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Development and Characterization of Prickly Pear Seed Oil Microcapsules in a Biopolymeric Alginate-Gelatin Matrix for Controlled Release

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Abstract

This study aimed to encapsulate the sensitive bioactive compounds of Prickly Pear Seed Oil (PPSO) (Opuntia ficus-indica) using a biopolymeric matrix composed of sodium alginate and gelatin. PPSO, which is rich in essential fatty acids and antioxidants, is highly susceptible to degradation caused by light, oxygen, and heat. Microcapsules were prepared using the complex coacervation technique to enhance oil stability and ensure controlled release under physiological conditions. The physicochemical, structural, and thermal properties of the microcapsules were evaluated using UV–Visible spectroscopy, FTIR, thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC). The results confirmed successful oil encapsulation and improved thermal stability compared to pure PPSO. The formulation containing 0.75 g of oil exhibited the highest encapsulation efficiency (93.51%) and the most favorable controlled release behavior. Release studies demonstrated low oil release in simulated gastric fluid (pH 1.2), followed by a sustained and controlled release in simulated intestinal media (pH 6.8 and 7.4). These findings highlight the effectiveness of the alginate–gelatin system for stabilizing PPSO, offering promising potential for applications in food, cosmetic, and pharmaceutical industries.

Keywords: *Alginate, Gelatin, Controlled Release, Encapsulation, Prickly Pear Seed Oil*

I. Introduction

Microencapsulation is a widely used technique in the food, cosmetic, and pharmaceutical industries for the protection, stabilization, and controlled delivery of sensitive bioactive compounds. This approach involves enclosing an active core material within a protective polymeric matrix, thereby reducing its exposure to environmental factors such as oxygen, light, moisture, and temperature fluctuations. As a result, microencapsulation enhances the stability, bioavailability, and shelf life of high-value natural ingredients, particularly oils rich in polyunsaturated fatty acids [01-03].

The successful application of microencapsulation is pivotal for preserving the functional integrity and extending the shelf life of high-value compounds, thereby enhancing product quality and efficacy [04]. Among the diverse methods available for microencapsulation, complex coacervation is recognized for its operational simplicity, high encapsulation efficiency, and ability to produce robust, high-density microcapsules using food-grade biopolymers [05]. This phase separation technique is fundamentally driven by the electrostatic attraction between two oppositely charged polymeric materials in an aqueous medium. A highly effective and industrially relevant coacervation system utilizes the positively charged protein, gelatin, and the negatively charged polysaccharide, sodium alginate [06]. By precisely controlling critical parameters, notably pH and temperature, the interaction between the carboxyl groups of alginate and the protonated amino groups of gelatin leads to the formation of an insoluble coacervate complex. This dense, biopolymeric liquid phase preferentially precipitates around the dispersed core material, forming a continuous microcapsule wall that is subsequently stabilized, often

through cross-linking, to yield a solid, protective structure [07]. The present study focuses on the stabilization of Prickly Pear Seed Oil (PPSO), extracted from the seeds of Opuntia ficus-indica. PPSO has garnered significant global attention for its superior composition, which is characterized by an exceptionally high content of polyunsaturated fatty acids, primarily linoleic acid (up to 70%), and potent lipophilic antioxidants, including a rich profile of tocopherols and phytosterols [08]. These bioactive components confer remarkable nutritional, anti-inflammatory, and skin-regenerating properties, positioning PPSO as one of the most valuable vegetable oils in the cosmetic and functional food markets [09].

However, the chemical profile responsible for PPSO's high value specifically its high degree of unsaturation also dictates its inherent instability. The numerous double bonds in the fatty acid chains are highly susceptible to autoxidation, a chain reaction drastically accelerated by exposure to light and atmospheric oxygen [10]. This oxidative degradation leads to the rapid depletion of beneficial antioxidants, the formation of undesirable volatile off-compounds (rancidity), and a significant loss in the oil's functional and sensory quality. Consequently, the commercial application of PPSO is severely limited by its short shelf life under ambient conditions.

