



Research Article

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## Physicochemical characteristics of activated charcoal derived from melon seed husk

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### ABSTRACT

*This work investigated the generation of activated carbon from melon seed husk - a low value agricultural waste, its characterization using volumetric methods and subsequent utility in the bleaching of vegetable oil. The production of activated carbon from melon seed husk was done by a chemical activating process involving the use of 1 M NaOH solution mixed with 20 g of the sample and steeped overnight. The steeped waste was filtered and air-dried and then carbonized in a muffle furnace at the temperature of 500°C for a residence time of 40 minutes to give an activated carbon. The resultant activated carbon in powder form was characterized for the particle size, pH, bulk density, iodine adsorption number, pore volume, porosity and moisture content. The activated carbon was equally applied for the bleaching of de-gummed sample of vegetable oil. The result of the characterization showed that the powdered activated carbon has good properties and compared favourably with other reference activated carbons. While the bleaching experiment result showed that the activated carbon successfully bleached the colour of the de-gummed vegetable oil, and that the extent of oil bleaching was related to the quantity of activated carbon used. This research work has added to proof that powder activated carbon produced from melon seed husk has great adsorptive capacity and can be used for both liquid and gaseous phase adsorption.*

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### INTRODUCTION

Adsorption is one of the most important percolations processes used in industry for the purification and separation of solutes from a fluid stream onto a surface [1]. Activated carbon also known as activated carbon or activated coal is the best known general purpose adsorption medium and the system using activated carbon requires little space than many competitive processes and is impervious to toxic wastes and produces little or no sludge or secondary pollution. Charcoal as produced by carbonization process is not a very active adsorption material either for liquids or vapours because its fine structures is blocked by tarry residues. The carbonized carbon is then converted to the activated form through physical or chemical activation processes. However, chemical activation is preferred over physical activation owing to the lower temperature and shorter time needed for activating material. Chemical activation carried out as one step process using moderate temperature results in the development of highly porous structures [2]. Activated carbon hence is a form of carbon that has been processed to make it extremely porous and having a very large surface area available for adsorption or chemical reactions. Activated carbon has a relatively high degree of micro-porosity, hence merely one gram of activated carbon has surface area of approximately 500 cm<sup>2</sup> equivalent to about 2.17 tennis courts, as determined typically by nitrogen gas adsorption [3]. Sufficient activation for useful applications may come solely from the high surface area, though further chemical treatment often enhances the absorbing properties of the material. Amongst the wide industrial applications of adsorption process, activated carbon presents an important operation, which finds its major use in solution purifications [4].

In the recent times, there is the dear need and search for various methods of conversion of agricultural residues to more useful products like activated carbon. The activated carbon from the agricultural residues are subsequently

used for various industrial applications such as in adsorption studies. Activated carbon with desired characteristics can be obtained from solid agricultural wastes if carefully controlled processes of dehydration, carbonization and activation of the material are undertaken [5]. Such activated charcoal is used in gas purification, medicine, sewage treatment, air filters in gas masks, filters in compressed air and many applications. They are also applied in vodka of organic impurities since the activated charcoal does not bind well to alcohols [6], the percentage of alcohol is not significantly affected, while the carbon will bind and remove many organic impurities which can affect taste, colour and odour. The activated carbon as obtained from these carbonaceous material or organic precursor have had their hydrocarbons removed to increase its powers of adsorption [7]. For example, agricultural raw materials such as mangrove wood, bone and wood [8], bamboo [9], peels of citrus *documana* [10], marine algae *Valoria bryopsis* [11], coconut shell [12] and palm kernel shell [13], have been studied for the production of activated carbon. Woody materials like bamboo are used to obtain high –grade activated carbon with inherent mechanical strength, high carbon (40 percent) and low ash content. There has been an increasingly high demand for activated carbon following the vast applications of activated carbon in industrial processes such as solvent recovery, gasoline vapour emission control canisters in automobile, clean-up of corn sugar solutions and removal of tastes and odours from water supplies, vegetable and animal fats and oils, alcoholic beverages chemicals and pharmaceuticals. As a decolourant, activated carbon, with its very large surface area and pore volume, is hundreds of times more efficient than charcoal and at least 40 times more than bone black. The amount of material adsorbed by activated carbon is surprisingly large, amounting frequently to from a quarter to an equal weight of such vapours as gasoline, benzene, carbon tetrachloride [14]. Activated carbon is able to absorb practically any organic solvent at about 35°C and release it when heated to 120°C or higher for solvent recovery [4].

Many reports have appeared on the development of activated carbon from cheaper and readily available agricultural materials. Throughout the world, agricultural by-products such as wood, coal, shells etc are some of the materials used in activated carbon production [9]; [13].

In this work, the husk from melon seed are used as sample for conversion into activated carbon. It is of particular interest to know why melon seed husk is the chosen material in our proposal than others. Melon seed husk are available abundantly at no or low costs, it therefore has a potential to provide a low cost adsorbent for cleaning our environment. This material arising from melon seed husk do not have any immediate use, are discarded as waste in many parts of the country or are used as land- fills, making soft surface for poultry animals, while in some places are burnt as source of fuel or just ordinarily burnt as a form of disposal method. The success in this research will no doubt open up new product line for the melon seed husk which is regarded as waste and a nuisance in the environment.

