Preparation of Activated Carbon with High Surface Area from *Pistacia Aatlantica Shell*

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Abstract

We report the preparation of activated carbon (AC) using phosphoric acid (H3PO4) as the activating agent from *Pistacia Atlantic* (PA) shell. The properties of AC were characterized by the scanning electron microscopy (SEM), nitrogen adsorption isotherm, low angle XRD and Fourier transforms infrared spectroscopy (FTIR). The obtained mesoporous activated carbon has a BET surface area of 1728.78 m2/g. Methylene blue (MB) was chosen as model dye compound to evaluate the adsorption behavior. The best conditions resulted in PAAC with a maximum monolayer adsorption capacity of 350 mg/g.

Keywords: Pistacia Atlantica shell, Activated carbon, Methylene blue

1. Introduction

There is a wide attention to the activated carbons with highly developed surface area to use in the various industrial applications such as separation/purification of liquids and gases, water treatment, as supporting substrate of catalytic materials, super-capacitors, electrodes, gas storage devices and *etc.*, [1].

Activated carbons are usually prepared from the carbonaceous raw materials by different activation reaction so that their quality and characteristics depend on the physical and chemical properties of the starting materials and employed activation method. Among many synthesis methods, which need to the high temperature and difficult reaction condition, the preparation of activated carbons from agricultural and natural wastes is introduced as a facile, low cost and promising technique towards the new environmental friendly technologies for the synthesis of the carbonaceous substrates with a high surface area [2]. The obtained products are prone to the adsorption activity of toxic materials from environment and as a result of this concept, are nominated as the effective and low cost adsorbents. This method is driven *via* impregnating the initial materials using a chemical agent *e.g.*, phosphoric acid (H_3PO_4) [3], zinc chloride ($ZnCl_2$) [4] and potassium hydroxide (KOH) [5]. The initial row materials can be been the pistachio-nut shell [2], grain sorghum [3], waste (chipboard and plywood), scrap tyres, urban sewage, and straw [6], coconut shells [7], pecan shell [8], Eucalyptus camaldulensis Dehn [9], pomegranate peel [10], bamboo scaffolding [11], herb residues [12], mangosteen Peel [13], lotus stalk [14], grape seeds [15].

As a matter of fact, there is no systematic study related to the preparation of these type adsorbents from *Pistacia Aatlantica* shell. In this study, we used the *Pistacia Aatlantica* shell as the starting material to prepare the activated carbon by phosphoric acid as a chemical driving agent. Then, the obtained product as an adsorbent substrate was employed to decolorize of organic toxic dye, Methylene blue (MB), from aqueous solution.

2. Experimental

2.1. Initial materials

All the chemicals were purchased and used as received without purification. *Pistacia Aatlantica* shell (PA) was obtained from the mountains of Azerbaijan-e gharbi province of Iran. Deionized water was used to prepare all the reagents and solutions.

2.2. Preparation of activated carbon

PA shells were collected, washed, dried in air, crushed by mechanical milling and screened for the tuning of the particle size between 1 and 5 mm. Then, the screened particles were impregnated by H_3PO_4 85% aqueous solution under mechanical stirring for 3 h and treated at 500 °C in air, which is lower activation temperature in comparison with the physical activations [16]. The residual phosphoric acid was eliminated from the prepared activated carbon by washing with distilled water several times. The “yield” of synthesis is defined as the ratio of as-prepared activated carbon weight (w_1) to the initial weight of dried PA shells (w_0):

$$\text{Yield\%} = (w_1/w_0) \times 100$$

2.3. Characterization

Chemical characterization of functional groups was detected by Fourier transform infrared spectrometer (FT-IR Shimadzu- 8400S Spectrometer) in the scanning range of 4000–400 cm^{-1}. The surface morphology was characterized using a scanning electron microscope, SEM, VEGA/TESKAN microscope with an accelerating voltage of 30.00 KV. The pore structural characteristics of the as-prepared activated carbon derived from the PA shells was determined by nitrogen adsorption at 77 K using a nitrogen sorption/desorption measurements with a Belsorp mini II. Prior to the analysis, the sample was degassed for 3 h under vacuum at 573 K. The sample was transferred to the analysis system where it was cooled in liquid nitrogen. Analysis was carried out at 77 K to obtain the nitrogen adsorption isotherm. The S_{BET} was calculated by the BET (Brunauer–Emmett–Teller) equation [17]. The total pore volume was estimated to be the liquid volume of adsorbate (N_2) at a relative pressure of 0.985. The mesopore and micropore size distributions were determined using the BJH model [18]. Powder X-ray diffraction (XRD) patterns were recorded on a JEOL X-ray diffractometer with monochromatized Cu Kα radiation (λ=1.5418 Å).

