International Journal of ChemTech Research
CODEN (USA): IJCRGG, ISSN: 0974-4290, ISSN(Online):2455-9555
Vol.9, No.12, pp 688-694, 2016

Characterization of activated carbon that's synthesis from green bean peels by using H_2SO_4 agent: Implementation in reactive blue dye removal

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Abstract : In this article used green bean peels (GBPs) that collected from Iraqi markets to synthesis activated carbon (AC). H_2SO_4 agent using in one step chemical activation method. The effect of volume (15, 25, 35 ml) and concentration (4, 6, 8 molarity) of H_2SO_4 on the activated carbon (AC) yield was studied by removal reactive dye blue from wastewater. Adsorption using synthesis activated carbon has been proven by calculated the dye removal efficiency and the amount of adsorbed dye. Activated carbon (AC) was characterized by laser particles size, XRD and FTIR. The best volume and concentration of H_2SO_4 using in this study are (15 ml volume) and (4 molarity concentration) that investigated by % efficiency of dye removal (90.3%) and the amount of adsorbed dye (11.31 mg/gm).
Keywords : GBPs, Sulfuric acid, Reactive dye Blue.

1. Introduction:

The great importance of the activated carbon (AC) is as an absorbent in the separation and purification processes to reduce environmental pollution in the air, water and soil. Activated carbon capacity of absorption does not depend on the AC surface area only, but also on (i) the internal structure of the pores, (ii) characteristic of the surface, and (iii) functional groups provide on the surface of the adsorbent. These criterions depend on the method used in the preparation and the precursor used also. Therefore, an appropriate adsorbent characterization is decisive to the separation and adsorption processes. It prefers to use local raw materials and cheap and which contain a small amount of ash and high amount of carbon in the preparation of activated carbon process to reduce the cost of the massive production[1]. Synthesis of activated carbon from agricultural waste deals from many researchers including broad bean peels[2], date palm fronds[3-5], apple pulp and apple peel[6], rice husk[7,8], pistachio hull[9], pistachio shell and apricot stone[10], hard cortex of apricot stones[11], spent tea leaves[12], banana peel[13], pineapple peel[21], dates kernels[22], residues of trimming peach trees[22], corn stalks[22], eucalyptus bark[23], leaves of anthacephalous cadamba[24] etc. Synthesis ACs used two common activation methods physical and chemical. In this method , which relies on the activation physicist where the preparation of activated carbon from any source containing carbonaceous material and the extent of high temperatures 700-1100 ° C and using an inert gas , while the next stage is activation by using agents like steam, O_2 and carbon dioxide. While, chemical activation method used lower temperature 400-700 ºC to carbonized the precursor materials by utilized activating chemical agent. In chemical method the activation and carbonization happens together. There are some benefits of chemical over physical activation method. Considering that the chemical method has a low cost process and since it's occur through a one step and at lower temperature range thus the produced activated

carbon has a higher carbon yields and better pores structure[3]. Many workers have applied the method of chemical activation to synthesis the ACs. Several chemical activating agents are used by researchers such as zinc chloride ($ZnCl_2$) [3,24], phosphoric acid (H_3PO_4) [4,6,7,22], potassium hydroxide (KOH) [8], calcium chloride ($CaCl_2$) [14], sulfuric acid (H_2SO_4) [15,21], sodium hydroxide (NaOH) [16], hydrochloric acid (HCl) [17,24], ($FeCl_3$) [20], nitric acid [23] etc.. Wastewater outflow from industries have different pollutant like dyes [18]. Many methods can be used for clearing of dyes from the medium including chemical, biological and physical processes; one of these methods is adsorption [19]. The goal of this research is to synthesize and characterization of activated carbon from green bean peels (GBPs) and test it for clear reactive dye blue from aqueous solution.

2. Experimental

2.1. Materials

GBPs (Figure 1) were collected from Iraqi market. GBPs were thoroughly washed by tap water to remove the impurities and dust. Microwave (LG, China) using to dried the washed GBPs for 20 min. to remove the moisture content. The result sample was mill by hand into powder. H_2SO_4 98% (J. T. Baker, Belgium) analytical grad chemical was used for activation. Distilled water was used for prepared solution and washing.

Figure 1. Green bean peels GBPs

2.2. Preparation of Activated Carbon

To prepare ACs, added different volumes 15, 25, 35 ml of H_2SO_4 solution to the GBPs powder. Also using Different concentrations such as 4, 6 and 8 M of H_2SO_4 solutions that prepared by dissolving H_2SO_4 in distilled water. 5 gm of GBPs powder was mixed with 15 ml, 4 M H_2SO_4 solution at room temperature and transfer the sample to the furnace (Kendro, Hanau, D-63450, Germany) at 110^{o}C for 2 h and after that carbonized the sample in the same furnace at 400^{o}C for 3 h. The result sample was washed by distilled water to remove the remain H_2SO_4 until reach PH 7 then dried the sample in microwave for 20 min to remove moisture.

