

PREPARATION, SURFACE AND POROUS CHARACTERIZATION OF ECO-FRIENDLY ACTIVATED CARBON PRODUCED FROM APRICOT STONES

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Abstract

This work presents a thermochemical approach toward controlled preparation of powdered activated carbons from lignocellulosic raw materials (apricot stones), as industrial byproducts and components of organic solid waste. The physicochemical changes of the biomass during thermochemical activation with H_3PO_4 obtained after carbonization at 500 C for 2 h have been investigated by means of Fourier Transform Infrared (FTIR) spectra, scanning electron microscopy (SEM) and Brunauer–Emmett–Teller (BET) technique. The BET surface area and total pore volume were as high as 1098.78 m²/g and 0.5 cm³/g, respectively. The experimental results indicated that the use of apricot stones as a precursor material for the preparation of activated carbon (AC) was feasible.

Introduction

Activated carbon is one of the most used and tested adsorbents in the removal of heavy metals, industrial organic compounds, pharmaceuticals and dyes. Different types of lignocellulosic materials was used as potential precursors in the production of low cost activated carbon, because these materials possess high carbon contents and are low in cost [1].

There are, two different processes for the preparation and production of activated carbon: physical and chemical. Chemical activation includes impregnating the lignocellulosic raw materials with chemical agents (H_3PO_4 , HNO_3 , H_2SO_4 and $NaOH$). After impregnation, the materials are carbonized and washed to eliminate the residues. The chemical activation, which was used in this study has two important advantages when compared to the physical activation. The first advantage is the lower temperature at which the process is conducted, and the second is that the yield (mass efficiency of activation) of the chemical activation tends to be greater [2].

In this study, AC was prepared from apricot stones, as industrial byproducts with H_3PO_4 as an activating agent in the complete absence of inert atmosphere. A detailed characterization of the obtained activated carbon was performed through various instrumental analyses, comprising of FTIR, SEM and BET.

Experimental

Apricot stones were obtained from fruit plantation located in Novi Becej, province of Vojvodina (Serbia). The stones were milled with a mechanical mill and < 3.0 mm fraction was chosen for the thermochemical conversion.

Preparation of activated carbon included the following steps: milled stones were washed with distilled water. Later, the fruit stones were impregnated with a solution of 50% H_3PO_4 . After impregnation, the solution was filtered to remove the residual acid. Subsequently impregnated

samples were air dried at room temperature. The samples were placed in a furnace and heated (10 °C/min) to the final carbonization temperature of 500 °C for 2 h without the use of nitrogen. After cooling, the adsorbent was washed with distilled water to achieve acid free conditions and its pH was monitored until the filtrate pH value exceeded 4.

A detailed Fourier Transform Infrared spectra (FTIR) study of prepared activated carbon was carried out to identify qualitative characteristics of carbon material and the different functional groups responsible for the adsorption. FTIR spectra of AC were recorded with a FTIR/NIR spectrophotometer Nexus 670 (Thermo Nicolet, USA), at wavenumbers from 400 to 4,000 cm^{-1} . The microstructures of the AC were determined by scanning electron microscope (SEM) JSM 6460LV instrument (JEOL, USA). Scanning electron micrographs were recorded without sample coating, with $\times 500$ and $\times 5,000$ magnification.

AC specific surface area determination was done by measurement of N_2 adsorption, applying Brunauer–Emmett–Teller technique (BET) and Autosorb iQ instrument (Quantachrome, USA).

Results and discussion

A review of the adsorption literature revealed that only materials with suitable physico-chemical characteristics are practical and cost effective for industrial scale applications [3]. The ideal properties to achieve the optimum adsorption performance include structural stability, porosity, large surface areas, hydrophilicity and appropriate functional groups [4].