Therefore, the primary objective of this work was to employ the gelatin-sodium alginate complex coacervation method for the efficient microencapsulation of PPSO. The goal is to construct a resilient biopolymeric shell that functions as an effective barrier to protect the oil from photo- and thermo-oxidative degradation. Through comprehensive physicochemical characterization, morphological analysis, and an assessment of the release kinetics in simulated media, this research seeks to demonstrate a viable strategy for stabilizing Prickly Pear Seed Oil, ensuring the preservation of its bioactivity for long-term industrial and consumer use.

II. Material and methods

2.1. Materials

The natural extract used was Prickly Pear Seed Oil (PPSO) (Opuntia ficus-indica), commercialized as Golden Brand Herbal Oil. The biopolymeric matrix was formed using Sodium Alginate (Alg) and Gelatin (Gel). Other chemicals included Polyethylene Glycol (PEG 6000), Polysorbate 80 (Tween 80), and various reagents used for pH control and solution preparation, such as Sodium Hydroxide (NaOH), Sodium Chloride (NaCl), Monopotassium Phosphate ($KH_2\ PO_4$), Hydrochloric Acid (HCl), and n-hexane.

2.2. Microcapsule Preparation

The microcapsules containing Prickly Pear Seed Oil (PPSO) Prickly pear seed oil (PPSO) microcapsules were prepared using the complex coacervation technique with sodium alginate (Alg) and gelatin (Gel) as wall materials. Sodium alginate was dissolved in deionized water at a concentration of 2% (w/v) under continuous magnetic stirring at 40 °C until complete solubilization. Gelatin was dissolved separately in deionized water at 2% (w/v) under the same conditions. The two polymer solutions were then mixed at an Alg/Gel mass ratio of 1:1. The pH of the biopolymeric mixture was carefully adjusted to pH 4.0 ± 0.1 using 0.1 M HCl, corresponding to the optimal pH range for electrostatic interaction between the negatively charged carboxylate groups of alginate and the positively charged amino groups of gelatin, thus inducing complex coacervation. Prickly pear seed oil was incorporated into the polymeric mixture at three different concentrations (0.5 g, 0.75 g, and 1 g) under high-speed mechanical stirring at 10,000 rpm for 10 minutes to form a stable oil-in-water emulsion. Tween 80 (1% w/v) was added as an emulsifying agent to enhance oil dispersion, while PEG 6000 (1% w/v) was used as a plasticizer to improve the flexibility and integrity of the microcapsule wall. Cross-linking of the formed coacervates was achieved by the dropwise addition of calcium chloride ($CaCl_2$, 0.2 M) under gentle stirring for 30 minutes, allowing ionic gelation of the alginate chains and stabilization of the microcapsules. The resulting microcapsules were collected by filtration, washed thoroughly with deionized water to remove unencapsulated oil and residual reagents, and then dried at 40 °C for 24 h. The dried microcapsules were stored in airtight containers at room temperature until further characterization.

Solutions buffered at controlled pH were prepared to assess the release in simulated physiological environments.

- **Simulated Gastric Fluid (SGF)**: pH 1.2, prepared using NaCl and HCl.
- **Simulated Intestinal Fluid (SIF, duodenum)**: pH 6.8, prepared using $KH_2\ PO_4$ and NaOH.
- **Phosphate Buffer Solution (PBS)**: pH 7.4, simulating other physiological fluids.

2.3. Characterization Techniques

The prepared microcapsules were characterized using several analytical techniques:

- **UV-Visible Spectroscopy:** by SpectroScan 50, used to establish the calibration curve of the PPSO and determine the **Encapsulation Efficiency (EE)** and concentration.

$$EE\% = 100 * \frac{Ccp}{Ci} \quad (1)$$

- **Infrared Spectroscopy (FTIR):** using SHIMADZU FTIR-8400S in the range of 4000-400 cm-1 with a resolution of 4 cm-1. Used to identify the functional groups of the oil and the biopolymers, confirming the successful encapsulation and interactions within the matrix.
- **Thermal analysis: Thermogravimetric Analysis (ATG) and Differential Scanning Calorimetry (DSC):** using a LINSEIS STAPT 1600 type thermogravimetric apparatus, in the temperature range from 20 -700 °C and a heating rate of 10 °C/min. Performed to evaluate the **thermal stability** and behavior of the raw materials and the final microcapsules.
- **Controlled Release Kinetics:** A volume of 100 mL of each buffered solution at pH1.2 (SGF), pH 6.8 (SIF), and pH 7.4 (PBS) was transferred into separate Erlenmeyer flasks. Subsequently, three microcapsule samples, each weighing 1g and corresponding to the optimal formulation prepared with 0.75g of oil, were introduced into the respective solutions.

