ENCAPSULATION NATURAL OILS PLANT BASED USING FREEZE-DRYING

Alyanis Mufid SWM[1*], Soraya Ratnawulan Mita[2], Patihul Husni[3]

[1-3]Padjajaran University

Email Korespondensi: alyanis21001@mail.unpad.ac.id

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ABSTRACT

Natural oils, such as essential oils and oils with high unsaturated fatty acids, can be derived and extracted from plant sources. Essential oils contain aromatic compounds commonly used in the food industry, while oils rich in unsaturated fatty acids, such as omega-3, -6, and -9, along with vitamin E, are highly beneficial in industrial sectors. Furthermore, natural oils possess antimicrobial and antioxidant properties that are valuable in daily life. However, degradation of natural oils is inevitable due to various external factors such as temperature, humidity, and light. Therefore, an effective technique is required to enhance the stability of natural oils, with one of the methods being freeze drying, using wall materials made from various polymers depending on the intended application. This review aims to summarize the development of natural oils encapsulated by freeze dryingThe literature search in this review was conducted using the search engines Google Scholar and ScienceDirect. The selected papers used for this review consist of original research articles published between 2021 and 2025, focusing on natural oils unsaturated fatty acids or essential oils as the primary subject of study. Stability can be improved by examining thermal analysis parameters in various natural oils, such as walnut, sacha inchi, rose damascena, mandarin, and boswellia sacra. Storage at low temperatures helps enhance the chemical stability of natural oils. In natural oils, both essential oils and oils with high unsaturated fatty acids, stability improvement can be achieved through freeze drying using polymers as wall materials such as polysaccharides, proteins, gums, lignin, or other polymers. The use of proper machine freeze drying parameter settings is also necessary to obtain the best results with high encapsulation efficiency

Keywords: Encapsulation, Essential Oils, Freeze Drying, Natural Product, Unsaturated fatty Acids.

INTRODUCTION

Essential oils (EOs) are volatile and aromatic substances that are naturally extracted from medicinal and aromatic plants (Grădinaru et al., 2018; Ni et al., 2021). These hydrophobic oils are abundant in bioactive compounds and are known for their potent antimicrobial properties . EOs have found broad applications in various fields, including food and non-food additives, flavorings, cosmetics, as well as medicinal and pharmaceutical products (Ni et al., 2021). Essential oils consist of intricate blends of volatile and semi-volatile compounds derived from various plant parts, including fruits, flowers, roots, seeds, leaves, and stems. They are characterized by their potent aroma, show good solubility in organic solvents, and are typically immiscible with water. Commonly employed as flavoring agents, essential oils also exhibit a wide range of biological activities, such as antimicrobial, antioxidant, and insect-repellent effects (de Sousa et al., 2023).

In addition to the presence of essential oils, which are known for their beneficial properties, there are other active components that are widely utilized in various industries due to their equally important functions—namely, natural oils/unsaturated fatty acids (UFAs). These compounds are found in certain plants that are rich in omega-3, omega-6, and omega-9 fatty acids, as well as vitamins such as A and E, depending on the source of the UFA. However, similar to essential oils, UFAs also exhibit limitations such as instability and susceptibility to degradation (Rodríguez-Cortina et al., 2022).

Nevertheless, when exposed to factors like light, oxygen, and elevated temperatures, these oils become highly prone to oxidation, chemical degradation, and polymerization (Turek & Stintzing, 2013), also the practical use of essential oils is often restricted by their high volatility, poor water solubility, chemical reactivity, instability, and tendency to oxidize easily (Shetta et al., 2019; Turek & Stintzing, 2013). the application of essential oils (EOs) is challenged by various limitations, including the loss of active constituents due to evaporation and their high vulnerability to oxidative degradation during storage or upon exposure to light, heat, air, and humidity (Turek & Stintzing, 2013). Given their physicochemical characteristics—such as their lipophilic nature, low or no solubility in water, volatility, strong aroma, and concentration-dependent activity—as well as their biological effects and potential health impacts, encapsulating or entrapping natural oils (Eos and UFA) becomes a good step. In this context, incorporating natural oils into polymer matrixes (Dajic Stevanovic et al., 2020) has shown great potential to: (i) protect them from oxidation and evaporation, (ii) improve their stability and water dispersibility, (iii) extend their shelf-life, (iv) enhance their bioavailability and therapeutic effectiveness, and (v) allow for controlled or sustained release. Such alterations can diminish their aromatic quality and significantly reduce their biological efficacy.

