Preparation Of Sulfurized Granular Activated Carbon From Beji Asphalt Using Concentrated H_2so_4

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Key words: sulfurized granular activated carbon, surface area, iodine number, volume activity**,** asphalt.

Abstract

In this investigation, sulfurized granular activated carbon of good quality was prepared from Beji asphalt by mixing with about 15% by weight of concentrated sulfuric acid (H_2SO_4). The overall yield of granular activated carbon was 80% by weight of asphalt. The activated granules have a total surface area (air-Permeability) of 4605 cm^2/g, iodine number of 965 mg/g, ash content of 5.3% maximum, hardness number of 99%, bulk density of 0.604 g/cm^3, and a volume activity of 582.9 mg/l. Such properties make the produced carbon suitable for waste water treatments and other applications.

Introduction

Activated carbon can be manufactured from many carbonaceous raw materials. High carbon content, low ash and low cost are raw material requirements. Wastes of various origins have been utilized for the production of activated carbons; such as sulfite liquors, lignin waste, and wastes resulted from processing petroleum and lubricating oils. Materials of vegetable origin and younger fossil fuels were some of the first materials used; wood, peat, sawdust, nut shells and fruit pits[1]. More recently, asphalt and bituminous coals have come into use. Hassler[2] (1974) mentioned 33 raw materials which have been used for the production of activated carbon. The final product characteristics depends on the raw materials, the type of manufacturing method - chemical or steam - and the manufacturing processes. Most of the raw materials and processes will allow the production of fine powdered carbons although product characteristics and costs will vary[3].

Asphalt is a sticky, black and highly viscous liquid or semi-solid that is present in most crude petroleums and in some natural deposits. There is some disagreement amongst chemists regarding the structure of asphalt, but it is most commonly modeled as a colloid, with asphaltenes as the dispersed phase and maltenes as the continuous phase. In U.S. terminology, asphalt (or asphalt cement) is the carefully refined residue from the distillation process of selected crude oils. Outside of North America, the product is called bitumen[4].

Due to the high pore volumes and adsorption capacities as well as their relative chemical inertness, activated carbons have been known as the most widely used adsorbents for liquid and gas phase treatments.

Activated carbon is a commonly used adsorbent in sugar refining, chemical and pharmaceutical industries, water treatment and wastewater treatment. The activated carbons is present in two well known forms, the powdered activated carbons (PAC) and granular activated carbons (GAC)[5].

The carbon activation process.[5] The carbon activation process creates or increases porosity on the carbon surface (Fig. 1). In reality, the surface of an activated carbon is not so simple. Pores are not idealized pits and crannies, and they can be more accurately portrayed as the imperfect junctions of carbon atoms at the surface of carbon units.

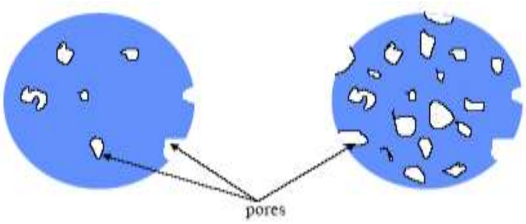

Figure (1): Two-dimensional representation of carbon activation[5].

Activation occurs by selectively removing carbon groups from the surface of carbons, creating more voids between carbon atoms which enlarge the pores, where molecules can be adsorbed or attaching functional groups to the surface of carbon. There are two conventional means of carbon activation: physical activation (or gasification) and chemical activation.

Physical Activation.

Physical activation processes involve the treatment of pyrolyzed carbonaceous materials at temperatures between 700-1000°C, where an oxidizing gas is used to induce gasification. Common activation gases include CO_2, steam, and air.

Chemical Activation.

The carbon-based raw material used in the chemical activation process is treated with a strong chemical agent, including but not limited to sulfuric acid, zinc chloride, phosphoric acid or potassium hydroxide, that dehydrates molecules in the raw material, and results in the formation of water that is removed from the raw material by moderate heat treatment. The activated carbon created by chemical activation has internal porosity developed primarily due to the action of the chemical dehydration agent[1].