The objective of this procedure was to initiate the controlled release study of the encapsulated oil under simulated physiological conditions (gastric, intestinal, and colonic) to evaluate the gastroprotective capacity and targeted delivery performance of the alginate-gelatin microcapsule system.

III. Results and discussion

3.1. UV-Visible Spectroscopy and Encapsulation Efficiency

UV-Visible spectroscopy was used to establish the calibration curve of the Prickly Pear Seed Oil at the maximum absorption wavelength of λ=278nm (Figure 1). Analysis of the supernatant allowed for the determination of the Encapsulation Efficiency (EE) (Table 1). The microcapsule formulation prepared with 0.75g of PPSO demonstrated the best encapsulation yield. The optimal encapsulation efficiency achieved with the 0.75g formulation was 93.51%. This high efficiency confirms the suitability of the alginate-gelatin system for retaining the oil. The same result was observed by M. A. Rahim et al. (2021), who noted that the combination of isolated soybean proteins (SPI) and soy polysaccharides (SPS) as wall materials offered a high encapsulation efficiency (63.55%) for seabuckthorn seed oil, highlighting the importance of choosing appropriate encapsulation materials [11].

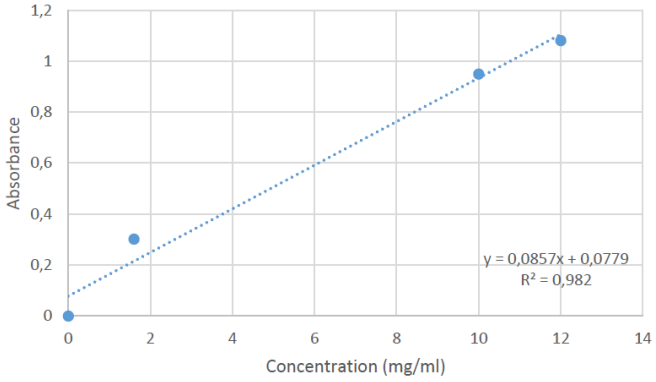

Figure 1. Calibration Curve of Prickly Pear Seed Oil (PPSO) at λ= 278 nm.

Table: Influence of PPSO concentration on encapsulation efficiency

Ci PPSO (mg/ml)	25	37,5	50
Ccp PPSO (mg/ml)	23,37	33,28	49,36
EE%	88,76 ±2	93,51 ±2	98,73 ±2

3.2. **Fourier-Transform Infrared Spectroscopy (FTIR)**

FTIR spectroscopy was employed to confirm the successful encapsulation of Prickly Pear Seed Oil (PPSO) within the alginate–gelatin matrix and to investigate possible intermolecular interactions between the core and wall materials (Figures 2 and 3). The FTIR spectrum of sodium alginate exhibited a broad absorption band in the range 3200–3400 cm^{-1}, attributed to O–H stretching vibrations, and a band at 2920 cm^{-1} corresponding to C–H stretching. The characteristic asymmetric and symmetric stretching vibrations of carboxylate groups ($–COO^-$) were observed at 1602 cm^{-1} and 1415 cm^{-1}, respectively, confirming the polysaccharide structure of alginate.[12]. The gelatin spectrum displayed typical protein-related bands, including a broad O–H/N–H stretching band at 3294 cm^{-1}, the Amide I band at 1641 cm^{-1} (C=O stretching), Amide II at 1544 cm^{-1} (N–H bending and C–N stretching), and Amide III at 1230 cm^{-1}, which are characteristic of peptide bonds.[13]. The FTIR spectrum of PPSO revealed the typical features of triglyceride-based oils. A strong absorption band at 1740 cm^{-1} was assigned to the ester carbonyl (C=O) stretching vibration, while bands at 2850–2950 cm^{-1} corresponded to symmetric and asymmetric stretching of aliphatic C–H groups. A weak band around 3006 cm^{-1} indicated the presence of unsaturated C=C–H stretching, and bands in the region 1160–1090 cm^{-1} were associated with C–O ester linkages.[14]. The FTIR spectra of the PPSO-loaded microcapsules (0.5 g, 0.75 g, and 1 g) showed the presence of characteristic peaks of both the biopolymeric matrix and the

