Advances in essential oils encapsulation: development, characterization and release mechanisms

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REVIEW PAPER

Advances in essential oils encapsulation: development, characterization and release mechanisms

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Abstract

Recent developments in micro and nanoencapsulation are promising tools to encounter the different limitations of essential oil formulations, enhance their functionalities, and protect them from the external environmental conditions. This review addresses the current studies and progresses related to the development of encapsulated essential oils using different systems and carrier material types. It also focuses on the formation methods used with the subsequent physicochemical characterization of the developed particles. Moreover, this review considers the factors affecting the release of essential oils with the different physicochemical release models. The choice of the appropriate formation method as well as the carrier material types and system forms were shown to highly depend on the intended purpose of the encapsulated essential oil formulation. Micro and nanoencapsulation are used to control essential oils' release properties, enhance the various characteristics of essential oils, and allow to expand applications in different fields. This review provides the optimal conditions for micro and nanoencapsulation of essential oil formulations based on the intended end uses.

Keywords Essential oil · Microencapsulation · Nanoencapsulation · Release mechanism · Physicochemical characterization

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Introduction

Researchers and consumers' interests have increased towards the use of biosourced essential oils (EOs) with relevant and diverse functional activities. EOs are active volatile hydrophobic compounds extracted as secondary metabolites from the different parts of edible, herbal and medicinal plants. Till present, 3000 different EOs were discovered and around 300 of which were commercially used in cosmetic, perfume, food, beverage, sanitary, agronomic, medicine, and pharmaceutical industries [1]. EOs global market is increasing day by day and it was found to exceed US$ 7.51 billion in 2018 with an estimated increase greater than 9% of its annual growth rate between 2019 and 2026 [2]. Over time, several authors have reviewed the use of EOs due to their remarkable biological and fragrance properties and have reported their anti-oxidant [3–6], anti-inflammatory [7–11], antifungal [12], pesticidal [13–15], antimicrobial [16–21] and anti-cancer [7, 22–24] properties.

EOs broad spectrum applications have increased as they derive from natural sources, degrade easily in soil and water, are environmentally-friendly and have shown low mammalian toxicity [25–27]. The use of such biosourced agents ensures a sustainable development, protection and respect for the environment. Nevertheless, the direct application of EOs in their free native forms is limited due to several reasons therefore, the main challenge is to develop new strategies able to encounter the different limitations of EOs. To achieve this, micro and nanoencapsulation could be powerful approaches that enhance the different potential functionalities of EOs while decreasing the amounts being used.

The novelty of this review lies in providing the most suitable approach for the micro and nanoencapsulation of EO formulations related to their intended applications. The objectives of this review are to discuss the recent progresses in micro and nanoencapsulation to overcome EO limitations, and to focus on encapsulation: (i) forms and carrier material types, (ii) methods and physicochemical characteristics, (iii) factors affecting EO formulations release and, (iv) the physicochemical release models of EOs.

Limitations of free essential oils and their active components

The application of EOs in their free forms could be hindered due to several critical factors as their: (i) low stability and susceptibility to degradation by volatilization and/or oxidation when exposed to external deteriorating factors as oxygen, light and temperature, (ii) low water solubility and (iii) potential interactions with food components.

Oxygen presence was mainly reported to induce considerable physicochemical and compositional alterations to EOs, reducing their stability, quality and potential functionalities [28]. The peroxide values indicating the amount of oxidized compounds of eucalyptus, lavender, may chang, pine, rosemary and turpentine

EOs increased mainly during the first month of storage indicating their oxidation and low stability [29]. Room storage of *Thymus daenensis* EO was shown to induce significant oxidation and evaporation of its major active compounds namely α-pinene, myrcene, *p*-cymene and γ-terpinene affecting thus its initial quality and limiting its use in cosmetic and pharmaceutical industries [30]. With an increase in storage period from 10 to 30 days, the concentration of geraniol, the main component of *Rosa x damascena* Mill. EO, decreased from 19.07 to 2.29%, reducing its application in flavoring and perfumery industries [31]. Also, upon air exposure, auto-oxidations of linalool and limonene were reported producing some oxidized compounds that increased skin irritation [32]. At room storage, lemon balm EO compounds having low boiling temperatures as neral, citronellal and geranial, evaporated and decreased from 18.9 to 4.0%, 25.8 to 12.6% and 27.0 to 4.6%, respectively [33]. Lemon EO samples stored in half-filled bottles containing air showed significant loss of γ-terpinene, limonene and citral [34].

Generally, light exposure induces also pronounced effects on the stability and the different potential functionalities of EOs. Storage of marjoram EO for more than 3 months in light, produced considerable changes in its chemical composition with an accumulation of oxidative compounds and a loss of its organoleptic properties making it unsuitable for use [35]. Whereas, storage in dark for 1 year did not induce any significant changes in both its chemical and organoleptic properties, suggesting that light accelerates chemical reactions and alters the stability of EOs [35]. Also, greater peroxide values were reported in rosemary and lavender EOs upon light exposure, when compared to storage in the dark at the same temperature [36]. However, in the same study, thyme EO showed pronounced stability when stored in different light and temperature conditions, which may be attributed to the high amounts of phenolic content as carvacrol and thymol that scavenge the free radicals formed during oxidation [36]. Fennel EO was extremely unstable throughout light storage with degradation of its monoterpene hydrocarbons and oxidation of *trans*-anethol, while lower oxidation was reported when stored in dark [37]. In another study, light and dark storages showed very similar effects on the degradation of lemon EO major compounds and the appearance of off-flavors and off-odors [34].

EOs stability was additionally found to be related to the temperature as generally, an increase in temperature accelerates chemical reactions and degradation of EOs contributing to their lower stability [28]. It was reported that after 12 weeks of storage at 38 °C, the peroxide values of rosemary EO were 8.2 times higher than the initial values, whereas at room temperature, the peroxide values were only 4.2 times greater than the initial ones, indicating the accelerated degradation of EOs at higher temperatures [36]. Also, heating nutmeg EO at 180 °C, induced significant loss of its major components (α-and ß- pinene and sabinene) with an increase in myristicin and safrole contents [38].

The direct incorporation of EOs into aqueous-based matrices and environments is limited due to their hydrophobic nature. As a result, their antimicrobial effectiveness and applications might be affected [39]. Higher concentrations will be required to achieve potential functionalities in water based matrices which may alter the organoleptic properties and exceed the acceptability thresholds

[40]. The addition of solubilizers to EO compounds as carvacrol, eugenol, linalool and 2-Pentanoylfuran enhanced their solubility from 938–1399 mg/L to 1.28–5.32 mg/L in aqueous media. Subsequently, their antimicrobial activities against *Escherichia coli*, *Staphylococcus aureus*, *Bacillus subtilis* and *Saccharomyces cerevisiae* were enhanced with lower amounts being used [40].

Another major limitation of EOs' applications, is their potential interactions with intrinsic food constituents as proteins, lipids and other compounds that impair their applications and reduces their effectiveness [1, 41]. For example, due to the strong binding between eugenol and fat globules present in milk, higher amounts of eugenol were needed to inhibit microbial growth in milk products compared to Tryptic Soy Broth culture medium [42]. Also, the antimicrobial activity of eugenol was more effective in skim milk compared to full fat milk. 3.5 g/L eugenol were needed to completely inhibit *E. coli* O157:H7 bacterial cells in skimmed milk (< 0.5% fat), while 5.5 g/L eugenol were needed for a complete bacterial inhibition in full fat (4%) and reduced fat (2%) milk [42]. In zero-fat hotdogs, 10 ml/L of thyme and clove EOs induced 0.86–1.72 log reductions of *Listeria monocytogenes*, while in full-fat hotdogs, lower bacterial reductions were induced (0.42–1.28 logs) due to the interaction of EOs with fats present in full-fat hotdogs [43]. The anti-listerial activity of oregano and thyme EOs was also reduced when incorporated with lipid oils and potato starch at concentrations higher than 5% [44]. All these examples of the reduced activities of EOs in food matrices may be explained by the presence of fats, proteins or starches that may form a layer around bacterial cells protecting them from the action of antimicrobial agents. Additionally, EO components may migrate to the fatty compounds in food due to their hydrophobic nature, allowing bacteria to develop freely in the aqueous fraction [43, 45, 46]. As the effect of EOs is reduced in food matrix systems, higher unacceptable concentrations will be required to compensate their interaction with food components and to perform their intended functionalities [47]. Thus, EOs may develop some unpleasant unacceptable odors and flavors that alter the sensorial properties and overall acceptability of food products limiting thus their incorporation and efficiency in several applications [39, 48]. As an example, the higher concentrations of *Mentha piperita* EO required to achieve an antimicrobial activity induced strong odors in minced meat that decreased their overall acceptability [49]. Also, the concentrations of *Salvia officinalis* L. and *Schinus molle* L. EOs required to induce significant anti-bacterial effect in minced beef meat had notable negative effects on the flavor and odor [50]. This further required the combination of EOs with other preservation methods to reduce the concentrations being used and thus their negative sensorial impacts.

Beside these different challenges, the high concentrations of free EOs probably needed for certain applications may increase the risk of developing resistance by several microorganisms [47]. The challenge is therefore to develop new delivery systems as micro and nanoencapsulation to overcome the different obstacles of free EOs and boost their effectiveness.

Micro and nanoencapsulation of essential oils

Encapsulation is the process in which one or more active agents (coated materials, core or internal phase) are loaded in a homogenous or heterogeneous matrix (shell, wall or carrier material) at micro (1–5000 µm) or nano (< 1 µm) scale [51–53]. The entrapped molecules are protected from the deteriorating external conditions such as degradation, evaporation and oxidation [13, 48, 54]. Due to their reduced size and increase in surface-to-volume ratio, micro and nanoparticles allow a greater bioavailability and a facilitated diffusion of EOs into their target sites enabling efficient long-term activities [48, 55–58]. In addition, lower amounts of EOs are used in the encapsulation process which achieves lower sensorial impacts on food products, reduces the probability of developing resistance by microorganisms, minimizes their toxic effects and reduces the economic costs.

Carrier material types

In micro and nanoencapsulation of EOs, different carrier materials could be used depending on their charge, on the encapsulated material as well as on the encapsulation process and the targeted applications [59]. Carrier materials could be homogenous or heterogeneous and solids or liquids [60]. For a better encapsulation efficiency and for environmental purposes, the used carrier materials are preferable to be biocompatible, biodegradable into non-toxic products, Generally Recognized as Safe (GRAS), commercially available, non-reactive with the encapsulated active ingredients, and easily administered [53, 58]. The carrier materials used for EOs encapsulation were natural biopolymers as polysaccharides, proteins, lipids as well as synthetic ones (Fig. 1). Moreover, in some encapsulation techniques, carrier materials used were a thin layer of surfactant molecules surrounding bioactive compounds and emulsified with an aqueous solution [56, 61–66]. Surfactants could be added to form more stable EO emulsions and reduce the interfacial tension between aqueous and oil phases [66].

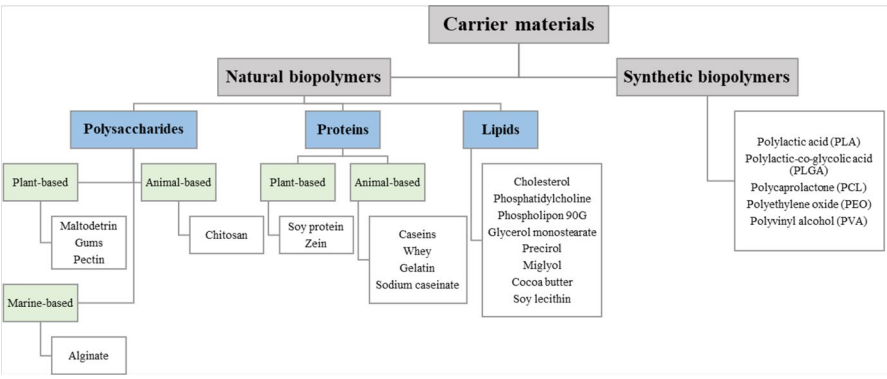

Fig. 1 The main types of carrier materials used for the encapsulation of essential oils

Polysaccharides used as carrier materials for EOs encapsulation derived mainly from plant, marine and animal sources. The polysaccharide-based carrier materials should be safe, non-toxic, biodegradable, have a good water solubility, a low viscosity at high concentrations and of low cost due to their abundancy in nature [53, 67, 68]. In comparison with other carrier types, polysaccharide-based matrices have a better thermal stability and are less sensitive to environmental changes as pH or ionic strength modifications [69].

Proteins derived from plant and animal sources are considered also good candidates for the delivery of EOs as they are widely available, easily modified, inexpensive, biodegradable, biocompatible and have good water solubility [51, 53, 67, 68]. However, most proteins are susceptible to pH, ionic strength or temperature changes, which limits their applications but could provide an advantage by enabling the controlled release of encapsulated active substances by a simple pH or heat trigger [69].

Several solid and liquid lipids were also used as carrier materials for the encapsulation of EOs. Lipid-based encapsulations showed high encapsulation efficiency, controlled release and potential applications at industrial levels [53, 58].

The other types of carrier materials used for the encapsulation of EOs were synthetic biopolymers. Most of these materials were found to be biodegradable, biocompatible, cost-efficient and non-toxic [70–72]. Some reviewers suggested that these polymers have shown better chemical and physical reproducibility with a higher purity than polysaccharide, protein and lipid-based carriers that presented certain limitations due to their broad range of molecular weight and variations from batch to batch [73, 74].

A combination of different types of carrier materials together has gained much attention recently in several applications. Carrier materials combination starts either by a direct interaction of materials together or by the layer-by-layer method which starts by the formation of particles made of one type of carrier material and then the adsorption of other carriers on the formed particles [75]. An example of a combination of carrier materials is whey proteins with maltodextrins that has improved thermal stability, emulsifying properties and transparency of eugenol nanocapsules [76]. Also, the combination of sodium caseinate and pectin has improved the stability and controlled release of citral from microcapsules [77]. Polylactic acid (PLA) combined with polyethylene oxide (PEO) [72], and zein combined with casein [78] ensured a controlled release of lavender, eugenol and thymol, respectively from nanocapsules. Thus, in general, the combination of different carrier materials together was proved to enhance encapsulation efficiency, loading capacity, thermal and mechanical resistance, controlled release, bio-availability and stability of the encapsulated bioactive compounds [53, 79, 80].

