Preparation of Activated Charcoal with High Surface Area from an Agriculture Wastes and Industrial Residues

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الخلاصة:

تركز العمل في هذا البحث على تحضير مادة مختبرية مهمة وهي مادة الكاربون المنشط (Activated Carbon) والتي تسمى ايضاً بالفحم المنشط Charcoal، وذلك بالاستفادة من مصدرين الاول: الكم الهائل للمخلفات الطبيعية المتمثلة بمخلفات نبات الذرة من سيقان وقشور والعرنوص الفارغ، ومخلفات ناتج حصاد نباتي الحنطة والشعير وأعشاب برية مختلفة. اما المصدر الثاني فهو المخلفات الصناعية المتمثلة بالورق (مسودات, كتب تالفة، اوراق امتحانية، الاوراق الرسمية للدوائر الحكومية، اوراق المصارف والصناديق الكارتونية) ونشارة الخشب التي تنتج في محلات النجارة وبكميات كبيرة حيث تم استخدام نشارة نوعين من الخشب هما (الصنوبر والزان). وقد تضمنت طريقة التحضير مفاعلة هذه المخلفات بنوعيها الطبيعي والصناعي مع وفر من هيدروكسيد الصوديوم وبنسبة وزنية (1 من المخلفات : 2 من هيدروكسيد الصوديوم) حيث تم حرقها لمدة ثلاث ساعات عند درجة حرارة 550 ± 25 م°. تلت عملية الحرق هذه تنقية المواد الكاربونية المتكونة عن طريق غسلها عدة مرات بالماء المقطر حتى الوصول الى حالة التعادل، ثم معاملة الناتج مع محلول حامض الهيدروكلوريك بتركيز 10 %. يخضع مزيج التفاعل لعملية التصعيد الحراري (Reflux) لمدة ساعة واحدة ويتم ترشيح الناتج وتغسل المادة الكاربونية بالماء المقطر حتى الوصول الى حالة التعادل مرة اخرى، ثم تترك مدة 24 ساعة عند درجة حرارة 110 – 120 م° لغرض التجفيف. تم تحديد العديد من المواصفات والخواص الفيزياوية للكاربون المنشط الذي تم تحضيره مثل الكثافة والرطوبة وكمية الرماد وقابلية الامتزاز ومقارنتها مع مواصفات الكاربون المنشط التجاري لشركة .B.D.H.

الكلمات المفتاحية: الكاربون المنشط ، الميثيلين الأزرق ، النفايات الزراعية

Abstract:

This work was concentrated on preparation of activated carbon which also call (charcoal) from natural agricultural wastes (leg & leaves and empty ear of corn, Wheat & Barley grass and herb residues) and industrial residues (Paper drafts and Sawdust). These residues were treated first with Sodium Hydroxide (1:2) (1 of the raw materials : 2 of NaOH), then burning at 550 ± 25 °C for three hours to complete the carbonization process. The reaction mixture was purified by washing with distillation water and neutralized using 10% Hydrochloric acid. Reflux for one hour, filtrate and washing with distilled water, and leaves to dry at 110 – 120 °C for 24 hours. The physical properties of preparing activated carbon were determined and compared with a commercial activated carbon which supply by B.D.H. chemical company.

Key words: Activated Carbon, Methylene Blue (MB), Agriculture Wastes.

1. Introduction:

Activated carbon which also known activated charcoal has a structure composed from carbon atoms this material has an ancient history and an important Lab's uses depending on its properties like, high surface area,[1] highly micro-porous.[2] This gave it a several applications in adsorption of many substances from aqueous solution,[3] moreover, was used as a material for separation of gases,[4] removable and cleaner for unwanted organic chemicals from different phases and systems as liquid and vapor [5] help to soak up the poisons in food poisoning cases as well as it used to purification of drinking water from organic pollutants, color and odors removal.[6] Activated carbon in the beginning had been produced using a vegetable material and used to refining of sugar.[7] After that, the producing was developed to preparing a powdered activated carbon from wood ((MDF) sawdust) which used widely in the industrial.[8, 9, 10]

Previously agricultural residue and waste materials had been successfully used as sources to production of activated carbon and applied for adsorption processes, like almond shells, [11] rice hulls [12], husks and coconut shells [13], scrap tyres [14], sulfonated coal [15], grape seeds [16] and pomegranate peel.[17]

There are many reports focused on the preparation of activated carbon, and these usually depend on source of the feed stock, and what included the carbonization process to produce a special type of activation carbon.

Al-Channam and Co-workers. [18] Early used a Mours Nigra to prepare an activated carbon using varied amounts of NaOH and the mixture was heated to 500 ± 25°C. Hu and Srinvasan [19] proposed other method to prepare activated carbon; this proposal was based on chemical activation of coconut shell in present of potassium hydroxide as an activated agent. Guo et al.[20] developed a route to preparation an activated carbon via treatment of rice husks with potassium hydroxide which also act as an activating agent.

The present work involves uses of agricultural and natural in addition to synthetic wastes to production of activated carbon. Then, the adsorption of Methylene Blue and Iodine had been applied in determination the activity of obtained product charcoal.