In studies involving natural oils, both essential oils and unsaturated fatty oils have been shown to possess high susceptibility to oxidation, as evidenced in several conducted investigations (Aranda Saldaña et al., n.d.; Avendaño et al., 2024; Fernandes et al., 2014; Guerrero et al., 2022; Tonon et al., 2011; Wang et al., 2021). research has shown that encapsulating sacha inchi oil through the freeze-drying technique enhances its energy-related properties, as reflected in the thermal analysis results, when compared to samples without treatment. This improvement indicates a strong binding affinity between the wall materials and the encapsulated sacha inchi oil. Comparable thermal behavior has also been observed in encapsulated sunflower and walnut oils, which displayed similar thermal analysis outcomes (Aranda Saldaña et al., n.d.; Erdem & Kaya, 2021; Rodríguez-Cortina et al., 2022; Vicente et al., 2017; Wang et al., 2024a). Likewise, essential oils exhibit parallel trends; for example, *Rose damascena* essential oil, when encapsulated, revealed higher energy characteristics and superior thermal stability than its unencapsulated counterpart. This pattern has also been reported in studies involving other essential oils, including *Boswellia sacra*, which demonstrated extended shelf life and enhanced thermal performance following encapsulation (Alabrahim et al., 2024; Khodadadi et al., 2024). To address these stability issues, encapsulation has emerged as a promising strategy to protect natural oils and preserve their functional properties (Shetta et al., 2019).

LITERATURE RIVEW

Encapsulation technology enables essential oils to be incorporated within a protective matrix, enhancing their water dispersibility, stability, and enabling controlled release. Various types of carriers—such as emulsions, liposomes, solid lipid particles, and biopolymeric systems—have been utilized for encapsulating essential oils. The selection of appropriate wall materials and encapsulation techniques is critical, as they directly affect the encapsulation efficiency, overall yield, and stability of the final product (Muhoza & Uriho, 2025). Each encapsulation system presents its own set of benefits and limitations. Among these, biopolymers are favored due to their natural origin, environmental degradability, and compatibility with biological systems. Encapsulation using coating materials has been suggested as a strategy to enhance the stability of natural oils unsaturated fatty acid (UFA), minimize undesirable flavors, delay lipid oxidation and enzymatic hydrolysis, and enable controlled release of the encapsulated components (Rubio-Rodríguez et al., 2010). This mechanism of controlled release may lead to improved bioavailability of food compounds and more efficient nutrient absorption in the body (Jafari, 2017).

The encapsulation process typically begins with the formation of an emulsion, where lipid-based droplets are dispersed within an aqueous continuous phase (Rubio-Rodríguez et al., 2010). Food-grade emulsions created through various technologies—such as high-speed blending, mixing, high-pressure homogenization, microfluidization, and ultrasound—have demonstrated strong resistance to destabilization (Ratti, 2012; Stratta et al., 2020). However, employing some of these techniques at an industrial scale can be costly and energy-intensive (Camacho et al., 2018; Valková et al., 2022). Among these, ultrasound-assisted emulsification is emerging as an efficient, sustainable, and eco-friendly alternative to conventional high-energy methods (Carpenter et al., 2018; Jafari, 2017; McClements, 2004; L. Zhou et al., 2021).

Spray drying and freeze drying are among the most widely used methods for drying essential oils encapsulated in different carrier systems. While spray drying offers advantages such as scalability and rapid processing, the use of high-temperature inlet air for moisture removal can degrade thermolabile essential oil components, including monoterpene hydrocarbons, oxygenated monoterpenes, sesquiterpene hydrocarbons, oxygenated sesquiterpenes, and phenolic compounds (Rezvankhah et al., 2020). Elevated temperatures can trigger free radical formation, which in turn promotes autoxidation and breakdown of hydroperoxides (Turek & Stintzing, 2013). In contrast, freeze drying operates under low-temperature conditions that can effectively preserve essential oil integrity by slowing or halting degradation pathways. Additionally, freeze drying results in powders with excellent rehydration capacity and has been shown to achieve high yields and effective retention of volatile compounds like eugenol and eugenyl acetate when encapsulated in carriers such as maltose, maltodextrin, and gum Arabic (Cortés-Rojas et al., 2014). Given the thermal sensitivity of essential oils and unsaturated fatty acids, freeze drying emerges as a preferred method due to its gentle processing conditions. Therefore, the development of robust essential oil delivery systems is crucial, with formulations designed to form thick interfacial membranes that can safeguard the oils during the drying process. Technologies such as nanoparticles, microcapsules, and biopolymeric matrices—particularly those based on proteins and polysaccharides—are commonly employed to enhance the retention and stability of encapsulated essential oils (Kumar et al., 2025)

