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Encapsulation of mint essential oil: Techniques and applications

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Abstract

Mint essential oil (MEO) is an outstanding antibacterial and antioxidant agent, that can be considered as a promising natural preservative, flavor, insecticide, coolant, and herbal medicine. However, the low solubility and volatility of MEO limits its extensive applications. In order to utilize MEO in different products, it is essential to develop treatments that can overcome these limitations. More recently, encapsulation technology has been developed as a promising method to overcome the shortcomings of MEO. In which, sensitive compounds such as essential oils (EOs) are entrapped in a carrier to produce micro or nanoparticles with increased stability against environmental conditions. Additionally, encapsulation of EOs makes transportation and handling easier, reduces their volatility, controls their release and consequently improves the efficiency of these bioactive compounds and extends their industrial applications. Several encapsulation techniques, such as emulsification, coacervation, ionic gelation, inclusion complexation, spray drying, electrospinning, melt dispersion, melt homogenization, and so on, have been emerged to improve the stability of MEO. These encapsulated MEOs can be also used in a variety of food, bioagricultural, pharmaceutical, and health care products with excellent performance. Therefore, this review aims to summarize the physicochemical and functional properties of MEO, recent advances in encapsulation techniques for MEO, and the application of micro/nanocapsulated MEO in different products.

Keywords: Mint essential oil, Menthol, Encapsulation, Controlled release

1. Introduction

Mints (*Mentha* spp.) are aromatic, medicinal, and economically important perennial plants that taxonomically belong to the Lamiaceae family. Various species of mints are cultivated worldwide, with *Mentha piperita*, *Mentha spicata*, Mentha haplocalyx, *Mentha longifolia*, and *Mentha pulegium* being among the most commonly used. Mints have broad spectrum applications in a range of industries mainly including perfumery, pharmaceutical, and food industries [1]. These plants are known to contain diverse phytochemical compounds with a wide range of biological and pharmacological activities. Among these bioactive phytochemicals, essential oils (EOs) have recently received particular attention mainly due to their antioxidant, antifungal, antibacterial, antiviral, and other biological properties [2]. As described by Başer and Demirci [3], "EOs, also known as essence, etheric oil, volatile oil, or aetheroleum, are complex mixtures of volatile compounds biosynthesized by the living organisms." EOs as secondary metabolites of botanical origin are commonly a blend of instable (easily decomposable) and highly volatile compounds [4].

Mint essential oil (MEO) is accumulated in the glandular trichomes, that are predominantly situated on the adaxial surface of leaves. Although, other plant organs such as stems and flowers are rich sources of EOs [5]. MEO has several health promoting effects and is used as an active ingredient in many medications for treating oral mucosa inflammation, respiratory tract disorders, enteritis, and gastritis [6]. It is also valued for its cooling and antimicrobial properties, making it a popular ingredient in toothpastes, mouthwashes, and chewing gums. The main constituents of MEO are menthol, menthone, neomenthol, and iso-menthone [7]. The extent of these individual compounds (ICs) is influenced by the species and cultivar of mint, its growing region, and fertilization.

MEOs are isolated by a range of extraction methods from different parts of the mint plant including leaves, flowers, and stems. Some of them are traditional and commonly-used/old-fashion techniques, such as hydro-distillation and steam distillation. While some of the extraction methods are based on more advanced technologies, e.g., ultrasound-assisted extraction, microwave-assisted extraction, and supercritical fluid extraction [8]. The presence of acceptable contents of EOs in mints has been demonstrated by extensive documentation [7]. MEOs represent a growing, special, and demanding market niche on a global scale with a substantial upsurge in product sales over the past years [9]. However, like other EOs, the highly volatile bioactive components of MEOs are known to have poor chemical stability and are susceptible to degradation [10]. These are considered among the potential bottlenecks/drawbacks/limitations in achieving the acceptable quality in products containing MEOs.

Potentially, encapsulation strategies represent a promising approach that could widely provide protection of MEOs from rapid degradation and improve/assure (at least partially) their stability and therefore their functionality and bioactivity. Recently, Weisany et al. [11], highlighted several potential advantages for encapsulation of EOs including prevention of their degradation, their higher stability in food formulation, increase in their bio-efficacy, ease of processing, enhancement of their functional activity, their site specific delivery, their higher absorption in body, and the conversion of EOs from liquid state to powder. In general, encapsulation is defined as a process of integration/entrapment of a sensitive substance (core material) in a particular coating medium (wall material) to preserve them against chemical and thermal degradations [12].

Encapsulation involves a wide range of available and innovative techniques that can be applied under different conditions to produce encapsulates, which are final products with varying sizes and structures. Micrometric (1.0–1000 μm) and nanometric scale (< 1.0 μm) encapsulations

are two principal procedures widely used in encapsulation practices for application in agri-food products [13]. The surface to volume ratio of nanocapsules is very high; consequently, the solubility, bioavailability, encapsulation efficiency, and controlled release of the core materials are higher than larger encapsulating structures. Both procedures are well-reviewed and their exclusive efficacy in improving functionality of bioactive compounds are supported by ample and growing body of evidence [14].

This review aims to provide a comprehensive and concise overview of various aspects of micro-/nano-encapsulation of MEOs, including the utilized encapsulation techniques (coacervation, emulsification, spray-drying, electrospinning and electrospraying, inclusion complexation and melt dispersion) and the techno-functional considerations, recent applications of encapsulated MEOs and finally the challenges and safety issues related to the encapsulation of MEOs.

2. Encapsulation of MEO

Encapsulation technology can increase the stability and control the release of bioactive compounds by physically isolating them from the surrounding environment [15]. In these systems, the coated or entrapped materials are also called core material, internal phase, or payload, and the coating material is also known as wall material, coating, membrane, carrier, capsule, or shell [16]. In this section, various wall materials and techniques for encapsulating MEOs and its ICs are discussed.

2.1. Selection of appropriate wall materials

The success of the micro/nano-encapsulation process is highly dependent on the selection of appropriate wall materials with ideal stability, solubility, surface activity, viscosity, and release behavior to protect the core material against harsh external stressors (e.g., light, moisture, oxidation, etc.) and completely release the active substance under different conditions [17]. Wall materials are usually composed of biodegradable polymers that are chosen from synthetic and/or natural materials [15].

The selection of appropriate wall materials for encapsulating food ingredients and nutraceuticals poses a significant challenge, as it necessitates strict adherence to regulations set by organizations such as the Food and Drug Administration (FDA) and the European Food Safety Authority (EFSA).[18]. In this respect, several points should be considered in selecting such materials to serve as an efficient delivery vehicle, including (1) biocompatibility of the wall material, (2) capability to disperse the core material and stabilize the resulting emulsion-based product, (3) high-loading of desired bioactive compounds, (4) lack of chemical reactivity with the core material during the encapsulation process, (5) capability to withstand environmental stresses during processing and storage, and (6) providing a controlled release mechanism [19, 20].

There are numerous studies reported the possible application of non-toxic and safe wall materials for the encapsulation of MEOs in food and pharmaceutical industries, which can be considered as Generally Recognized as Safe (GRAS) materials [21-23]. These materials include synthetic polymers, such as poly (lactide-co-glycolic acid) (PLGA), poly (vinyl alcohol) (PVA), and poly (ε-caprolactone) (PCL), or natural polymers, such as carbohydrates (e.g., starch, cellulose, cyclodextrin, maltodextrin, sodium or calcium alginate, gum Arabic (GA), and chitosan), proteins (e.g., whey protein, casein, zein, and gelatin), and lipids (e.g., beeswax and candelilla wax) in their native or modified forms, as well as emulsifiers, such as lecithin, Tweens,

Spans [24-32]. Furthermore, it is important to note that a single membrane may not possess all the desired features of an ideal wall material. As a result, it is often recommended to utilize a combination of polymers, such as carbohydrate-carbohydrate (e.g., GA-starch, chitosan-alginate, maltodextrin-alginate, etc.) and carbohydrate-protein (e.g., chitosan-gelatin, GA-whey protein, GA-gelatin, etc.) complexes [33-35].

2.2. Encapsulation methods

Various methods exist for encapsulating bioactive compounds, each with its own unique encapsulation system. These systems can range from a single coating membrane to tubular, spherical, or irregular walls/membranes, multi walled structures with similar or different compositions, or even multiple cores within the same walled structure. However, regardless of the specific technique employed, there are three common steps involved in all encapsulation techniques. These steps include (1) the formation of a wall material around the core material, (2) ensuring that no leakage occurs, and (3) removing any undesired substances. [16]. These technologies are generally classified by drying methods, such as spray-drying, spray-chilling, fluid-bed coating, freeze drying and so on, due to the liquid nature of the core materials (or active agents) [36]. However, the encapsulation techniques can also be categorized into coacervation, inclusion complexation, and emulsification techniques based on the presence of core materials in a complex matrix or emulsion systems, or can be divided into electrospinning and electrospraying techniques based on the use of professional instruments [20]. Most of these techniques are also dried by different drying methods in order to use in various sectors of food, agricultural, pharmaceutical, and cosmetic industries.

2.2.1. Emulsification

Emulsification is an effective approach to improve the solubility and consolidation of hydrophobic materials such as EOs in an aqueous phase. Oil-in-water (O/W) emulsions enhance the dispersion of EOs in food and non-food matrices, increase their stability and controlled release, antimicrobial activity, and antioxidant properties. Emulsions are divided into 3 groups based on their droplet size: coarse emulsions (0.5-100 μm), nanoemulsions (50-500 nm), and microemulsions (10-100 nm) [37]. Also, according to their emulsifier types, emulsions are categorized into molecular-emulsifier-based emulsions and particle-emulsifier-based emulsions, also known as Pickering emulsions. The molecular-based emulsifiers, are surface-active agents, that are adsorbed on the oil–water interface, and reduce the interfacial tension. While, the particle-based emulsifiers are usually amphiphilic solid particles that are adsorbed at the oil-water interface. Solid particles may not have a significant impact on reducing interfacial tension; they act as mechanical barriers that effectively prevents the coalescence of droplets [38, 39].

2.2.1.1. Molecular emulsifier-based emulsions

Emulsifiers play an important role in the formation and stabilization of emulsions by adsorption onto the oil–water interface. Emulsifiers can be classified considering their molecular weight into low molecular weight emulsifiers (LMWE) and high molecular weight emulsifiers (HMWE). The former has a hydrophilic head and a lipophilic tail. It includes natural polar lipids such as lecithins and synthetic emulsifiers such as polysorbates, mono- and diglycerides, spans (sorbitan esters), Tweens (polyoxyethylene sorbitan esters), sucrose esters, and citric, lactic, and

acetic acid esters of mono- and diglycerides. The latter refers to amphiphilic biopolymers that contain both hydrophilic and lipophilic groups within their chain. Modified starches, modified celluloses, GA, galactomannans, and some types of pectin are examples of HMWE [40].

