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**Chapter**

# Encapsulation of Essential Oils and Their Use in Food Applications

*Hamdy A. Shaaban and Amr Farouk*

## Abstract

Due to the modern lifestyle and consumers' interests, demands toward healthy foods and nutraceuticals were increased, among them essential oils (EOs) characterized by different biological activities. However, the use of EOs in foods and pharmaceuticals may be limited due to the hydrophobicity nature in addition to the instability and cause of degradation upon exposure to environmental conditions, e.g., oxygen, temperature, and light. Therefore, encapsulation in various colloidal systems such as microcapsules, nanospheres, nanoemulsions, liposomes, and molecular inclusion complexes, seem to be the solution for such issues. New trends in food packaging have also been focused on exploiting capsulated bioactive EOs constituents for extending foods' shelf life due to their potent antimicrobial agents and the great activity against pathological bacteria. Micro and nanoencapsulation of EOs may affect their biological activities based on the technique used. In the current chapter, different subjects have been discussed, like techniques used for the encapsulation of EOs, potential applications in food, and their behaviors/trends after encapsulation.

Moreover, the benefits of encapsulation, namely bioavailability, controlled release, and protection of EOs against environmental stresses, are discussed. The applications of encapsulated EOs are also summarized in this chapter. Also, the relevance of the encapsulation of EOs as antimicrobial agents and their incorporation into food packaging are discussed.

**Keywords:** essential oils, encapsulation, biological activities, food preservation

## 1. Introduction

Essential oils (EOs) can be extracted from any part of plants and are considered secondary metabolites. They usually comprise a complex mixture of alkaloids, flavonoids, isoflavones, monoterpenes, phenolic acids, carotenoids, and aldehydes [1]. EOs consist of a broad spectrum of components in which the efficacy as antimicrobial, antioxidants, etc., comes from the synergistic effect of many components. These components are responsible for the ability of EOs to be introduced and incorporated in many applications, such as in cosmetics, nutraceuticals, and food products. The application of EOs industrially is often limited. They are susceptible to environmental conditions such as light, oxygen, and temperature; they easily evaporate, are nearly insoluble in water, and have strong lipophilicity and volatility [2]. As a result, exploring the potential to extend their applications has become a key research issue.

Encapsulation has been introduced to improve EOs applications. It allows for the preservation of bio-functional properties of EOs, enhancing their stability against harsh conditions, giving benevolent masking effect, and providing controlled release of EOs. In a study by Shetta et al. [3], it was found that encapsulation significantly enhances the thermal stability of encapsulated peppermint and green tea EOs around 2.18 and 1.74 folds, respectively, pure EOs. Encapsulation can be achieved by many techniques and divided into 1) chemical method, 2) physicomechanical method, and 3) physicochemical method. The encapsulation process might involve more than one technique [4]. The selection of the most feasible technique would depend on the type of coated material, the operational cost, and the application of the encapsulation products. Encapsulation parameters such as encapsulation efficiency, encapsulation yield, payload/loading capacity, and surface loading are commonly used as primary indicators to reflect the performance of the encapsulation process and quality of encapsulation products (encapsulates).

Packaging protects foods from environmental factors and microbial contamination to maintain food quality and safety [5]. Using bioactive packaging avoids food spoilage and poisoning, which seriously affects public health and extends the shelf life of food products, especially those susceptible to microbial spoilage [6]. Unlike routine packaging, which only avoids the exchanges of air gases, moisture, and aromatic compounds between the food and the environment around [7], bioactive packaging provides antimicrobial activity to extend shelf life and food safety [8].

The safety and quality of packaged food by incorporating natural antimicrobial compounds and natural antioxidant compounds [9] is now an active research area [10–14]. Unfortunately, their use in raw form in food packaging materials is restricted by the hydrophobicity nature and the low stability against the environmental conditions during the processing, distribution, and storage of foods [15]. Also, the uncontrolled release of volatile active constituents of EOs can significantly negatively affect their biological benefits [15]. To overcome such limitations, appropriate carriers and encapsulation techniques were designed.

