

ACTIVATION OF MESOPHASE BY OXYGEN PLASMA

A. R. Coutinho

UNIMEP, Technological Center
13450-000, Santa Bárbara D'Oeste, SP, Brazil,

C. Otani, M. Massi, S. Otani and H. S. Maciel

ITA-Physics Department
12280.000 - São José dos Campos, SP, Brazil

C. A. Luengo

UNICAMP, IFGW,
13083-970, Campinas, SP, Brazil.

Intruduction

Active carbons (AC) are characterized by large specific surface areas and high porous volumes, their main property is to adsorb gases and liquids [1]. Due to their microporous structure, AC are applied for several adsorption processes such as water treatment, gas mixture separation, chemical products purification, and others. The conventional process of AC production utilizes coal and biomass, which are carbonized at high temperature and activated by steam, air or CO₂. Recently, AC are obtained from new raw materials, cellulose, carbon fibers, mesocarbon microbeads [2-4]. Moreover, new techniques like chemical processes and plasma corrosion were also employed [5-8].

Experimental

In this work, naphthalene commercial mesophase, from Mitsubishi Gas Chemical Co [9-11] was heated in inert atmosphere at 350°C with a heating rate of 0.07°C/min then to 800°C, using 1.0°C/min. The carbonized mesophase (MFC) was crushed, screened and compacted in discs, 25 mm diameter and 1.0, 2.0 mm thickness. The MFC discs were submitted to plasma treatments. The glow discharges were generated by RF at 13.6 Mhz. Pressure during treatments was kept at 380 mTorr and oxygen gas flowing at a rate of 185 sccm. The RF powers used were: 25, 50, 75, 100 and 150 Watts and treatment times: 1, 2, 3 and 4 hours. In each case, the MFC sample was named: MFC-P1, MFC-P2, MFC-P3 and MFC-P4, respectively. The activated mesophase (AMF) was characterized by XRD, Scanning Electron Microscopy and Porosimetry.

Results

Table 1. Physical and chemical properties of green and carbonized MF.

properties	green MF	carbonized MF
melting point (°C)	260	-
anisotropy (%)	100	-
toluene insolubles (%)	99	-
volatiles (%)	18.9	1.2
fixed carbon (%)	81.1	98.7
ash (%)	0.01	0.01
density (g/cm ³)	0.65	1.4

Figure 1 shows weight losses of MFC, treated by O₂ plasma, as a function of RF power. Treatment induced larger losses in samples with smaller thickness. In those cases, treatments were of four hours.

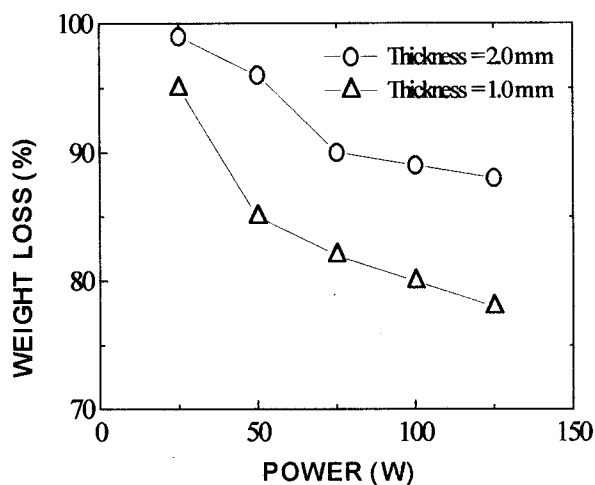


Figure 1. Weight loss of MFC treated by O₂ plasma

Plasma treated MFC present amorphous structure. The parameters L_c and d_{002} determined by XRD of 2,0 mm, were treated using 150 W of RF power. It is shown that layer spacing decrease with increasing exposure time.

Table 2. XRD parameters of MFC treated by O_2 plasma

Sample	d_{002} (Å)	L_c (Å)
MF original	3.50	30.79
MF Carbonized	3.47	31.97
MFC-P1	3.45	33.58
MFC-P2	3.42	32.07
MFC-P3	3.40	33.81
MFC-P4	3.37	33.56

The pore distribution of MFC-P4 samples, treated by O_2 plasma at 150 W, is shown in figure 2 and porous concentration in figure 3, both as a function of pore radius.

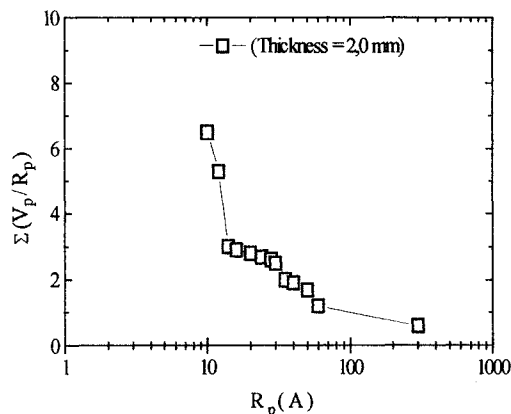


Figure 2. Porous distribution of MF treated by O_2 plasma, as a function of pore radius

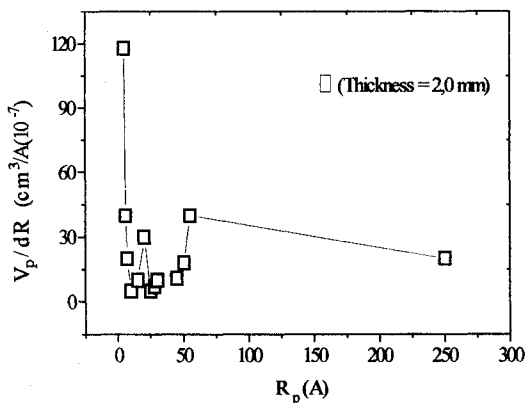


Figure 3. Pore concentration of MF treated by O_2 plasma, as a function of pore radius

Scanning electron microscopy of MFC-P4, treated by oxygen plasma, at 150 W, is showed in figure 4.

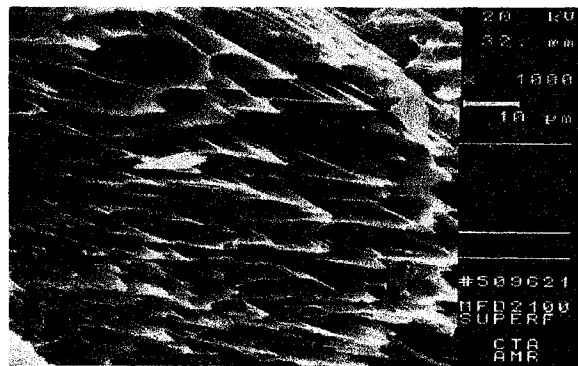


Figure 4. Micrograph of MFC treated by O_2 plasma.

Conclusions

Spacing d_{002} values decrease with increasing exposure time which may indicate that MF samples lose weakly bounded atoms. However, amorphous structure persists. Microscopy shows important pore corrosion, caused by oxygen reaction with carbon surface. The main micropore concentration was observed between 10Å to 50Å , with a small contribution of meso and macropores.

Aknowledgements

The authors wish to thank FAPESP, for its financial support (Process:95/9627-6)

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