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Biopolymer-Based Microencapsulation of Bioactive Compounds: Evaluation of the Impact of Encapsulated Compound Characteristics on Process Efficiency

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Abstract: Complex coacervation using proteins and polysaccharides enables efficient microencapsulation with high thermal stability, facilitating continuous core component release and yielding coacervates with superior properties for diverse applications. This study investigates the use of casein and pectin for microencapsulating *Ocimum basilicum* L. essential oil (EO) and phenolic extract (PE). Microencapsulation yield and efficiency were 85.3% and 89.8% for EO microcapsules (EO-MC) and 53.1% and 70.0% for PE microcapsules (PE-MC). Optical microscopy revealed spherical microcapsules; EO-MC had smooth surfaces, while PE-MC had porous surfaces. Thermal analysis showed stability, with both types exhibiting two stages of weight loss. XRD analysis indicated increased crystallinity in EO-MC and high crystallinity in PE-MC due to phenolic interactions. FTIR spectroscopy confirmed molecular interactions, including hydrogen bonding between phenolic compounds and the biopolymer matrix and amide bonds between the carboxyl groups of pectin and the amino groups of casein, ensuring the successful encapsulation of the bioactive compounds. These findings highlight the potential of casein and pectin for microencapsulating extracts, particularly EOs, for food industry applications.

Keywords: encapsulated compounds; biopolymer; freeze-drying; thermal behavior; crystallinity

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1. Introduction

The genus *Ocimum*, Lamiaceae, commonly known as basil, showcases remarkable diversity and is predominantly cultivated in Mediterranean regions [1–4]. Among its

various species, *Ocimum basilicum* L., or sweet basil, stands out as a popular culinary herb available in fresh and dried forms, owing to its multifaceted pharmacological effects [5,6]. Basil, with its extensive range of applications, demonstrates potent antimicrobial, antifungal, antioxidant, and anti-inflammatory properties, making it a promising candidate for therapeutic interventions in both human and animal health [1,7–9]. *Ocimum basilicum* L. comprises numerous active compounds within its essential oil (EO), including phenols, alcohols, esters, and oxides, contributing to its distinct aroma and therapeutic potential [10,11]. Furthermore, the EO of sweet basil finds applications in pharmaceuticals for treating various ailments such as coughs, headaches, and gastrointestinal issues [12,13], while also being extensively utilized in the food and perfume industries due to its flavoring and aromatic properties [14–16]. Extracts derived from *Ocimum basilicum* L. leaves encompass a wide array of bioactive components, including rosmarinic acid, a phenolic compound renowned for its antioxidant qualities and therapeutic benefits against oxidative stress-related disorders and cancer proliferation [17–19]. Additionally, rosmarinic acid demonstrates hepatoprotective, lung-protective, and cardioprotective effects, attributed to its antioxidant and anti-inflammatory activities [20–23]. All these findings underscore the immense therapeutic potential of basil extracts and highlight the importance of further research to harness their benefits effectively [24,25].

Polysaccharides, particularly pectin, are pivotal renewable resources with diverse applications in the food and pharmaceutical industries. Pectin, renowned for its abundance, biodegradability, and versatility, acts as a crucial component within the plant cell wall, facilitating structural integrity and cell adhesion [26]. Beyond its role in plant physiology, pectin serves as a multifunctional ingredient in food production, contributing to texture and stability while serving as a gelling agent, thickener, emulsifier, and sugar substitute [27,28]. In pharmaceuticals, pectin functions as a valuable drug carrier, offering diverse applications in gel beads, matrices, and film-coated tablets [29,30].

Conversely, casein, a major milk protein, showcases remarkable properties such as exceptional surface activity and insolubility in aqueous mediums at low pH levels, making it invaluable across various applications [29,31]. Its unique structure endows it with excellent barrier properties against oxygen and non-polar molecules, rendering it suitable for the production of protein-based edible capsules [32]. Furthermore, its compatibility with other encapsulation materials and its potential to stabilize acidified dairy drinks highlight its versatility and importance in industrial applications [30]. The interest in studying the interactions between pectin and casein has surged, particularly in enhancing their physicochemical properties and exploring novel applications in the food and pharmaceutical sectors. Research has elucidated various factors influencing their interactions, including pH, temperature, ionic strength, and the presence of oil droplets, particularly in the development of acidic milk drinks [31].

Complex coacervation, a phenomenon driven by electrostatic interactions between oppositely charged biopolymers, finds wide applications in the food and pharmaceutical industries [26,33]. Influenced by factors like pH, temperature, and biopolymer ratios, it enables the encapsulation of bioactive compounds, ensuring protection and controlled release [34,35]. Despite being miscible in solvents, coacervates form through solute interactions, offering potential in food processing and encapsulation [36]. The process, crucial for creating coacervates, is utilized in various food applications and offers innovative solutions for enhancing food functionality and shelf-life through the improved stability of bioactive compounds [37–39]. This technology, driven by electrostatic attraction between proteins and polysaccharides, holds promise in protecting bioactive compounds and improving their stability during processing and storage, thereby offering innovative solutions for enhancing food functionality and shelf-life [40]. Several researchers delved into the mi-

croencapsulation of basil extracts. Ozdemir et al. [41] explored the microencapsulation efficiency of basil essential oil through various combinations of gum arabic, whey protein isolate, and maltodextrin. Similarly, Paul et al. [42] and Fadel et al. [43] examined the impact of coating materials such as maltodextrin, sodium caseinate, carboxymethyl cellulose, sodium alginate, and chitosan on the microencapsulation of basil oil using spray drying techniques. Expanding on this, chitosan and sodium alginate biopolymers were utilized by Thuekeaw et al. [44] to microencapsulate basil essential oil, employing a hot air oven for the drying process. Meanwhile, Tomé and da Silva [45] obtained basil ethanolic extract microcapsules using sodium alginate as the wall material, dried via air drying.

While numerous studies have explored the microencapsulation techniques, there is a paucity of investigation about understanding the potential of complex coacervation for encapsulating *Ocimum basilicum* L. essential oils and phenolic extract efficiently. This novel study aims to fill this gap by investigating the efficacy of complex coacervation employing casein from bovine milk and pectin from citrus peels as wall materials. The choice of freeze-drying as the drying method underscores the commitment to preserving the delicate bioactive compounds inherent in basil extracts. This innovative approach not only addresses a significant research gap but also holds promise for unlocking novel applications in various industries. What sets this study apart is its innovative approach to combining complex coacervation with these specific biopolymers for the encapsulation of basil oil and phenolic extract. By employing sophisticated physicochemical characterizations such as thermogravimetric and differential scanning calorimetry analysis, X-ray diffraction, and FTIR spectroscopy, this research delves into elucidating the moisture content, thermal behavior, crystallinity, and chemical structure of the resulting microcapsules. Additionally, the meticulous evaluation of particle size and distribution adds depth to this study's findings, contributing valuable insights into the potential applications of these microencapsulated basil extracts in various sectors.

