



King Saud University  
Arabian Journal of Chemistry

www.ksu.edu.sa  
www.sciencedirect.com



## ORIGINAL ARTICLE

# Characterization of activated carbon prepared by phosphoric acid activation of olive stones

S.M. Yakout \*, G. Sharaf El-Deen

Hot Labs Center, Atomic Energy Authority, P.C. 13759 Cairo, Egypt

Received 26 June 2011; accepted 10 December 2011

## KEYWORDS

Activated carbon;  
Yield;  
Olive stones;  
Phosphoric acid;  
Porous texture

**Abstract** The effects of activating agent concentration on the pore structure and surface chemistry of activated carbons derived from olive stone with chemical activation method using phosphoric acid as the activating agent were studied. Mass changes associated with the impregnation, carbonization and washing processes were measured. With  $H_3PO_4$  dilute solutions (60, 70, and 80 wt%  $H_3PO_4$ ), the loading of substance on CS increases with concentration. The concentration of the  $H_3PO_4$  solution seems to control the processes of impregnation, carbonization and washing in the preparation of AC from olive stones by  $H_3PO_4$  chemical activation. ACs have been characterized from the results obtained by  $N_2$  adsorption at 77 K. Moreover, the fractal dimension ( $D$ ) has been calculated in order to determine the AC surface roughness degree. Optimal textural properties of ACs have been obtained by chemical activation with  $H_3PO_4$  80 wt.%. The BET surface areas and total pore volumes of the carbons produced at  $H_3PO_4$  80 wt.% are  $1218 \text{ m}^2/\text{g}$  and  $0.6 \text{ cm}^3/\text{g}$ , respectively.

© 2011 King Saud University. Production and hosting by Elsevier B.V. All rights reserved.

## 1. Introduction

Olive stones are the most extensively cultivated fruit crop in the world. Olive cultivation is particularly widespread throughout the Mediterranean region and plays an important role in its rural economy, local heritage, and environment protection.

The largest producing countries are located in the Mediterranean and Middle East regions providing 98% of the total cultivated surface area, and 99% of the total olive fruit production (Niaounakis and Halvadakis, 2006). The world production of olives, for the year 2007, was 17.4 Mton, of which 12.6 Mton came from Europe (FAOSTAT, 2009).

Activated carbon is also among the potential conversions, with applications in the removal of dyes, odors, tastes, and contaminants, in water purification and other decontamination processes (Matos et al., 2010). Olive stone is a lignocellulosic material, with hemicellulose, cellulose and lignin as the main components. Olive stones could be a very adequate feedstock to obtain active carbons with good adsorptive properties and hardness, which could be of interest in future environmental protection programs.

Activated carbon (AC) is widely used on an industrial scale as an adsorbent mainly in the purification/separation of liquids and gases and also as a catalyst and catalyst support

\* Corresponding author. Tel.: +20 0482807763.  
E-mail address: sobhy.yakout@gmail.com (S.M. Yakout).

1878-5352 © 2011 King Saud University. Production and hosting by Elsevier B.V. All rights reserved.

Peer review under responsibility of King Saud University.  
doi:10.1016/j.arabjc.2011.12.002



Production and hosting by Elsevier

(Olivares-Marin et al., 2006). Furthermore, newer applications are ever emerging, particularly those concerning environmental protection and technological development.

There are basically two methods for activated carbons' production: physical and chemical activations. Physical activation includes the carbonization of the starting material and the activation of the carbonize using steam and carbon dioxide. In the chemical activation method, phosphoric acid and zinc chloride are used for the activation of lignocellulosic materials, which have not been carbonized previously; whereas metal compounds such as potassium hydroxide are used for the activation of coal precursors or chars. When compared to zinc chloride, phosphoric acid is the most preferred because of the environmental disadvantages associated with zinc chloride. Problems of corrosion and inefficient chemical recovery are also associated with it. Moreover, the carbons obtained using zinc chloride cannot be used in pharmaceutical and food industries as they may contaminate the product (Prahas et al., 2008). Although potassium hydroxide develops large microporosity, the yield of activated carbon impregnated by potassium hydroxide is lower than those activated with zinc chloride or phosphoric acid, and at high temperature, i.e.  $> \pm 650$  °C, the carbon content is less than fixed carbon in the initial precursor. The presence of metallic potassium will intercalate to the carbon matrix (Prahas et al., 2008), yielding lower yield of activated carbon, less than the carbon content of the raw material.  $H_3PO_4$  is the most widely used impregnation agent. Using this substance, most of the numerous studies carried out so far on the preparation of AC have focused on the influence of concentration of the impregnation solution and soaking temperature on the porous structure. Because of the highly polar character of  $H_3PO_4$  and hence the control of the physical and chemical interactions occurring in the bulk of the solution and with the substratum during the impregnation treatment, the solution concentration is likely to be the primary factor of the activation process. Here, dilute and concentrated solutions of  $H_3PO_4$  are used in the impregnation of an AC precursor, the influence of phosphoric acid concentration on the loading of  $H_3PO_4$  is investigated. This work deals with the influence of the treatment methods of olive stones and SEOP on the structure and surface properties of the produced activated carbons.

