

## Preparation Activated Carbon from Biji Refinery Asphalt Treated with Sulfur and Waste Polymers

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### Abstract

The objectives of this work were to develop a modified method to produce activated carbon from system (Asphalt: Polymer). The activated carbon prepared by treated asphalt with two kind of waste polymer in presence sulfur, under fixed laboratory conditions of temperature and time as well as certain percentages of polymers additives, used potassium hydroxide to process primary carbonization and solution (10%) hydrochloric acid to remove any trace of the ions and reduction of the metal components. observed improved adsorption properties of activated carbon prepared by use the polymers compared with model activated carbon prepared from pure asphalt material. Many tests have been conducted on prepared activated carbon such as determining the activity of the samples gained through adsorption measure against the aqueous solutions for iodine and methylene blue. The mechanical resistance has been measured for some prepared samples rather than achieving some other measures such as density, ash content and humidity.

**Keyword:** Activated carbon, adsorption, waste polymers.

### INTRODUCTION

According to the great importance of the activated carbon and its use in the field of industry and pollution control, so it has been prepared by different means because no certain form of activated carbon exists which can be used for all purposes. Activated carbon is known as a material with a high content of Carbonic and structure of follicular various, making it the active structure toward the adsorption of chemicals from their midst [1]. Activated carbon also known as the class of carbonate material with the structure of a sophisticated and varied of follicular with large surface area, Activated carbon has different types of pores vary in size and forms when compared to the size of the pores almost fixed in the zeolite [2].

The acquisition of reactivated carbon of these qualities made it a material with a high adsorption capacity for many of the chemical materials. The ability of activated carbon known to remove colored substances from solutions since the fifteenth century, however, this property is not used on an industrial scale, but at the end of the fifteenth century when it was used in the sugar industry in the UK and has expanded the use of

this material to include the kinds of industrial and chemical processes [3, 4]. Activated carbon has a broad distribution in the size of pores and types, although the form of these pores changed when compared to the size of the fixed pores in Zeolite and activated carbon is generally different from the others porous materials such as silica gel and alumina some of the most important attributes: -

1. Activated carbon containing all types of Pores from large and medium that can be observed in electron microscope and even micro size that participate in the adsorption process.
2. The non-polar of the activated carbon lead to link of adsorption process and to a large extent the nature of the porous compositions of the material (Porosity) [3, 5].

The activated carbon can be classified into several types: -

- A. Powdered Activated Carbon Properties of carbon activated powdered depends on the type of raw material and the process of carbonization and method of activation, although the size of particles carbon between (50-100) nm almost and radius ranging between (15-75) Micrometers, as well as having large internal surface area [6].
- B. Granulated Activated Carbon is used for adsorption of gaseous materials and vapors because it allows the passage of gas through the layers is also used as supporting material in the production of catalysts [7].
- C. Spherical Activated Carbon this type of prepared in the form of balls as it softens the asphalt by using naphthalene and turn asphalt into balls by effect of soaking and the presence of naphthalene, then these balls are placed in contact with naphthalene distilled solution at atmospheric normal pressure and that work on the extraction of naphthalene from these balls and formation of porous structures, the balls are heated to temperatures between (100-400) °C with presence of oxidizing gas (CO<sub>2</sub>), or water vapor, and then raise the degree of heating temperature to (700) °C for the purpose of activated it. This type of Carbon is characterized by high mechanical resistance and high adsorption capacity for nitrogen oxides and sulfur [8].
- D. Plated Activated Carbon this type of activated carbon uses in adsorption sulphated gases and marcabtans

selectively [9]. The Plated activated carbon prepared by soak carbon with several types of epithelial material as well as its use in the removal of H<sub>2</sub>S from natural gas [10].

- E. Polymer coated activated carbon its prepared from the packaging carbon activated by polymeric materials to give satin surface and be permeable so that this case does not allow the occurrence of diverticulitis of the pores and is used in filtration processes [5, 11].
- F. Activated carbon in the form of molecular sieve (CMS this is the kind of carbon activated who has form sieves molecular and its uses for adsorption and removal of very low concentrations of the adsorbent material in the liquid and gaseous phases as it is used in the adsorption the polluting gases that emissions from electric power stations as well as the separation of hydrogen gas and oxygen in a manner adsorption oscillator it was adsorption at room temperature [9].
- G. Fiber activated carbon this type of activated carbon is prepared from the process of developing the non-crystalline structure of primary material such as rayon and asphalt and polymers phenolic, etc. at (800) °C [10].

