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A novel strategy for the synthesis of polyamide-6 microspheres

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ABSTRACT

In this study, we develop a novel method to prepare polyamide-6 (PA6) microspheres with designed morphology. The diameter range of the PA6 microspheres is controlled at 200 nm ~ 500 nm by adding a certain amount of sodium dodecyl sulfate (SDS) surfactant in the polyamide-6/polyethylene glycol (PA6/PEG) system. The key strategy for the method is to induce phase inversion by PEG in the synthesis process. PEG and SDS are removed with water and without any toxicity to environment. Scanning electron microscope (SEM) and transmission electron microscope (TEM) have confirmed that the PA6 spherical morphologies. The crystallization behaviour of PA6 microspheres is investigated using X-ray diffraction (XRD) and Differential scanning calorimeter (DSC). We believe this strategy can be applied to prepare a variety of microspheres materials.

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1. Introduction

Polymer microspheres are a novel functional material with excellent properties, because of its special structure, they have the characteristics of large specific surface area, strong adsorption, large agglutination and strong surface reaction ability and so on. Especially after introducing various functional groups on polymer microspheres, the functional microspheres show good applications in many high-tech fields, such as solid-phase diagnostics [1,2], biosensors [3–5], biocatalysts [6,7], bioseparation [8–10] et al. Polyamide-6 (PA6) microspheres with functional groups (such as amine and carboxyl group), which are easy to be modified, so are of particular interest for their direct and potential applications in fields of bioengineering, selective separation and industrial materials which enable it a tempting carrier. However, the reported preparation methods are either limited to quite complex techniques [11], either to huge amounts of organic solvent [12–14], thus are difficult to make big progress. New methods for materials fabrication are of great significance in our technology societies [15–21]. To date, phase inversion technology is a relatively new approach for the synthesis of PA6 microspheres. For example, Pei et al. [22] synthesized PA6 microspheres with a diameter ranging from 7 to 80 μm via adjusting the polystyrene (PS) content of the PA6/PS alloy by phase inversion technology. But this process requires abundant tetrahydrofuran (THF) solvent to remove the PS phase, the pollution to environment would also exercise a great influence for the potential applications. Furthermore, the size of PA6 microspheres obtained is in micron scale. Size provides important control over many of the physical and chemical properties of polymer materials [23]. Therefore, the development of a new method for preparation of nanoscale PA6 microspheres without the use of organic solvent should be great interests to materials scientists.

Herein we report a novel method for the preparation of PA6 microspheres with tunable nanoscale diameters according to the method presented in our patent [24].The diameter range of the PA6 microspheres was controlled at 200 nm to 500 nm by adding a certain amount of sodium dodecyl sulfate (SDS) surfactant in the PA6/PEG system. In this work, PA6 microspheres were prepared via an *in situ* anionic ring-opening polymerization of ε-caprolactam (CL). This strategy is based on PEG induced self-assembly and phase inversion mechanism. Interesting, the phase inversion occurs at an extremely low PEG content (20 wt%), and traditional melting mixing method for the polymer pairs alloy is impossible to do it. Another characteristic is that no organic solvent is used in the synthesis process. We believe this strategy can be applied to prepare a variety of microspheres materials.

This strategy is sketched in Fig. 1 and detail synthesis materials and experimental steps in this approach are provided in the Electronic Supplementary Information (ESI).

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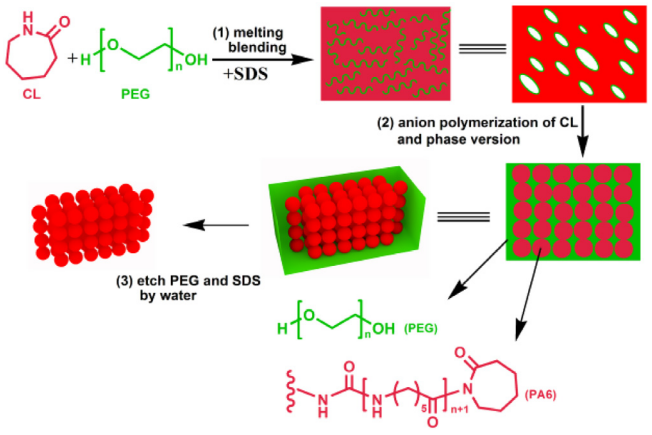

Fig. 1. Schematic outline for the preparation of PA6 microspheres.