Carrier system forms

Several micro and nanocarrier systems have been adopted for the delivery of EOs and their bioactive compounds into specific target sites. The most common delivery system forms used are capsules, hydrogels, emulsions, liposomes, solid-lipid nanoparticles (SLNs) and nanostructured lipid carriers (NLCs) (Fig. 2). Each of these

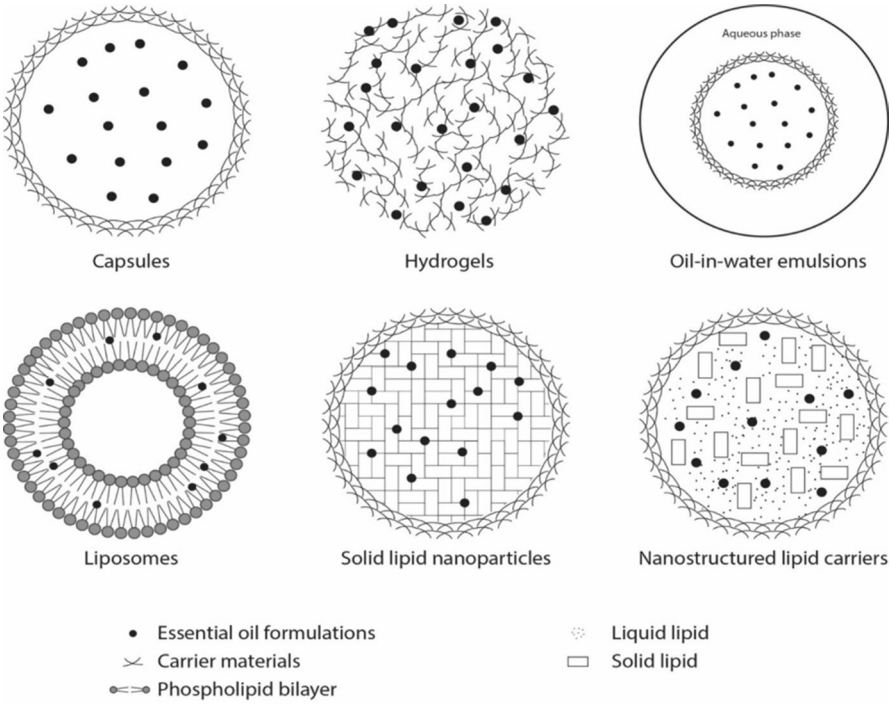

Fig. 2 Schematic representation of the main carrier system forms used for the encapsulation of essential oil formulations

systems has a specific architecture and physicochemical characteristics depending on the materials used for their development and on their formation methods [53].

Capsules

Capsules are hollow vesicular systems in which bioactive compounds are entrapped within a membrane of carrier materials that forms a protective shell [58]. Proteins like whey protein, zein, casein, gelatin; polysaccharides such as maltodextrin, chitosan, pectin, alginate, cashew gum, gum Arabic; and other carrier materials as PLA, PEO, polycaprolactone (PCL), and polylactic-co-glycolic acid (PLGA) were used for the micro and nanoencapsulation of EOs in capsules [21, 42, 68, 70–72, 78, 80–82]. Capsules have shown enhanced anti-oxidant and antimicrobial activities, stability and a controlled release of the entrapped bioactive compounds [78, 83].

Hydrogels

Generally, micro and nanohydrogels are made up of oppositely charged carrier materials that are cross-linked to form a three-dimensional (3D) structure that has the ability to hold a great amount of water in different severe conditions [53]. Usually, a filled hydrogel particle contains bioactive compounds dissolved in an oil phase and

entrapped within the carrier materials network. When exposed to various external stimuli, hydrogel particles ensure an effective controlled and sustained release of bioactive components while maintaining their network structure making them effective delivery systems [53, 84]. Hydrogels have also presented a high stability, high loading capacity, biocompatibility and a protection from chemical degradations for both hydrophilic and hydrophobic bioactive compounds [58, 85]. Polysaccharides like alginate, chitosan, pectin; proteins like soy and whey proteins; lipids such as soybean oil and other carrier materials as polyvinyl alcohol (PVA) were used for the encapsulation of EOs in hydrogels [60, 86, 87].

Emulsions

Emulsions are formed by the dispersion of two immiscible phases: one phase (dispersed phase) is spread as droplets into another phase (continuous phase) [53]. Emulsions could be water-in-oil (W/O) or oil-in-water (O/W) emulsions depending on the location of water and oil phases. O/W is the most suitable emulsion for the encapsulation of EOs. The incorporated bioactive compounds in the dispersed phase will be protected by the continuous phase from the different environmental conditions. Carrier materials used for the formation of EOs emulsions were polysaccharides like sodium alginate, gum Arabic; lipids like soybean oil or surfactant solutions like Surfynol, Tween, Span, Brij or lecithin [39, 61, 64, 65, 88–90]. Micro and nanoemulsions of EOs exhibited enhanced stability, controlled release, antioxidant and antimicrobial activities when compared to free EOs [39, 91]. Microemulsions were shown to be thermodynamically stable and appeared usually as white opaque droplets while nanoemulsions with smaller sizes, appeared as transparent to translucent droplets with a better kinetic stability favoring their use in food products [53, 66].

Liposomes

Liposomes are spherical vesicles made up of one or several phospholipid bilayers enclosing usually an internal aqueous phase [92]. Due to their amphiphilic nature, liposomes can be used to encapsulate, at the same time, both hydrophilic molecules within their internal aqueous compartment, and hydrophobic molecules as EOs within the lipid bilayer [93, 94]. Only lipid based carrier materials such as phosphatidylcholine, cholesterol and lecithin were used for the nanoencapsulation of EOs in liposomes [55, 95–97]. Liposomes enhanced the antimicrobial and antioxidant activities of the incorporated bioactive compounds [55, 95, 97]. In addition, they have shown targeted delivery properties [98]. However, the use of liposomes was limited due to their high production cost and as they have shown poor loading capacity and poor physicochemical stability [53, 58, 92]. Their low stability and its subsequent consequences may be improved by coating liposomes with additional carrier materials or trapping them within other carrier systems [99].

Solid lipid nanoparticles

Solid lipid nanoparticles (SLNs) are spherical nanocolloidal carriers made of completely crystallized lipid droplets in which hydrophilic or hydrophobic compounds are directly incorporated or dissolved [100]. They are prepared from oil-in-water emulsions made up of solid lipids with processing temperatures above the melting points of lipids [99]. Solid lipids used for the encapsulation of EOs in SLNs were glycerol monostearate and precirol [24, 101, 102]. SLNs encapsulating EOs were mainly stabilized by surfactant based carrier solutions as Tween 80, Span 80, Poloxamer 188 and Miranol Ultra C32 [13, 24, 54, 102]. Compared to liposomes and emulsions, SLNs presented more protection against different chemical reactions and a prolonged controlled release since the encapsulated bioactive compounds were immobilized in a solid matrix [103]. The formation of SLNs is simple and inexpensive [58]. However, they have shown under acidic conditions, poor stability, high tendency for aggregation and growth of particles during drying, possible gelation, low encapsulation load due to the crystalline structure and transitions in their fat crystalline structures during storage leading to explosions [58, 103].

Nanostructured lipid carriers

Nanostructured lipid carriers (NLCs) are a form modification of the SLNs and contain an inner phase made of a combination of lipids in both solid and liquid states where bioactive compounds are melted and/or solubilized. Five to 40% of the solid lipid phase in SNLs is replaced by liquid lipid in NLCs allowing a better solubilization of bioactive components [58, 103]. The main lipid carriers used for the formation of NLCs encapsulating EOs were cocoa butter, precirol, miglyol, virgin olive oil and sweet almond oil combined with surfactant solutions [104–107]. NLCs were developed to overcome the limitations of SNLs as they had a smaller size, presented higher loading capacity and prevented the formation of crystals and thus subsequent expulsion [103, 108]. However, before the formation of both SLNs and NLCs, lipids need to be melted at temperatures above those of the lipid melting temperature which might cause a degradation of heat-sensitive EO components [69]. Additionally, both SLNs and NLCs have a low stability under acidic conditions and their formation is not cost-effective at an industrial scale [69, 109].

As a summary for the carrier system forms used, capsules, hydrogels and emulsions were mainly used for the formation of both micro and nanoparticles with different types of carrier materials. They exhibited enhanced functional activities and controlled release when compared to EOs in their free native forms. Whereas, liposomes production was limited to lipid-based carrier materials and only nanosized particles were produced. In SLNs and NLCs, lipids combined with surfactant solutions were mainly used as carrier materials. The latter carrier systems have shown lower stability and higher production costs at industrial scale. However, compared to emulsions and liposomes, SLNs and NLCs showed better prolonged controlled and targeted release with higher encapsulation efficiency of bioactive compounds [109]. Thus, the selection of the carrier system form depends mainly on the entrapped bioactive compounds and the target of encapsulation.

Table 1 Appropriate encapsulation methods used to develop each form of carrier system

Encapsulation method	Different forms of carrier systems					
	Micro and nano			Nano		
	Capsules	Hydrogels	Emulsions	Liposomes	SLNs[a]	NLCs[b]
Spray drying	×		×			
Thin film hydration				×		
Extrusion	×	×				
Coacervation	×					
Ionic gelation	×	×				
Nanoprecipitation	×					
Emulsification (including high pressure homogenization, ultrasonication and microfluidization)	×	×	×	×	×	×

[a]SLNs: solid lipid nanoparticles

[b]NLCs: nanostructured lipid carriers

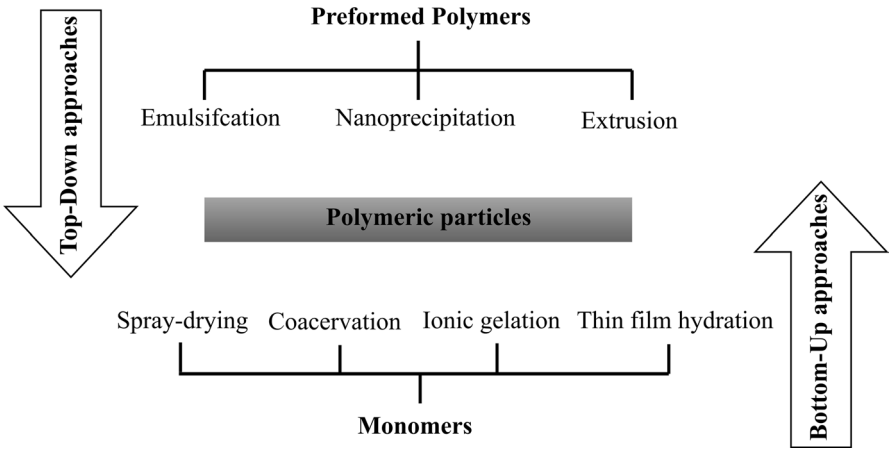

Fig. 3 Top-down and bottom-up approaches for the development of polymeric particles

Encapsulation methods

Various techniques have been developed for the encapsulation of bioactive compounds in different carrier system forms (Table 1). The selection of the encapsulation method is highly essential and is based on the physicochemical properties of the entrapped active compounds, carrier material types, and the intended application of particles [58, 110].

The encapsulation methods could be divided into top-down as well as bottom-up approaches (Fig. 3). The top-down approaches utilize high energy mechanical external tools to reduce the size and shape of the developed particles into smaller

dimensions [98]. Whereas in bottom-up approaches, low energy techniques are applied to build a molecular assembly through the association of different molecules or particles together [111, 112].

Nanoprecipitation

Nanoprecipitation, also known as solvent displacement method, relies on the addition of an organic phase containing the bioactive compound(s), organic solvent and the dissolved carrier material into a surrounding aqueous phase. Then, the carrier material immediately precipitates and the organic solvent diffuses into the aqueous phase and is then removed by evaporation (Fig. 4). PCL, PLA and PEO were used as carrier materials for the production of nanocapsules of thyme, oregano and lavender EOs by nanoprecipitation [70, 72]. This method was mainly used for the formation of nanocapsules for food and textiles applications.

Spray-drying

Spray-drying is one of the most common and adopted techniques used for the encapsulation of EOs. It is a mechanical dehydration method that converts a liquid into dried solid powder through a heating process. A primary emulsion mixture is formed and then atomized through a nozzle in a heated air chamber. The solvent will then evaporate rapidly when in contact with hot air, and thus fine solid droplets of bioactive compounds will be obtained [85, 113, 114] (Fig. 5). In fact, the drying conditions play a major role in the determination of the encapsulation efficiency and quality, thus an optimization of the process conditions is required to incorporate efficiently EOs and avoid their volatility [112, 115]. Eugenol, thymol, carvacrol, citral

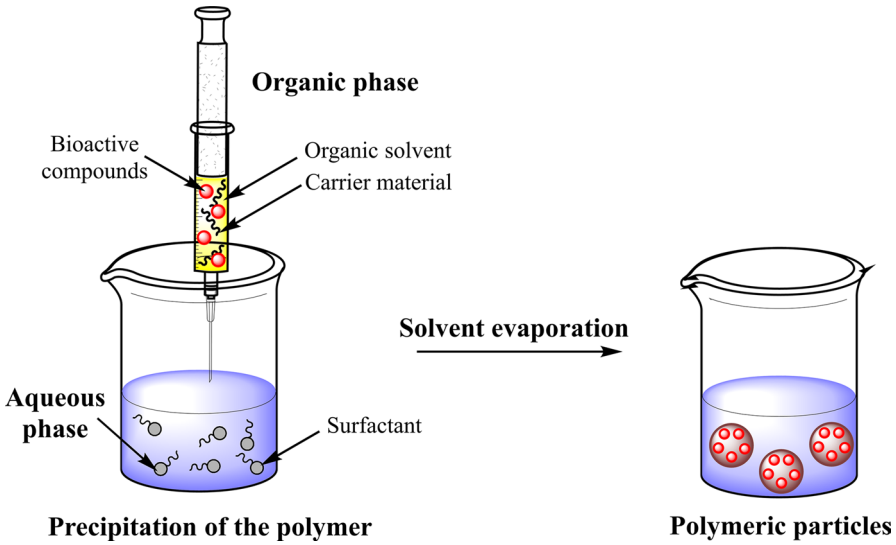

Fig. 4 Schematic representation of the nanoprecipitation technique

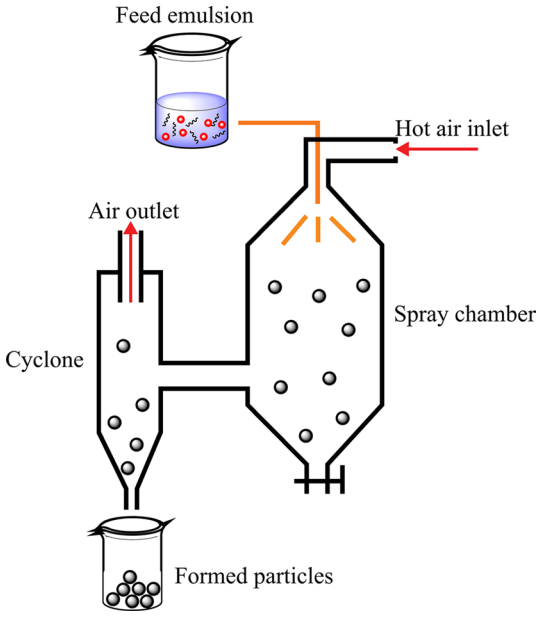

Fig. 5 Schematic representation of the spray-drying technique

and *Lippia siloides* EOs were encapsulated in micro and nanocapsules using spray-drying [78, 80, 81]. Also, *Cymbopogon citratus* emulsions were prepared using this technique [90]. Spray-drying was mainly used for the formation of emulsions and capsules used for food, nutraceutical and larvicidal applications. Only polysaccharides and proteins were used as carrier materials in this method.