Yan, Z. et al. [6], developed a method of manufacturing hard activated carbon pellets in which a lignocellulose material is chemically activated to form a char and heated to a temperature above about 360°C. The char is ground and then agglomerated in a pin mixer followed by pelleting the agglomerated acid char in the presence of an activatable binder. The pelleted hard activated carbon is then subjected to a final heat activation to yield a product with high density and activity.

Shawabkeh, R. et al. [7], disclosed a method of producing an activated carbon using fractured Pecan hulls, chemically activated in an acid solution, such as sulfuric acid, phosphoric acid, hydrochloric acid, etc. to provide an activated carbon capable of adsorbing large molecules, such as dyes, and ionic molecules, such as metal ions.

Hideharu, I. et al. [8] activated carbonaceous materials, to obtain activated carbonaceous materials with an oxidizable chemical, such as, zinc chloride, phosphoric acid, or sulfuric acid at a temperature greater than 100°C and less than 500°C.

Johnson, B. et al. [9], made granular activated carbon from dried, bituminous, sub-bituminous, and lignite coal by contacting the starting materials with mineral acids such as, hydrochloric acid, nitric acid and sulfuric acid. The amount and time used were sufficient to produce activated carbon with significantly higher Iodine, 750 or greater, and Carbon tetrachloride, 40.0 or greater, numbers.

Meng M., and Jiang, Z. [10], studied systematically the factors influenced the properties of activated carbon such as activating agent, additives, power of microwave and activating time by means of orthogonal test. The optimized factors of manufacturing activated carbon were obtained. Bagasse were infused 48 h in 15%～25% phosphoric acid, in which 2%～4% sulfuric acid and 1%～2% hydrochloric acid were added, and radiated 15～20min by microwave at 720W.The experiment showed that yield of activated carbon was 47.5%, methylene blue decoloring ability reached 15.5mL/0.1 g, and drying loss was 3.1%.

This study is aimed to produce an inexpensive and effective sulfurized granular activated carbon (SGAC) from asphalt for removing pollutants and heavy metals from drinking water using single step method by treating asphalt with concentrated H_2SO_4 (in presence of oxygen O_2) which decomposes asphalt into carbon, CO_2, and H_2O. Activation process occurred with the assistance of librated CO_2 and heat. These represent activating agents which most commonly used in physical activation.

By impregnating with concentrated H_2SO_4 more effectively sulfonated carbon atoms can be made at the active sites to increase porosity of activated carbon and increase its adsorptive capacity.

Experimental

Asphalt from Beji refinery was used for the preparation of activated carbon in this study. Some physical properties of that asphalt were mentioned in Table (1)and some chemical properties elementary composition and fraction composition of that asphalt and carbon produced were mentioned in table (2):

Table (1): Some physical properties of Beji asphalt used in the study

No.	Test	Result	ASTM	Range
1	Specific gravity	1.0		
2	Loss on heating,%	0.5		
3	Ash content,%	0.16		
4	Softening point, ºC	50	D449-79	40-60
5	Ductility, cm	100$^+$	D946-83	≥ 100
6	Flash point, ºC	290	D946-83	≥ 232
7	Solubility,%	99.6	D449-79	≥ 99

Table (2): Some chemical properties of Beji asphalt used in the study and the carbon produced .

No.	Elementary Composition of Asphalt		Fraction Composition of Asphalt		Composition of Carbon produced	
1	C, %	84.0	Asphaltene, %	9.5	C, %	83.80
2	H, %	9.5	Saturates, %	8.1	H, %	8.02
3	N, % + O, %	1.2	Naphthene Aromatics, %	65.5	N, % + O, %	1.68
4	S, %	5.3	Polar Aromatics, %	16.5	S, %	6.5

A- Preparation and Purification of Activated Carbon:

a) (200 g) of asphalt was heated to (90ºC) in an oven to simplify mixing.

b) (150 ml) of concentrated (98% ≡ 36N) sulfuric acid were added to the asphalt in three portions with continuous mixing until the starting of carbonization step, formation of foams and CO_2 gas was librated.

