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انتاج الكاربون المنشط من ثمار نبات اللزيق بالمعاملة الكيميائية مع هيدروكسيد البوتاسيوم وتحديد خواصه الفيزيائية ودراسة صفاته الامتزازية

م . د. حيدر ابراهيم حامد سليم الطه

وزارة التربية / المديرية العامة لتربية نينوى

haideribrahim259@gmail.com

المستخلص:

الغرض من هذه الدراسة هو تحضير كاربون منشط له صفات امتزازيه جيدة يمكن استخدامه في تنقية الماء والاهواء, تم تحضير الكاربون المنشط من ثمار نبات اللزيق بالمعاملة الكيميائية مع نسب مختلفة من هيدروكسيد البوتاسيوم, و تم قياس صفاته الفيزيائية (الكثافة , الرطوبة , الرماد) و خصائص الامتزاز من خلال امتزاز صبغة المثلين الزرقاء وكذلك اليود لجميع النماذج المحضرة. خلال نتائج امتزاز صبغة المثلين الزرقاء واليود تم اخذ افضل نموذجين (S_3 ,S_4) و تم اجاء قياس المساحة السطحية وحجم المسامات لهما من خلال فحص BET وهذا الفحص اظهر ان النموذج (S_3) يمتلك اكبر مساحة سطحية و كذلك اكبر حجم للمسامات التي يمتلكها, وقد كانت هذه القياسات مطابقة تماما لخصائص الامتزاز من حيث امتزاز صبغة المثليـن و اليود للنمـاذج (S_3 ,S_4).

الكلمات المفتاحية : كاربون منشط ، اللزيق ، هيدروكسيد البوتاسيوم.

Production of activated carbon from the fruits of the Xanthium spinosum plant by Chemical treatment with potassium hydroxide and determination of its physical and adsorption properties

Lec.Dr.Haider Ibrahim Hamed Salim Al-Taha

Ministry of Education /Directorate General of Education Nineveh

haideribrahim259@gmail.com

Abstract

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The purpose of this study is to prepare activated carbon with good adsorption properties that can be used in water and air purification. Activated carbon was prepared from the fruits of the Xanthium spinosum plant by chemical treatment with different proportions of potassium hydroxide. Its physical properties (density, moisture, ash) and adsorption properties were measured by adsorption of methylene blue dye as well as iodine for all prepared samples. During the results of the adsorption of methylene blue dye and iodine, the two best models (S3, S4) were taken and the surface area and pore volume were measured through BET examination. This examination showed that the model (S3) had the largest surface area and also the largest pore volume, and these measurements were completely consistent with the adsorption properties in terms of the adsorption of methylene blue dye and iodine for the models (S3, S4.(

Key words: Activated Carbon, Xanthium Spinosum, Potassium hydroxide.

Introduction

Coal has been known for a long time, as activated carbon prepared from plant sources was used until 2000 years BC. The ancient Egyptians used it to purify water used for medical purposes, and large quantities of it were produced in the seventeenth and eighteenth centuries. It was prepared from the partial combustion of wood in limited amounts of air, producing what is known as (wood charcoal). Coal was also extracted from special mines deep under the surface of the earth, which is known as (bituminous coal), whose specifications and heat of combustion can vary greatly depending on its characteristics (Daridsos, 1968; Parakash et al., 1974).

At the start of the 20th century, activated carbon was made from coconut shells. However, due to the growing demand for carbon and the scarcity of these shells, producers tried to make it from other organic materials, such as coal, coal tar, various wood types, bitumen, and polymers, bones, asphalt materials, and other materials (Othmer et al ., 1974).

A flaw in its crystalline structure and a hydrogen shortfall occur during the formation of activated carbon, an amorphous porous substance. Because of this flaw, pores that are unstable in terms of their energy content or efficacy arise. The carbon granules' exterior surfaces are where these pores

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are primarily located. In other situations, the activator is internal, and because the pores are larger than those found in typical carbon types, it has a higher capacity for adsorption. As a result, the extremely high adsorption capacity of activated carbon has been demonstrated (Green, 1986; Othmer et al., 1964).

