INFLUENCE OF THE PRECURSOR'S COMPOSITION ON THE PREPARATION OF PITCH-BASED CARBONS WITH MOLECULAR SIEVE PROPERTIES

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Introduction

A previous study [1] has shown that activated carbons with developed microporous properties can be produced from the toluene-insoluble fraction of heat-treated pitches. However, these properties appeared to depend strongly influenced on the composition of the pitch, mostly in the first stage of activation (burn-off < 40%). The present study was focused on the relation between pitch composition and the microporous structure developed during the activation process.

Experimental

The experimental procedure for the preparation of activated carbons from raw A240 petroleum pitch is given elsewhere [1]. Here, two heat-treated and toluene-fractionated pitches were chosen as precursors. The properties of such precursors depend mainly on their content of low molecular weight species, which have been partially removed during toluene extraction. The fractionated pitches EP1 and EP2 have residual solubilities in toluene of respectively 12 and 5% in weight. Activation was carried out with carbon dioxide at 900°C for various periods of time, and activated carbons with burn-off ranging from 10% to 60% were obtained. These materials were characterized by high-pressure CO_2 adsorption and immersion calorimetry [2].

Results and Discussion

The main difference between precursors EP1 and EP2 is the higher content of γ-resins in EP1. Large isotropic domains, surrounding the anisotropic β-resins particles, were observed and they are thought to be responsible for the strong inhibition of microporosity development during the activation of EP1 [1]. The average micropore width *Lo,* determined from nitrogen adsorption, was also found to depend strongly on the composition of the precursor, a higher γ-resin content leading to larger *Lo* values.

The determination of pore size distributions (PSD) by both, high pressure carbon dioxide adsorption and immersion calorimetry, led a better understanding of the influence of the precursor's composition. As shown in Figures 1 and 2, the PSDs obtained by CO2 adsorption for EP1(12%) and EP2 (13%) were very similar, with average pore widths *Lo* around 0.7-0.8 nm. The real differences between the two structures was only revealed by immersion calorimetry. EP1(12%) is a genuine molecular sieve with a narrow pore size distribution, whereas EP2(13%) shows a gate effect with a major proportion of micropore sizes in the range of 0.3-0.5nm.

As shown in Figures 3 and 4, these specific microporous structures were eliminated by stronger activation and for a 37wt% burn-off, the PSD of carbons EP1 and EP2 are the similar as those usually observed for carbon dioxide activation (broader distributions).

Conclusions

By removing species of low molecular weight, toluene fractionation helps to produce activated carbons with high micropore volumes [1]. However, the extraction procedure influences the microporous structure of the subsequent active carbons. After activation with CO2 at low burn-off, pure β-resins precursors such as EP2, obtained by strong toluene fractioning, lead to unusual microporous structures with gate-effects. Such molecular sieve properties appear to depend on the composition of the precursor, as well as on the degree of gasification.

References

1. Daguerre E, Guillot A and Py X. Elaboration of activated carbons using T.I. fraction of A240 heat-treated pitch. Extended Abstract, 23th biennal conf. on carbon, PennState, American Carbon Society, 199X;156-157.

2. Stoeckli HF. Characterization of Microporous Carbons by Adsorption and Immersion Techniques. In: Patrick JW, editor. Porosity in Carbons ,Great Britain: Edward Arnold, 1995:67-92.

Samples	Wo (cm3/g)	Lo (nm) N_2 ads.	Lo (nm) CO_2 ads.
EP1-BO12%	0.07	0.92	0.7
EP2-BO13%	0.17	0.71	0.7
EP1-BO37%	0.14	0.11	0.83
EP2-BO37%	0.28	0.14	0.9

Table 1. Microporous properties of activated carbons

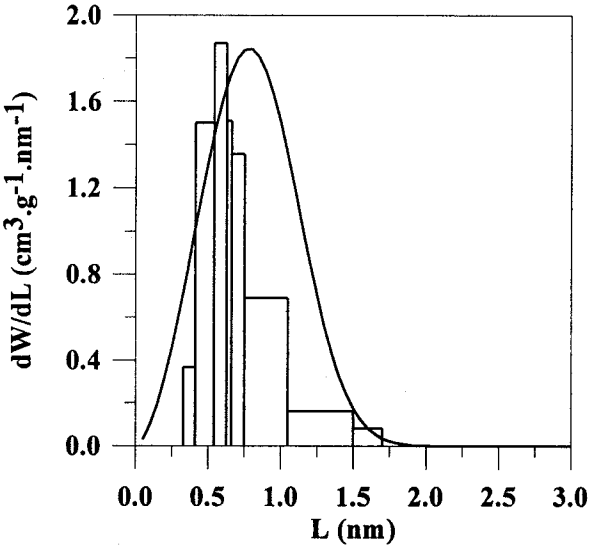

Figure 1. Pore size distribution for activated carbon EP1 (burn-off 12%) determined by CO_2 adsorption and immersion calorimetry.