2. Materials and Methods

2.1. Material and Reagents

Ocimum basilicum L. aerial parts were collected (Lakhdaria province of Bouira, Algeria), and air-dried for a week. An electrical grinder (SAYONA) was used to grind the plant into a fine powder, which was then sieved through a sieve shaker with a porosity of 100 µm. The powder was stored in an amber flask until use. Ethanol (C_2H_6O), hexane (C_6H_{14}), methanol (CH_3OH), casein from bovine milk ((Purity: 87–94% protein basis, high molecular weight phosphoprotein), (C7078, Lot ♯BCCD1666)), pectin (Ps) from *citrus* peel ((Purity: galacturonic acid ≥74.0% dried basis, poly-D-galacturonic acid methyl ester, molecular weight may range from 10 kDa to 300 kDa), (P9135, Lot ♯SLCB2455)), trichloroacetic acid ($C_2HCl_3O_2$), sodium sulfate (Na_2SO_4), sodium hydroxide (NaOH), and hydrochloric acid (HCl) were purchased from Sigma-Aldrich (Brøndby, Denmark). Sodium phosphate monobasic ($NaH_2PO_4.H_2O$) and sodium phosphate dibasic ($Na_2HPO_4.12H_2O$) were purchased from VWR Chemicals (Fontenay-sous-Bois, France).

2.2. Extraction of Bioactive Compounds

The bioactive compounds extracted from basil powder in this study employed unconventional extraction methods. Basil essential oil (EO) was obtained through microwave-assisted extraction using a Samsung ME6124ST (Samsung, Seremban, Malaysia) (2450 MHz, 1000 W) oven with interior dimensions of 373 mm × 233 mm × 393 mm (W × H × D) [46]. Following the protocol outlined by Bellik et al. [47], 20 g of basil powder was mixed with 200 mL of distilled water in a flat-bottom flask, and then subjected to microwave irradiation

at 500 W for 15 min. Subsequently, the EO was separated from the water and stored in an amber flask at 4 °C.

The phenolic extract of basil (PE) was acquired via ultrasound-assisted extraction [48]. An amount of 10 g of basil powder was combined with 100 mL of ethanol (70% v/v) and set to ultrasound treatment in a bath operating at 40 kHz and 40 °C for 30 min, as described by Pedrosa et al. [49]. The resulting extract was obtained through centrifugation at 4025× g for 15 min using a Sigma 3–16 L centrifuge (Osterode, Germany), followed by filtration [50]. After solvent evaporation, the phenolic extract was dried using lyophilization (Christ Alpha 1-2 LD plus, Osterode am Harz, Germany) and stored at 4 °C.

2.3. Preparation of Microcapsules by Complex Coacervation and Freeze-Drying

The microcapsules containing basil EO and PE were prepared based on the methodologies outlined by Medeiros et al. [51] and Guazelli et al. [52], with adaptations. The essential oil was added directly while the PE extract was hydrated in deionized water. Biopolymer solutions were prepared by dispersing pectin and casein in distilled water, with a solid content of 10% w/v (5% casein and 5% pectin), and left to disperse fully at 4 °C overnight.

To create the microcapsules, the extracts were added to the casein solution (pH 8) at proportions of (1:10) and (5:10) substance-to-polymer for basil EO and PE, respectively. The mixture was homogenized at 362× g for 10 min using an Ultra-Turrax (OS40-IBX instrument, IKA-Werke GmbH & Co. KG, Staufen im Breisgau, Germany). The pectin solution was then slowly added while the mixture was agitated. Subsequently, the mixture was stirred continuously and the pH was gradually reduced to 3.5 using a solution of TCA (1.0 M).

To ensure successful complex coacervation and microcapsule precipitation, the mixture was left overnight at 4 °C. After filtration, the mixture was frozen at −25 °C in a Liebherr Comfort-NoFrost freezer (Ochsenhausen, Germany) before undergoing lyophilization for 48 h using the Christ Alpha 1-2 LD plus freeze dryer (0.1 mbar, −60 °C).

2.4. Determination of the Encapsulation Yield and Efficiency

The yield (Y%) of the freeze-dried microcapsules was determined using Equation (1) as described previously by Mebarki et al. [53], with modifications. Encapsulation efficiency was assessed by dispersing 500 mg of basil EO microcapsules (EOMC) and phenolic extract microcapsules (PEMC) in 2 mL of hexane and shaking the mixture for 10 min. After filtration (0.22 µm syringe filter), the amount of basil EO and PE on the microcapsule surfaces was quantified using a spectrophotometer (UV–visible, Optizan Pop, Daejeon, Republic of Korea) at 280 nm and 330 nm, respectively.

Alternatively, 1.5 mL of 2.0 M HCl was added to the microcapsules and stirred (161× g, 37 °C) for 5 min. Subsequently, 2 mL of ethanol was added, and the mixture was centrifuged at (2516× g, 25 °C) for 5 min using a Sigma centrifuge (Osterode, Germany). The amounts of basil EO and PE in the supernatant were determined spectroscopically at 280 nm and 330 nm, respectively, using calibration curves (EO: y = 8.7153x + 0.0132, R^2 = 0.99/PE: y = 0.5168x + 0.0307, R^2 = 0.9958). Encapsulation efficiency (EE %) was calculated using Equation (2) [53–55].

$$\mathrm{Y}(\%) = \frac{\text{Dried microcapsules (g)}}{\text{Initial amounnt of substance (g)} + \text{Polymers (g)}} * 100 \quad (1)$$

$$\mathrm{EE}(\%) = \frac{\text{Total amount of substance} - \text{Surface amount substance}}{\text{Total substance amount}} * 100 \quad (2)$$

2.5. Morphology and Size Characteristics

The morphology and size of the microcapsules were assessed through optical microscopy (Axiom 2000, OPTICA, Ponteranica, Italy) and scanning electron microscopy (Thermo Fisher Scientific (formerly FEI Company), FEI Quanta 250, Hillsboro, Oregon, USA), following the procedures outlined by Benammar et al. [56]. Additionally, the size distribution of the dried microcapsules was analyzed using specialized software.