Ionic gelation

Ionic gelation starts by the atomization or dripping of the carrier material solution into an ionic solution under constant agitation. Then, bioactive compounds are added and dissolved in the carrier solution. Drops of this solution reaching the ionic solution are converted to spherical gel particles [116] (Fig. 6). This method is based on the interaction between oppositely charged ions to form intramolecular and inter cross-linkages. By cross-linking, particles acquire a higher mechanical strength, stability and chemical resistance which causes a controlled release of the encapsulated compounds [117]. The entrapped active components are further released by gel phase changes triggered by different stimuli as osmotic or mechanical forces, enzymes and/or pH changes [118]. The main cross-linker agents used for the encapsulation of EOs using this technique were glutaraldehyde, transglutaminase, tripolyphosphate, calcium chloride, and formaldehyde. Only chitosan based carrier materials were used for the encapsulation of EOs using ionic gelation. *Carum copticum* hydrogels, peppermint, green tea, carvacrol and ginger nanocapsules were produced by ionic gelation and used in food, nutraceutical, cosmetic and medical applications [83, 86, 119, 120].

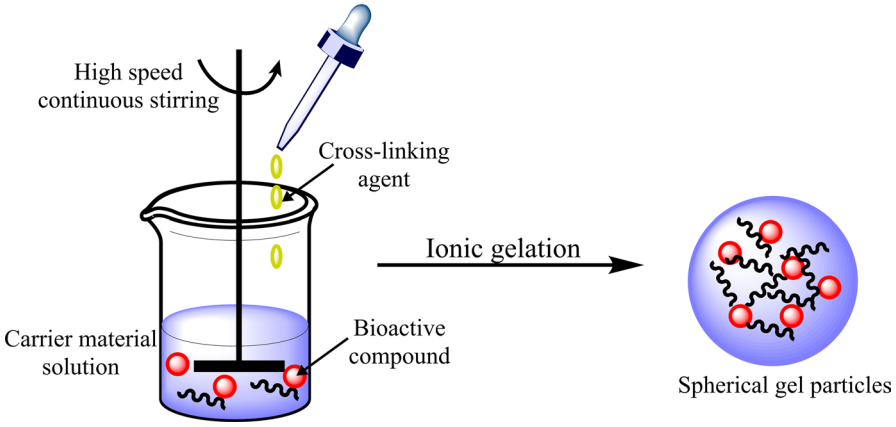

Fig. 6 Schematic representation of the ionic gelation method

Thin film hydration

Thin film hydration method was only used for the formation of liposomes. In this technique, a mixture of lipids (usually cholesterol, phosphatidylcholine or soy lecithin) is dissolved in an organic solvent which was further removed by rotary evaporation to form a dry lipid thin film [121]. The lipid film was then hydrated in an aqueous buffer solution under high agitation to form liposomes (Fig. 7). Although it is a simple technique, thin film hydration produces liposomes with low stability and wide size distribution [85]. This needs an exposure of the produced liposomes to further treatments as ultrasonication, freeze-thawing or extrusion in order to obtain

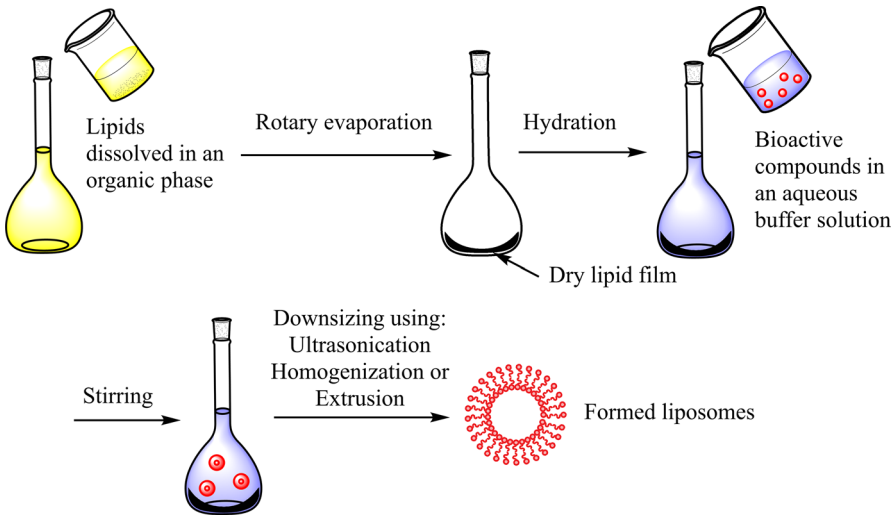

Fig. 7 Schematic representation of the different steps involved in the thin film hydration method

homogeneous particles with reduced size. Liposomes encapsulating clove, cinnamon, carvacrol, thymol and γ-terpinene using this method were mainly used in food, cosmetic and medical applications [55, 95, 97].

Emulsification

Emulsification is another common technique used to entrap bioactive components with both hydrophobic and hydrophilic properties [58]. Two immiscible liquid phases (dispersed and continuous) are homogenized to produce emulsions. In the dispersed phase, the bioactive compounds are entrapped, while the continuous phase protects the entrapped components from the external conditions (Fig. 8). Droplet particles are either directly used in the liquid state, or freezed and spray-dried to form solid powder particles. Different types of polysaccharide, protein, lipid-based and surfactant carrier materials were used for the encapsulation of EOs using this method. Emulsification includes high energy emulsification approaches as high pressure homogenization (HPH), ultrasonication and microfluidization [58]. HPH forms a liquid coarse emulsion by high shear mixer and then forces it through a narrow gap at high speed and pressure (100–2000 bar), causing the formation of smaller droplets [79, 85]. This technique is considered the most effective method for the production of SLNs and NLCs [24]. *Cymbopogon flexuosus* nano-emulsions, orange hydrogels, peppermint NLCs and citral SLNs were produced by HPH [24, 54, 60, 61, 91, 105]. In ultrasonication, high frequency ultrasonic waves (> 20 kHz) are generated after the immersion of a probe in a coarse emulsion to produce intensive disruptive forces leading to the formation of small liquid droplets with uniform size [79]. *Zataria multiflora* SLNs, Eucaplytus, oregano, *Nigella sativa* L. and *Thymus daenensis* nanoemulsions were formed using ultrasonication [22, 62, 66, 101, 122, 123]. In microfluidization, two flows of coarse emulsions pass under high pressure through a channel and then interact in a chamber where intensive disruptive forces are produced resulting in the formation of small emulsion droplets [58]. Lemongrass, thyme and sage micro and nanoemulsions were produced by microfluidization [124, 125]. In general, the above emulsification methods are highly reproducible at large scale with no use of toxic organic solvents or high temperatures. The formed nanoemulsions present a high kinetic stability due to their extreme small

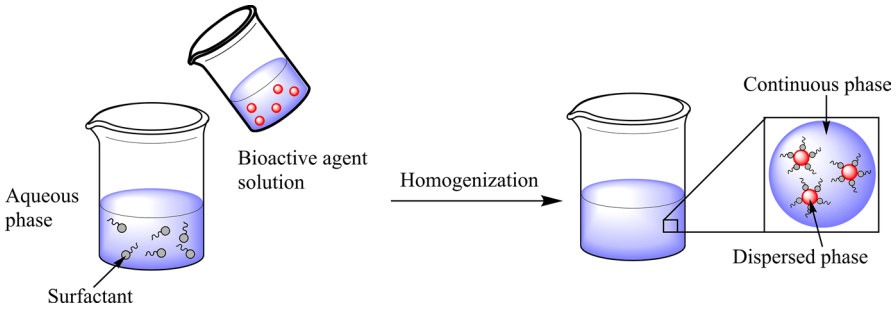

Fig. 8 Schematic representation of the emulsification technique

size which further exhibits a better retention of EOs content on the surface of the droplets [111]. However, these high energy emulsification methods require a lot of energy consumption and sophisticated equipment. On the other hand, the low energy spontaneous emulsification is more economic, simple and does not require the use of sophisticated special equipment. However, it produces unstable emulsions when exposed to chilling, heating, drying, and requires the use of high amounts of surfactants [58]. Several EOs were encapsulated in microcapsules (citronella, thymol, carvacrol…) and in micro and nanoemulsions (eugenol, carvacrol, cinnamon bark, *Pelargonium graveolens* and *Eucalyptus globulus*…) using spontaneous emulsification [63, 65, 82, 89, 126–128]. Low and high energy emulsification methods were the only techniques that produced all the different forms of carrier systems for the encapsulation of EOs.

Coacervation

The main objective of coacervation, also known as phase separation, is to produce particles based on the separation of two immiscible liquid phases in a colloidal solution [129]. Coacervation could be simple if it involves the phase separation of a single polymer dissolved in an aqueous or an organic phase, or complex if the separation occurs between a mixture of oppositely charged polymers dissolved in an aqueous phase [130, 131]. To induce phase separation in simple coacervation, water miscible non-solvent or inorganic salts are added [132]. Whereas for complex coacervation, temperature or pH changes cause electrostatic attraction between oppositely charged carrier materials leading to phase separation [133]. The two separate phases produced are a coacervate phase made of concentrated insoluble carriers and another dilute or equilibrium phase which is almost free from carrier materials and contains the solvent in which the coacervate is dispersed [71]. After phase separation, the newly formed coacervate is deposited around the bioactive compounds and a hardening agent, a cross-linker, is added to consolidate particles [71] (Fig. 9). PLGA, gelatin and gum Arabic carrier materials along with octamethylcyclotetrasiloxane (OCMTS), formaldehyde, transglutaminase, glutaraldehyde or tannic acid cross-linkers were used for the formation of thyme, citronella, *Zanthoxylum limonella* and lavender microcapsules by coacervation [14, 15, 68, 71]. Coacervation is a simple method that does not require the use of high temperatures or solvents, however, it has a difficulty to control particles size. The formed coacervates are stable

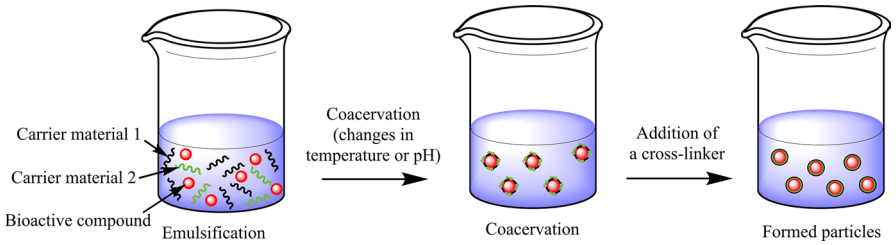

Fig. 9 Schematic representation of the different steps involved in the coacervation method

in a very narrow range of temperature and pH, and most cross-linking agents used are toxic chemicals that are forbidden in the food industry [134, 135]. As complex coacervation exhibits better functionalities and higher loading capacity than simple coacervation, it is considered a better choice for food and pharmaceutical applications [58].

Extrusion

In extrusion, a solution containing bioactive compounds and carrier material(s) passes to a gelling environment through a nozzle, syringe, pipette or atomizing disk for the solidification of particles at low temperatures [136]. The solution drops fall into a bath of a gelling agent leading to the formation of particles [137] (Fig. 10). This technique allows the encapsulation of both hydrophobic and hydrophilic heat sensitive compounds with no use of organic solvents [58, 138]. Cinnamon, thyme and clove microcapsules, in addition to thyme hydrogels were produced using this technique for pesticides and pharmaceutical applications [12, 87]. In previous studies, polysaccharides like alginate and sodium alginate, and proteins like soy proteins were the only carrier materials used in the extrusion process for the encapsulation of EOs [12, 87].

Combination of different methods

Several methods have been combined for EOs encapsulation. High energy emulsifications were the main methods combined with other techniques to provide particles with a smaller size. Microfluidization of thyme, lemongrass and sage coarse emulsions was done following HPH in order to reduce their size into nanoemulsions

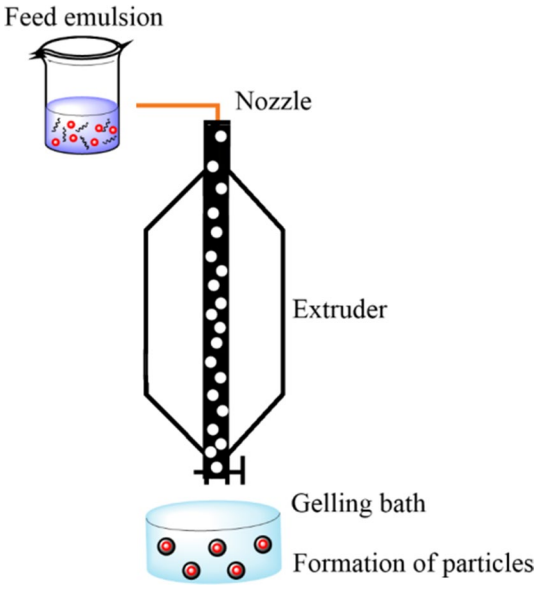

Fig. 10 Schematic representation of the extrusion technique for the encapsulation of essential oils

[125]. Cinnamon particles and citral coarse emulsions were also subject to ultrasonication after HPH to obtain smaller NLCs and nanoemulsions, respectively [88, 104]. Citral coarse emulsions formed by HPH were also spray-dried to produce microcapsules [77]. Carvacrol and thymol liposomes formed by thin film hydration were subject to ultrasonication to reduce and homogenize their sizes [96]. Thus, some combined encapsulation methods could be used to obtain targeted reduced size of particles.