### Preparation of Activated Carbon

All the primary materials used in the preparation of activated carbon must be contain a high percentage of carbon moreover all chemical treatments should include two main reactions : -

1. carbonization reaction to remove hydrogen atoms from carbon initial material used in the preparation of activated carbon such as coal, bitumen, asphalten, wood, coconut shells and other materials. the temperature at which carbonization process taking place between (500- 750) °C and get the weight loss of initial material between (60-70) % and are mostly in the form of CO<sub>2</sub> and volatile materials [12, 13].
2. activation process that works to increase efficiency of adsorption for activated carbon [14], this process includes two different methods:-

#### A. Chemical Activation

In this type of activation is saturation material carbonate with one type or more of the catalyst activation such as [Zinc chloride, carbonate alkaline, sulfuric acid, phosphoric acid and etc.], then article carbonate thermal treatment this process leading to the removal of water also leads to some changes in the chemical structure of carbon and leading to the formation of carbon with a clear efficacy with some type of chemicals material [3].

#### B. Physical Activation

The physical activation process are the treatment of carbonate material thermally at extent thermal ranges between (800-1000) °C the existence of suitable active gases such as steam, CO<sub>2</sub> and air [1].

### Uses of Activated Carbon

Activated carbon used in industry and in everyday life due to having large surface area and capacity of high adsorption and the effectiveness of its surface also contains the structure of porous accurate [3], so it has been used in treatment industrial drinking water for its ability to remove the odor, color and pollutants. this process is very useful in solutions containing organic materials that high molecular weight, low polarity and low solubility in the water [16]. as used in the adsorption of gases that cause environmental pollution and toxic gas during the war [15, 17]. it was used to remove the colors and purification of liquids [17, 18] has also been used in the removal of heavy metals from water [19], other uses for activated carbon in support of catalysts due to the characteristics of chemical and mechanical excellent that are available in which such inactivity to chemicals and corrosion resistance and high hardness, also has a variety of medical uses [20, 21].

## EXPERIMENTAL PART

### Raw Materials Used to Prepare the Activated Carbon

The modified asphaltens in sulfur were prepared at 180 °C and 1h by mixing 1% sulfur with asphalt, then treated Asphalt-Sulfur Mixtures (ASM), with two kind of waste polymers: Polystyrene(PS) and Tires Rubber (R), in a fixed ratio [100g: 1g] [ASM: polymer] respectively and treated thermal heating at 180 °C and 2h with constantly stirring, then compute percentage of asphalten as shown in step II.II.

Then took (100 g) of the sample and distilled under normal atmospheric pressure to remove the maximum amount of light components, then the resulting residue was distilled under reduced pressure of (20 mm) Hg, the remaining material was used in the preparation of activated carbon.

### Calculation the percentage of Asphalten

Taking one gram of asphalt plants with mixtures polymer in a glass beaker and add (40 ml) of petroleum ether by [40:1] (Weight:size) and shake the solution by using a shaking electrical device for 3 hours at (Zero) °C, then asphalten separation by filtration and washing the precipitate(asphalten) by sufficient quantities of petroleum ether, the precipitate dry at room temperature and then calculated the percentage of asphalten in measured models [22].

### Primary Carbonization for Preparation of Activated Carbon

The article prepared mixed with potassium hydroxide by [2:1] [raw material: potassium hydroxide] and added to it (15-20)ml of distilled water and heated the mixture gradually until the 350 °C for 2 h.

### Final Carbonization and Activation

The resulting material took from the primary carbonation and heated to a temperature  $(550 \pm 25)$  °C for 3h and then cooled carbonated material to the room temperature.

### Purification of Activated Carbon II.III.

For the purpose of purifying the pollutes activated carbon by alkali and metal components was conducted by following:

1. Wash models with distilled water several times for the purpose of removing non-interactive potassium hydroxide and make sure that the result of washing process is equivalent.
2. Treated carbon resulting from the above step with a solution (10)% hydrochloric acid and by using thermal escalation process for 2 hours to remove any trace of the ions and the reduction of metal components to as little as possible [23].
3. Wash the carbon output from the previous step with distilled water several times while making sure it is free of any trace of acid and making sure that the result of washing process is equivalent.
4. dried models carbonate resulting from step above at  $(110-120)$  °C for 24 hours and then crushed and riddled by using sieves size (20-40) Mesh, and kept in the desiccators.