Natural oils possess a wide range of beneficial properties, making them attractive for use across various industries such as food, beverages, cosmetics, and pharmaceuticals. However, their application is limited due to their chemical instability when exposed to factors like oxygen, light, and elevated temperatures. Several studies have demonstrated the degradation of key compounds such as linolenic acid, linoleic acid, oleic acid, limonene, citral, beta-ionone, and linalool during storage, with significant losses observed over an 8-week period. Similarly, other volatile components like ß-pinene and p-cymene have been shown to degrade over time at room temperature. To address these stability issues, encapsulation has emerged as a promising strategy. The use of biopolymers as encapsulating agents is particularly appealing due to their low cost, biodegradability, and compatibility with biological systems. For example, encapsulating citral essential oil within an emulsion stabilized by whey protein and gum Arabic significantly enhanced its resistance to oxidation and thermal degradation. Modifying interfacial properties using biopolymers has also been associated with better protection against chemical degradation of essential oils (Djordjevic et al., 2008; Erdem & Kaya, 2021; Wang et al., 2024a; Z. Zhang et al., 2024).

Various carriers, including polysaccharides (e.g., starch, alginate, gums, cyclodextrins, chitosan), proteins (e.g., gelatin, milk and plant proteins), and natural fibers, either individually or in combination, have been used to encapsulate essential oils. Given the diverse compositions, structures, and surface characteristics of these biopolymers, their interactions with essential oils differ, which in turn influences encapsulation efficiency, retention capacity, and overall storage stability (Silva-Espinoza et al., 2020).

Polysaccharides, composed of glycosidically linked monosaccharides, are particularly valued for their hydrophilic nature and resilience under varying environmental conditions such as changes in pH, ionic strength, and temperature. Among them, gums are notably hydrophilic and, due to their molecular properties, have facilitated the development of advanced delivery systems such as nanoemulsions, nanoparticles, and nanocomplexes for encapsulating essential oils and other bioactive compounds (Taheri & Jafari, 2019).

This review seeks to summarize and find out the current advancements in the freeze-drying to influence of natural oils encapsulated within polymer-based carriers. It further examines the influence of wall materials, encapsulation techniques, and freeze-drying parameters on the characteristics and stability of essential oils and unsaturated fatty acids embedded in polymeric systems.

RESEARCH METHODOLOGY

The literature search in this review was conducted using the search engines Google Scholar and ScienceDirect. The selected papers used for this review consist of original research articles published between 2021 and 2025, focusing on natural oils unsaturated fatty acids or essential oils as the primary subject of study. The encapsulation method employed in these studies involved freeze-drying, and each article included discussions and data related to the characteristics of the resulting encapsulated products.

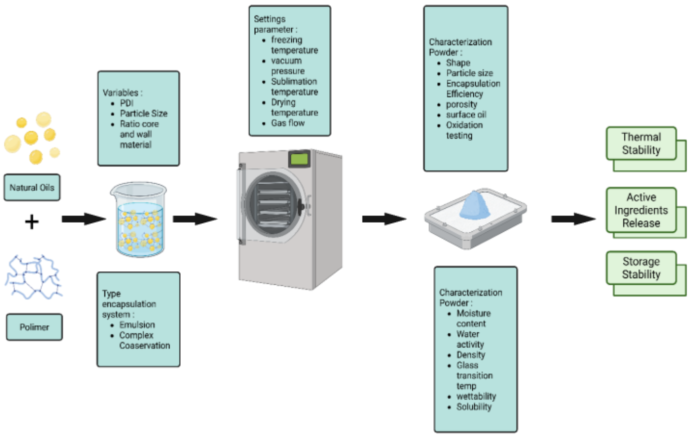

Figure 1. Representation Processing Freeze Drying Natural Oils (Created In Biorender, Https://Www.Biorender.Com/ , Accessed On 24 June 2025)

Table 1. Characteristic and Stability Essential Oil with Unsaturated Fatty Acids Encapsulated