c) Asphalt was impregnated with strong dehydrating agent (concentrated H_2SO_4) for (4-5 hrs) at (25ºC), or for (3 hrs) at (120ºC) until the foams settled and sulfurized activated carbon was obtained.

d) The resultant activated carbon was washed with water many times till the washing water was neutral (pH=7), to remove the un-reacted excess acid.

e) The washed AC was dried by oven at (150ºC) for (1 hr).

f) The dried AC was grounded and sieved in mesh size range of (20-40 mesh) or (0.84-0.42 mm).

g) Then it was further activated thermally by electric furnace at (500°C) for (1 hr).

h) The sulfurized granular activated carbon was cooled to ambient temperature in wheaton dry-seal desiccator and packed with sealed bottle.

Figure (2), explain the process of producing sulfurized granulated activated carbon.

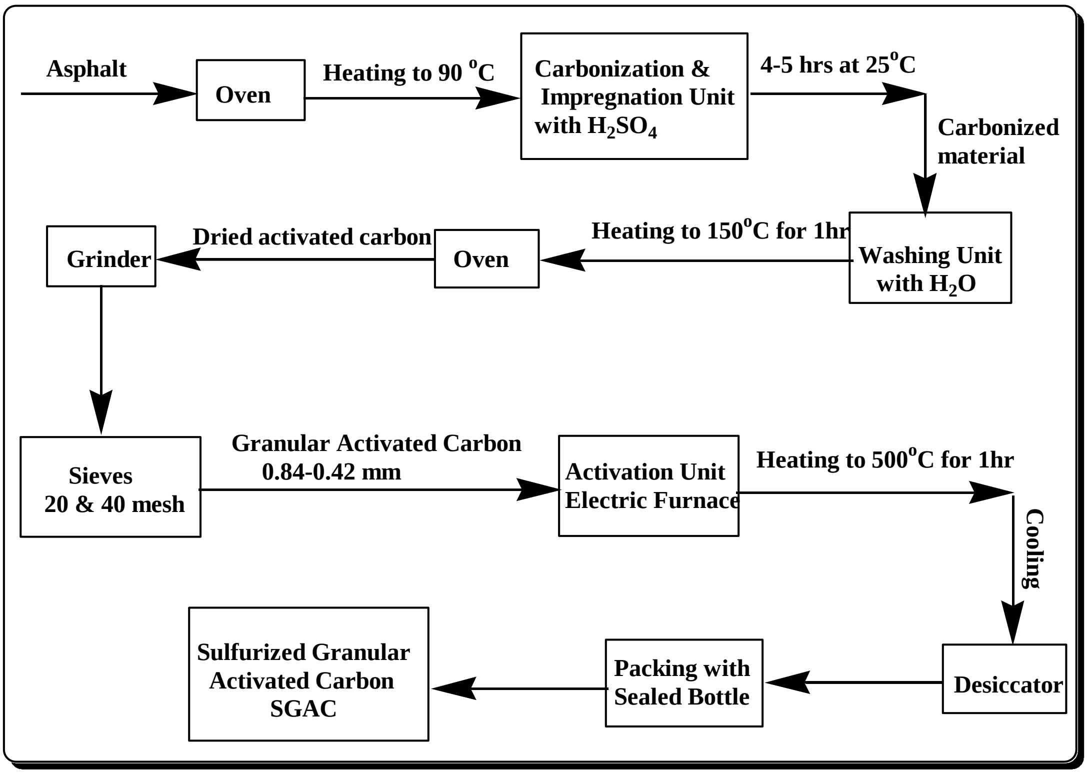

Figure (2): Flow Diagram for Sulfurized Granular Activated Carbon Production.

B- Activated Carbon Measurements:

1- Adsorption Properties:

1a- Determination of Carbon Activity by Iodine Adsorption Method (Iodine Number)

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

1 - One gram of dried activated carbon transferred to a (250 ml) Erlenmeyer flask.

2 - To the flask, (10 ml of 5%) HCl was added and swirled until the carbon is wetted.

