



Preparation and characterization of activated carbon from plant wastes with chemical activation



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ARTICLE INFO

Article history:

Received 18 February 2014

Received in revised form 10 July 2014

Accepted 13 July 2014

Available online 21 July 2014

Keywords:

Activated carbon
Chemical activation
Plant wastes
Zinc chloride

ABSTRACT

Activated carbon has been widely used in the sorption of chemical species from aqueous solutions as a versatile adsorbent with optimal sorption properties. However, production and regeneration of commercial activated carbons are still expensive and so the importance of activated carbon production by using low-cost raw materials and methods are still up to date. In this study, a one-step chemical activation process by zinc chloride was used to obtain activated carbon from plant wastes (PWs) such as pine sawdust (PS), rose seed (RS), and cornel seed (CS). The effect of activation parameters such as carbonization temperature, impregnation (ZnCl_2/PW) ratio, and impregnation time on the properties of final products were investigated. The produced activated carbons were characterized by nitrogen adsorption isotherms at 77 K. The present results showed that the surface area and methylene blue index of activated carbons, which were determined by adsorption tests, were achieved as high as 1825 m²/g and 300 mg/g in their highest value, respectively.

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1. Introduction

The adsorption has been found to be superior as compared to other traditional treatment methods for wastewater treatment due to its low-cost, easy availability, simplicity of design, high efficiency, ease of operation and ability to treat dyes and metal ions [1,2]. The most widely used adsorbent to remove of wastes by adsorption is activated carbon. Activated carbon is a water industry's standard adsorbent which has highly developed porosity, specific surface area of more than 400 m²/g, surface functional groups, especially oxygen groups and relatively high mechanical strength [3–5]. Therefore, it has been extensively used for separation of gases, recovery of solvents, removal of organic pollutants from drinking water and a catalyst support. As environmental pollution is becoming a more serious problem, the need for activated carbon is growing [6]. However, production and regeneration of commercial activated carbons are still expensive [2] and there are many continuing researches for potential materials and methods so the importance of production of activated carbon by using low-cost raw materials and methods is still up to date [7,8]. Thus, it is of extreme relevance to find suitable low-cost raw materials that are economically attractive and at the same time present similar

or even better characteristics than the conventional ones [9,10]. Currently, there are many studies on the production of activated carbons from industrial or agricultural products, by-products and/or wastes [9]. Basically, there are two different processes for the preparation of activated carbons which have developed pore network and so high adsorption capacity: physical activation and chemical activation. In comparison with physical activation, there are two important advantages of chemical activation. One is the lower temperature in which the process is accomplished. The other is that the global yield of the chemical activation tends to be greater since burn-off char is not required [11].

The aim of this study is to evaluate the utility of some plant wastes, pine sawdust (PS), rose seed (RS) and cornel seed (CS) as abundant and/or cheap precursors for activated carbon production. The influence of the experimental parameters such as carbonization temperature, impregnation ratio and impregnation time on the carbon characteristics was evaluated. The surface areas of the samples prepared in this study were compared with the results published in the literature.

2. Material and methods

2.1. Materials

Pine (*Pinus sylvestris*) sawdust, rose (*Rosa canina*, Gümüşsu Inc.) seed, and cornel seed (*Cornus mas* L.) obtained from the east of

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Turkey were selected as the precursor to production of activated carbon. The precursors were dried, crushed and sieved to a particle size fraction of 1.7 mm (seeds) and 5 mm (sawdust). Elemental composition of materials (carbon, nitrogen, hydrogen, and sulfur) was determined by using the LECO CHNS-932 elemental analyzer. The oxygen content was estimated by difference [100 – (% C + % H + % N + % S)]. The proximate and elemental analyses of raw materials are shown in Table 1. The high carbon, low ash and low sulfur content of the precursors are positive factors and therefore, these PWs can be potential starting materials for the production of active carbon [12,13]. Zinc chloride (98%) and methylene blue (MB) were obtained from Sigma Aldrich and Park respectively. A commercial activated carbon (Merck-2183) was used as a reference material.

2.2. Method

Different amounts of ZnCl₂ (5, 7.5, 10, 15, and 20 g) were dissolved in 150 mL of distilled water, and then 10 g of dried PWs was mixed with the zinc chloride solution and stirred at approximately 85 °C for 2 h. The mixtures were dehydrated in an oven at 110 °C for about 24 h. The ZnCl₂ impregnated PWs were placed in a ceramic crucible and pyrolyzed in a horizontal tubular furnace under the nitrogen flow. The samples were heated to final temperatures of 300–800 °C with 10 °C/min heating rate for residence time of 1 h. The resultant activated carbon was washed with 3 M HCl solution by heating at around 90 °C for 30 min, filtered and rinsed by warm distilled water until neutral pH, dried at 105 °C in an oven for about 12 h and weighed to calculate the yield [14]. The activated carbon production yields were calculated from the weight of activated carbons divided by the weight of the dried PWs.