oil, confirming successful encapsulation. Notably, the ester C=O band of PPSO at 1740 cm^{-1} was preserved in all formulations, indicating that the chemical structure of the oil remained intact after encapsulation.[15]. A slight shift of the alginate carboxylate asymmetric stretching band from 1602 cm^{-1} (pure alginate) to approximately 1594–1597 cm^{-1} in the microcapsules suggests electrostatic interactions between alginate carboxyl groups and protonated amino groups of gelatin. Additionally, changes in the intensity of the O–H/N–H stretching band (3200–3400 cm^{-1}) indicate the formation of hydrogen bonding interactions within the alginate–gelatin–oil system. Among the three formulations, the microcapsules containing 0.75 g of PPSO exhibited the highest intensity of oil-related bands (2920 cm^{-1} and 1740 cm^{-1}), suggesting a more homogeneous oil distribution and improved encapsulation efficiency. In contrast, the 1 g formulation showed comparatively lower relative intensity, which may indicate partial matrix saturation and less uniform dispersion of the oil at higher loading levels. Overall, the FTIR results confirm the successful encapsulation of PPSO within the alginate–gelatin matrix through physical interactions, without chemical degradation of the oil. The 0.75 g formulation provides the most favorable interaction balance.[16] and [17].

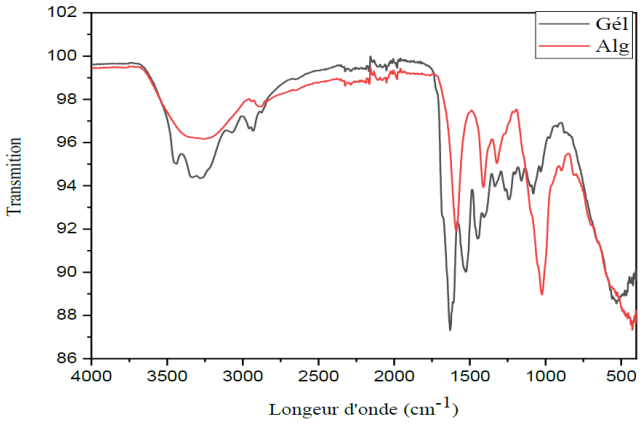

Figure 2. Infrared spectrophotometer analysis of Alginate (Alg) and Gelatin (Gel)

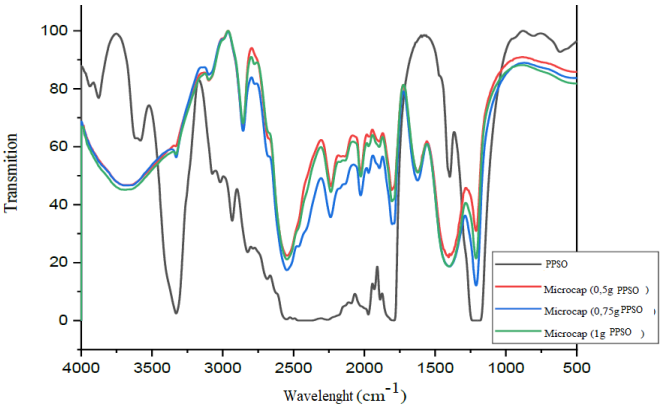

Figure 3. Infrared spectra of PPSO (oil), Alg (alginate), Gel (gelatin), and microcapsules containing 0.5 g, 0.75 g, and 1 g of PPSO.

