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Short communication

Effect of carbothermal reduction on the microstructures of porous carbons from the mixture of coal-tar pitch and nano-MgO

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ABSTRACT

Porous carbons were prepared by pyrolysis of the mixture of coal-tar pitch and nano-MgO, and the effect of carbothermal reduction between nano-MgO and carbons on their microstructures was studied. Pyrolysis behavior of the mixture was characterized by simultaneous thermogravimetry–differential scanning calorimetry (TG–DSC) and X-ray diffraction (XRD). In addition, the microstructures of resulting porous carbons were studied using scanning electron microscopy (SEM), transmission electron microscopy (TEM), Raman spectroscopy and nitrogen adsorption-desorption measurement. Results show that the carbothermal reduction occurs when the temperature is beyond 1300 °C, greatly influencing the microstructures of the resulting porous carbons. With the increasing of temperature, the porous carbon prepared possesses a thinner thickness of pore wall and a higher specific surface area with a less mesopore volume. When the temperature arrives at 1800 °C, the porous carbon prepared exhibits three-dimensional network structures consisting of few-layered graphene sheets.

1. Introduction

Porous carbons have attracted increasing attention due to their high specific surface area, low density, large pore volume, well-developed pore structure, high thermal stability, good electronic conductivity, and so on [1–3], and therefore they are widely used in many fields ranging from gas storage, absorbent, catalyst supports to electrode materials [4–7]. As is well known, properties of porous carbons are closely related to their pore structures. Porous carbons, which possess well-controlled pore structures, have excellent properties and good application prospect. In recent years, the methods for preparing porous carbons mainly include physical or chemical activation [3], direct carbonization of polymers or their mixtures [8], organic carbon-based gel method [9] and template method [10], among which template method has been demonstrated to be quite effective to prepare porous carbons with well-controlled pore structures.

Nano-sized magnesium oxide (nano-MgO) has been considered as an appropriate template for preparing porous carbons with well-controlled pore structures, which can be attributed to its characteristics like low cost, easy removal and good recycling [10]. It is usually selected as a hard template mainly because of its thermal and chemical stabilities, compositional and structural stabilities, and lack of reaction with carbon precursors up to carbonization temperature [11]. However, a series of carbothermal reductions between MgO and carbons can occur when the temperature arrives at a certain value [12]. To our knowledge, there are few related reports on preparing porous carbons using nano-MgO or its precursors as templates at relatively high temperatures. In this work, we attempt to investigate the effect of carbothermal reduction on the microstructures of porous carbons from the mixture of coal-tar pitch and nano-MgO.

2. Experimental

2.1. Raw materials

The raw materials used were commercial coal-tar pitch (softening point, 270 °C; coking value, 70%; quinoline insoluble content, 20%; toluene insoluble content, 65%; mean particle size,75 μm) and nano-MgO (purity >99.9%; mean particle size, 50 nm).

2.2. Preparation of porous carbons

5 g of coal-tar pitch was mixed with 7.5 g of nano-MgO by thorough grinding in a mortar. The mixture obtained was transferred to a graphite crucible with a lid, placed in a furnace and then degassed to remove entrapped air under high vacuum (<0.01 Pa). After that it

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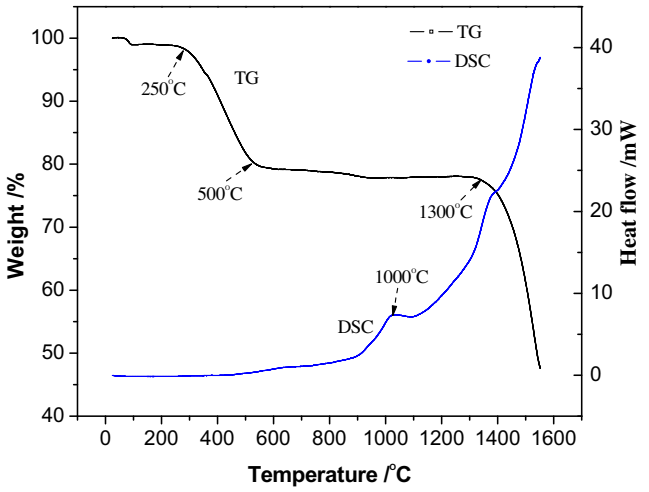

Fig. 1. TG-DSC curves of the mixture of coal-tar pitch and nano-MgO.

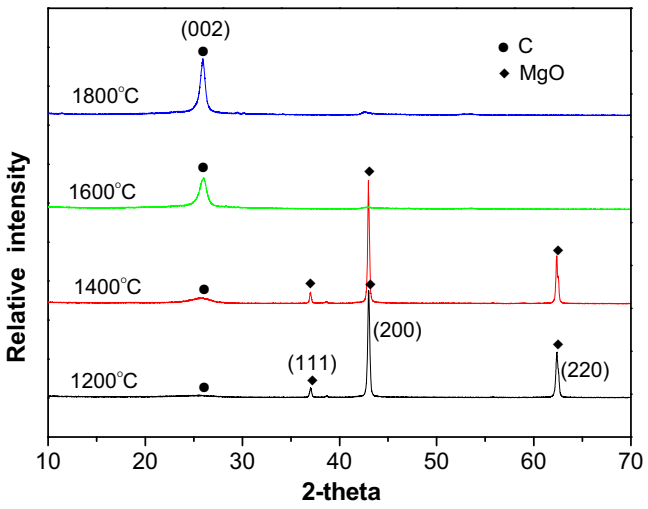

Fig. 2. XRD patterns of the carbonized products from the mixture of coal-tar pitch and nano-MgO at different temperatures.