The design of the encapsulation method on the form of essential-oil-loaded particles is a complex process with interrelated steps [5] based on many factors like choosing the wall material, technique used, and the intended matrix in which essential oils are to be incorporated [8]. Basically, the nanoencapsulation process is the coating or trapping EOs as a core material by biopolymers to avoid the limitations of using EOs as a natural food preservative. Accordingly, different techniques could be used for the nanoencapsulation of EOs, such as nanoemulsion and liposomes. In the specific case of essential oil nanoemulsion, the preparation consists of a biphasic liquid system of one liquid solution dispersed in a continuous medium, and no polymer shells are used [12]. The presence of EOs in stable nanoemulsions helps enhance their dispersibility in aqueous solutions, avoid the interaction with other food ingredients or environment, keep their organoleptic properties, and improve their absorption and bioavailability. Therefore, nanoemulsions ofEos as a natural powerful food conservator became a potential target with respect to the encapsulation technique, leading to the instability or the inefficiency of the produced emulsion. A better understanding of the EOs encapsulation phenomenon would widen the knowledge of possible alternatives to consider while designing green food preservatives for future research. Accordingly, this chapter covers a general description of the EOs and encapsulation techniques along with evaluation for these methods and a comparison between nano- and microencapsulation. Finally, the effect of the nanoemulsion technique used on the EOs constituents was discussed based on recently published studies [16].

## 2. Essential oils

EOs are natural substances consisting of mixtures of different volatile and aromatic constituents. They are widely found in herbs and spices such as garlic, black cumin, cloves, cinnamon, thyme, basil, bay leaves, coriander, mustard, rosemary, sage, and others [17, 18]. The EOs constituents produced as secondary metabolites have many functions, such as insecticidal, antimicrobial agents, or attracting insects to help in flower pollination [18]. Flowers, leaves, stems, roots, fruits, and even seeds could be sources for EOs. Different techniques are used to extract, such as steam and hydro-distillation. Organic solvents extraction such as ethanol, acetone, and methanol are also used based on the polar solubility of the different constituents of EOs [19–21]. Distillation of EOs depends on their density, which is mostly less than 1, despite a few exceptions, e.g., cinnamon, sassafras, clove, and vetiver [22]. Based on the structure of their different constituents, the bioactivity of EOs showed various potential uses and applications as antimicrobial, antioxidant, and antifungal agents against yeasts and filamentous fungi, which represented a potential natural and potential healthy use as food preservatives [23–25]. Due to their antioxidant activity, the application of different EOs in food industries, especially fats and oils, to avoid lipid peroxidation caused by free radicals represented a potential target [26]. Lipid peroxidation results in many negative impacts for food products, including unpleasant aroma and flavors, deterioration of the food quality, decreasing the nutritional value of food, and severe health issues [27]. Based on the modern lifestyle and consumer demands, using EOs as natural antioxidants is favored over synthetic antioxidants such as butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), and tert-butylhydroquinone whose applicability has been discouraged due to safety, health, and environmental concerns [28].

Among others, phenols, esters, terpenes, sesquiterpenes, aldehydes, ethers, alcohols, and phenylpropanoids, constitute the main classes in EOs responsible for bioactivity and sensory properties [22, 25]. For example, thymol, carvacrol, α-terpinene, eugenol have antioxidant effects [29], while other constituents such as limonene, eugenol, pinene, carvone, and linalool carvacrol have been suggested as agents responsible for the antimicrobial efficiency against foodborne pathogens [30]. In the same context, eugenol exhibited an efficient bactericidal activity against *Salmonella enterica* serovar *Typhimurium* as well as carveol, citronellol, and geraniol which presented anti bactericidal activity against E. coli, while terpineol had good activity versus S. aureus strains [31]. The presence of hydroxyl groups is responsible for the previously described compounds' higher antimicrobial activity [31]. Meanwhile, other compounds belonging to different classes such as benzoic acids, benzaldehydes, and cinnamic acid have shown up to 50% inhibition of *Listeria monocytogenes* under anaerobic conditions [32]. The EOs of similar plants have been reported to have differences in composition depending on the geographical location that the plant is found [33]. Notably, the composition and yield of EOs can vary with environmental conditions, climate, harvesting stages, planting, preparation methods, and genetics [34]. For example, weather parameters like rains and temperature have influenced the oil content and its constituents [35].

