Solvent Induced Large Compound Vesicle of [60]Fullerene Containing Poly(tert-butyl methacrylate)

Chung How Tan¹, Palaniswamy Ravi¹,², Sheng Dai¹,², Kam Chiu Tam¹,² *, Leong Huat Gan³

¹School of Mechanical and Production Engineering, ²Singapore-MIT Alliance,
³Natural Sciences, National Institute of Education, Nanyang Technological University, Nanyang Avenue, Singapore 639798, Republic of Singapore.

Experimental Section

1. Synthesis details:

The C₆₀ has been successfully grafted with poly(methyl methacrylate) and polystyrene using ATRP previously.¹⁶ In this study, the –Cl terminated PtBMA with narrow molecular weight distribution was first synthesized as reported from our earlier publication.¹⁵ Using the PtBMA-Cl as macroinitiator, the C₆₀ was grafted in presence of CuCl/HMTETA catalyst system with 1:2 molar ratio of PtBMA-Cl: C₆₀ in 1,2-dichloro benzene at 90°C. After the polymerization, the catalyst was removed by passing through alumina. The un-reacted C₆₀ was completely removed by dissolving the polymer in THF and filtered and passed through the alumina column. The filtrate was concentrated and precipitated in excess of water/methanol (1:1) mixture. The procedure was repeated three times to ensure the complete removal of un-reacted C₆₀. It was found that the average molecular weight (Mₙ) of FBMA is 14000. In addition, the molecular weight distribution as obtained from GPC is 1.22, indicating only the
absence of multi-arms grafted with one C₆₀ molecule. This was further confirmed by TGA through determining the weight fractions of the PtBMA and C₆₀.

2. Equipment

Dynamic light scattering (DLS) studies were conducted by a Brookhaven BI-200SM goniometer equipped with a 9000AT digital correlator. The light source is a power-adjustable argon ion laser with the wavelength of 488nm. The inverse Laplace transformation routine of REPES supplied with the GENDIST software was used to analyse the time correlation functions with the probability of reject setting to 0.5. The viscosities and refractive indexes of different solvent mixtures were measured by an LS 40 viscometer and an Atago 3T refractometer. Static light scattering (SLS) was conducted using the same instrument with scattering angles ranging from 20 to 100°. The refractive index increment of dn/dc for the PtBMA₉₈–g-C₆₀ solutions, measured by a BI-DNDC differential refractometer, varied from 0.09523 to 0.09248 in 0.7 to 0.96 ethyl acetate composition at 25°C. Rᵧ values were obtained from the Zimm plot. Transmission electron microscopy (TEM) studies were conducted using a JEOL JEM-2010 transmission electron microscope operating at 200kV. Copper grid precoated with carbon film was placed on a filter paper. A drop of polymer solution was placed onto the copper grid and allowed to dry naturally overnight.
Figure S1: Static light scattering of C\textsubscript{60}-b-PrBMA in the mixed solvents

Figure S2: Relaxation time distribution of 0.7 wt\% C\textsubscript{60}-b-PrBMA in different solvent mixtures
Figure S3: TEM Micrographs of LCVs for C60-b-PtBMA in 75 and 84 mole% ethyl acetate

Figure S4: TEM Micrographs of LCVs for C60-b-PtBMA in 88 and 92 mole% ethyl acetate