Besides the various encapsulation methods proposed, none of them could be considered as an optimal absolute method for the encapsulation of bioactive compounds. The choice of the encapsulation method is related to the properties of the entrapped bioactive agents and carrier materials, the forms of carrier systems and the purpose of the encapsulation. The different advantages and limitations of each encapsulation method should be taken into account for choosing the ultimate method for the encapsulation of bioactive compounds (Table 2).

Carrier system forms could also predict the preferable encapsulation method, as some system forms are limited to specific formation methods. Emulsification was the only technique that is able to produce all types of carrier system forms. For EOs encapsulation, methods that use solvent evaporation and uncontrollable heating as thin film hydration should be avoided since EOs are heat-sensitive and volatile compounds [85]. For food applications, nanoprecipitation, thin film hydration and coacervation methods must be avoided as they use toxic organic solvents or toxic chemical cross-linkers. Additionally, the choice of the encapsulation method could be related to the intended purposes. For an enhanced stability, ionic gelation, spray-drying, nanoprecipitation, HPH, ultrasonication and thin film hydration techniques could maintain or improve the stability of the encapsulated EOs [64, 70, 72, 77, 83, 95, 107, 123]. While, extrusion and coacervation techniques did not ensure an enhanced stability of the encapsulated bioactive compounds, as the extruded particles had a large and porous structures and the coacervates were highly unstable in several conditions [58]. Regarding encapsulation efficiency and according to research studies discussed in this review, thin film hydration followed by ionic gelation methods exhibited the lowest encapsulation efficiencies of 4.16–29.2% and 4.7–45%, respectively [55, 86, 95, 97, 119, 120]. While higher encapsulation efficiency ranges of 70–96%, 55–99.6%, 84–99.84%, 57–98.2% and 72–94% were exhibited by nanoprecipitation, spray-drying, emulsification, coacervation and extrusion, respectively. The intended size of particles could also predict the choice of the encapsulation method as micro-sized particles were only formed by spray-drying and extrusion techniques. Whereas, nano-sized particles were formed by all the different encapsulation methods except of extrusion technique. Some of the studies reporting EOs encapsulation forms and methods used for different applications are presented in Table 3.

Physicochemical characteristics

After micro and nanoencapsulation of bioactive compounds, several physicochemical characteristics of the formed particles need to be evaluated as they may have

Table 2 Advantages and limitations of the most common methods used for the encapsulation of essential oils

Encapsulation methods	Advantages	Limitations	References
Nanoprecipitation	• Simple and fast	• High encapsulation efficiency only for lipophilic compounds	[69, 70, 72, 149]
	• Economic	• Mediated by organic solvents	
	• Good reproducibility		
	• High encapsulation efficiency		
Spray-drying	• Continuous and industrial scale production	• Uses high temperatures that can degrade heat sensitive products	[58, 112, 135, 138, 150]
	• Simple and fast	• Low product yield	
	• Economical		
	• Good stability		
	• Relatively high encapsulation efficiency		
Ionic gelation	• Good encapsulation efficiency with long term retention	• Particles with large size distribution	[112, 118, 151]
	• Simple and of low cost		
	• High production capacity		
	• Avoids the use of toxic organic solvents and high temperatures		
Thin film hydration	• Simple	• Particles with low stability and wide size distribution	[85, 152]
		• Not suitable for large scale productions	
Emulsification	• Simple	• Low stability	[150–152]
	• Low cost	• Poor controlled release	
	• Relatively high loading capacity	• Low control over particles' size and shape	
	• High throughput	• Low encapsulation efficiency	
Coacervation	• Simple	• Low control over particles' size	[51, 112, 133, 135, 150]
	• Cost effective	• Unsustained release of encapsulated agents	
	• High loading capacity	• Products' agglomeration	

Table 2 (continued)

Encapsulation methods	Advantages	Limitations	References
	• High reproducibility	• Low mechanical strength of particles walls	
	• No need for sophisticated equipment	• Uses toxic cross-linking agents	
	• Can be used at an industrial scale		
Extrusion	• Low operating temperatures	• Relatively large particles' diameter	[58, 115, 138, 151]
	• Easy to conduct in laboratories	• Porous particles	
	• Chemical and mechanical stability	• Limited types of carrier materials	
		• Relatively expensive	
		• Low production rate	

Table 3 Examples of different applications of essential oil formulations developed using several methods and carrier system forms

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
Nanocapsules	Ionic gelation	Chitosan + Tween 80/Tripolyphosphate	Peppermint (0.12–0.48 g), Green tea (0.12–0.48 g)	Food, Pharmaceutical	Improved antioxidant activity and lower minimum bacterial concentrations (MBC) against *Staphylococcus aureus* and *Escherichia coli* compared to their free forms	[83]
	Ionic gelation followed by Ultrasonication	Chitosan/Tripolyphosphate	Cardamon (10–25%)	Pharmaceutical	Excellent prolonged antimicrobial activity against *E. coli* and *S. aureus* for 7 days	[142]
	Nanoprecipitation	Polycaprolactone (PCL) + + Sorbitan monostearate + Polysorbate 80	Thyme (310 mg), Oregano (310 mg)	Food	Stable with varied time and temperature of storage, and lower minimum inhibitory concentration (MIC) values against *S. aureus* and *E. coli* compared to their free forms	[70]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
	Spray-drying	Whey protein isolate + Maltodextrin	Eugenol (20%)	Food (milk)	Enhanced antimicrobial activity against *E. coli* O157:H7 and *Listeria monocytogenes* in milk having three fat levels but not in tryptic soy broth	[42]
	Spray-drying	Zein + Casein	Eugenol (1 g), Thymol (1 g)	Food (milk)	Controlled and sustained release during 24h, with an improved long-term antimicrobial activity for 48h after encapsulating the mixture of EOs	[78]
	Nanoprecipitation	Polylactic acid (PLA) + Polyethylene oxide (PEO)	Lavender (0.4 µl/ml)	Textiles (footwear)	Stable up to 23 days under the optimal formation conditions with a controlled release of EO based on PLA molecular weight	[72]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
	Ionic gelation	Chitosan + Tween 80/ Pentasodium Tripolyphosphate or Sodium Hexametaphosphate	*Carum copticum* (0–0.625 g)	Nutraceutical, Cosmetic, Pharmaceutical	Sustained release and enhanced antimicrobial activity against *S. aureus, Staphylococcus epidermidis, Bacillus cereus, E. coli, Salmonella typhimurium* and *Proteus vulgaris*	[119]
	Spray-drying	Alginate + Cashew gum + Tween 80	*Lippia siloides*	Larvicide	Formulations with higher proportion of cashew gum showed a higher and faster release	[81]
	Ionic gelation	Chitosan + Tween 60/ Pentasodium Tripolyphosphate	Carvacrol (0–0.60 g)	Pharmaceutical, Biomedical, Cosmetic, Food	Inhibition of the growth of *B. cereus, S. aureus* and *E. coli* at MIC values with a faster release of carvacrol at acidic pH, followed by basic pH and then neutral solutions for up to 2 months	[120]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
Microcapsules	Emulsification	Chitosan + Coconut oil	Citronella	Healthcare	Slower release of EO with an increase in chitosan wall membrane thickness, and with a thermal treatment at 80 °C compared to 40 and 60 °C	[82]
	Extrusion	Sodium alginate/Calcium Chloride	Clove (10 g), Thyme (10 g), Cinnamon (10 g)	Pesticides	Prolonged antifungal activity after encapsulation without a significant difference compared to free EOs against *Aspergillus niger* and *Fusarium verticillioides*	[12]
	Emulsification	Polylactic-co-glycolic acid (PLGA) + Polyvinyl alcohol (PVA)	Thymol (20%)	Food	Higher release of thymol at a higher relative humidity with a complete inhibition of *S. aureus* and *E. coli* at 15 mg/mL	[128]
	Coacervation	Gelatin + Gum arabic/Glutaraldehyde, Transglutaminase or Tannic acid	Lavender (0–3%)	Perfumes	Optimization of the conditions for the preparation of microcapsules	[68]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
	Coacervation	PLGA + Tergitol/Octamethylcyclotetrasiloxane (OCMTS)	Thyme (0.2 ml)	Cosmetics	Potent antimicrobial activity against *P. aeruginosa* with a controlled release mechanism over 4 days	[71]
	Coacervation	Gelatin /Formaldehyde	Citronella	Larvicide	Controlled and sustained release profile	[15]
	Emulsification	Gum Arabic + Tween 20	Thymol (1–10%), Carvacrol (1–10%)	Food packaging	Controlled release and significant antimicrobial activity against *E. coli* O157:H7, *L. innocua, S. aureus* and *A. niger* at MIC values	[126]
	Coacervation	Gelatin + Tween 80/ Glutaraldehyde	*Zanthoxylum limonella* (3–15 ml)	Larvicidal	Controlled release of EO with different concentrations of carrier materials and cross-linkers	[14]
	High Pressure Homogenization (HPH) followed by Spray-drying	Maltodextrins, Sodium caseinate and/or Pectin	Citral (2.5–5.0%)	Food preservative	Improved stability of primary emulsions after the addition of a second layer of pectin to sodium caseinate	[77]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
	Spray-drying	Pectin + Alginate + Tween 80	Carvacrol	Food, Nutraceuticals	Encapsulation did not reduce nor improve the antioxidant activity and the antimicrobial activity of carvacrol against *E. coli*	[80]
Hydrogels	Ionic gelation	Chitosan + PVA + Soybean oil + Tween 80 + Span 80/ Sodium Tripolyphosphate	Ginger (1–5%)	Pharmaceutical (wound healing)	Prolonged and sustained release of EO over a period of 2880 min	[86]
	HPH	Whey protein isolate + Pectin + Tween 80	Orange (5%)	Flavor retention	Efficient encapsulation and retention of orange flavors	[60]
	Extrusion	Alginate + Soy protein / Calcium chloride dihyrate	Thyme (3.0g)	Pharmaceutical (drug release in gastrointestinal tract)	Controlled release of thyme	[87]
Emulsions	Spray-drying	Gum Arabic / Sodium Trimetaphosphate	*Cymbopogon citratus* (5%)	General applications	Formation of emulsions with optimal physical and chemical characteristics	[90]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
Microemulsions	Emulsification	Surfynol 485 W	Eugenol (0.9%) Carvacrol (0.7%)	Food	Significant reduction of *E. coli* O157:H7 and *L. monocytogenes* strain 101 biofilms and an inhibition of *L. monocytogenes* strain Scott A biofilms	[65]
	Emulsification	Soybean oil + Tween 80	Cinnamon bark (313–5000 ppm), Eugenol (313–5000 ppm) or Thyme (313–5000 ppm)	Food (milk)	Microemulsions of cinnamon bark showed better antimicrobial activity with lower MIC values when compared to microemulsions of eugenol and thyme	[89]
	Emulsification	Tween 20	*Cinnamomum cassia* (2.5%) and/or *Salvia officinalis* (5%)	Food contact surface (Stainless Steel)	Reduction of > 3 log of *S. aureus* ATCC 43387 biofilm grown in different culture media	[56]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
Micro and Nanoemulsions	Ultrasonication, HPH	Sodium alginate + Span 20 + Tween 20 + Tween 40 or Tween 80	*Cinnamomum zeylanicum* (1%) *and/or Piper nigrum* (1%)	Food	Ultrasonication produced emulsions with smaller droplets size and higher antimicrobial activity compared to HPH against *L. monocytogenes, Salmonella enterica, S. aureus* and *E. coli*	[39]
Nanoemulsions	Spontaneous emulsification,Microfluidization, Ultrasonication	Sodium alginate + Tween 80	Lemongrass (1%)	Food	Microfluidization achieved more log reductions of *E. coli* compared to spontaneous emulsification, while ultrasonication completely inhibited the antimicrobial activity	[124]
	Emulsification	Tween 80	*Citratus medica L. var. sarcodactylis* (10g)	Food (tofu)	Enhanced antioxidant activity and antimicrobial activity against *S. aureus*	[4]
	Ultrasonication	Tween 80	Oregano (0.05 or 0.1%)	Food	Significant antimicrobial activity against *E. coli* O157:H7, *S. typhimurium* and *L. monocytogenes*	[122]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
	Ultrasonication	Polysorbate 80	*Nigella sativa* L. (3%)	Therapeutic	Significant reduction of viable breast cancer cells and apoptosis to Michigan Cancer Foundation-7 (MCF-7) cancer cells	[22]
	HPH, Microfluidization	Sodium Alginate + Tween 80	Thyme (1%) Lemongrass (1%) Sage (1%)	Food	Significant decrease in *E. coli* cells after treatment with thyme nanoemulsions compared to sage nanoemulsions. Lemongrass nanoemulsions did not show any antimicrobial activity	[125]
	Ultrasonication	Tween 80	Eucalyptus (16.66%)	Food	100% inhibitory activity against *B. cereus*	[66]
	HPH	Span 80 + Tween 80	*Cymbopogon flexuosus* (5%)	Pharmaceutical	Enhanced antimicrobial activity against *Candida albicans*, *Cryptococcus grubii*, *S. aureus* and *P. aeruginosa* planktonic and sessile cells	[61]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
	Ultrasonication	Tween 80 + Lecithin	*Thymus daenensis* (2%)	Food	Stable over a 6-months period with an enhanced antimicrobial activity against *E. coli*	[64]
	Emulsification	Polysorbate 80 + Sorbitan monooleate	*Pelargonium graveolens* (5%)	Hospital catheters (polyethylene and polyurethane)	Enhanced antifungal activity against *Candida albicans, Candida tropicalis* and *Candida glabrata*	[63]
	Ultrasonication	Tween 80	*Ocimum basilicum* (6%)	Food	Stable for 1 month with an inhibition of *E. coli* viable cells	[62]
	Ultrasonication	Tween 80 + Lecithin	*Thymus daenensis* (2%)	Pharmaceutical	Stable over a 6-months period with lower MIC values against multi-drug resistant *Acinetobacter baumannii* and an enhanced inhibition of *A. baumannii* biofilms	[123]
	HPH	Span 80 + Tween 80	*Cymbopogon flexuosus* (5%)	Pharmaceutical	Enhanced antimicrobial activity against *Mycobacterium species* with a controlled release	[91]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
	HPH followed by Ultrasonication	Span 85 + Brij 97	Citral (10%)	Food, Cosmetics and Drug industry	Potent antimicrobial activity against *S. aureus, E. coli, P. aeruginosa, Enterococcus faecalis, S. typhimurium* and *L. monocytogenes*	[88]
	Emulsification	Span 80 + Tween 80	*Eucalyptus globulus* (5%)	Pharmaceutical	Efficient antifungal activity, while lower MIC values were reported for free EO compared to nanoemulsions against *Candida species*	[127]
Liposomes	Thin film hydration followed by Ultrasonication	Soy Phosphatidylcholine	Thymol (0.106 g), Carvacrol (0.106 g)	Food (milk)	Efficient antimicrobial activity while free EO components showed lower MIC values compared to liposomes against *S. aureus* and *Salmonella*	[96]
	Thin film hydration	Soy lecithin + Cholesterol	Clove (2–6 mg/ml)	Food (tofu)	Stable and narrow size distribution with a significant reduction of *S. aureus*	[95]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
	Thin film hydration	Soy lecithin + Cholesterol	Cinnamon (0.5–8 mg/ml)	Therapeutic	Enhanced antibiofilm activity against methicillin-resistant *S. aureus* on stainless style, nylon, gauze and non-woven fabrics	[55]
	Thin film hydration	Phospholipon 90G + Cholesterol	*Salvia tribola* (5–20 µl/ml), *Rosmarinus officinalis* (5–20 µl/ml)	General application	Enhanced antioxidant, antiinflammatory and antimicrobial activity against *Klebsiella pneumoniae*	[11]
	Thin film hydration	L-α-phosphatidylcholine + Cholesterol	Carvacrol (4 ml), Thymol (4 ml), γ-Terpinene (4 ml)	Food, Cosmetics, Medical industries	Enhanced antioxidant and antimicrobial activities	[97]
Solid Lipid Nanoparticles (SLNs) + Emulsions	HPH	Poloxamer 188 or Miranol Ultra C32	*Artemisia arborescens* (1.0%)	Pesticides	Lower evaporation with decreased cumulative release of EOs from SLNs	[13]
SLNs	HPH	Tween 80 + Span 80	Citral (0.2–5%)	Beverages (orange)	Excellent sustained release properties and protection from degradation and formation of off-flavors	[54]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
	HPH	Glycerol Monostearate (Imwitor 900 K) + Poloxamer 188	Citral (1%)	Pharmaceutical	Optimization of the SLN formulation with a long term stability at room temperature	[24]
	HPH followed by Ultrasonication	Glyceryl Monostearate + Precirol + Tween 80 and/or Poloxamer 188	*Zataria multiflora* (0.03%)	Agriculture	Enhanced antifungal activity against *Aspergillus* species, *Rhizophus stolonifer, Rhizoctone solani* and *Alternia solani*	[102]
	Ultrasonication	Glyceryl Monostearate + Precirol + Stearic acid + Tween 80 and/or Poloxamer 188	*Zataria multiflora*	Food, Medicine, Agriculture industries	Stability over 4-months period	[101]
Nanostructured Lipid Carriers (NLCs) + Emulsions	Emulsification	Precirol + Miglyol + Tween 80 + Poloxamer	Citral (200 mg)	Food preservative	Enhanced antimicrobial activity compared to emulsions against *S. aureus*, *B. cereus* and *E. coli* with a relative stability for up to 90 days of storage	[107]

