

Preparation and characterization of pure activated carbon from Resorcinol formaldehyde polymer

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Abstract

A pure activated carbon was prepared from resorcinol – formaldehyde polymer (RF) via aqueous poly condensation of resorcinol (R) with formaldehyde (F) in presence of dil. NaOH as a catalyst followed by subcritical drying and carbonizing at 550°C then activated at different conditions of temperature and time.

Iodine and methylene blue were chosen as solutes for their well established value in determination of adsorption capacities of activated carbon as well as other physical properties has been determined and compared with commercial sample from B.D.H company.

Key words: Resorcinol formaldehyde (RF), Activated carbon, Pyrolysis, Adsorption capacity, Surface area, Methylene blue (MB)

°550

. (B.D.H.)

Introduction

Almost any carbonaceous material with low organic volatile content, high elementary carbon, and sufficient strengths can be converted into activated carbon. Commercial activated carbons are prepared from a variety of carbonaceous materials and there are many studies in the literature relating to the preparation and characterization of activated carbons from agricultural waste⁽¹⁾

The aim of this work is to prepare activated carbon from resorcinol – formaldehyde polymer (RF) via pyrolysis which have a higher porous materials with possibilities for technical applications such as deionization devices, filtration media, or catalyst supports^(2,3). Because of their adjustable pore and particle size as well as their high electrical conductivity, they are ideal electrode materials in different electrochemical application e.g. fuel cells and supercapacitor⁽⁴⁻⁶⁾.

RF resin are produced via aqueous polycondensation of resorcinol (R) with formaldehyde (F) in presence of dil. NaOH as catalyst followed by subcritical drying⁽⁷⁻⁹⁾

The porosities of carbon, as initially prepared by carbonization at 500 °C are not sufficiently developed for most application and some a melioration is prerequisite step. This is done in several way involving creation of further porosity. One of these methods is thermal activation made of either carbon dioxide or steam or mixture of these two gases. Carbon atoms can be removed from within porous carbons by gasification using carbon dioxide or water vapor, usually at 800-900 °C⁽¹⁰⁾

Iodine and methylene blue (MB) were chosen as solutes for their well-established value in determination of adsorption capacities of the activated carbons.

Experimental

The starting point of resorcinol formaldehyde polymer (RF) preparation was an aqueous solution of resorcinol and formaldehyde with a molar ratio (1:1.5). The catalyst used was sodium hydroxide. After a preinvestigation in order to find a homogenous samples we prepared a dilution of 1% sodium hydroxide.

After gelation the cure temperature kept at 60 °C for 1 hr. then at 75 °C for 24 hr.,

the solid RF resin was crashed to small pieces then carbonized at 500 °C for 1/2 hr., In the final step the carbonized RF were converted into activated carbon via pyrolysis in a constant steam flowing at different temperature and times.

Determination of iodine number for activated carbon⁽¹¹⁾:

This test method covers the determination of the relative activation level of

activated carbons by adsorption of iodine from aqueous solution. The amount of iodine adsorbed (in milligrams) per 1g of carbon called iodine number (I.N).

1g of activated carbon was boiled with 10 ml of 5% HCl for 1/2 hr. cooled to room temperature then treated with iodine solution (0.1N 100 ml) shaken for 1/2 hr. by electrical shaker.

50ml of the filtered solution was titrated with 0.1N sodium thiosulfate in presence of starch as indicator.

Calculation of iodine number (I.N):

$$X = A - [2.2B \times A]$$

$$A = N_1 \times 12693$$

$$B = N_2 \times 126.93$$

Where:

X = the amount of iodine absorbed by activated carbon in milligram

N_1 = normality of iodine solution (0.1 N)

N_2 = normality of sodium thiosulfate (0.1 N)

2.2 = dilution factor ;
(100+10)/50

$$I.N = X / M.D$$

Where:

M = weight of activated carbon used

D = correction coefficient (it's nearly 1)

Determination of methylene Blue (MB) for activated carbon:

The adsorption of MB was performed by agitating for 24 hr. a known quantity of the adsorbent (0.1g) with (100 ml of 20 mg/l MB) stock solution until equilibrium was obtained. A calibration curve was set up in the concentration range of 1-20 mg/l in order to determine the dye concentration in solution after adsorption⁽¹²⁾ by spectro photometric method.

