

Preparation of Fullerene/Polystyrene Nanoparticles by Emulsion Polymerizations

Kun-Ji Kim, Sooyeon Park, and Myong-Hoon Lee

Dept. of Polymer.Nano Science and Technology, CHONBUK NATIONAL UNIVERSITY,
Jeonju, Jeonbuk, 561-756, Korea

Tel.:82-63-270-2337, E-mail: mhlee2@chonbuk.ac.kr

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Abstract

Fullerene/polystyrene nanoparticles having the average size of 300 nm ~ 1 μ m were prepared by emulsion polymerization in aqueous medium. Poly(vinyl pyrrolidone) and potassium persulfate were used as a dispersant and an initiator, respectively. The contents of fullerene in the nanoparticle were controlled to be from 10 to 57 wt% by varying the feed ratio, which was confirmed by IR-spectroscopy, thermogravimetric analyses, and elemental analyses. Dynamic light scattering experiments revealed the particles have a broad size distribution. Further characterizations of the nanoparticles were performed by using SEM and TEM observation. The high content of fullerene in the particles will find applications in photovoltaic and organic semiconducting area.

1. Introduction

Fullerene (C_{60}) is a widely studied material due to its unique optical, electrical, and chemical properties. However, one of the issues in the practical application of fullerene comes from its extremely low solubility in organic solvents. Besides, C_{60} tends to form aggregated clusters which hinder the homogeneous dispersion in the molecular level. Encapsulated particles consisting of an organic or inorganic functional material and a polymer can make advanced applications with better control of optical properties, charge carrier mobility, surface chemistry, stability, and etc. Since the functional materials are encapsulated by a polymer with controlled particle size in nanometer scale, enhanced physical properties are to be expected. The dispersion stability of composite particles themselves is determined by the contribution of three main factors: van der Waals attractive force, electrostatic forces, and steric repulsive force between particles. In this study, polystyrene (PS) and C_{60} composite nanoparticles were prepared by emulsion polymerization of styrene in the presence of C_{60} in aqueous medium.

2. Experimental

C_{60} /PS particles were prepared by using potassium persulfate (KPS) as an initiator and poly(vinyl pyrrolidone) (PVP) as a dispersing agent in aqueous medium. Firstly, a mixture of styrene monomer and 10 wt% (to styrene) of divinylbenzene (DVB) as a crosslinker in aqueous solution of PVP was treated ultrasonically for 1 h to obtain a fine emulsion. Another emulsion was prepared by ultrasonifying C_{60} dispersed in an aqueous PVP solution for 1 h. Then, the two solutions were mixed, and ultrasonified for 1 h to obtain a homogeneously dispersed emulsion. The mixture was stirred at 300 rpm for 15 min, the temperature was increased to 70 $^{\circ}$ C, and aqueous KPS solution was slowly added. The mixture was stirred for 24 h while keeping the temperature variation in less than ± 5 $^{\circ}$ C. The resulting polymer particles were washed with deionized water repeatedly.

3. Results and discussion

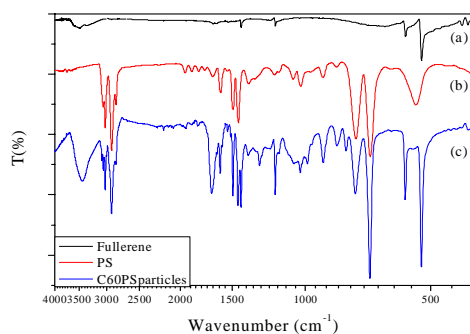
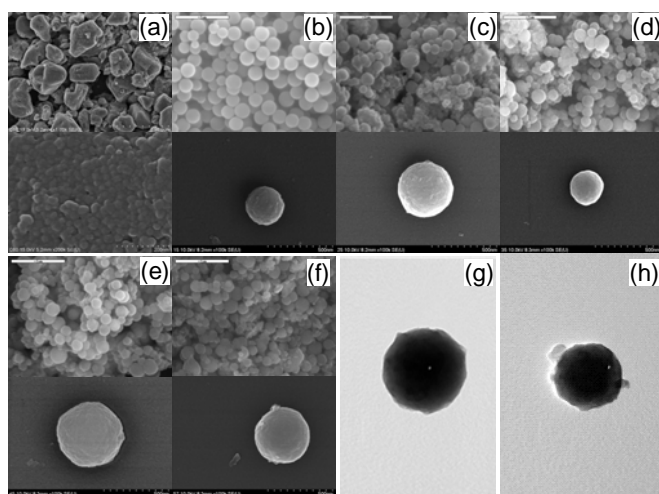
The resulting particles were analyzed by using Fourier transform infrared spectroscopy (FT-IR), thermogravimetric analyses (TGA), elemental analyses (EA), scanning electron microscopy (SEM), tunneling electron microscopy (TEM), and dynamic light scattering (DLS).

The FT-IR of C_{60} /PS particles showed characteristic vibrational bands of both PS and C_{60} implying that the prepared particles are consisted of PS and C_{60} (Figure. 1). The C_{60} contents in the C_{60} /PS particles were determined by measuring the char yields at 600 $^{\circ}$ C from the TGA and estimations from the EA results, of which the results are reasonably coincided with each other. C_{60} content in the particles increased with initial C_{60} feed ratio, ranging from 10 to 56 wt%. The results are summarized in Table 1.

Table 1. Fullerene content (wt%) in C₆₀/PS particles estimated by TGA and elemental analysis.

Sample Name	FP-1	FP-2	FP-3	FP-4	FP-5
Fullerene in feed (wt%)	8.47	17.24	26.31	35.71	45.45
TGA (wt%)	15	24	32	45	57
EA (wt%)	10.39	17.66	29.73	42.48	56.11

By using DLS, average particle size was measured to be 300 nm ~ 1 μm with broad particle-size distribution in most cases. Slight decrease of particle size with increasing amount of C₆₀ was observed. The surface morphology was observed by using SEM and TEM. As shown in Figure 2(a-f), homogeneous particles were obtained for lower C₆₀ contents, while particle size distribution became wider for high C₆₀ contents. TEM revealed that the particle is composed of small aggregates (~30 nm) with irregular spherical shape as shown in Figure 2(g-h).

**Fig 1. FT-IR spectra of (a) untreated C₆₀(KBr pellet), (b) Polystyrene, and (c) C₆₀/PS particles****Fig 2. SEM photographs of (a) pristine fullerene, (b) FP-1, (c) FP-2, (d) FP-3, (e) FP-4, (f) FP-5, and TEM photographs of (g) FP-4, and (h) FP-5.**

4. Summary

We report the first preparation of C₆₀/PS composite particles with high C₆₀ contents. C₆₀/PS composite particles containing more than 15 wt% of C₆₀ have never been reported to our knowledge. We expect the C₆₀/PS nanoparticles with high fullerene contents can find many application areas such as dielectrophoretic displays, photovoltaic cells and organic microelectronics.

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5. References

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