Production of Activated Carbon from fibers (*Cocus nucifera L.*) via Addition Of Adipic Acid and Chemical Activation

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Abstract

This research consists to produce of activated carbon from fibers (cocus nucifera L.) via some additions. A different percentages of Adepic acid (10-40%) related to the raw material carbonization and activation had been done by using Sodium hydroxide (1:2). The research concept was based on acid decomposition during carbonization and the probability of bi-sodium salts formation for adepic acid. That leads to a increasing the efficiency of carbonization. This process gives a positive results for the size and the number of internal and external holes for prepared activated carbon. The efficiency of carbonization increased by increasing of the ratio of the added adepic acid and that was proved by the results of the research. The Iodine number was estimated methylene blue adsorption, measurement of ash content, density, humidity and comparing it with one of commercial samples of B. D. H company.

Key words: Activated carbon, chemical activation, adipic acid

Introduction

Activated carbon are widely used as adsorbents in gas and liquid-phase separation processes, purification of products and water cleaning operations. One of the most important field in terms of consumption in water and waste water treatment, where activated carbon with a relatively high surface area and well developed porosity are needed (21).

Usually the production of activated carbons involves two stages, the carbonization of the raw materials followed by a high temperature activation at 800-1000 °C of the resulting chars. The method used in this study combines the two stage into a single one, while the treatment temperature is considerably lower between 600-800 °C. This method is preferable to the two stage treatment from an economic point of view (5,12).

Chemical activation of various carbonaceous precursors with phosphoric acid has long been known and used for the production of active carbon in industry (11,14).

Among the other raw materials used as precursors to make activated carbon are sawdust, peat, lignite, coal, cellulose residues (10) and petroleum coke, spent ion exchange resins such as styrene-divinyl benzene polymers (23).

Phenol-formaldehyde resins (19) and old automobile tires (17) and sewage sludge (8). Various binding agents may be added to improve the structure (4). Commercial sources appear to be made from a variety of precursors, activating agents and binders (22).

In case of Looking at the literatures we found many research has been done as given below:

Gergova et al. (5) have used one step pyrolysis steam activation to produce activated carbons from coal and agricultural by products, 50 gm sample was heated in a tube furnace at heating rate 10°C/min at atmospheric pressure and a steam flow rate of 0.5 dm³/hr. The experiments were curried out in a temperature range of 600-700 °C. The samples were heated at the final temperature for 1, 2 and 3 hr. Activated carbon were produced from apricot stones at 700 °C for 2 hr., had the highest surface (1175 m²/gm).

Patra Panyawafanakit (13) produced activated carbon from palm oil shell. The processes of carbonization and activation with super heated steam were studied. The

palm oil shells were carbonized at 400 °C for 1 hr. Next, the charcoal was activated with super heated steam. The optimum condition for activation was 0.850-0.355 mm of charcoal size at 900 °C for 1 hr.

The resulting characteristics were yield of 19.31% iodine number of 779 mg/gm, methylene blue number of 136.96 mg/gm, total surface area of $670.1 \text{ m}^2\text{/gm}$.

Hiavaly and Kavan (6) prepared activated carbon by using carbon source and metallic powder of Na, Li and K by employing the catalyst as colloidal materials in toluene or normal hydrocarbon solvent.

The activity of metal ion for carbonization is K > Na > Li, Measurements indicate that the carbon obtained in the study have a good adsorption property and large surface area.

Carbonization of brown coal using hydroxide of Na and K at 500-700 °C was carried out by Kobagashi et al. (9). The measurement of the characters of the activated carbon prepared such as surface area which is found to be $1000 \text{ m}^2/\text{gm}$.

Experimental

Production of Activated Carbon:

10 gm of fibers (*Cocus nucifera L.*) was mixed with different percentage of (10-40) % adipic acid. The mixtures were mixed with twice the weight of sodium hydroxide and 10 ml of distilled water. The mixtures were heated gradually to 450-500 °C with continuous stirring.

The mixtures were allowed to stand at 450-500 $^{\circ}$ C for 3 hrs. 100 ml of distilled water were added to the carbonized products, refluxed with 10% HCl for 1 hour, filtered and washed with distilled water. The products were dried at 120 $^{\circ}$ C and allowed to cool to room temperature. The samples were used in the next steps .

Activated Carbon Measurements:

A. Measurement of Density

The density of the prepared activated carbon were determined by weighting 10 cm³ of the carbon sample using graduated cylinder (1).

B. Determination of Ash Content

The Ash content was measured by heating one gram of the prepared activated carbon sample in a porcelain crucible using an electrical furnace for five hours at temperature of 1000-1100°C. the remained residue was weighed and considered as the ash content (2).

C. Measurement of Humidity

One gram (exactly weighed) of the activated carbon was heated in oven at 150° C for 3hr. the difference weight before and after heating was calculated as H_2O vapour in the sample (7).

D. Determination of Carbon Activity by Methylene Blue Adsorption Method

An exactly weight (0.1 gm) of the prepared activated carbon sample was added to an aqueous solution of 20 ppm methylene blue pigment in a conical flask. The solution was shacked by an electrical shaker for 24 hr at a temperature of 25°C till adsorption of methylene blue from its aqueous solution was completed.

