Preparation Of Phosphorized Granular Activated Carbon From Beji Asphalt Using Concentrated H_3po_4

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Abstract

In the present study, the activation features of phosphoric acid have been investigated using asphalt as the raw material in the production of granular activated carbon. The quality of the prepared carbon was tested by measuring its iodine number and ability to adsorb methylene blue. The density, moisture content, hardness, effective size, and total ash content were also evaluated. The obtained results were compared with a carbon of a commercial grade supplied by the BDH chemical company. The comparison proved that the prepared carbon is of a good quality and comparable to the commercial one.

Introduction

Activated carbon has several important uses including solution purification (as in the clean-up of cane,beet and corn-sugar solutions), removal of tastes and odors from domestic and industrial water supplies,vegetable and animal fats and oils,alcoholic beverages,chemicals and pharmaceuticals and in the waste water treatment. It also finds use in purification of gases, liquid phase recovery, separation processes, petroleum industries,mining and as catalyst and catalyst supports[1].

The source of the most commonly used activated carbon in commercial practice is peat, asphalt, bituminous coals, lignite, wood, resins, used tires and coconut shell. In recent years, many different agricultural residues were used to produce activated carbon, e.g. palm stone, rice hulls, rice straw, sugarcane bagasse, pecan shells, and from natural precursors (jute and coconut fibers)[2]. On the other hand agriculture by products are lignocelluloses wastes that may offer an inexpensive additional source of activated carbons[3].

One of the most important physical properties of activated carbon is the surface area. Activated carbons have an extensive internal pore structure that presents a high surface area available for adsorption. Figure (1) presents an artist's rendition of the cross section of an activated carbon particle. Note that almost all of the surface area available for adsorption is associated with its internal pore structure. Also note the relative change in pore diameters, going from very large at the granule surface boundary, to much smaller within the particle interior[4].

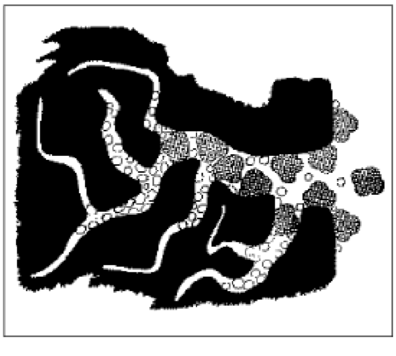

Figure (1): An artist's rendition of the cross section of an activated carbon particle[4].

Usually the surface properties of the produced activated carbon are affected by the starting material and activation scheme. The effect of starting material is due to the fact that carbon materials present important differences in their properties and their reactivity. Activated carbons are produced by three methods, physical, chemical and combined (physical and chemical) procedures[1]. Physical activation involves carbonization (thermal treatment) of raw materials at 950°C in an inert atmosphere followed by an activation step with inert gas like (CO_2, or N_2) at the same temperature, whereas in chemical activation, the raw material is impregnated with chemical reagent such as, KCl, $ZnCl_2$, or H_3PO_4 then heat-treated at moderate temperatures. In general, chemical activation processes are carried out at lower temperatures and shorter time than physical activation, which may benefit the development of a microporous structure. In general the residues from carbonization and activation have a large total pore volume and a high internal surface area of the range between 300 and 2500 m^2g^{-1}[5].

Asphalt, which is the raw material in this work, 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[6].

Massoud R. et. al.[7], produce low-cost adsorbent carbons from Illinois coal and evaluate the suitability of these materials for natural gas storage. Granular (20-100 mesh) activated carbons were produced by chemical activation with H_3PO_4,. The product was characterized for its BET surface area, micropore volume, bulk density, and gravimetric/volumetric methane adsorption/storage capacities.

Baksi, S. et. al. [8] carried out an investigative study on converting waste bamboo to produce usable activated carbon By impregnating with H_3PO_4 doping agent.

Ichcho, S. et al. [9], studied the use of Moroccan oil shale for the preparation of adsorbents by chemical activation with phosphoric acid. The results indicate that this material is promising for this application. The effect of different conditions of preparation on the yield and surface area is discussed. These parameters are H_3PO_4/shale weight ratio, carbonization temperature, carbonization time and concentration of H_3PO_4.