Object	Wall Material	Freeze-drying Condition	Encapsulation Efficiency (%)	Thermal Analysis	Particle Size	Storage Temperature/ Time	Storage Stability	Reference
Sacha Inchi Oil	Sodium caseinat + maltodextrin	-40°C	60	200-350°C 350°C - 490°C	Not provided	Not provided	Not provided	(Rodríguez-Cortina et al., 2022)

Walnut Oil	Sodium caseinat + maltodextrin	−15° C	76.43 ± 1.03	175-350° C, 375-500° C	415.18 ± 29.97 μm	Not provided	Not provided	(Wang et al., 2024b)
Canola Oil	Alginate + maltodextrin	−40° C	Not provided	Not provided	496-734μm	Not provided	Not provided	(Guerrero et al., 2022)
Sunflower Oil	Soy protein isolate	-55°C	Not provided	50.49°C, 58°C	100.58 ± 5.03 - 152.09 ± 7.60 μm	Not provided	Not provided	(Erdem & Kaya, 2022)
Fennel Essential Oil	Porous starch, sodium alginate and chitosan	-70°C	70	Not provided	171 μm	Not provided	Not provided	(Sun et al., 2021)
Rose Damascene Essential Oil	Lignin	−15° C	73-99	100-200° C, 300-400° C	311 nm	25°C / 21 days	≥80%	(Khodadadi et al., 2024)
Mandarin Essential Oil	Whey, gum Arabic and maltodextrin	−40° C	92	96°C, 271° C, 363° C	728 nm	50°C / 30 days	22.8 %	(Mahdi et al., 2021)
Boswelia Sacra Essential Oil	hydroxypropyl-beta-cyclodextrins	−20° C	96	113° C, 343° C	448.6 ± 0.6244 nm	−4°C / 540 days	≥ 90%	(Alabrahim et al., 2024)

RESULTS AND DISCUSSION

Encapsulation refers to a method used to protect sensitive bioactive compounds—such as essential oils—by enclosing them within a protective wall material. This approach enhances the compounds' resistance to environmental stressors like heat, light, and oxygen, while also enabling controlled or prolonged release under varying conditions. Numerous encapsulation strategies are available, including emulsification, coacervation, molecular inclusion, the formation of nano- and microparticles, freeze drying, spray drying, melt extrusion, and fluidized bed coating. While a wide range of carriers can be employed for essential oils, this review specifically emphasizes systems such as emulsification, coacervation, molecular inclusion, and nano/microparticles in combination with freeze-drying techniques (Delshadi et al., 2020).

Encapsulating natural product oils, is an essential strategy to improve their stability, water solubility, and biological effectiveness, thereby broadening their potential uses in multiple sectors. Although essential oils exhibit a wide range of valuable activities—including antibacterial, antifungal, antiviral, and antioxidant effects—their high volatility and susceptibility to degradation limit their direct application. Encapsulation technologies address these limitations by shielding the oils from environmental stressors and enabling controlled release, which helps preserve their functional properties and prolong shelf life. The subsequent sections will discuss various encapsulation methods and their respective applications (Delshadi et al., 2020; Kumar et al., 2025; Ratti, 2012; Valková et al., 2022).

Utilization of encapsulated oils for some sectors, they are: food sector, encapsulated essential oils (EOs) are integrated into active food packaging systems to extend product shelf life and preserve food quality. Advanced nanocarriers, including liposomes and solid lipid nanoparticles, are employed to enhance EO stability and enable controlled release within food matrices (Ben-Fadhel et al., 2024). Second one is pharmaceutical and medical applications, incorporating EOs into carriers like zeolites and nanostructured lipid systems improves their therapeutic potential by enhancing both stability and targeted delivery. This has proven effective in areas such as aromatherapy and topical formulations for treating bacterial infections (Ferreira et al., 2022), also micro- and nano-encapsulation techniques allow for the incorporation of plant-derived essential oils into food products, thereby enriching their nutritional value and boosting functionalities like antimicrobial and antioxidant effects (Delshadi et al., 2020).

Lyophilization, commonly referred to as freeze drying, is a dehydration technique highly effective in preserving the quality of natural oils. The process entails freezing the material followed by reducing the ambient pressure, enabling the frozen moisture to transition directly from solid to vapor through sublimation. This technique is extensively applied due to its capability to retain the product’s structural features, nutritional value, and bioactive constituents, rendering it a suitable method for handling natural product oils. Although freeze drying requires substantial energy input, it offers notable benefits in terms of maintaining product stability and overall quality (Yao et al., 2023). Process freeze drying natural oils representation it can be seen in Figure 1.