3 - The flask was placed on hot plate to bring the contents to boil for exactly (30 s).

4 - The flask and content was allowed to cool to room temperature, then (100 ml) of standardized (0.1 N) iodine solution was added to the flask.

5 - The flask was immediately plugged and shaked vigorously for (30 min).

6 - The contents of the flask were filtered by gravity after shaking through filter paper.

7 - The filtrate was mixed in a beaker with a stirring rod and (50 ml) of the filtrate was transferred via pipette into (250 ml) Erlenmeyer flask.

8 - (50 ml) sample was titrated with standardized (0.1 N) sodium thiosulfate solution until the yellow color has almost disappeared. About (1 ml) of starch was added and the titration is continued until the blue indicator just disappear. The volume of sodium thiosulfate solution used was recorded. The standard test method for iodine number is ASTM D4607-94 [11].

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

Where:

m = weight of activated carbon in grams.

X = A – (2.2B × ml of thiosulfate solution used).

$A = N_1 \times 12693$

$B = N_2 \times 126.93$

N_1 = normality of iodine solution

N_2 = normality of sodium thiosulfate solution

D = correction factor.

1b- Methylene Blue Test for Determination of Carbon Activity (Maximum Adsorption Capacity and Specific Surface Area)

An exactly weight (0.1 g) of prepared activated carbon sample was added to an aqueous solution of (20 ppm) methylene blue pigment in a conical flask. The solution was shaked by an electrical shaker for (24 hr) at a temperature of (25°C) till adsorption of methylene blue from its aqueous solution was completed and a state of equilibrium was reached. The absorbance of the solution was determined using (CECIL-3021 Spectrophotometer) at (λ_{max} = 665 nm). The final concentration of methylene blue value was calculated as the number of milligrams of methylene blue adsorbed by one gram of activated carbon using calibration curve, between concentration of methylene blue (1–30 ppm) and absorbance, specially performed for this reason[12]. The Adsorption of methylene blue allows the determination of the specific surface area of activated carbon directly[13].

1c- Total External Surface Area (air-Permeability)

This method covers determination of fineness of a material, using the MIC-313 Blain air permeability apparatus, in terms of the specific surface expressed as total external surface area in square centimeters per gram of the material. This method represents a measurement of the external surface area of activated carbon as a function of the porosity of the bed. Its also a measure of external macropore structure (> 500ºA).

The standard test method for total external surface area (air-Permeability) is C204-78a[14].

2- Physical Properties:

2a- Measurement of Bulk or Apparent Density (A.D.)

The density of the prepared activated carbon was determined by weighing (10 cm^3) of the carbon sample using graduated cylinder The standard test method for apparent density is ASTM D2854-96 [15].

2b- Volume Activity (Volume Iodine Capacity)

Since granular activated carbons are used in adsorbers of fixed volume, apparent density values can be used to calculate volume activity, which may help determine the work capacity of an adsorber with alternative carbon loadings. For example, assume that carbon A adsorbs iodine to produce a standardized Iodine Number of 1100 mg/g., and has an A.D. of 0.4 g./cc. Carbon B has an Iodine Number of 950 mg./g and an A.D. of 0.5 g./cc. Multiplying the A.D. by the weight basis activity value, carbon A has a volume iodine capacity of 440 mg./cc. while carbon B has a value of 475 mg./cc. Therefore, carbon B, which has lower activity, might actually do more work and therefore have a longer service life than carbon A of an equal volume. If the price of carbon B permitted filling a given adsorber with the greater weight required, it could thus be the most economical of these adsorbents on a net cost basis[16].