## 2. Materials and methods

### 2.1. Active carbon preparation

As indicated above, olive stones (OS) were used as a precursor. OS as received was ground, dried and sieved, and only the fraction of particle sizes comprised between 1 and 2 mm was selected for the preparation of activated carbons. Approximately, 50 g of conditioned OS was impregnated with 200 ml of phosphoric acid solutions with concentrations of 60, 70 and 80 wt.% mixture stirred at 85 °C, maintaining for 4 h to ensure the access of  $H_3PO_4$  to the interior of the olive stones. After mixing, carbonization of acid-impregnated OS was carried out in a stainless steel reactor provided with gas inlet and outlet, which was placed in a vertical cylindrical furnace. The temperature within the furnace was first calibrated and the length and position of the constant temperature hot zone determined. Heating from room temperature to the maximum heat treatment temperature 500 °C (i.e., carbonization

temperature) in inert atmosphere of nitrogen (flow rate = 200 mL/min. The heating rate was 3 °C/min and holding time 2 h). After heating, the system was cooled down to room temperature under the same flow of nitrogen and the product obtained was washed with distilled water until pH 6 was reached in the residual liquid and finally oven-dried at 120 °C. OS6, OS7, OS8 were used as a label for activated carbon prepared from olive stones at  $H_3PO_4$  60wt.%, 70wt.%, 80wt.% concentrations, respectively.

Low temperature-ramping rate of 3 °C/min was used in this stage to minimize the temperature difference between the furnace chamber and activation mixture as well as maintain the mixture at a specific temperature range for enough time so that perceptible activation extent could be obtained and an optimized activation temperature range could be determined.

### 2.2. Active carbon characterization

Nitrogen adsorption/desorption isotherms were measured at 77 K on an automatic adsorption instrument (Quantachrome Instruments, Model Nova1000e series, USA) in relative pressure ranging from  $10^{-6}$  to 0.999. Prior to the measurement, all the samples were crushed and powdered to shorten the time required for reaching equilibrium in the isotherm study and degassed at 250 °C under nitrogen flow for 16 h.

The nitrogen adsorption–desorption isotherms were used to determine the following parameters: specific surface area SBET (according to the BET equation), total pore volume  $V_{tot}$  (calculated from the nitrogen uptake at relative pressure of 0.95), total micropore volume  $V_{micro}$ , according to simplified equations (Mehandjiev et al., 1994; Nickolov and Mehandjiev, 2000, 1995), total mesopore volume  $V_{mes}$  (determined by subtracting the micropore volume from the total pore volume), mean pore radius  $r_p$  (calculated using the total pore volume and the BET surface area, assuming a cylindrical pore model), the micropore size distribution parameter  $X_o$  (the half-width of the distribution curve maximum) was calculated according to simplified equation of the Horvath–Kawazone (HK) method (Ustinov and Do, 2002).

The fractal dimension is often used as an index of roughness or irregularity of the surface of ACs (Diaz-Diez et al., 2004) among other materials. In this work, the fractal dimension was determined by applying the FHH (Frenkel–Halsey–Hill) equation (Halsey, 1948) to the adsorption isotherm.

Apparent (bulk) density of all samples was calculated as the ratio between weight and volume of packed dry material.

The FTIR spectra of the samples were recorded between 4000 and 450  $cm^{-1}$  in a Perkin-Elmer 1720 spectrometer. Pellets were prepared by thoroughly mixing carbon and KBr at the 1:400 carbon/KBr weight ratio in a small size agate mortar. The resulting mixture was compacted in a Perkin-Elmer manual hydraulic press at 10 ton for 3 min.

The sample morphology was observed using a scanning electron microscope (SEM) model JEOL 5400. The specimens for SEM observation were prepared by depositing the fibers onto specimen-stubs with conductive double sticky copper tapes, and then sputter-coating (model Polaron SC7640, Quorum Technologies Ltd., UK) the sample surface with Au–Pd to prevent electrical charging during examination. Imaging was done in the high vacuum mode under an accelerating voltage of 15 kV, using secondary electrons.

Download English Version:

<https://daneshyari.com/en/article/5142212>

Download Persian Version:

<https://daneshyari.com/article/5142212>

[Daneshyari.com](https://daneshyari.com)