### Measurement of the Internal Surface Area of Activated Carbon

Is the number of milligrams iodine adsorbed by one gram of activated carbon, took one gram of activated carbon and put in a dry flask capacity (250 ml) was added to it (10 ml) of solution (5% hydrochloric acid) and heating the flask with its contents to the boil for 30 minutes. put the flask to cool at laboratory temperature and added to it (100 ml) of iodine solution (0.1 N), stir the contents of the flask for 30 minutes by using electric shaking device and then filtered by using dry filter paper .neglected (20ml) of solution at the beginning of the filtration process and collect the other in the conical flask. taking (50ml) and titration versus the stander solution of sodium thiosulfate (0.1 N) presence (1 ml) of starch, then calculate the iodine weight adsorbed by applying the following equation:

$$X = A - [2.2 B \times \text{ml of sodium thiosulfate Used}]$$

$$A = N_1 \times 12693$$

$$B = N_2 \times 126.93$$

Where:

X = weight of iodine adsorbed (mailgram) by 1 g of activated carbon.

N1 = standardized iodine solution.

N2 = standardized sodium thiosulfate.

The iodine number is calculated from the following equation [24].

$$I_n = \frac{X}{M} D$$

Where:

M= weight of the activated carbon used

D= Correction Factor.

### Measuring the external surface area of the active carbon

Taking (0.1 g) of activated carbon and put it in the conical flask and then added to it a known amount of methylene blue dye of concentration (20ppm), shake the flask with its contents in the shaking electric machine for 24 hours in the laboratory temperature and when the disappearance of color added another quantity of the dye solution to be access to the case of an increase of dye is non-adsorbed. then the dye separated by centrifuge and then take the transparent solution and put in an absorption cell and the absorbance was measured at a wavelength of 665 nanometers (methylene blue dye absorbs at this wavelength).

the concentration of the dye removed calculated by using a standard curve which has been prepared for this purpose by taking different concentrations of the dye solution [20,15,10,5 and 25]ppm and measuring the absorbance at (665 nm) and a graph line between values absorbance and concentration [25].

### Measuring of Density

A certain amount of activated carbon was putting in the cylinder of capacity (5 ml) after that, weight the activated carbon in the cylinder using a sensitive balance and density calculated as follows [26].

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

### Ash Content

one grams of activated carbon was putting in Jaffna ceramic then put it in an electric oven at 1000 m for 3 hours and then cold model and weight by sensitive balance and from the difference weights had calculated the percentage of ash in the prepared activated carbon samples [27].

### Humidity Content

one gram of carbon models has been weighed and lifted exposed to the atmosphere for 168 hours, after that put models in an electric oven at a temperature of  $(150)^\circ\text{C}$  for 3 hours, and calculate the percentage of humidity [28].

## DISCUSSION

Use the asphalt in the preparation of activated carbon that is a great economic feasibility material used in industrial areas and in the control of environmental pollution. the goal from this studies that is prepare activated carbon by using asphaltic materials with waste polymer as shown in the (II.I) experimental part, which are often in the form of waste affecting environmental, since this material is increasing the carbon content of the asphalt as organic materials and table (1) shows the prepared activated carbon models specifications and compare it with the commercial model of the company B.D.H.

Note from the prepared models that chemical activation method by using KOH gave very good results, as note that the iodine number values were excellent and also the adsorption of blue methylene dye for models prepared and this explains the role of the hydroxide to increase the number of pores formed, since a potassium hydroxide is affecting factor in the activation process which develops porous structure in the production of activated carbon since the presence of the hydroxide can cause necrosis process in the structure of the raw material, leading to the development of the porous structure of the activated carbon product [25]. activation uses to refer to the all operations that produce effective carbon stimulus have the ability to adsorption of some materials physically or chemically from diluted solutions or from the gas phase. therefore, the main goal of the activations is to increase the number of pores per unit area, leading to increased final surface area as this study shows the impact of Activation and additives polymeric on the quality of activated carbon prepared of asphalt materials. the adsorption properties of the activated carbon prepared improved by existence of polymeric mixtures in the model which led to increase the proportion of asphalten due to increased condensation nuclei aromatics and Aliphatic that containing to the alkali compensators as well as hybrids atoms contained asphalten, since the structure of carbon consists of aromatic sheets three-dimensional arrangement and an arrangement similar to the structure of graphite, Figure (1) shows the structure of graphite.