2c- Hardness Number

The harder the activated carbon, the less it will crumble into fine particles during handling and use. The fine particles would then escape from the system, resulting in losses. To define this property, a certain amount of activated carbon is put into a pan, together with some steel balls, and shaken for a defined period of time. That is why this property is often known as Ball-Pan Hardness. Weighing the carbon before and after the shaking determines the amount of loss. The % of the original carbon that is left after the shaking is the Hardness Number. 95% and above is a good Hardness Number. The standard test method for Ball-Pan Hardness of activated carbon is ASTM D3802-79[17]

2d- Measurement of Humidity

One gram of the activated carbon was heated in an oven at (150ºC) for (3 hr). The difference in weight before and after heating operation was calculated as percentage H_2O vapor in the sample which represent a practical limit for the level of moisture in activated carbon· The standard test method for moisture is ASTM D2867-83 [18].

3- Determination of Total Ash Content

The ash content was measured by heating one gram of the dry prepared activated carbon sample in a porcelain crucible using electrical furnace for five hours at temperature of (1000 - 1100ºC). The remained residue was considered as the ash content expressed as a percent of the original weight of activated carbon. The standard test method for total ash content is ASTM D2866-94 [19].

4- pH Value

The pH Value of activated carbon is a measure of whether it is acidic or basic. Activated carbon, when coming out of the kiln tends to be a bit basic, with a pH of around 8. Too high pH indicates too much contaminant. To low pH, especially for acid washed activated carbon, means that the acid has not been properly rinsed away. Most activated carbon is specified for a pH of (6-8).

The standard test method for a pH of activated carbon is ASTM D3838-83[20].

5- Particle Size Distributions

Particle size distributions are important in carbon systems because they influence handling of the activated carbon material. For example, in granular carbon, the particle size affects hydraulic loading and backwash rates for a filter. On the other hand, particle size is often important because of its effect on adsorption rates as well. The standard test method for Particle size distributions is ASTM D2862-82 [21].

6- Effective Size

The effective size is the diameter for which 10% by weight of granules are smaller. It is an indicator of the filtration and pressure drop performance of the carbon. A lower effective size will filter smaller particles from the influent water and have a greater pressure drop. The standard test method for effective size is ASTM D2862-97[22].

RESULTS & DISCCUSION

Extensive research activities are in progress worldwide to increase the adsorption capacity and selectivity of various activated carbons. There are two very well known techniques to produce activated carbons namely gaseous or physical and chemical activation methods. In the chemical activation technique, due to the contribution of water to decompose the chemical structure of the raw material, the activation may be achieved in a lower temperature. Furthermore, the chemical activating agent improves the micropores formation and increases the surface area to a large extent.

Activated carbon is made up of random amorphous graphite plates which give the carbon its very large surface area and unique property of adsorbing many different compounds. However, this structure is difficult to characterize, especially for obtaining information for the design of carbon adsorption systems. In addition, differences in the carbon structure can make a very large impact on the performance of activated carbon for a particular application.

Activated carbon in this investigation was produced by impregnation

with concentrated sulfuric acid as a doping agent. Therefore carbonization and activation were carried out at the same time. Activated carbon so produced generally exhibits a very ‘open’ pore structure, ideal for the adsorption of large molecules.

The results of the study and comparison with commercial activated carbon are shown in Table(3).

Total Mass Loss and Yield: Total mass losses and yield were determined after sample processing. Total mass loss is defined in terms of the raw material mass:

$$\textbf{Total Mass Loss Percent} = \frac{\mathbf{M_{raw} - M_{processed}}}{\mathbf{M_{raw}}} \times 100$$

and yield is the total process yield based on raw material mass:

$$\textbf{Yield} = 100 - \left(\frac{\mathbf{M_{raw} - M_{processed}}}{\mathbf{M_{raw}}} \right) \times 100 = 100 - \textbf{Total Mass Loss Percent}$$

Hardness/Abrasion Number:

Hardness Number is a measure of activated carbon's resistance to attrition, and it is an important indicator of activated carbon to maintain its physical integrity and withstand frictional forces imposed by backwashing etc. There are large differences in the hardness of activated carbons, depending on the raw material and activity level, also it is affected by the granulometry for granular carbon. The hardness number is a better indicator for gas phase applications.