2. Results and discussion

There are three steps in this approach as shown schematic outline in Fig. 1. Firstly, the reactive monomer CL dissolves PEG and SDS into transparent homogeneous solution. Secondly, the microspheres are synthesized via *in situ* anion polymerization of CL by PEG induced phase inversion technology. Thirdly, the above microspheres are etched with water to remove PEG continuous phase and SDS surfactant. Then the PA6 nano-microspheres are obtained. In synthesis process, the role of PEG is inducing phase inversion of system. While SDS plays the role of reducing the particle size of PA6 microspheres.

The system includes two phases, that is, the pre-product is PA6/PEG polymer pair alloy. In the synthesis process, the key step is polyethylene glycol (PEG) induce the system phase inversion. When PEG content is below 15 wt%, the system is PEG dispersed/PA6 matrix. That is, the PA6 microspheres are not formed. Once PEG content is between 15 wt% and 30 wt%, phase inversion starts

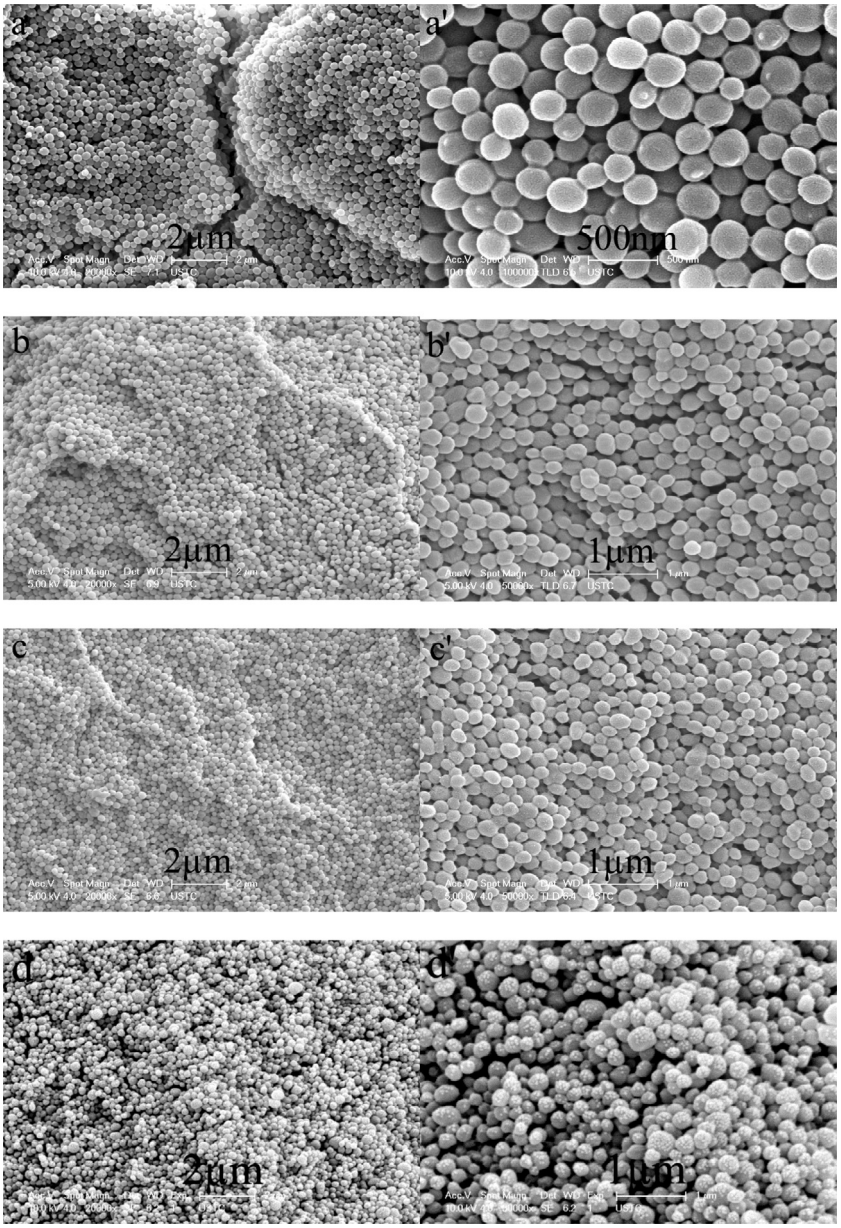

Fig. 2. SEM of PA6 nano-sized microspheres: a) S0; b) S0.5; c) S1; d) S1.5 and their correspondingly magnifying a′ ~ d′, respectively.

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