2.6. Moisture Content

Moisture content in the freeze-dried microcapsules was performed according to the protocol outlined by Bakry et al. [57]. Basil EOMC and PEMC samples were weighed and placed in glass Petri dishes, then heated in an oven at 105 °C until a constant weight was achieved, indicating complete evaporation of moisture from the microcapsules.

2.7. Thermal Behavior, Crystallinity, and Structure of Microcapsules

The thermal properties of casein (CN), pectin (PN), EOMC, and PEMC were evaluated according to the procedure described by Chaib et al. [58]. Thermogravimetric analysis (TG-DSC) was performed using a SDT Q600 analyzer (TA Instruments, New Castle, Delaware, USA) under a nitrogen atmosphere (N_2). Each sample (20 mg) was heated from 25 °C to 650 °C at a rate of 10 °C/min. The microcapsules' crystalline structures and their phase compositions were examined using X-ray diffractometry with a panalytical-Empyrean X-ray diffractometer (Almelo, Netherlands). The instrument was equipped with a copper tube and utilized Cu-Kα radiation with a wavelength of 1.5418 Å at 45 kV and 40 mA. The analysis involved examining samples across a diffraction angular range of 5–70° with a step of $2\theta = 0.0262°$. The peak position (2θ), full width at half maximum (FWHM), crystallite size (nm), and lattice strain (%) of the samples were obtained using X-PertHighScore software (version 3.0e (3.0.5)) with the Scherrer equation (Equation (3)) [52]. Also, the degree of crystallinity (Xc) of the pectin/casein complex was calculated by Equation (4) [26].

$$D = \frac{K.\lambda}{\beta.cos\theta} \tag{3}$$

$$Xc = \frac{Area\ of\ crystalline\ peaks}{Area\ of\ all\ peaks\ (Crystalline + Amorphous)} \tag{4}$$

The infrared spectra were collected using an attenuated total reflectance (ATR) cell in an infrared spectrum (FTIR) instrument (JASCO Corporation, Jasco-ATR PRO 450-FT/IR4200, Tokyo, Japan) in a wavenumber range of 4000 to 400 cm^{-1} and a resolution of 1 cm^{-1} [59,60].

2.8. Statistical Analysis

All experiments were conducted in triplicate (n = 3), and the results are expressed as mean ± standard deviation (SD). Statistical analysis was performed using two-way ANOVA, followed by Sidak's multiple comparisons test to assess differences among groups. Graphs and statistical evaluations (assuming a normal distribution) were generated using Origin (version 2019b (9.6.5.169)) and GraphPad software (9.5.0 (730)). A p-value of less than 0.05 was considered statistically significant.

3. Results

3.1. Encapsulation Yield and Efficiency

Microencapsulation yield and efficiency are critical parameters in the encapsulation process, determining the success and cost-effectiveness of the technique. Yield refers to the proportion of core material successfully encapsulated within the microcapsules,

typically expressed as a percentage of the initial amount of the core material [61]. A high yield indicates efficient encapsulation, minimizing wastage and ensuring the maximum utilization of materials. On the other hand, efficiency encompasses various factors such as encapsulation rate, uniformity of capsule size, and stability of the encapsulated material. Achieving high efficiency implies not only a high yield but also consistent and reliable encapsulation performance, leading to desired product characteristics such as prolonged release profiles, improved stability, and enhanced bioavailability [62,63].

In this study, the microencapsulation of EO and PE yielded distinct results in terms of both yield and efficiency (Table 1). The yield of EO microencapsulation (EO-MC) was notably higher at 85.3% compared to PE microencapsulation (PE-MC) at 53.1%. Similarly, the efficiency of EO-MC was substantially higher at 89.8 ± 1.4% compared to 70.0 ± 1.01% for PE-MC.

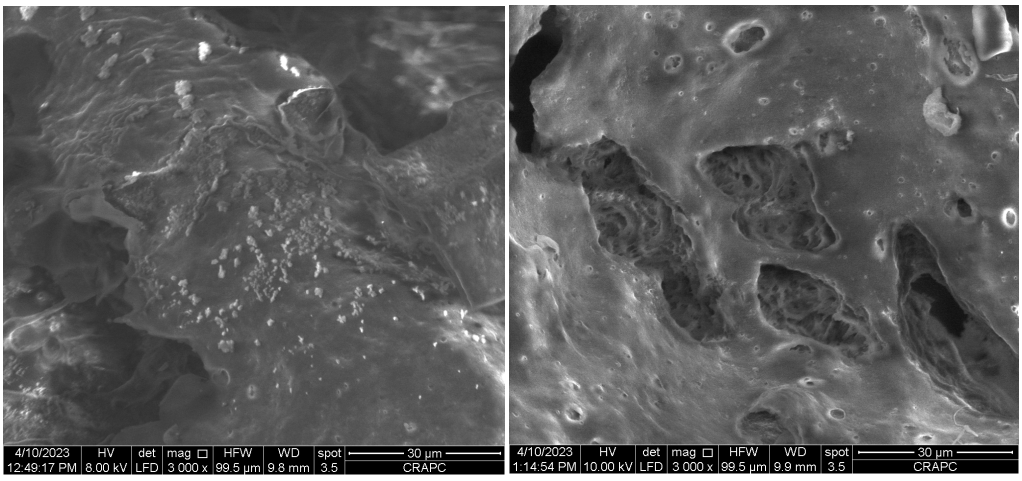
Table 1. Characteristics of EO and PE microcapsules.

Sample	Yield (%)	Efficiency (%)	Moisture (%)	Size (µm)
EO-MC	85.3 ± 1.8 ^{a}	89.8 ± 1.4 ^{a}	4.08 ± 0.47 ^{a}	9.70 ± 2.82 ^{a}
PE-MC	22.297 ± 0.5 ^{b}	70.0 ± 1.01 ^{b}	4.41 ± 0.25 ^{a}	6.95 ± 1.94 ^{a}

Values with different letters indicate statistically significant differences ($p < 0.05$).

3.2. Microcapsule Morphology and Particle Size Distribution

The microcapsules derived from both EO-MC and PE-MC exhibited a characteristic spherical shape, as evident from the results obtained through SEM (Figure 1a,b), indicative of the typical outcome of microcapsules produced via complex coacervation.



(**a**) (**b**)

Figure 1. Microcapsules observation under scanning electron microscopy: (**a**) EO-MC; (**b**) PE-MC.

The SEM analysis of basil PE-MC unveiled a notably porous surface texture (Figure 1b), contrasting with the smoother surface of EO-MC. These figures further illustrate the successful microencapsulation process utilizing casein and pectin as encapsulating agents through coacervation, with clear evidence of a protective layer enveloping the core, indicative of the presence of the extracts.