Marit J. [10] showed that the reaction of a lignocellulosic precursor with a strong chemical reagent, such as phosphoric acid (H_3PO_4), at temperatures up to 350ºC or above, can lead to the formation of a porous carbon. Depending on the reagent, its concentration, and the carbon precursor material, the carbon will possess a highly developed internal pore structure and be an effective adsorbent for molecules in the gas or liquid phase. The objectives were to improve the understanding of the relationship between activated carbon properties and the structure of the precursor, and to clarify the reaction chemistry and mechanisms.

Gonzalez-Serrano[11], showed that activated carbons with a high BET surface area and a well-developed porosity have been prepared from pyrolysis of H_3PO_4-impregnated lignin precipitated from Kraft black liquors. Impregnation ratios within the range of 1-3 and activation temperatures of 623-873K have been used, giving rise to carbons with different porous and surface chemical structure. Increasing the activation temperature and the impregnation ratio leads to a widening of the porous structure with a higher relative contribution of mesoporosity.

Tawalbeh, M. et al. [12], experimentally investigated the production of chemically activated carbon from Jojoba seed residue using a laboratory-scale static bed reactor. The effects of process variables such as activation time, activation temperature, particle size, chemical reagents (KCl, $ZnCl_2$ and H_3PO_4) and impregnation ratio on adsorption capacity of activated Jojoba seed residue were studied.

Kim D.S. [13], In his study, investigated the activation features of phosphoric acid using waste peach stones as the raw material in the production of granular activated carbon. The activated carbon produced was found to be as good as that of the commercial ones.

Phosphoric acid activation has been applied to coconut shell, coals, hardwoods, shells of nuts like almond, pecan, black walnut, macadamia nut, cotton stalks, and palm date pits[14].

The present work investigated the feasibility of producing phosphoric acid impregnated granular activated carbon using asphalt, for liquid and gaseous pollutant control. The strong chemical agent– phosphoric acid–dehydrates molecules in asphalt 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. It is believed that carbonization and activation step proceeds simultaneously in chemical activation.

EXPERIMENTAL

In this study, asphalt from Beji refinery was used for the preparation of activated carbon. 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:

Preparation steps of phosphorized granulated activated carbon PGAC are as followed:

1. (24 g) of asphalt or bitumen were carbonized on a burner till all evolved gases are finished.
2. (15 ml) of concentrated ortho phosphoric acid was added (dropwise) with mixing and heating at (300-400ºC) for about (30 min) until the foams were settled down.
3. The phosphorized carbon must be water washed to remove any residual phosphoric acid. This was done by washing the carbon with water many times till the washing water became neutral (pH=7). The cooled carbon was then dried in oven at (200ºC) for (2-3 hrs).
4. The phosphorized carbon was then grinded and sieved to (20-40) mesh, (0.84-0.42 mm).
5. Activation of phosphorized granular carbon was accomplished thermally in furnace at (500ºC) for (1 hr).
6. The final activated carbon was cooled in a desiccator to the ambient temperature and packed in a sealed bottle.

Figure (2), shows the flow diagram of producing phosphorized granulated activated carbon (PGAC).