The porous structure is the distinctive characteristic of activated carbon, as the great diversity in the shape and size of these pores has led to its diversification of uses. Activated carbon has a large surface area, ranging from (300-2000) m^2/gm and this large surface area indicates the high percentage of these structures. Porosity (Hart et al., 1967).

Activated carbon also has a high ability to adsorb certain substances such as methylene blue dye, urea, carbon tetrachloride, ammonia, nitrogen oxides, and others (Shreve et al., 1976).

Many types of organic carbon materials are used to prepare activated carbon as raw materials, including wood, bones, brown and dark charcoal, coconut shells, black paper ash, heavy oil residues, semi-carbonized materials, sugar, and organic sediments resulting from wastewater treatment, as well as It is produced from some manufactured materials such as various organic polymers, and materials with a high carbon content are preferred in the manufacture of activated carbon (Baharudin et al., 1987).

The structure of activated carbon contains a group of atoms that are foreign to the basic structure. These atoms are known as hybrid atoms, such as oxygen, nitrogen, and sulfur. These atoms affect the characteristics of the activated carbon and its adsorption properties, as the adsorption of nitrogen on the surface of the activated carbon is considered a physical adsorption, unlike the chemical adsorption of oxygen, which is difficult. Removing it from the surface, it is removed by intense heating to expel oxygen in the form of oxides (CO_2, CO) and then this removal leads to a loss in the carbon percentage (Steenberg, 1944).

Experimental

1- Preparing Samples of wood for Carbonization Purpose:-

The Xanthium spinosum plant's fruits were allowed to dry in the air before being placed in a furnace set between 110 and 130 degrees Celsius

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until they reached equilibrium weight. Then, using the base KOH, the fruits were cut into small pieces to achieve the carbonize-able size (ASTM D 2854-70, 1898).

2- Primary Carbonization by Soaking Method:-

After 48 hours of soaking in a base (KOH), carbonising agent, and activating material in different ratios (0.5, 1, 1.5, 2), the fruit of the Xanthium spinosum plant was filtered normally without the need of filter paper. After drying the models at 110 to 130 degrees Celsius and calculating their weights, all of the dried samples were stored for the final carbonising step (Austin, 1992).

3- The final activation of the various samples and final thermal carbonisation:

The material left over after the main carbonisation process was heated for three hours over a direct flame to a temperature of roughly 550±50Co. The samples were then allowed to cool to room temperature.

4- The Purification and Activation of Carbon:-

1- Distilled water was used to wash the produced carbon samples in order to eliminate any sodium hydroxide that had not reacted., and till the confining of the equivalence of the resultant water of the washing process by using sunflower paper as an evidence. Then all the samples dried out in a furnace at (110-130C^{o}) for a period of 3 hours to obtain a constant weight for the sample.

2- After the washing and drying processes were finished, the carbon sample was put in a round flask. Next, a solution of 10% hydrochloric acid was added. The solution was boiled for 30 minutes to dissolve the carbon, and then distilled water was used to clean it until it was as clean as the washing water. The sample was then dried at 110 to 130 degrees Celsius until its weight stayed consistent. It was then packed and stored in a container until it was needed for measurements again.

5-Identification of Activated Carbon's Internal and External Pores:

-5.1- Iodine adsorption from aqueous solution is used to measure the internal area of activated carbon:

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Measuring the quantity of iodine absorbed by the activated carbon samples is a simple and rapid method of determining the inner surface dimension. The following is implied when one gramme of activated carbon is used to calculate the amount of adsorbed iodine in milligrammes (McGraw-Hill, 2005).