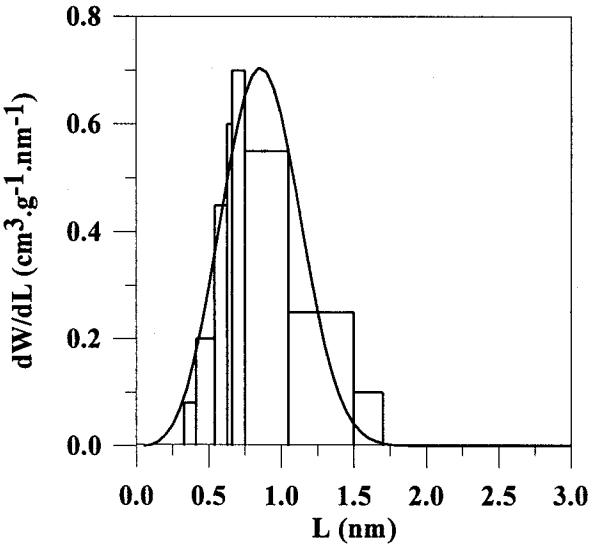

Figure 3. Pore size distribution for activated carbon EP1 (burn-off 37%) determined by CO_2 adsorption and immersion calorimetry.

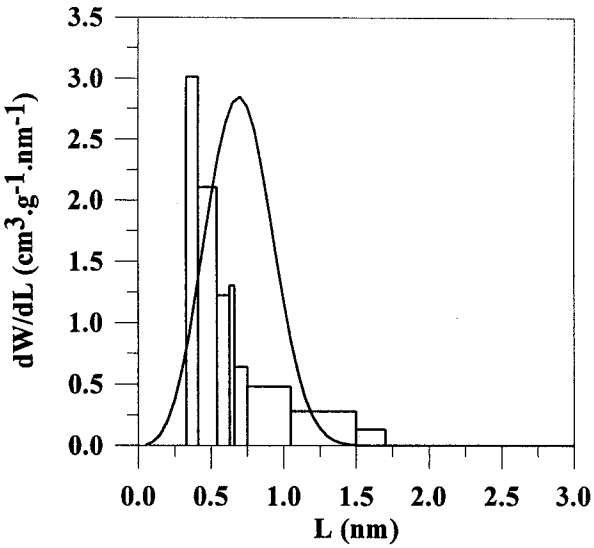

Figure 2. Pore size distribution for activated carbon EP2 (burn-off 13%) determined by CO_2 adsorption and immersion calorimetry.

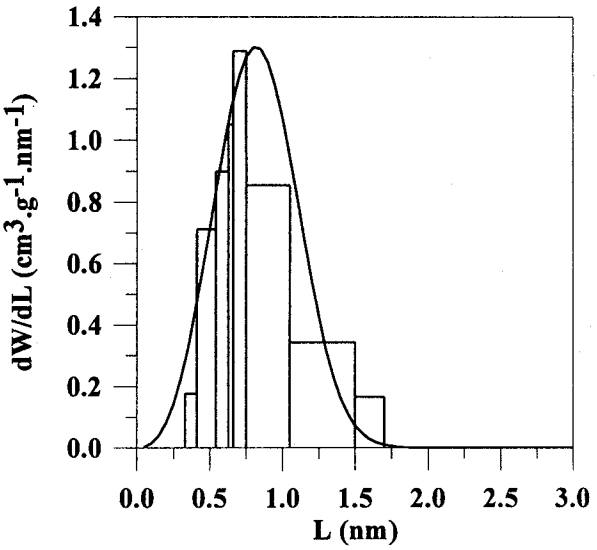

Figure 4. Pore size distribution for activated carbon EP2 (burn-off 37%) determined by CO_2 adsorption and immersion calorimetry.

Acknowledgements

This study was part of a CNRS-ECOTECH contract # 94N80/0086 with the support of ADEME.