Evaluation of the thermal stability of β-cyclodextrin inclusion complexes with four essential oils

Deming Li [1, a], Wenjing Tao [2, b], Chuan Cao [1, c, *]

[1] Anhui Vocational and Technical College, Hefei 230011, China

[2] Beijing Meizheng Bio-tech Co., Ltd Beijing, 102200

[a] 36369806@qq.com, [b] wenjingtao5235@126.com, [c] 877542357@qq.com

Abstract. Using Thermogravimetric Analysis (TGA-DSC), the thermal characteristics of the essential oil/β-CD inclusion complexes were assessed. The findings showed that, in comparison to the pure essential oils, the inclusion complexes had noticeably improved thermal stability. This enhancement is ascribed to the encapsulating effect of β-CD, which offers a shielding structure that stops the heat deterioration of the essential oils. The study comes to the conclusion that essential oils are better suited for applications that demand more thermal resistance because β-CD effectively increases their thermal stability.

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; TGA, Thermogravimetric Analysis ;

1. Introduction

The expression "microcapsule technology" describes the process of using synthetic or natural polymer materials for the wall and various core elements embedded in them to create a sealed or semi-sealed material [1]. It could improve the stability of essential oils' active constituents, lessen the influence of the outside world on them, and produce long-lasting antimicrobial effects and sustained release [2]. β-Cyclodextrin (CD) generates inclusion complexes with tiny essential oil molecules, enhancing their water solubility and offering protection and prolonged release due to its distinct, unique outer hydrophilic and inner hydrophobic cavity chemical structures [3]. Because the embedded components solidify into powders or particles, they make transportation easier, and because of their superior dispersibility , they can be combined with other materials or ingredients to maximize usage efficiency. Essential oils' economic worth is significantly increased by the creation of essential oil microcapsules that merely retain the aromatic components of the oils in addition to successfully regulating their delayed release of active compounds [4].

The thermal stability and thermodynamic properties of the inclusion complex between β-cyclodextrin and essential oils can be quickly assessed using TGA and DSC studies.By examining the quality change curve, ascertain the inclusion compound's volatility and thermal decomposition temperature, and confirm if the essential oil's thermal stability has been enhanced. The weightlessness phase changes allow for the differentiation of inclusion complexes from physical mixtures.By using endothermic and exothermic peaks, DSC can verify the thermodynamic properties of inclusion interactions and assess the thermal consequences of inclusion complexes.The thermal stability, volatility, and phase transition behaviour of inclusion complexes can be thoroughly assessed by combining TGA and DSC. This provides a crucial foundation for enhancing essential oil stability and inclusion condition optimisation.

2. Experiment

2.1 Materials

Essential Oils: Shanghai McLean Biochemical Technology Co., Ltd.'s lemon and laurel; Shanghai Yuanye Biotechnology Co., Ltd.'s Atractylodes macrocephala and Camellia sinensis. β-CD: From Chemical Reagent Co., Ltd., from China National Pharmaceutical Group.

2.2 Preparation of inclusion complexes

Inclusion complexes were prepared by dissolving 3 g of β-CD in 200 mL distilled water at 55℃ for 30 minutes, cooling to room temperature, and adding essential oils to achieve various mole fractions. The mixture was vortexed for 3 minutes, stirred in the dark at 30 ° C for 4 hours, and then equilibrated overnight at 4 ℃. The complexes were obtained by freeze-drying.

2.3 Thermal performance analysis

Thermodynamic properties of the sample were analyzed using the synchronous thermal analyzer TG-DSC (TA-SDT650, American). .Raise the temperature by 15℃per minute from 50℃ to 600℃ while maintaining a nitrogen flow rate of 50 mL/min.

3. Results and discussion

3.1 Thermo gravimetric analysis (TGA)

A typical thermal analysis method is TGA. Figure 1 displays the TGA for all of the materials. At 50–260 °C, the weight loss of free essential oil molecules reaches over 95%. The weight loss temperature is LAEO<LEEO<LCEO<CAEO, indicating that CAEO has the best thermal stability, while LAEO has relatively poor thermal stability. However, overall, mass loss occurs at lower temperatures, and the thermal stability of essential oils is poor. Essential oils have limited thermal stability. It is obvious from the graph that the inclusion complex mainly consists of two stages for weight loss. First, there is a weight loss of 12.3% to 20.6% at lower temperatures, which is associated with water loss during the heating process; second, there is a weight loss of 74.6% to 82.8% at 305–345 °C, which corresponds to the main weight loss stage and is associated with the complex's thermal decomposition during this stage [5]. Because of the host-guest interaction that occurs after the development of an inclusion complex, the typically volatile guest molecules within the complex become more stable. Furthermore, the slower rate of weight loss and lower overall weight loss of the inclusion complex in comparison to CAEO demonstrate that molecules have more opportunities for entanglement and aggregation at higher temperatures[6]. In the complexes of inclusion for LAEO/β-CD, the thermal stability that exhibits the highest weight loss temperature is the best; However, the lowest weight loss temperature is found at CAEO/β-CD complexes[7]. The inclusion compound's weight loss percentage is in order of magnitude. LAEO/β-CD>LCEO/β-CD>LEEO/β-CD>CAEO/β-CD, respectively. The presence of highly loaded guest molecules in these inclusion complexes increases with the percentage of weight loss [8]. This is almost in the same order as the calculated embedding rate of essential oil molecules. In conclusion, it should be noted that inclusion complexes may enhance the thermal stability of molecules in essential oils in addition to encouraging the formation of inclusion complexes.