## 3. Encapsulation

Encapsulation of active ingredients or a core in solid walls or carriers represents a potential solution to control their release during storage or application and protect them from environmental conditions or interactions with the matrix around. As EOs are hydrophobic, emulsifying or dispersing in an aqueous solution represents the important primary step in the whole process. Following the emulsifying process, encapsulation can be performed by different techniques; chemical techniques like molecular inclusion or interfacial polymerization; physicochemical techniques like conservation and liposome encapsulation; and physical techniques like spray drying, spray chilling/cooling, co crystallization, extrusion, or fluidized bed coating [36]. Based on the technique and energy used, capsules can be found in micro and nanoscales, where microcapsules range between a few micrometers and a few millimeters while nanocapsules are found in the range of 53.8–415.2 nM. Other factors affect the size and physical properties of the capsules, like the natural or synthetic polymers used as wall materials and the core used. This system can increase the passive cellular absorption mechanisms, reduce mass transfer resistances, and increase antimicrobial activity due to its subcellular size [37].

## 4. Encapsulation of EOs

Natural EOs and extracts have limited applicability [38] because of drawback reactions during processing, transporting, or storage like oxidation, hydrolysis, crystallization, or enzymatic deterioration in the presence of oxygen and light [39, 40]. The lower thermal stability during food processing causes loss of EOs active components' biological functionalities [41] and significantly deteriorate their flavor and solubility. For example, the pomegranate peel extract associated with easy oxidation causes color deterioration and other instability issues [42], while Satureja hortensis EO drastically changes composition upon heating over 160°C [43]. The intense flavor of EOs, which is used as preservatives, may be transferred to the packed foods and negatively affect the final product's sensory properties[44, 45], so encapsulation is required to avoid the volatility of EOs bioactive components [46]. Consequently, many researchers have encapsulated them into other protection materials in order to make full use of their antioxidant and antimicrobial properties [47]. Nanoencapsulation of bioactive components to apply in food packaging materials are a potential target, growing steadily [48], since it can protect the components and therefore their biological efficiency against oxidative degradation upon exposure to air or high temperatures and during food processing [49, 50], in addition, to control their releasing [51]. For example, encapsulating thyme EO into cyclodextrin/ε-polylysine can reduce undesirable deficiencies such as volatility and hydrophobicity of its bioactive components [52]. While carvacrol, characterized by its antimicrobial activity, can be protected/encapsulated in a starch fiber matrix to avoid direct contact with food and reduce the effects on sensory features [53]. Encapsulation in zein microparticles improved the thermal stability of polyphenols from maqui fruit extract when exposed to high temperatures related to processed foods [54]. Orange and thyme oil adsorbed in halloysite or montmorillonite clay and then encapsulated in a polyethylene/polyamide/polyethylene multilayer film prolongated aroma release [55]. Encapsulation of black pepper (*Piper nigrum* L.) EO into sodium alginate and gelatin by complex coacervation avoids the loss of the main volatile from EOs preserved (80% of their original content) [56].