Table 3 (continued)

Carrier system form	Encapsulation methods	Carrier materials/cross-linkers	Essential oil (EO) formulations	Applications	Main findings	References
NLCs	Emulsification followed by HPH and Ultrasonication	Cocoa butter + Virgin olive oil + Tween 80	Cardamon (275–4400 µg/ml)	Food supplements	Enhanced antimicrobial activity against *E. coli* and *S. aureus*	[106]
	Emulsification followed by HPH	Precirol + Miglyol + Poloxamer	Peppermint (200 mg)	Pharmaceutical (wound healing)	Potent antimicrobial activity with lower MIC values against *S. typhimurium, P. aeruginosa* and *L. monocytogenes* compared to their free forms. While against *E. coli, S. aureus* and *S. epidermidis* free EO showed lower MIC values compared to NLCs. Both free EO and NLCs had similar MIC values against *Bacillus anthracis* and *Streptococcus pneumonia*	[105]
	HPH, Ultrasonication	Cocoa butter + Almond oil + Chitosan + Tween 80	Cinnamon (100 mg)	Food (milk)	Enhanced antioxidant activity	[104]

an impact on the efficacy, physical stability and release mechanisms of bioactive agents [139].The most important physicochemical characteristics include size, size distribution, surface charge and morphology of the particles which depend on several external factors like pH, relative humidity and temperature, in addition to the processing operations and materials [133].

Size and size distribution

Particles size and their size distribution dictate the stability of particles over a period of time. A decrease in particles size increases the surface to volume ratio which further enhances the bioavailability and the different functionalities of the encapsulated EOs and decreases the attractive forces between droplets leading to a better stability [66, 102, 139]. The degradation rate of a carrier material is also affected by the size of particles, as an increase in particle size increases the degradation rate of particles walls [59]. In the studies reported in this review, the size of micro and nanoparticles ranged between 1.02–1880 μm and 10–811 nm, respectively.

Polydispersity index (PDI) is a measure of the particles size distribution which also predicts the stability of particles. PDI values vary between 0 and 1, with low values near zero indicating a narrow size and stable distribution of the particles. A PDI below 0.3 reveals the uniformity and homogeneity of particles [61]. Most of literature studies, reported stable particles as PDI values were below 0.3, except few studies where PDI values were between 0.358 and 0.726 indicating the non-uniformity and non-stability of the developed particles [39, 54, 102, 107, 125]. Dynamic light scattering (DLS) was the most common method used for the measurement of particle size and PDI [56, 61, 62, 64, 91, 95, 96, 102, 123, 127]. Other techniques were also used as photon correlation spectroscopy (PCS) and laser diffraction instrument [13, 77, 140].

Surface charge (zeta potential)

In an ionic solution, most particles have a layer of oppositely charged ions. When particles diffuse into a solution, this layer comes in contact with a second outer layer composed of loosely associated ions to form an electrical double layer [141]. The potential measured at the surface of this double electric layer is known as zeta potential. It is reported that zeta potential values above 30 mV (either positive or negative) indicate a good electrical stability and promote more repulsive interactions avoiding the aggregation or collision between particles [59, 83, 96, 123]. All studies indicating zeta potential values above absolute 30 mV showed a long term stability with no aggregation of particles encapsulating EOs [13, 39, 55, 81, 95, 96, 102, 123, 125, 142]. Whereas studies showing zeta potential values below absolute 30 mV presented a lower and shorter term stability of particles with a faster aggregation [61, 83, 88, 105, 120]. Zeta potential measurements were done using zeta sizer [13, 39, 77, 81, 95, 142], phase-analysis light scattering (PALS) [124, 125], and electrophoretic mobility technique [61, 70, 91, 127].

Morphology and structure

The developed particles morphology refers to their external as well as their internal structures that depend highly on the carrier materials used and on the operating conditions. Usually particles encapsulating bioactive agents are spherical but they may present some other shapes as cylindrical, ellipsoid or irregular. The shape of particles might have an impact on the stability, aggregation, optical and release characteristics of the encapsulated compounds [99]. Different microscopic techniques as phase contrast microscopy [66], atomic force microscopy (AFM) [55, 63, 66, 78, 142], scanning electron microscopy (SEM) [143–145], and transmission electron microscopy (TEM) [61, 62, 91, 102, 123] were used for the determination of particles topography, morphology and structure. In addition to the visualization of particles shape, the different microscopic techniques allowed an observation of the presence or absence of aggregations or fusion between particles [146].

Factors affecting essential oils release

Delivery systems release the entrapped bioactive compounds at appropriate time and site, in response to particular triggers. pH, relative humidity, temperature, particles' size, carrier systems forms, ratio and properties of carrier materials and cross-linking agents are considered the main factors that may alter the release of the encapsulated compounds.

It is shown that pH changes alter the charge of particles encapsulating EOs [119]. In several studies, as pH changes, the degree of ionization of the different functional groups of carrier materials changes leading to charge modification. For example, at acidic pH (pH = 3), nanocapsules of peppermint, green tea, *Carum copticum* and carvacrol showed a significant higher release of bioactive compounds when compared to the release at higher pH values (between 7 and 11) [83, 119, 120]. The higher release of EOs in acidic medium was attributed to the ionic repulsion between the protonated amino groups (NH_3^+) on the neighboring chitosan chains of carrier materials, increasing the surface area of nanocapsules exposed to the external medium. Whereas at neutral and basic pH values, the deprotonation of NH_3^+ groups on chitosan chains caused more aggregation between particles lowering the release rate of the encapsulated active components [120]. Also, the electrostatic attraction between combined carrier systems is related to pH changes [60]. At pH values above the proteins' isoelectric point (pI), most of amino and carboxyl groups are deprotonated resulting in net negative charges. Whereas below pI of proteins, most of these groups are protonated leading to net positive charges [60]. Thus, in systems based on a combination of proteins with other carrier materials, the attraction or repulsion between carriers is related to the pI of proteins and pH values. As an example, when proteins are positively charged (pH below pI of proteins), they will be attracted to oppositely charged polysaccharides and thus bioactive compounds will be retained inside the carrier system. Whereas, negatively charged proteins are repulsed from anionic polysaccharides leading to the release of bioactive compounds [99, 147].

Relative humidity (RH) was also found to be related to the release of bioactive compounds from carrier systems. For example, with an increase in RH from 50 to 90%, the release of thymol from PLGA microcapsules increased from 41.54 to 61.13% [128]. This increased release was mainly related to the increase in water absorption by microcapsules causing a faster degradation of carrier material and thus a greater release of the entrapped components [128].

Temperature can also alter carrier materials contraction and mobility thus affecting the release of the entrapped bioactive compounds. Based on the carrier material type and the applied temperature, different release rates have been observed. As for example, a gradual increase in environmental temperature from 40 to 80 ºC, caused a contraction of chitosan polymeric chains, a reduction of the space between particles' pores and thus an inhibition of the release of citronella EO from microcapsules [82]. On the contrary, the release of thymol from PLGA microcapsules was greater at 25 ºC than at 4 ºC [128]. This was mainly explained by the mobility of the carrier material at higher temperatures ensuring a higher release of EOs.

The bioactive compounds release may also be related to the carrier materials and to the cross-linkers properties and ratios. An increase in chitosan carrier concentration from 0.5 to 1.5% decreased the release rate of citronella from microcapsules [82]. This was accounted for the increase in thickness of the surrounding membrane and subsequent reduction in pores sizes between chitosan molecules. Also, *Zanthoxylum limonella* oil release rate from microcapsules decreased when both the gelatin carrier amount and the molecular concentration of glutaraldehyde cross-linker increased [14]. An increase in PLA molecular weight from 4.5 to 10 KDa increased the release of lavender oil by 5 to 40% from nanocapsules [72]. Capsules made of PLA with higher molecular weight of 10 KDa were smaller and thus increased the release due to the higher surface area [72]. The ratio of alginate to cashew gum influenced also the release rate of *Lippia siloides* EO from nanocapsules. A 1:3 alginate/cashew gum ratio increased the release while a 3:1 ratio decreased the release of EO. Cashew gum was found to increase the hydrophilic properties of particles and thus increase EOs release, whereas alginate provided a more complex structure retaining EOs [81]. In addition, the bioactive compounds release was found to be related to particles size. It was reported that a decrease in microcapsules size from 225 to 11 μm, increased the release rate of citronella from approximately 22 to 50% [82]. This was mainly explained by the increase in total surface area of smaller particles allowing a faster release of bioactive components from microcapsules [82]. Controlled release of bioactive compounds was additionally found to be related to the carrier system form. SLNs and NLCs showed the most prolonged controlled release of EOs as bioactive compounds were entrapped within solid lipid carrier materials retarding their release.

Physicochemical release mechanisms

An effective encapsulation ensures a protection of the encapsulated bioactive compounds from the external conditions. After a specific trigger condition, bioactive compounds will be released at an appropriate time, concentration and speed. Several

factors affect the release of encapsulated bioactive compounds including the interactions and ratio between carrier and core materials, the size as well as the viscosity of the developed particles [115]. When exposed to environmental trigger conditions, the release of encapsulated EOs from carrier systems is regulated by one or more of the following physicochemical mechanisms:

Diffusion

Diffusion is the most common release mechanism of bioactive compounds from the different carrier systems [108]. The diffusion of molecules is driven by the concentration gradient from medium of high concentration to medium of lower concentration. Generally, in diffusion-controlled release mechanisms, there is a high initial release followed by a decrease in diffusion as the distance of bioactive compounds to the surface of particles increases. Diffusion rate depends on the size of the entrapped molecules, the thickness and molecular weight of the carrier material and the medium of dispersion. Larger molecules diffuse slower than smaller ones, a thick membrane wall will also retard the release of components, and the diffusion rate is slowed in a viscous medium.

Swelling

Swelling release mechanism mainly occurs in hydrophilic based carriers like proteins and polysaccharides and particularly in hydrogels that have the ability to absorb a great amount of water [108]. Some environmental conditions may cause changes in the repulsive or attractive interactions between polymeric carrier materials leading to pores size changes and swelling of particles as a result of the absorption of fluids from the surrounding medium [99]. The increase in volume due to swelling, increases pores size and thus the entrapped active components will be released from particles by simple diffusion.

Dissolution or melting

Water-soluble carrier materials are easily dissolved in the presence of moisture or a suitable solvent, whereas lipid-based carriers release compounds upon melting by heating [79]. Once the surrounding carrier is dissolved or melted, bioactive components are released and come in contact with the external medium. The release rate depends on the thickness and nature of the carrier material [148].

Degradation

Particles made of biodegradable carriers as proteins, polysaccharides and lipids will eventually undergo enzymatic degradation under particular environmental conditions [79, 108]. As the carrier material degrades, the surrounding medium enters inside particles and leads to the release of bioactive compounds from the entrapped inner core.