On the other hand, particle size holds significant importance, particularly concerning the utilization of food ingredients, as excessively large particles can yield an unpleasant mouthfeel. Conversely, there are cases where large particles are pursued, such as when aiming for visible particle presence in products. Furthermore, larger particles have the potential to facilitate an extended release of active compounds [61,64]. Both EO-MC and PE-MC displayed comparable sizes on average, with EO-MC showing a slightly larger

average size of 9.70 ± 2.82 µm (Table 1, Figure 2a). On the other hand, PE-MC exhibiting a slightly smaller average particle size of 6.95 ± 1.94 µm (Figure 2b).

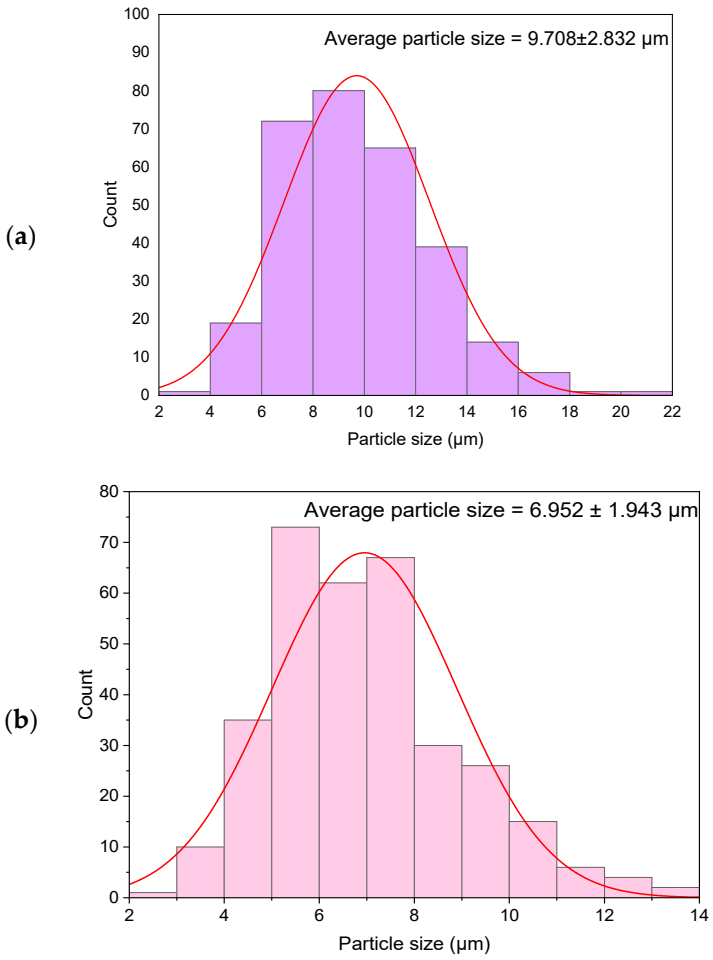


Figure 2. Particle size average and distribution of the microcapsules: (**a**) EO-MC; (**b**) PE-MC, the red line in both figures represents the normal distribution of particle size.

3.3. Moisture Determination Results

The moisture content of microcapsules obtained through complex coacervation was determined to be 4.08 ± 0.47% for EO-MC and 4.41 ± 0.25% for PE-MC (Table 1). These results align closely with the minimum moisture content specifications required in the food industry for dried powders, which typically range between 3% and 4% [65].

3.4. Thermal Behavior

The thermal behavior of wall materials (casein and pectin) and the obtained EO-MC and PE-MC were evaluated using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The TGA curves exhibited three distinct stages of thermal degradation, characterized by weight loss relative to temperature. The wall material curves were previously presented in Hamid et al. [55]. Briefly, two distinct thermal events were observed for pectin (50–100 °C and 200–250 °C), attributed to thermal salvation and degradation of pectin chains. The weight losses for these stages were 11% and 62%, respectively, with a total weight loss of 73%. Casein exhibited two thermal stages between 50–250 °C and 280–370 °C, resulting in a total weight loss of 76%, with maximum loss at 280 °C.

The thermal stability of the microcapsules EO-MC and PE-MC is illustrated in Figure 3a,b. EO-MC presented two stages of weight loss with a total weight loss of 69.68%.

The first stage occurred from 20 °C to 135 °C, with a weight loss percentage of 4.24%, and the second from 125 °C to 650 °C with 65.62%. The PE-MC showed similar behavior, with two major stages of weight loss: 8.97% from 25 °C to 190 °C and 70.17% from 200 °C to 650 °C, with a total weight loss of 79.14%.

