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Experimental and theoretical approach to evaluation of nanostructured carbon particles derived from phenolic resin via spray pyrolysis

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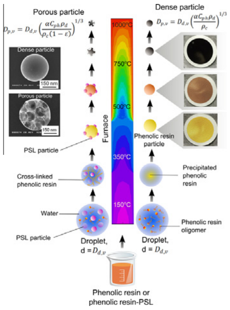
HIGHLIGHTS

- Droplet diameter was measured using laser diffraction technique.
- Pyrolysis conversion of phenolic resin to nanostructured carbon was evaluated.
- New equation to predict carbon particle size was developed from mass balance.
- Shrinkage of carbon particle is included in the proposed equation.
- Particle diameters from proposed equations and measurement are in good agreement.

GRAPHICAL ABSTRACT



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ABSTRACT

Dense and porous nanostructured carbon particles were successfully synthesized via spray pyrolysis of phenolic resin and polystyrene latex. An approach to estimate the size and size-distribution of the synthesized carbon particles is presented here. The size of dense carbon particles was found to be mainly determined by the concentration of phenolic resin in the precursor. Meanwhile, the size and morphology of porous carbon particles were found to be affected by the concentration of the polystyrene latex particles used as the template. A residual ratio was introduced as a variable to represent the shrinkage due to phenolic resin decomposition. New correlations were developed that permit estimation of the mean diameter and size-distribution of the synthesized carbon particles from the phenolic resin concentration. Good agreement was observed between predicted values and experimental results.

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1. Introduction

The aerosol process allows scalable synthesis of functionalized, ultrafine particles with particular physical and chemical properties from molecular precursors [1,2]. In this method, particles are produced by either a droplet-to-particle or a gas-to-particle conversion process [3]. Spray drying is one of the droplet-to-particle methods for a wide range of applications, e.g. pharmaceuticals, foods, ceramics or catalysts [4–6]. Here, structured particles are produced from a particle suspension or a colloidal mixture that is atomized to form ultrafine droplets whose volumes are mostly occupied by solvent. The solvent is then evaporated by heating, causing the remaining components to precipitate after the mixture reaches a super-saturated state. This whole process can be carried out in several seconds [7]. The produced particles may exhibit a range of sizes and morphologies according to the characteristics of precursor, the size of the droplets and other operating conditions. Particles with a narrow size-distribution are preferable to obtain homogeneous properties, for example for understanding flow and dispersion [6,8,9]. In light of this preference, ultrasonic atomization—rather than two-phase atomization—tends to be used

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for droplet production since it can produce tightly-controlled, micrometer-sized droplets [10].

Ultrafine particles with a narrow size-distribution and controllable morphology can also be synthesized by spray pyrolysis. Although the basic principle is similar to that for spray drying, the presence of chemical decomposition at elevated temperatures differentiates spray pyrolysis [11], as shown in Fig. 1. The precursor of the spray pyrolysis process is usually metal salts or simple organic materials [12]. For example, phenolic resin was successfully used as the starting material for carbon particle synthesis by pyrolysis [13–16], and also has potential for scaled-up carbon particle production as it is a relatively low-cost raw material.

In the scaled-up production of nanostructured carbon particles, the particle surface area is a key quality objective. For example, for particles used as catalysts a higher surface area is desired because it is one of the factors that determine catalytic activity. Because particle size and morphological structure are among the determining factors of particle surface area, a number of works have studied these factors. Morphological control of carbon particles, including control of the pore size and structure, has been carried out using a number of techniques, such as employing a template and using different derivations of phenolic resin [17,18]. Controlling the particle size requires an adequate understanding of the factors that contribute to the particle size and the correlation between them. Through such correlation, the concentration of phenolic resin that is required to attain certain particle size can be determined.

Correlation between the final particle size and the starting material in the case of spray drying has been studied with the particle size found to be predicted by the following equation [19,20]:

$$D_{p,v} = D_{d,v}\left(\sum_{i=1}^{n}\frac{C_i M_i}{\rho}\right)^{1/3} \qquad (1)$$

$D_{p,v}$, $D_{d,v}$, C, M, ρ denote the average particle diameter, the average droplet diameter, the droplet concentration, the molecular weight of droplet and the particle mass density, respectively. The subscript i corresponds to the component sequence. Although some disagreements were reported at certain levels of starting material concentration [19], most of experimental data for both dense and porous particles showed good agreement with the correlation in terms of particle size and particle size distribution [7,20].

