ORIGINAL ARTICLE

Formulation and Characterization of Poly(methyl methacrylate) Microcapsules Encapsulating Peppermint and Origanum Essential Oils

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Abstract

Introduction: Peppermint and origanum essential oils are known for their strong antimicrobial, antifungal, and antibiofilm properties. Despite their effectiveness, their clinical use is limited due to high volatility, poor water solubility, and instability under environmental conditions. Microencapsulation using biocompatible polymers like poly(methyl methacrylate) (PMMA) can help overcome these limitations by improving stability and enabling controlled release. **Materials and Methods:** Microcapsules were prepared using a modified solvent evaporation method. A mixture of peppermint and origanum oils in a 100:75 ratio was combined with PMMA in dichloromethane. This organic phase was emulsified into an aqueous phase containing 1% polyvinyl alcohol and 2 g sodium lauryl sulfate. The mixture was stirred for solvent evaporation, followed by centrifugation to isolate the microcapsules. The capsules were then analyzed for ultraviolet (UV) absorbance, particle size, morphology, yield, and entrapment efficiency (EE). **Results:** UV-visible spectrophotometry showed λmax at 253 nm for peppermint oil and 276 nm for origanum oil. BIOVIS microscopy revealed that 99.24% of the capsules were in the 0.5–5 µm range. SEM images showed spherical particles with smooth surfaces and no cracks. The yield was 70%, with 1050 mg recovered from 1500 mg. EE ranged from 33.39% to 50.64%, with 37.83–45.00% for peppermint oil and 27.58–58.00% for origanum oil. **Discussion:** The PMMA microencapsulation method effectively produced uniform and stable microcapsules. The particle size and surface morphology support their potential for controlled delivery. Moderate entrapment efficiencies suggest room for optimization, possibly influenced by differences in the physicochemical properties of the oils. Overall, the method showed promise in enhancing oil stability and reducing volatility. **Conclusion:** PMMA-based microencapsulation successfully stabilized peppermint and origanum oils, offering good yield, structure, and encapsulation efficiency. This system holds potential for future applications in controlled-release formulations for antimicrobial therapies.

Key words: Entrapment efficiency, microencapsulation, origanum oil, peppermint oil, poly(methyl methacrylate)

INTRODUCTION

Microencapsulation is a well-established strategy for enhancing the stability, bioavailability, and delivery efficiency of volatile and labile compounds such as essential oils (EOs). It involves entrapping active agents within a polymeric matrix or shell to protect them from environmental degradation and allow for controlled release.[1] The technique

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has been widely applied in pharmaceuticals, cosmetics, food, and nutraceuticals.

Among available polymers, poly(methyl methacrylate) (PMMA) is widely recognized for its biocompatibility, mechanical strength, and chemical stability.[2] PMMA microcapsules have been used in various drug delivery systems, including antibiotics, antifungals, anticancer drugs, and ophthalmic agents, due to their ability to provide localized and sustained release.[3-5] EOs, composed mainly of terpenoids and phenolic compounds, are known for their antimicrobial, antifungal, antioxidant, and anti-inflammatory effects.[6-8] However, challenges such as volatility, low aqueous solubility, and sensitivity to environmental conditions limit their direct use.[9,10] Microencapsulation offers a promising solution by improving shelf life, masking strong odors, and allowing site-specific delivery.[10-13]

Peppermint oil (*Mentha piperita*) is rich in menthol and demonstrates antibacterial, antifungal, and antibiofilm properties, particularly against *Staphylococcus aureus* and *Candida albicans*.[14,15] Origanum oil (*Origanum vulgare*) contains carvacrol and thymol, which exert antimicrobial action by disrupting microbial membranes and inhibiting biofilm formation.[16-18]

PMMA's compatibility with various drug classes and its versatility in formulation technologies make it a suitable candidate for encapsulating EOs. Table 1 presents a comparison of common PMMA preparation techniques, highlighting their benefits and limitations.

This study aims to develop and characterize PMMA-based microcapsules encapsulating peppermint and origanum EOs, to enhance their stability and enable controlled release for pharmaceutical and cosmeceutical applications.

MATERIALS AND METHODS

Materials

The materials used in this study included heat-cure acrylic resin (PMMA), specifically Trevalon® obtained from Dentsply India, serving as the primary polymer matrix. For active components, EOs such as peppermint oil (Himedia) and origanum oil were procured from Suyash Herbs to provide antimicrobial and therapeutic properties. Dichloromethane (DCM) was employed as the organic solvent in the microencapsulation process. To stabilize the emulsion system, polyvinyl alcohol (PVA) and sodium lauryl sulfate (SLS) were used as stabilizers.

