Influence of the generation process on the textural characteristics of activated carbons produced from olive stones by thermal activation, chemical activation, and chemical – wet activation

Abdelmottaleb OUEDERNI,
Mahla ELAMAMI, Souad NAJAR and Naima JABER
Research Unit: Chemical Reactor and Process Control

Chemical Engineering Department, National School of Engineers of Gabes, Gabes University, St. Omar Ibn El Khattab, 6029 Gabes, Tunisia
Mottaleb.ouederni@enig.rnu.tn

Introduction

Agriculture industry by products, as olive stones, are largely used as precursor for activated carbon production. Physical or chemical activation are the most common processes applied in activated carbon generation from carbonic precursors. The physical process involves two stages: carbonisation of the raw material by pyrolysis in inert atmosphere at (600 – 1000°C) followed by thermal activation using vapour, CO_2 or O_2 at high temperature up to 1000°C. In the chemical process, phosphoric acid, zinc chloride or potassium hydroxide are used as activation agent. The precursor is impregnated in concentrated aqueous solution of the chemicals at a given temperature, than a thermo chemical activation is conducted at 400 to 600°C or more. Among the differences in operating conditions and in raw materials, this two generation ways result in different characteristics of activated carbon.

Activated carbon is mostly applied in liquid and gas adsorption operations; also it is used in catalysts production and some other specific applications. The main performances of activated carbon used in different fields depend on textural and physical-chemical surface proprieties. The main challenge in activated carbon production process is the control of the pore size distribution, the internal specific area and the surface chemical proprieties of the product in goal of each application.

This work compares the principal textural characteristics of activated carbons produced from olive stones by three different processes.

Experimental

Three activated carbons (ACs) are generated from olive stones by different process: physical (ACP), thermochemical with H_3PO_4 activation (ACC) and wet thermochemical (ACW).

In the physical process the precursor is previously carbonized in nitrogen stream at 600°C and than activated with steam streamed in nitrogen flow at more than 800°C. The chemical process uses phosphoric acid and occurred in two steps. Prior to the thermochemical activation, the granular olive stones is impregnated within aqueous phosphoric acid solution. Activation was carried out by heating the impregnated solid under a pure nitrogen continuous stream in rising temperature to 410°C. The mixed process follows the chemical one and instead pure nitrogen stream, a mixture of steam and vapor is applied during thermal activation step.

Vertical fixed bed gas solid laboratory scale reactor is used for different operating conditions which are well controlled and optimized in other works.

Results and Discussion

Textural characteristics are investigated by using nitrogen isotherms adsorption-desorption at 77K performed in Autosorb AS1-C Quantachhrome apparatus. The BET specific areas estimated from nitrogen isotherms are reported in table 1.

Table 1. Specific area of different AC

Activated carbon	ACP	ACC	ACCW
Specific area (m^2/g)	**800 to 900**	**1150 to 1250**	**1600 to 1700**

The ACC and ACCW are essentially microporous with more than 95% of pore volume occupied by pores with less than 2 nm depth. The ACP adsorption-desorption isotherms shows hysteresis loop in desorption back; the isotherm shape is intermediate between type I and type II. The ACC exhibit an isotherm shape of type I.

MEB micrographics show different microstructure shapes. The physical process transforms and cuts the raw cellular enrolled tubular shape of the raw lignocellulosic material in empty hallow “potates” with some regular perforations on the wall (fig. 1). The length of those native shapes is much shorter than the raw lignocellulosic cells. We observe everywhere some regular perforations on the wall. We note that hallows were observed in different SEM micrographic of activated carbons made by physical process from olive stones, it seems to be a specific print of this process [1].

Activated carbon made via phosphoric acid activation, has completely different microscopic texture, the original lignocellulosic cells shape disappears and gives micro granular particles, in micro size range, within quite regular spherical shape in less than 1µm diameter (fig.2). Some of these microgranules are integrally detached from the original cell wall; others are compiled and aggregated and appear in distorted wall shape. Introduction of vapour in the process (ACPW) gives stacked lamellar shapes (fig. 3). EDAX survey spectrum of the ACC indicates the presence of three distinct peaks due to carbon, oxygen and phosphorus. The displacement of the X-ray probe on the ACC surface shows the presence of equally distributed phosphorus compounds, this observation of phosphorus insertion in AC structure is cofirmed in other published works [2,3]