Although Eq. (1) demonstrates the correlation for spray drying, to the best of our knowledge a correlation between particle size and precursor properties specifically applicable to nanostructured carbon particle synthesis by spray pyrolysis has not yet been developed. Indeed, Eq. (1) may not be applicable for carbon particles derived from phenolic resin using spray pyrolysis because the equation only considers water evaporation during particle generation. However, in the spray pyrolysis of phenolic resin, several gases are also released through polymeric reactions which have important implications for predicting particle size [21]. Notably, the loss of these gases will decrease the mass of the synthesized particles, causing the particle to shrink after carbonization. Understanding the decomposition stoichiometry is one approach to evaluate the particle size but this is difficult for the phenolic resin decomposition where the stoichiometry is difficult to determine. Therefore, another approach is needed to evaluate the carbon particle size.

This paper aims to define an approach to establish new correlations to estimate the size and size-distribution of dense and porous carbon particles formed by spray pyrolysis. The correlations include a parameter to represent phenolic resin decomposition in spray pyrolysis and pores formation due to the addition of a template molecule.

2. Materials and methods

2.1. Carbon particle synthesis

Two types of carbon particles—dense and porous—were prepared. Dense carbon particles were prepared from an aqueous solution containing resole-type phenolic resin (Sumitomo Bakelite Co., Ltd., Tokyo, Japan) as the precursor. Five concentrations of the phenolic resin were used; 0.06, 0.125, 0.25, 0.5 and 1 wt%. A mixture of phenolic resin and negatively-charged polystyrene latex (PSL; particle size of ~230 nm) was prepared for porous carbon particle production. The phenolic resin concentration was maintained at 0.25 wt% while the mass ratio of PSL to phenolic resin was studied at 0.8, 1.6 and 3.2.

Spray pyrolysis experiments were carried out using the setup shown in Fig. 2. The apparatus consists of an ultrasonic nebulizer (1.7 MHz, NE-U17, Omron Healthcare Co., Ltd., Kyoto, Japan) for droplet production, a tubular furnace (length: 1100 mm; inner diameter: 13 mm) and a filter where sample was collected. The furnace was divided into low- and high-temperature zones, with each zone including two sections. The upper and lower sections in the low-temperature zone were maintained at a temperature of 350 and 150 °C, respectively. The temperature in the high-temperature zone was determined following the preliminary experiments detailed below. Nitrogen (N_2), flowing at 0.8 L min^{-1}, was used as the inert carrier gas.

2.1.1. Preliminary experiments

Preliminary experiments were carried out to determine the optimum temperature configuration for the high-temperature zone. According to thermogravimetric analysis of phenolic resin

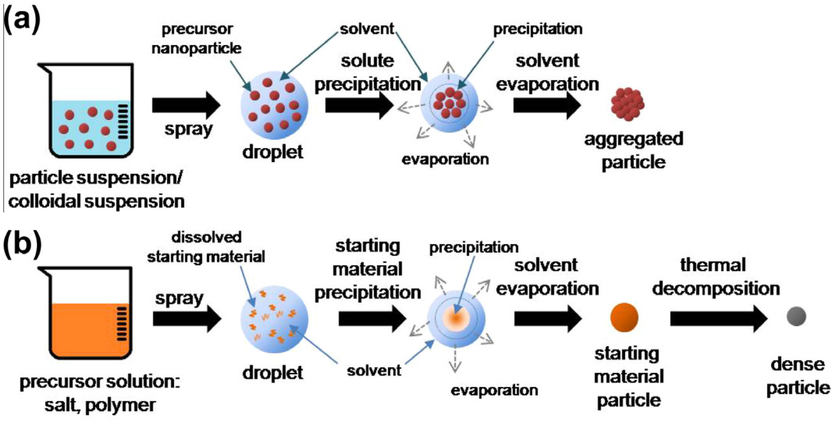

Fig. 1. Illustration of spray drying (a) and spray pyrolysis (b).

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