Iodine Number:

It is the most fundamental parameter used to characterize activated carbon performance. It is a measure of activity level (higher number indicates higher degree of activation). It is a measure of the micropore content of the activated carbon (0 to 20 Å, or up to 2 nm) by adsorption of iodine from solution. It is equivalent to surface area of activated carbon between 900 m^2/g and 1100 m^2/g. It is the standard measure for liquid phase applications. In general, the higher the iodine number, the higher the activity, the higher the internal surface area.

Methylene Blue:

The methylene blue number is based on how much a sample of activated carbon can de-colorize a solution of this aromatic dye and is another indicator of adsorption capacity. It is a measure of mesopore structure (20 – 500Å). It was observed that when the equilibrium concentration of methylene blue is more than (20 ppm), there is a decrease of methylene blue adsorption in mg/g, this may be due to the desorption of the methylene blue molecules. A schematic of the interaction between the methylene blue molecule and SGAC surface is shown in Figure (3). It was assumed that there is a complete adsorption of methylene blue as a mono layer into the surface of SGAC as shown by the plateau of the adsorption isotherm.

Bulk Density:

Higher density provides greater volume activity and normally indicates better quality activated carbon. The density is very dependent on the raw material used in the production of the activated carbon. For example, wood based carbons have a lower density than coal based carbons. As the carbon adsorbs compounds, the density increases and therefore it is useful for determining the degree of saturation of exhausted carbons and the effectiveness of reactivation when the density goes down again. The apparent density is measured by allowing the carbon to fall into a measuring cylinder at a fixed rate from a vibrating tray to allow settling of the granules. The apparent density is useful in sizing adsorbers for gas phase applications.

Table (3): Properties of Activated Carbon

No.	Technical Specifications	Prepared GAC	Commercial AC	Test Method
1	Yield of activated carbon, % (min.)	80		
2	Iodine No., mg/g, (min.)	965	1100	ASTM D4607-94
3	Maximum adsorption capacity (q_{max}), (by methylene blue) mg/g, (max.)	165.94	618	
4	Specific Surface Area (by Methylene Blue), m^2/g, (max.)	526. 8	1962	
5	Total Surface Area (air-Permeability), cm^2/g, (min.)	4605.4	4832.6	ASTM C204-78a
6	Bulk Density, g/cm^3 or g/ml, (avg.)	0.604	0.343	ASTM D2854-96
7	Hardness, %, (min.)	99	91.5	ASTM D3802-79
8	Moisture Content, %, (max.)	3.07	3.54	ASTM D2867-83
9	Total Ash Content, %, (max.)	5.3	1.5	ASTM D2866-94
10	pH, (avg.)	8.1	6.5	ASTM D3838-83
11	Mesh size (U.S. Sieve series, 20*40), %, (min.) Less than No. 20, % (max.) Greater than No. 40, % (max.)	85 5 10		ASTM D2862-82
12	Effective Size	0.635-1.270 A or 0.417-0.833 B		ASTM D2862-97
13	Volume Activity (Volume Iodine Capacity), mg/l	582.9	377.3	

Ash Content:

It reduces the overall activity of activated carbon. It reduces the efficiency of reactivation. The metal oxides (Fe_2O_3) can leach out of activated carbon resulting in discoloration. In drinking and waste water applications, the ash content is a useful indicator of whether mineral have deposited on the carbon during operation. This can include aluminum or iron coagulants or calcium carbonate coming out of solution. In gas phase applications such solvent recovery, certain ash constituents can assist in the decomposition of organic compounds such as acetone to acetic acid which makes recovery of the product more difficult.

The surface area and porosity values are considerably high due to the sulfur enrichment which is significant. This suggests that most of the pores are reacted with (-SO_3H) and enlarge their sizes but not clogged with sulfur atom. This carbon is called sulfurized activated carbon (SAC). This would be expected to alter the adsorption properties of the carbons due to the electrostatic effects which promote formation of hydrogen linkage. Some fundamental measured IR absorption frequencies of sulfurized activated carbon are given in Table (4). A suggested schematic of the interaction occurred between methylene blue molecule and sulfurized activated carbon surface (C-SO_3H) for this work is shown in Fig. (3).