Conclusions and future perspectives

A growing interest in EOs as potent biosourced and ecofriendly alternatives to the synthetic sanitizers has been evident during the last years due to their different physicochemical, biological, and functional properties. Micro and nanoencapsulation of EOs were investigated as novel promising delivery systems that could offer several benefits and overcome the different limitations of free EOs. Successful encapsulation of EOs ultimately depends on the intended purposes and applications, in addition to the selection of the carrier materials types, forms and development methods. The combination of appropriate GRAS, biodegradable and non-toxic carrier materials was found to offer advantageous formulations for the encapsulation of EOs. Several carrier system forms and encapsulation methods have highlighted the feasibility to produce micro and nano-sized stable EO particles enabling their integration and application in several fields without causing detrimental effects. SLNs and NLCs present outstanding controlled release properties but with higher production costs and lower particles stability. Whereas, the formation of capsules, hydrogels and emulsions is inexpensive and able to enhance the different functional activities of EOs but presents lower controlled release properties. It was also shown in this review to preferably avoid thin film hydration, nanoprecipitation and coacervation methods for the encapsulation of EOs as they require the use of organic solvents.

Beside the numerous advantages of encapsulation, it has raised a number of regulatory, safety and environmental issues concerning its impact on the environment and on human health. Their safety aspects are still unclear till present and require further exploration as no specific international legislation has been applied. Thus future studies should emphasize (i) the safety aspects and risk assessments of the use of micro and nanoencapsulation of EOs in several applications, (ii) the improvement of existing encapsulation methods and their production on an industrial scale, (iii) the optimization of factors affecting the release of bioactive compounds for an enhanced and more pronounced activity, and (iv) the exploration of further direct applications of EOs particles particularly in biological and food systems.

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Declarations

Conflict of interest The authors have no relevant financial or non-financial interests to disclose.

References

1. da Silva BD, Bernardes PC, Pinheiro PF, Fantuzzi E, Roberto CD (2021) Chemical composition, extraction sources and action mechanisms of essential oils: natural preservative and limitations of use in meat products. Meat Sci 176:108463. https://doi.org/10.1016/j.meatsci.2021.108463
2. Sharmeen JB, Mahomoodally FM, Zengin G, Maggi F (2021) Essential oils as natural sources of fragrance compounds for cosmetics and cosmeceuticals. Molecules 26:666. https://doi.org/10.3390/molecules26030666
3. Ahmed AF, Attia FAK, Liu Z, Li C, Wei J, Kang W (2019) Antioxidant activity and total phenolic content of essential oils and extracts of sweet basil (*Ocimum basilicum* L.) plants. Food Sci Human Wellness 8:299–305. https://doi.org/10.1016/j.fshw.2019.07.004
4. Lou Z, Chen J, Yu F, Wang H, Kou X, Ma C (2017) The antioxidant, antibacterial, antibiofilm activity of essential oil from *Citrus medica* L. var. sarcodactylis and its nanoemulsion. LWT 80:371–377. https://doi.org/10.1016/j.lwt.2017.02.037
5. Marín I, Sayas-Barberá E, Viuda-Martos M, Navarro C, Sendra E (2016) Chemical composition, antioxidant and antimicrobial activity of essential oils from organic fennel, parsley, and lavender from Spain. Foods 5:18. https://doi.org/10.3390/foods5010018
6. Petretto GL, Maldini M, Addis R, Chessa M, Foddai M, Rourke JP, Pintore G (2016) Variability of chemical composition and antioxidant activity of essential oils between Myrtus communis var. Leucocarpa DC and var. Melanocarpa DC. Food Chem 197:124–131. https://doi.org/10.1016/j.foodchem.2015.10.056
7. Afoulous S, Ferhout H, Raoelison EG, Valentin A, Moukarzel B, Couderc F, Bouajila J (2013) Chemical composition and anticancer, antiinflammatory, antioxidant and antimalarial activities of leaves essential oil of Cedrelopsis grevei. Food Chem Toxicol 56:352–362. https://doi.org/10.1016/j.fct.2013.02.008
8. Avoseh ON, Oyedeji OOO, Aremu K, Nkeh-Chungag BN, Songca SP, Oluwafemi SO, Oyedeji AO (2015) Chemical composition and anti-inflammatory activities of the essential oils from *Acacia mearnsii* de Wild. Nat Prod Res 29:1184–1188. https://doi.org/10.1080/14786419.2014.983504
9. Coté H, Boucher MA, Pichette A, Legault J (2017) Anti-Inflammatory, antioxidant, antibiotic, and cytotoxic activities of *Tanacetum vulgare* L. essential oil and its constituents. Medicines 4:34. https://doi.org/10.3390/medicines4020034
10. Loizzo MR, Menichini F, Conforti F et al (2009) Chemical analysis, antioxidant, antiinflammatory and anticholinesterase activities of Origanum ehrenbergii Boiss and *Origanum syriacum* L. essential oils. Food Chem 117:174–180. https://doi.org/10.1016/j.foodchem.2009.03.095
11. Risaliti L, Kehagia A, Daoultzi E et al (2019) Liposomes loaded with Salvia triloba and Rosmarinus officinalis essential oils: In vitro assessment of antioxidant, antiinflammatory and antibacterial activities. J Drug Deliv Sci Technol 51:493–498. https://doi.org/10.1016/j.jddst.2019.03.034
12. Soliman EA, El-Moghazy AY, El-Din MSM, Massoud MA (2013) Microencapsulation of essential oils within alginate: formulation and in vitro evaluation of antifungal activity. JEAS 3:48–55. https://doi.org/10.4236/jeas.2013.31006
13. Lai F, Wissing SA, Müller RH, Fadda AM (2006) *Artemisia arborescens* L. essential oil-loaded solid lipid nanoparticles for potential agricultural application: preparation and characterization. AAPS PharmSciTech 7:E10. https://doi.org/10.1208/pt070102
14. Maji T, Baruah I, Dube S, Hussain M (2007) Microencapsulation of Zanthoxylum limonella oil (ZLO) in glutaraldehyde crosslinked gelatin for mosquito repellent application. Biores Technol 98:840–844. https://doi.org/10.1016/j.biortech.2006.03.005
15. Solomon B, Sahle FF, Gebre-Mariam T, Asres K, Neubert RHH (2012) Microencapsulation of citronella oil for mosquito-repellent application: formulation and in vitro permeation studies. Eur J Pharm Biopharm 80:61–66. https://doi.org/10.1016/j.ejpb.2011.08.003
16. Amaral VCS, Santos PR, da Silva AF, dos Santos AR, Machinski M, Mikcha JMG (2015) Effect of carvacrol and thymol on *Salmonella* spp. biofilms on polypropylene. Int J Food Sci Technol 50:2639–2643. https://doi.org/10.1111/ijfs.12934
17. de Oliveira MMM, Brugnera DF, do Nascimento JA, Batista NN, Piccoli RH (2012) Cinnamon essential oil and cinnamaldehyde in the control of bacterial biofilms formed on stainless steel surfaces. Eur Food Res Technol 234:821–832. https://doi.org/10.1007/s00217-012-1694-y

18. dos Santos Rodrigues JB, de Carvalho RJ, de Souza NT et al (2017) Effects of oregano essential oil and carvacrol on biofilms of *Staphylococcus aureus* from food-contact surfaces. Food Control 73:1237–1246. https://doi.org/10.1016/j.foodcont.2016.10.043
19. Nostro A, Roccaro AS, Bisignano G et al (2007) Effects of oregano, carvacrol and thymol on *Staphylococcus aureus* and *Staphylococcus epidermidis* biofilms. J Med Microbiol 56:519–523. https://doi.org/10.1099/jmm.0.46804-0
20. Sadekuzzaman M, Mizan MFR, Kim HS, Yang S, Ha SD (2018) Activity of thyme and tea tree essential oils against selected foodborne pathogens in biofilms on abiotic surfaces. LWT 89:134–139. https://doi.org/10.1016/j.lwt.2017.10.042
21. Yammine J, Gharsallaoui A, Fadel A, Mechmechani S, Karam L, Ismail A, Chihib NE (2022) Enhanced antimicrobial, antibiofilm and ecotoxic activities of nanoencapsulated carvacrol and thymol as compared to their free counterparts. Food Control 143:109317. https://doi.org/10.1016/j.foodcont.2022.109317
22. Periasamy VS, Athinarayanan J, Alshatwi AA (2016) Anticancer activity of an ultrasonic nanoemulsion formulation of *Nigella sativa* L. essential oil on human breast cancer cells. Ultrason Sonochem 31:449–455. https://doi.org/10.1016/j.ultsonch.2016.01.035
23. Yang C, Chen H, Chen H, Zhong B, Luo X, Chun J (2017) Antioxidant and anticancer activities of essential oil from Gannan Navel orange peel. Molecules 22:1391. https://doi.org/10.3390/molecules22081391
24. Zielińska A, Martins-Gomes C, Ferreira NR, Silva AM, Nowak I, Souto EB (2018) Anti-inflammatory and anti-cancer activity of citral: optimization of citral-loaded solid lipid nanoparticles (SLN) using experimental factorial design and LUMiSizer. Int J Pharm 553:428–440. https://doi.org/10.1016/j.ijpharm.2018.10.065
25. Hui X, Yan G, Tian FL, Li H, Gao GY (2017) Antimicrobial mechanism of the major active essential oil compounds and their structure–activity relationship. Med Chem Res 26:442–449. https://doi.org/10.1007/s00044-016-1762-0
26. Kavanaugh NL, Ribbeck K (2012) Selected antimicrobial essential oils eradicate *Pseudomonas* spp. and *Staphylococcus aureus* biofilms. Appl Environ Microbiol 78:4057–4061. https://doi.org/10.1128/AEM.07499-11
27. Teixeira RF, Balbinot Filho CA, Borges CD (2022) Essential oils as natural antimicrobials for application in edible coatings for minimally processed apple and melon: a review on antimicrobial activity and characteristics of food models. Food Packaging and Shelf Life 31:100781. https://doi.org/10.1016/j.fpsl.2021.100781
28. Turek C, Stintzing FC (2013) Stability of essential oils: a review. Comp Rev Food Sci Food Saf 12:40–53. https://doi.org/10.1111/1541-4337.12006
29. Turek C, Stintzing FC (2011) Evaluation of selected quality parameters to monitor essential oil alteration during storage. J Food Sci 76:1365–1375. https://doi.org/10.1111/j.1750-3841.2011.02416.x
30. Rowshan V, Bahmanzadegan A, Saharkhiz MJ (2013) Influence of storage conditions on the essential oil composition of Thymus daenensis Celak. Ind Crops Prod 49:97–101. https://doi.org/10.1016/j.indcrop.2013.04.029
31. Sharma S, Kumar R (2016) Effect of temperature and storage duration of flowers on essential oil content and composition of damask rose (Rosa×damascena Mill.) under western Himalayas. J Appl Res Med Aromatic Plants 3:10–17. https://doi.org/10.1016/j.jarmap.2015.10.001
32. Christensson JB, Forsström P, Wennberg AM, Karlberg AT, Matura M (2009) Air oxidation increases skin irritation from fragrance terpenes. Contact Dermatitis 60:32–40. https://doi.org/10.1111/j.1600-0536.2008.01471.x
33. Najafian S (2014) Storage conditions affect the essential oil composition of cultivated Balm Mint Herb (Lamiaceae) in Iran. Ind Crops Prod 52:575–581. https://doi.org/10.1016/j.indcrop.2013.11.015
34. Nguyen H, Campi EM, Roy Jackson W, Patti AF (2009) Effect of oxidative deterioration on flavour and aroma components of lemon oil. Food Chem 112:388–393. https://doi.org/10.1016/j.foodchem.2008.05.090
35. Misharina TA, Polshkov AN, Ruchkina EL, Medvedeva IB (2003) Changes in the composition of the essential oil of Marjoram during storage. Appl Biochem Microbiol 39:311–316
36. Turek C, Stintzing FC (2012) Impact of different storage conditions on the quality of selected essential oils. Food Res Int 46:341–353. https://doi.org/10.1016/j.foodres.2011.12.028