Several types of emulsifiers have been applied for the emulsification of MEO in delivery systems. For instance, Gorjian et al. [41] studied the effect of non-ionic emulsifiers, namely Tween 20, Tween 40, and Tween 80, on the formation and characteristics of spearmint EO nanoemulsions produced by ultrasonic emulsification. The nanoemulsions were stored for 35 days at 4 °C and their average particle size, zeta potential, polydispersity index, pH, viscosity, turbidity, and stability were monitored during storage. The results demonstrated that the type of emulsifiers had significant effects on the properties of nanoemulsions and the samples prepared with Tween 20 had the highest stability and the lowest particle size. Moreover, Zhong et al. [42] prepared MEO microemulsions and evaluated the effect of processing conditions (mixing order and temperature) and surfactant type on the release profile of MEO. The emulsifiers included Cremophor EL (CrEL), Brij-35, Span-20, Tween 20, lecithin, and sodium bis(2-ethylhexyl) sulfosuccinate (AOT). The results unveiled that the formation of a MEO microemulsions was dependent on the type and ratio of surfactants. A 1:1 mixture of AOT:CrEL was the most effective surfactant that reduced the amount of surfactant needed and produced stable microemulsions with encapsulation efficiency of 78.4% and average particle size of 20 nm. However, the processing parameters such as the order of mixing the emulsion components and temperature, did not show significant effects on the properties of microemulsions. Furthermore, Salvia-Trujillo et al. [43] produced nanoemulsions loaded with various EOs, including mint, thyme, sage, clove, lemongrass, tea tree, marjoram, geranium, rosewood, and palmarosa with tween 80 and sodium alginate by microfluidization method. The nanoemulsions presented higher negative z-potential values

compared to coarse emulsions indicating a strong repulsive force between MEO droplets in the aqueous media and their droplet size was below 20 nm. In addition, the whiteness index and transparency of emulsions improved after microfluidization due to the size reduction of droplets. However, their viscosity decreased as a result of the mechanical stress during microfluidization. Moreover, the nanoemulsions exhibited remarkable antimicrobial activity against *E. coli*, suggesting their potential use as biopreservatives in various products.

Oil-in-water *Mentha spicata* EO nanoemulsions were produced by mixing MEO (10% w/w), water (85% w/w), and Tween 80 (5% w/w) with an ultrasonic homogenizer and its antimicrobial effects against foodborne pathogens including *S. Typhimurium, E. coli, S. aureus, L. monocytogenes*, and *B. cereus* were compared with free MEO. The SEM images of bacterial cell membranes and growth behavior of bacteria revealed that nanoemulsion were more efficient than free MEO in destruction of pathogens. Both free MEO and its nanoemulsion showed better antimicrobial properties against Gram-positive bacteria than the Gram-negative bacteria and presented the highest antimicrobial effects against *L. monocytogenes.* Also, the antioxidant activity of nanoemulsions was higher and the IC50 value was lower than free MEO. Therefore, the nanoemulsions comprising MEO have the potential to be used as natural preservative [44].

2.2.1.2. Pickering emulsions

The application of molecular emulsifiers is limited under certain pH, temperature, ionic strength, and shearing conditions; because they cannot prevent the destabilization of emulsions. Furthermore, some synthetic emulsifiers may have toxic effects or cause environmental pollutions [45]. Pickering emulsions have a long-term stability, owing to the irreversible attachment of solid particles at the oil-water interface of emulsions droplets. Inorganic particles such as metal oxide,

metal sulfate, metal hydroxide, clay, silica, and carbon particles have traditionally been applied for the stabilization of Pickering emulsions. However, there has been an increasing interest in using organic particles in Pickering emulsions in recent years. Biopolymeric micro/nanoparticles from proteins and polysaccharides are widely used in Pickering emulsions. Polysaccharide particles include starch granules, chitosan, cellulose, and their nanomaterials, while proteins include zein, casein, whey, gelatin, etc. These natural particles are nontoxic and biodegradable, thus can be used in foods, drugs, and cosmetics. Pickering emulsions are promising vehicle for the delivery of EOs [46]. In a study by Cheng et al. [47], resveratrol-zein-pectin nanoparticles were used to prepare peppermint essential oil (PEO) emulsions. The physicochemical and antimicrobial properties of PEO-loaded Pickering emulsions were investigated and the results revealed high encapsulation efficiency of PEO. Additionally, it was demonstrated that increasing the concentration of pectin decreased the droplet size of emulsions, improved their chemical and physical stability, and antimicrobial properties. As shown in Fig. 1, Lai et al. [46] fabricated PEO-loaded Pickering emulsions with chitosan coated silica nanoparticles and studied its physicochemical properties. They used different concentrations (0.1, 0.25, 0.5, 1, 2.5, and 5%) of chitosan to improve the hydrophobicity of silica nanoparticles and observed that chitosan effectively modified the surface of silica nanoparticles and facilitated their adsorption at the oil-water interface. Also, the investigations disclosed that increasing the concentration of chitosan prolonged the stability, improved the sustained release, increased the antimicrobial activity, and decreased the particle size of emulsions. Therefore, chitosan-decorated silica nanoparticles are appropriate shells for PEO-loaded Pickering emulsions with long-term antimicrobial activity.

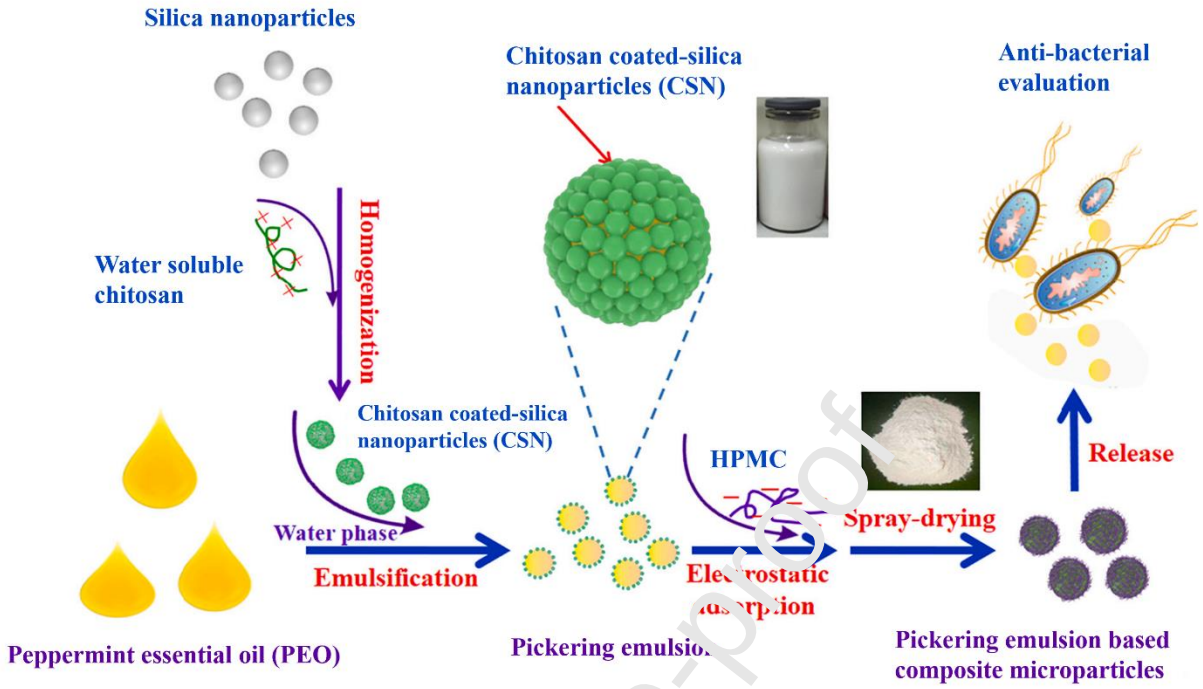

Fig. 1. The schematic image of PEO laded Pickering emulsions stabilized by chitosan-decorated silica nanoparticles; adapted from Lai et al. [46].

2.2.2. Ionic gelation

Ionic gelation is highly recommended for the encapsulation of EOs. This process is based on the ionic interactions between oppositely charged polymers or between a polymer and a polycation or polyanion. These interactions form a polyelectrolyte complex bond on the surface of particles and increase their mechanical strength. In this process, an emulsion of EOs in a polymeric solution is prepared and poured into a cross-linking solution where gelation is carried out on the droplet surface until the complete formation of the microspheres [48, 49]. The gels obtained from ionic gelation can improve the solubility of hydrophobic compounds; therefore, they are great carriers for EOs. In addition, they have high stability, loading capacity, and controlled release. These gels have a three-dimensional structure in which polymer chains form a cross-linked

network. Non-degradable, synthetic degradable, and biopolymers can be used for the fabrication of capsules in ionic gelation. However, biopolymers are more appreciated than other compounds due to their non-toxicity, biodegradability, biocompatibility, and solubility. As shown in Fig. 2, Shetta et al. [50] used ionic gelation for the encapsulation of PEO. MEO emulsions were prepared by mixing chitosan, Tween 80 and different concentrations of MEO (0.12, 0.24, 0.36, and 0.48 g) and then Sodium tripolyphosphate (TPP) was added to the emulsions for the cross-linking of chitosan by ionic gelation. The obtained pellets were washed several times and dispersed in distilled water and freeze-dried. The transmission electron microscopy (TEM) images illustrated spherical nanoparticles with an average size ranging from 20 to 60 nm. The loading capacity of MEO was 22.2% and the in-vitro release profile showed a Fickian diffusion behavior. The antioxidant activity and the stability of total phenolic content were improved by encapsulation. The thermogravimetric analysis (TGA) of encapsulated MEO revealed that the peak temperature for thermal degradation increased up to 350 °C. In a similar study, a core-shell system was devised by Bonda et al. [33], which involved the creation of inverse ionic gelation-based systems for PEO. The process entailed carefully dripping an emulsion (consisting of PEO and an aqueous solution of hydroxyethylcellulose and $CaCl_2$) into an alginate solution with the addition of a secondary excipient (either shellac gum or maltodextrin) at different concentrations. It was observed that modifications made to the composition of the capsule shell had minimal effect on the PEO content, which remained at levels above 50% w/w. However, these changes did influence various other characteristics of the dried capsules, including their size, shell thickness, hardness, and swelling behavior. The wet capsule particles exhibited an approximate size of 4 mm, unaffected by the type or quantity of the secondary excipient. Upon drying, the particle size was reduced to around 2 mm. While the presence of shellac gum did not affect the dimensions of the dried capsules, an increase

in maltodextrin content led to larger diameters. The hardness of the capsules decreased with higher amounts of the secondary excipient in the formulations. Additionally, the inclusion of maltodextrin in the formulations influenced the capsules' swelling behavior in water, suggesting its potential as an excipient for modulating swelling based on specific application requirements.