Humidity test⁽¹³⁾

1g of activated carbon was exposed to the ambient temperature for 24 hr., then dried at 110 °C for 2hr., cooled in desiccator and weighed. The difference in weight is calculated as humidity percentage .

Ash percentage⁽¹⁴⁾

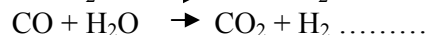
1g of activated carbon was transferred in crucible heated in muffle furnaces at 1000 °C for 3hr., cooled in desiccators the remaining weight was supposed to be ash contain then the percentage was calculated.

Results and Discussion

Activated carbon is a highly carbonaceous material with high surface area. Table 1, 2 and 3 show the adsorption characteristic of ACS prepared. As regards of I.N. this test is complementary to $N_2/77k$ adsorption

isotherm and is used to measure surface area of micropores $\geq 10A^2$ ⁽¹⁵⁾

In contrast, our samples demonstrate an increase I.N. when both temperature and time increased (Table 1 and 2). Whilst Table 3 illustrate that the adsorption characteristics of the activated carbons which have been prepared using steam during the thermal activation shows a higher degree of I.N. comparing to which prepared without steam. This means that more C atoms were released from the pores and also new pores were formed. And this might be the reason of increasing I.N. as shown in the following reactions⁽¹⁶⁾:



The adsorption of MB is a good test for the ability of AC for removing dyes from their aqueous solution. Thus, the method concentration of MB in solution was analyzed using spectrophotometer (Unico Type) at 665 nm.

To determine the amount of adsorption, we used the following equation:

$$q_e = (C_0 - C_e) V / W$$

Where q_e is the adsorption quantity of dye at equilibrium, C_0 and C_e is the initial and equilibrium liquid phase concentrations respectively (mg/l) while V is the volume of the solution and W is the weight of carbon used in grams .

We also found that the density of ACS has been decreased when the temperature raised, We also found a decline in the density when a long period of time was applied whether by using a steam or not; as shown by the tables I, 2, and 3. by contrast, we realized that the other types of ACS contain a certain percentages of Ashes. However, the ACS which we prepared was ash less because they were prepared synthetically from pure polymers. Therefore, they differ from

the others by physical characteristic such as mechanical resistances, and higher conductivity⁽⁶⁾. This is due to the nature of high cross linked polymers.

Conclusion

a. The high purity of activated carbon prepared is important

characteristic reason for food industrial application.

b. The ash less is the unique characteristic of this activated carbon which could be used as electrode material in different electrochemical application.

Table 1. The specification of the prepared activated carbon at 1:30 hr. at different temperatures

Sample	Temp.	AC %	I.N mg/g	M.B mg/g	Hum. %	Ash %	Density
1	650	63.8	156	7.5	1.491	0	0.799
2	700	60.6	208	8.0	1.626	0	0.731
3	750	59.3	305	9.0	1.700	0	0.712
4	800	58.2	414	10.0	1.773	0	0.690
5	900	57.6	497	12.0	1.799	0	0.675

Table 2. The specification of the prepared activated carbon at 750° C at different times

Sample	Time	AC %	I.N mg/g	M.B mg/g	Hum. %	Ash %	Density
1	1.5	59.24	304	9.0	1.700	0	0.712
2	3	54.08	588	15	2.183	0	0.581
3	4.5	45.70	620	16.5	2.261	0	0.560
4	6	35.80	670	18	2.329	0	0.543

Table 3. The specification of the prepared activated carbon at 750° C at

different times using vapor steam system

Sample	Time	AC %	I.N mg/g	M.B mg/g	Hum. %	Ash %	Density
1	1.5	44.06	736.10	27	2.431	0	0.502
2	3	40.25	761.58	40	2.452	0	0.500
3	4.5	30.30	820.50	49.5	2.796	0	0.450
BDH	—	—	837.0	80	0.8	3.1	0.540

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