37. Misharina TA, Polshkov AN (2005) Antioxidant properties of essential oils: autoxidation of essential oils from Laurel and Fennel and of their mixtures with essential oil from Coriander. Appl Biochem Microbiol 41:610–618. https://doi.org/10.1007/s10438-005-0111-8
38. Tomaino A, Cimino F, Zimbalatti V, Venuti V, Sulfaro V, De Pasquale A, Saija A (2005) Influence of heating on antioxidant activity and the chemical composition of some spice essential oils. Food Chem 89:549–554. https://doi.org/10.1016/j.foodchem.2004.03.011
39. Jiménez M, Domínguez JA, Pascual-Pineda LA, Azuara E, Beristain CI (2018) Elaboration and characterization of O/W cinnamon (Cinnamomum zeylanicum) and black pepper (Piper nigrum) emulsions. Food Hydrocolloids 77:902–910. https://doi.org/10.1016/j.foodhyd.2017.11.037
40. Liang H, Yuan Q, Vriesekoop F, Lv F (2012) Effects of cyclodextrins on the antimicrobial activity of plant-derived essential oil compounds. Food Chem 135:1020–1027. https://doi.org/10.1016/j.foodchem.2012.05.054
41. Niu F, Pan W, Su Y, Yang Y (2016) Physical and antimicrobial properties of thyme oil emulsions stabilized by ovalbumin and gum arabic. Food Chem 212:138–145. https://doi.org/10.1016/j.foodchem.2016.05.172
42. Shah B, Davidson PM, Zhong Q (2013) Nanodispersed eugenol has improved antimicrobial activity against *Escherichia coli* O157:H7 and *Listeria monocytogenes* in bovine milk. Int J Food Microbiol 161:53–59. https://doi.org/10.1016/j.ijfoodmicro.2012.11.020
43. Singh A, Singh RK, Bhunia AK, Singh N (2003) Efficacy of plant essential oils as antimicrobial agents against *Listeria monocytogenes* in hotdogs. LWT Food Sci Technol 36:787–794. https://doi.org/10.1016/S0023-6438(03)00112-9
44. Gutierrez J, Barry-Ryan C, Bourke P (2008) The antimicrobial efficacy of plant essential oil combinations and interactions with food ingredients. Int J Food Microbiol 124:91–97. https://doi.org/10.1016/j.ijfoodmicro.2008.02.028
45. Cava R, Nowak E, Taboada A, Marin-Iniesta F (2007) Antimicrobial activity of clove and cinnamon essential oils against *Listeria monocytogenes* in pasteurized milk. J Food Prot 70:2757–2763. https://doi.org/10.4315/0362-028X-70.12.2757
46. Rattanachaikunsopon P, Phumkhachorn P (2010) Assessment of factors influencing antimicrobial activity of carvacrol and cymene against *Vibrio cholerae* in food. J Biosci Bioeng 110:614–661. https://doi.org/10.1016/j.jbiosc.2010.06.010
47. Hyldgaard M, Mygind T, Meyer RL (2012) Essential oils in food preservation: Mode of action, synergies, and interactions with food matrix components. Front. Microbiol 3. https://doi.org/10.3389/fmicb.2012.00012
48. Avila Gandra E, Radünz M, Helbig E, Dellinghausen Borges C, Kuka Valente Gandra T (2018) A mini-review on encapsulation of essential oils. J Anal Pharm Res 7:00205. https://doi.org/10.15406/japlr.2018.07.00205
49. Smaoui S, Hsouna AB, Lahmar A et al (2016) Bio-preservative effect of the essential oil of the endemic Mentha piperita used alone and in combination with BacTN635 in stored minced beef meat. Meat Sci 17:196–204. https://doi.org/10.1016/j.meatsci.2016.03.006
50. Hayouni EA, Chraief I, Abedrabba M, Bouix M, Leveau JY, Mohammed H, Hamdi M (2008) Tunisian *Salvia officinalis* L. and *Schinus molle* L. essential oils: their chemical compositions and their preservative effects against *Salmonella* inoculated in minced beef meat. Int J Food Microbiol 125:242–251. https://doi.org/10.1016/j.ijfoodmicro.2008.04.005
51. Aguiar J, Estevinho BN, Santos L (2016) Microencapsulation of natural antioxidants for food application: the specific case of coffee antioxidants—a review. Trends Food Sci Technol 58:21–39. https://doi.org/10.1016/j.tifs.2016.10.012
52. Chouhan S, Sharma K, Guleria S (2017) Antimicrobial Activity of some essential oils—present status and future perspectives. Medicines 4:58. https://doi.org/10.3390/medicines4030058
53. de Souza SL, Madalena DA, Pinheiro AC, Teixeira JA, Vicente AA, Ramos OL (2017) Micro- and nano bio-based delivery systems for food applications: in vitro behavior. Adv Coll Interface Sci 243:23–45. https://doi.org/10.1016/j.cis.2017.02.010
54. Tian H, Lu Z, Li D, Hu J (2018) Preparation and characterization of citral-loaded solid lipid nanoparticles. Food Chem 248:78–85. https://doi.org/10.1016/j.foodchem.2017.11.091
55. Cui H, Li W, Li C, Vittayapadung S, Lin L (2016) Liposome containing cinnamon oil with antibacterial activity against methicillin-resistant *Staphylococcus* aureus biofilm. Biofouling 32:215–225. https://doi.org/10.1080/08927014.2015.1134516
56. Campana R, Casettari L, Fagioli L, Cespi M, Bonacucina G, Baffone W (2017) Activity of essential oil-based microemulsions against *Staphylococcus aureus* biofilms developed on stainless steel

surface in different culture media and growth conditions. Int J Food Microbiol 241:132–140. https://doi.org/10.1016/j.ijfoodmicro.2016.10.021
57. Niknejad H, Mahmoudzadeh R (2015) Comparison of different crosslinking methods for preparation of docetaxel-loaded albumin nanoparticles. Iran J Pharm Res 14:385–394
58. Shishir MRI, Xie L, Sun C, Zheng X, Chen W (2018) Advances in micro and nano-encapsulation of bioactive compounds using biopolymer and lipid-based transporters. Trends Food Sci Technol 78:34–60. https://doi.org/10.1016/j.tifs.2018.05.018
59. Bhatia S (2016) Natural polymers vs synthetic polymer. In: Natural polymer drug delivery systems. Springer, Cham, pp 95–118
60. Kwan A, Davidov-Pardo G (2017) Controlled release of flavor oil nanoemulsions encapsulated in filled soluble hydrogels. Food Chem 250:46–53. https://doi.org/10.1016/j.foodchem.2017.12.089
61. da Silva GS, De Souza ME, Quatrin PM et al (2018) Nanoemulsions containing Cymbopogon flexuosus essential oil: development, characterization, stability study and evaluation of antimicrobial and antibiofilm activities. Microb Pathog 118:268–276. https://doi.org/10.1016/j.micpath.2018.03.043
62. Ghosh V, Mukherjee A, Chandrasekaran N (2013) Ultrasonic emulsification of food-grade nanoemulsion formulation and evaluation of its bactericidal activity. Ultrason Sonochem 20:338–344. https://doi.org/10.1016/j.ultsonch.2012.08.010
63. Giongo JL, De Almeida VR, Fausto VP et al (2016) Anti- Candida activity assessment of Pelargonium graveolens oil free and nanoemulsion in biofilm formation in hospital medical supplies. Microb Pathog 100:170–178. https://doi.org/10.1016/j.micpath.2016.08.013
64. Moghimi R, Ghaderi L, Rafati H, Aliahmadi A, McClements DJ (2016) Superior antibacterial activity of nanoemulsion of Thymus daenensis essential oil against *E. coli*. Food Chem 194:410–415. https://doi.org/10.1016/j.foodchem.2015.07.139
65. Pérez-Conesa D, Cao J, Chen L, McLandsborough L, Weiss J (2011) Inactivation of *Listeria monocytogenes* and *Escherichia coli* O157:H7 biofilms by micelle-encapsulated eugenol and carvacrol. J Food Prot 74:55–62. https://doi.org/10.4315/0362-028X.JFP-08-403
66. Sugumar S, Nirmala J, Ghosh V, Anjali H, Mukherjee A, Chandrasekaran N (2013) Bio-based nanoemulsion formulation, characterization and antibacterial activity against food-borne pathogens: bio-based nanoemulsion against food-borne pathogens. J Basic Microbiol 53:677–685. https://doi.org/10.1002/jobm.201200060
67. Gharsallaoui A, Roudaut G, Chambin O, Voilley A, Saurel R (2007) Applications of spray-drying in microencapsulation of food ingredients: an overview. Food Res Int 40:1107–1121. https://doi.org/10.1016/j.foodres.2007.07.004
68. Xiao Z, Liu W, Zhu G, Zhou R, Niu Y (2014) Production and characterization of multinuclear microcapsules encapsulating lavender oil by complex coacervation: microcapsules encapsulating lavender oil. Flavour Fragr 29:166–172. https://doi.org/10.1002/ffj.3192
69. Fathi M, Vinceković M, Jurić S, Viskić M, Režek Jambrak A, Donsì F (2019) Food-grade colloidal systems for the delivery of essential oils. Food Rev Intl 37:1–45. https://doi.org/10.1080/87559129.2019.1687514
70. Granata G, Stracquadanio S, Leonardi M et al (2018) Essential oils encapsulated in polymer-based nanocapsules as potential candidates for application in food preservation. Food Chem 269:286–292. https://doi.org/10.1016/j.foodchem.2018.06.140
71. Moreira ACG, Martins IM, Fernandes I, Barreiro MF, Rodrigues AE (2016) Microencapsulation of red and white thyme oil in poly(lactic-co-glycolic) acid: assessment of encapsulation efficiency and antimicrobial capacity of the produced microcapsules. Can J Chem Eng 94:469–475. https://doi.org/10.1002/cjce.22411
72. Popiolski TM, Otsuka I, Halila S, Muniz EC, Soldi V, Borsali R (2016) Preparation of polymeric micelles of poly (Ethylene Oxide-b-Lactic Acid) and their encapsulation with lavender oil. Mat Res 19:1356–1365. https://doi.org/10.1590/1980-5373-mr-2016-0430
73. George A, Shah PA, Shrivastav PS (2019) Natural biodegradable polymers based nano-formulations for drug delivery: a review. Int J Pharm 561:244–264. https://doi.org/10.1016/j.ijpharm.2019.03.011
74. Mora-Huertas CE, Fessi H, Elaissari A (2010) Polymer-based nanocapsules for drug delivery. Int J Pharm 385:113–142. https://doi.org/10.1016/j.ijpharm.2009.10.018
75. Rehman A, Ahmad T, Aadil RM et al (2019) Pectin polymers as wall materials for the nano-encapsulation of bioactive compounds. Trends Food Sci Technol 90:35–46. https://doi.org/10.1016/j.tifs.2019.05.015

76. Shah B, Davidson PM, Zhong Q (2012) Encapsulation of eugenol using Maillard-type conjugates to form transparent and heat stable nanoscale dispersions. LWT 49:139–148. https://doi.org/10.1016/j.lwt.2012.04.029
77. Wang J, Khelissa OS, Chihib NE, Dumas E, Gharsallaoui A (2019) Effect of drying and interfacial membrane composition on the antimicrobial activity of emulsified citral Food Chem 298:125079. https://doi.org/10.1016/j.foodchem.2019.125079
78. Chen H, Zhang Y, Zhong Q (2015) Physical and antimicrobial properties of spray-dried zein–casein nanocapsules with co-encapsulated eugenol and thymol. J Food Eng 144:93–102. https://doi.org/10.1016/j.jfoodeng.2014.07.021
79. Saifullah MD, Shishir MRI, Ferdowsi R, Tanver Rahman MR, Van Vuong Q (2019) Micro and nano encapsulation, retention and controlled release of flavor and aroma compounds: a critical review. Trends Food Sci Technol 86:230–251. https://doi.org/10.1016/j.tifs.2019.02.030
80. Sun X, Cameron RG, Bai J (2019) Microencapsulation and antimicrobial activity of carvacrol in a pectin-alginate matrix. Food Hydrocolloids 92:69–73. https://doi.org/10.1016/j.foodhyd.2019.01.006
81. de Oliveira EF, Paula HCB, de Paula RCM (2014) Alginate/cashew gum nanoparticles for essential oil encapsulation. Colloids Surf B 113:146–151. https://doi.org/10.1016/j.colsurfb.2013.08.038
82. Hsieh WC, Chang CP, Gao YL (2006) Controlled release properties of Chitosan encapsulated volatile Citronella Oil microcapsules by thermal treatments. Colloids Surf, B 53:209–214. https://doi.org/10.1016/j.colsurfb.2006.09.008
83. Shetta A, Kegere J, Mamdouh W (2019) Comparative study of encapsulated peppermint and green tea essential oils in chitosan nanoparticles: encapsulation, thermal stability, in-vitro release, antioxidant and antibacterial activities. Int J Biol Macromol 126:731–742. https://doi.org/10.1016/j.ijbiomac.2018.12.161
84. Hajebi S, Rabiee N, Bagherzadeh M et al (2019) Stimulus-responsive polymeric nanogels as smart drug delivery systems. Acta Biomater 92:1–18. https://doi.org/10.1016/j.actbio.2019.05.018
85. de Matos SP, Lucca LG, Koester LS (2019) Essential oils in nanostructured systems: challenges in preparation and analytical methods. Talanta 195:204–214. https://doi.org/10.1016/j.talanta.2018.11.029
86. Ngampunwetchakul L, Toonkaew S, Supaphol P, Suwantong O (2019) Semi-solid poly(vinyl alcohol) hydrogels containing ginger essential oil encapsulated in chitosan nanoparticles for use in wound management. J Polym Res 26:224. https://doi.org/10.1007/s10965-019-1880-8
87. Volić M, Pajic-Lijakovic I, Djordjevic V et al (2018) Alginate/soy protein system for essential oil encapsulation with intestinal delivery. Carbohydr Polym 200:15–24. https://doi.org/10.1016/j.carbpol.2018.07.033
88. Lu WC, Huang DW, Wang CC et al (2018) Preparation, characterization, and antimicrobial activity of nanoemulsions incorporating citral essential oil. J Food Drug Anal 26:82–89. https://doi.org/10.1016/j.jfda.2016.12.018
89. Ma Q, Davidson PM, Zhong Q (2016) Antimicrobial properties of microemulsions formulated with essential oils, soybean oil, and Tween 80. Int J Food Microbiol 226:20–25. https://doi.org/10.1016/j.ijfoodmicro.2016.03.011
90. Ribeiro FWM, Laurentino LDS, Alves CR et al (2015) Chemical modification of gum arabic and its application in the encapsulation of Cymbopogon citratus essential oil. J Appl Polym Sci 132:8. https://doi.org/10.1002/app.41519
91. Rossi GG, Guerres KB, Bonez PC et al (2017) Antibiofilm activity of nanoemulsions of Cymbopogon flexuosus against rapidly growing mycobacteria. Microb Pathog 113:335–341. https://doi.org/10.1016/j.micpath.2017.11.002
92. Sahli C, Moya SE, Lomas JS, Gravier-Pelletier C, Briandet R, Hémadi M (2022) Recent advances in nanotechnology for eradicating bacterial biofilm. Theranostics 12:2383–2405. https://doi.org/10.7150/thno.67296
93. Laouini A, Jaafar-Maalej C, Limayem-Blouza I, Sfar S, Charcosset C, Fessi H (2012) Preparation, characterization and applications of liposomes: state of the art. J Coll Sci Biotechnol 1:147–168. https://doi.org/10.1166/jcsb.2012.1020
94. Prakash B, Kujur A, Yadav A, Kumar A, Singh PP, Dubey NK (2018) Nanoencapsulation: an efficient technology to boost the antimicrobial potential of plant essential oils in food system. Food Control 89:1–11. https://doi.org/10.1016/j.foodcont.2018.01.018
95. Cui H, Zhao C, Lin L (2015) The specific antibacterial activity of liposome-encapsulated Clove oil and its application in tofu. Food Control 56:128–134. https://doi.org/10.1016/j.foodcont.2015.03.026