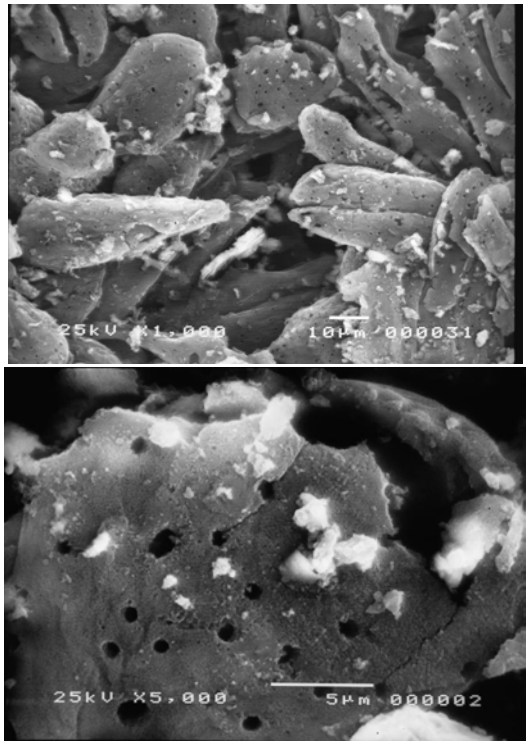

Fig.1 Micrograph MEB of AC produced from olive stones by physical process

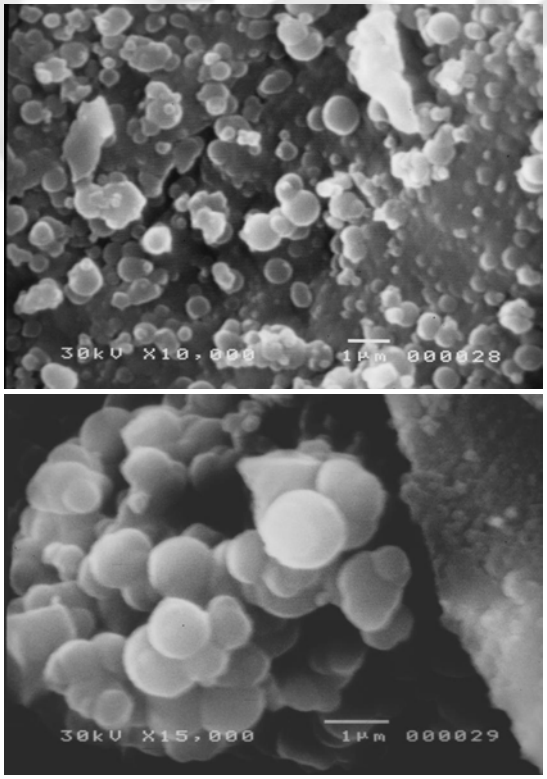

Fig.2 Micrographs MEB of AC produced from olive stones by chemical process

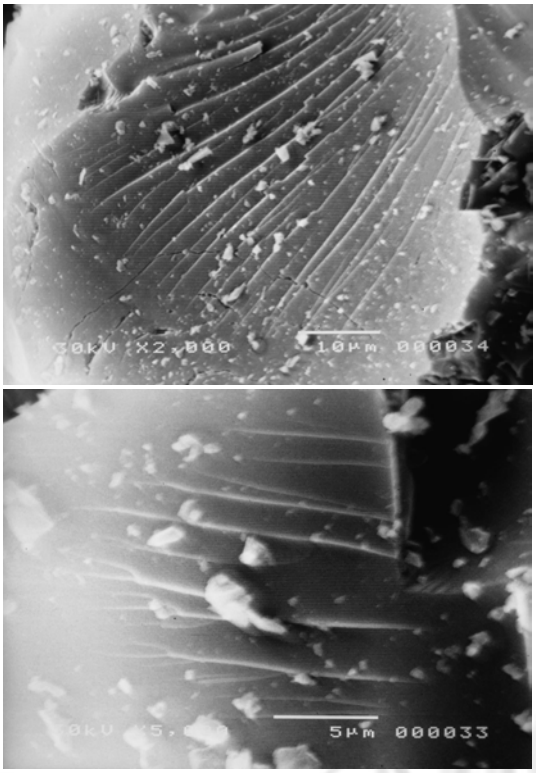

Fig.3 Micrograph MEB of AC produced from olive stones by chemical – wet process

Conclusion:

The process performed to convert olive stones in activated carbon is determinant for final textural characteristics of the product. Among different specific area and porosity proprieties, the microscopic shape of micrometric skeleton is clearly influenced by operating conditions specific to each process. This offers different ways in development of specific applications of thus activated carbons.

References

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