2. **Experimental**:

2.1. Carbonization of natural and synthetic residues:

To carbonization and activation for the starting materials, the residue (20 gm) had been mixed with excess amount of sodium hydroxide (40 gm), and then the mixture was heated to 550 ± 25 °C for three hours.

2.2. Purification of Charcoal:

After complete the carbonization process, the resulting products had been cooled and washed well with distilling water to get the neutralized which checked by the using litmus paper. This resulting carbon had been treated with hydrochloric acid (10%), refluxed for 60 min, filtered and washed well with distill water (4 x 200 ml). Finally, the result of this process was dried in oven for 24 hours at 110 – 120 °C to give a pure activated carbon in high yeild.

2.3. The Properties of prepared Activated Carbon:

2.3.1. Activated Carbon Density Measurement:

There are a several methods to Measurement the density of activated charcoal. In this work the density of the prepared activated carbon were determined by: an empty 5 mL volumetric flask was weighed and the mass was recorded (10.2 gm). Then, amount of prepared activated charcoal was placed to it followed by a slight shake till the powder molecules will be in one level and up to the mark, and was weighed (12.7 gm). The difference between the mass of the filled flask and the empty flask was the exact mass for the used activated charcoal. [21]

The density calculator uses the formula D (g/cm^3) = Wt / V

D= density

Wt= weight

V= volume

2.3.2. Ash Content:

The ash content of the prepared activated charcoal has been a measured via burning (1 g) of the carbon sample by heating for at less (5 hrs) between 1000-

1100 °C in an electrical furnace, to get a residue which consider as an ash content of activated carbon. [22]

2.3.3. Measurement of Humidity:

In order to determine the humidity of prepared charcoal, one gram of the sample has been heated at 150 °C for three hours using an oven. The weight should be exactly (1 gm) and the difference between primary sample weight before heating and final weight after heating was calculated and it refer to the water vapor in the carbon sample.[23]

2.3.4. Adsorption of Methylene Blue in determination of charcoal activity:

To determinate the carbon activity, adsorption of methylene blue (MB) method's had been applied using (0.1 gm) from the prepared charcoal which mixed with (20 ppm) of aqueous solution of methylene blue in a rotary shaker. The reaction mixture was left to shaking for 24 hours at room temperature (25 °C). This limited time determined the equilibrium case for the adsorption of the methylene blue from its aqueous solution. UV-Visible spectrophotometer has been used to determine the absorbance of the resulting solution, and applied on the different samples which were prepared for comparison purposes. Finally in the calculation, for the each prepared sample of the activated charcoal has been taken the final value of concentration of the methylene blue (MB). Each milligram of methylene blue was refer to the amount that absorbed by one gram of carbon.[24]

2.3.5. Pore Volume:

From each sample (1g) was transferred into a beaker then10 ml of deionized water was added and boiled for 5 min. The beaker contents were then filtered, and superficially dried. Finally resulting products was weighed and use to determine the pore volume of the samples, by dividing the increase in weight of the sample by the density of water. [25]

2.3.6. Iodine adsorption number test to determination of charcoal activity:

Iodine number (In) adsorption method was used to estimate surface area of activated charcoal. In this test each amount of iodine in its aqueous solution (in milligram measured) adsorbed by a gram of activated carbon and the method include a few steps:

First step, In 250 ml flask hydrochloric acid 5% (10 ml) was added to a dry Prepare an activated charcoal (1 g) until it became a wet, then the mixture was heated to boil for exactly half hour (30 minute). The mixture of flask contents was cooled down to room temperature followed by the addition of 0.1 N iodine solutions (100 ml).

Second step involved, in a closed system by using a stopper for the flask, the mixture was shaken vigorously for not less than 30 minute and followed by a filtration (Filter by gravity) using filter paper. The mixture was poured into beaker and leave to stir for 10 min, and then 50 ml of the filtration product had been taken and transferred to (250 ml) conical flask.

Finally, the choosing sample (50 ml) was titrate with a standardized (0.1N) of sodium thiosulfate solution was added until disappearing the yellow color and it became aclear colorless solution. Following this, starch solution (1 ml) was added as indicator to give a blue color, and continuous in titration process until disappearance of the color. The value of sodium thiosulfate which used has been recorded and apply in calculates.[26]

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

X = a - [2.2 b * volume of thiosulfate solution that has been used(in ml)]

a = Normality of the iodine solution * 1269.0

b = Normality of the sodium thiosulfate solution * 126.93

m = weight (in gram) of activated charcoal.

D = correction factor.

3. Results and discussion:

It's known that the active carbon is a common compound and consider as an important product which used widely in industrial and in the chemist labs, in addition to uses in various aspect of modern civilization. As described above, there are a number of ways to produce but it usually the producing process required a carbon feedstock availability.

