

Improvement of sphericity of thick-walled polystyrene shell



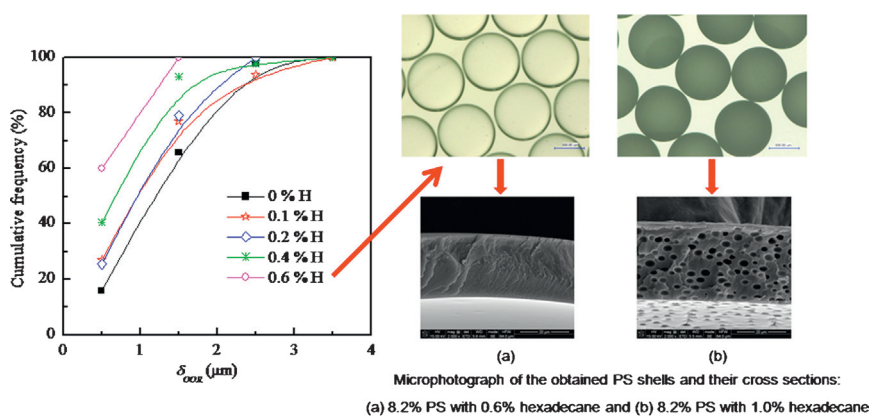
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HIGHLIGHTS

- The sphericity and the wall thickness uniformity of PS shells are improved by introduction of a little hexadecane into the oil phase.
- The W1/O and the O/W2 interfacial tensions increase with the hexadecane mass fraction increasing in the oil phase.
- Mechanisms of hexadecane affecting the sphericity and the wall thickness uniformity are proposed.

GRAPHICAL ABSTRACT



Effects of the hexadecane concentrations on the quality of PS shells

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ABSTRACT

Thick-walled polymer shells with high sphericity are always needed in the inertial confined fusion (ICF) experiments. The introduction of hexadecane (H) into the oil phase (O) was used to explore possible effects in improving the sphericity of polystyrene (PS) shells. The experimental results showed that not only the interfacial tension increased, but also the density matching levels between the inner water phase (W1) and the oil phase, and that between the W1/O compound droplets and the outer water phase (W2) were improved, probably due to the hydrophobic characteristic and low density of H. Therefore, the batch yield of PS shells with the out-of-round (δ_{OOR}) values less than 1 μm increased from 16% to 60% with increasing H mass fraction in the O phase from 0 to 0.6%. For wall uniformity, the yield of the PS shells with Δt_w less than 3 μm also increased from 45% to 67%, but there was no further increase when H mass fraction in the O phase was more than 0.2%. However, when the H mass fraction was 1.0%, the PS shells became opaque, since there were many voids in the shells resulting from very low solubility of H in water. Therefore, the H concentrations in the O phase should be controlled according to the requirements of the structure. Obviously, the introduction of H into the O phase provides a novel and effective method for preparing PS shells with high sphericity.

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1. Introduction

With increasing requirements of energy in the world, the inertial confined fusion (ICF) experiments, as one of the most promising methods to control the reaction of nuclear fusion, have attracted a great deal of interest [1]. Thick-walled polymer shells such as polystyrene (PS) and deuterated polystyrene (DPS) are always needed for preparing the target of the ICF experiments [2,3]. Since the shape of these polymer shells such as the sphericity and wall thickness uniformity is important to the symmetry and hydrodynamic stability in the implosion, there are stringent specifications on the sphericity and wall thickness uniformity for shells used for the ICF targets [4,5]. Taking a shell with 1000 μm diameter and 25 μm wall thickness for example, the out-of-round (δ_{OOR}) and wall thickness variation (Δt_w) should be less than 1 μm .

However, as shown in Fig. 1, an initial compound droplet generated by a triple orifice droplet generator is not a perfect spherical drop and the inner phase is not in the center of the middle phase. Due to gravitational force, drag force and so on, the non-spherical and eccentric phenomenon probably becomes worse during the solidifying process. With increasing wall thickness, both the deformation of the O phase and the movable space of the inner phase also increases, so it is more difficult to make a thick-walled compound droplet be spherical and concentric. Over the past two decades, to obtain polymer shells with high sphericity and uniform wall thickness, a lot of research work has been done. The theoretical analysis has showed that there are many factors, such as interfacial tension, density and viscosity, influencing the deformation of droplets [6–9]. However, how to precisely optimize these factors to improve the sphericity and wall thickness uniformity in experiments has been being a challenge, especially for thick-walled polymer shells [10].