Analytical methods

Preparation of stock solution

A combined stock solution of Peppermint and Origanum EOs in the ratio P100O75 (Peppermint: Origanum = 0.36 g: 0.24 g) was prepared at 100 µg/mL using methanol.

Determination of λmax

To evaluate total phenolic content, 0.5 mL of the methanolic EO mixture was mixed with 2.5 mL of 10% Folin–Ciocalteu reagent and 2.5 mL of 7.5% sodium bicarbonate. The mixture was incubated at room temperature for 45 min, and absorbance was measured using a UV-visible spectrophotometer. λmax values were 253 nm for peppermint oil, 276 nm for origanum oil, and both peaks were observed in the P100O75 blend, confirming the presence of phenolics from both oils.[25] Calibration curve of Peppermint and Origanum EOs was done to find our regression coefficient.

Table 1: Advantages and limitations of common PMMA microcapsule preparation techniques

Preparation technique	Advantages	Drawbacks	Ref.
Emulsion polymerization	Good particle size control; high yield; suitable for hydrophobic drugs	Requires surfactants; residual monomers may affect biocompatibility	[19]
Solvent evaporation	Simple; widely used; ideal for encapsulating essential oils and volatile drugs	Involves organic solvents; lower loading of hydrophilic drugs	[20]
Spray drying	Rapid and scalable; dry, free-flowing powder	High temperatures may degrade heat-sensitive compounds; high equipment cost	[21]
Precipitation polymerization	No surfactants needed; uniform particle size	Limited to water-insoluble monomers; narrow drug compatibility range	[22]
Suspension polymerization	Scalable; controlled bead size	Requires stabilizers; less suitable for volatile/thermolabile drugs	[23]
Miniemulsion polymerization	High encapsulation efficiency; narrow particle size distribution	Complex formulation; energy-intensive (ultrasonication or high shear required)	[24]

PMMA: Poly (methyl methacrylate)

Preparation of PMMA Microcapsules by Solvent Evaporation Method

PMMA microcapsules containing a peppermint–origanum EO blend (P100O75) were prepared using a modified solvent evaporation method with a polymer-to-oil ratio of 2:1.2. The stepwise process is illustrated in Figure 1.

The aqueous phase was prepared by dissolving 1% w/v PVA and 2 g SLS in 300 mL of distilled water. The organic phase consisted of 1 g PMMA dissolved in 30 mL DCM, into which 0.6 g of the EO mixture was added.

The organic phase was slowly added to 100 mL of the aqueous phase under vigorous stirring (1200 rpm) to form an oil-in-water emulsion. After 30 minutes, the emulsion was added to the remaining 200 mL of aqueous phase and stirred at 40°C to facilitate solvent evaporation and capsule formation.

The microcapsules were collected by centrifugation (2000 rpm), washed with distilled water to remove residual surfactant or polymer, air-dried, and stored in a desiccator for further evaluation. This technique enhances the stability and controlled release of EOs, making it suitable for pharmaceutical and cosmeceutical applications.[26,27]

Evaluation of PMMA microcapsules

The prepared PMMA microcapsules were evaluated for particle size, morphology, and process efficiency.

Particle size and morphology

Digital imaging microscopy (BIOVIS)

Dried microcapsules were dispersed in distilled water and observed under a BIOVIS microscope. Particle diameters were measured using image analysis software to assess size distribution and uniformity, which influence release behavior.[28]

Scanning electron microscopy (SEM)

Air-dried microcapsules were gold-coated and examined under SEM. High-resolution images provided insights into shape, surface texture, and integrity. Features such as smoothness, sphericity, or cracks were evaluated, as they impact encapsulation efficiency and release properties.[29]

Percentage yield

Process efficiency was calculated as the percentage yield using the formula:

$$\text{Percentage Yield} = \left(\frac{\text{Mass of Microcapsules Obtained}}{\text{Total Weight of Drug + Polymer}} \right) \times 100$$

Percentage yield and entrapment efficiency (EE)

Percentage yield was calculated as:

$$\% \text{ Yield} = (\text{Mass of microcapsules obtained/Total weight of polymer + oil}) \times 100$$

A high yield reflects minimal material loss during emulsification, solvent evaporation, and recovery, indicating process efficiency and scalability.[30]

