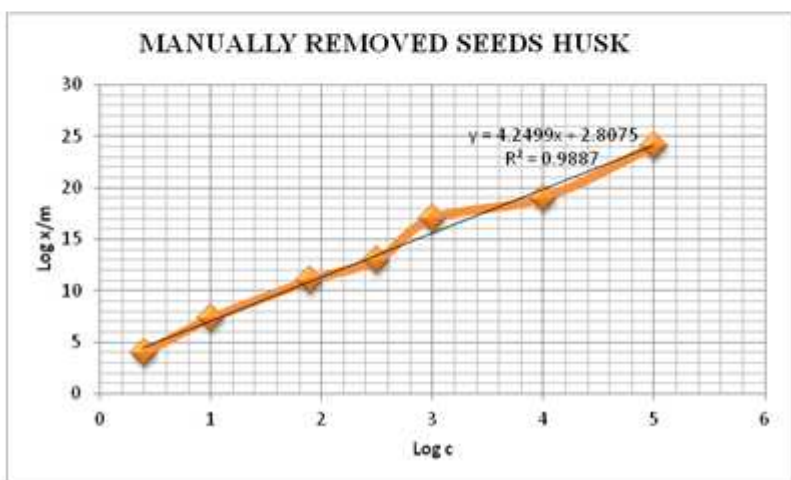


Figure 2 Langmuir Adsorption Isotherm of Sample 2.

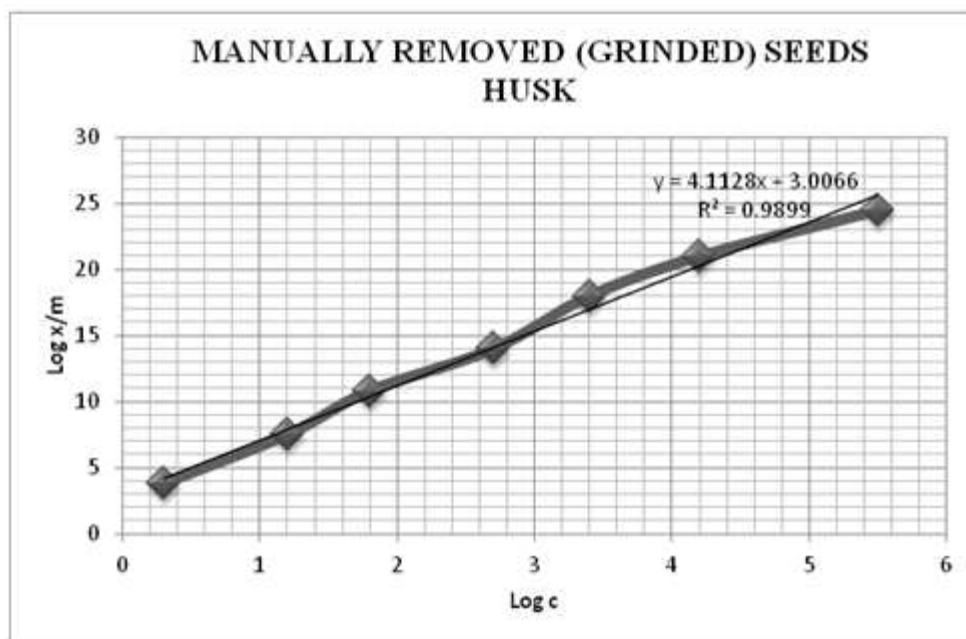


Figure 3 Langmuir Adsorption Isotherm of Sample 3.

3.3 Iodine Test

The iodine test is carried out to determine the iodine number which gives indication of the internal surface area of activated carbon. It is defined as the number of milligrams of iodine adsorbed from an aqueous solution by 1 g of activated carbon. The higher the iodine number, the higher the surface area of the activated carbon. From the results obtained, it can be concluded that three samples give good results of adsorption as shown in Figure 4, 5, and 6 for samples 1, 2, and 3, respectively. The Iodine number for three samples is tabulated in Table 2.