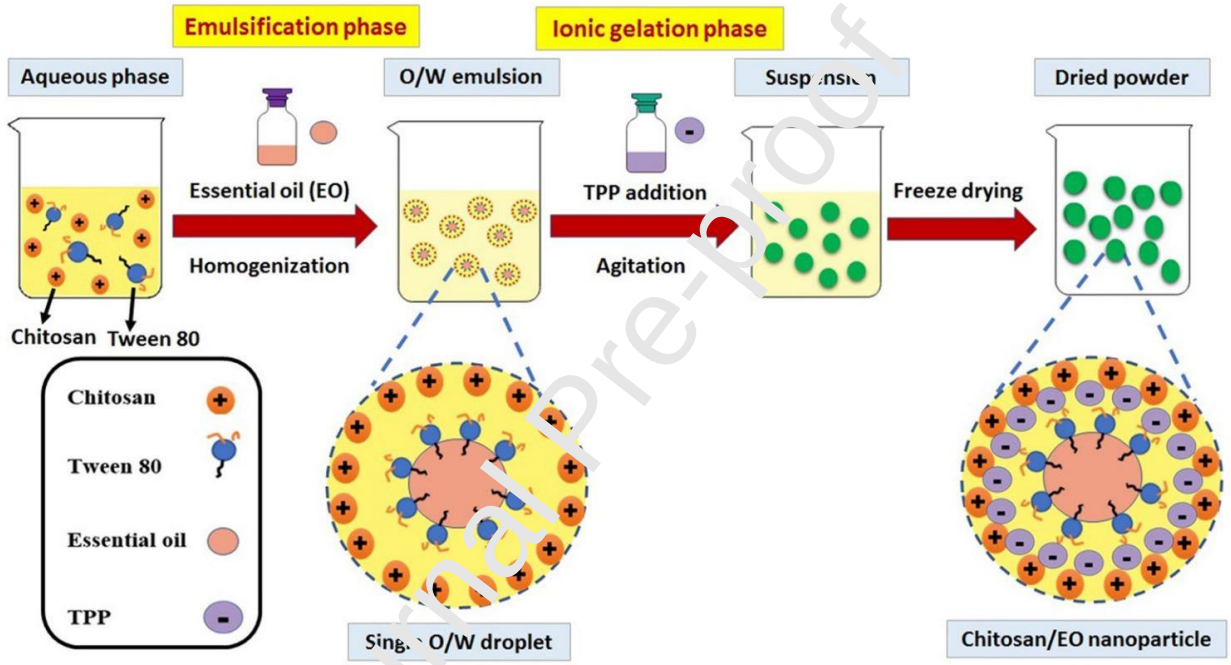

Fig. 2. Schematic image of nanocapsulation of EOs by ionic gelation method; adapted from Shetta et al. [50].

2.2.3. Coacervation

Coacervation is an alternative technique utilized for encapsulating bioactive and heat-sensitive compounds, available in two forms: simple coacervation and complex coacervation. Complex coacervation microcapsules, typically exhibiting low water solubility, are known for

their high heat resistance. Complex coacervation involves the mixing of oppositely charged polyelectrolytes in an aqueous environment, and in the formation of phase-separated entities known as polyelectrolyte complexes, coacervates, or complex coacervates. Fig. 3 illustrates the schematic diagram of the encapsulation of EOs by complex coacervation. Various factors including the concentration of the polyelectrolyte, temperature, and pH significantly affect the complex coacervation. Abundant and environmentally friendly natural polymers are often preferred when it comes to the preparation of polymeric encapsulating systems as they have high levels of biodegradability as well [34]. The complex coacervation method was utilized for the encapsulation of MEO by several researchers. Deka et al. [34] use complex coacervation of the encapsulation of PEO, by chitosan and alginate. Electron microscopy, thermal analysis, and FTIR spectroscopy were used for the characterization of the resulting complexes. Results showed that the formation of the polyelectrolyte complex was dependent on factors such as the amount of used crosslinker (glutaraldehyde), the ratio between alginate and chitosan, and pH. Where the optimum amount of these parameters was 1 mL, 5 to 4, and 3.6, respectively. Using a phosphate-buffered saline system with a pH of 7.4 as well as buffers with pH values of 4, 7, and 9 showed that the swelling microparticles and release of oil in a solution were affected by pH, and higher pH value caused higher oil release. Additionally, the antimicrobial effect of the complexes against *Staphylococcus aureus, Bacillus subtilis, Enterobacter aerogenes*, and *Proteus mirabilis* was evaluated. Where the *Bacillus subtilis* culture showed the largest inhibition zones, measuring 25 ± 0.5 mm. In another study, the complex coacervation was used to encapsulate PEO by tannic acid serving as the hardening agent for GA/gelatine microcapsules. The study examined the impact of various parameters on the efficiency of encapsulation and particle size. The parameters included the concentration of emulsifier (Tween 80) and tannic acid, as well as the concentration of the core

material and the wall material. The resulting spherical microcapsules had diameters ranging from 19 to 66 micrometers. The findings revealed that the higher concentrations of wall and core materials lead to higher particle sizes while increasing the levels of emulsifier and tannic acid decreased the particle size. While the concentrations of tannic acid and emulsifier did not have significant effects on the encapsulation efficiency, higher concentrations of the wall and core materials improved the encapsulation efficiency. Under optimal conditions of 0.02% emulsifier, 0.75% tannic acid, 5% core material, and 4% wall material, the maximum efficiency was achieved, which was 82%. Simulated gastric and intestinal fluids were used to investigate the release behavior of the microcapsules. Simulated gastric fluid with a pH of 1.2 demonstrated a significant release of the core material from the microcapsules [35]. To develop a temperature-sensitive release microcapsule to encapsulate menthol, a study was conducted to extend the duration of the fresh sensation provided by PEO. The wall material of the microcapsules consisted of, chitosan, gelatin, carboxymethyl kappa carrageenan and a cross-linking agent (lutensol). This encapsulation method offered a chemical modification approach to control the release of PEO components into the surrounding environment. The coacervation method was employed for the microencapsulation process. The morphology of the PEO microcapsules was analyzed using SEM images, while the influence of temperature on the morphology of microcapsules was examined through optical microscope images. Gas chromatography spectrum profiles were used to assess the impact of heating temperature on the release of menthol from microcapsules. The SEM analysis demonstrated that the microcapsules had a rounded shape in the absence of heating but became fragmented when subjected to heat. Moreover, the release profile of the PEO components varied with different heating temperatures of the microcapsules. Specifically, the amount of each PEO

component released was higher when the microcapsules were heated to 39°C compared to 36°C [51].

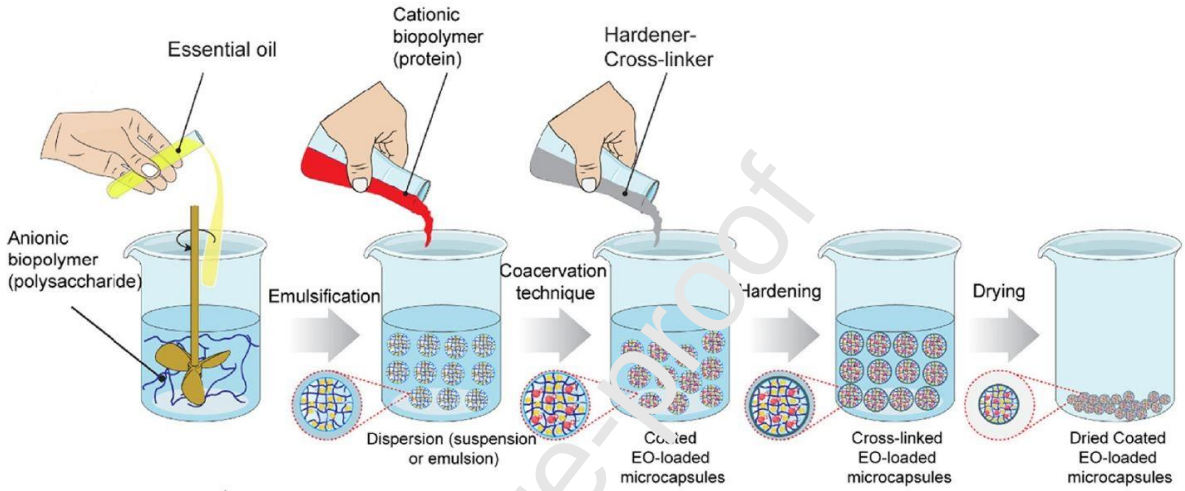

Fig. 3. Schematic image of encapsulation of EOs by complex coacervation; adapted from Weisany et al. [11].

2.2.4. Spray-drying

Emulsification and dispersion are simple methods for the encapsulation of sensitive compounds. However, the aqueous solutions obtained by these methods are not usable in tablets or capsules [52]. Spray-drying of EO-loaded emulsions is an effective, simple, and economical technique to preserve the EOs. The type of wall material is one of the major factors affecting the encapsulation efficiency and release profile of spray-dried MEO. Modified starches are one of the most widely used wall materials for spray-drying encapsulation of MEO. In this regard, Baranauskiene et al. [53] studied the effects of commercial modified starches for the encapsulation of PEO by spray-drying. To prepare the encapsulated flavors, different types of starches namely

HI-CAP 100, ENCAPSUL 855, CAPSUL, N-LOK, CRYSTAL TEX 627, CIEmCap 12633, CIEmCap12634, and CIEmCap 12635 were dispersed in deionized water to produce 30% w/w solutions and mixed with MEO (15.25%w/w of solids). Subsequently, the emulsified mixture was homogenized and spray dried and parameters such as emulsification efficiency, encapsulation efficiency, particle size distribution, flavor retention, water activity (aw), moisture content, and aroma release during storages were determined. Generally, all of the modified starches efficiently retained the MEO. However, the best results were obtained in CIEmCap 12633, followed by CIEmCap 12634, and CAPSUL. While the poorest emulsification efficiency and flavor retention were detected in the samples encapsulated by CRYSTAL TEX 627 and ENCAPSUL 855. In another study, Wang et al. [54] investigated the effect of octenyl succinic anhydride (OSA) substitution level of starch on the spray drying encapsulation of MEO. The results showed a greater flavor retention (86.68 and 90.10 %) in higher degree of substitution (DS) (0.0287 and 0.0379). The flavor release had a negative correlation with DS of starch during simulated oral processing. This might be due to the improvement of emulsifying capacity and formation of an effective interface with the increase of DS. The sustained release experienced during oral processing can be attributed primarily to the wall material's increased enzymatic resistance, microstructural stability, and lower friction coefficient.

Mehran et al. [55] optimized the spearmint EO encapsulation conditions by spray drying. The concentration of EO (4–8% w/w), the solid content (15–35% w/w), and the inlet air temperature of spray drier (110–150 °C) were the variables and the optimization was performed using response surface methodology (RSM). The wall materials including GA and inulin with the ratio of 25:75 % w/w hydrated overnight and then mixed with MEO using Ultra-turrax followed by ultrasound homogenization and then spray dried. The maximum oil retention and encapsulation

efficacy were obtained in the samples with 35 % solid content, 4% MEO, and the inlet temperature of 110 °C. The release behavior of encapsulated MEO was studied in four food simulants including distillated water, 3% acetic acid, 10 % ethanol, and 50 % ethanol. The optimized encapsulated sample showed quicker release in 50 % ethanol indicating better solubility of the wall material. Additionally, the EO release was studied by Higuchi, Korsmeyer-Peppas, and Peppas-Sahlin models and best model for describing the release behavior of EO was the Peppas-Sahlin. In another study, Mortenson and Reineccius [56] studied the effect of OSA substitution level of different carbohydrates on the properties of spray-dried encapsulated L-menthol. The hydrocolloids including dextrin, dextrinized waxy maize starch, and two types of GA obtained from *Acacia senegal* and *Acacia seyal* and were modified by different levels (1, 2, and 3%) of OSA. The investigations indicated that the modification of carbohydrates by OSA increased the encapsulation efficiency of menthol and this parameter was improved by increasing the substitution level. The solid content of spray dryer feed, had significant effects on the particle size of microcapsules. As higher solid content resulted in larger microcapsules. However, the density was not affected by the carbohydrates type and OSA degree of substitution. They stated that the increase of flavor retention and the decrease of surface menthol is due to the improvement of emulsifying properties of OSA-modified wall materials. In another study, they determined the effects of wall material on the release characteristics of menthol in different spray-dried samples using dynamic real-time analysis. The study revealed that the menthol release profile was significantly affected by the type and OSA substitution level of the wall material. Increasing the OSA content caused a greater burst of menthol release and after the characteristic burst, a decrease was observed in menthol release which might be due to the collapse of wall material [57]. In a further study, Pilicheva et al. [58] used different concentrations (10-20% w/w) and ratios of GA:

maltodextrin (100:0, 75:25, 50:50, 25:75, and 0:100) for the encapsulation of PEO (2.5% and 5%) by spray-drying. MEO was emulsified with GA, maltodextrin, Span 80, and Tween 80 and spray-dried at 170°C, 7.5 mL/min feed flow, and 35 m^3/h air flow. The particle size, morphology, production yield, and flow properties of microcapsules were investigated. The analysis indicated that the encapsulates were spherical with a mean diameter of 2.41-5.99 µm. The production yield was 71-84%, and the angle of repose was more than 35° in all of the samples indicating acceptable flowability. In conclusion, both the concentration and type of wall material, significantly affected the properties of encapsulation particles and GA alone was not able to form a stable wall material around the MEO droplets. The incorporation of maltodextrin formed a continuous film and improved the morphological properties of particles. A GA: maltodextrin ratio of 75:25 was optimal for encapsulation of MEO and higher polymer concentration (20%) resulted in higher oil retention. In addition, Sarkar and Singhal [59] and Sarkar et al. [60] esterified GA and guar gum hydrolysate (GGH) with OSA and oleic acid and used them as wall materials for the encapsulation of MEO by spray drying. The time and temperature of reaction and the concentration of acid were optimized by RSM to obtain the highest DS. The DS values obtained forGA-OSA, GGH-OSA, and GGH-oleate were 0.070, 0.072, and 0.061, respectively. The emulsion stability, particle size, and viscosity of modified hydrocolloids were investigated to determine their suitability as wall materials in encapsulation. The findings revealed that both OSA and oleic acid improved the emulsifying properties of hydrocolloids. However, OSA was more effective in improving the emulsifying potential of GGH and make it a promising alternative to GA for encapsulation of EOs. In conclusion, GA-OSA was the best wall material followed by GGH-OSA, GA, and GGH-oleate. In a further study, Sarkar et al. [23] used irradiated and enzymatically depolymerized guar gum (GG) and GA as wall materials for the microcapsulation of MEO by spray drying. The MEO

retention of microcapsules during eight weeks of storage was monitored with principal component analysis (PCA). The results indicated that the radiation depolymerized guar gum (RDGG) was a better wall material compared to enzymatically depolymerized guar gum (EDGG) for MEO encapsulation as it could better retain MEO compounds during storage. Also, the combination of GA and RDGG (90% GA and 10% RDGG) exhibited better MEO retention than GA alone in spray-dried microcapsules. The encapsulation of L-menthol in GA, HI-CAP 100 and CAPSUL modified starches by spray drying was investigated by Soottitantawat et al. [61]. Different ratios (1:9, 2:8, and 3:7) of L-menthol: wall material were examined and the results revealed that L-menthol was more stable in higher concentration of wall materials. HI-CAP 100, showed higher L-menthol retention compared to other wall materials. Additionally, the release rate of L-menthol increased by the increase of temperature and relative humidity due to the damage of wall materials.

2.2.5. Inclusion complexation

Inclusion complex systems are non-covalent molecular devices for encapsulating flavor compounds. These systems can be formed by the aggregation of starch molecules or their derivatives, such as cyclodextrins [62, 63].

2.2.5.1. Starch inclusion complex

Starch has a semi-crystalline structure that is mainly composed of two homopolymers of glucose, linear amylose (70 to 85 %) and branched amylopectin (15 to 30 %), which linked through α-(1→4)- and α-(1→6)-glycosidic linkages [64, 65]. Amylose and some linear side chains of amylopectin can form inclusion complexes with a wide range of small molecules, including iodine,

alcohols, fatty acids and their esters, as well as aroma compounds [66]. In this respect, Zhang et al. [67] reported that L-menthol and menthone can be successfully encapsulated by corn starch with the inclusion ratios of 54.7 and 43.8 %, respectively (Table 1). However, the complexing ability of starches from different botanical sources is varied and is highly dependent on their amylose content and chain length. In comparison with amylopectin, amylose molecules can form stronger complexes with guest compounds, especially non-polar molecules with a non-dominant polar group, such as menthol and menthone [66, 68]. In these complex systems, amylose is aggregated and forms a left-handed single helix structure with a hydrophobic inner helical cavity and a hydrophilic outer surface to accommodate the guest molecules [69]. The structure of amylose inclusion complexes can be investigated by the X-ray diffraction (XRD) technique, which shows a V-type crystalline pattern with major diffraction peaks around 7, 13, and 20°, also known as V-amylose. In addition, the dimensions of the V-amylose helix are controlled by the size of the guest molecule, resulting in helices with 6, 7, or 8 glucose residues per helical turn [66]. According to Ades et al. [25] findings, both menthol and menthone are able to form V-amylose complexes, and the complexation yield can improve with increasing the amylose content of the system. However, starches with various V-type crystalline patterns (i.e., V_6, V_7, and V_8-types) represented different menthol release behavior, due to their different association mechanisms with the guest molecule, such as physical adsorption in the amorphous phase or on the surface of the starch, as well as inter- and intra-helical entrapment [69]. Overall, the starch inclusion technique can be considered as a promising method for the encapsulation of MEOs, especially with high-amylose starches.

Table 1. Encapsulation of MEO and its ICs by inclusion complexation technique

Core material	Wall material	Processing conditions	Main results	Reference
L-menthol and menthone	Corn starch	0.15 g starch was heated in distilled water (15 mL) for 1 h at 100 °C. Afterward, the aroma compound (host-to-guest ratios of 10:0.6-10:1.0 g/g or mL) were slowly added and heated at 30-70 °C for 0.5 h, then freeze-dried	□ The resulting inclusion ratios were 54.7 and 43.8 % for L-menthol and menthone, respectively □ The major peak of C=O at 1771 cm^{-1} revealed the inclusion complex formation	[67]
Menthone	Mung bean, tapioca, and rice starches	0.8 % w/v starch dispersion was stirred at 140 °C for 1 h. Then, 0.1 % v/v menthone was added at 80 °C and left for slow cooling	□ Mung bean and tapioca complexes showed higher menthone entrapment (4 %) than rice (<1 %) □ Mung bean starch was the best wall material for menthone encapsulation	[68]

L-menthol and menthone	Amylose or waxy, normal, and high-amylose corn starches	600 mg amylose or defatted starches was stirred in 100 mL KOH at 90 °C. Then, 60 mg menthol or 60 μL menthone was added and stirred at 60 °C for 0.5 h. Finally, the suspension was acidified (pH 4.8) and held at 60 °C for 24 h	□ Both menthone and menthol were able to form V-amylose complexes □ The starch-aroma complexes were stable at high temperatures and variety of pH □ Complexation improved with increased in amylose content	[25]
L-menthol	High-amylose corn starch	V-type high-amylose corn starch and L-menthol (1:1) were heated at 70 °C for 0.5 h	□ V_6, V_7, and V_8-types showed different release rates, due to their different association mechanisms with menthol □ V_6-type amylose could encapsulate menthol more effectively	[69]
L-menthol	α-, β-, and γ-CDs	10-30 % maltodextrin solution was heated to 80 °C and cooled to 25 °C. Then, 400 ppm L-menthol and α-, β-, and γ-CDs were added to maltodextrin solution and left for 12 h	□ Increased in the retention of menthol □ β-CD is the best encapsulant material	[70]

Mentha piperita EO	α-, β-, and γ-CDs	CDs were polymerized with epichlorohydrin (EP). Soluble polymers were dialyzed overnight to remove all impurities, while insoluble polymers were sieved and the fraction <125 μm were used during the study	□ Menthol, menthone, pulegone, and eucalyptol were identified as the major components □ β-CD could perform the controlled release of aroma compounds	[71]
L-menthol	α-, β-, and γ-CDs	1 mmol α-, β-, or γ-CDs and 8 mmol KOH solution were stirred 3.5 h, then ultrasonic dispersion for 0.5 h. The solution was filtered and 50 mL L-menthol was added and allowed to crystallize for 3-5 weeks	□ β-CD showed a better thermal stability, with an initial thermal degradation temperature of 253 °C □ Encapsulation efficiency and menthol content were 22.54 and 21.76 %	[72]
D- and L-menthol	β-CD	10 mL β-CD and 0.5 mL D- or L-menthol (15.6 mg, 0.01 mM) were stirred at 60 °C until complete solution and left for slow cooling	□ Formed inclusion complexes with both enantiomers of menthol □ The stability constants of both complexes were the same (163 M^{-1})	[73]
L-menthol	β-CD	10 g β-CD and 3 g L-menthol were stirred for 3 h at 45 °C. Then, the suspension was stored at 5 °C for 24 h	□ Increased in the thermal stability of menthol from 120 to 267.5 °C □ The minimum binding energy was -116.7 kJ/mol at -0.8 × 10^{-10} m	[74]

L-menthol	β-CD	1 mmol β-CD and 8 mmol KOH solution was filtered, followed by methanol diffusion at 25 °C for 3-5 weeks. Then, L-menthol was added in a sealed pressure vessel for continuous heating	□ Increased in the thermal stability of menthol □ Encapsulation efficiency and menthol content were 30.6 and 27.1 %	[75]
Mentha x villosa EO	β-CD	*Mentha x villosa* EOs and β-CD were complexed with a ratio of 1:9 by co precipitation and kneading methods in a hydroethanolic medium	□ 22 compounds were totally or partially complexed, while 5 compounds were not complexed □ Encapsulation efficiency and volatiles retention were 13.6 and 77 %	[76]
L-menthol	Hydroxypropyl-β-CD	10 g hydroxypropyl-β-CD was dissolved in 20 g water at 45 °C. Afterward, 2 g L-menthol were added and stirred at 45 °C for 2 h, then freeze-dried	□ Two menthol release peaks were observed at 69.3 and 279.1 °C □ The minimum binding energy was -127 kJ/mol at 3.5×10^{-10} m	[77]

CD: Cyclodextrin; EO: Essential oil.

2.2.5.2. Cyclodextrin inclusion complex

Cyclodextrin (CD) complexation is another technique to improve the solubility, stability, bioavailability, and releasing behavior of bioactive compounds. CDs are water-soluble macrocyclic oligosaccharides, which can be obtained from starches using enzymatic treatment (i.e., CD glycosyltransferase). Similar to starch inclusion complexes, the CD-based complexes are also represented a hydrophobic interior and a hydrophilic exterior due to their cone-shaped structure, which is favorable for the encapsulation of non-polar molecules [78, 79]. CDs are mainly composed of 6, 7, or 8 glucose units, which are called α-, β-, and γ-CDs (Figure 4). Consequently, the molecular dimensions of their hydrophobic cavity are varied for each type of CD. The type of CD should be selected based on the size of the guest molecule to achieve the maximum encapsulation efficiency [80].

Generally, the effective complex formation between CDs and flavor compounds is highly dependent on the hydrophobicity of the guest molecules to form hydrogen bonds or van der Waals forces with CDs and their geometric accommodation into the CD cavity [81]. Compared with α- and γ-CDs, β-CD showed better thermal stability, controlled release behavior, and efficiency for the encapsulation of MEO compounds [70-72]. Furthermore, Ceborska et al. [73] reported that β-CD can successfully form inclusion complexes with both enantiomers of menthol (i.e., D- and L-menthol). Additionally, Zhu et al. [74] showed that the menthol-β-CD inclusion complexes had significantly higher thermal stability compared to non-capsulated menthol EOs with the minimum binding energy of -116.7 kJ/mol at -0.8×10^{-10} m. Similar results were reported by Hu et al. [75], who demonstrated that the controlled release and thermal stability of menthol were improved using β-CD metal-organic framework delivery systems. However, in some cases, β-CD represented the lowest water solubility among other kinds of CDs [82]. Martins et al. [76] stated that among the

28 identified compounds in MEO, only 12 of them were completely complexed with β-CD, while 11 were partially complexed, and the rest were not complexed. In this regard, several strategies have been applied to overcome the shortages of β-CDs and improve their functional abilities. For instance, Zhu et al. [77] evaluated the possible application of hydrophilic synthetic derivative of β-CD, hydroxypropyl-β-CD, for the encapsulation of L-menthol. The results showed that the shelf-life, stability, solubility, heat release, and activation energy of L-menthol were significantly improved in L-menthol-hydroxypropyl-β-CD inclusion complexes.