3.3. Thermal Stability (ATG/DSC)

Thermogravimetric analysis was performed to evaluate the thermal stability and degradation behavior of pure PPSO, the individual wall materials (alginate and gelatin), and the PPSO-loaded microcapsules containing 0.5 g, 0.75 g, and 1 g of oil (Figure 4).The TGA curves of sodium alginate and gelatin exhibited an initial mass loss of approximately 8–12% between 30 °C and 150 °C, which can be attributed to the evaporation of physically and chemically bound water. The main degradation stage of both biopolymers occurred between 230 °C and 380 °C, corresponding to the decomposition of the polymer backbone. Pure PPSO showed a single major degradation step starting at an onset temperature (Tonset) of approximately 300 °C, with rapid mass loss up to 450 °C, indicating its high susceptibility to thermal degradation. In contrast, the PPSO-loaded microcapsules exhibited enhanced thermal stability compared to pure oil. The onset degradation temperatures were shifted to lower but more gradual degradation profiles, with Tonset values of approximately 225 °C, 239 °C, and 245 °C for microcapsules containing 0.5 g, 0.75 g, and 1 g of PPSO, respectively. This behavior indicates that the polymeric matrix effectively delays and moderates oil degradation by acting as a thermal barrier. Moreover, the residual mass at 700 °C increased with increasing oil content, reaching its highest value for the 1 g PPSO formulation, which suggests improved char formation and enhanced structural stability due to stronger polymer–oil interactions. Although the 1 g formulation exhibited the highest thermal resistance, the 0.75 g formulation presented a favorable balance between thermal stability and encapsulation homogeneity. These results confirm that encapsulation within an alginate–gelatin matrix significantly improves the thermal resistance of PPSO, making the system suitable for applications involving moderate thermal processing.[18].

DSC analysis of the microcapsules

Differential scanning calorimetry was employed to further investigate the thermal transitions and stability of the PPSO-loaded microcapsules (Figure 5).

All formulations exhibited a first endothermic peak between 50 °C and 100 °C, which is attributed to the evaporation of residual moisture entrapped within the biopolymeric matrix. This endothermic event was less intense for the microcapsules containing 1 g of PPSO, indicating reduced water affinity due to increased hydrophobicity. A major exothermic transition was observed between 350 °C and 470 °C for all microcapsule formulations, corresponding to the

thermal decomposition of the alginate–gelatin network and the encapsulated oil. The position and intensity of this peak varied with oil concentration. The microcapsules containing 1 g of PPSO exhibited the highest peak temperature, indicating superior thermal stability, whereas the 0.5 g formulation showed a broader and less intense peak, suggesting lower resistance to thermal stress. The 0.75 g PPSO formulation displayed intermediate thermal behavior, with a well-defined exothermic peak and moderate enthalpy change, reflecting a stable and homogeneous microcapsule structure. This formulation combines sufficient hydrophobicity, high encapsulation efficiency (93.51%), and satisfactory thermal resistance, making it the most suitable candidate for controlled release applications. Overall, DSC analysis corroborates the TGA results and demonstrates that increasing PPSO content enhances the thermal stability of the microcapsules. However, an intermediate oil loading (0.75 g) provides the optimal compromise between thermal performance and encapsulation efficiency [14].

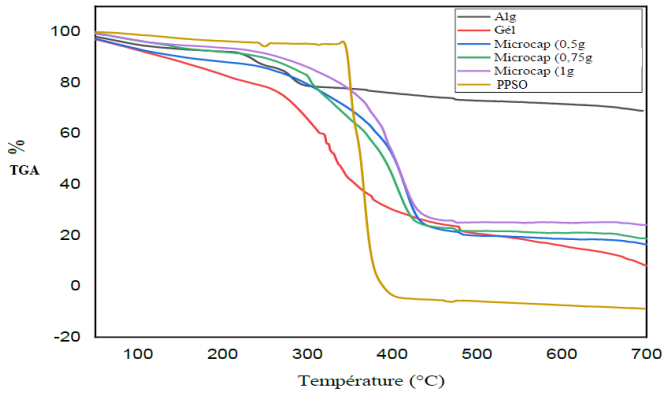

Figure 4. TGA thermal analysis of PPSO (oil), Alg (alginate), Gel (gelatin), and microcapsules (0.5, 0.75, and 1 g PPSO)