## EXPERIMENTAL SECTION

### Collection and preparation of samples

Melon seed were purchased from Keffi central market. The seeds were de-husked and the husks were used as samples. The melon seed husk was washed severally with distilled water in order to remove dirt's and dusts. They sample was further sun-dried for 4 days and later blended using Keenwood electric blender in order to obtain a powder form of the waste. The powder waste was then sieved with 500 µm mesh [15]. This portion of the powder sample was then kept in air-tight containers for further analysis.

### Chemical activation and carbonization

Activated carbon was generated from the melon seed husk using chemical activation method.

Powdered melon seed husk sample (50 g) was treated with 1 M NaOH and steeped overnight. This was done to activate the waste prior to carbonization. The steeped waste was filtered to remove excess alkali, air-dried and then carbonized in a muffle furnace at the carbonization temperature of 540°C for a residence time of 40 min. to give an activated carbon. The activated carbon produced was washed with 0.5 M acetic acid solution, rinsed thoroughly with distilled water, sun-dried and sieved. The portion retained on the mesh were oven dried for 1 h and stored in an air-tight container.

### Characterization of activated carbon

The formed activated carbon was characterized for moisture content, ash, particle size, pH, adsorption capacity, iodine adsorption number (IAN), bulk density and pore volume. Characterization of activated carbon is very important in order to classify activated carbon for specific uses.

**(i) Moisture Content Determination**

A 1.0 g of the activated carbon sample was collected and dried in an oven for four hours at 150°C, until the weight of the sample became constant [19]. The moisture content was calculated from the relationship;

$$X_o = \frac{W_1 - W_2}{W_1}$$

**Where:**

$X_o$  = Moisture content on wet basis

$W_1$  = Initial weight of sample, g

$W_2$  = Final weight of sample after drying( g)

**(ii) Particle Size**

For the particle size determination, lots of samples were weighed and placed on top of a set of sieves (7) ranging from 75 to  $1.4 \times 10^3$   $\mu\text{m}$ . The sieves were shaken manually for two minutes, after which the weight percent of the active carbon retained on the sieves and bottom pan was determined [16].

**(iii) pH**

1 g of the sample was weighed and dissolved in 3 ml of de-ionized water. The mixture was heated and stirred for 3 minutes to ensure proper dilution of the sample. The solution was filtered and out and its pH was determined using a digital pH meter.

**(iv) Iodine Adsorption Number (IAN)**

1 g sample was weighed into a beaker and 25 ml of standard iodine solution (0.023 M) added. The mixture was swirled vigorously for 10 minutes and filtered by means of a funnel impregnated with clean ashless glass wool. 20 ml of the clear filtrate was titrated with the standard (0.1095 M) thiosulphate solution to the persistent of a pale-yellow colour. 5 ml of freshly prepared starch indicator was added and titration resumed slowly until a colourless solution appeared, the procedure was carried out two more times. The titrations were also repeated with 20 ml portions of the standard iodine solution not treated with the sample to serve as the blank titration. The iodine number (IAN) was calculated from the relationship

$$\text{IAN} = \frac{12.69 N (V_2 - V_1) \text{ mole iodine/g sample}}{W}$$

**Where:**

$N$  is the normality of thiosulphate solution

$V_1$  is the volume of the thiosulphate (ml) used for the titration of the sample –treated aliquot.

$V_2$  is the volume of the thiosulphate (ml) used for the blank titration,

$W$  is the mass of the sample used (g).

**(v) Determination of Porosity and Bulk Density**

The sample (1 g) was dispersed in 20 ml water in a graduated cylinder with the aid of a shaker, this was further centrifuged for 10 minutes. The resulting volume of the water was read a  $V_T$  and recorded. The equation below was used for the calculation of the porosity and bulk density as the case may be.

**Porosity** =  $V^w/V_T$ ,

**Density** =  $\text{raw}/(1 - \alpha)$  while

**Raw** =  $\text{Ma}/V_w$

**(vi) Pore Volume**

The sample (1 g) was collected and transferred completely into a 10 ml measuring cylinder in order to get the total volume of the sample. The sample was then poured into a beaker containing 20 ml of deionized water and boiled for 5 min. the content in the beaker was then filtered, superficially dried, and weighed. The pore volume of the sample was determined by dividing the increase in weight of the sample by the density of water [8].

**(vii) Bleaching Treatment**

The activated carbon (0.5 g) was added into a beaker containing 50 g of degummed oil sample. The mixture was heated to a constant temperature of 120°C, with stirring for 10 min. and then filtered at the same temperature using a funnel and a vacuum pump. The filtrate was kept for colour measurement. For the colour measurement, the filtrate was placed in the optical cell and placed in the Lovibond equipment while moving the coloured filters to the right. The red, yellow and blue filters were adjusted in correct proportion until the correct colour match was obtained. Two reference columns were filled up with de-ionized water to the mark. The sample column was filled up with the oil to

the mark and then placed in the sample compartment in between the two reference cells. With one eye closed, it was observed through the top of the machine while adjusting the two knobs of the reference samples until hardly one can differentiate the colour of the two reference samples and the sample. The reading of the lowest value was recorded for the sample [17]. 2.5 g and 5.0 g of the activated carbon was repeated for the above procedure. Also the same procedure was repeated for the remaining two samples.