These studies led to produce an activated carbon with an excellent quality and this would be achieved by carbonizing of raw materials (natural and synthetic residues) by treatment with an excess amount of sodium hydroxide in limited temperature around 550 ± 25°C for three hours reaction time

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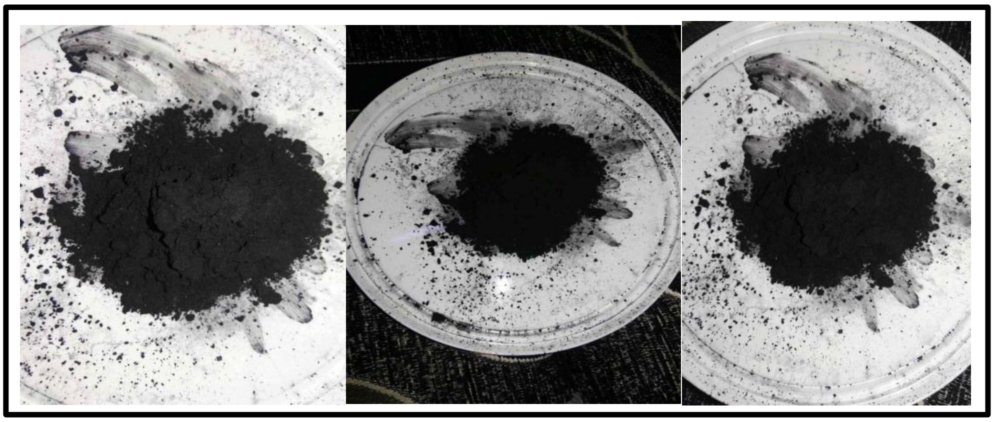

Figure (1): Samples of the prepared activated charcoal in this study

Hydroxide ion of the sodium hydroxide work on etching the raw materials structure and make it easy to porous, while sodium ion in this method help to determination the size of the porous.[27] The results obtained from these experiments were reported in the table (1).

Table (1): The Properties of Activated Carbon

Sample Name	Density	Ash %	Humidity	Iodine Number mg /gm	Methylene Blue mg /gm	Charcoal percentage %
corn leg & leaves	**0.510**	**1.020**	**0.689**	**110**	**11**	**14**
Empty corn ear	**0.488**	**1.019**	**0.692**	**100**	**10**	**8**
Wheat & Barley grass	**0.377**	**1.798**	**1.310**	**330**	**26**	**22**
herb residues	**0.373**	**1.794**	**1.303**	**326**	**25**	**17**
Selective Sawdust (beech and pine)	**0.320**	**1.152**	**2.062**	**943**	**121**	**29**
Paper drafts	**0.375**	**1.789**	**1.307**	**325**	**26**	**19**

Commercial Activated carbon from B. D. H [28]	**0.356**	**3.210**	**0.805**	**908**	**90**	

Many factors had been taken to prepare of activated carbon, such as, easy available, inexpensive, high density and with high carbon but low ash.

This study involved the successful preparing of activated carbon which confirmed by the Table (1) results, and it shows the all the raw materials are suitable to use but best one is (**beech and pine Sawdust**), which has 29%, and it is the highest carbon content between the feedstock. Moreover, and based on the obtained high density for the preparing activated carbon which was (0.496 gm/cm^3), indicates that coal has high adsorption and efficiency.

Scanning electron microscopy (SEM):

The scanning electron microscopy analysis (which available in faculty of education for pure sciences (Ibn al-Haytham)), was performed in this study. The SEM image (Figure 2) proves that, the prepared activated carbon contains more pores providing for adsorption.

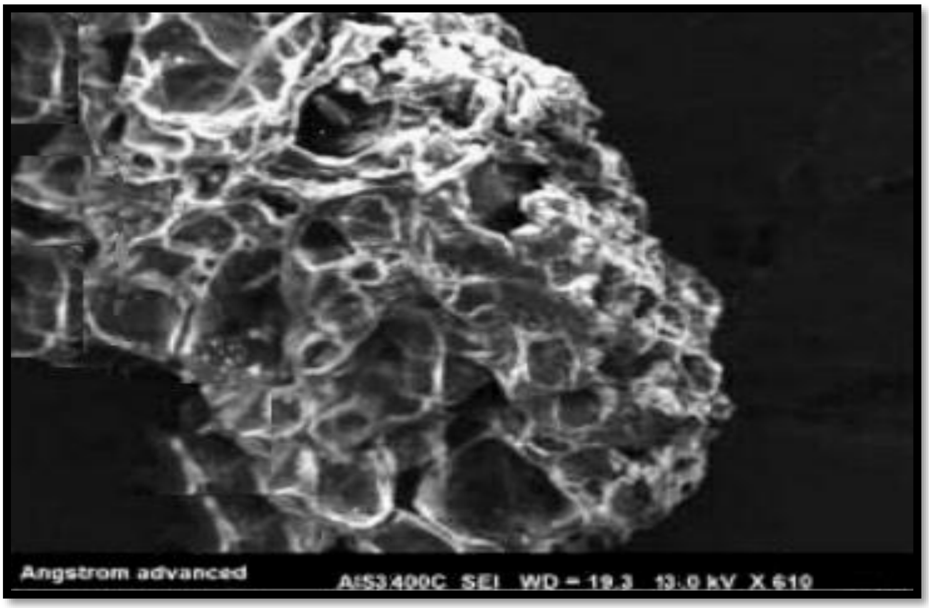

Figure (2): SEM image of activated charcoal sample**.**

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