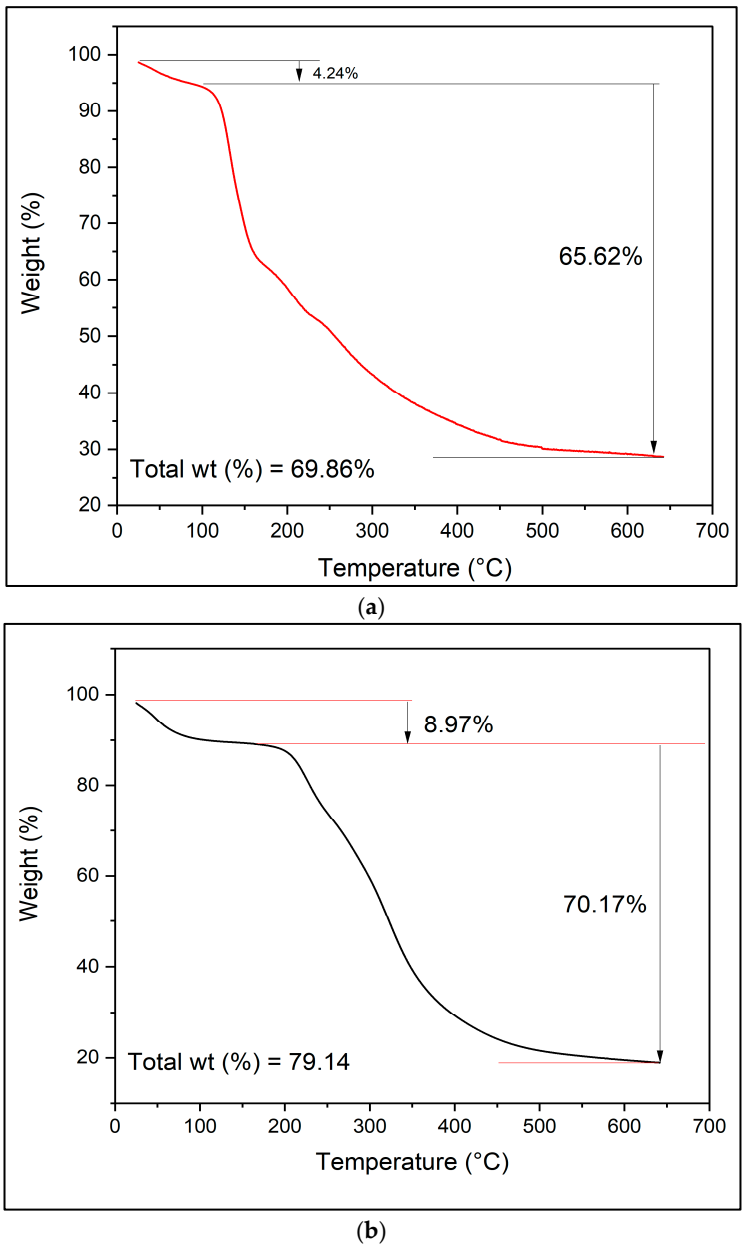


Figure 3. Thermogravimetric analysis (TGA) of EO-MC (**a**) and PE-MC (**b**) (for interpretation of the references to color in this legend, the reader is referred to the web version of this article).

3.5. Crystallinity (X-ray Diffraction)

The crystalline behavior and phase composition of the pectin/casein complex, basil essential oil microcapsules (EO-MC), and phenolic extract microcapsules (PE-MC) were investigated through X-ray diffraction (XRD) analysis.

Figure 4a,b illustrates the XRD patterns, while Table 2 summarizes the crystallographic parameters for these materials. In Figure 4a, the XRD pattern of the pectin/casein complex reveals two broad peaks spanning the range of 9.9° to 19.9°, with an additional small peak observed at $2\theta = 21.75°$. These features are indicative of the presence of amorphous structures, attributed to the casein component [66]. The XRD pattern of EO-MC exhibits three

new peaks at 31.8°, 45.5°, and 56.6°, corresponding to characteristic peaks of pectin [67], suggesting the presence of pectin crystals, likely formed due to essential oil-induced recrystallization. Additionally, the peak at 21.75° shifts to 22.2° and becomes more intense, indicating changes in casein's crystallinity within the polymer complex.

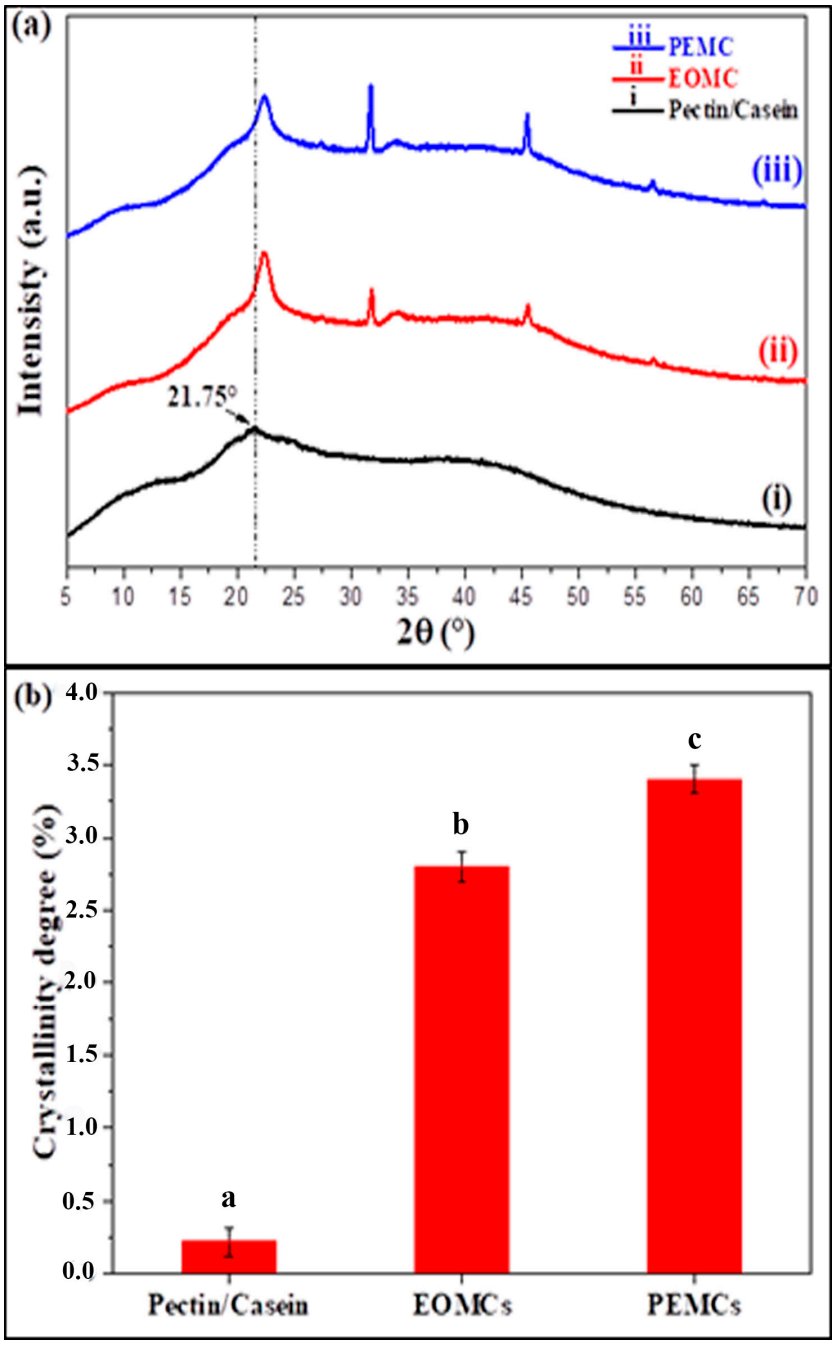


Figure 4. XRD patterns (**a**) of pectin/casein, EO-MC, and PE-MC. Effect of different microcapsule formulations on the degree of crystallinity of the pectin/casein complex (**b**); values with different letters indicate statistically significant differences ($p < 0.05$).

Table 2 shows the decreased full-width at half maximum (FWHM), increased crystallite size, and reduced lattice strain for this casein peak, supporting enhanced crystallinity in the pectin/casein complex due to the microcapsule formulation.

Table 2. Crystallographic parameters of samples obtained by XRD pattern.