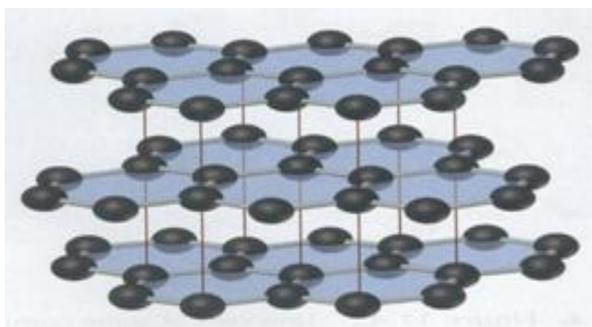
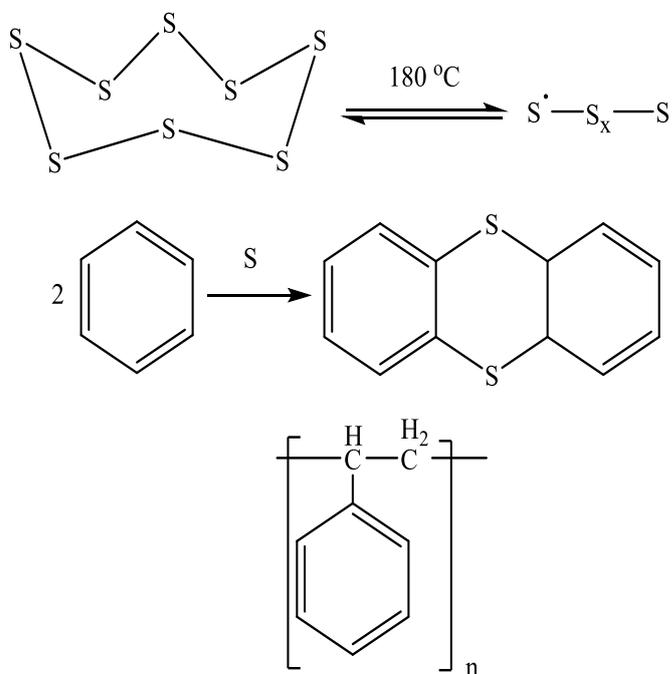


Figure 1: shows the structure of graphite

The most structure in form of activated carbon is hexagonal rings and maybe this is the reason to improvement of the properties of the activated carbon to increase the proportion of asphalten. note from the table (1) that most of the models have low values from the ash as thermal escalation process by using a solution of 10% hydrochloric acid cause to remove most of the metal components in the raw material or that resulted from the preparation of activated carbon process [29]. The intensity and humidity values are a relative measure that mean the ability to adsorption of water is difference. as for the values of mechanical resistance, we find it is less with increase in iodine number for activated carbon models because the increased the values of iodine number and methylene blue dye means an increase in the porous activated carbon and then increase the fragility of the activated carbon and decreasing the mechanical resistance.



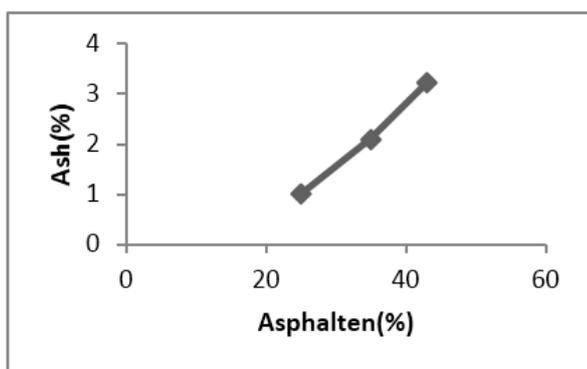
## CONCLUSION

This work provides a simple method to obtain activated carbon that high adsorbents and showing the effect of polymeric materials to improvement the adsorbents properties, Because the polymer particles causes condensed aromatic rings and increase carbon-content, conclude from this that the preparation of activated carbon not only dependent on the condition of preparation but dependent on the modification the chemical properties of row material (asphalt).