EE was determined by dissolving 10 mg of microcapsules in 10 mL methanol, followed by vortexing and centrifugation. The supernatant was analyzed at 253 nm (peppermint) and 276 nm (origanum) using a UV-visible spectrophotometer. Oil content was quantified via calibration curves, and EE was calculated as the percentage of oil encapsulated relative to the initial amount used.[31]

Calculation:

The EE% was calculated using the following formula:

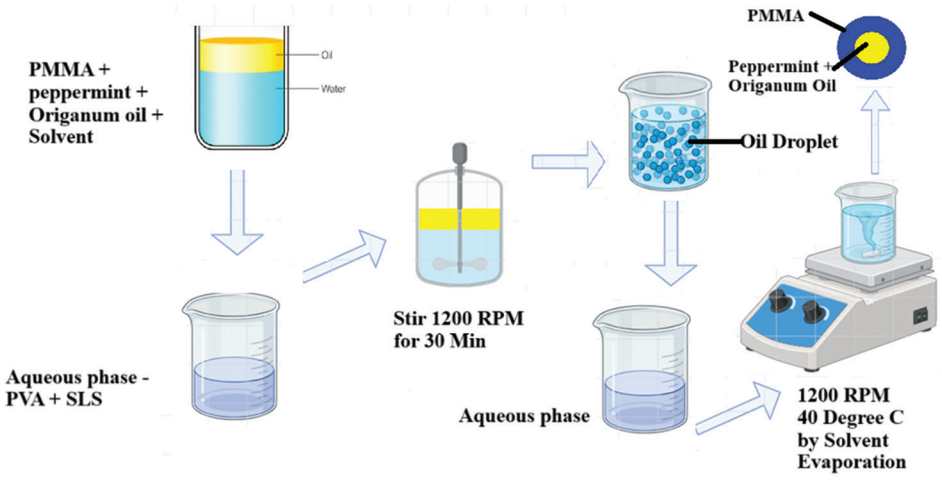

Figure 1: Schematic diagram of the preparation of poly(methyl methacrylate microcapsules with encapsulated Peppermint oil by the solvent evaporation method in O/W

$$\text{Entrapment Efficiency}(\%) = \left(\frac{\text{Actual Drug Content}}{\text{Theoretical Drug Content}} \right) \times 100$$

Where Actual Drug Content is the amount of EO recovered from microcapsules (via UV analysis), while Theoretical Drug Content is the total oil used, assuming full encapsulation.

This calculation assesses oil retention versus loss during processing.[32] High EE is vital for volatile oils and can be improved by optimizing factors like polymer-to-oil ratio, solvent type, and stirring speed.

RESULTS

UV-visible spectrophotometric analysis of λmax

UV-Visible spectrophotometry was used to determine the maximum absorbance wavelengths (λmax) of peppermint and origanum EOs for use in entrapment and release studies.

Each oil was diluted in ethyl acetate and scanned over 200–400 nm. Peppermint oil showed a λmax at 253 nm [Figure 2], attributed to menthol and menthone. Origanum oil exhibited a λmax at 276 nm [Figure 3], corresponding to thymol and carvacrol.

These λmax values reflect the characteristic phenolic and aromatic constituents of each oil and were used in all subsequent spectrophotometric quantifications.

Figure 4 shows the calibration curve of peppermint oil at λmaxis 253 nm, with the regression equation y = 0.000x and a correlation coefficient $R^2 = 0.981$, indicating strong linearity.

Figure 5 represents the calibration curve for origanum oil at λmax = 276 nm, with the equation y = 0.015x + 0.093 and $R^2 = 0.955$, confirming reliable linearity.

Figure 2: Ultraviolet spectrometric analysis of Peppermint essential oil

Both curves were used to quantify the respective EOs in microcapsules during EE and release studies, as determined by UV-visible spectrophotometry.

Particle size and morphology analysis

BIOVIS microscopy of 1974 PMMA microcapsules showed that 99.24% were within the 0.5–5 µm range, confirming a uniform, ultrafine particle distribution. Minor proportions were between 5.01–25 µm, with no particles above 25 µm, indicating monodispersity and process consistency [Figure 6].