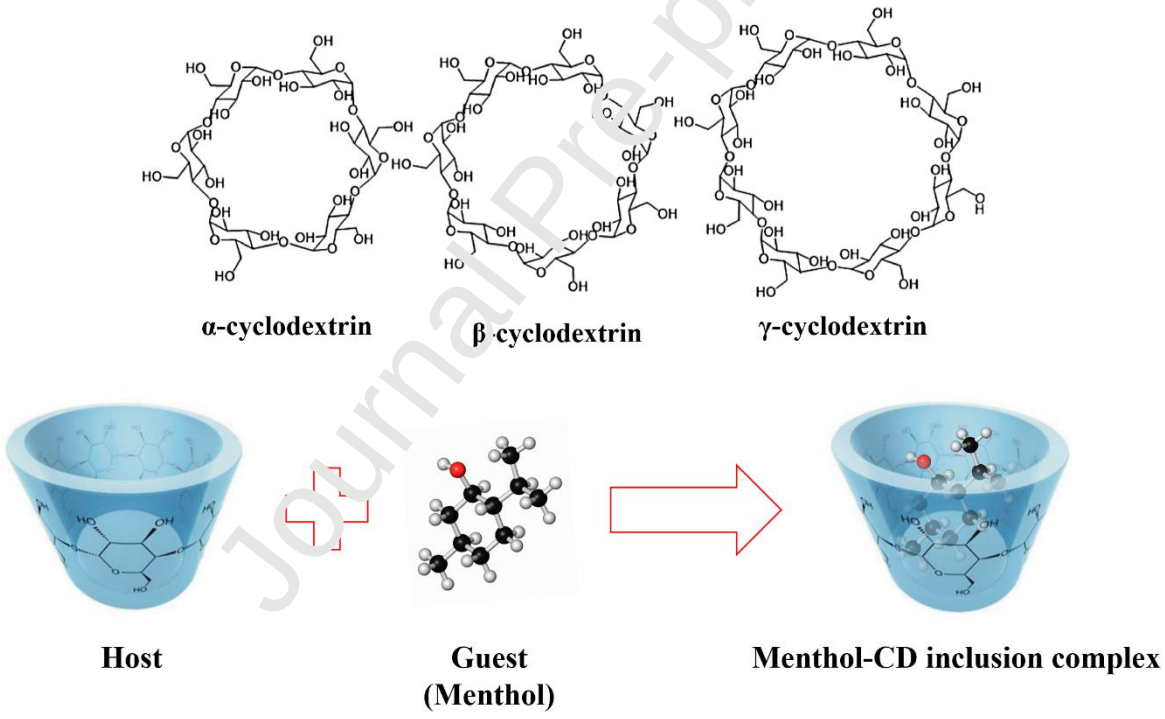

Fig. 4. The structure of α-, β-, and γ-cyclodextrins (CDs) and menthol-CD inclusion complex.

2.2.6. Electrospinning and electrospraying

Electrospinning and electrospraying are emerging electrohydrodynamic approaches that have garnered significant attention in recent years for the encapsulation of bioactive compounds, including MEO and its ICs. Electrospinning, in particular, is a straightforward method that offers several advantages, such as the ability to precisely control fiber properties like diameter, morphology, porosity, and scalability. [83]. The process involves transmitting polymer through a nozzle tip via a pump and using high voltage between the nozzle and collector to create an electric field that results in the formation of a Taylor cone and fiber collection on the collector surface [84]. This process is performed at ambient temperature and leads to the production of fast and efficient release systems.

Various researches have been conducted in the field of electrospinning in encapsulating MEO and its ICs and have revealed promising results, as shown in Table 2. For instance, Ye et al. [85] used coaxial electrostatic atomization to encapsulate L-menthol in silk fibroin (SF). They fabricated menthol-loaded fibers and capsules with an average diameter of 1-2 μm, and stated that the spinnablity and structural features of the electrospun/electrosprayed fibers or core-shell particles can be amended by changing the ratio of components in the core and shell materials. Additionally, the loading capacity and encapsulation efficiency of fibers were higher than particles. In another study, Mira et al. [86] produced Poly (methyl vinyl ether-alt-maleic anhydride) (PMVEMA) electrospun nano/microfibers loaded with 16% of terpene menthol. PMVEMA with molecular weights of 216 and 1080 kDa was applied for the fabrication of fiber mats and bead-free and uniform microfibers were developed. The results showed that fibers obtained from the high M_W PMVEMA were ribbon-like with high encapsulation efficiency while the fibers obtained from low M_W PMVEMA were cylindrical with lower encapsulation efficiency. Also, according to the in vitro biological assays, cytotoxicity was not detected in none of the

samples. Besides, Rezaeinia et al. [21] employed the electrospinning technique to fabricate a multilayered fiber mat loaded with menthol using Balangu seed gum (BSG) and gelatin. They conducted a comparative analysis of the menthol release rate during the oral phase between the multilayered mat and a gelatin monolayer mat. In sandwich structures, menthol was loaded into gelatin layer and sandwiched between PVA/BSG hybrid layer. While, the monolayer structure was composed of a menthol-loaded gelatin fiber mat. Atomic Force Microscopy (AFM) revealed that the electrospun mats had smooth surfaces and mesh-like structures. The multilayered mats increased the contact angle, and dissolution time but decreased the bioadhesive strength. XRD, DSC, and FTIR tests proved the thermal stability and successful entrapment of MEO in fiber mats.

Table 2. Encapsulation of MEO and its ICs by electrohydrodynamic techniques

Electrospun/electrosprayed polymer(s)	Polymer(s) concentration	Menthol concentration	Processing conditions						Reference
			Syringe (mL)	Flow rate (mL/h)	Distance from tip to collector	Voltage (kV)	Particle diameter	Encapsulation Efficiency (%)	
SF	1–10 wt%	50–100 wt%	10	0.2	10 cm	10	963 nm	46	[85]
PMVEMA	12 %	2.3%	2	0.25	10 cm	15.5	837-1369 nm	68-92	[86]
BSG and PVA/ gelatin	0.5% w/v BSG + 10% w/v PVA/ (20, 25, and 30%) gelatin	3% v/w	1	0.5	150 mm	25	218.28-237.28 nm	92.15	[21]
HPγCD and HPβCD	160% w/v	-	1	0.5 and 1	10-15 cm	10-15	590-1005 nm	-	[87]
PS/ α-, β- and γ-CDs	20% w/w α-CD, 24% w/w β-CD,	3.3%	1	1	10 cm	15	0.30-4.25 µm	-	[88]

	and 27% w/w γ-CD*								
PMMA/ α-, β- and γ-CDs	10% w/v PMMA/ 31% w/w α-CD, 36% w/w β-CD, and 41% w/w γ-CD	5% W/W	1	1	10 cm	5	543-907 nm	-	[89]
Alginate and pectin	80% alginate + 20% pectin	-	1	0.5	10 cm	5	1.58-3.24 µm	82	[90]
BSG and PVA	0.25% BSG + 1% PVA	0.015 g	1	1	150 mm	25	-	77.56-87.82	[91]
PEG	7.31 %	10.7%	5	1	15 cm	17	1136 nm	-	[92]

BSG: Balangu seed gum; PEG: Polyethylene glycol; PMVEMA: Poly (methyl vinyl ether-alt-maleic nhydride); PVA: Poly vinyl alcohol; PMMA: Poly (methyl methacrylate); PS: Polystyrene; CD: Cyclodextrin; SF: Silk fibroin; HPγCD: Hydroxypropyl-γ-cyclodextrin; HPβCD: Hydroxypropyl-β-cyclodextrin.

* Each type of CD forms 1:1 (molar ratio).

In another study, Yildiz et al. [87] produced menthol/CD-inclusion complex with hydroxypropyl-γ-cyclodextrin (HPγCD) and hydroxypropyl-β-cyclodextrin (HPβCD) and used them to fabricate electrospun nanofibers (Fig. 5). The modeling and phase solubility studies indicated that the highly concentrated solutions of HPβCD or HPγCD and menthol in a molar ratio of 1:1 produced stable inclusion complexes which increased the water solubility of menthol. The concentrated solutions were successfully electrospun and produced uniform and bead-free nanofibers with the average diameter of 590±230 to 1005±285 nm. The menthol release from nanofiber was slow and its thermal stability was improved as the evaporation was observed at very high temperatures (up to 275°C). Moreover, Uyar et al. [88] produced Polystyrene (PS) fibers comprising cyclodextrin (α-, β- and γ-CDs)-menthol inclusion complexes by electrospinning to create functional fibrous webs. Complexation of menthol with CDs enhanced the thermal stability of menthol up to 350°C, while the fibers without CD-menthol complex were not heat stable. The highest thermal stability and the best release properties of menthol were observed in γ-CD compared to α-CD and β-CD. In a further study, Uyar et al. [89] studied the electrospinning of poly (methyl methacrylate) (PMMA) nanofibers containing α-, β- and γ-CDs-menthol inclusion complexes for producing functional nanofibers. The mass spectrometry results exhibited that the menthol evaporation occurred at high temperatures and in a wide temperature range. This behavior indicated the successful complexation of menthol in the CD cavity. The size of CD cavity affected the thermal stability of menthol, γ-CD had the largest cavity and the highest stability followed by β-CD and α-CD.

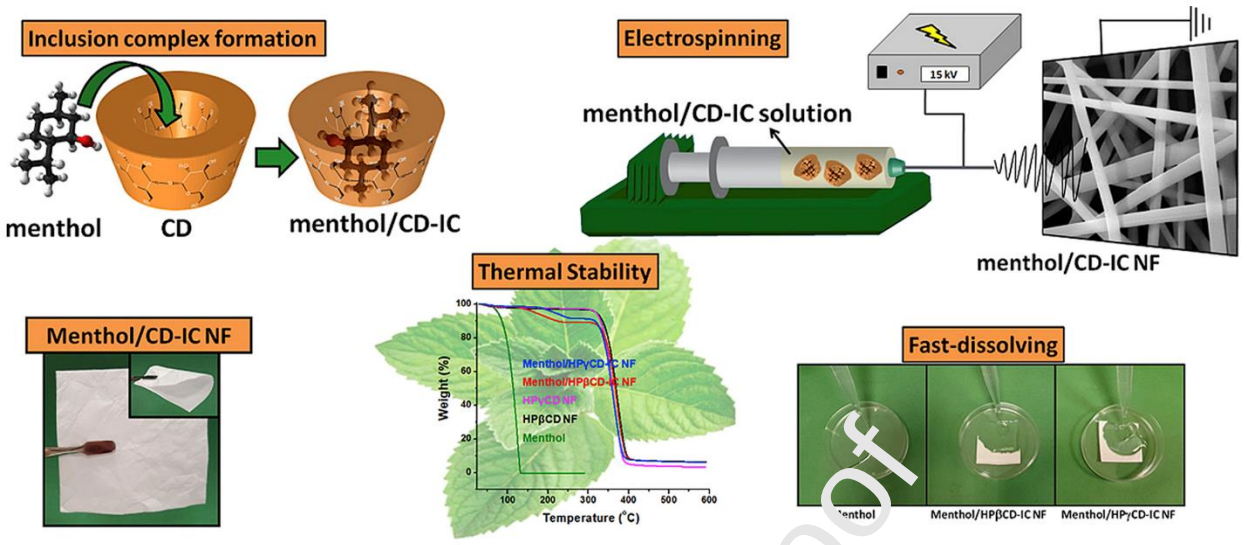

Fig. 5. Encapsulation of MEO into hydroxypropyl-γ-cyclodextrin (HPγCD) and hydroxypropyl-β-cyclodextrin (HPβCD) by electrospinning technique; adapted from Yildiz et al. [87].