was heated up to a certain temperature (1200 °C, 1400 °C, 1600 °C, or 1800 °C) at a rate of 10 °C/min and maintained for 2 h in nitrogen atmosphere, and then cooled naturally to room temperature. The carbonized product was dispersed in 500 mL of HCl solution (0.5 mol/L) at 50 °C and stirred for 4 h. Afterwards, it was washed with hot distilled water (85 °C) until the pH of the filtrating solution was adjusted to 7, and then dried in vacuum at 100 °C for 12 h to obtain porous carbon. The porous carbons prepared were labeled as PC-1200, PC-1400, PC-1600, and PC-1800 when the carbonization temperature was 1200 °C, 1400 °C, 1600 °C, and 1800 °C, respectively. The yield of the porous carbon was calculated:

$$Y(\%) = W_1/W_0 \times 100, \quad (1)$$

where W_0 is the mass of coal-tar pitch and W_1 the mass of the porous carbon. Here the yields of PC-1200, PC-1400, PC-1600, and PC-1800 were 52.6%, 40.1%, 27.5%, and 14.3%, respectively.

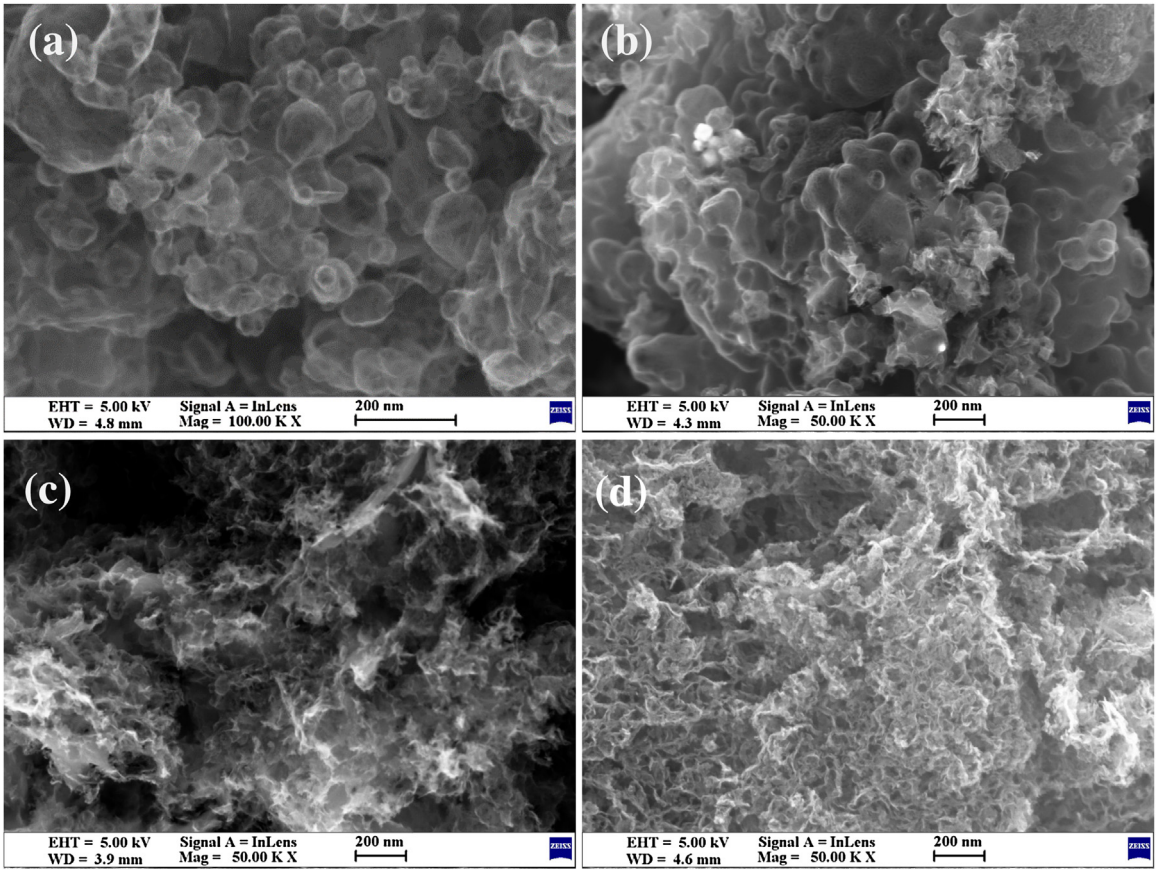

Fig. 3. FESEM images of the four porous carbons prepared. (a) PC-1200; (b) PC-1400; (C) PC-1600; (d) PC-1800.