2.3. Analytical methods

Concentrations of dye were obtained by using a double-beam UV spectrophotometer (Jenway, 6800, China). Measurements were getting at the λmax values (600 nm) against a blank of distilled water. Calibration curve (Figure 2) must be obtained from Absorbance values and standards dye concentrations and using it to convert the absorbance to the concentration.

FTIR spectra (Alpha, Germany) were limited in the area of (4000 – 400 cm^{-1}) and done at H_2SO_4 concentration (4M) and volume (15 ml). X-Ray diffractometer (Shimadzu-6000, Japan) and Laser particle size (Bettersize 2000, China) were done at H_2SO_4 conditions 4M and 15 ml.

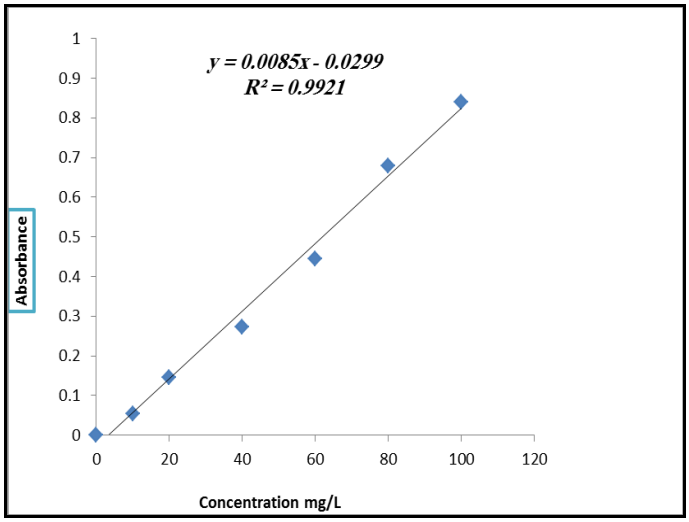

Figure 2 Calibration curve of reactive blue dye.

2.4. Adsorption studies

100 mg of reactive blue dye ($C_{45}H_{44}N_3NaO_7S_2$; Mwt. 825.97) was dissolved in 1 litter of distilled water to prepare stock solution of dye. 0.4 gm of synthesis AC was added to the 50 ml of dye stock solution then adjusted PH to 2.5 and magnetically stirred for 30 min. The sample was filtered after that using UV to analyzed dye absorbance and using calibration curve (Figure 2) to get dye concentration. Calculated the amount of adsorbed dye (Q) (mg/gm) and the efficiency of removal dye (E) by using the following equations:

$$Q = \frac{(C_i - C_f)V}{ms} \quad (1)$$

$$E = \frac{C_i - C_f}{C_i} x\,100 \quad (2)$$

Where V is the solution volume (L), C_i illustrate the initial concentration of dye (mg/L), C_f final concentration of dye (mg/L), and m_s is the adsorbent mass (gm).

3. Results and Discussion

3.1. Results of dye removal

To test the performance of synthesis AC, using it to remove reactive blue dye from aqueous solution and table (1) show the efficiency of removal dye and the amount of dye adsorbed by using different concentration of H_2SO_4 agent with constant volume 15 ml and table 2 show its with different volume of H_2SO_4 agent at constant concentration (4 M).

Table 1. Efficiency of removal dye (E) and the amount of dye adsorbed (Q) for reactive blue dye at different concentration of H_2SO_4 with constant H_2SO_4 volume 15 ml (C_i= 100 mg/L, time= 30 min., V= 50 ml, m_s= 0.4 gm).

Molarity of H_2SO_4 at 15ml	Absorbance	Concentration of dye (mg/L)	Removal dye efficiency (E %)	Amount of dye adsorbed (Q) (mg/gm)
4	0.0530	9.75	90.3	11.31
6	0.2984	38.62	61.5	7.7
8	0.3017	39.01	61.1	7.66

Table 2. Efficiency of removal dye (E) and the amount of dye adsorbed (Q) for reactive blue dye at different volume of H_2SO_4 with constant H_2SO_4 concentration 4M (C_i= 100 mg/L, time= 30 min., V= 50 ml, m_s= 0.4 gm).

Volume of H_2SO_4 (ml) at 4M	Absorbance	Concentration of dye (mg/L)	Removal dye efficiency (E %)	Amount of dye adsorbed (Q) (mg/gm)
15	0.0530	9.75	90.3	11.31
25	0.4371	54.94	45.20	5.66
35	0.5890	72.81	27.37	3.43

The desired values of removal dye efficiency and the amount of dye adsorbed that we can see from table 1 and 2 are 90.3% and 11.31 mg/gm respectively at 4M concentration and 15 ml volume of H_2SO_4.

3.2. Results of XRD test

Figure (3) shows the XRD pattern of the prepared activated carbon after carbonization at 400 °C for 3h. It shows that the prepared sample is poor crystalline with weak peaks at 25°, 26°, and 31° which are diffracted from the planes indexed to graphite.