Koo et al. [90] used electrospraying for the microencapsulating of PEO in an alginate-pectin matrix. They analyzed the composition and properties of the resulting microcapsules, and found that the ratio of alginate to pectin had an impact on both encapsulation efficiency and stability (the maximum efficiency was obtained with the 80% alginate and 20% pectin combination). The study's conclusion highlights that this microencapsulation method can produce a suitable alginate-pectin microcapsule with appropriate rheological properties and encapsulation efficiency. In another study, Rezaeinia et al. [91] nanocapsulated *Mentha longifolia* L. EO into BSG by electrospraying process (Fig. 6). The electrosprayig solutions was composed of MEO (0.015 g), PVA (0.5 and 1%), BSG (0.25 and 0.5% w/w), and Tween-20 (0.06, 0.08 and 0.1%). The optimal formulation contained 1% PVA, 0.25% BSG, and 0.08% Tween-20. The Peppas-Sahlin model was used to predict the EO release profile, which followed a Fickian diffusion

mechanism with a burst release within the first 3 minutes and sustained release for 180 minutes. Besides, AALI et al. [92] evaluated the possible preparation of menthol/polyethylene glycol (PEG) micro/nanoparticles with various menthol (10, 15, and 20% (wt)) and PEG (5, 10, and 15% (wt)) concentrations using electrospraying method. According to the scanning electron microscopy (SEM) images, the particle diameters increased with increasing the concentrations of PEG and menthol. Also, the best menthol and PEG concentrations, were 10% (wt) and 7.31% (wt), respectively, using RSM.

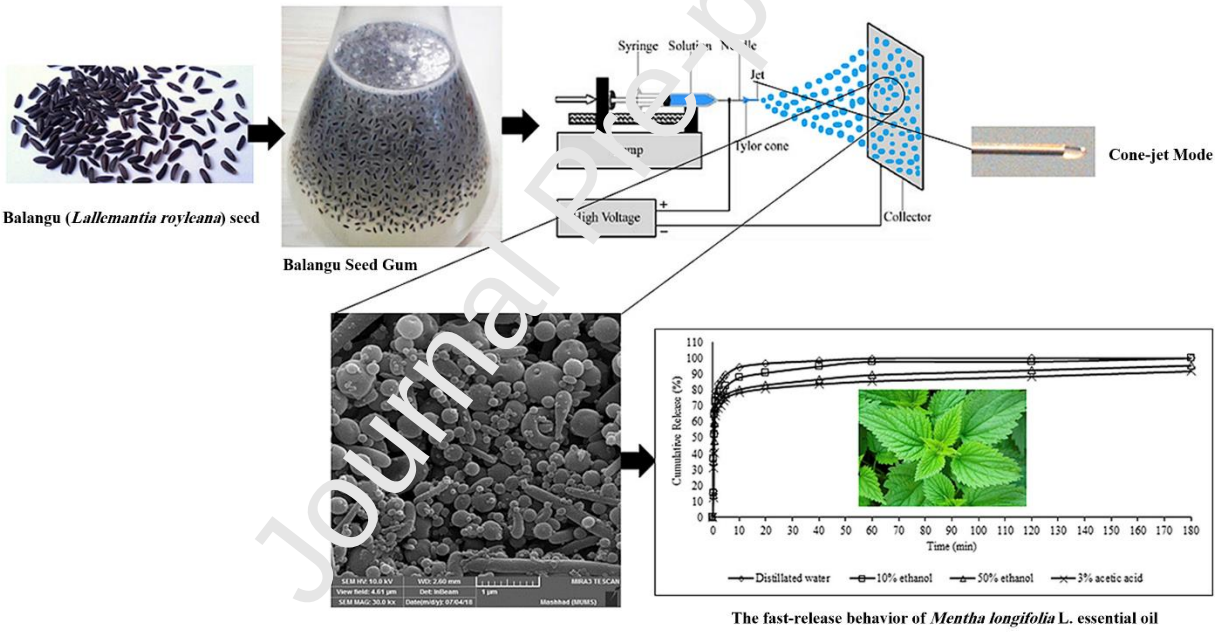

Fig. 6. Encapsulation of MEO into Balangu seed gum (BSG) by electrospraying technique; adapted from Rezaeinia et al. [91].

2.2.7. Melt dispersion and melt homogenization

Melt dispersion is a simple, fast, convenient, and inexpensive encapsulation method. In this method, the EOs are finely dispersed in molten wall material and then solidify, grind, and pass through a sieve to produce powder microparticles. The operating temperature in this technique is quite low; therefore, it is appropriate for encapsulation of heat sensitive and volatile compounds such as EOs. The encapsulation of EOs by melt dispersion offers several advantages such as high oil load, slow-release properties, and enhanced stability. In this method, the wall materials with thermal stability at 30-200°C, low melt viscosity, and low melting point (32-85°C) are required. Waxes, fatty acids, fatty alcohols, glycerides, and their derivatives are commonly used in melt dispersion. Polyethylene glycol (PEG) is one of the most widely used materials in the formation of solid particles by melt dispersion due to its low melting points (55–65°C), low cost and non-toxicity [93, 94]. In this regard, Yuliani et al. [95] investigated the properties of MEO encapsulated in polyethylene glycol (PEG 6000) by melt dispersion technique. PEG 6000 was heated to melt at 65°C, subsequently different concentrations (5, 7.5, 10, and 12.5%) of MEO were added to PEG and mixed on a magnetic stirrer. The molten mixtures were transferred to plates and solidified by cooling to room temperature. The samples were crushed and passed through a sieve to obtain encapsulated MEO. The size, morphology, and thermal properties of encapsulates were evaluated and it was found that the particles had irregular flake shapes with an average size of 10 μm. However, the TEM images revealed that the oil droplet size ranged 200-500 nm and samples with higher MEO content had higher particle sizes which indicate the formation of agglomerated and large particles at high levels of MEO. The melting enthalpy of encapsulated MEO was higher than free mint oil, indicating the improvement of heat stability in encapsulates. The samples containing up to 10% MEO were free-flowing powders with great heat stability, high yield, and small particles. In a similar study, Kumar et al. [96] prepared PEO nanoparticles by the melt dispersion

technique. PEG 6000 was heated to 65°C to melt, mixed with 5, 7.5, or 10.0% (w/v) MEO and cooled to 25°. After solidification, the encapsulates were grinded and sifted to obtain encapsulated MEO particles. The encapsulates showed a wide range of particle sizes and high polydispersity index. It is a disadvantage that has negative impact on the controlled release of MEO. Furthermore, the polydispersity index (PDI) can be reduced by incorporating homogenization and suitable surfactants into the mixture of molten wall material and EOs; this process is known as melt homogenization. In this respect, Ghodrati et al. [97] and Khezri et al. [98] encapsulated *Mentha pulegium* EO into nanostructured lipid carriers (NLCs) by melt homogenization method. MEO was dissolved in Miglyol, mixed with melted Poloxamer, dissolved in water, homogenized at 70°C, and then cooled to 24°C to produce solid lipid nanoparticles. The results revealed that the NLCs have high entrapment efficiency and MEO loading and low polydispersity index indicating that melt homogenization is an appropriate method for encapsulation of MEO. In a similar study, Piran et al. [99] fabricated menthol-loaded NLCs by melt homogenization method and compared the morphology, size distribution, particle size, encapsulation efficiency, stability, and antimicrobial properties of NLCs with conventional menthol-loaded emulsions. The menthol-loaded NLCs were spherical with an average particle size of 115.6 nm and narrow size distribution. The menthol encapsulation efficiency was very high (98.73%), and its stability in NLCs after 90 days of storage was very high. The antibacterial tests indicated that NLCs presented higher antimicrobial activity than the conventional emulsion.

3. Recent applications of encapsulated MEOs

MEO, being a bioactive compound, possesses a wide range of functionalities, making it a suitable ingredient for the formulation of various products such as foods, drugs, healthcare items,

and agro-biological products. While MEO is non-toxic to humans, it has been proven to exhibit high toxicity towards pests and pathogens. The antimicrobial activity of MEO stems from its ability to disrupt the lipid components within the membrane, resulting in altered permeability and subsequent release of intracellular substances. [100]. Therefore, MEO can be used in foods and therapeutic products as an antimicrobial agent or a natural preservative. Additionally, MEO is a larvicidal, pesticidal, and insect repellent compound. MEO is a great antioxidant in human body or food matrix that reduces the detrimental effects of reactive oxygen species. Consequently, MEO can be considered as an anticancer supplement for humans or as a natural antioxidant that can prevent the spontaneous oxidation of foods. Also, MEO compounds such as menthol have cooling effects that can be used in chewing gums, mouthwashes, toothpastes, and oral care products. However, the rapid release and degradation of MEO is the main challenge that prevent its extensive applications. Several studies have revealed that encapsulation is an effective technique in increasing the stability of MEO and expanding its applications, which are reviewed in this section.

3.1. Edible coatings and films

Due to their high moisture content, fruits and vegetables are prone to bacterial and fungal infestations, resulting in spoilage and loss. To mitigate this issue, the application of suitable treatments, such as antimicrobial coatings and films, can effectively minimize postharvest losses and maintain the safety and quality of fruits and vegetables throughout storage. Natural agents such as EOs can be added to the films and coatings to reduce the decay of fruit and vegetables. MEO, particularly in encapsulated form, is an effectively antimicrobial agent that increases the shelf life of fresh fruits and vegetables. In a study by Yang et al. [101], encapsulated menthol was added to layer-by-layer (LBL) self-healing edible coatings and used to increase the shelf life of

apples. The coatings were produced from sodium alginate (SA) and β-cyclodextrin grafted chitosan (β-CD-g-CS). Menthol was encapsulated to the β-CD-g-CS solution and LBL coatings were deposited on glass slides with alternating layers of SA and menthol loaded β-CD-g-CS. Subsequently, the coatings were separated from the slides and stored at relative humidity of 56% and temperature of 22°C for 24h. The results indicated that the encapsulated menthol increased the smoothness, transparency, self-healing, and mechanical properties of edible coatings, Additionally, a controlled release of menthol was observed during storage. The coatings preserved the firmness, decreased the weight loss, and effectively improved the shelf life of apples. In another study, Beyki et al. [102] encapsulated different concentrations (100-1200 ppm) of *Mentha piperita* EOs into chitosan–cinnamic acid nanogel and used it as an antifungal coating for tomatoes. The tomatoes were inoculated by *A. flavus* and stored in a refrigerator for 30 days. The control tomato sample (inoculated by *A. flavus* without menthol coating) started to decay after 3 days of storage. While, the samples treated by 800 and 900 ppm of free menthol deteriorated after 10 and 14 days, respectively. The application of 100ppm encapsulated MEO preserved the post-harvest quality of tomatoes during 30 days of storage.

