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Fabrication of high-strength macroporous carbons with tunable pore size by a simple powder process using phenolic resin microspheres

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ABSTRACT

High-strength macroporous carbons with tunable pore sizes were fabricated by the simple mixing–pressing–hardening–carbonization of phenolic resin microspheres as a matrix with acid-catalyzed furfuryl alcohol as a binder. Pore-size tuning with the open cell structures was possible by changing the diameters of the phenolic microspheres in the range of 8–100 μ m. After pressing the mixture of phenolic resin microspheres and binder, hardening at 170 °C for 5 h in air, and carbonizing at 1000 °C for 1 h in nitrogen flow, the macroporous carbon was formed. The mean pore sizes of the macroporous carbons were 1.8, 6.6, and 27.5 μ m for 8-, 25-, and 100- μ m-diameter phenolic microspheres, respectively, and the corresponding bulk densities were 1.01, 1.00, and 0.94 g/cm³. The compressive strengths of the fabricated macroporous carbon samples were 106.8, 83.5, and 43.6 MPa for the 8-, 25-, and 100- μ m-diameter phenolic microspheres, respectively, which were notably higher than those of typical carbon foams. The simple fabrication process with easily tunable pore size, which yields macroporous carbons with remarkably high mechanical strength, has potential for large-scale production and many applications.

1. Introduction

Porous carbons such as carbon foams are used as catalyst supports, filters, refractories, electrodes, and energy storage materials in various fields [1–6], owing to their light weight and excellent properties, such as high temperature resistance, chemical inertness, and tailorable electrical and thermal conductivities. The remarkable properties of porous carbons are based on their varying pore sizes from the microscale (diameter $< 2\ \mathrm{nm})$ to macroscale (diameter $> 50\ \mathrm{nm})$ and porosities, which can reach 95%. Therefore, it is necessary to select appropriate raw materials and establish manufacturing methods that allow for easy control of the pore structure of such carbons through simple and appropriate processes.

Since 1964, when carbon foams were first prepared by Ford via carbonizing phenol-formaldehyde [7], various materials including phenolic resin [8,9], polyurethane [10], petroleum pitch [11], and biomass [12,13] have been considered as raw materials for carbon foams. These carbon precursors can be classified into two main groups: hydrocarbon derivatives and polymers. Polymers generally have lower carbon yields than aromatic hydrocarbons because they contain elements such as chlorine, oxygen, and nitrogen in addition to hydrogen

and carbon. However, they are easier to process as they require no modification or pretreatment before use. According to Reitz et al. [14], the carbon yields after carbonization were high in coal tar pitch (52.5%), phenol-formaldehyde (52.1%), and liquid furfuryl polymer (49.1%), while they were relatively low in epoxy resin (10.1%) and urethane resin (8.2%). Polymers such as phenol-formaldehyde that have high carbon yields are more economic and hence preferred as raw materials for porous carbons.

The most critical factor in tailoring the pore structure of porous carbons is the manufacturing method. Several methods have been explored, including the carbonization of porous polymers through heat treatment [10,15], templating methods using silica or other materials [16,17], and chemical reactions [18,19]. However, polymer carbonization usually causes large deformations of the pore structure [10], and templating methods require additional steps for template removal [16, 17]. Meanwhile, chemical reaction methods are challenging in terms of controlling the pore structure [20].

Porous carbons such as carbon foams have inferior mechanical properties owing to their sponge-like cell structures, which limit their applications [21,22]. The improvement of the mechanical properties of porous carbons by reinforcing them with fibers or powders has been

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