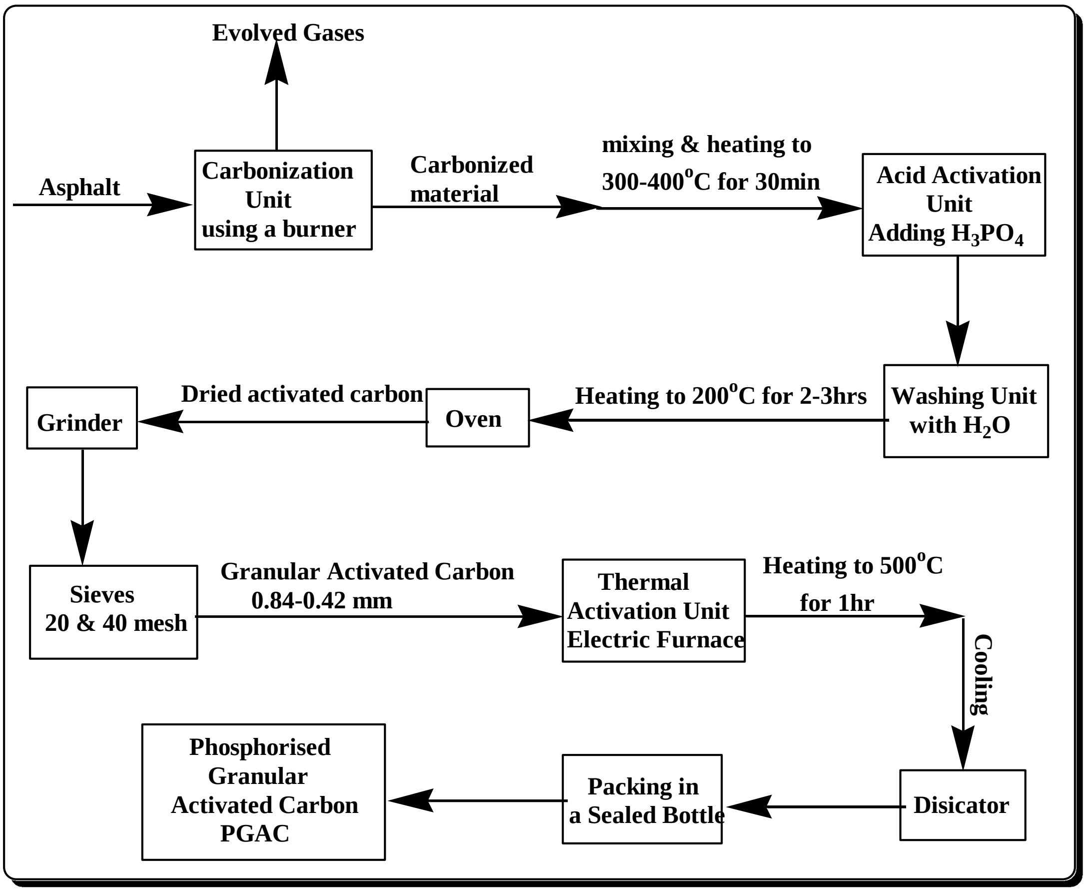

Fig (2): Flow Diagram for phosphorized Granular Activated Carbon Production.

B- Activated Carbon Measurements

1- Adsorption Properties:

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

Iodine number (IN), is the amount (in milligrams) of iodine adsorbed from its aqueous solution by one gram of activated carbon[15]. The standard test method for iodine number is ASTM D4607-94 [15].

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

An exact 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 (25ºC) 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[16] .

The Adsorption of methylene blue allows the determination of the specific surface area of activated carbon directly[17].

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

The standard test method for total external surface area (Air-Permeability) is C204-78a[18].

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 [19].

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 to determine the working capacity of an adsorber with alternative carbon loadings[20].

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. The standard test method for Ball-Pan Hardness of activated carbon is ASTM D3802-79[21] .

2d- Measurement of Humidity

The standard test method for moisture is ASTM D2867-83[22].

3- Determination of Total Ash Content

The standard test method for total ash content is ASTM D2866-94 [23].

4- pH Value

The pH Value of activated carbon is a measure of whether it is acidic or basic. The standard test method for a pH of activated carbon is ASTM D3838-83[24] . The pH value was measured with (analysts omnimeter model OM-1B).

5- Particle Size Distributions

A standard test procedure for particle size distribution (PSD) is defined in ASTM D2862[25]. Particle size distributions are important in carbon systems because they influence handling of the activated carbon material.

6- Effective Size

The effective size is an indicator of the filtration and pressure drop performance of the carbon. The standard test method for effective size is ASTM D2862-97[26].