Benefits of freeze drying for natural product oils, they are: retention of nutrients and bioactive compounds, freeze drying, due to its low-temperature operation, effectively preserves the nutritional and functional components in natural oils. This is especially advantageous for oils that are rich in thermosensitive compounds such as antioxidants. For example, buriti oil processed through freeze drying retains significant levels of carotenoids and antioxidant activity

(Pereira de Oliveira et al., 2022). Second one is to improved oxidative stability, this technique also contributes to enhanced oxidative stability (Oliveira et al., 2023). Studies on freeze-dried black soldier fly larvae oil reported lower peroxide values compared to those subjected to oven drying, indicating improved long-term preservation of oil quality (Hurtado-Ribeira et al., 2024), also freeze drying can be synergistically applied with encapsulation to improve both stability and ease of handling. For instance, using wall materials such as inulin and gum arabic, buriti oil has been successfully converted into stable microparticles that maintain their structural and chemical integrity (Pereira de Oliveira et al., 2022).

Challenging and key consideration of freeze-dry, they are: high energy demand, freeze drying is characterized by significantly higher energy consumption—ranging from four to ten times greater than that of traditional drying methods—resulting in increased operational costs. However, these costs are often offset by the superior quality of the resulting product (Bhatta et al., 2020; Yao et al., 2023). Optimization also the second challenge, enhancements in freeze-drying efficiency can be achieved through process optimization. For instance, incorporating dry ice during the freezing stage has been shown to reduce energy usage by up to 43%, as observed in the freeze drying of habanero peppers (González-Toxqui et al., 2020). Broad product applications the last one, freeze drying is highly adaptable and applicable to a diverse range of natural materials, including plant-derived foods and oils. It has been successfully employed to generate stable, high-quality products from sources like olive leaves and oregano processing by-products, leading to increased retention of bioactive compounds and enhanced antioxidant capacity (Koutra et al., 2025).

Although freeze drying provides significant advantages in preserving natural product oils, it is essential to weigh the trade-off between quality and economic feasibility. The method's high energy requirements and operational costs present challenges for its implementation on an industrial scale. Nevertheless, the increasing consumer preference for high-quality, minimally processed natural products is likely to encourage further technological advancements and refinements in freeze drying processes, potentially improving their cost-effectiveness and environmental sustainability. Moreover, combining freeze drying with other efficient physical techniques may enhance overall process efficiency and reduce expenses, thereby expanding its use across the food and pharmaceutical sectors (Koutra et al., 2025).

Based on Table 1, various types of polymers are used as wall materials in blending processes for emulsification systems and coacervation before undergoing freeze drying. The most commonly used wall material according to Table 1 is maltodextrin, which has favorable characteristics such as low viscosity at high concentrations, making it easier for the freeze-drying process as it does not interfere with the formation of the dry pore matrix structure. It also has film-forming capabilities, enabling maltodextrin to form a stable and good dry structure, which is crucial for freeze drying since the final product needs to have low moisture content while maintaining the physical and chemical stability of the active ingredient. Additionally, it has high solubility, making it easy to form a homogeneous solution with core materials such as vegetable oils or other bioactive compounds. Maltodextrin is biocompatible, non-toxic, and easily digestible, which allows it to encapsulate active materials such as essential oils or unsaturated fatty acids effectively, improving encapsulation efficiency and reducing volatility loss during freeze drying. Furthermore, maltodextrin is cost-effective and widely available, making it highly suitable for industrial-scale applications. However, maltodextrin's emulsifying capacity is low, so it works best when combined with other polymers to enhance the stability of the physical mixture (Lekshmi et al., 2021). One polymer that combines well with maltodextrin is alginate. Alginate is a natural polysaccharide that is water-soluble, economical, non-toxic, and compatible with oil systems (Ghaedi et al., 2020).

