Development of microencapsulation technology of some plant-derived bioactive compounds

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Introduction

Microcapsules are spherical particles ranging from 1-1000 μm in diameter, each containing a solid, liquid, or gaseous core encased within an individual coating produced by various microencapsulation techniques. Since the introduction of microencapsulation in the late 1930s and subsequent technological progression in the 1960s and 1970s, it has remained a field of intensive study. Over recent decades, interest in microencapsulation has significantly increased across scientific fields such as pharmaceuticals, cosmetics, and food industries due to advancements in techniques and materials. Complex coacervation is especially valued for its simplicity and cost-effectiveness among the diverse array of microencapsulation methods. This method produces microcapsules fully enclosed by a protective shell that shields the core material from environmental factors like moisture, light, and oxidation.

Objectives

The main objectives of this research were to cultivate aromatic medicinal plants under the region's specific pedoclimatic conditions, extract essential oils from the harvested biomaterial through steam distillation, and assess the essential oil quality by GC-MS analysis according to international standards. Additionally, this study aimed to produce essential oil-containing microcapsules through complex coacervation, convert them into a solid form via freeze-drying, characterize the freeze-dried microcapsules using microscopic and spectroscopic methods, and optimize the microencapsulation process using various optimization techniques.

General Methodology

Essential oils were extracted via steam distillation using a 2 L glassware laboratory unit and a semi-industrial stainless steel system with a 50 L capacity. Microencapsulation was performed through complex coacervation, and freeze-drying was applied to solidify the coacervates. The optimization of complex coacervation was conducted using OFAT (One Factor At a Time) and DoE (Design of Experiments) methodologies.

Study 1 – Cultivation of Aromatic Medicinal Plants, Extraction, and Characterization of Essential Oils by GC-MS

Objective

This study aimed to cultivate sage (*Salvia officinalis* L.), lavender (*Lavandula angustifolia* Mill.), and yarrow (*Achillea millefolium* L.), extract their essential oils through steam distillation and analyze these oils by using GC-MS to evaluate their quality according to international standards, commercially available samples, and literature data.

Conclusions

The composition of sage essential oil varied over consecutive years, with compounds such as α-thujone, 1,8-cineole, and camphor showing fluctuations likely due to storage conditions and the natural degradation of volatile compounds, affecting ISO standard parameters. Lavender essential oil largely adhered to ISO 3515:2002 and European Pharmacopoeia (Ph. Eur.) standards for key components like linalyl acetate and linalool, though minor components showed variability, possibly due to storage effects. Yarrow essential oil exhibited high levels of chamazulene and germacrene D, with self-produced samples containing higher chamazulene compared to commercial samples from Bulgaria.

Study 2 – Microencapsulation of Essential Oils: Optimization by OFAT and Analysis of Microcapsules

Objective

This study aimed to encapsulate essential oils within protein-polysaccharide systems using gelatin and gum arabic as shell materials, optimize the microencapsulation process with three grades of gelatin type A, and investigate formulation and process-related factors. Further objectives included characterizing the microcapsules using microscopic and spectroscopic techniques and evaluating essential oil entrapment efficiency.

Conclusions

Optical and electron microscopy confirmed successful formation of core-shell microcapsules with a range of particle sizes. Different gelatin grades influenced both microcapsule size and structural characteristics, with higher Bloom values resulting in larger, less compact particles. Further analyses, including FT-IR, DSC, UV-VIS, and GC-MS, verified encapsulation with minimal core-capsule interaction, preserving essential oil composition. GC-MS analysis noted minor losses of volatile components post-encapsulation, though key constituents remained stable.

Study 3 – Optimization of Complex Coacervation of Lavender Oil by Response Surface Methodology (RSM)

Objective

Based on findings from Study 2 regarding particle size characteristics using OFAT optimization, this study aimed to further examine gelatin grade effects alongside other parameters to potentially modulate particle size through Design of Experiments methodology. A D-optimal design with 18 experimental runs was employed, focusing on core/shell ratio, gelatin grade, and stirring speed. Responses included particle size characteristics, sedimentation properties, and GC-MS analysis of lavender oil components.

Conclusions

Microcapsules with diverse morphological characteristics were produced, ranging from small (less than 10 μm) to large (over 200 μm) diameters, and varying from mono- to polynucleated cores and round to oval shapes. Sedimentation properties exhibited similar variability. Gelatin Bloom value was critical for particle size and sedimentation, while rotational speed was essential for even particle size distribution. The model showed limited predictive power for monoterpenes and monoterpene esters, but was effective for sesquiterpenes.