Sample	2θ (°)	FWHM (°)	Crystallite Size (nm)	Strain (%)
Pectin + Casein	21.747	0.461	14.6	1.047
EO-MC	22.204	0.358	23.1	0.796
PE-MC	22.297	0.614	13.4	1.359

FWHM: full width at half maximum.

The XRD pattern of basil PE-MC shows an increase in the intensity of characteristic pectin peaks, suggesting the enhanced crystallinity of pectin chains, likely due to physical interactions between the phenolic substances and the pectin. However, there is a slight decrease in the intensity of the casein characteristic peak, potentially caused by reduced pectin/casein interface interactions as a result of increased pectin crystallinity promoted by the phenolic molecules [68]. This is supported by an increase in the value of FWHM and the lattice strain, along with a decrease in crystallite size, corresponding to the characteristic peak of casein (as indicated in Table 1).

Figure 4b illustrates the impact of various microcapsule formulations on the degree of crystallinity of the pectin/casein complex, with values marked by different letters indicating statistically significant differences ($p < 0.05$). The data reveal that the unmodified pectin/casein complex exhibits a low crystallinity, characteristic of an amorphous structure, while the introduction of specific microcapsule formulations, such as EOMCs and PEMCs, substantially increases the degree of crystallinity. This enhancement suggests that the encapsulation process induces a more ordered molecular arrangement within the biopolymer matrix, likely due to interactions between the encapsulated components and the pectin/casein network. Notably, the formulation differences between EO-MCs and PE-MCs further imply that subtle variations in the encapsulation protocol or the nature of the additives can significantly influence the structural ordering of the complex, which may, in turn, affect the stability and release properties of the encapsulated bioactive compounds. This observation underscores the pivotal role of these bioactive compounds and their synergistic interactions in modulating the crystalline architecture of the complex [69]. Notably, such structural modifications are anticipated to influence the controlled release profile of the encapsulated drug. Enhanced crystallinity reinforces the structural integrity of the pectin/casein matrix, thereby reducing molecular diffusion, impeding the release kinetics of the active ingredient, and improving the overall stability of the microcapsules [70].

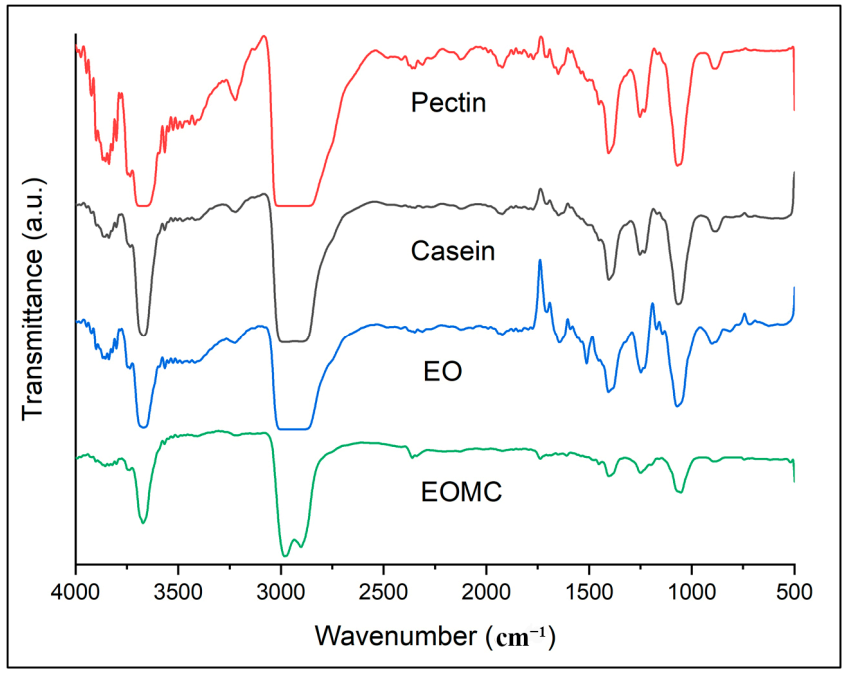
3.6. Infrared Spectroscopy

Figure 5a,b illustrates the FTIR spectrum of microencapsulated samples (EO-MC and PE-MC) alongside a comparison of the wave numbers from the FTIR spectra corresponding to the basil compounds and wall biopolymers.

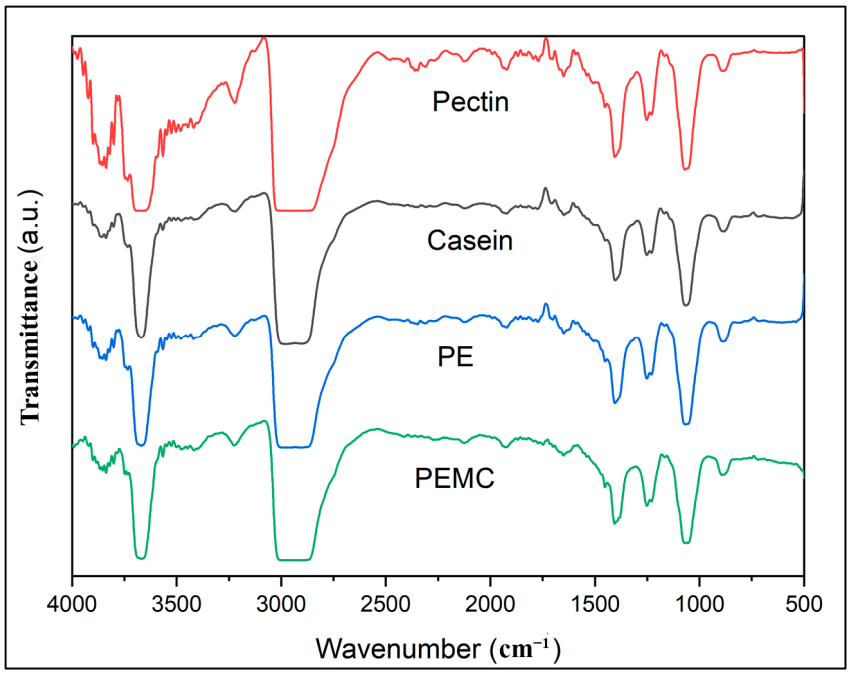
The FTIR analysis validated the successful encapsulation process by detecting the presence of basil EO and phenolic extract in the casein/pectin microcapsules. This analysis revealed similarities among the samples, with variations observed in peak width and intensities, likely due to differences in the type and quantities of compounds used to formulate the microcapsules. The basil EO displayed distinctive peaks, revealing its chemical composition. These included broad peaks in the ranges of 3000–2800 cm^{-1} and 3750–3500 cm^{-1}, indicating C–H stretching vibrations and O–H/N–H vibrations, respectively [58]. A strong C–O stretching vibration band was observed between 1200 and 1100 cm^{-1}.

