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Solvent induced morphology evolution from microrods to monodispersed microspheres based on 1,2-diphenyl-4-(4-methoxyphenyl)-1,3-cyclopentadiene

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ABSTRACT

The uniform microrods, microspikes, and monodispersed microspheres of 1,2-diphenyl-4-(4-methoxyphenyl)-1,3-cyclopentadiene (PMPCP) were prepared by a facile solvent evaporation method. The effect of reaction conditions on the morphology evolution of PMPCP aggregates was investigated. It is believed that the different nucleation and growth processes in different solvents should be responsible for the formation of different microstructures. The photoluminescence properties of these particles were also investigated.

1. Introduction

Organic micro/nanomaterials have drawn more and more attentions due to their unique electronic, optical, and magnetic properties as well as their practical applications in the many fields such as photonics and optoelectronic devices [1–5]. Various morphologies of organic materials, such as sphere, rod, wire, and cube, have been prepared by different methods [3,6–13]. However, the controlled morphology evolution of organic particles is still a major challenge currently. On the other hand, conjugated cyclopentadiene derivatives (CPDS) have drawn great concern for their good electrical and optical properties and they are widely used in electroluminescent devices [14–16]. The fabrication of their micro/nanostructures might be helpful in enhancing their properties because different aggregation behaviors of molecules have effect on charge transfer transitions and energy levels. To date, only one example of assembling aggregate of CPDS was reported, in which nanoribbon structure of 1,2,3,4,5-pentaphenyl-1,3-cyclopentadiene exhibits multicolor emission properties [17]. Based on our continuing research on conjugated organic molecules [18,19], our research interest is focus on aggregate structures and properties of CPDS. Herein, we report the morphology evolution of 1,2-diphenyl-4-(4-methoxyphenyl)-1,3-cyclopentadiene (PMPCP) from crystalline microrods to monodispersed microspheres by a solvent evaporation method. The crystal structure and luminescent property of PMPCP with different morphologies were characterized.

2. Experimental

2.1. Preparation

All reagents are off-the-shelf commercial products. The solvents were purified with standard methods and dried as needed. The PMPCP was synthesized according to reported procedure [19]. The solvent evaporation method was used to fabricate PMPCP particles (Fig. 1). In a typical procedure, a 50 μL aliquot of PMPCP in organic solvent was dropped on a substrate located in a glass container with a cover. After evaporation of the solvent, the samples were obtained on the substrate.

2.2. Characterization

The as-prepared samples were characterized by XRD (Rigaku-DMax 2400 with Cu Ka radiation). The morphologies of as-prepared samples were observed with scanning electron microscope (SEM, JEOL-6360LV, operated at 20 kV). Photoluminescence spectra (PL) were recorded with a JASCO FP-6300 spectrofluorimeter.

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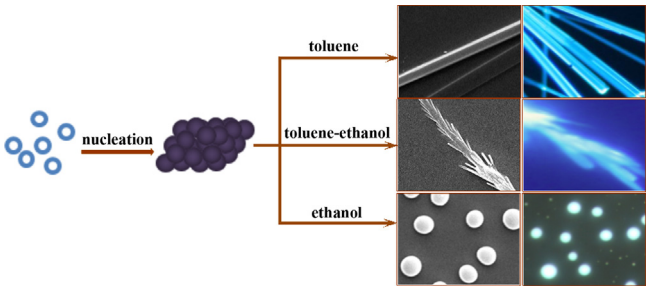

Fig. 1. Illustration of the proposed mechanism for PMPCP with different morphologies.

Microscopic images were observed on an OLYMPUS BX51 fluorescent microscope.

X-ray crystal structure determinations were performed on a Bruker SMART APEX CCD diffractometer with graphite monochromated Mo Kα radiation (λ = 0.71073 Å) using the SMART and SAINT programs. The structures were solved by direct methods and refined on F^2 by full-matrix least-squares methods with SHELXTL version 5.1. All non-hydrogen atoms were refined anisotropically. Crystallographic data has been deposited in the Cambridge Crystallographic Data Centre with reference numbers 828075. A summary of the crystallographic data and structure refinements are tabulated in Table 1.

Table 1
Crystallographic data and structure refinements for PMPCP.