| Encapsulation method | Description | Nanoencapsulation | Microencapsulation |
|---|---|---|---|
| Emulsification | Emulsification is a process of mixing two immiscible solvents, and the resulting product is referred to as an emulsion. It can be divided into top-down approaches (high-shear stirring, high-pressure homogenization, microfluidization, and ultrasonication) and bottom-up (phase inversion temperature, emulsion phase inversion, and spontaneous nano emulsification) approaches. | Vitamin E encapsulated by Tween-80; vanillin encapsulated in poly(lactic acid) nanoparticles | Curcumin encapsulated by Tween 80 and polyglycerol polyricinoleate; lycopene encapsulated in plant (soy and pea) or dairy (whey and sodium caseinate) proteins |
| Spray drying | The basic theory of spray-drying is to feed the liquid into a drying chamber in the form of tiny droplets containing biologically active compounds, supplying hot air to the drying chamber, forming microcapsules in the drying chamber, and recovering them through a cyclone. | Folic acid encapsulated by whey proteins and resistant starch; curcumin encapsulated by chitosan/Tween 20 | Propolis extracts bioactive compounds encapsulated by maltodextrin matrices with or without nature gums; cocoa volatile compounds encapsulated by maltodextrins and modified starch |
| Freeze drying | The basic principle of freeze-drying is to freeze water contained in a solution or suspension and then evaporate the water molecules from the solution or suspension. | Fish oil encapsulated by poly-e-caprolactone and Pluronic F68 | Blackberry by-product extract encapsulated by maltodextrins; flaxseed oil encapsulated by sodium alginate, whey protein, and maltodextrin |
| Extrusion | Extrusion technique involves the injection of a bio-based solution into another solution to promote gelation and produce a hard and dense encapsulation system. | Seed oils encapsulated by sodium alginate and high methoxyl pectin | Canola oil encapsulated by alginate and high methoxyl pectin; quercetin encapsulated by carnauba wax, shellac, or zein |
| Complex coacervation | Coacervation is a well-known implemented technique to produce micro- and nanosystems. The basic mechanism is the formation of an emulsion by electrostatic attraction between oppositely charged molecules to produce the encapsulating structure. | Folic acid encapsulated by casein nanoparticles; anthocyanins encapsulated by whey protein isolate and beet pectin | Algal oil encapsulated by soy protein isolate and chitosan; β-carotene encapsulated by casein and gum tragacanth |
| Electro-spinning and electro-spraying | They are two modes of electrohydrodynamic processes that use a charged jet to rotate or spray a polymer solution to produce fibers or particles. | Rosehip seed oil encapsulated by zein prolamine fiber; β-carotene encapsulated by zein prolamine fiber | D-limonene encapsulated by seed gum and tween 20; fish oil encapsulated by a composite zein fiber |

**Table 1.**
*Summary of recent studies on micro- and nanoencapsulation of food bioactive compounds.*

## 5. Encapsulation process evaluation method

Generally in encapsulation, the idea of quantifying EO upon encapsulation process is 1) to calculate encapsulation efficiency and other encapsulation parameters; 2) to perform a controlled release study and understand the kinetics of release [57]; as well as 3) to evaluate the stability of encapsulates based on how much oil is left in the encapsulates [58], or how much oil is released to the releasing media [59], and still adhered to the surface [60]. Besides that, it is crucial to determine the components that are successfully encapsulated and responsible for the bio-function of EOs exactly. These components or types of EOs would have effects on encapsulation evaluation parameters. In a study by ref. [61], different encapsulation efficiency values were obtained when encapsulating kaffir lime oil from peels (KLO-P) and twigs oil fraction (KLO-TF) using chitosan as wall material. It was found that the encapsulation efficiency of KLO-TF is greater than KLO-P. The encapsulation efficiency difference was attributed to the components presented in each kaffir lime oil in which KLO-TF contains more oxygenated monoterpene components while the hydrocarbon monoterpenes components dominate KLO-P. Oxygenated monoterpenes components are more likely to interact with the functional group (active site) in the encapsulate, and as a result, more KLO-TF was successfully encapsulated.

Determination of EO in encapsulates can be done gravimetrically through direct measuring [62] or the distillation process. However, drawbacks associated with such techniques are that a large amount of formulation is required, improper extraction, and chances of loss of EO due to volatilization. To overcome these issues, reliable techniques using analytical methods such as chromatographic or spectrophotometric methods are introduced and expected to exhibit higher values than when the thermogravimetric analysis is used [63]. When employing these analytical methods, sometimes, digestion of the wall material is required to be achieved physically, chemically, or enzymatically [64]. **Table 1** below shows different types of EOs and commonly used solvents and methods to digest encapsulated walls. Subsequently, EO is extracted using an organic solvent such as hexane [65], petroleum ether, ethanol [66], or non-ionic surfactant; tween-80 before quantification using appropriate analytical methods. These analytical methods also have some disadvantages, such as possible experimental error, chances of loss of EO due to volatilization, and the possibility that the method selected is not convenient. For example, in cases where digestion of encapsulated walls is needed, the digested wall materials might somehow interfere with the spectrometric reading of EO. However, this could be resolved by using appropriate solvent and technique. Tolun et al. [66] used hexane to extract Moxa oil from encapsulates since gelatine and Gum Arabic used as encapsulating material did not interfere with the measurement process as they were insoluble in hexane. Meanwhile, Fraj et al. [67] used derivative spectrophotometry for quantitative analysis of core material since wall materials used (vitamin C and genipin) were also soluble in ethanol.