Alginates have three different linear arrangements of blocks. The G blocks consist of L-guluronic acid, which reacts with cations to provide greater gel strength, while the M blocks are made of D-mannuronic acid, and the MG blocks are soluble in lower pH (Tavassoli-Kafrani et al., 2016). Therefore, alginate can crosslink in M and G blocks to form an eggbox model, which is an essential structure for forming capsules under specific conditions with divalent cations such as Ca, Ba, Mn, Cu, and Zn (Priyadarshi et al., 2021). In addition to alginates, maltodextrins, which are polysaccharides, are commonly used in food systems due to their high solubility, low viscosity, and colorless nature. Maltodextrins are also used as encapsulating agents in spray drying processes, highly compatible with alginate to serve as the outer shell and wall of capsules (Maqsoudlou et al., 2020). Moreover, maltodextrin is highly digestible, releasing inner compounds under various gastrointestinal conditions (González et al., 2020).

Sodium caseinate is also a good wall material when combined, known for its excellent emulsifying properties due to its amphiphilic nature, meaning it has both hydrophilic (water-attracting) and hydrophobic (oil-attracting) parts (X. Liu et al., 2023). This makes sodium caseinate highly effective as an emulsifier, maintaining the stability of emulsions between oil and water before drying (e.g., vegetable oil emulsions in an aqueous solution). It forms stable layers between the oil and water phases, which is essential for preserving droplet integrity during freeze drying (Ma & Chatterton, 2021).

Another good wall material combination is soy protein isolate (SPI), a by-product of the soybean oil industry (Ye et al., 2019). SPI has garnered increased attention due to its large-scale production, cost-effectiveness, excellent film-forming ability, good gelling capacity, and emulsifying properties (dos Santos Paglione et al., 2019). SPI is made up of approximately 90% globulin proteins, with the major fractions being ß-conglycinin (7S) and glycinin (11S), corresponding to 37% and 31% of the total protein extract, respectively (H. Zhang & Mittal, 2010).

Another polymer that can be used as a wall material is lignin, the second most abundant natural organic polymer on Earth. This aromatic biopolymer has attractive properties such as biodegradability, non-toxicity, amphiphilic nature, and renewability (Ganewatta et al., 2019). Despite its many advantages, the use of lignin in valuable applications is limited due to its irregular structure. Lignin nanoparticles (LNPs) expose hydrophilic functional groups on the outside, shielding hydrophobic groups internally. LNPs can be easily dispersed in water, opening the possibility for their use in various applications (Alqahtani et al., 2019; Wijaya et al., 2021).

GA (Gum Arabic) is also an effective encapsulation agent due to its colloidal functionality. GA is compatible with most starches, gums, proteins, and carbohydrates and forms the most stable emulsions with oils across a wide pH range (Alves et al., 2014). However, due to the rising demand, the cost of GA has increased, and researchers are exploring suitable alternatives to replace it (Charve & Reineccius, 2009).

WPI (Whey Protein Isolate) is another excellent coating agent for oils and volatile compounds because of its hydrophilic and hydrophobic amino acids, which assist in encapsulating hydrophobic substances (Karrar et al., 2021). WPI also helps stabilize emulsions by reducing interfacial tension and creating strong competitive interactions. The significance of WPI in emulsion viscosity stabilization and flow behavior modulation has been documented (Du et al., 2021). Additionally, WPI is a functional protein with unique drying characteristics, such as high yield, shielding, and smooth flow (Khalifa et al., 2019).

The most widely used cyclodextrins are α, β, and γ cyclodextrins, differentiated by the number of glucopyranose units in their structure (LOFTSSON & DUCHENE, 2007; Pinho et al., 2014). Due to their unique structure, cyclodextrins can host various molecules to form inclusion complexes, improving the bioavailability, stability, and release profile of the encapsulated molecules (LOFTSSON & DUCHENE, 2007; Waleczek, 2003). Hydroxypropyl functional groups can be added to β-cyclodextrins to form hydroxypropyl-beta-cyclodextrins (HPβCD), which have greater safety and solubility (Garnero et al., 2010). HPβCD is approved by the FDA as an excipient in oral and intravenous solutions (Braga, 2019). Additionally, cyclodextrins, including HPβCD, have been recognized as safe (GRAS) by the FDA (Becktel et al., 2022) and are used in food products in Europe and the U.S. Freeze-dried natural unsaturated fatty acids and essential oils encapsulated in polymeric matrices have found numerous applications in food matrices.

