

Synthesis of Microspheres of Poly (pyrrolyl methine) by Interfacial Polymerization

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Abstract:

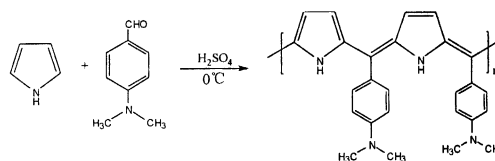
Conducting microspheres of poly (pyrrolyl methine) (PPDMABE-MS) were synthesized by the interfacial polymerization. An optimal condition was found under which spheres with 0.5–1 μm in diameter were obtained. The morphology of PPDMABE-MS was proved by SEM, and their backbone structure was characterized by FT-IR. The measurement results indicated that the molecular structures of the PPDMABE-MSs were identical to that of PPDMABE synthesized by solution method.

Keywords: Interface preparation; Conducting polymer; Microspheres

1. Introduction

The conjugated polymer's morphology is found to have strong influence on its properties and application [1]. So, much work has been done to improve its morphology. Morphologies like rice grains, needles, fibers, tubules and spheres were reported [2]. In an interfacial polymerization (IP) reaction, the poly condensation reaction takes place where both reactants meet each other, exactly at the interface. In general, it is assumed that most polymers form and grow at the organic solvent side of the interface [3]; only a few authors believe that reactions might also occur at the aqueous side [4].

We wish to report the synthesis of poly [(pyrrole-2,5-diyl)(p-dimethylaminobenzyliene)] micro spheres (PPDMABE-MS) by the static IP reactions of pyrrole with N,N-dimethylaminobenzaldehyde under the condition of ice-bath. In this reaction, the polymer microspheres were form and grow at the aqueous side.



Scheme 1 The synthetic route of PPDMABE-MS

2. Experimental

The synthetic route of PPDMABE-MS is shown in **Scheme 1**. Pyrrole (8×10^{-3} mol) and N,N-dimethylaminobenzaldehyde (8×10^{-3} mol) were dissolved in 10 mL 1,2-dichloroethane. Then 22 mL diluted sulfuric acid (2 mL concentrated sulfuric acid dissolved in 20 mL deionized water) was added slowly. The mixture was placed in an ice-bath for 12 hours, and then was filtered. The residue film was washed with water and methanol several times, and finally dried under vacuum at room temperature for 24 h.

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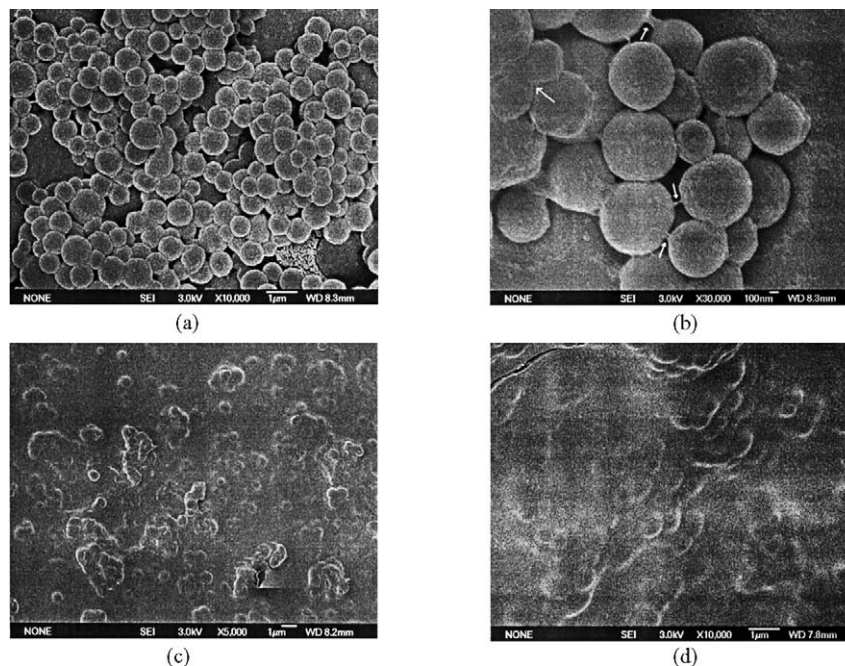


Fig.1 SEM images of PPDMAE-MS synthesized by the interfacial polymerization
(a) 12 hours (b) 12hours (c) 48hours (d) 72hours

3. Results and discussion

It must be pointed that the materials we obtained by IP reaction were polymer membranes. These polymer membranes were composed of microspheres under certain conditions. In order to find the optimal conditions for formation of PPDMAE-MS by the IP reaction, the different synthetic time were investigated. As seen in Figure 1, the synthetic time have influence on the formation of the polymer spheres. For instance, when the reaction time was 12h, the polymer microspheres were form perfectly, as shown in Fig.1a. But if the reaction time was too long, the microspheres would be incoalescent and it was difficult to get the individual polymer microsphere (Fig.1(c)(d)).

The typical SEM images of polymer microspheres are shown in Fig1(b). The spheres diameter is in the range of 0.5~1µm. It can be seen that some spheres connect with each other to form dumbbell or calabash structure, as pointed by arrows. We think this phenomenon means these spheres' walls are make up of reactive polymers, they can continue to react with each other to form bigger spheres. The mechanism of the formation of the PPDMAE-MS is under studying.

FT-IR spectrum of PPDMAE-MS shows the

appearance of a strong absorption at 1632cm^{-1} which is attributed to the stretching vibration of conjugated C=C and the stretching vibration of aromatic C=C in phenylene. A distinct peak near 780cm^{-1} is due to the out-of-plane vibration of $\text{C}_\beta\text{-H}$ characteristic of the α -linkage in pyrrole rings. The FT-IR spectrum of PPDMAE-MS is the same as that of PPDMAE synthesized by general polymerization described in the literature [5]. It means the synthetic microsphere is PPDMAE.

The conductivity of doped microsphere with iodine falls in the range of a semiconductor.

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