Preparation and Properties of Poly(ε-caprolactone) Nanocapsules Containing Phytoncide Oil by Emulsion-diffusion Method(1)

유화확산법을 이용한 피톤치드오일 함유 폴리입실론카프로락톤 나노캡슐의 제조와 성질(1)

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Abstract

Poly(ε-caprolactone) nanocapsules(nanoPCL) containing phytoncide oil were synthesized by emulsion diffusion method using ethyl acetate as organic solvent. The influence of the degree of hydrolyzation of poly(vinyl acohol) used as an emulsion stabilizer, and the different weight ratio of core material to wall material on the particle size, morphology, and emulsion stability was investigated to design nanocapsules. The encapsulated nanoPCL were characterized by FT-IR spetrometry, Scanning electron microscope, Differential scanning calorimetry, and Thermogravimetry analysis, respectively.

1. Introduction

A detailed investigation into the mechanisms of nanocapsule formation by means of the two stages "emulsion-diffusion" process is reported. An emulsion of oil, polymer and ethyl acetate is fabricated as a first step; dilution with pure water allows ethyl acetate to diffuse out from the droplets, leaving a suspension of nanocapsules at the end. It has been shown that the size of nanocapsules was related to the chemical composition of the organic phase and the size of primary emulsion through a sample geometrical relationship. As a consequence, most of the properties of the nanocapsules were decided at the emulsification step.

In this study, Phytoncide oil that was antimicrobial allelochemic volatile organic compounds derived from plants was encapsulated by emulsion-diffusion method using Poly(ε-caprolactone) (PCL) as wall materials. The influence of the degree of hydrolyzation of poly(vinyl acohol) used as an emulsion stabilizer, and the different weight ratio of core material to wall material was investigated to design nanocapsules.

2. Experiment

2.1 NanoPCL Preparation

NanoPCL were prepared by emulsion-diffusion method. First, mutually saturated aqueous and organic phases were prepared. The saturated water contains 8.3% of ethyl acetate and the saturated solvent contained 3% of water. PVA was dissolved in saturated water at 50℃ for 2hr. PCL was dissolved in saturated ethyl acetate at 50℃ during 2 hr and oil was added when the solution has cooled back to room temperature. The resulting organic solution was poured into the aqueous phase and emulsified with a homogenizer.

Table 1. Description of nanoPCL composions.

NanoPCL	PVA (Hydrolyzation degree, %)	Core/wall ratio	Emulsification speed(rpm)
Nano1	99.9	3.5	10000
Nano2		2.5	
Nano3		1.75	
Nano4		1	
Nano5		0.5	
Nano6	89.8	3.5	
Nano7		2.5	
Nano8		1.75	
Nano9		1	
Nano10		0.5	
Nano11	89.8	2.5	8000
Nano12			10000
Nano13			12000

The oil-in water emulsion (O/W) formed at room temperature. The dispersed droplets were converted into nanocapsules in the second step of solvent diffusion. The addition of a large volume of water (4 times the volume of the O/W) to the emulsion under gentle stirring with a magnetic bar allowed the ethyl acetate to leave the droplets. The organic solvent and a part of the water were thereafter removed by evaporation under reduced pressure to afford a purified and concentrated suspension.

2.2 Characterization of nanoPCL

The structure of the obtained PCL nanocapsules containing phytoncide was studied by Fourier transform infrared (FT-IR) spectroscopy using a Impact 400D(Nicolet). The particle size distribution was determined by laser light scattering system 34mW He-Ne laser high speed correlator (Brookhaven Instruments Corporation). Electron

microscopy techniques were used to assess the morphology of nanocapsules. Scannig Electron Microscopy (SEM) was performed with a HITACHI S-4200 (Jeol Corporation, Hitachi).

3. Results and discussion

NanoPCLs containing Phytoncide oil were prepared by emulsion-diffusion method. An increase in a ratio of core material to wall material could give a smoother surface and smaller particle size. An increase of emulsification speed gave smaller NanoPCLs. As the degree of hydrolyzation of poly(vinyl acohol) used as an emulsion stabilizer decreased, the mean particle size of the NanoPCL became more regular, the thermal stability of the wall membrane increased, and the particle size distribution was more uniform.

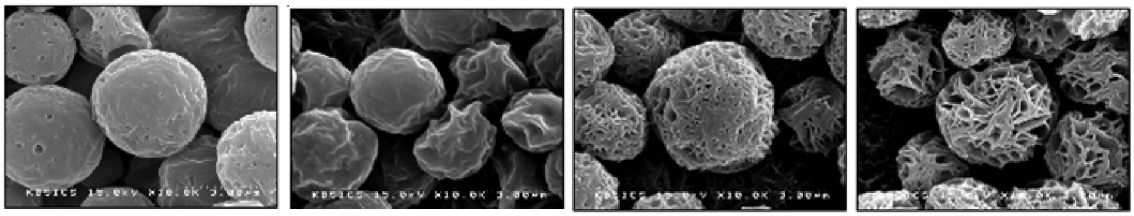

Fig. 1. SEM image of nanocapsules : (a) Nano1, (b) Nano3, (c) Nano4, (d) Nano5

Reference

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