Preparation and Characterization of Natural Fragrant Microcapsules

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Abstract: Natural fragrant microcapsules with ethyl cellulose (EC) as a shell and lavender oil as a core were prepared by emulsify-solvent diffusion method. The characters, including particle size, encapsulation efficiency and oil loading capacity, were tested and analyzed by using orthogonal design. The processing parameters of the ratio of an oil phase to water phase, the ratio of core material to shell material, concentration of PVA and stirring speed were evaluated. The morphology and structure features of microcapsules were studied by SEM, FT-IR and etc. The results showed that the microcapsules were in sphere shape and most of the particle size was about 1μm with a good formation. Encapsulation efficiency and the oil loading capacity are high with a satisfied fragrant releasing rate. This product shows a promising application on garment as well as functional textile industry.

Keywords: textile fragrant treatment, microcapsule, emulsify-solvent diffusion

1. Introduction

Many fragrant fabrics have been developed nowadays due to the reason of enjoying a healthy life style, and these novel products often possess additional functionalities which are good for human health. Recently, people pay great attention to medical effect of "forest bath", aromatic therapy, plant sterilization and fragrant plant essential oil. Not only does plant essential oil gives off a pleasant smell, but also the functions of antiseptic, antiphlogistic and emotional calming. The health care function of natural fragrant fabrics can satisfy people fashion, comfortable life style as well as health needs. Underwears incorporated with fragrant fabrics can provide a comprehensive effect of anti-bacterial, stimulate nerves system to make people calm, awakening and so on [1-4]. Indoor textiles treated by the technology, such as sheet, quilts, curtain, carpet can eliminate fatigue, improve sleep quality, also a romantic atmosphere for family can be provided[8-10]. The concept of "forest bath" was firstly put forward by Japanese scientists in the fragrant product development.

Microencapsulation has become a mainstream technology of fragrant finishing. In late 1980s, KANEBO.Ltd, Japan, developed fragrant fabrics by using microencapsulation technology, and a series products of 'incense of flower' caused an enormous effect on global market[5-7]. The SNC208 nano microcapsule was made by HERST groups, in which natural plants and plant's extracted essence were used. Microcapsule particle size is only in a range of 20 ~ 100 nm; the product had been applied on textile finishing, net printing and coating process. [11-15]

A new emulsify-solvent diffusion method is reported in this paper, ethyl cellulose (EC) is selected as a shell material. The Microencapsulation process and processing parameters, including particle size, encapsulation efficiency and the oil loading capacity are analyzed.

2. Experiment

2.1 Materials

Natural fragrance oils (lavender flavour): Dry lavender was bought from traditional Chinese medicine store, then grinded to fine particles;

Natural fragrance oils were extracted with n-hexane solvent by an ultrasonic device and distillation. The ratio of lavender to the solvent was 1:12, and extracting process lasted for 2h with temperature of 50^{o}C, the power ultrasonic device was 80W.

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Ethyl cellulose (EC), BR degree, purchased from Chinese medicine and chemicals Co. LTD;

OP-10, AR, Tianjin HongYan chemicals plant; Ethyl acetate, AR, Tianjin DengFeng chemical reagent plant;Polyvinyl alcohol PVA, NO 6 of Tianjin chemical reagent plant; Distilled water, self-preparation. Magnetic stirrer, model 85-2, Shanghai Pudong physical optics instrument plant; KQ-100E ultrasonic generator; Kunshan ultrasonic instruments Co. LTD.

2.2 Preparation of Natural Fragrant Microcapsule

Natural fragrant microcapsule preparation process is described by the following steps.

A. Ethyl cellulose and ethyl acetate with proper weight were put into a conical flask with cover, heating and stirred to dissolve them, then cooled down to the room temperature. Lavender oil was added and stirred to form an oil phase. Dissolve polyvinyl alcohol (PVA) in a saturated ethyl acetate (10%) water solution; a small amount of emulsifier was then added to make a water phase. Put the oil phase slowly to the water phase with stirring, and the oil-in-water emulsion was finished.

B. Diffusion and capsule formation:

put the oil-in-water emulsion slowly onto distilled water, magnetic stirrer was used at the room temperature; the stirring speed was controlled continuously, so ethyl acetate could constantly diffuse in water to reduce the organic solvent. Therefore, the ethyl cellulose was slowly forced to separate out from the emulsion and deposited onto the interface of the oil droplets. Microcapsule of the lavender essence oil enclosed by ethyl cellulose (EC) was prepared accordingly.

In this study, the ratio of oil phase to water phase, the ratio of core material to shell material, concentration of PVA, concentration of emulsifier and stirring rate were evaluated by a number of index, such as particle size, encapsulation efficiency (EE%) and the oil loading capacity.

2.3 Characterizations

2.3.1 Particle Size and Its Distribution

A drop of microcapsule suspension was added onto a glass slide and made it dry, the microcapsule was observed by an optical microscope; To get a mean size and size distribution, 500 individual microcapsules were selected randomly and accurate measurements were taken for statistics analysis.

2.3.2 Encapsulation Efficiency and The Oil Loading Capacity

Three microcapsule samples were weighed accurately. Then rinse them by ethanol, followed by grinding them with a mortar to completely dissolve the fragrance oils. Leave aside for a moment, then filtrated by a filter paper. The filtrate was measured for the optical absorbance at 340nm wavelength by ultraviolet spectrophotometer according to the standard curve spectrophotometry. The oil content of microcapsule can be calculated by a regression equation. The encapsulation efficiency (EE%) and the oil loading capacity can be determined by the following formula.

$$EE\% = \frac{\text{The contents of oils of microcapsule}}{\text{Total natural fragrance oils}} \times 100\%$$

$$\text{Oil loading} = \frac{\text{amount of oils in microcapsule}}{\text{Total fragrance oils}} \times 100\%$$

2.3.3 The Morphology Observation

Put the fragrance microcapsule onto a glass slide, drying at vacuum, and coated by a layer of gold. Then its appearance observation could be obtained by RMRAY - 1000B scanning electron microscope (SEM).