

Production of Activated Carbon via Oxidation with $K_2Cr_2O_7$ and Chemical Treatment

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Abstract

In this work activated carbon of good quality prepared using oxidation condensation process.

The oxidation of the asphaltic materials was conducted using several percentage of $K_2Cr_2O_7$ and in the presence of a stream of air or oxygen. The reaction mixture was heated at $350^\circ C$ for 3hrs. This followed by removal of uncarbonized materials under reduced pressure. The condensation of the fraction obtained is expected to recombine or rereact to give a high carbon content. The carbonization and activation was conducted at $550\pm 25^\circ C$ for 3hrs. and the ratio of the feedstock to NaOH was (1:2).

After the carbonization was completed, the reaction mixture was subjected to purification by treatment with 10% HCl and distiller water till it gives neutral test to the litmus paper. The samples were dried at $120^\circ C$ for 24hrs. and its physical properties were determined.

The research work indicated that a good quality activated carbon can be obtained when the oxidation of sample was conducted using 3% $K_2Cr_2O_7$.

350

(%5-1)

$K_2Cr_2O_7$

o

25 ± 550 [NaOH :] [2:1]

HCl %10

Introduction

Activated carbon, also called charcoal, is more involved in our life than we might expect. It is employed in the production of many products may need every days, to remove impurities that cause an objectionable colour, taste, odour, heavy metals from drinking water and nutrients. Removal of lethal gas in the chemical ware fare (gas masks) and on purification of products in chemical and pharmaceutical industries.

Activated carbon covers a family of materials made from carbon which have the ability to attract and hold certain substance on the carbon surface. This ability is called "adsorption". The adsorption process is an example of what is termed "Chemical Filtration" where certain dissolved substance are removed from water. Filter floss on the other hand performs "mechanical filtration" by trapping solid particles suspended in water. The feedstock most commonly used to produce the carbon are coal, petroleum residue aspartmes, peat, lignite, and wood. These feedstock containing recognizable amount of carbon. Hydrogen and other unrequited components are driven of by heating the feedstock under vacuum to produce a (char). The char is then activated by exposure to extremely hot gas, usually at about 800-1000°C which may help to open the pores of all its type, by reacting with unrequited atom in the structure.

The feedstock selected plus the manner in which its carbonized and activated, determines the characteristics of the finished activated carbon product and its cost. There are dozens of different kinds of activated carbon produced, of these only a

handful will be well valuable to any particular application⁽¹⁾. The literature contains many research work concerning activated carbon, which may include source of the feedstock, natural of carbonization processes, type of activation methods and texture of the product activated carbon which is given as follow:

Garcia, *et. al.* prepared activated carbon from pine wastes gasified in a pilot reactor with the activity reagent of KOH, using different alkali/char ratios from 1/1 to 4/1 in wt/wt at 725-800°C⁽²⁾. Al-Ghannam, *et. al.*, prepared activated carbon from *Morus nigra* by using excess amount of NaOH (1:2) at 550±25°C for 3hrs⁽³⁾. Ptrov, *et. al.*, prepared activated carbon by one-step pyrolysis of agricultural wastes, apirot stones, cherry stones and grapeseed, in presence of steam at 700°C. The adsorption capacities towards Ni, Mg and As ions of the activated carbons prepared have been determined. The sample obtained from cherry stones has shown the highest adsorption capacity increasing in the order As < Mg < Ni⁽⁴⁾. Kim, *et. al.*, prepared activated carbon from Rice husks by heating at a rate of 5°C up to 700°C and maintaining at 700°C for 2hrs. The carbons obtained were subsequently activated at temperature between 750 and 900°C in a steam of moisture containing nitrogen⁽⁵⁾. Rhamadhan, *et. al.*, prepared activated carbon from Beje asphalt using several percentage of V₂O₅ and in the presence of a stream of air or oxygen at 350°C for 3 hrs., the carbonization and activation was conducted at 550±25°C for 3 hrs. using excess amount of NaOH⁽⁶⁾. Al-Ghannam, *et. al.*, prepared activated carbon from Heet asphalt using several

percentage of polyethylene in the presence of air at 350°C for 3hrs. and then complete the carbonization and activation at 550±25°C using excess amount of NaOH⁽⁷⁾.

Experimental

1. Preparation of feedstock for the preparation activated carbon

The feedstock was obtained as residual materials from Beji refinery.

2. Oxidation and condensation of feedstock

Exactly weighed sample of the asphalt was treated with various quantities of K₂Cr₂O₇ (1-5%) weight percent. The mixture was heated gradually until 350°C and kept for 3hrs. at this temperature. The oxidized asphalt was cooled to room temperature. Removal of the uncarbonized materials was carried out by distillation vacuum. The residual materials after distillation was considered as the feedstock for the activated carbon preparation.