The adsorption capacities of the samples were evaluated by methylene blue adsorption. A 100 mg amount of the carbon samples was added to 50 mL vials with 25 mL of methylene blue at a concentration of 1200 mg/L in 5% acetic acid-distilled water solution [15]. Blanks not containing methylene blue (MB) were used for each series of experiments. The vials were shaken for 24 h at 25 °C. The concentration of MB in the supernatant solution after and before adsorption was determined with a 1.0 cm light path quartz cells using Shimadzu 1201 UV spectrophotometer (Shimadzu) at 664 nm (λ_{\max}). The amount of methylene blue adsorbed by per gram of adsorbent was calculated as following equation:

$$q = \frac{(C_0 - C) \cdot V}{w}$$

where q is methylene blue index (mg/g), V is the volume of solution (L), and w is the dry weight of the added adsorbent (g).

The surface characteristics were determined by N₂ adsorption/desorption isotherms at 77 K using an analyzer Micromeritics Gemini V. Prior to the adsorption analyses, the samples were pre-degassed at 120 °C for 3 h under vacuum to remove moisture and other impurities. In these analyses, nitrogen was used as adsorbate at liquid nitrogen temperature. Single point surface area (S , mg/g) was determined at $P/P_0 < 0.35$. The average pore widths

of samples were determined by Barrett-Joyner-Halenda (BJH) method.

2.3. Mathematical modeling

In this study, models were developed to predict surface areas and methylene blue indexes of activated carbon samples based on their preparation conditions (carbonization temperature, impregnation ratio, and impregnation time). Firstly, the fitting of experimental data to various models such as linear, logarithmic, inverse, power, cubic, compound, growth, and exponential was investigated for selecting the best equation. Then the final version of the chosen model with further changes which will increase the R^2 values was determined by non-linear regression using the SPSS 11.5 package program. The coefficient of determination R^2 was one of the main criteria for selecting the best equation [16]. In the derived models, the carbonization temperature, impregnation ratio, and impregnation time were coded as X_1 , X_2 , and X_3 , respectively.

3. Result and discussion

3.1. The effect of carbonization temperature

Effects of carbonization temperature on the methylene blue index (q), surface area (S), and yield (%) of activated carbon were evaluated under the conditions of the impregnation ratio of 1.5 g/g and impregnation time of two hours (Table 2).

The carbonization temperature and impregnation ratio can be assumed as the most important process parameters affecting the yield of activated carbon. As seen from Table 2, the yield of activated carbon decreased with the increasing of carbonization temperature for all the raw materials. This could be attributed to the promotion of carbon burning-off, tar volatilization and the devolatilization of biomass would be enhanced due to increase of temperature. Hence, the char yield was reduced and the volatile (liquid and gas) yield was increased [14,17].

The methylene blue indexes of the samples sharply increased with the increase of carbonization temperature from 300 to 400 °C, whereas after 400 °C there was no significant change. The similar results were also found for the surface area of activated carbon prepared from PWs. For instance, the surface areas of the activated carbons prepared from PS sharply increased with temperature up to 400 °C and then slightly decreased. However, the highest surface areas for the produced activated carbon from RS and CS are observed at 500 °C. It highlighted that there are three stages of pore development. They are: opening of previously inaccessible pores, creation of new pores by selective activation and widening of the existing pores [18]. Thus, it can be hypothesized that the first two stages are the dominant process on the pore development until temperature where the surface area is maximum. However, at the higher temperature, it can be concluded the widening of the existing pores. Decrease in the surface areas can be attributed to widening of the existing pores as a result of severe thermal treatment, which causes the breakdown of

Table 1
The proximate and elemental analyses results of raw materials.

Precursor	Elemental					Proximate*			
	C	H	N	S	O	Moisture	Fixed carbon	Volatile matters	Ash
PS	46.41	6.27	0.06	0.03	47.23	6.02	21.30	72.23	0.45
RS	41.36	6.03	1.20	0.10	51.31	5.50	25.37	66.34	2.79
CS	43.67	6.43	0.40	–	49.50	5.40	27.34	64.67	2.59

* As received.

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