Additionally, peaks from 1600 to 1000 cm^{-1} confirmed the oil's aromatic nature, with C=C stretching of a benzene ring, O–H bending, and C–O stretching of an aromatic group [69]. In the case of basil PE, bands identified within the wavenumber range of 1800 to

750 cm^{-1} are associated with polyphenols found in plants, such as C–OH, C–O–C, and C–C stretching vibrations [71]. The absorption bands of the aromatic ring stretching vibrations of C–H were observed at 2900 cm^{-1} [72]. The band detected between 3700 and 3300 cm^{-1} is attributed to hydrogen bonding and the stretching of the O–H bond in carbohydrates and carboxylic acids [32,73]. The functional groups of the wall biopolymers (casein and pectin) were previously detailed in our study [55]. FT-IR analysis confirmed the generation of amide bonds due to the interaction between the free carboxyl groups of pectin and the amino groups of casein in the complex.



(**a**)



(**b**)

Figure 5. FTIR spectra of pectin and casein, basil essential oil and phenolic extract, and the obtained microcapsules (**a**) EO and (**b**) PE.

4. Discussion

Basil extracts exhibited significant biological activity, attributed to their rich composition of phenolic and volatile compounds. The basil phenolic extract (PE) demonstrated potent antioxidant properties, while the basil essential oil (EO) exhibited remarkable antimicrobial efficacy. These findings highlight the promising bioactivity of basil extracts, suggesting their potential applications in health and culinary contexts (Erez et al., 2024 [1], Chu 2022 [6], Pedrosa et al., 2021 [49], Moussa et al., 2024 [74], Darrag et al., 2022 [75], Fernandes et al. [76], Fayoumi et al. [77], and Rezzoug et al. [78]. This underscores the utility of basil extracts, not only as functional ingredients in food but also in therapeutic applications, further supporting the relevance of encapsulation techniques to enhance their stability and bioactivity.

In terms of microencapsulation, the results indicated that the encapsulation of the essential oil (EO) was more efficient than the phenolic extract (PE). The higher yield and efficiency of EO microcapsules (EO-MC) suggest a more successful encapsulation process, likely due to the inherent chemical properties and solubility of essential oils compared to phenolic compounds. Essential oils are typically composed of smaller, volatile compounds with low molecular weights, which make them more readily encapsulated in contrast to larger, more complex phenolic compounds. Furthermore, the hydrophobic nature of essential oils facilitates their encapsulation in hydrophobic matrices such as those formed by pectin and casein in complex coacervation [27]. In line with this, García-Saldaña et al. [79] encapsulated broccoli seed extract rich in sulforaphane using complex coacervation with two biopolymer combinations (gelatin/pectin and gelatin/gum Arabic), reporting higher yield values of 81% and 85%, respectively. However, their encapsulation efficiency was lower than that observed in this study, with values of 18% and 12%, respectively. Similarly, De Souza et al. [61] achieved encapsulation yield and efficiency values close to those of our study, with values of 65% and 70%, respectively, when encapsulating cinnamon bark extract in gelatin and κ-carrageenan. In contrast, Chaib et al. [58] encapsulated *Thymus vulgaris* essential oil in gelatin B and gum Arabic, reporting an efficiency of 98%, which aligns closely with our results, although the yield (66%) was slightly lower than ours.

These differences are consistent with the typical outcomes observed in microencapsulation studies using complex coacervation. The porous surface observed on the PE-MC microcapsules, in contrast to the smoother surface of EO-MC, can be explained by the physical and chemical characteristics of the encapsulated compounds. Phenolic compounds, being larger and more complex than compounds present in essential oils, may influence the formation and structure of the microcapsules during coacervation. The uneven distribution or aggregation of phenolic compounds within the microcapsule core likely contributes to the creation of void spaces or pores on the surface during drying. Additionally, the solubility and interaction differences between the encapsulating biopolymers (casein and pectin) and the core compounds (essential oil and phenolic extract) may lead to variations in the surface morphology. Essential oils, composed of smaller and more homogeneous molecules, may result in a smoother surface due to more uniform interactions with the encapsulating agents, whereas phenolic extracts, with their larger and more heterogeneous composition, tend to create a more porous surface. This observation is supported by previous studies that have examined the morphology of microcapsules encapsulating essential oils and phenolic extracts from various plant sources [80–84].

Notably, the particle sizes observed in this study were considerably smaller compared to those reported in the literature. For example, Silva et al. [85] encapsulated green tea phenolic extract using cashew gum/maltodextrin and reported an average particle size of 10 μm. Campini et al. [81] observed microcapsules of *Cinnamomum cassia* essential oil encapsulated in polylactic acid with an average particle size of 80 μm. In comparison,

our study achieved smaller microcapsules, especially for essential oils, demonstrating the effectiveness of the current encapsulation technique and the biopolymer combination used. This result is also consistent with our previous study on the microencapsulation of *Citrus limon* EO, which yielded spherical-shaped microcapsules with an average size of 25 µm [37], highlighting the suitability of the chosen biopolymers for achieving smaller particle sizes.

The particle size of microcapsules plays a crucial role in determining their functionality and application potential. In this study, the microcapsules exhibited a particle size of less than 10 µm, which, while beneficial for enhancing surface area, solubility, and bioavailability, may also lead to increased interparticle interactions that could affect their flowability and dispersibility. To address these potential challenges, various strategies can be employed, including the incorporation of anti-caking agents, surface modifications, or controlled agglomeration during processing. Moreover, the specific application of these microcapsules will dictate whether their small size presents a limitation or an advantage. In pharmaceutical and food formulations, for example, finer particles can facilitate improved dissolution rates and controlled release properties, making them highly desirable. Therefore, despite potential concerns regarding interparticle interactions, the reduced particle size of the microcapsules remains advantageous for their intended applications.

Moisture content is a critical factor influencing the stability and shelf life of microcapsules. Complex coacervation generally results in microcapsules with lower moisture content due to water evaporation during the encapsulation process. In our study, moisture content was relatively low in both EO-MC and PE-MC microcapsules. This is in contrast to the findings of Silva et al. [86], who reported a moisture content of 5.96% in microcapsules of lycopene encapsulated in gelatin/pectin. Vonghirundecha et al. [87] reported a moisture content ranging from 5.10% to 6.65% for Moringa oleifera phenolic extract encapsulated in maltodextrin, which was notably higher than our values for PE-MC. Conversely, Wang et al. [88] observed a moisture content of 3.70% in microcapsules of tuna oil encapsulated in gelatin/sodium hexametaphosphate. Low moisture content in microcapsules is particularly beneficial in food applications, as it helps prevent microbial growth and reduces biochemical reactions, thereby enhancing the shelf life of products [28].