3. Primary carbonization of the oxidized feedstock

The materials from step (2) were mixed with twice its weight of the carbonizing agent (NaOH) and (30-50) ml of distilled water. The mixture was heated gradually with continuous stirring until a thick bulk of materials were obtained. The carbonized bulk was heated with a little exposure to atmosphere at 550±25 °C for 3hrs. The product was cooled to room temperature and purified as in the next step.

4. Purification of the activation carbon from metallic impurities

The carbonized materials were crashed carefully and mixed with sufficient amount of 10% HCl solution. The mixture was heated under reflux for 1hr., cooled, filtered and washed with distilled water. The

washing continued until a negative litmus paper was indicated. The sample obtained was dried at 120°C for 24hrs., and kept for properties measurements.

5. Activated carbon measurements

A. Measurement of Density

The density of the prepared activated carbon were determined by weighing 10 cm³ of the carbon using graduated cylinder⁽⁸⁾.

B. Determination of ash content

The ash content was measured by heating one gram of the prepared activated carbon at a temperature of 1000-1100°C. The remained residue was considered as the ash content⁽⁹⁾.

C. Measurement of humidity

One gram of the activated carbon was heated in an oven at 150°C for 3hrs. The difference in weight before and after heating was calculated as H₂O vapour in the sample⁽¹⁰⁾.

D. Determination of carbon activity by methylene blue adsorption method

An exactly weight (0.1 gm) of the prepared activated carbon sample was added to an aqueous solution of 20 ppm methylene blue pigment. The solution was shaken for 24 hrs. at 25°C until adsorption of methylene blue was completed. The absorbance of the solution was determined using (UV- Visible Spectrophotometer) at λ_{max} 665 nm. The procedure was carried out with different types of the prepared samples for comparison purposes. The final concentration of methylene for each activated carbon sample was calculated as the number of milligrams of methylene blue adsorbed by one gram of carbon⁽¹¹⁾.

E. Determination of carbon activity by iodine adsorption method

1. The iodine number (In), which is the amount (in

milligrams) of iodine adsorbed from its aqueous solution by one gram of activated carbon was determined for each sample using the following equation⁽¹²⁾:

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

Where:

m = is the weight of the activated carbon in grams

X = A - [2.2 B x ml of thiosulfate solution used]

$$A = N_1 \times 12693$$

$$B = N_2 \times 126.93$$

N₁ = normality of iodine solution

N₂ = normality of sodium thiosulfate solution

D = correction factor

Results and Discussion

Active carbon is a very important industrial product due to its uses in various aspect of modern civilization. Production of active carbon usually carried out by carbonization of any organic compound that has high carbon content by a routine carbonizing method or modified ones. The choice of feedstock for carbonization processes is of a virtual important to the uses and application, since activated carbon was used in the removal of pollutants and support for catalysts, therefore the aim of this work was to produce activated carbon of a good quality by carbonizing the materials through oxidation condensation process which employed K₂Cr₂O₇ in different percentage and in the presence of stream of air at about 350°C for 3hrs.

Oxidation process is designed to introduce oxygen in the structure and to form oxygenated linkage or carbon-oxygen linkage, which will

lead to increase the viscosity and density of the product and finally increase the molecular weight, asphaltene content, resin content and carbon content. In this method of preparation of activated carbon the oxidized materials were mixed with twice its weight of NaOH and homogenized with little amount of water. The product was purified by 10% HCl. The product washed with distilled water and dried at 120 °C for 24 hrs. The results of the study are given in Table (1).

The results in the Table employing K₂Cr₂O₇ as an oxidizing agent in the presence of oxygen indicated that the ash content in all the prepared samples are less than the commercial sample from B.D.H. Company (treated prepared activated carbon by 10% HCl solution leads to remove a large amount of mineral materials in the samples)⁽¹³⁾. The density of the activated carbons as comparable to that of the B.D.H. commercial one, while the humidity content is slightly higher than the commercial one and this can be reduced by elevating the temperature to 250°C with out exposure to the air to get a little loss in the carbon.

Measuring iodine number and methylene blue when 3% K₂Cr₂O₇ was used is better than the commercial sample. On the other hand increasing the amount above 3% may result unstable oxygenated species which dose not react to increase the carbon content, therefore the optimum amount for catalysts is 3%.

Table 1: Chemical and physical measurements of the prepared activated carbon

Sample	K ₂ Cr ₂ O ₇ %	Density gm/cm ³	Ash %	Humidity %	Iodine number mg/gm	Methylene Blue mg/gm
1	Zero	0.390	1.000	1.121	300	15
2	1	0.350	1.121	1.315	600	50
3	2	0.346	1.211	1.216	750	100
4	3	0.300	1.321	1.432	950	150
5	4	0.345	1.421	1.621	700	73
6	5	0.355	1.451	1.533	650	65
B.D.H	-	0.345	3.200	0.800	908	90

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