

Synthesis and characterization of porous carbon spheres with controlled size

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Abstract

The goal in this study is to use a facile surfactant assisted polymerization approach to synthesize microporous carbon spheres with different sizes. Carbon micro spheres have been attractive because of their importance in a wide range of applications including adsorption, membrane separation, drug delivery and catalysis [1, 2]. Irreversible aggregation and lack of control of spheres size and porosity are the major issues in conventional synthesis methods like hydrothermal treatment of sugar or glucose. In a recent study, Yao and his coworkers have used triblock copolymers, Pluronics, as the structure directing agents and formed poly (furfuryl alcohol) (PFA) spheres using a two step polymerization process [3]. However no study had been carried out on the effect of surfactant and monomer concentration on the size of the spheres or morphology of the latex particles in such a system.

In this study, the size of the carbon spheres was successfully controlled by changing different parameters contributing in synthesis: surfactant, furfuryl alcohol (FA) and HCl (polymerization catalyst) concentration in the initial mixture. Spherical PFA particles were formed because of polymerization of FA inside micelles. Spherical particles were then solidified using strong sulfuric acid and washed and centrifuged using distilled water to separate all the remained monomer which may lead to agglomeration in drying and pyrolysis steps. These PFA spheres were then carbonized under argon and high temperature to transform them to carbon spheres. To examine size and morphology, resultant carbon particles have been characterized using Field Emission Scanning Electron Microscopy (FE-SEM), Transmission Electron Microscopy (TEM). Nitrogen physisorption was used to determine pore size distribution and porosity of the samples. Characterization results showed that they are highly mono-dispersed spheres with smooth surfaces and completely microporous. Surface area of as synthesized spheres was measured to be $450 \text{ m}^2/\text{g}$ which can be increased to $1500 \text{ m}^2/\text{g}$ by activation under CO_2 . SEM image of a typical carbon sphere sample synthesized with this method with the average size of 890 nm is shown in Figure 1.

The novelty of our work is that we can control the size of the spheres and make monodispersed carbon spheres with the average size ranging from 1 μm or higher to 50 nm. The result of one set of experiments conducted to determine the effect of surfactant concentration on the size of the spheres, is shown in Figure 2. The whole region in the pseudo-ternary phase diagram of solvent/surfactant/monomer system was studied and the interesting regions which lead to regular polymeric/carbonaceous structures were determined.

References

- [1] H. Wang, B.A. Holmberg and Y. Yan, Journal of Material Chemistry, **12** (2002) 3640-3643.
- [2] X. Sun, Y. Li, Angewandte Chemie-Internatioanl Edition, **43** (2004) 597-601.
- [3] J. Yao, H. Wang, et. al., Carbon, **43** (2005) 1709-1715.

Figures

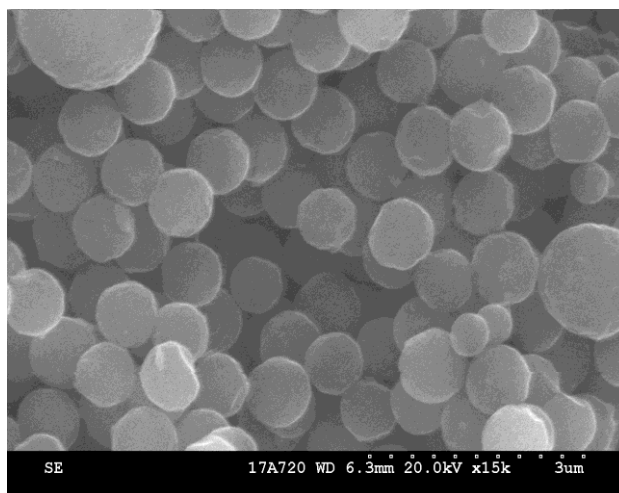


Figure 1- SEM of carbon spheres made at the surfactant to monomer ratio of 1, average size: 890 nm

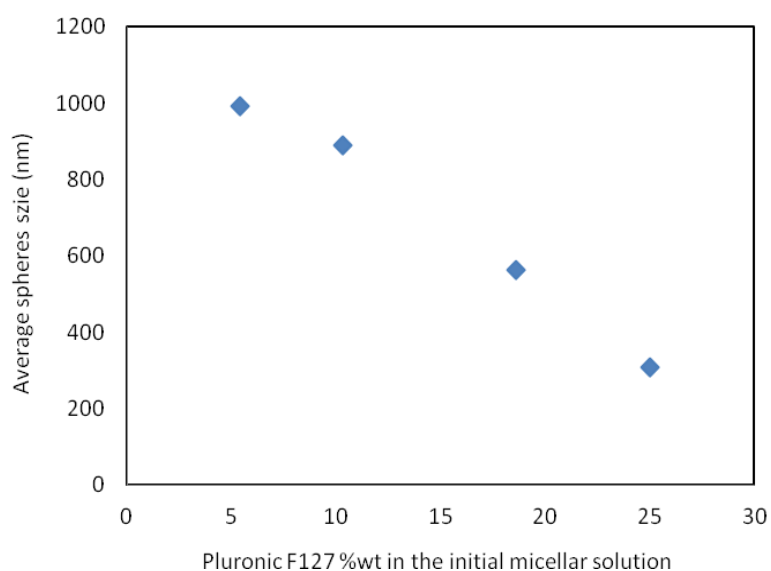


Figure 2- Effect of surfactant concentration in the micellar solution on the resultant spheres' size