The thermogravimetric analysis (TGA) of microcapsules revealed distinct thermal events corresponding to various stages of degradation. The first stage of weight loss is attributed to the evaporation of residual water and the release of volatile compounds that are not encapsulated. This initial loss reflects heat absorption and is consistent with typical moisture loss or desorption phenomena observed at lower temperatures [51]. In the second stage, both the encapsulated compounds and the wall materials undergo thermal degradation, involving the breakdown of proteins and polysaccharides. This is indicative of the thermal decomposition of the biopolymers that make up the wall materials, leading to significant mass loss. This observation is consistent with the findings reported in the literature, such as those of Radünz et al. [69] and Tavares and Zapata Noreña [89], who describe similar degradation processes for proteins and polysaccharides under elevated temperatures. The thermal degradation patterns observed in this study align with previous research on the microencapsulation of volatile and phenolic compounds, which has demonstrated that encapsulation enhances the thermal stability of bioactive substances [51,90–92]. The microcapsules' ability to protect the encapsulated compounds from degradation at high temperatures supports the notion that encapsulation is an effective strategy for improving the thermal stability of sensitive bioactive agents.

An X-ray diffraction (XRD) analysis of the microcapsules revealed important crystallographic changes. The broad peaks observed in the pectin/casein complex suggest an amorphous structure, likely due to the high casein ratio and the preparation method, which inhibits crystallization [53,66]. The appearance of new peaks in the EO-MC pattern, partic-

ularly at 31.8°, 45.5°, and 56.6°, suggests pectin crystallization induced by the essential oil. The shift and increased intensity of the peak at 21.75° further indicate changes in casein's crystallinity, reflecting the impact of the encapsulation process on the polymer complex. The enhanced crystallinity of pectin in the PE-MC formulation is likely a result of interactions between phenolic substances and pectin, promoting a more ordered structure [53]. However, the decreased intensity of the casein peak in the PE-MC pattern may be due to reduced interactions between pectin and casein, caused by the increased crystallinity of pectin [68]. These crystallographic changes are crucial because they affect the microcapsules' structural integrity, potentially influencing the release profiles of the active ingredients. Enhanced crystallinity in the polymer matrix may provide a better barrier effect, slowing down the release and contributing to the stability of the microcapsules [93].

Finally, the results of FTIR spectroscopy confirmed the successful encapsulation of basil essential oil (EO) and phenolic extract (PE) within the pectin/casein microcapsules. The characteristic peaks corresponding to the molecular structures of the essential oil and phenolic compounds were observed in the spectra of the microcapsules, confirming their presence. Specifically, the basil EO exhibited distinctive peaks in the 3000–2800 cm^{-1} and 3750–3500 cm^{-1} ranges, attributed to C–H stretching vibrations and O–H/N–H vibrations, respectively. The strong C–O stretching vibration band was detected between 1200 and 1100 cm^{-1}, while peaks from 1600 to 1000 cm^{-1} confirmed the oil's aromatic nature. Similarly, the presence of phenolic compounds in the microcapsules was confirmed by absorption bands in the 1800–750 cm^{-1} range, corresponding to C–OH, C–O–C, and C–C stretching vibrations. Furthermore, modifications in the spectra of the microcapsules, particularly the reduction or shifting of peaks associated with the functional groups of pectin and casein, suggest molecular interactions between the encapsulated compounds and the biopolymer matrix. The disappearance or shift of peaks in the regions associated with pectin's free carboxyl groups (-COOH, ~1730 cm^{-1}) and casein's amine groups (-NH_2, ~1550 cm^{-1}) indicates the formation of amide bonds, confirming electrostatic interactions between pectin and casein in the coacervation process. Additionally, the hydrogen bonding interactions between phenolic compounds and the biopolymer network are supported by changes in the 3700–3300 cm^{-1} region, associated with O–H stretching vibrations. These results are consistent with previous studies that have demonstrated successful encapsulation processes for volatile and phenolic compounds [78,94]. The successful encapsulation process and the chemical stability of the bioactive compounds further support the potential applications of these microcapsules in pharmacology and nutraceuticals [88,94].

5. Conclusions

This study effectively evaluated the complex coacervation method using a combination of biopolymers (casein and pectin) for the microencapsulation of bioactive compounds derived from *O. basilicum* L. The resulting microparticles of basil essential oil and phenolic extract exhibited promising characteristics. EO-MC demonstrated superior microencapsulation yield (85.3%) and efficiency (89.8%) compared to PE-MC (53.1% and 70.0%) for yield and efficiency, respectively, while both types displayed spherical morphology with PE-MC exhibiting a notably porous surface texture. An analysis revealed smaller particle sizes for PE-MC (6.95 ± 1.94 μm) compared to EO-MC (9.70 ± 2.82 μm). The moisture content aligned with the food industry standards for dried powders with values less than 5% for both EO-MC and PE-MC. Thermogravimetric and differential scanning calorimetry analysis indicated two stages of weight loss for both EO-MC and PE-MC, reflecting the evaporation of residual water and volatile compounds followed by the decomposition of microencapsulated compounds and wall materials. X-ray diffraction analysis showed enhanced crystallinity in EO-MC due to essential oil-induced re-crystallization and increased

pectin crystallinity in PE-MC attributed to interactions with phenolic substances, impacting drug release and microcapsule stability. The results of FTIR spectroscopy confirmed molecular interactions and the formation of new chemical bonds between encapsulated compounds and wall materials. Specifically, hydrogen bonds are likely formed between phenolic compounds and the biopolymer matrix, and amide bonds are generated through interactions between the carboxyl groups of pectin and the amino groups of casein, facilitating the encapsulation of the bioactive compounds. Overall, these findings underscore the potential of the complex coacervation method using these biopolymers for the effective microencapsulation of bioactive compounds from plant sources, especially essential oils, with implications for various applications in food, nutraceuticals, and pharmaceuticals.

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Abbreviations

The following abbreviations are used in this manuscript:

XRD	X-ray diffraction
TGA	Thermogravimetric analysis
FTIR	Fourier transform infrared
EO	Basil essential oil
PE	Phenolic extract
PE-MC	PE microcapsules
EO-MC	EO microcapsules

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