RESULTS & DISCCUSION

In the manufacture of activated carbons, a wide variety of raw materials and widely varying quality specifications are used. While the raw material itself determines many of carbons physical properties, its adsorption capacity is dependent on a precise and carefully controlled activation process. The most popular aqueous phase carbons are bituminous based because of their hardness, abrasion resistance, pore size distribution, and low cost, but their effectiveness needs to be tested in each application to determine the optimal product[2]. Activated carbon products can be characterized by physical properties and activity properties. Important physical properties are surface area, product density; mesh size, abrasion resistance and ash content. The results of the study and comparison with commercial activated carbon are shown in Table (3). It is inferred from the results obtained that the acid treatment of the asphalt starting material slows down the activation rate and allows the asphalt sample being activated to develop a larger capacity for iodine and methylene blue adsorption. It would appear that the mechanism for this increased capacity is related to an increased activation of the inner pores accompanied by reduced surface oxidation. While it is theorized that acid treatment of the asphalt starting material may remove compounds which would catalyze or promote rapid oxidation during activation, or that the acid treatment may add or replace acid groups normally expected to slow the rate of activation[13].

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:

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

Table (3): Properties of Activated Carbon

No	Technical Specifications	Prepared GAC	Commercial AC	Test Method
1	Yield of activated carbon, %(min.)	60		
2	Iodine No., mg/g(min.)	1103.3	1100	ASTM D4607-94
3	Maximum adsorption capacity (q_{max}), (by methylene blue) mg/g (max.):	562.08	618	
4	Specific Surface Area (by Methylene Blue), m^2/g (max.):	1784.62	1962	
5	Total External Surface Area (air-Permeability), cm^2/g (min.):	4695.4	4832.6	ASTM C204-78a
6	Bulk Density, g/cm^3 or g/ml (avg.):	0.513	0.343	ASTM D2854-96
7	Hardness, % (min.):	90	91.5	ASTM D3802-79
8	Moisture Content, % (max.):	5	3.54	ASTM D2867-83
9	Total Ash Content, % (max.):	7.4	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	566	377.3	

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

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

Yield percent in this work is 60% minimum, which is fair enough for bitumen starting material.

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. The hardness number of the prepared PGAC is good as compared with the commercial one.

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 28 Å, or up to 2.8 nm) by adsorption of iodine from solution.It is equivalent to surface area of activated carbon between 900 m²/g and 1100 m²/g. It is the standard measure for liquid phase applications. In this investigation the iodine number is almost equal to the commercial activated carbon which means it has high activity.The iodine number is also a measure of the volume present in pores up to 28Å in diameter. Carbons with a high percentage of pore sizes in this range would be suitable for adsorbing lower molecular weight substances from water. Carbons with a high iodine number are the most suitable for use as vapor phase carbons, as water molecules tend to effectively block off and isolate pore sizes less than 28 Å.

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 (30–500 Å). 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. Methylene blue number of prepared PGAC is less than that of the commercial one which means a decrease in mesopore structure. A schematic of the interaction between the methylene blue molecule and

PGAC surface is shown in Figure (4). . It was assumed that there is a complete adsorption of methylene blue as a mono layer into the surface of PGAC 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. Here, the bulk density is greater than that of the commercial carbon, this may be due to use asphalt the heavy substance as the raw material. 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.

Ash Content:

It reduces the overall activity of activated carbon, and reduces the efficiency of reactivation. The high ash content of the prepared GPAC may be due to the minerals and oxides present in the starting material (asphalt).

Particle Size Distribution:

Generally, the rate of adsorption will increase as the particle size decreases, as the process step of diffusion to the carbon surface should be enhanced by the smaller particles. Note that another critical aspect of rate of adsorption is the pore size distribution, and development of “transport pores” within the particle that allow effective migration of contaminants to the point of adsorption. However, particle size may not be that important in all cases, as the porous nature of the carbon particles results in large surface areas in all sizes of carbon particles. Head loss through a carbon bed increases as the carbon particle size decreases and as the uniformity coefficient increases. The surface area and porosity values are considerably high due to the phosphorus enrichment which is significant.This suggests that most of the pores are reacted with (-PO_3H_2) and enlarge their sizes but not clogged with phosphorus atom. This carbon is called phosphorized activated carbon (PAC). This would be expected to alter the adsorption properties of the carbons due to the electrostatic effects which promotes formation of hydrogen linkage. Some fundamental IR absorption frequencies of phosphorized activated carbon are given in Table (4) and it's IR chart is given in Fig(3). A suggested schematic of the interaction occurred between methylene blue molecule and phosphorized activated carbon surface (C-PO_3H_2) is shown in Fig. (4).

