

Synthesis of nanoporous carbon: An in situ template approach

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Abstract

A new approach for synthesis of nanoporous carbon is reported. This approach involves: (1) synthesis of the template of iron nanoparticles in situ with formation of carbon by thermal pyrolysis of $\text{Fe}(\text{CO})_5$ and C_2H_2 , and (2) removal of the template by air oxidation and acid treatment. The obtained nanoporous carbon had a high specific surface area of $729 \text{ m}^2/\text{g}$ and pore sizes of 1–10 nm. The present template approach is simpler than previous ones involving separate preparation of template materials.

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1. Introduction

The synthesis of nanoporous carbon has been an intensive research area for many years. This is because such material has important applications in many areas, such as adsorption, separation, catalysis, and electrode materials [1–6]. A number of methods for the preparation of nanoporous carbon have been developed, with the traditional one being the carbonization-activation route [7,8] used to produce activated carbon. However, the controllability of the porosity parameter of the nanoporous carbon is limited, as the carbon precursors are generally in solid state [9]. Recently, there has been a growing interest in using nanostructured silica as a template for preparation of ordered and uniform nanoporous carbon materials [10–14]. This method usually contains five steps: (1) preparation of the nanoporous silica; (2) removal of the structure directing agent (surfactant); (3) synthesis of a carbon precursor/silica nanocomposite; (4) carbonization; and (5) removal of the silica template. The synthesis of the template is a separate step from the formation of carbon, and the template filling process is sophisticated, resulting

in complication and time-consuming of the whole procedure [15,16]. In this study, we report a new template method for the preparation of nanoporous carbon. Iron nanoparticles being the template were synthesized in situ with the formation of carbon so that the procedure was much simpler than those reported before.

2. Experimental

The initial sample was fabricated by thermal pyrolysis. A gas mixture of C_2H_2 and iron pentacarbonyl [$\text{Fe}(\text{CO})_5$] was introduced into a vertical tube furnace with N_2 as a diluting and carrying gas. In order to add $\text{Fe}(\text{CO})_5$, N_2 and C_2H_2 gases flowed through the liquid carbonyl which was held at 0°C . Typical flow rates were 60 L/h for N_2 and 10 ml/min for C_2H_2 . The operating pressure in the quartz tube (\varnothing 30 mm) was slightly larger than 1 atm and the temperature was set at 700°C . The initial sample was collected at the bottom of the quartz tube. To remove the iron template, the initial sample was oxidized in air at 350°C for 1 h first and then ultrasonicated in 37 wt.% HCl for 8 h. The sample was finally filtered and dried for analysis. In order to investigate the yield of nanoporous carbon and the amount of remnant iron, the final sample was completely oxidized in air at the temperature of 800°C for 1 h. The mass change in each step was recorded.

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The microscopic feature of the initial sample was studied by transmission electron microscopy (TEM), selected area diffraction (SAD), high resolution TEM (HRTEM) and energy dispersive X-ray spectroscopy (EDS). HRTEM specimens were prepared by dispersing the sample in alcohol and then dipping the solution on a copper grid with a carbon film. X-ray diffraction (XRD) was performed to study the crystallization of carbon and iron phases contained in the sample.

N₂ adsorption experiments using BELSORP measuring instruments (BEL, Japan, Inc.) were carried out in order to investigate the porosity of the samples before and after template removal. All the samples were outgassed at 200 °C under nitrogen flow for about 4 h prior to measurement. The nitrogen adsorption data were recorded at the liquid nitrogen temperature (77 K). The specific surface area (SSA) was calculated using the Brunauer–Emmett–Teller (BET) equation. The micropore size distributions