The storage stability and release of essential oils depend on the composition of the wall material and encapsulation efficiency. The thermal stability of freeze-dried natural oils encapsulated in polymeric matrices has been investigated using Thermogravimetric Analysis (TGA) (Sun et al., 2021). The findings, as shown in Table 1, demonstrate that the degradation temperatures range from 200–350°C for Sacha Inchi oil, 175-375°C for Walnut oil, 100–200°C for Rose Damascena Essential Oil, 271°C for Mandarin Essential Oil, and 113°C for Boswellia Sacra Essential Oil. These temperatures represent the degradation of the wall material, which prevents direct degradation of the encapsulated natural oil and limonene (Olmedo et al., 2015a). Additionally, the surface amount of essential oils is mainly affected by powder porosity and encapsulation efficiency. The degradation temperatures between 350–490°C for oils like Sacha Inchi and Walnut show the influence of wall materials on the thermal behavior during freeze drying. High molecular weight polymers and complexes of proteins and polysaccharides offer relatively high thermal stability. The variation in thermal conditions across different natural oils is due to the differing compositions of each oil. For instance, Sacha Inchi has a high omega-3 content (45-50%), omega-6, omega-9, vitamin E, and phenolic compounds (Aranda Saldaña et al., n.d.; Soimee et al., 2020). Walnut oil is rich in omega-6 (50–60%), omega-3 (10–15%), omega-9 (13–17%), and contains phenolic compounds and vitamin E (Xu et al., 2023). Canola oil contains omega-9 (± 61–64%), omega-6 (± 20–22%), omega-3 (± 8-11%), vitamin E, and phytosterols (Correndo et al., 2024). Sunflower oil contains omega-6 (60–75%), omega-9 (15–30%), phytosterols, squalene, and vitamin E (Kurtulmuş, 2021). Fennel Essential Oil contains anethole (15–30%) and fenchone (5-25%) (Abd El-Kareem et al., 2025). Rose Damascena Essential Oil has citronellol (20–45%), geraniol (5-20%), and phenylethyl alcohol (10-30%) (Ahadi et al., 2023). Mandarin Essential Oil contains a large amount of limonene (65–95%) (Lin et al., 2021). Boswellia Sacra Essential Oil contains limonene (5–20%) and α-pinene (25–50%) (Miran et al., 2022).

In addition to freeze drying, the wall material and composition that affect thermal properties, functional groups also influence these thermal behaviors. Canola, Sunflower, Walnut and Sacha Inchi oils have ester and alkena groups. Ester groups in triglycerides in vegetable oils have good stability at low temperatures. Freeze drying does not cause ester hydrolysis because there is no exposure to high temperatures that could break the ester bond. Alkenes (C=C): Double bonds in alkenes like oleic, linoleic, and α-linolenic acids are more susceptible to oxidation when exposed to air, but freeze drying at low temperatures reduces oxygen exposure. This helps maintain the stability of double bonds that are sensitive to high temperatures and oxidation (Correndo et al., 2024; Erdem & Kaya, 2022; Guerrero et al., 2022; Kurtulmuş, 2021; D. Zhou et al., 2017; Zuleta et al., 2012). Fennel Essential Oil contains aromatic ether, ketones, and alkenes. Aromatic ethers in essential oils (like anethole) are relatively stable at low temperatures. Freeze drying helps preserve the stability of ether compounds, which would degrade if exposed to high temperatures. Ketones (Fenchone) are stable at low temperatures, and freeze drying helps maintain their structure without unwanted chemical changes. Alkenes (Limonene) are also stable at low temperatures, and the freeze-drying process helps maintain the integrity of alkenes (Abd El-Kareem et al., 2025; Sun et al., 2021). Rose Damascena Essential Oil contains alcohols, alkenes, and aromatic alcohols. Alcohols (Citronellol, Geraniol) in essential oils are stable at low temperatures. Freeze drying preserves these alcohols without affecting their quality, allowing the preservation of these compounds without degradation. Alkenes (Geraniol and Nerol) with double bonds in geraniol and nerol are relatively stable at low temperatures, so freeze drying preserves their chemical structure. Aromatic alcohols (Phenylethyl alcohol) remain stable at low temperatures and do not degrade during freeze drying (Ahadi et al., 2023; Khodadadi et al., 2024). Mandarin and Boswellia Sacra Essential Oils (Alkenes, Esters). Alkenes (Limonene in Mandarin, α-pinene in Boswellia) are double bonds (alkenes) in limonene and α-pinene, which remain stable at low temperatures, and freeze drying does not affect their double bonds or hydrocarbon structure. Esters (Incensole acetate in Boswellia Sacra) are also stable at low temperatures, so freeze drying preserves their presence without any changes (Lin et al., 2021; Mahdi et al., 2021; Miran et al., 2022).