Shahbazi et al. [103] evaluated the effect of encapsulated *Mentha longifolia* L. EO into carboxymethyl cellulose-gelatin (CMC-GE) nanofibers by electrospinning on extending the shelf-life of peeled freshwater prawns stored for 14 days at 4°C. Adding MEO to fibrous films improved their properties, particularly CMC-GE-MEO 1% and CMC-GE-MEO 2%, showed lower force and tensile strength but higher water vapor permeability, swelling index, antimicrobial, and antioxidant activities. Incorporating MEO (0.5-2%) into CMC-GE nanofibrous films extended the shelf-life of peeled freshwater prawns compared to the control group. CMC-GE-MEO 2% prolonged the shelf-life of prawn to 14 days under refrigeration. These nanofibrous films with antimicrobial and

antioxidant activities have a high potential for active packaging of raw freshwater prawns. Moreover, Eghbalian et al. [104] fabricated sodium caseinate-gelatin (SC-GE) electrospun fiber mats loaded with *Mentha spicata* L. EO (0, 0.5, and 1%) and nano-magnesium oxide (MgO; 0 and 0.1%) and assessed their impact on prolonging the shelf-life of trout fillets. The nanofibers exhibited significant in vitro growth inhibition against *L. monocytogenes* and *S. aureus*. The trout samples packaged in SC-GE + MgO 0.1 % + MEO 0.5 % and SC-GE + MgO 0.1 % + MEO 1% had significantly lower microbial populations, thiobarbituric acid, peroxide, and total volatile base nitrogen than the control group after 13 days of storage. Therefore, inclusion of MEO and MgO to the SC-GE nanofibers is a promising method to enhance the effectiveness of antimicrobial agents in food packaging.

3.2. Chewing gum

In chewing gums, achieving a sustainable and controlled release of flavoring agents is crucial. Moreover, there has been a recent development of medicated chewing gums, specifically designed for the administration of dietary supplements and drugs to children. However, these components often possess an unpleasant taste that necessitates effective masking [105]. Encapsulation is an appropriate method to obtain a palatable and pleasant formulation in chewing gums. Santos et al. [106] co-encapsulated xylitol and menthol by double emulsion method and complex coacervation and incorporated the capsules into chewing gums. The concentration of xylitol in the emulsions was 50 g/100g emulsion; while, the menthol content was 2 or 6 g/100g. The microcapsules showed spherical shapes and high encapsulation efficiency. The samples with 6 g of encapsulated menthol showed the highest encapsulation efficiency and the lowest release rate. Additionally, co-encapsulation prolonged the cooling effects of mint-flavored chewing gums.

Furthermore, Yoshii et al. [107] studied the release behavior of menthol encapsulated by different wall materials (GA, β-CD, γ-CD, CAPSUL, and HI-CAP 100 modified starches) in chewing gums. Based on the obtained results, the chewing gums incorporated with menthol encapsulated into γ-CD showed the longest releasing time and the highest activation energy; while the encapsulation of menthol in β-CD showed the shortest release time and the lowest activation energy.

3.3. Pesticide and insecticide

The control of agricultural pests and vector-borne diseases by using synthetic insecticides has increased the pesticide-resistant organisms, and imposed adverse effects on human health and ecosystem [108]. On the other hand, the consumer awareness of the detrimental effects of synthetic pesticides has increased in recent years and led to the development of natural pesticides. The botanical insecticides can be used as a promising alternative to synthetic materials. Several studies have revealed that MEO and its ICs have insect repellent, insecticidal, and larvicidal properties. However, the rapid evaporation and poor water solubility of MEO have limited the extensive application of this natural compound. Encapsulation is an effective approach in increasing the stability, water solubility and controlled release of MEO. In this regard, Mohafrash et al. [108] obtained *Mentha spicata* EO nanoemulsions by ultrasonic emulsification and Tween 80 as an emulsifier. Subsequently, they used the emulsions as a larvicidal substance against *Musca domestica* and *Culex pipiens*. Sonication for 10 min and MEO: Tween 80 ratio of 1:1 (v/v) produced emulsions with an average droplet size of 97.8 nm. These nanoemulsions showed great larvicidal properties against *M. domestica* (LC50, 65.13 μg/mL) and *C. pipiens* (LC50, 43.57 μg/mL) compared to free MEO. Additionally, these nanoformulation showed a controlled release during storage and were not toxic for the treated rats. Therefore, these nanocapsules can be used

as effective, safe, and eco-friendly formulations to control larvae. In addition, Rajkumar et al. [109] encapsulated PEO in chitosan nanoparticles with ionic gelation and investigated its insecticidal activity against *S. oryzae* and *T. castaneum* as two common grain pests. The particle size, Zeta potential, and encapsulation efficiency were 563.3 nm, −12.12 mV, and 64%, respectively. The natural pesticide showed significant effects against both pests compared to the control. However, it was more effective against *S. oryzae* than *T. castaneum*. In another study, Faraji et al. [110] encapsulated *Mentha pulegium* and *Ferula gummosa* EOs in nanoliposome and compared their effects against *Tribolium castaneum* with free EOs. The results indicated that the encapsulation efficiency of *M. pulegium* and *F. gummosa* EOs were 99.38% and 96.41%, respectively. The release (%) of EOs was 46% for *M. Pulegium* and 33 %, for *F.gummosa* nanoliposomes. Generally, these findings revealed that the fumigant toxicity of EOs against *T. castaneum* was increased in nanoliposomes in comparison with the free EOs. Besides, *Mentha piperita* EO was encapsulated by melt-dispersion. The nanocapsules contained 5–10%, w/v MEO with an encapsulation efficiency of 78.2–83.4%. The lethality rate of the MEO-loaded nanoparticles against housefly larvae was 100% in lab [96]. In a further study by Kavetsou et al. [111], *Mentha pulegium* EO was encapsulated in commercial baker's yeast and used against *Myzus persicae* as a common agricultural pest. They reported that the loading capacity of microparticles was 29-36% and the insecticidal activity of encapsulated MEO was prolonged to 3 days.

3.4. Dental care products

Mint is widely recognized and favored for its flavor in toothpaste and mouthwash formulations. To enhance the stability and achieve a sustained release of mint in these products, various encapsulation techniques can be employed. Encapsulation serves to physically isolate the

MEO from the surrounding environment, guarding against potential chemical reactions and bolstering its stability throughout storage. Ashrafi et al. [26] developed chitosan nano-gels loaded with *Mentha piperita* EO (MPEO) for their antibiofilm properties against *Streptococcus mutans*, a bacterium associated with dental plaque formation. The nano-gels were synthesized using a sol-gel method with tripolyphosphate (TPP) acting as a linking bridge. To characterize the MPEO-loaded chitosan nanoparticles (MPEO-CNs), their physicochemical properties were assessed using various techniques such as FTIR, SEM/EDX, DLS, and zeta potential measurements. The release kinetics of MPEO from the nano-gels, as well as the minimum inhibitory concentration (MIC) and minimum bactericidal concentration (MBC), were determined. Furthermore, the impact of sub-MIC concentrations of MPEO-CNs on the expression of biofilm-associated genes was investigated. The adherence of bacterial cells to the MPEO-loaded nano-gels exhibited higher sensitivity compared to unloaded chitosan nano-gels. The MPEO-CNs demonstrated antibiofilm activity against *S. mutans* at concentrations of 50 µg/mL, while unloaded nano-gels required a concentration of 400 µg/mL. Among the biofilm synthesis genes, some genes showed minor changes upon treatment with MPEO-CNs, while others experienced significant down-regulation in the presence of both unloaded and MPEO-loaded nano-gels. This study highlights the potential of MPEO-CNs as an effective nano-formulation, specifically targeting key glycosyltransferase genes involved in extracellular polymers, and suggests their potential use as an anti-biofilm agent in toothpaste or mouthwash formulations. In another study, Xue and Zhang [112] encapsulated PEO with GA and whey protein by complex coacervation at different pH values (4, 5, and 7) for dental care applications. They reported an encapsulation efficiency of 85% at pH 3 and 4 and 80% at pH 7. After 7 days of storage the samples encapsulated at pH 4 showed the highest stability while pH 7 were not stable and preserved only 20% of menthol.

3.5. Topical administration

3.5.1. Wound healing

Bacteria are the predominant microorganisms found on the surface of intact skin as well as in wounds. Consequently, the colonization of bacteria poses a significant challenge in wound healing, often causing delays or even hindering the process entirely. Wound infections are commonly attributed to pathogens like *Staphylococcus aureus* and *Pseudomonas aeruginosa*, which can cause considerable pain and, in severe cases, even lead to fatality. To address this issue, innovative wound dressings have been developed to administer therapeutic compounds, including natural antibacterial agents, directly into infected wounds.MEO is a natural antimicrobial that can be loaded into wound dressings. However, the EOs are unstable and should be encapsulated to be effective in wound healing. In this respect, Khezri et al. [98] produced NLCs for protecting *Mentha pulegium* EO (MPO) and increasing its antimicrobial activity in wound dressings. The MPO-NLCs showed significant antibacterial effect against *Staphylococcus epidermidis*, *Staphylococcus aureus* and *Listeria monocytogenes* as gram positive bacteria and *Pseudomonas aeruginosa* and *Escherichia coli* as gram negative bacteria. The inflammatory phase of wounds was shortened by the topical administration of MPO-NLCs and its proliferative phase was improved compared with the control group. In addition, Ghodrati et al. [97] conducted a study to investigate the effectiveness of NLCs laded with PEO in wound dressings. The wound healing of PEO-NLC dressings was studied in mice model. The results showed that the PEO-NLC-treated animals had higher rate of wound contraction, collagen deposition, fibroblast infiltration, and re-epithelialization. Moreover, Unalan et al. [113] fabricated electrospun fiber mats with different concentrations of PEO incorporated to PCL, to produce antibacterial fibers for wound healing applications. The

electrospun fiber mats had a smooth, bead-free, and uniform morphology, with a reduced fiber diameter when PEP was added. The fibers showed antibacterial properties against *Escherichia coli* and *Staphylococcus aureus* and improved the cell viability in normal human dermal fibroblast (NHDF) cells. The study suggests that MEO loaded PCL fiber mats could be a promising candidate for wound healing applications.

3.5.2. Skin care products

MEO and its compounds such as menthol can be used in several skincare products as natural coolants, antiseptic, antipruritic, analgesic, and anti-inflammatory agents. However, the water insolubility, volatility, low penetration, and irritancy prevent the topical application of these products. Yingngam et al. [114] used nanoprecipitation technique for the encapsulation of menthol in lipid-core nanoparticles and studied its skin application. Box-Behnken design was used to explore the effect of proportions of menthol, poloxamer 188, and PCL on the zeta potential, hydrodynamic diameter, and encapsulation efficiency. The optimized formulation contained 150 mg menthol, 125 mg poloxamer 188, and 84 mg PCL. The physicochemical characteristics, cytotoxicity, irritancy permeation, and skin localization of menthol-loaded nanocapsules were investigated and the results revealed that the nanocapsules have great performance and are safe for topical applications. In another study, Uhlířová et al. [115] developed an innovative nanofiber patch loaded with *Mentha piperita* EO for acne vulgaris treatment. The minimum inhibitory concentration (MIC) and minimum bactericidal concentration (MBC) were determined to investigate the antimicrobial activity of patches against *Cutibacterium acnes* and *Staphylococcus epidermidis*. The MEO were integrated into gelatin nanofibers by electrospinning and showed a strong antibacterial activity against *S. epidermidis* and *C. acnes*. Therefore, the antimicrobial

nanofiber patches containing MEO are suitable for the local treatment of acne vulgaris. Moreover, Mishra et al. [94] produced an ointment containing microcapsulated *Mentha spicata* L. var. viridis EO as an antifungal agent. They used the simple coacervation method to encapsulate MEO in chitosan and optimized the processing conditions. The optimum microcapsules with the highest encapsulation efficiency were obtained at MEO:chitosan ratio of 5.4:5, cross-linker content of 1.62% w/v and mixing speed of 1000 rpm. The microcapsules retained 96.92% of MEO even after 8 weeks of storage. Therefore, microcapsulation can be used to increase the therapeutic efficacy of MEO.