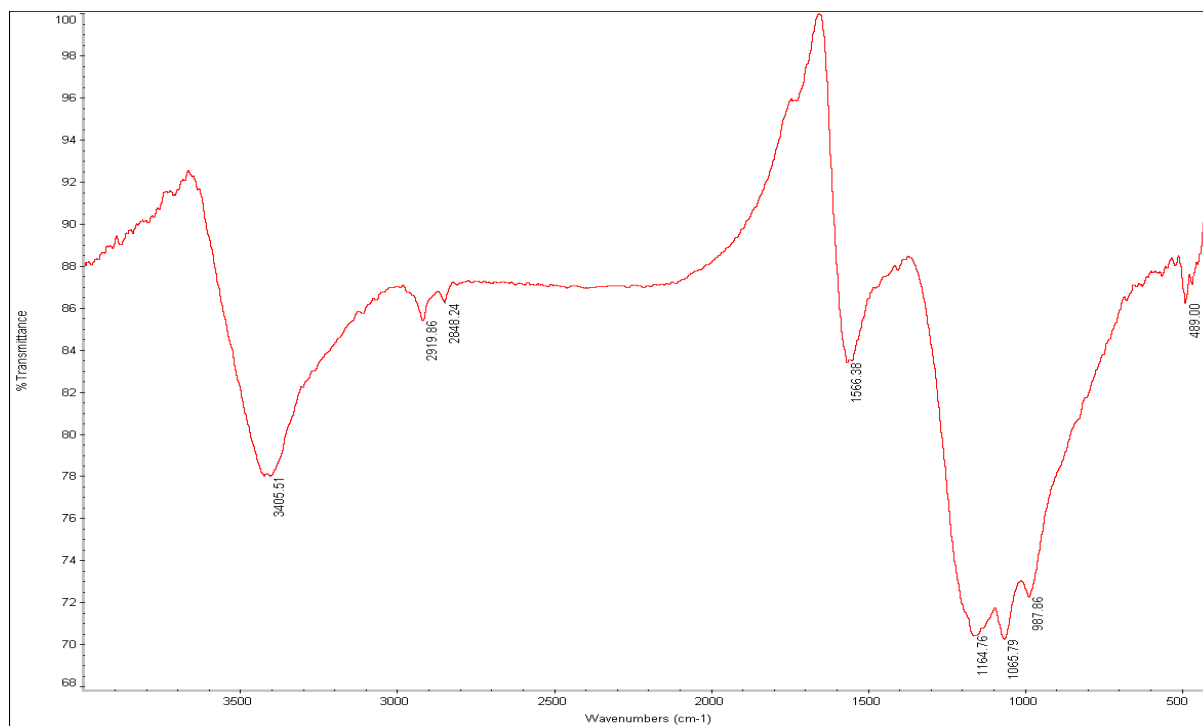


Figure 1. FTIR spectra of the activated carbon (AC)

The FTIR spectra of adsorbent (Figure 1) showed a broad band at 3405.91 cm^{-1} due to O–H stretching vibration and a peak at 489.00 cm^{-1} due to O–H bending vibration. Peaks between the range of 3700 and 3200 cm^{-1} represent the overlapping peaks of stretching vibrations of O–H and N–H groups. The distinct absorption peaks at 2919.86 cm^{-1} and 2848.24 cm^{-1} could be assigned to -CH stretching vibrations of -CH₂ and -CH₃ functional groups. The adsorption peak at 1566.38 cm^{-1} could be characterized by primary and secondary amide bands. The sharp bond

within $1164.76 - 987.86 \text{ cm}^{-1}$ is attributed to the C–O groups, which confirms the lignin structure of the activated carbon.