Empirical formula	$C_{24}H_{20}O$
Formula weight	324.40
Crystal system	Orthorhombic
Space group	*Pbca*
a (Å)	7.3962(15)
b (Å)	20.723(4)
c (Å)	23.358(5)
Volume (Å^3)	3580.0(12)
Z	8
D_{Calc} (mg/m^{-3})	1.204
μ (mm^{-1})	0.072
F(0 0 0)	1376
R_{int}	0.1236
Data/restraints/parameters	4096/0/235
GOF on F^2	1.001
R_1 [$I > 2\sigma(I)$][a]	0.1051
wR_2 [$I > 2\sigma(I)$][a]	0.2544
R_1 (all data)[a]	0.1843
wR_2 (all data)[a]	0.3049

[a] $R_1 = \sum \frac{||F_o|-|F_c||}{\sum |F_o|}$; $wR_2 = \left\{ \frac{\sum [w(F_o^2-F_c^2)^2]}{\sum [w(F_o^2)^2]} \right\}^{1/2}$.

3. Results and discussion

3.1. SEM and fluorescence microscopy images

Fig. 2 shows the SEM and fluorescence microscopy images of the samples obtained in different solvents. For PMPCP in toluene, the

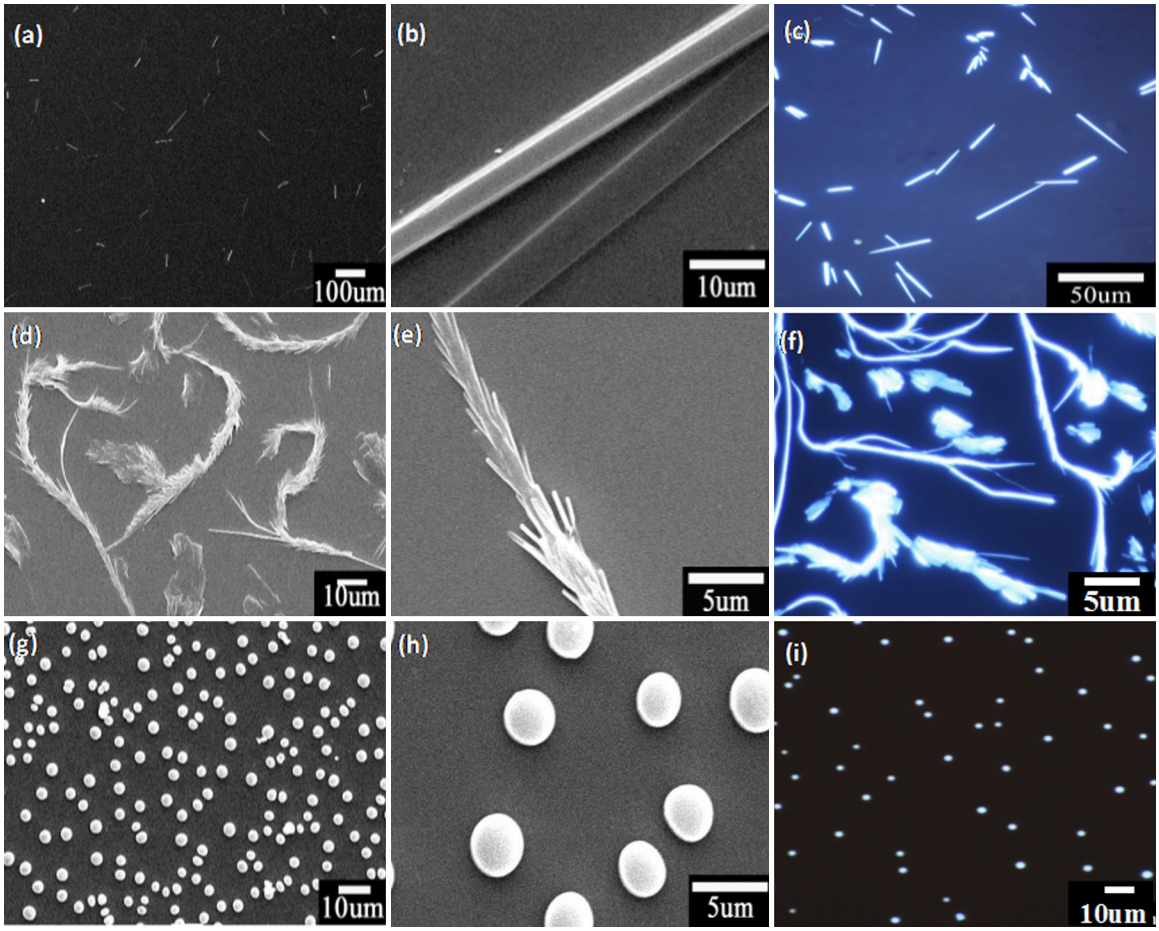

Fig. 2. The SEM and fluorescence microscopy images of PMPCP microstructures: microrods from toluene (a–c), microspikes from toluene doped with ethanol (d–f), and microspheres from ethanol (g–i).