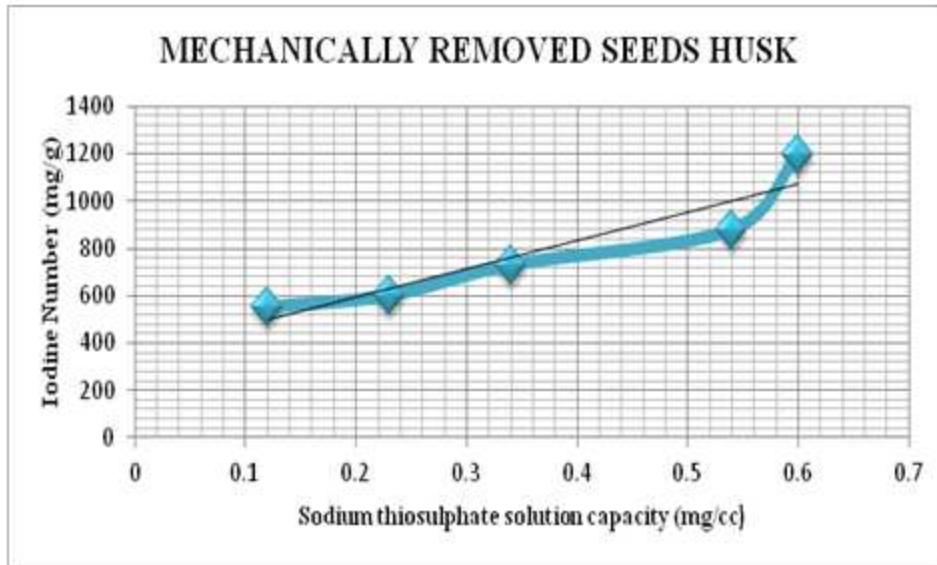


Figure 4 Iodine Number of Sample 1.

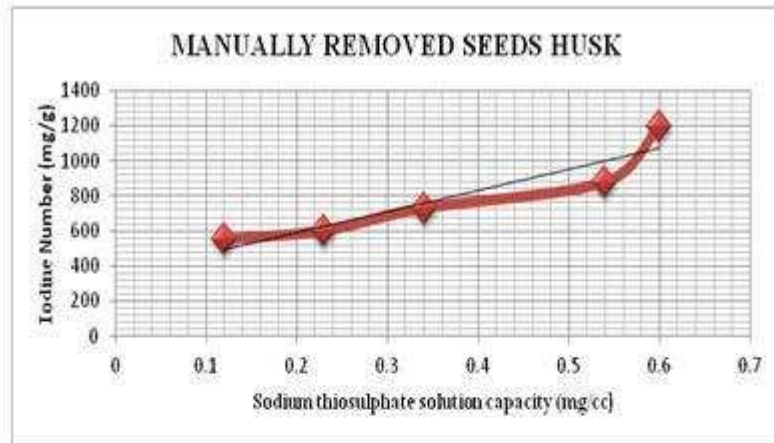


Figure 5 Iodine Number of Sample 2.

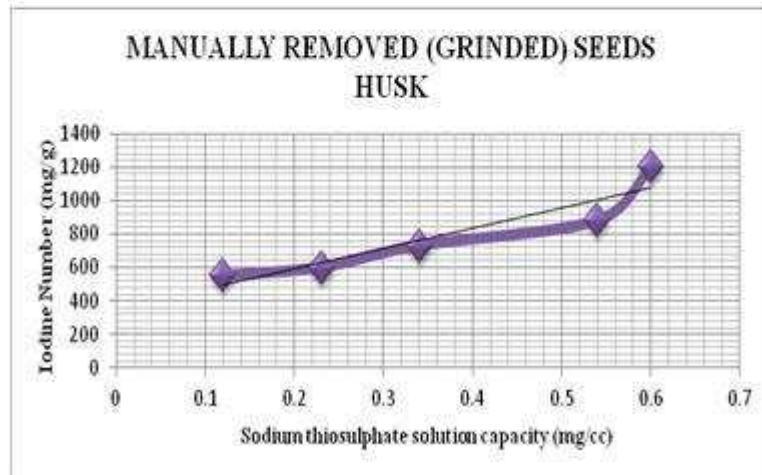


Figure 6 Iodine Number of Sample 3.

Table 2, Properties comparison

ACTIVATED CARBON	SAMPLE 1	SAMPLE 2	SAMPLE 3	COMMERCIAL PRODUCT
Surface Area (m ² /g)	0.003	0.002	0.0026	0.0013
Adsorption (Value of R ²)	0.9891	0.9887	0.9899	0.9881
Iodine Number (mg/g)	1260	1250	1270	1240

4. Conclusion

Activated carbon was produced from *Moringa oleifera* seeds husks. The properties of activated carbon produced such as: surface area and adsorption capacity showed good results compared to the commercial activated carbon available in the markets.

The results showed that activated carbon can be produced from *Moringa oleifera* seeds husks in one stage steam pyrolysis without any chemical addition. The mechanically removed seeds husks has the highest surface area and the fine size of manually removed husks presented the highest iodine number, and the manually removed husks showed lowest properties.. More research work is recommended to be carried out at less than 600°C activation temperatures at different heating range. Different seeds husks can be used for optimization.

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