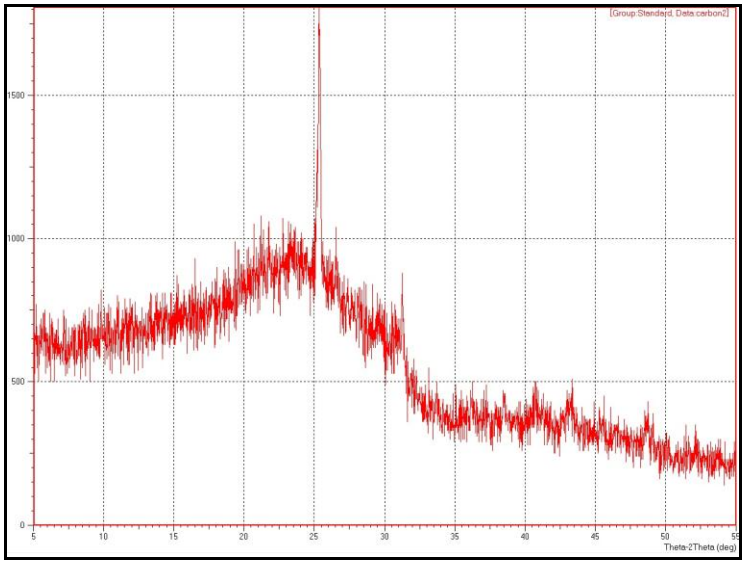

Figure 3. XRD of synthesis AC.

3.3. Results of FTIR test

The FTIR spectra of the prepared activated carbon after carbonization at 400 °C for 3h are shown in figures (4). Table (3) summarizes the interpretation of this spectrum.

Table 3. FTIR spectrum interpretation

Band (cm^{-1})	Corresponding to
1116	O-H (hydroxyl or carboxyl) stretching vibration and C-OH bending vibrations
1370	Stretching vibration of the C-H in the carbonyl
2360, 2220, 2181	OH stretching of HSO_4 group
875–750	aromatic C–H out-of-plane bending vibrations
1554	stretching vibration of benzene ring skeleton
3614	Free OH stretching
3862-3737	vibration absorption spectra of OH
450-750	in-plane and out-of-plane aromatic ring deformation vibrations
602 and 674	out-of-plane C-H bending mode

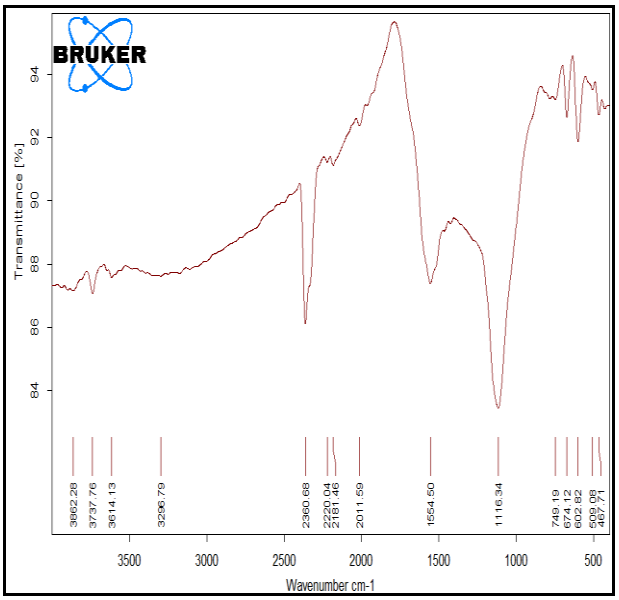

Figure 4. FTIR spectrum of synthesis AC.

3.4. Results of particles size distribution test

Figure (5) shows the result of analysis of particles size distribution for the prepared activated carbon after carbonization at 400 °C for 3h. It can be observed that the prepared sample has wide particle size distribution from around 0.5µm to 200µm with avarge particle size of around 40µm.

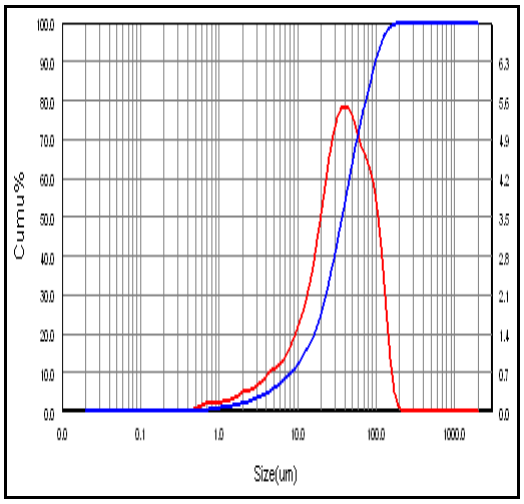

Figure 5. Particles size distribution of synthesis AC.

Conclusions

Activated carbon was synthesised from GBPs at different H_2SO_4 concentration (4, 6, 8 M) and volume (15, 25, 35 ml). The 4 M concentration and 15ml volume of H_2SO_4 agent are the optimum values at which the higher efficiency of dye removal 90.3% and the amount of adsorbed dye 11.31 mg/gm. For environmental and economic considerations prefers to use GBPs for the production of AC and also because it is abundant agricultural waste product. In XRD test weak peaks at 25°, 26°, and 31° which appear and diffracted from the planes indexed to graphite. For particles size distribution test the average size of particle was around 40µm.

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