Structural analysis of β-cyclodextrin using four essential oil microcapsules

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Abstract. This study used the co-precipitation method to create microcapsules containing four essential oils (camellia, lemon, laurel, and litsea pepper) with β-cyclodextrin (β-CD) as the wall material. The microcapsules were then characterised using X-ray diffraction (XRD) and scanning electron microscopy (SEM). The SEM results demonstrated that the essential oil molecules were successfully filled into the β-CD lumen, as evidenced by the inclusion complex formed by the essential oil and β-CD having an irregular polyhedral cubic shape. The contact between the essential oil molecule and β-CD was confirmed by XRD analysis, which also confirmed the creation of the inclusion complex.

Keywords: β-CD；essential oil；inclusion complexes.

Abbreviations: β-CD, β-Cyclodextrins; LCEO, litsea cubeba essential oil; CAEO, Camellia sinensis essential oil; LEEO, Lemon essential oil; LAEO, Laurel essential oil; SEM scanning electron microscopy ; XRD, X-ray diffraction.

1. Introduction

There are numerous techniques to prepare microcapsules using β-CD as the wall material, including co-precipitation, spray drying, grinding, and ultra sonication. By adding the core material to a saturated solution of the wall material and applying ultrasonic waves to it, the wall material envelops the core material, which is subsequently cooled, filtered, cleaned, dried, and crushed to produce microcapsules. Nevertheless, the process requires an extended period of time and can be complex[1]. A core material solution that has been dissolved in the solvent is added and fully processed into a paste using the following grinding method: β-CD is added to water for grinding. Although minimal, labor-intensive, and time-intensive, and requiring many steps to prepare, the embedded material can be made by low-temperature drying, washing, and re-drying [2].The spray drying method is a commonly used embedding method in the food industry, which generally requires emulsification, homogenization, atomization, and other steps. However, the process also calls for skilled atomization instruments and has high operator requirements [3]. The process of co-precipitation involves mixing β-CD solution with core material, stirring, and stirring and it's heated. The required material microcapsules can be obtained by filtering and freeze-drying the precipitate formed after cooling. The core material has a high retention rate of essential oils and the process is straightforward [4].

The co-precipitation approach was employed for preparing the essential oils/β-CD microcapsules for use as feed additives, fruit and vegetable preservation, and other applications.SEM and XRD were used to describe the microcapsules. This involved theoretical information about the application of essential oils in feed, food, and other industries.

2. Experiment

2.1 Materials

Atractylodes essential oil and tea tree essential oil are available from Shanghai Yuanye Biotechnology Co., Ltd. β -CD was supplied by China National Pharmaceutical Group Chemical Reagent Co., Ltd. Moreover, Shanghai McLean Biochemical Technology Co., Ltd. provides essential oils of lemon and laurel.

2.2 Preparation of inclusion complexes

The inclusion complexes were prepared according to a previous report [5].

2.3 An analysis of inclusion complexes

2.3.1 SEM

Analyze the sample granules and their microscopic morphology with a SEM. (SU8010, Hitachi, Japan). According to the previous method [6], the sample was sprayed with a magnification of 1000 times.

2.3.2 Determining the Inclusion Complex's Crystal Structure

According to previous research[7], X-ray diffraction (XRD) was used to determine the sample's crystal structure, with a scanning area of 2 – 50° and a scanning rate of 0.5°/min.

3. Results and discussion

3.1 Examination of SEM

In Figure 1, the inclusion complexes and β -CD surface morphology are displayed.The inclusion complex's powder has an uneven polyhedral cubic shape, whereas CD has an irregularly structured block-like structure. Studies in the past that included eucalyptus or orange essential oils investigated the morphology of β -CD. They found that the inclusions' particle sizes and shapes differed from those of pure β -CD, indicating the development of complexes [8]. The inclusion complex has an uneven shape, as seen in Figure 1, but its surface does not have a fractured aggregate structure. This may be due to the hydrophilicity of β -CD and its cavity structure, which can effectively encapsulate essential oil molecules and provide a protective barrier for their attachment.