SEM analysis (10,000×) revealed smooth, spherical, and

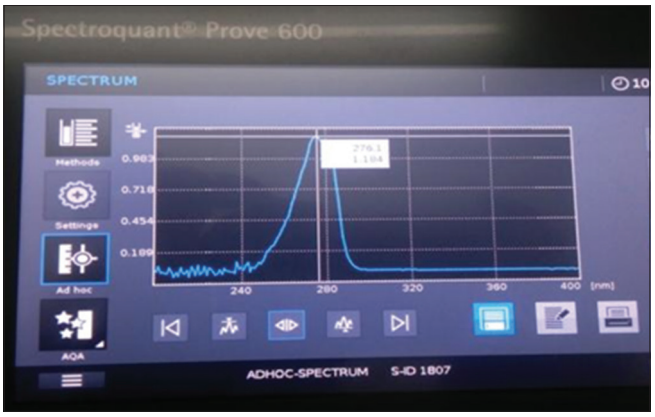

Figure 3: Ultraviolet spectrometric analysis of Origanum essential oil

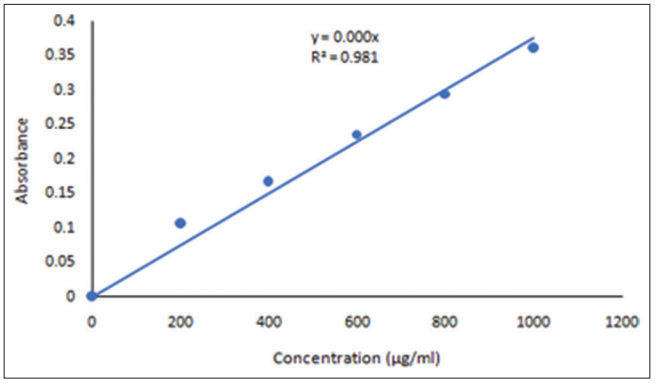

Figure 4: Standard plot of Peppermint oil, λmaxis 253 nm

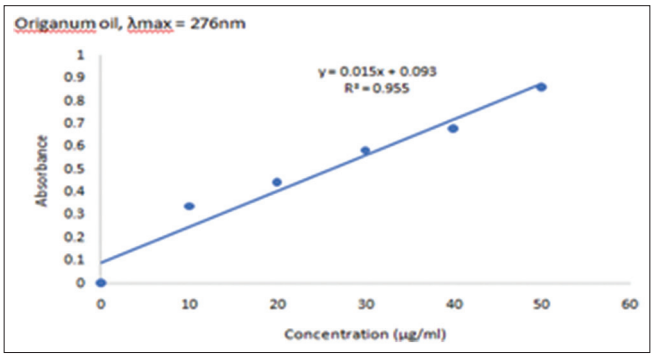

Figure 5: Standard plot of Origanum oil in ethyl acetate λmaxis 276 nm

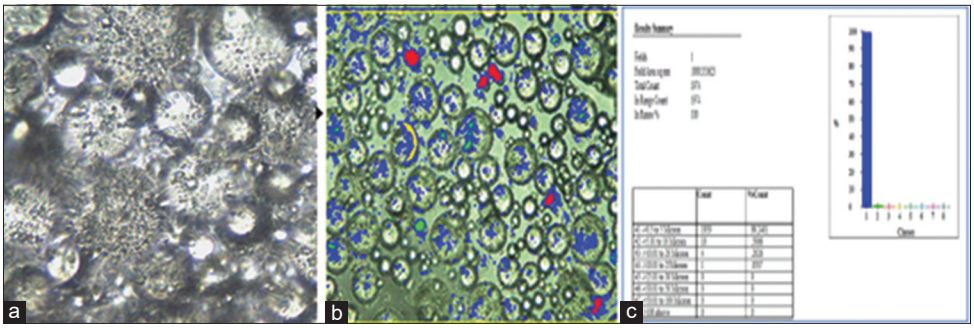

Figure 6: (a-c) BIOVIS particle size analyzer 40× magnification

discrete microcapsules with no surface pores or aggregation, confirming successful emulsification and a stable morphology – ideal for controlled drug delivery applications.

The smoothness and sphericity of the particles indicate proper polymer precipitation and solidification, consistent with an efficient solvent evaporation process. In addition, the absence of cracks, deformation, or surface roughness confirms the structural integrity of the microcapsules, which is critical for the protection and sustained release of the encapsulated EOs [Figure 7].

Overall, SEM analysis confirms that the formulation technique produced high-quality PMMA microcapsules, making them suitable for pharmaceutical or cosmeceutical applications where consistent particle morphology is essential.

Percentage yield and EE

The microencapsulation process yielded 1050 mg of PMMA microcapsules from 1500 mg of raw materials, resulting in a 70% yield. This reflects good process efficiency with minimal material loss, validating the suitability of the solvent evaporation method for encapsulating volatile EOs.