The thermal stability of freeze-dried essential oils encapsulated in biopolymeric matrices has been extensively examined using Thermogravimetric Analysis (TGA) by various researchers (Muhoza et al., 2020). Degradation process occurs in two major temperature ranges: 100–200°C and 200–400°C. The lower range (100–200°C) corresponds to the breakdown of essential oils present on the particle surface, which is largely governed by the volatility and boiling points of individual constituents. For example, camphor, citronellal, and carvacrol exhibit higher degradation temperatures compared to alpha-pinene and limonene (Muhoza et al., 2020). The quantity of surface oil is significantly influenced by the powder's porosity and encapsulation efficiency (Q. Liu et al., 2020).

The higher temperature range (200–400°C) reflects the thermal decomposition of both the core essential oils and the wall materials, which is determined by the thermal behavior of the encapsulating biopolymers. For instance, gelatin begins to degrade at around 200-300°C, natural gums at ≥260°C, milk proteins between 200–400°C, and plant proteins from 200–320°C. In general, high-molecular-weight biopolymers and protein-polysaccharide complexes demonstrate superior thermal stability (Olmedo et al., 2015).

Stability of freeze-dried essential oils at different temperatures (4, 25, 40, and 50°C) over durations ranging from 20 to 540 days. Results indicate that powders stored at 4°C maintained stability for up to 360 days, whereas the other one using at 50°C significantly accelerated oxidative degradation. After approximately one year of storage at 4°C, essential oil retention was around 90%, in contrast to only 25% under accelerated storage at 50°C. Elevated temperatures increase the permeability of the wall matrix, facilitating both the evaporation of volatile oils and the diffusion of oxygen, thereby accelerating oxidative deterioration and reducing oil retention (Alabrahim et al., 2024; Miran et al., 2022).

Furthermore, freezing at very low temperatures (−5 to −16°C) can lead to the formation of large ice crystals, resulting in porous powders with cracks and larger pore sizes. These structural characteristics enhance diffusion rates, thereby reducing long-term stability. Hence, optimizing the interfacial membrane properties such as thickness, permeability, mechanical strength as well as controlling the freezing process, is crucial for achieving stable freeze-dried powders (Q. Liu et al., 2020; Mahdi et al., 2021; Muhoza et al., 2020).

Essential oils that have been freeze-dried and encapsulated within biopolymeric matrices are engineered to provide controlled and targeted release under diverse environmental conditions, including variations in pH, temperature, ionic strength, mechanical forces, and humidity. Encapsulated powders were tested across pH values from 2 to 8 and temperatures of 4°C, 25°C, 50°C, 60°C, and 80°C. The extent of release under these conditions ranged between 20% and 70%, with higher temperatures and prolonged exposure times generally promoting greater release of the core essential oils. Nonetheless, several factors such as the type of shell material, membrane thickness, porosity, and interactions between the wall matrix and the essential oils significantly influence the release profile (Dima et al., 2016).

For instance, freeze-dried *Coriandrum sativum L.* essential oil encapsulated in a chitosan matrix showed maximum release at pH 2.5 and 25°C, whereas encapsulation in an alginate matrix led to a higher release at pH 6.5. These differences were associated with the specific behavior of the wall material, including its molecular conformation, swelling capacity, and solubility under given conditions. Furthermore, when eucalyptus oil was encapsulated in a crosslinked hydrophilic biopolymer, it exhibited a slow and sustained release over 240 minutes at 40°C. This sustained release was attributed to the degree of crosslinking, which influences the structural integrity and barrier function of the interfacial membrane (Dima et al., 2016; Noppakundilograt et al., 2015).

CONCLUSION

In natural oils, both essential oils and oils with high unsaturated fatty acids, stability improvement can be achieved through freeze drying using polymers as wall materials such as polysaccharides, proteins, gums, lignin, or other polymers. The use of proper machine freeze drying parameter settings is also necessary to obtain the best results with high encapsulation efficiency. Stability improvement can be observed in thermal analysis testing, which shows that degradation starts with the encapsulation system first, followed by degradation in the core. Future studies are recommended to incorporate comprehensive in vitro evaluations of each natural oil to assess and compare their biological activities in relation to their physicochemical stability profiles. These findings should be further correlated with the entrapment efficiency values, providing a clearer understanding of the relationship between encapsulation performance and bioactivity.

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