## RESULTS AND DISCUSSION

The characteristics of the powdered activated carbon produced in this work from melon seed husk are presented in Table 1 and are observed to fall within some reference characteristics. Table 2 represents the reference characteristics of activated carbon results from other authors derived from other different agro- wastes while Table 3 is the result of bleaching of vegetable oil with activated carbon.

**Table 1: Activated carbon characteristics of melon seed husk at 540°C carbonization temperature**

Parameter	Activated Carbon 540°C Melon seed husk
Bulk density (g/cm <sup>3</sup> )	0.54
Porosity	0.54
Pore volume (ml)	1.10
Mean Particle Size (µm)	22.73
Moisture Content (%)	27.61
Iodine number (mg/g)	966
PH	8.90

**Table 2: Activated carbon characteristics comparison**

Parameter	Activated carbon sample at 540°C	Reference activated carbon at 700°C
Bulk density (g/cm <sup>3</sup> )	0.54	0.40 – 0.51 [8]; [16]; [18]
Porosity	0.56	0.12 – 0.21 [8]; [16]; [18]
Pore volume	1.10	0.50 – 1.11 [19]; [16]; [18]
Mean particle size (µm)	22.73	5 – 50 [20]; [16]; [18]
Moisture content (%)	27.61	0.9 – 8.0 [21]; [16]; [18]
pH	8.90	6.0 – 7.10 [21]; [16]; [18]
Iodine absorption number (mg/g)	966	500 – 1200 [3 ]

**Table 3: Result of bleaching of vegetable oil using activated carbon**

Sample	Colour (Lovibond Tintometer)	Colour (Lovibond Comparator)
Degummed Oil	Yellow	>3
	<b>0.5g Activated Carbon Bleaching</b>	
Melon Seed Husk a.c	Yellow	>3
	<b>2.5g Activated Carbon Bleaching</b>	
Melon Seed Husk a.c	Light yellow	<3
	<b>5.0 g Activated Carbon Bleaching</b>	
Melon Seed Husk a.c	Bleached	<1

The activated carbon produced in this work from the melon seed husk sample had acceptable properties and compared favourably with reference activated carbon which is an indication of quality of adsorbent that can be used for both liquid and gaseous purification operations.

### Iodine adsorption

The highest iodine adsorption number obtained for the sample was 966 mg/g at the carbonization temperature of 540°C. It is known that activated carbon do absorb iodine very well and hence the iodine adsorption number is an indication of the total surface area of the activated carbon. It is a measure of activity level, the higher the number the higher the degree of activation. It is also a measure of the microspore content of the activated carbon (0 to 20 Å or up to 2 nm) by adsorption of iodine from solution [3]. The smaller the volume of iodine adsorbed the bigger the iodine number adsorption. Consequently, the bigger the surface area and the more effective it is [21].

### Particle size

The particle size value obtained in this work for the sample was 22.73(µm) at 540°C activation temperature. The value fall within those of the reference values. The significance of particle sizes is that it aided the fast diffusion of solvent polluted to get to the active part of the activated carbon or carbonaceous material. The finer the particle size of an activated carbon, the better the access to the surface area and the faster the rate of adsorption kinetics. Careful consideration of particle size distribution can provide significant operating benefits. This may fasten adsorption of contaminants to the surface of adsorbents.

**pH**

The pH also affects the rate of activated carbon adsorption. The value of pH obtained in this work is high at the pH 8.90. Activated carbon is more effective at low pH than high pH.

**Moisture content**

Activated carbon is generally priced on a moisture free basis. Some activated carbon when stored under humid conditions will absorb considerable amount of moisture over a period of time and will still appear dry, therefore airtight containers are most ideal for storing it. For many purposes, the moisture content does not affect the adsorptive power of activated carbon, but they obviously dilute the carbon.

**Bleaching test**

The bleaching performance of the activated carbon derived from melon seed husk with respect to colour reduction is shown in Table 3. It was observed for the sample that colour reduction was proportional to the quantity of activated carbon used. For instance, using 2.5g of the activated carbon on the degummed oil, the colour was reduced to light yellow from the original yellow colour. When the amount was increased to 5.0g the colour of the oil was completely discharged (Table 3). The observed increase in colour reduction with the quantity of activated carbon is probably due to the availability of more adsorption sites until saturation point is attained. This could also be as a result of an increase in the collision frequency between the molecules of the colouring matter and the activated carbon [23]. Bleaching is a complex mechanism and involves more than simple adsorption of colour pigments. Bleaching has two factors working in its favour- adsorption and colour fading by oxidation and two factors working against it –new colour formation by oxidation [24].

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