Earlier in 1998, taking advantage of a simple model and theoretical calculation, Cook et al. pointed that either increasing interfacial tension or reducing density mismatching level would make the drop be spherical [6]. Then, Takagi et al. reported that the sphericity of poly(α -methylstyrene) (P α MS) shells were greatly improved by using poly(acrylic acid) (PAA) instead of poly(vinyl alcohol) (PVA) in the external water phase (W2), which substantially increased the interfacial tension between the oil phase and external water phase (O/W2) [11,12]. However, the wall thickness uniformity of P α MS shells was not significantly improved [13]. Lattaud et al. found that foam shells with low non-concentricity were obtained when the density of the W1 phase was slightly higher than that of the O phase [14]. Our previous work also confirmed that appropriate density matching benefited improving both the wall thickness uniformity and sphericity of the PS shells [15]. Therefore, it is promising to obtain PS shells with high sphericity and uniform wall thickness by increasing both interfacial tension and density matching level.

Hexadecane (H), as the third component, is always added in some emulsions to improve its stability [16,17]. It has also reported that the addition of hexadecane into tetralin not only increased the interfacial tension between the mixed oils and water, but also increased the stability of the emulsion [17]. Moreover, the density of hexadecane is less than that of fluorobenzene (FB). Therefore, with introduction of H into PS/FB solution, which is used as the O phase in the preparation of PS shells, it would probably not only improve the interfacial tension, but also reduce the density of the solution, benefiting the density matching level, which can improve the sphericity and the wall thickness uniformity. However, as far as we know, there is no report about the effects of H on the sphericity of polymer shells. With this in mind, in this study, a small amount of H was added in the O phase and we explore whether the proposed effects of hexadecane improves the

sphericity and the wall thickness uniformity of the PS shells or not.

2. Experimental

2.1. Materials

PS ($\bar{M}_w = 250 \text{ kg mol}^{-1}$, $\rho_s = 1.05 \text{ g cm}^{-3}$, Acros Organics Inc.), PVA ($\bar{M}_w = 13 \text{ kg mol}^{-1} \sim 23 \text{ kg mol}^{-1}$, 87–89% mole hydrolyzed, Aldrich Company), H ($\bar{M}_w = 226 \text{ g mol}^{-1}$, Tianjing Kermel Reagent Ltd.) and anhydrous calcium chloride (CaCl_2) (Chengdu Kelong Chemical Reagent Factory) were all used as received without further purification. FB (Shanghai Jingchun Reagent Ltd.) was purified by distillation. Distilled, deionized water was used in the preparation of all aqueous phases.

2.2. Preparation of PS shells

PS shells were prepared by the microencapsulation technique. Details of the fabrication process were in the Ref. [15]. Distilled water was used as W1 phase. The PS/FB solutions with 8.2% PS mass fractions and different hexadecane mass fractions (0.1%, 0.2%, 0.4%, 0.6% and 1.0%) were used as the O phases in this work. Aqueous solution with 2.0% PVA mass fractions and 0.5% CaCl_2 mass fractions was used as the W2 phases.

To limit the possible effects of the diameter and wall thickness on the density matching, the corresponding inner diameter (ID) and outer diameter (OD) of the W1/O compound droplets were controlled by changing the W1, O, and W2 flow rates. The experimental results showed that the ID and wall thickness of resulting shells are in the range of $900 \mu\text{m} \pm 30 \mu\text{m}$ and $30 \mu\text{m} \pm 3 \mu\text{m}$, respectively.

2.3. Characterization

2.3.1. Density

The densities of the W1, O and W2 phases were measured precisely by a densitometer (Anton Paar®, DMA 5000) from 5 °C to 70 °C at intervals of 5 °C. The densitometer accuracy is $10^{-6} \text{ g cm}^{-3}$ for density measurement.

Due to the higher viscosity and lower fluidity of PS/FB solution with high mass fractions of more than 30%, it is difficult to measure its density by the densitometer. Therefore, to obtain the density of PS/FB solution with high mass fractions, an exponential fit was used to correlate the mass fraction of the PS/FB solution with its density. The coefficient of determination with the density of the solution at 55 °C is more than 0.999, while its *P*-value is less than 0.001, which indicates the fit correlates well. Then, the density of PS/FB solution with high mass fractions can be obtained.

Based on the dimension of the W1/O compound droplets, the density of the O phase ($\rho_o(C_{\text{PS}})$) and that of the W1 phase (ρ_{W1}), the average density of the W1/O compound droplets ($\rho_{\text{W1/O}}$) during the solidifying process can be calculated by [18,19]

$$\rho_{\text{W1/O}}(C_{\text{PS}}) = \frac{m_{\text{W1}} + m_o(C_{\text{PS}})}{V_{\text{W1}} + V_o(C_{\text{PS}})} \quad (1)$$

where *m* is the mass and *V* is the volume. Therefore, $m_o(C_{\text{PS}})$ and $V_o(C_{\text{PS}})$ are the mass and volume of the oil phase when the mass fraction of PS in the oil phase is C_{PS} , respectively. Assuming that the mass of the W1 drop in the W1/O droplet, and the mass of PS in the oil phase do not change during the solidifying process, V_{W1} and m_{W1} are calculated by

$$V_{\text{W1}} = \frac{4}{3} \pi r^3 \quad (2)$$

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