## 6. Nanoencapsulation versus microencapsulation

A comparison of micro- and nanoencapsulation functionality has been reported by [68], as shown in **Figure 1**. The main functionalities of microencapsulation taken into consideration are protecting active ingredients, including the extension of shelf

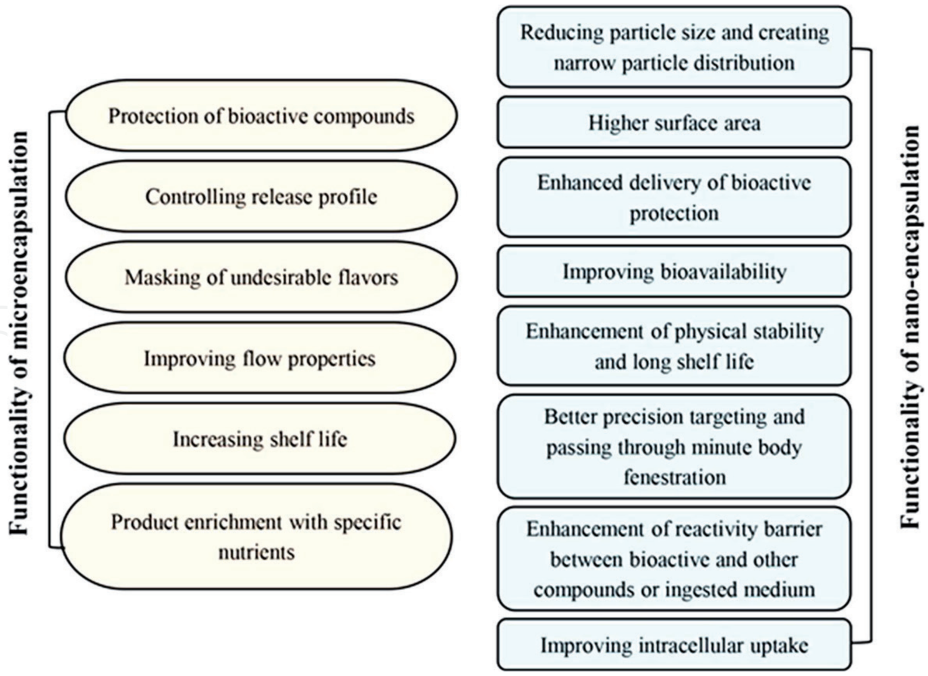


**Figure 1.**
*Advantagesofnano-andmicroencapsulation [69].*

life and controlling the release of bioactive components. While for nanocapsules, more attention is given to the functionals related to the size reduced like higher surface area and improving intracellular uptake. According to the authors, the formulation in nanoscales may improve bioavailability; however, this may depend on the technique used, as discussed later in section 7 of this chapter.

Particle size is an important factor affecting the functional characteristics of capsules [70]. Nanoencapsulation is the formulation of capsules with less than 1 micron (1000 nm), possessing different properties than ordinary encapsulation. According to the literature, capsules should be less than 100 nm to be considered nanocapsules [71]. The nanometric size of delivery systems can increase the surface area and, consequently, the food matrices' dispersion to form uniform and stable colloidal suspensions and may have better sustained-release effects than microcapsules. Based on their smaller size, nanocapsules can increase the passive cellular absorption mechanisms, promoting the effective release of active substances inside the target cells and consequently increasing the efficiency of active substances and their bioavailability.

Meanwhile, nanoparticles may penetrate the tissues (such as the liver) through the capillaries and are absorbed by the cells in the tissues; thus, the active substance can be efficiently delivered to the target cells in the body [72]. In the case of emulsions-based delivery systems, some interesting physical properties can distinguish nano and microemulsions. Microemulsions generally exhibit multiple scattering of visible light, which means they have an opaque white appearance. Conversely, nanoemulsions are much smaller than visible wavelengths, and therefore, they appear almost optically transparent, making them easily applied in the beverage industry [73].