Table (4): Some fundamental IR absorption frequencies of phosphorized activated carbon

No.	Band position cm^{-1}	Possible assignments
1	1173	C-P Stretching, small, sharp and doublet due to the P groups (-H_2PO_3) bonded to activated carbon
2	960	P=O Stretching
3	800	P-O-P Asymmetric stretching
4	925	P-OH Bending, small and sharp
5	640	P-P Stretching
6	2320	C=C-H Stretching, doublet, sharp and small
7	2730	C=C-H Stretching, singlet and medium
8	2920	O-H Stretching, weak, and very broad-bonded

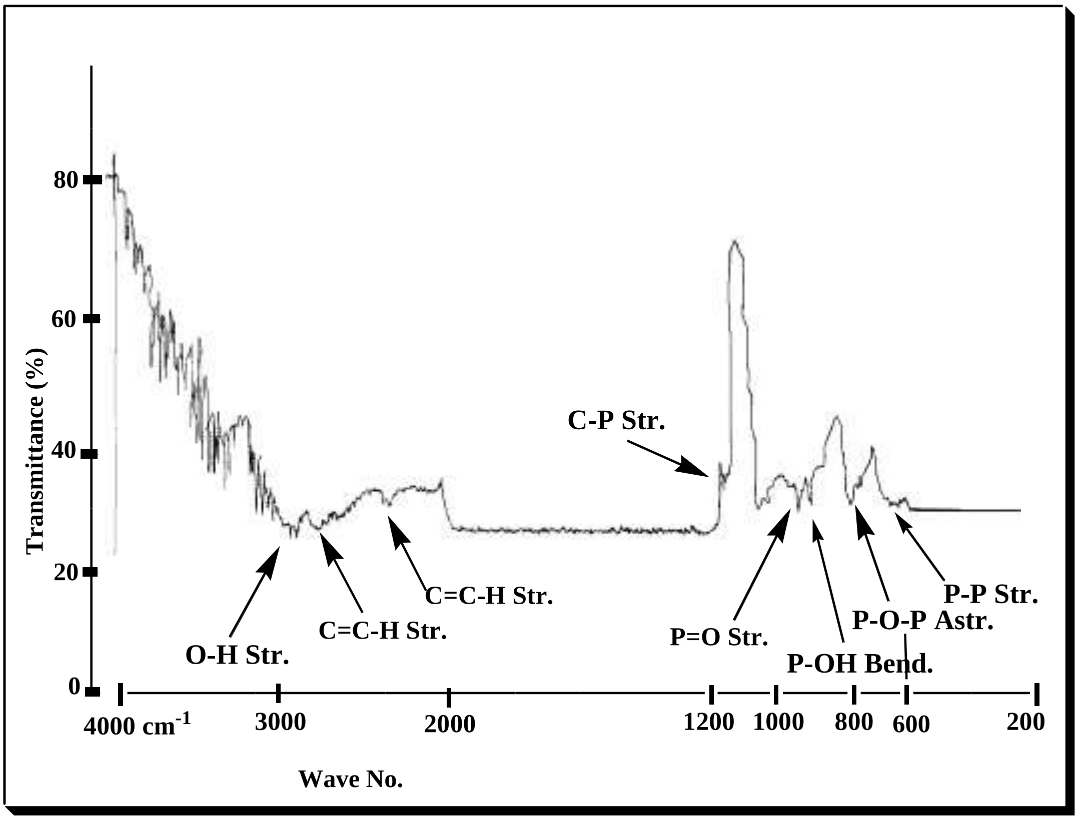

Fig (3): IRChart for phosphorized Granular Activated Carbon Production.