EE, determined through UV spectrophotometry at 253 nm (peppermint) and 276 nm (origanum), ranged from 33.39% to 50.64%, with individual oil EE between 27.58% and 58.00%. The moderate EE is attributed to the oils' volatility and possible loss during processing. Further optimization of formulation parameters may improve retention.

DISCUSSION

This study successfully formulated PMMA microcapsules containing peppermint and origanum EOs using a solvent evaporation technique, yielding stable microspheres with suitable properties for drug delivery.

UV-visible λmax determination

UV analysis confirmed λmax at 253 nm (peppermint) and 276 nm (origanum), corresponding to key constituents

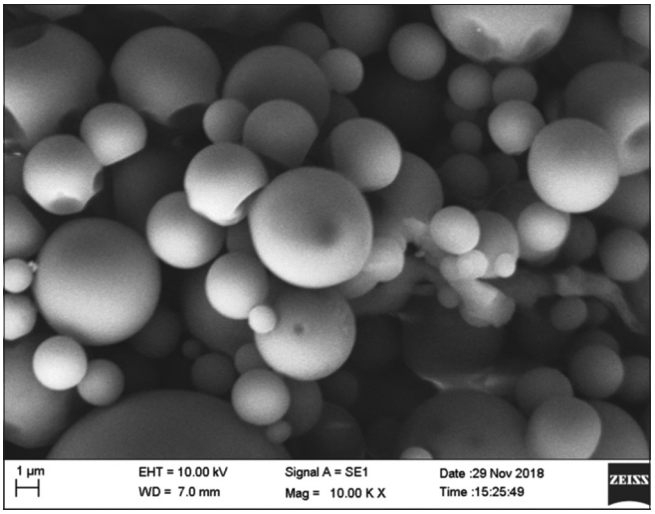

Figure 7: Microcapsules were examined using scanning electron microscopy

such as menthol and thymol. These values support accurate quantification during entrapment and release studies, validating UV spectrophotometry for EO tracking.[29,33,34]

Particle size and morphology

BIOVIS analysis showed 99.24% of particles within 0.5–5 µm, indicating a uniform and monodispersed system. SEM confirmed spherical, smooth, non-porous particles, ideal for controlled release. These outcomes align with previous findings on polymer-based EO encapsulation.[24,26,30,35,36]

Percentage yield

A 70% yield reflects minimal material loss and efficient process control, consistent with literature reports for solvent evaporation methods.[28] Controlled stirring and optimized surfactant concentration likely contributed to this outcome.

EE

EE ranged from 33.39% to 50.64%, with individual oils between 27.58% and 58.00%. Moderate EE is expected due

to oil volatility and loss during evaporation. Optimizing oil-to-polymer ratio and exploring alternative encapsulation strategies may enhance EE further.[31]

CONCLUSION

The present study successfully demonstrated the formulation, characterization, and evaluation of PMMA-based microcapsules encapsulating peppermint and origanum EOs using a modified solvent evaporation method. The EOs were selected for their well-documented antimicrobial and therapeutic properties, while PMMA served as a robust, biocompatible polymer matrix offering controlled release and protection of volatile actives.

UV-Visible spectrophotometric analysis confirmed the λmax values of peppermint and origanum oils at 253 nm and 276 nm, respectively, which served as the basis for further analytical quantification. The microcapsules exhibited a uniform particle size distribution predominantly in the range of 0.5–5 µm, with spherical morphology and smooth surfaces confirmed via Digital Imaging Microscopy (BIOVIS) and SEM. These features support the integrity, stability, and potential for controlled delivery of the encapsulated oils.

The process yielded approximately 70% of microcapsules, indicating good material recovery and efficiency of the encapsulation technique. EE ranged from 33.39% to 50.64%, demonstrating moderate encapsulation capacity. While promising, these results suggest the need for further process optimization to improve oil retention, especially given the volatility of EO components.

Overall, this formulation strategy offers a promising platform for the delivery of EOs in pharmaceutical and cosmeceutical applications, combining controlled release properties with enhanced stability. Future studies may focus on in vitro release kinetics, antimicrobial efficacy, and scalability for commercial development.

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AUTHORS' CONTRIBUTIONS

All authors have accepted responsibility for the manuscript's content and consented to its submission. They have thoroughly reviewed all results and unanimously approved the final version of the manuscript.

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