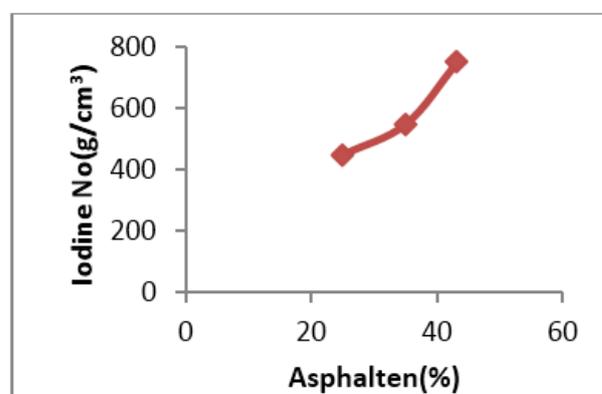
**Table 1:** The specifications of activated carbon models prepared and compare it with the business model.

Sample of carbon	The percentage of Additives used (1:1) (ASM : polymer)	Asphalten %	Ash%	Density (g/cm <sup>3</sup> )	Iodine number(mg/g)	Humidity %	Methylene blue (mg/g)
C <sub>0</sub> *	----	25	1.000	0.380	410	1.100	48
C <sub>1</sub>	ASM + PS	35	3.01	0.299	566	2.00	59
C <sub>2</sub>	ASM +R	38	2.08	0.321	810	1.01	68
C <sub>B.D.H</sub>	----	---	3.200	0.345	908	0.800	90

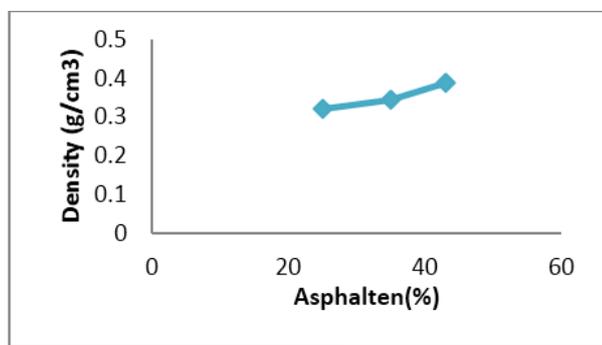
C\* activated Carbon model prepared from Baiji asphalt after a distillation.



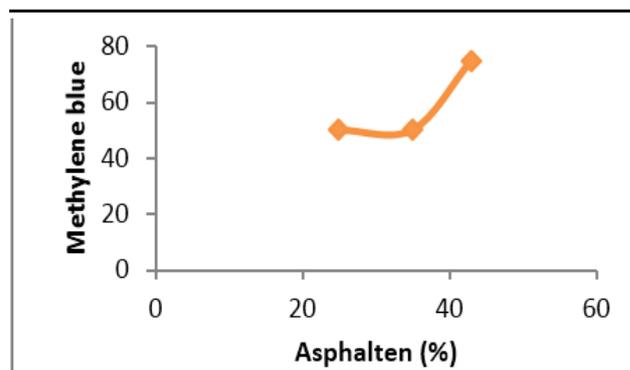
**Figure 1:** Ash percentage with asphalten content



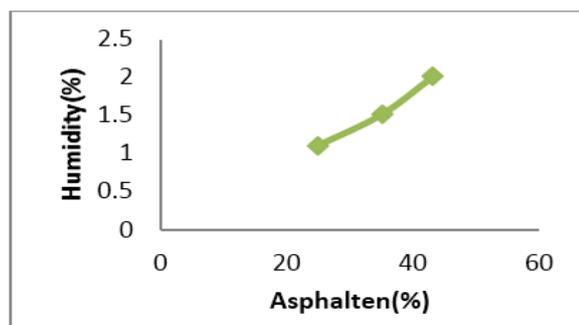
**Figure 4:** Humidity percentage with asphalten content



**Figure 2:** Density with asphalten content



**Figure 5:** Methylene blue with asphalten content



**Figure 3:** Humidity percentage with asphalten content

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