1. Put one gram of activated carbon into a conical flask and add 10 ml of (10% HCl) hydrochloric acid.
2. After bringing the mixture to a boil for 30 minutes, let it cool to room temperature.
3. A flask containing 100 millilitres of 0.1N iodine solution was sealed with a light stopper and left in an electrical agitating device for 30 minutes. After filtering the flask's contents, the remainder was gathered in a dry flask.
4. A 250 ml conical flask was filled with 50 ml of the filtered material. After that, it was made pale yellow by diluting it with a normal sodium thiosulphate dihydrate (0.1N) solution. When we finally added one millilitre of starch pilot, the solution's colour changed from blue to yellow, depending on the size of the sodium thiosolfate dihydrate (0.1N). Then, we added another millilitre of starch pilot, which caused the solution's colour to change to blue. The admixing process was then continued until the blue colour vanished based on the sodium thiosolfate dihydrate that was consumed through the size differences.

5- The weight of iodine adsorbed by the activated carbon has been calculated using the following formula.:-

"X = A - [2.2B × ml volume of sodium thiosulfate dihydrate]

A = N1 × 12693…..

B = N2 × 126.93….

Whereas:-

X = the adsorbed iodine weight in ml, by the activated carbon

N1 = Iodine solution concentration

N2 = sodium thiosolfate dihydrate concentration which (N1 = N2) equivalent to (0.1N)

The following formula is used to determine the iodine number:

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I.N = $\frac{X}{M} D$ …..

M = the activated carbon sample weight used (1gm)
D = correction factor"

5.2- Utilising the Adsorption of Methylene Blue from Aqueous Solutions to Determine the External Surface Area of Activated Carbon:-

The analysis of methylene blue adsorption from its aqueous solution provides insight into molecules with a high molecular weight; this technique represents the activated carbon's outer surface area. While 0.1 gramme of activated carbon was taken after adding a specific volume (10ml) of methylene blue (20 ppm) and placed in a conical flask, one gramme of activated carbon can be used to define this value as the number of milligrammes of methylene blue that were extracted from its a solution by its adsorption on the outer surface of the carbon. The flask attached to theelectrical shaker device for 24 hours ,with a shaking speed 100 RPM (Revolutions Per Minute) at the laboratory temperature until the colour is disappeared, at this case, an additional amount of the pigment solution is added, and the shaker continues until the colour is fixed, following which a specific volume of the solution is when the purified solution is taken and put in a centrifugal apparatus for three to five minutes to remove the carbon particles, the absorption is measured and the value of the absorption as it relates to the dyes solution at the wave length of 665 nm (the wave length where the dyes is being absorbed.

The standard curve, which is made especially for this purpose, determines the removed pigment concentration from the aqueous solution by taking various standard concentrations from the pigment solution (5, 10, 15, 20, 25 pmm), measuring the absorption of these solutions at the wave length (665 nm), and drawing diagrammatic lines between the concentrations and the absorption lines (Mochida et al., 1997).

6.1 - Physical Properties of Activated Carbon Measured: Calculating the Humidity Content:-

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Following the weighing of one gramme of the wet activated carbon samples, they were baked at 150 degrees Celsius for three hours, the weight difference was used to calculate the humidity percentage.

$$\%\text{Humidity} = \frac{\text{weight of the wet activated carbon} - \text{weight of the dry activated carbon}}{\text{weight of the dry activated carbon}} \times 100$$

6.2- Percentage of Ash:-

To determine the percentage of ash in each sample, one gramme of activated carbon was placed in a crucible and placed for three hours at 1000 degrees Celsius in an electric oven. For each sample of greenish activated carbon, the crucible was then leave for 3 hours at laboratory temperature, the crucible is then weighed and the difference in weight represents the ash content (Naito, 2018)

$$\%\text{Ash} = \frac{\text{weight after born}}{\text{weight befor born}} \times 100$$

6.3- Determination of apparent Density:-

A certain amount of activated carbon is added to determine the density (after being crashed and sieved in specific sieves size 80 mmash) in volumetric bottle (5 ml) the weight is calculated from the following law:- (Toth et al., 2001).

$$\text{Density} = \frac{mass}{volume} \quad \text{gm/cm}^3$$

Discussion of Results

In this research, different ratios of the base were taken with the Non-activated carbon in order to activate. The base (KOH) ratios taken are (base: non-activated carbon, 0.5:1 , 1:1 , 1.5:1 and 2:1).