2.4. Adsorption performance

Adsorption tests were undertaken in a 100 mL of dye solutions containing 0.01 g of the synthesized adsorbent within the initial concentration range of 10–50mg/L. The mixture was agitated at 125rpm and room temperature to reach the equilibrium and then, the residual concentration of MB dye was determined using a UV–Vis spectrophotometer (Shimadzu, Mini 1240) at 668 nm. MB uptake at equilibrium, q_e (mg/g), was calculated by:

$$Qe = \frac{(C0 - Ce)V}{W}$$

Where, C_0 and Ce (mg/L) are the liquid-phase concentrations of dye in initial and equilibrium time, respectively. V (L) is the volume of the solution, and W (g) is the mass of adsorbent.

3. Results and discussion

SEM images of the prepared activated carbon by chemical treatment on the crushed PA shells as a natural waste are shown in Fig. 1. It can be clearly observed that the texture surface of the product is cylindrical (Fig. 1b) is resulted from evaporating the H_3PO_4 molecules and leaving the spaces on the surface of compound. The sharp peak at 2θ of 1° in low angle XRD pattern (shown in Fig. 2) demonstrated the formation of the activated carbon without any impurities. FTIR spectra of the precursor before heating in furnace (char) and final product (PAAC) were presented in Fig.3. The broad band at 3600–3000 cm^{-1} is related to the vibration frequency of the C-H, N-H and O-H groups. The vibration frequencies at 2360 and 2270 cm^{-1} are assigned to the C≡C stretching band of alkynes. The presence of -COOH dimmer shows a signal at 2343 cm^{-1}, while the peak at 1995 cm^{-1} can be ascribed to the vibration of C≡N band. In addition, the vibration frequencies at 1700-1800cm^{-1}, 1425 and 1276 cm^{-1} are attributed to the C=O and C-O-C groups, respectively.

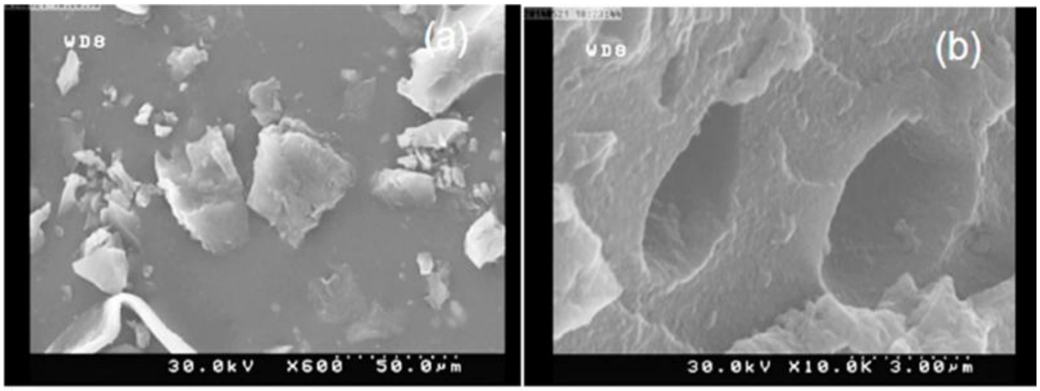

Fig.1. SEM images of product.

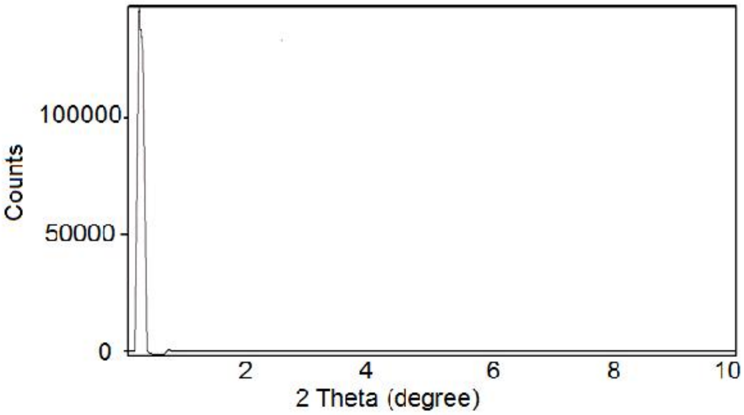

Fig.2. Low angle XRD pattern of product.