Despite the numerous technologies for encapsulating biologically active compounds studied, only a few techniques, namely spray-drying and freeze-drying, are widely applied in the food industry [74]. Emulsification represents the first step of encapsulation. There are two types of approaches used to produce emulsions: a top-down approach and a bottom-up approach. The top-down approach involves reducing coarse particles' size through intensive mechanical destructive forces like high-pressure and high-shear homogenization, microfluidization, and microchannel homogenizers [75]. On the other hand, the bottom-up approach generally includes self-assembly, phase inversion, and spontaneous emulsification, which are affected by pH, temperature, concentration, and ionic strength [69].

Low-energy methods are used to prepare emulsions before other nanoencapsulation methods, e.g., spray-drying, complex coacervation, extrusion, electro-spinning, and electro-spraying [76]. However, low-energy methods require more stabilizers and surfactants to reduce the size and easily disperse the active ingredients [69]. Choosing the primary encapsulation technique is interrelated with many factors like the core and wall material properties, solubility, emulsification, particle size distribution, and food matrix composition [76]. **Table 1** summarizes the commonly used encapsulation techniques to formulate nano- and microcapsules.

## 7. Effect of Encapsulation by the Intensive-energy techniques on the structure and bioactivity of EOs components

Literature dealing with the encapsulation of EOs focused on the physical stability and biological activity of the micro or nanoparticles but not on the changes in the volatile constituents of the capsules. Few studies have reported that the formulation based on energy-intensive techniques like high-pressure and high-shear homogenization may lead to Ostwald ripening, flocculation, or coalescence of the emulsion with changes in its physical stability and biological activity [77]. Ali et al. [78] studied the effect of nanoencapsulation on volatile components and the bioactivity of Algerian *Origanum glandulosum* Desf. essential oil, a significant quantitative difference was observed in the level of monoterpenes between hydrodistilled oil and its nanocapsules. Additionally, the majority of sesquiterpenes were not detected in the nanocapsules extract. They owed that to the intensive-energy homogenization at 18000 rpm. Also, they reported that essential oil exhibited a higher antioxidant activity than nanocapsules and nanoemulsions, while nanocapsules showed the most potent cytotoxic effect on liver cancer cell line Hep-G2 in comparison to HD oil and nanoemulsions. In the same context, thymol and carvacrol were detected as predominates in the nanoemulsion of Algerian

*Saccocalyx satureioides* Coss. et Durieu oil was prepared by high-pressure homogenization, while borneol and α-terpineol were the major compounds detected in the same hydrodistilled oil, which affected the bioactivity of the oil and nanoemulsion [79]. Also, *Citrus sinensis* L.peel essential oil exhibited antifungal activity against *A. niger*, *A. ochraceus*, *Fusarium* spp., and *Penicillium* spp. Its nanoemulsion displayed lower antifungal activity, based on the changes in the chemical constituents due to homogenization by high-intensity ultrasound [80]. Further studies are necessary in order to explain the behavior of bioactive components during different encapsulation processes, especially the intensive-energy ones, and thereby evaluate the compatibility of the different encapsulation techniques for EOs.

## 8. Conclusions

Encapsulation represented an efficient approach to protect the EOs against environmental conditions that lead to oxidation or volatilization and reduced biological activities. Moreover, encapsulation solves the problem of EOs hydrophobicity and controls their release. Spray drying and emulsification are the most versatile and commercially available techniques used widely for EOs encapsulation. The encapsulated EOs showed enhanced antimicrobial, antifungal, antioxidant, antiviral, and insecticidal activities. The use of encapsulated EOs in food, cosmetics, and pharmaceutics could be an economic benefit and fulfill consumer concerns regarding safety. Energy-intensive techniques may negatively affect the structure-activity relationship of EOs bioactive components; therefore, further studies are necessary to find out the compatibility of encapsulation techniques for EOs.

## Funding sources

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

## Conflict of interest

The authors declare that there is no conflict of interest.

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