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Influences of impregnation ratio and activation time on ultramicropores of peanut shell active carbons

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ABSTRACT

We studied effects of impregnation ratio and activation time on ultramicropores of KOH-activated peanut shell active carbons (ACs). The ultramicropores were characterized in terms of ultramicropore volume ($V_{<0.7\ nm}$), ultramicropore fraction ($V_{<0.7\ nm}/V_t$), and pore size distribution. The ultramicropores of the ACs were mostly in the range of 0.45–0.70 nm. Increasing the impregnation ratio from 1 to 3 first increased the $V_{<0.7\ nm}$ and $V_{<0.7\ nm}/V_t$, and then decreased them. Prolonging the activation time from 1 h to 2 h resulted in a minimum $V_{<0.7\ nm}/V_t$, but hardly affected the $V_{<0.7\ nm}$. The AC with the highest $V_{<0.7\ nm}$ (0.11 ml/g) was obtained by activation for 1–2 h at an impregnation ratio of 2. The $V_{<0.7\ nm}$ bore a linear relationship with the CO_2 uptakes at 0.1 and 0.2 bar (0 °C). This research was significant for preparation of ACs with developed ultramicropores.

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1. Introduction

Preparation of active carbons (ACs) from peanut shells has aroused much interest [1–5], due to the abundant availability of the shells and wide applications of the ACs [6–9]. So far, peanut shell ACs have been prepared by activation using various agents including KOH [2–5,7,8], with much attention paid to effects of process parameters on pore properties like surface area and pore volume. However, the influences of process parameters on ultramicropores of peanut shell ACs remain unknown.

According to IUPAC classification, ultramicropores are the pores with sizes less than 0.7 nm [10]. Such pores have been found highly important for adsorption of some gases like CO_2 [10,11] and H_2 [12–14]. Hence, knowing how ultramicropores vary with process parameters is necessary for improving gas separation or storage.

This research aimed to investigate effects of impregnation ratio and activation time on ultramicropores of KOH-activated peanut shell ACs. The ACs were characterized in terms of surface morphology and pore properties such as ultramicropore size distribution, ultramicropore volume, and ultramicropore fraction. The research could provide guidelines for preparation of highly ultramicroporous ACs.

2. Experimental

Active carbons (ACs) were prepared by KOH activation of peanut shell char (PSC), with the KOH and PSC described in Text S1 of Supplementary material (SM). To prepare ACs, 4.00 g of PSC with sizes of 0.28–0.90 mm was mixed with KOH dissolved in 14-ml distilled water at an impregnation ratio (KOH/PSC weight ratio) of 1–3. After being shaken for 5 h, the mixture was dehydrated at 140 °C under N_2 until constant weight. Subsequently, the dried mixture was heated in N_2 flow first to 400 °C with a retention time of 30 min at a heating rate of 8 °C/min, and then to 780 °C with a retention time of 1–2 h at 10 °C/min. The as-obtained sample was cooled, washed repeatedly with distilled water until the washings were about neutral, and finally dried at 110 °C overnight. The prepared active carbon was named AC*x*–*y* h, where *y* h and *x* denoted activation time in hours and impregnation ratio, respectively. For example, AC1–2 h represented an active carbon prepared by activation for 2 h at an impregnation ratio of 1 using the aforementioned process.

Surface morphology of samples was observed using a scanning electron microscope (SEM, S4800, Hitachi Corp., Japan). N_2 adsorption/desorption isotherms at 77 K were measured by a static

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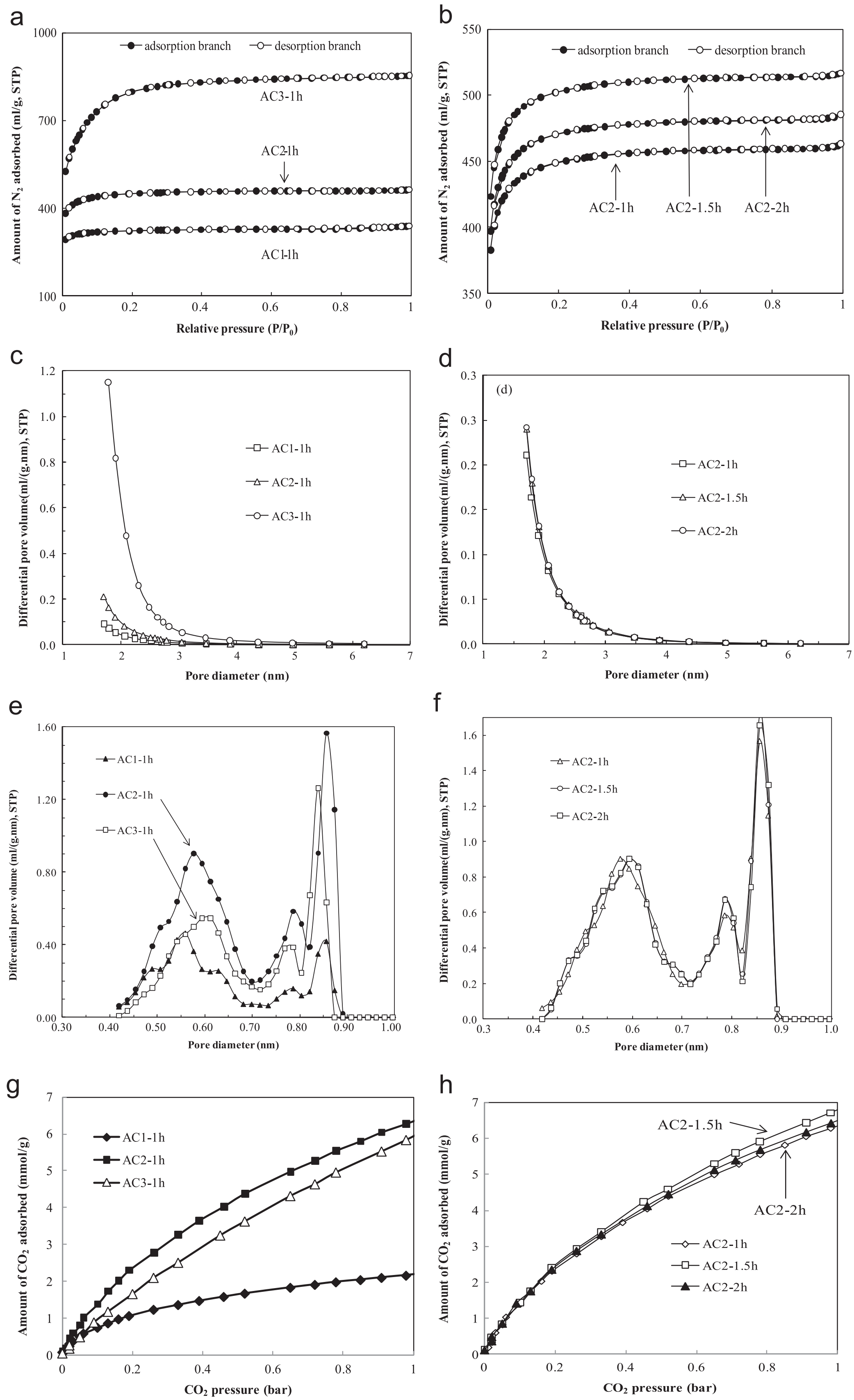

Fig. 1. N_2 adsorption/desorption isotherms (a, b), mesopore size distributions (c, d), ultramicropore size distributions (e, f), and CO_2 adsorption isotherms at 0 °C (g, h) of active carbons activated for 1–2 h at impregnation ratios of 1–3.

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