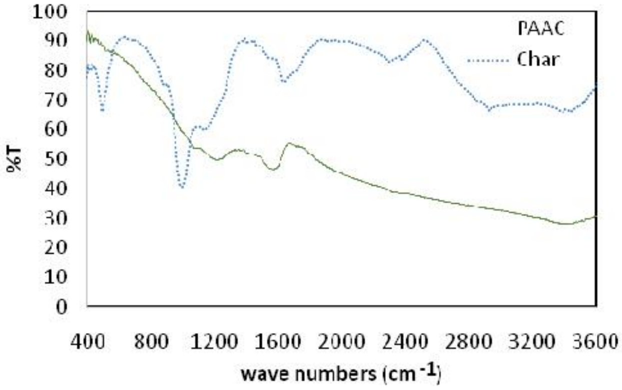

Fig.3. FTIR spectra of the Char and PAAC.

Nitrogen adsorption–desorption curve provides qualitative information on the adsorption mechanism and porous structure of the carbonaceous materials [19]. The isotherm (Fig. 4) features a combination of type I and type II isotherms, in accordance with the IUPAC classification. This adsorption behavior exhibits a combination of microporous–mesoporous structure. The obtained activated carbon has a BET surface area of 1728.78 m2/g. The pore diameter between 1.5 to 5 nm with an average pore size of 2.9077 nm was determined using the BJH model (Fig. 4).

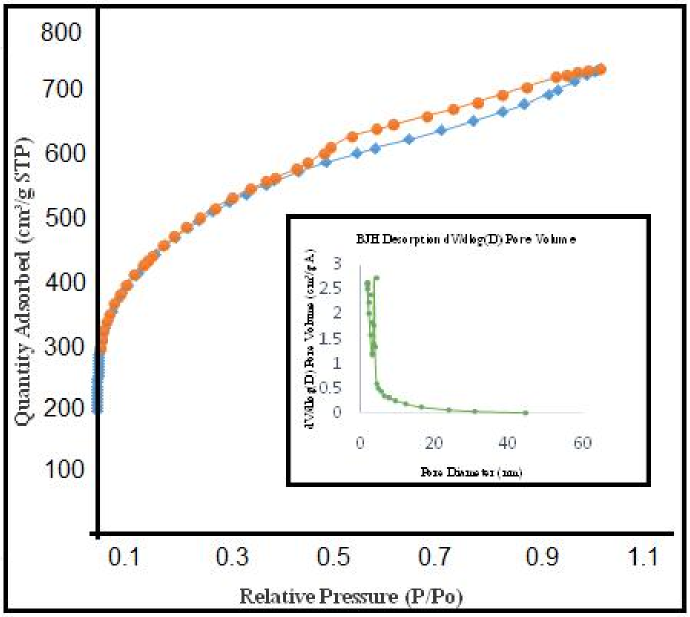

Fig.4. Nitrogen adsorptions–desorption and pore size distribution curves of product.

3.1. Adsorption process

The adsorption capacity curve, q_t *vs.* t, in the initial concentration range of 10–50 mg/L is displayed in Fig.5. Due to overcome the driving force to the mass transfer resistance between the aqueous and solid phases, at first, the number of dye molecules adsorbed onto the carbon surface rapidly increased and then, the active sites on the surface of adsorbent are saturated and adsorption process slowed down and reached a plateau level. The adsorption equilibrium, q_e increased from 99.23 to 350.4 mg/g with an increase of initial concentration to 50 mg/L. the results indicated a superior capability of the product for the removal of dye pollutants from water by adsorption mechanism.