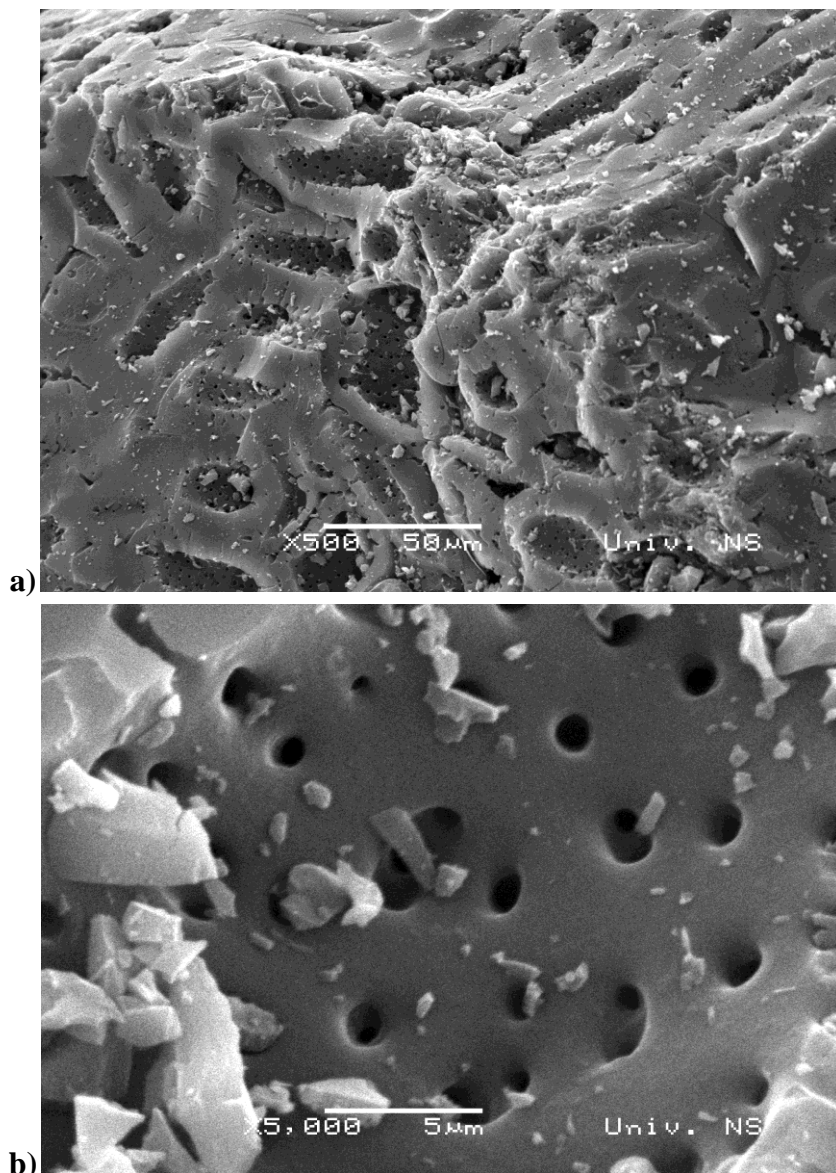


Figure 2. SEM images of the activated carbon (AC) with different magnification

Figure 2 display the SEM images of the surface structure for carbonized material at different magnification. The SEM micrographs of the activated carbon surface show homogenous and widely porous structure of AC after thermochemical activation by phosphoric acidmaking it suitable for trapping more species of pollutants with different sizes. In other words, the presence of various sizes of pores and large extent of surface area make the AC suitable for the adsorption of diverse species.

Surface area of the AC was determined by Brunauer–Emmett–Teller (BET) equation within the relative pressure range from 0.006 to 0.10. The micropore volume were obtained by the Horvath–Kawazoe (HK) method. The total pore volume was calculated from the amount of N_2 adsorbed at a relative pressure. Mesopore volume was calculated by subtracting the micropore volume from

the total pore volume. The textural properties of AC were summarized in Table 1. The BET surface area of AC was evaluated to be $1098.78 \text{ m}^2 \text{ g}^{-1}$. The BJH adsorption cumulative volume of pores for AC was $0.022 \text{ cm}^3 \text{ g}^{-1}$. The total pore volume and micropore volume of AC were determined to be $0.5 \text{ cm}^3 \text{ g}^{-1}$ and $0.391 \text{ cm}^3 \text{ g}^{-1}$, respectively. As can be seen from Table 1, both micropore (<2 nm) and mesopore (2– 50 nm) structures are present in the AC but the micropore volume occupies approximately the 80% of the total pore volume which is great larger than the mesopore volume. The smaller particle sizes of a porous carbon, the greater rate of diffusion and adsorption[5]. As seen from BET analysis, the AC has porous structure and this evidence supports the enhancement of the surface area representing a good sorption capacity of such materials.

Table 1

Textural properties of the synthesized activated carbon

| | BET surface area ($\text{m}^2 \text{ g}^{-1}$) | Total pore volume ($\text{cm}^3 \text{ g}^{-1}$) | Micropore volume ($\text{cm}^3 \text{ g}^{-1}$) | Mesopore volume ($\text{cm}^3 \text{ g}^{-1}$) | Mesopore surface ($\text{m}^2 \text{ g}^{-1}$) | Max. mesopore diameter (nm) |
|----|--|--|---|--|--|-----------------------------|
| AC | 1098.78 | 0.5 | 0.391 | 0.022 | 16.867 | 31.97 |

Conclusion

The activated carbon was synthesized from apricot stones by chemical activation with phosphoric acid. The present study has shown that the activated carbons prepared from apricot stone have a suitable surface characteristics, which can positively affect the adsorption properties with this material.

Acknowledgements

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References

- [1] H. Demiral, C. Gungor, J. Clean. Prod. 124 (2016) 103.
- [2] V. Hernández Montoya and A. Bonilla Petriciolet, Lignocellulosic Precursors Used in the Synthesis of Activated Carbon, InTech, Rijeka, Croatia, 2012.
- [3] I.B. Rae, S.W. Gibb, S. Lu, J. Hazard. Mater. 164 (2009) 1601.
- [4] B. Volesky, B. Volesky, Sorption and Biosorption, BV Sorbex Inc., Montreal-St., Lambert, Que., Canada, 2003.
- [5]. J. Acharya, J.N. Sahu, B.K. Sahoo, C.R. Mohanty, B.C. Meikap, Chem. Eng. J. 150 (2009) 25.