96. Engel JB, Heckler C, Tondo EC, Daroit DJ, da Silva MP (2017) Antimicrobial activity of free and liposome-encapsulated thymol and carvacrol against *Salmonella* and *Staphylococcus aureus* adhered to stainless steel. Int J Food Microbiol 252:18–23. https://doi.org/10.1016/j.ijfoodmicro.2017.04.003
97. Liolios CC, Gortzi O, Lalas S, Tsaknis J, Chinou I (2009) Liposomal incorporation of carvacrol and thymol isolated from the essential oil of *Origanum dictamnus* L. and in vitro antimicrobial activity. Food Chem 112:77–83. https://doi.org/10.1016/j.foodchem.2008.05.060
98. Liao W, Badri W, Dumas E, Ghnimi S, Elaissari A, Saurel E, Gharsallaoui A (2021) Nanoencapsulation of essential oils as naturalfood antimicrobial agents: an overview. Appl Sci 11:5778. https://doi.org/10.3390/app11135778
99. McClements DJ (2018) Encapsulation, protection, and delivery of bioactive proteins and peptides using nanoparticle and microparticle systems: a review. Adv Coll Interface Sci 253:1–22. https://doi.org/10.1016/j.cis.2018.02.002
100. Salvi VR, Pawar P (2019) Nanostructured lipid carriers (NLC) system: a novel drug targeting carrier. J Drug Deliv Sci Technol 51:255–267. https://doi.org/10.1016/j.jddst.2019.02.017
101. Nasseri M, Golmohammadzadeh S, Arouiee H, Jaafari MR, Nemati H (2020) Preparation and comparison of various formulations of solid lipid nanoparticles (SLNs) containing the essential oil of Zataria multiflora. J Horticulture Postharvest Res 3:73–84. https://doi.org/10.22077/JHPR.2019.2570.1068
102. Nasseri M, Golmohammadzadeh S, Arouiee H, Reza M, Neamati H (2016) Antifungal activity of Zataria multiflora essential oil-loaded solid lipid nanoparticles in-vitro condition. Iran J Basic Med Sci 19:1231–1237
103. Katouzian I, Faridi Esfanjani A, Jafari SM, Akhavan S (2017) Formulation and application of a new generation of lipid nano-carriers for the food bioactive ingredients. Trends Food Sci Technol 68:14–25. https://doi.org/10.1016/j.tifs.2017.07.017
104. Bashiri S, Ghanbarzadeh B, Ayaseh A, Dehghannya J, Ehsani A (2019) Preparation and characterization of chitosan-coated nanostructured lipid carriers (CH-NLC) containing cinnamon essential oil for enriching milk and anti-oxidant activity. LWT 119:108836. https://doi.org/10.1016/j.lwt.2019.108836
105. Ghodrati M, Farahpour MR, Hamishehkar H (2019) Encapsulation of Peppermint essential oil in nanostructured lipid carriers: In-vitro antibacterial activity and accelerative effect on infected wound healing. Colloids Surf A 564:161–169. https://doi.org/10.1016/j.colsurfa.2018.12.043
106. Keivani Nahr F, Ghanbarzadeh B, Hamishehkar H, Samadi Kafil H (2018) Food grade nanostructured lipid carrier for cardamom essential oil: preparation, characterization and antimicrobial activity. J Funct Foods 40:1–8. https://doi.org/10.1016/j.jff.2017.09.028
107. Mokarizadeh M, Kafil H, Ghanbarzadeh S, Alizadeh A, Hamishehkar H (2017) Improvement of citral antimicrobial activity by incorporation into nanostructured lipid carriers: a potential application in food stuffs as a natural preservative. Res Pharm Sci 12:409. https://doi.org/10.4103/1735-5362.213986
108. Fathi M, Mozafari MR, Mohebbi M (2012) Nanoencapsulation of food ingredients using lipid based delivery systems. Trends Food Sci Technol 23:13–27. https://doi.org/10.1016/j.tifs.2011.08.003
109. Wang T, Xue J, Hu Q, Zhou M, Luo Y (2017) Preparation of lipid nanoparticles with high loading capacity and exceptional gastrointestinal stability for potential oral delivery applications. J Colloid Interface Sci 507:119–130. https://doi.org/10.1016/j.jcis.2017.07.090
110. Dua JS, Rana AC, Bhandari DAK (2012) Liposome: methods of preparation and applications. Int J Pharm Stud Res 3:14–20
111. Sharma N, Roy PSD, Majeed A et al (2022) Nanoencapsulation- A novel strategy for enhancing the bioactivity of essential oils: a Review. Indian J Agric Res 56:241–248. https://doi.org/10.18805/IJARe.A-5806
112. Zubair M, Shahzad S, Hussain A, Pradhan RA, Arshad M, Ullah A (2022) Current trends in the utilization of essential oils for polysaccharide- and protein-derived food packaging materials. Polymers 14:1146. https://doi.org/10.3390/polym14061146
113. Al-Maqtari QA, Rehman A, Mahdi AA et al (2021) Application of essential oils as preservatives in food systems: challenges and future prospectives: a review. Phytochem Rev 21:1209–1246. https://doi.org/10.1007/s11101-021-09776-y

114. Tewa-Tagne P, Briançon S, Fessi H (2007) Preparation of redispersible dry nanocapsules by means of spray-drying: development and characterisation. Eur J Pharm Sci 30:124–135. https://doi.org/10.1016/j.ejps.2006.10.006
115. Mechmechani S, Khelissa S, Gharsallaoui A, Omari KE, Hamze M, Chihib NE (2022) Hurdle technology using encapsulated enzymes and essential oils to fight bacterial biofilms. Appl Microbiol Biotechnol 106:2311–2335. https://doi.org/10.1007/s00253-022-11875-5
116. Pisoschi AM, Pop A, Cimpeanu C, Turcuş V, Predoi G, Iordache F (2018) Nanoencapsulation techniques for compounds and products with antioxidant and antimicrobial activity: a critical view. Eur J Med Chem 157:1326–1345. https://doi.org/10.1016/j.ejmech.2018.08.076
117. Zhang W, Li X, Yu T et al (2015) Preparation, physicochemical characterization and release behavior of the inclusion complex of trans -anethole and β-cyclodextrin. Food Res Int 74:55–62. https://doi.org/10.1016/j.foodres.2015.04.029
118. Kurozawa LE, Hubinger MD (2017) Hydrophilic food compounds encapsulation by ionic gelation. Curr Opin Food Sci 15:50–55. https://doi.org/10.1016/j.cofs.2017.06.004
119. Esmaeili A, Asgari A (2015) In vitro release and biological activities of Carum copticum essential oil (CEO) loaded chitosan nanoparticles. Int J Biol Macromol 81:283–290. https://doi.org/10.1016/j.ijbiomac.2015.08.010
120. Keawchaoon L, Yoksan R (2011) Preparation, characterization and in vitro release study of carvacrol-loaded chitosan nanoparticles. Colloids Surf, B 84:163–171. https://doi.org/10.1016/j.colsurfb.2010.12.031
121. Asbahani AE, Miladi K, Badri W et al (2015) Essential oils: from extraction to encapsulation. Int J Pharm 483:220–243. https://doi.org/10.1016/j.ijpharm.2014.12.069
122. Bhargava K, Conti DS, da Rocha SRP, Zhang Y (2015) Application of an oregano oil nanoemulsion to the control of foodborne bacteria on fresh lettuce. Food Microbiol 47:69–73. https://doi.org/10.1016/j.fm.2014.11.007
123. Moghimi R, Aliahmadi A, Rafati H, Abtahi HR, Amini S, Feizabadi MM (2018) Antibacterial and anti-biofilm activity of nanoemulsion of Thymus daenensis oil against multi-drug resistant *Acinetobacter baumannii*. J Mol Liq 265:765–770. https://doi.org/10.1016/j.molliq.2018.07.023
124. Salvia-Trujillo L, Rojas-Graü MA, Soliva-Fortuny R, Martín-Belloso O (2014) Impact of microfluidization or ultrasound processing on the antimicrobial activity against *Escherichia coli* of lemongrass oil-loaded nanoemulsions. Food Control 37:292–297. https://doi.org/10.1016/j.foodcont.2013.09.015
125. Acevedo-Fani A, Salvia-Trujillo L, Rojas-Graü MA, Martín-Belloso O (2015) Edible films from essential-oil-loaded nanoemulsions: physicochemical characterization and antimicrobial properties. Food Hydrocolloids 47:168–177. https://doi.org/10.1016/j.foodhyd.2015.01.032
126. Guarda A, Rubilar JF, Miltz J, Galotto MJ (2011) The antimicrobial activity of microencapsulated thymol and carvacrol. Int J Food Microbiol 146:144–150. https://doi.org/10.1016/j.ijfoodmicro.2011.02.011
127. Quatrin PM, Verdi CM, de Souza ME et al (2017) Antimicrobial and antibiofilm activities of nanoemulsions containing Eucalyptus globulus oil against *Pseudomonas aeruginosa* and Candida spp. Microb Pathog 112:230–242. https://doi.org/10.1016/j.micpath.2017.09.062
128. Zhu Z, Min T, Zhang X, Wen Y (2019) Microencapsulation of thymol in poly(lactide-co-glycolide) (PLGA): physical and antibacterial properties. Materials 12:1133. https://doi.org/10.3390/ma12071133
129. Eghbal N, Choudhary R (2018) Complex coacervation: encapsulation and controlled release of active agents in food systems. LWT 90:254–264. https://doi.org/10.1016/j.lwt.2017.12.036
130. Dubey R, Shami TC, Rao KUB (2009) Microencapsulation technology and applications. Defense Sci J 59:82–95
131. Gu XL, Zhu X, Kong XZ, Tan Y (2010) Comparisons of simple and complex coacervations for preparation of sprayable insect sex pheromone microcapsules and release control of the encapsulated pheromone molecule. J Microencapsul 27:355–364. https://doi.org/10.3109/02652040903221532
132. Dias DR, Botrel DA, Fernandes RVDB, Borges SV (2017) Encapsulation as a tool for bioprocessing of functional foods. Curr Opin Food Sci 13:31–37. https://doi.org/10.1016/j.cofs.2017.02.001
133. Bakry AM, Abbas S, Ali B, Majeed H, Abouelwafa MY, Mousa A, Liang L (2016) Microencapsulation of oils: a comprehensive review of benefits, techniques, and applications. Comp Rev Food Sci Foody Saf 15:143–182. https://doi.org/10.1111/1541-4337.12179

134. Peng C, Zhao SQ, Zhang J, Huang GY, Chen LY, Zhao FY (2014) Chemical composition, antimicrobial property and microencapsulation of Mustard (Sinapis alba) seed essential oil by complex coacervation. Food Chem 165:560–568. https://doi.org/10.1016/j.foodchem.2014.05.126
135. Suganya V, Anuradha V (2017) Microencapsulation and nanoencapsulation: a review. Int J Pharm Clin Res 9:233–239. https://doi.org/10.25258/ijpcr.v9i3.8324
136. Nedovic V, Kalusevic A, Manojlovic V, Levic S, Bugarski B (2011) An overview of encapsulation technologies for food applications. Procedia Food Sci 1:1806–1815. https://doi.org/10.1016/j.profoo.2011.09.265
137. Lucía C, Marcela F, Ainhoa L (2017) Encapsulation of almond essential oil by co-extrusion/gelling using chitosan as wall material. J Encapsulation Adsorpt Sci 7:67–74. https://doi.org/10.4236/jeas.2017.71004
138. Jia Z, Dumont MJ, Orsat V (2016) Encapsulation of phenolic compounds present in plants using protein matrices. Food Biosci 15:87–104. https://doi.org/10.1016/j.fbio.2016.05.007
139. Pal SL, Jana U, Manna PK, Mohanta GP, Manavalan R (2011) Nanoparticle: an overview of preparation and characterization. J Appl Pharm Sci 7:228–234
140. Donsì F, Annunziata M, Sessa M, Ferrari G (2011) Nanoencapsulation of essential oils to enhance their antimicrobial activity in foods. LWT Food Sci Technol 44:1908–1914. https://doi.org/10.1016/j.lwt.2011.03.003
141. Clogston JD, Patri AK (2011) Zeta Potential Measurement. Characterization of nanoparticles intended for drug delivery. Humana Press, Totowa, pp 63–70
142. Jamil B, Abbasi E, Abbasi S et al (2016) Encapsulation of cardamom essential oil in chitosan nano-composites: in-vitro efficacy on antibiotic-resistant bacterial pathogens and cytotoxicity studies. Front Microbiol 7. https://doi.org/10.3389/fmicb.2016.01580
143. Alanchari M, Mohammadi M, Yazdian F et al (2021) Optimization and antimicrobial efficacy of curcumin loaded solid lipid nanoparticles against foodborne bacteria in hamburger patty. J Food Sci 86:2242–2254. https://doi.org/10.1111/1750-3841.15732
144. Das S, Singh VK, Chaudhari AK, Dwivedy AK, Dubey NK (2022) Co-encapsulation of Pimpinella anisum and Coriandrum sativum essential oils based synergistic formulation through binary mixture: Physico-chemical characterization, appraisal of antifungal mechanism of action, and application as natural food preservative. Pesticide Biochem Physiol 184:105066. https://doi.org/10.1016/j.pestbp.2022.105066
145. Khelissa S, El-Fannassi Y, El Amrani MA, Gharsallaoui A, Barras A, Chihib NE (2021) Water-soluble ruthenium (II) complex derived from optically pure limonene and its microencapsulation are efficient tools against bacterial food pathogen biofilms: *Escherichia coli*, *Staphylococcus aureus*, *Enteroccocus faecalis*, and *Listeria monocytogenes*. Front Microbiol 12:711326. https://doi.org/10.3389/fmicb.2021.711326
146. Mozafari MR (2010) Nanoliposomes: preparation and Analysis. In: Liposomes. NJ: Humana Press, Totowa, pp 29–50
147. Rios-Mera JD (2019) Encapsulation optimization and pH- and temperature-stability of the complex coacervation between soy protein isolate and inulin entrapping fish oil. LWT Food Sci Technol 116:108555. https://doi.org/10.1016/j.lwt.2019.108555
148. Choudhury N, Meghwal M, Das K (2021) Microencapsulation: an overview on concepts, methods, properties and applications in foods. Food Front 2:426–442. https://doi.org/10.1002/fft2.94
149. Nair A, Mallya R, Suvarna V, Khan TA, Momin M, Omri A (2022) Nanoparticles: attractive carriers of antimicrobial essential oils. Antibiotics 11:108. https://doi.org/10.3390/antibiotics11010108
150. Mehta N, Kumar P, Verma AK et al (2022) Microencapsulation as a noble technique for the application of bioactive compounds in the food industry: a comprehensive review. Appl Sci 12:1424. https://doi.org/10.3390/app12031424
151. Reis DR, Ambrosi A, Luccio MD (2022) Encapsulated essential oils: a perspective in food preservation. Future Foods 5:100126. https://doi.org/10.1016/j.fufo.2022.100126
152. Mukurumbira AR, Shellie RA, Keast R, Palombo EA, Jadhav SR (2022) Encapsulation of essential oils and their application in antimicrobial active packaging. Food Control 136:108883. https://doi.org/10.1016/j.foodcont.2022.108883.