Preparation and characterization of activated carbon from olive stones

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Introduction & Objectives:

This work aims to develop a new efficient and cheap activated carbon. The raw material consists on a by agricultural product: olive stones. The optimal conditions of activation such as temperature and phosphoric acid concentrations were determined. The surface parameters were determined according B. E. T. analysis and scanning electron microscopy (S. E. M.).
A good development of porosity confirmed high values of surface area and microporous volume.

Methodology (Material and methods):

Preparation of AC

As-received olive stones were first washed with boiled distilled water, dried during 14 hours in oven at 110°C. Then, stones are washed by chloridric acid solution 0.1N during 1hour at 60°C. Stones are dried, crushed then sieved to 0.2-0.4 mm. the resulting material is impregnated by different concentration H_3PO_4 solutions (20 to 85%). The mixture was agitated at 85°C for 2 H, filtered and dried.
The resulting solid was then carbonized within an inert condition under the flow of N_2 at 410 in a modified carbonization reactor.

Characterization of activated carbon (AC)

N_2 adsorption and desorption characterization: The specific surface area and porosities of washed and dried samples were performed using a "COULTER" 3100 AS model porosimeter by nitrogen gas adsorption at 77 K. The micropore volume was measured by t-plot method [1], the total specific pore volume was calculated by liquid N_2 adsorbed at a relative pressure of 0.99 [2] and the pore size distribution calculated using the BJH model [3, 4].

Surface morphology: surface morphology was taken by JSM-5500 scanning electron microscope.

Results and Discussion

BET results

Adsorption isotherms (fig. 1) indicate that the N_2 adsorption occurs essentially at low pressures, especially for C1, C2, C3 and C5 which indicates the formation of microporous material with a very narrow pore size distribution [5]. For 85%, adsorptive capacity increases and hysteresis is extended until low relative pressures. Such result reveals a very developed microporosity (2).

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Specific surface area and microporous volume development increase with H_3PO_4 concentration and reach a maximum of 1874.63 m^2/g and 0.77cm^3/g respectively (Table 1).

Table 1. Specific surface area, (S), microporous volume, (V_m), and total porous volume (diameter below 100 nm), W_0.

Sample	C1	C2	C3	C4	C5	C6
[H_3PO_4](%)	20	30	45	60	75	85
S_{BET} (m^2/g)	279.02	571.68	629.45	922.08	1233.46	1874.63
V_m (cm^3/g)	0.00593	0.075	0.158	0.263	0.556	0.770
W_0 (cm^3/g)	1.864	0.746	0.263	0.687	0.709	1.755

Figure 1: Adsorption isotherms of different carbons

Scanning electron microscopy (SEM)

Initial morphology disappears completely when H_3PO_4 concentrations are over 45%. Picture of C4 carbon, shows a good formation of pores and an irregular surface formed by crevices,. The SEM image C6 (Fig. 3) show the development of capillary-like pore structures on the AC

surface. Some particulate matter appears on the external surface of the activated carbon, which may be related to the activation agent. The best porosity is developed by C6.

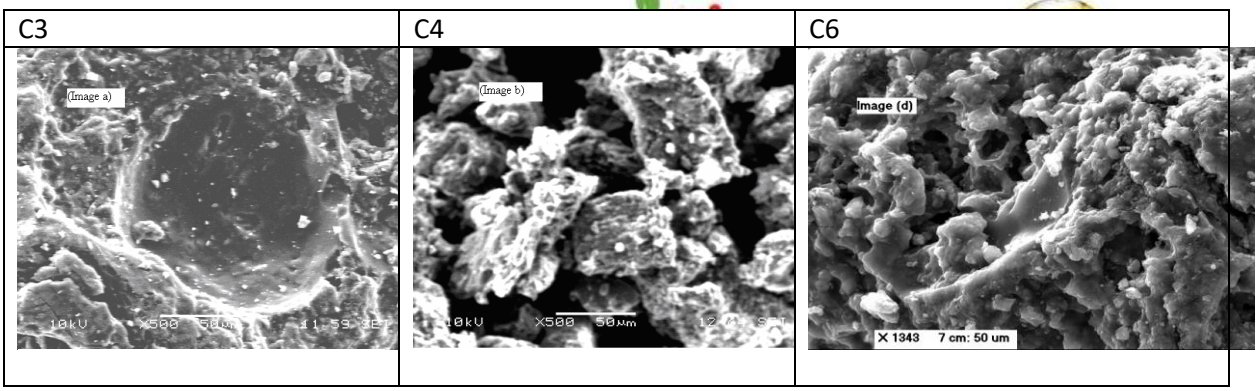

Figure 3: Scanning electron micrographs of C3, C4 and C6

Conclusion

Olive stones material is a suitable raw when activated with H_3PO_4 to prepare AC. When washed stones particle sizes between 0.2-0.4 mm. the optimized conditions for the preparation of AC are as follows: H_3PO_4 varies from 45 to 85 %, activation time 120 min at 410 °C. BET surface area increases from 629.45 to 1874.63 $m^2\ g^{-1}$. The AC is mainly microporous.

Key words: activated carbon (AC), surface area, activation, phosphoric acid.

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