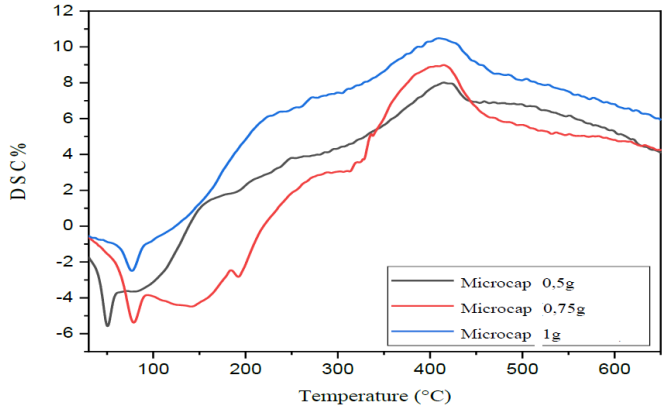

Figure 5. DSC analysis curves of microcapsules containing 0.5, 0.75, and 1 g of PPSO.

3.4. Controlled Release Kinetics

Figure 6 presents the release profile of the encapsulated oil from microcapsules containing 0.75g of oil in three media simulating different physiological conditions: an acidic pH (pH 1.2– Simulated Gastric Fluid, SGF), an intestinal pH (pH 6.8– Simulated Intestinal Fluid, SIF), and a colonic pH (pH 7.4– Phosphate Buffer Solution, PBS). The release kinetics show two distinct phases: Initial Phase (0 to 200 minutes): A rapid burst release is observed at pH 6.8 and 7.4, primarily attributed to the diffusion of loosely retained or surface-adsorbed oil from the polymeric matrix. This behavior is typical of biopolymeric systems encapsulating essential oils, as reported by M. C. Otálora et al (2023), who observed an initial rapid release due to weak surface interaction between the oil and the polymers [18]. Prolonged Phase (400 to 1440 minutes): The release becomes slower and more controlled, particularly at pH 6.8 and 7.4. This is explained by a gradual disintegration of the alginate-gelatin network under the influence of the physiological pH, enabling a more targeted and sustained release of the encapsulated oil. This mechanism is consistent with the work of Soukoulis et al. (2025), who showed that alginate-gelatin-based systems ensure protection in the gastric environment and adapted release under intestinal conditions [19]. At pH 1.2, the release remains relatively low throughout the experiment, highlighting the gastroprotective capacity of the matrix, which remains compact and poorly permeable in the acidic medium. This stability is reinforced by the high encapsulation efficiency observed (93.51%), indicating that the encapsulating system is suitable for protecting the oil in the gastric environment while permitting a controlled release in the intestinal and colonic areas, as desired for targeted oral applications.

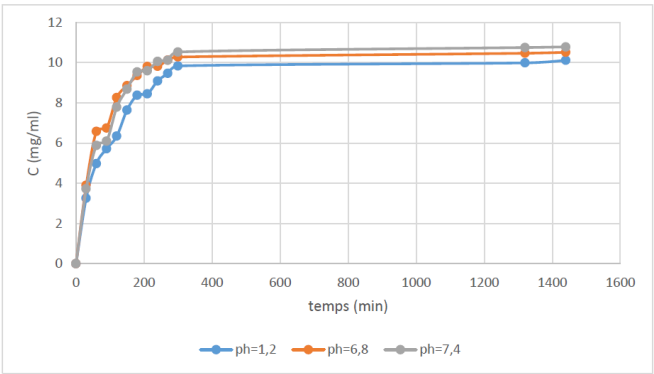

Figure 6. Release profile of the 0.75 g microcapsules in different pH media

IV. Conclusions

This study demonstrated the successful encapsulation of prickly pear seed oil using an alginate-gelatin biopolymeric matrix through the complex coacervation technique. The encapsulation process effectively protected the oil's sensitive bioactive compounds and significantly improved its thermal stability. Among the tested formulations, the microcapsules containing 0.75 g of PPSO exhibited the best compromise

between encapsulation efficiency, thermal performance, and controlled release behavior. Release studies confirmed the gastroprotective nature of the matrix under acidic conditions and its ability to provide sustained release in intestinal environments. These results suggest that alginate–gelatin microcapsules represent a promising strategy for stabilizing and controlling the delivery of PPSO in functional food, cosmetic, and pharmaceutical applications.

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