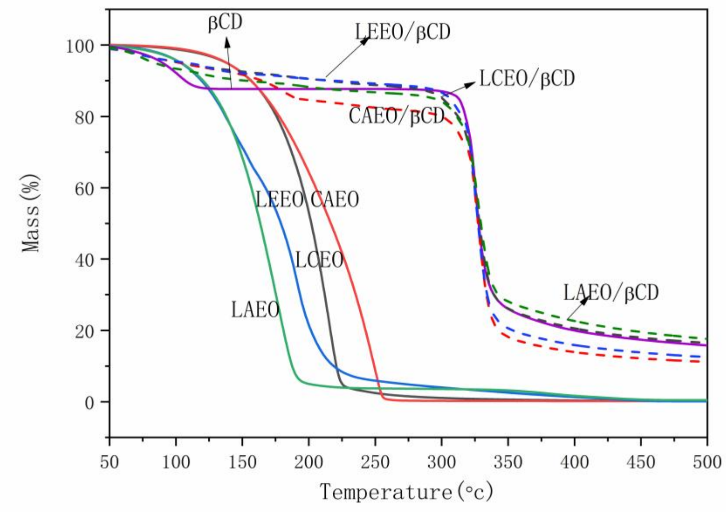

Fig.1.TGA analysis of β-CD, essential oils and inclusion complexes

3.2 Differential scanning calorimetry (DSC)

The thermal characteristics of inclusion complexes and essential oils could be shown in Figure 2. Because of the different shapes of the guest molecules in this study, LCEO, CAEO, and LAEO all showed endothermic peaks at 217.6 °C, 251.1 °C, and 178.4 °C, which are the temperatures at which the substances break down. Specifically, LEEO exhibits two endothermic peaks at 142.8 °C and 193.3 °C, which may be related to the stepwise degradation of the substance during the heating process[9]. We have observed that there are two distinct stages to the whole heating process in the DSC examination of inclusion compounds: the endothermic stage and the breakdown stage. The inclusion complex's endothermic peak at lower temperatures, which is associated with the endothermic peak of water loss in the composite, is LCEO-β-CD (64.6 °C, 101.6 °C), CAEO-β-CD (71.1 °C, 107.6 °C), LAEO-β-CD (77.7 °C, 115.7 °C), and LEEO-β-CD (68.5 °C, 122.6 °C). There was a difference between the DSC curves of inclusion complexes and guest molecules. It was discovered that when the guest molecules were put into the β-CD cavity, the endothermic peak of the essential oil molecules' thermal breakdown went away at the same time that the inclusion complex formed[10]. This suggests that β-CD and essential oil interact strongly, and compared to free essential oil molecules, the inclusion complex has better thermal stability.

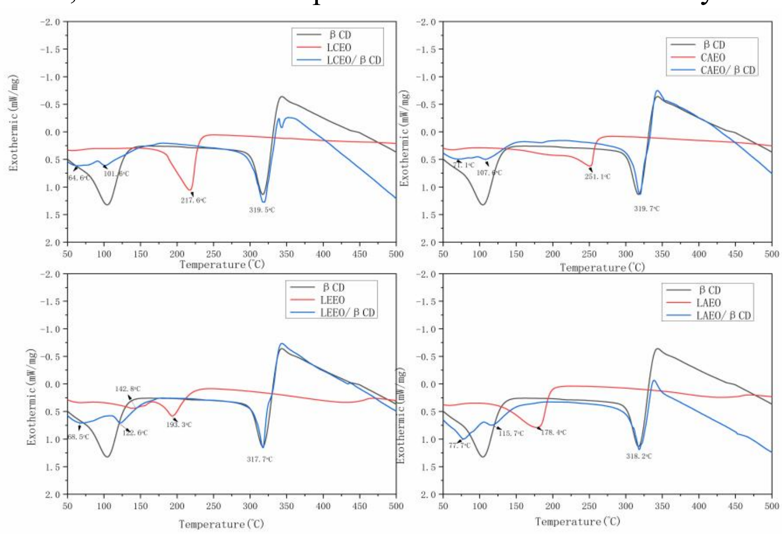

Fig.2. Differential scanning calorimetry analysis of β-CD, essential oils and inclusion complexes

4. Conclusions

This work used thermogravimetric analysis to examine the thermal characteristics of essential oil complexes with β-cyclodextrin (β-CD). In contrast to the pure essential oils, the microcapsules demonstrated superior heat stability, according to the results. This improved stability is ascribed to β-CD's encapsulating function, which shields the essential oils from heat deterioration. The study finds that essential oils are more suited for applications that need greater thermal resistance because β-CD effectively increases their thermal stability.

Acknowledgments

This study received support from the Caochuan Project, an excellent young teacher cultivation project in 2024, with project number YQZD2024082.

Conflict of interest statement

The authors declare no conflicts of interest.

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