From Table (3), it was shown that the volume activity of the prepared SGAC is higher than that of the commercial activated carbon, which indicates that the commercial AC actually do more work and therefore have a longer service life than prepared SGAC of an equal volume.

Table (4): Some fundamental measured IR absorption frequencies of sulfurized activated carbon.

No.	Band position cm-1	Possible assignments
1	1167	C=S Stretching, small, sharp and doublet due to the sulfur groups (-SO_3H) bonded to activated carbon
2	1111	S=O Stretching
3	640	S-S Stretching.
4	2925	C=C-H Stretching, doublet, sharp and small
5	2854	C=C-H Stretching, singlet, sharp and medium
6	2940	OH Stretching, weak, and very broad-bonded

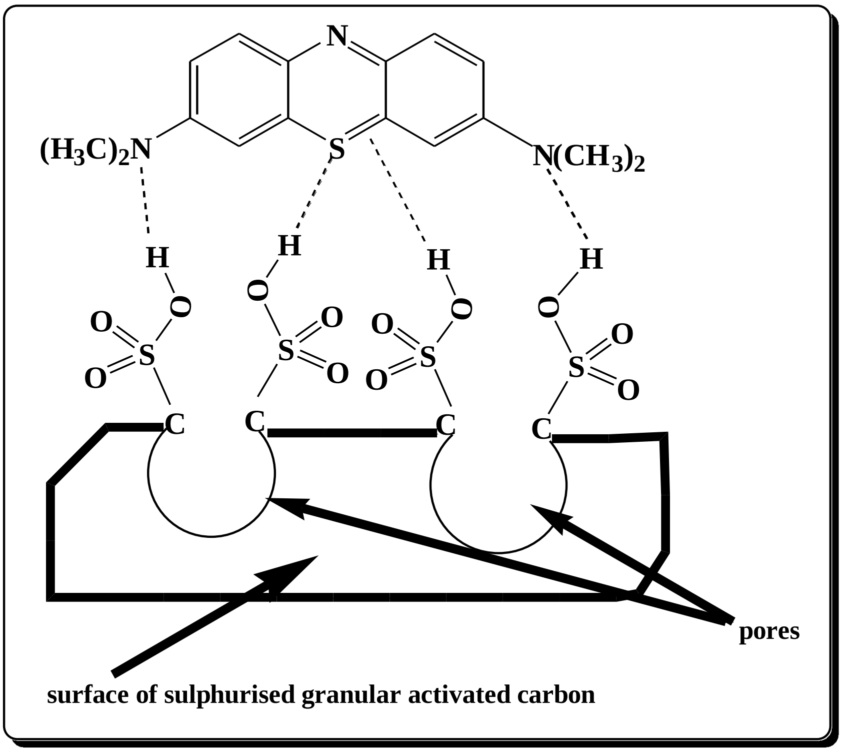

Figure (3): Suggested schematic model of methylene blue and sulfurized granular activated carbon.

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تحضير كاربون حبيبي منشط مكبرت من أسفلت بيجي باستخدام حامض الكبريتيك المركز

أروى محمد عثمان

قسم الصناعات الكيمياوية ، المعهد التقني–الموصل ،الموصل ، جمهورية العراق

الملخص:

في هذه الدراسة تم تحضير كاربون حبيبي منشط مكبرت ذو نوعية جيدة من أسفلت بيجي بواسطة المزج مع (١٥%) وزنا من حامض الكبريتيك المركز. نسبة الناتج الكلية كانت (٨٠%) من المادة الأولية المستخدمة، المساحة السطحية الكلية بطريقة نفاذية الهواء كانت (٤٦٠٥ cm^2/g)، الرقم اليودي (965 mg/g)، محتوى الرماد (٥,٣%) كحد أعلى، رقم صلادة ٩٩%، الكثافة الكلية (0.604 g/cm^3) ، وفعالية حجمية قيمتها (582.9 mg/l). هذه المواصفات تجعل هذا الكاربون المحضر مناسبا لعمليات معالجة مياه الفضلات والاستخدامات الأخرى.