Table (1) shows the percentages of base (KOH) used in activation and the weight of the non-activated carbon before the final carbonization, as well as the weight of the final activated carbon after activation with the base and the weight loss, as well as the percentages of the resulting activated carbon, (١٠)grams of unactivated carbon was taken and initially carbonized and

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activated with different proportions of potassium hydroxide base. The weight loss was very small and the final activated carbon yield was high. It is noticeable that the higher the percentage of base added for activation, the lower the percentage of final activated carbon produced.

Samples	Ratio non-activated carbon : KOH	Weight non-activated carbon before finally carbonization gm.	The weight of the final activated carbon after activation with the base gm.	Reducing the weight of activated carbon gm.	The percentages of activated carbon produced %
S_1	٠.٥: ١	10	9.7	0.3	97
S_2	1 : 1	10	9.3	0.7	92
S_3	١.٥1 :	10	9.2	0.8	91
S_4	٢: ١	10	8.8	1.2	89

Table (1) :- Weight change of the activated carbon sample following final activation with the base KOH

The physical properties examined for the four samples (density, moisture and ash content) were close to the values of the samples, The density examination shows that increasing the percentage of base used in the chemical activation process led to a decrease in the density and thus an increase in the number and size of pores in the final prepared activated carbon. This is completely consistent with the increase in the adsorption capacity of the samples. The lower the density, the greater the adsorption capacity of methylene blue and iodine, as shown in the lower density models 3 and 4, which are more adsorbent to methylene blue and iodine. Moisture values also increased with increasing levels of activated base, indicating that the base played a significant role in increasing the internal and surface area of the final activated carbon. Ash values were within the academically acceptable range.

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Testing the adsorption of methylene blue dye and iodine adsorption, we note that models (3) and (4) gave the highest adsorption values, as shown in Table (2). That is, the activated chamber worked to increase the surface area and internal area of the activated carbon. this means an increase in the number and size of pores present in the prepared activated carbon.

samples / Measurements	S_1	S_2	S_3	S_4	BDH*
Density gm./cm^3	0.127	25١0.	0١0.1	0.081	0.350
Humidity %	0.401	0.514	0.352	0.411	0.800
Ash %	٦٧2.0	2.150	1.255	2.190	3.200
M.B mg/g	245	437	536	521	90
I.N mg/g	122	216	249	234	908

Table (2):- The value of the physical properties and the results of the adsorption test of methylene blue and the iodine absorption of the prepared activated carbon.

BDH* = Commercial Model (Hamdoon et al., 2005).

The test (BET) conducted on the two best models (models 3 and 4) gave values that were completely consistent with the adsorption values of methylene blue and iodine.

Model 3 gave the largest surface area and the largest pore volume.

Model (3) gave the largest BET surface area (291.834 m^2/g), and Model (4) had the largest surface area (253.451m2/g). Also, the Pore Volume

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and Pore Size (width) values were given preference to model (3), as it gave the largest values for these tests, Physical values (density, humidity, and ash) are within academically acceptable limits. As shown in Table 3:

Samples / measurements	S_3	S_4
BET Surface area m^2/g	291.834	253.451
Pore Volume cm^3/g	0.1572	0.1484
Pore Size (width) Nm	7.8621	5.1916

Table (3):- Value BET Surface area, Pore Volume and Pore Size (width) for activated carbon models.

figure (Fig 1) show the Xanthium spinosum plant: The following

Conclusions and Recommendations

1. The ratio 1 base : 1 non-activated carbon was determined as the best activation ratio.
2. The percentage of the prepared activated carbon was very high when using a base ratio of 1: 1 un-activated carbon, Table (1)
3. Internal area We note that Model S3 (base 1: 1 un-activated carbon) gave the largest internal area as indicated by iodine adsorption, Table (2). the reason for this is that the increase in the base has destroyed the internal pores of the activated carbon.
4. The external surface area was the preference for model S3, which gave the largest adsorption of methylene blue dye, Table (2).
5. The results of the BET assay (Table 3) were completely identical to the measurements of iodine adsorption and adsorption of methylene blue, (Table ٢).

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