The absorbance of the solution was dtermined by using (UV- Visible Spectrophotometer) at λ max 665 nm.

The procedure was carried out with different types of the prepared samples for comparison purposes. The final concentration of methylene blue value for each activated carbon sample was calculated as the number at milligrams of methylene blue adsorbed by one gram of carbon (20).

E. Determination of Carbon Activity by Iodine Adsorption Method

The iodine number which is the amount (in milligrams) of iodine adsorbed from its aqueous solution by one gram of activated carbon.

The method involve:

One gram of the dried activated carbon transferred to 20 ml Erlenmeyer flask. To the flask, 10 ml of 5% HCl was added and stirred until the carbon was wetted.

Place the flask on a hot plate, bring the contents to a boil and allow to boil for exactly 30 minute. Allowed the flask and contents to cool to room temperature and add 100 ml of standardized 0.1 N iodine solution to the flask.

Immediately stopper flask and shake the contents vigorously for 30 miunte.

Filter by gravity immediately after the 30 minute shaking period through filter paper.

Mix the filtrate in the beaker with a stirring rod and pipette 50 ml of the filtrate into 250 ml Erlenmeyer flask.

Titrate the 50 ml sample with standardized 0.1 N sodium thiosulfate solution until the yellow color has almost disappeared. Add about 1 ml of starch solution and continue titration until the blue indicator color just

disappears. Record the volume of sodium thiosulfate solution used (3).

Iodine number =
$$\frac{X}{m}$$
 D

Where:

m = is the weight of the activated carbon in grams

X = A - [2.2 Bx ml of thiosulfate solution used]

 $A = N_1 \times 1269.0$

 $B = N_2 \times 126.93$

 N_1 = normality of iodine solution.

 N_2 = normality of sodium thiosulfate solution.

D = correction factor.

Results and Discussion

Active carbon is a porous carbonaceous material, prepared by carbonizing and activating organic substances of mainly biological origin. Active carbon are sufficiently specific in nature. The adsorb certain substances tenaciously while they show less affinity for others (16).

In our study we aimed to production activated carbon from fiber (*Cocus nucifera L.*) which contain large lignin content by mixing with different percentage of adipic acid.

The carbonization carried out by mixing with excess amount of sodium hydroxide at 550 ± 25 °C for 3 hrs.

The results of the study are given in the Table below.

Production of activated carbon using sodium hydroxide leds to evolution hydrogen then H_2O formation .

Whereas sodium ion led to the pores structure and determined the holes size (18) from the Table we show.

1. Ash Content:

The ash content which is explain the amount of metal in the sample is slightly low in all the sample compared with B.D.H. sample.

2. Humidity Content:

Humidity content of the sample is slightly higher than the commercial one and this can be reduced by elevating the temperature to 250 °C without exposure to the air to get little loss in the carbon.

3. Iodine Number and Methylene Blue:

Measuring internal porous in the form of iodine number in mg/gm, external porous which is termed as macropores in term of the adsorption methylene blue indicate that the percentage 40% of adipic acid gives the best results. The results due to the dissociation of adipic acid in the carbonization process and formed $(NaOOC(CH_2)_4COONa)$ which leds to increase the carbonization activity.

Sample	Adipic acid (%)	Iodine No. (mg/gm)	Methylene blue (mg/gm)	Ash (%)	Density (gm/cm ³)	Humidity (%)	Surface area* (m²/gm)
1	0	575.3	20	0.45	0.17	6.90	507
2	10	598.0	26.3	0.51	0.15	8.01	542
3	20	670.2	35.8	0.56	0.16	8.78	592
4	30	750.1	42.7	0.61	0.12	9.62	647
5	40	802.0	48.0	0.63	0.15	9.95	682
B.D.H	-	908	90	3.2	0.325	0.8	755

^{*} Calculated by equation adapted from reference (21)

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إنتاج كاربون منشط من ألياف ثمرة جوز الهند (Cocus nucifera L.) بإضافة حامض الادبيك والتنشيط الكيمياوي

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الملخص

يتضمن البحث تحضير الكاربون المنشط من ألياف ثمرة جوز الهند مع بعض المضافات، إذ استخدمت نسب مختلفة من حامض الادبيك (١٠-٠٤%) نسبة إلى المادة الأولية، وأجريت عملية الكربنة والتتشيط باستخدام هيدروكسيد الصوديوم بنسبة (١:١). اعتمدت فكرة البحث على تفكك الحامض أثناء عملية الكربنة واحتمالية تكون أملاح الصوديوم الثنائية لحامض الادبيك مما يزيد كفاءة عملية الكربنة والذي انعكس إيجابا على حجم وعدد المسامات الخارجية والداخلية للكاربون المنشط المحضر والتي ازدادت بزيادة نسبة حامض الادبيك المضاف وهذا ما دلت عليه النتائج المستحصلة. ولقد تم تقدير عدد اليود وامتزاز صبغة المثيلين الزرقاء وقياس محتوى الرماد والكثافة والرطوبة ومقارنتها مع احد النماذج التجارية لشركة B. D. H.