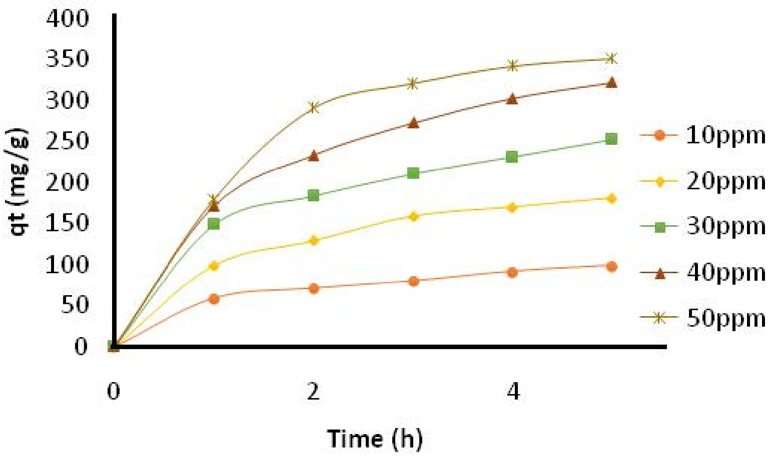

Fig.5. The effect of contact time on the adsorption of MB onto PAAC at room temperature.

4. Conclusion

In a summary, we successfully prepared a well-developed porous texture of activated carbon by using natural waste of (PA) shells. It was found that *Pistacia Aatlantica* (PA) shell can serve as a potential suitable natural waste for manufacturing the activated carbon with a high surface area, which can operate as a capable adsorbent for the water treatment. The BET surface area of the product (1728.7848 m^2/g) represented a higher surface area in comparison with the porous substrates. The adsorption behavior of MB dye on the product surface indicated a maximum monolayer adsorption capacity of 350 mg/g at neutral pH, and room temperature.

Acknowledgments

The financial support of this study, by Iran University of Science and Technology and Iranian Nanotechnology Initiative, is gratefully acknowledged.

References

[1] Kunbin Yang , Jinhui Peng , C. Srinivasakannan , Libo Zhang , Hongying Xia , Xinhui Duan, Bioresource Technology 101 (2010) 6163.

[2] Aik Chong Lua, Ting Yang, Journal of Colloid and Interface Science 290 (2005) 505.

[3] Yulu Diao, W.P. Walawender, L.T.Fan, Bioresource Technology 81 (2002) 45.

[4] Dilek Angin, Fuel 115 (2014) 804.

[5] Long Lin, Shang-Ru Zhai, Zuo-Yi Xiao, YuSong, Qing-Da An, Xiao-Wei Song, Bioresource Technology 136 (2013) 437.

[6] Sainz-Diaz, C.I., Griffith, A.J., Fuel 79 (2000) 1863.

[7] Zhonghua Hu, M.P. Srinivasan, Yaming Nia, Carbon 39 (2001) 877.

[8] Yanping Guo, David A. Rockstraw, Bioresource Technology 98 (2007) 1513.

[9] Phussadee Patnukao, Prasert Pavasant, Bioresource Technology 99 (2008) 8540.

[10] Nevine Kamal Amin, Journal of Hazardous Materials 165 (2009) 52.

[11] Edward L.K. Mui, W.H. Cheung, Marjorie Valix, Gordon McKay, Separation and Purification Technology 74 (2010) 213.

[12] Juan Yang, Keqiang Qiu, Chemical Engineering Journal 167 (2011) 148.

[13] Foo, K.Y., Hameed, B.H., Chemical Engineering Journal 180 (2012) 66.

[14] Hai Liu, Peng Dai, Jian Zhang , Chenglu Zhang , Nan Bao , Cheng Cheng , Liang Ren, Chemical Engineering Journal 228 (2013) 425.

[15] Diana Jimenez-Cordero, Francisco Heras , Noelia Alonso-Morales, Miguel A. Gilarranz, Juan J. Rodríguez, Fuel Processing Technology 118 (2014) 148.

[16] Benadjemia, M., Millière, L., Reinert, L., Benderdouche, N., Duclaux, L., Fuel Processing Technology 92 (2011) 1203.

[17] Gregg, S.J., Sing, K.S.W., Adsorption, Surface Area and Porosity, Academic Press, New York, 1982, p. 85.

[18] Barrett, E.P., Joyner, L.C., Halenda, P.H., Journal of American Chemical. Society. 73 (1951)373.

[19] Foo, K.Y., Hameed, B.H., Chemical Engineering Journal 184 (2012) 57.