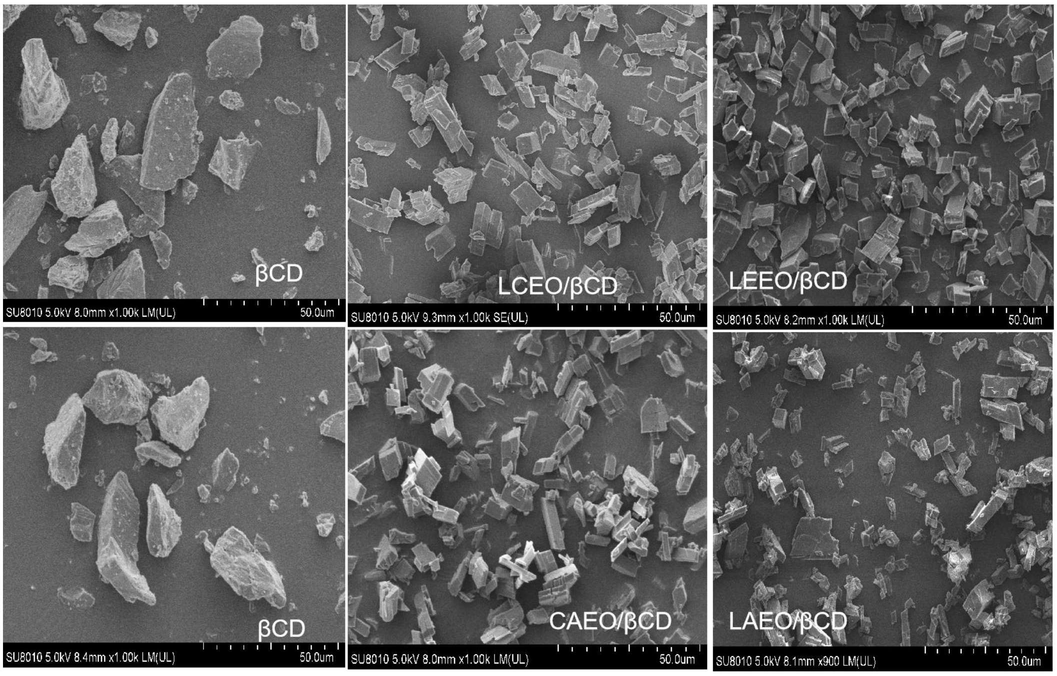

Fig. 1. Scanning electron micrographs of β-CD and inculsion complex,observed at 1000 ×

3.2 XRD analysis

The X-ray diffraction patterns of the complex formed by β-CD and essential oil are distinct, with varying crystal structures (Figure 2). Strong, sharp diffraction peaks are seen in both CD and inclusion complexes, displaying typical crystal properties [9]. 2θ =12.85° and 22.85° are the diffraction peaks that are thought to be β-CD; they show notable characteristic peaks associated with a cage-like crystal structure. The encapsulation effect in cage-shaped crystal formations is often dictated by the β-CD molecules' channel configuration. Guest molecules enter the columnar channels and stack and arrange with one another in this configuration. β-CD on 2θ = 15 to 25°, there are multiple continuous diffraction peaks that decrease when inclusion complexes form[10]. These crystal formations' distinctive diffraction peaks combine to generate a single, broader diffraction peak. A new diffraction peak emerged in 2θ = 11.85°, 12.15°, and 11.75°, respectively, in four inclusion complexes. LEEO/β-CD and CAEO/β-CD 's diffraction peaks are about equal in position and intensity, suggesting that their crystal structures may be similar. The changes in crystal properties brought about by the interactions between components can be characterized by measuring the movement or disappearance of characteristic diffraction peaks, the appearance of new diffraction peaks, and changes in the degree of crystallization or amorphization[11]. These measurements show that the reaction has produced a new solid phase, which supports the formation of inclusion complexes.

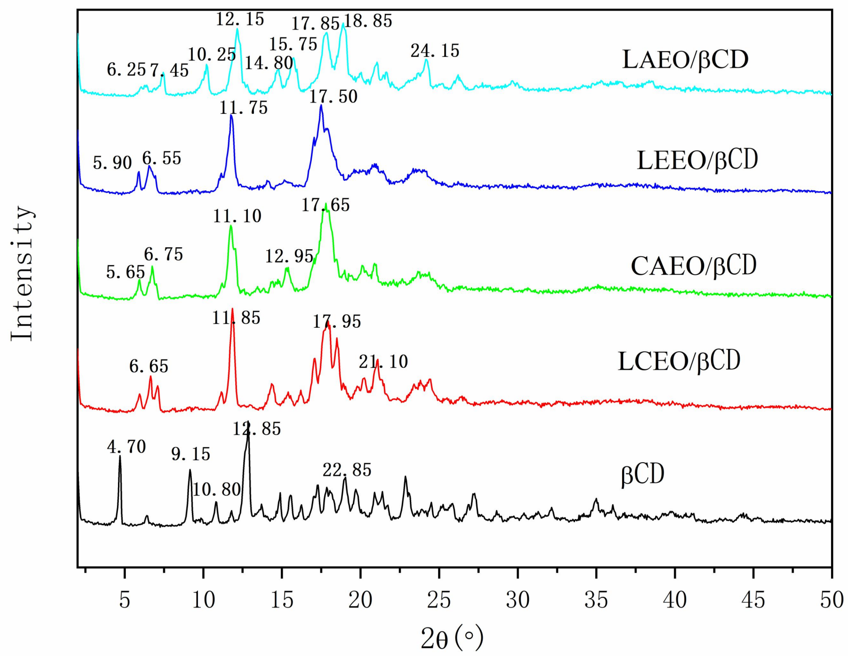

Fig. 2 . XRD analysis of β-CD, essential oils, and inclusion complexes.

4. Conclusions

X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to characterise the microcapsules made of essential oils and β -cyclodextrin (β -CD). According to SEM measurements, the microcapsules' surface had an irregular polyhedral cubic shape, suggesting that the essential oil molecules were successfully inserted into the β -CD lumen.

To elucidate the essential oil's embedding efficiency, XRD analysis compared the crystal structure before and after the inclusion, validated the interaction between the molecules of the essential oil and β -CD, and further confirmed the creation of the inclusion complex.According to these findings, β -CD can successfully increase the stability of essential oils as wall materials and offer the perfect structural foundation for essential oil release that is controlled.

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Conflict of interest statement

The authors declare no conflicts of interest.

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