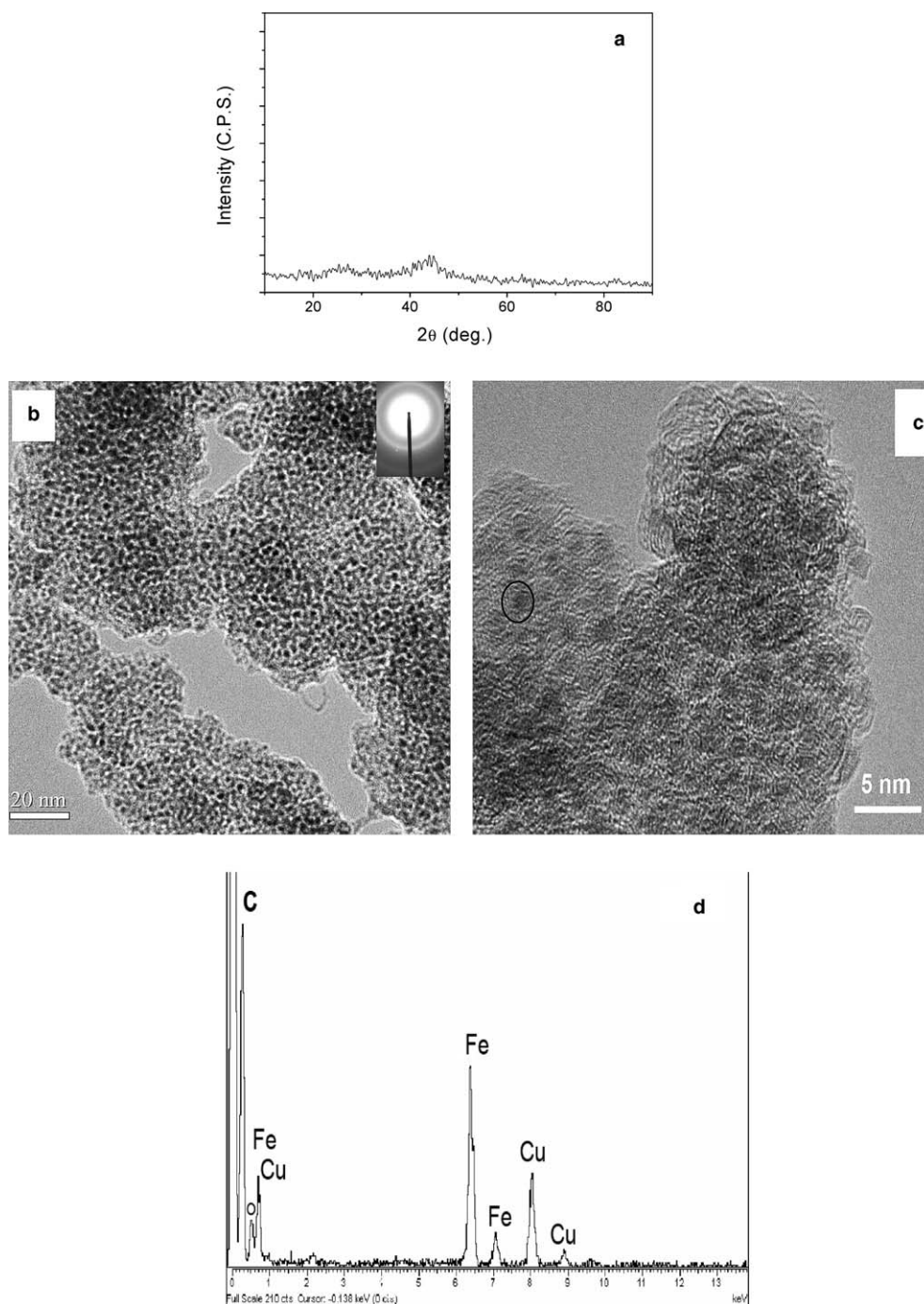


Fig. 1. XRD (a), TEM image with SAD (b), HRTEM image (c) and EDS analysis (d) of the initial sample, showing carbon matrix (grey) with embedded iron particles (black) and their poor crystallization. The EDS analysis (d) was performed on the circled dark region (Fig. 1c), confirming the existence of iron. The Cu peak may result from the TEM Cu grid, and that of O may be caused by oxidation of the initial sample when it was exposed in air.

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