Infrared investigations showed that carbons activated with phosphoric acid may be regarded as prospective cation exchangers for the removal of heavy metals from water solutions. The acidic properties of synthetic phosphoric acid activated carbons may be related to phosphorus-containing compounds formed during carbonization. It should be stressed that the acidic character of synthetic chemically activated carbons cannot be ascribed to an excess of phosphoric acid remaining in pores after carbonization, because the carbons were extensively washed with water until a neutral pH was attained. Typically, such a treatment lasted for (0.5 – 1 hr). Also, oxygen-containing surface groups of acid character (carboxylic, phenolic) should not be ruled out as possible contributors to the cation-exchange properties of synthetic phosphoric acid activated carbons. Although the most acidic oxygen-containing groups are known to begin to decompose at temperatures above (250ºC) and thus would not withstand carbonization at (500ºC), some acidic groups might be formed as a result of oxidation induced by self-heating and/or subsequent hydrolysis after the cooled samples were exposed to air. Infrared spectroscopy provides information on the chemical structure of the adsorbent material[27].

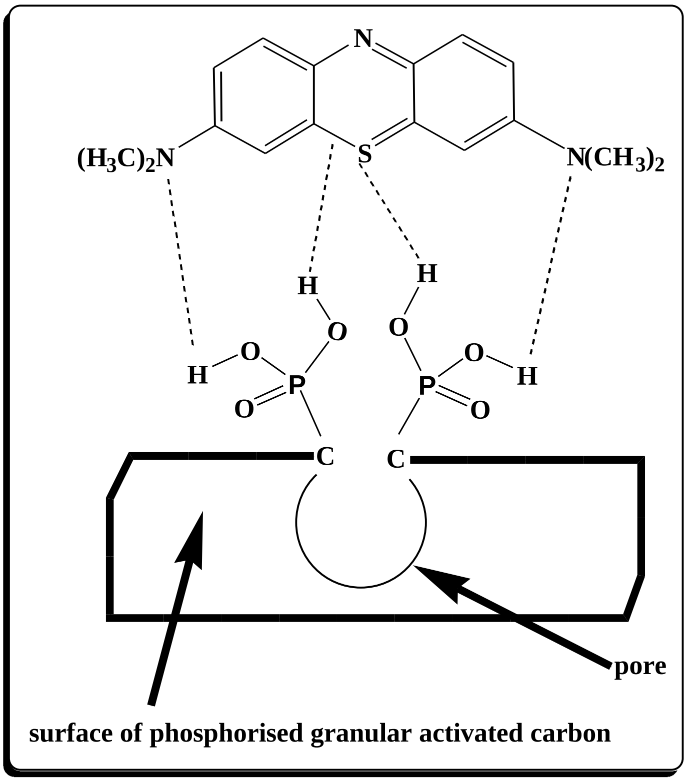

Figure (4): Schematic model of methylene blue and phosphorized granular activated carbon

The IR spectra show absorption band at (2920 cm^{-1}) which assigned to the O–H stretching mode of hydroxyl groups and adsorbed water. The position and asymmetry of this band at lower wave numbers indicate the presence of strong hydrogen bonds. The IR spectrum shows absorption bands due to aliphatic (2320 cm^{-1}) and aromatic structures (2730 cm^{-1}). The absorption peak at (1173 cm^{-1}) reported the appearance of C-P bonds in carbon obtained from activation by phosphoric acid, this peak was assigned to phosphates. P-OH bending, P-O asymmetric stretching, and P=O stretching were shown in phosphate-carbon complexes[27].

From Table (3), it was shown that the volume activity of the prepared PGAC 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 PGAC of an equal volume.

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تحضير كاربون منشط حبيبي مفسفر من أسفلت بيجي باستخدام حامض الفسفوريك المركز

رافع رشدي محمد و إعتماد إدريس طه و أروى محمد عثمان

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

الملخص:

تم في هذه الدراسة تحري ميزات استعمال حامض الفسفوريك المركز لتحضير كاربون منشط حبيبي من الإسفلت كمادة أولية. نوعية وجودة الكاربون المحضر اختبرت بواسطة قياس العدد اليودي وقابلية امتزاز صبغة المثيلين الزرقاء. تم أيضا حساب الكثافة، محتوى الرطوبة، الصلادة، الحجم المؤثر ومحتوى الرماد الكلي. نتائج الفحوصات المستحصلة للكاربون الحبيبي المفسفر تمت مقارنتها مع مثيلاتها **من** المواصفات لكاربون تجاري مجهز من قبل شركة BDH. المقارنة أثبتت أن الكاربون المحضر يمتلك مواصفات جيدة مقارنة بالكاربون التجاري.