Zague et al. [116] compared the cooling intensity of topical emulsions incorporating free or encapsulated menthol. The findings indicated that the cooling intensity of emulsions with encapsulated menthol was not reduced during 28 days of storage at 37°C. It shows that these emulsions are highly stable and can be used in skincare products. While, the emulsions containing free menthol showed a significant reduction in the cooling intensity. In another study, Song et al. [117] produced a topical emulsion loaded with PEO as an anti-itching and antifungal product and examined its effect on pigs. The bacterial exopolysaccharide (EPs) extracted from *Bacillus vallismortis* were used as emulsifier in MEO-laded O/W emulsions. It was demonstrated that EPs had great emulsifying properties at a concentration of 1% w/v. The emulsions were stable and their average particle size was 16.3 μm. The antifungal tests demonstrated that the MIC of the emulsified MEO was 4.0 mg/mL, while this value was 16 mg/ml for the free MEO against *Candida albicans*. These findings indicate that MEO emulsions exhibited superior effectiveness compared to free MEO in inhibiting fungal growth. The antipruritic test demonstrated that the emulsions significantly raised the threshold for itchiness in pigs when exposed to phosphate histamine. Moreover, the MEO emulsions successfully reduced the frequency of scratching incidents

observed in pigs.In a further study, Lamarra et al. [118] produced nanofiber mats based on PVA loaded with PEO for topical applications. Different rations (100:0, 87.5:12.5, 82:18, and 75:25) of PVA:emulsified MEO were mixed with each other and nanofibers were fabricated by electrospinning. SEM images displayed that increasing the ratio of emulsions decreased the fiber diameter and their mechanical properties. Moreover, MEO improved the elongation and structural integrity of nanofibers compared to single PVA fiber mats. The optimum formulation with the best mechanical properties was found to be the sample with 82% PVA and 18% MEO emulsion. It was used for topical applications and came in direct contact with the porcine skin explant. Confocal microscopy revealed the localization of MEO particularly on the stratum corneum after 1h of contact. However, after 7 h, MEO was distributed in the viable epidermis. Therefore, it has the potential to be used for topical or transdermal treatments as a skin penetration enhancer.

3.6. Cancer treatment

Cancer is a major cause of death and one of the most important public health problems. There are numerous treatments such as chemotherapy, radiotherapy, and surgery for cancer therapy. However, these treatments have several disadvantages and side effects such as drug resistance, nausea, and vomiting. As a result, plant-derived drugs such as EOs have received a significant amount of attention for the treatment of cancer. However, these natural compounds are less effective than synthetic drugs. Developing nanoformulations is a promising strategy to improve the effectiveness of EO-based drugs. Several studies investigated the influence of EO-based drugs on treatment of different types of cancer in recent years. For instance, in a study by Kelidari et al. [119], the anticancer effect of *Mentha longifolia* and *Mentha pulegium* EOs and their nanocapsules was investigated against melanoma A-375 cell line and breast cancer MCF-7

and MDA-MB-468 cell lines. Even at the highest concentration (1200 μg/mL) of free EOs a desirable cytotoxic effect was not detected on the tested cell lines as the cell viability was above 55%. On the other hand, a concentration of 600 μg/mL of the encapsulated MEOs reduced the cell lines viability to approximately 10%. Therefore, the MEO-based nanoformulations could be suitable candidates for further investigation in the in-vivo research and also as a supplementary medicine for cancer therapy. In addition, Abedinpour et al. [120] evaluated the anticancer properties of *Mentha piperita* EO and its nanoemulsions against three breast cancer cell lines during 24, 48, and 72 h incubation periods. They produced the nanoemulsions by mixing 25 μL MEO, 10-200 μL tween 20, and 10–200 μL ethanol and then added phosphate-buffered saline to reach 5000 μL. The best nanoemulsion with the highest anticancer properties with a mean droplet size of 136 ± 2 nm contained 10 μL tween 20, and 4965 μL phosphate-buffered saline (PBS). The anticancer effect of this formulation was considerably better than non-emulsified MEO. The effectiveness of MEO nanoemulsion after 24h exposure was significantly higher than that of MEO even after 72 h of exposure.

In a further study by Nirmala et al. [121], the effectiveness of *Mentha arvensis* EO nanoemulsions against anaplastic thyroid cancer cells was investigated. The nanoformulations were developed by sonication of MEO, water and tween 80. The ratios of oil:surfactant were 1:1, 1:2, and 1:3 and the oil content of all of the samples was 6%. The optimal conditions for the production of nanoemulsion was sonication time of 20 min, and oil: tween 80 ratio of 1:3. The anticancer effect of MEO nanoemulsions was investigated by colony formation assay, MTT, and Annexin V apoptotic assay. The result of Annexin V-FITC assay, evidently showed the induction of early apoptosis in anaplastic/aggressive thyroid cancer cell line (HTh-7). Besides, Azadi et al. [122] encapsulated *Mentha pulegium* EO into nanoemulsion with a droplet size of 7.7 nm.

Subsequently, carboxymethyl cellulose (2% w/v) was added to the nanoemulsions to produced nanogels. Different concentrations of nanoemulsions were applied on human A375 melanoma cells. Cell viability was studied by the MTT assay, and flow cytometry was applied to confirm the cell apoptosis. Furthermore, qPCR (quantitative Polymerase Chain Reaction) was used to evaluate the gene expression of apoptotic and anti-apoptotic cells, such as Bcl-2 and Bax. The results revealed that treatment with 300 μg/mL of nanogel reduced the that cell viability by 90%; while, this value was 45% for nanoemulsion. The flow cytometry, indicated that this behavior was due to apoptosis. The gene expression analysis indicated that the expression of Bax genes increased while the Bcl-2 gene expression decreased. Consequently, MEO loaded nanogels can be considered as powerful anticancer agents. In another study, Tubtimsri et al. [123] produced spearmint oil loaded nanoemulsions by phase inversion temperature technique and used the nanoemulsions against oral cancer cells. Different formulations of nanoemulsions were produced by using various concentrations and types of surfactants, oil loading, and MEO/virgin coconut oil (VCO) ratios. The nanoemulsions comprising polyoxyethylene sorbitan fatty acid esters (PSF80) and polyoxyethylene castor oil derivatives (Cremophor®RH40; PCO40, Kolliphor®EL; PCO35, Eumulgin®CO60; PCO60) displayed 100% creaming after temperature cycling test, whereas the samples containing PCO40 revealed more transparency and stability. Increasing the concentration of PCO40, decreased the droplet size and addition of more than 5% (w/w) PCO40 formed nano-sized (<1000 nm) droplets. Moreover, the MEO-VCO loading and ratio affected the droplet size as the nanoemulsions were developed at 25% MEO-VCO and MEO-VCO ratio of 40:60 to 80:20. The MTT assay results revealed that the MEO-VCO nanoemulsions had considerable cytotoxic effects against oral carcinoma (KON) cell line.

4. Challenges and safety issues in encapsulation of MEOs

Since the encapsulated MEO and its ICs are used in functional food products or nutraceutical supplements, a thorough understanding of their safety and the related mechanisms is essential. Therefore, the safety of bioactive compounds (core materials), wall materials, and encapsulation techniques should be carefully evaluated and monitored.

The highest recommended daily dose of MEO in the European Union (EU) is 1.2 mL [124]. According to the FDA, menthol is generally considered as a safe material for using in topical over-the-counter (OTC), cosmetics, and food products. The safety profile of menthol in concentrations up to 16% has been confirmed by various vitro and in vivo studies for OTC external use [125]. However, a conservative maximum daily exposure of 0.0074 mg/kg was determined for cosmetics [100]. In addition, the acceptable intake of menthol is quoted as 0.2 mg/kg per day [126]. These findings reveal the low potential of menthol to cause toxicity in humans. However, some cases of menthol poisoning has been reported due to the excessive consumption of menthol (50-150 mg/kg), which eventually led to coma and fatal intoxication [127, 128]. In addition to menthol, some other MEO compounds, such as menthone, menthofuran, and pulegone also showed toxic effects in certain conditions, which can cause hepatotoxicity, nephrotoxicity, carcinogenicity, or mutagenicity in animal tests at high concentrations [124, 129]. This is why it is so crucial to determine the chemical composition of newly introduced mint species before consumption.

The other important factor regarding the toxicity and safety of micro- or nano-carriers of mint bioactive compounds is the composition and structure of the wall material, which is comprehensively explained in Section 2.1. Generally, the membranes should be selected among the GRAS materials, which consist of a wide range of synthetic and natural ingredients, especially

where the encapsulated material directly deals with human health, such as food and pharmaceutical industries.

Nanotechnology has provided promising solutions to enhance the delivery of bioactive compounds in the pharmaceutical, agricultural, food, and cosmetic industries [130]. However, the use of sub-micron materials raises toxicity and safety issues, as nano-sized materials exhibit different physical and chemical characteristics compared to their macro-scale counterparts [131]. Although the FDA has confirmed the possible application of nano-based approaches in the food industry, the potential hazards of nano-materials cannot be ignored due to their highly reactive nature. It also has been hypothesized that the safe substances may show toxicity when nano-sized, leading to doubts as to whether these nano-materials deserve distinct regulatory frameworks [132, 133]. On the other hand, the potential harmful effects of nano-particles and nano-encapsulated materials, their pathways, and the way in which they interact with the human body are still unclear.

Overall, the micro- or nano-encapsulation of mint bioactive compounds is an effective and safe way to improve their functional, biological, and delivery properties, especially by using green wall materials and encapsulation techniques. However, more clinical research is required to investigate the safe daily intake of these encapsulated compounds for long-term consumption and their actual mechanisms in the human body.

5. Concluding remarks and future trends

Numerous researches indicated the great prospects for mint essential oil (MEO) as a bioactive ingredient in foods, cosmetics, pharmaceuticals, fragrances, and agrobiological products. Additionally, its nutritional, biomedical, insecticidal and herbicidal effects are well-documented.

However, MEO is easily damaged due to its sensitivity to oxygen, light, heat, and reactions with other compounds. The volatility, rapid release, and degradation prevent the extensive application of MEO in commercial products. For the successful commercialization of MEO and to overcome its sensitivity to the environmental conditions, encapsulation is suggested.

Encapsulation provides protection against these undesirable conditions and facilitates the application, storage, and transfer of MEO. Numerous methods such as emulsification, coacervation, ionic gelation, inclusion complexation, spray drying, electrospinning, melt dispersion, and melt homogenization have been used for the encapsulation of MEO and its compounds particularly menthol. It has been confirmed that these techniques could improve the stability, bioactivity and controlled-release properties of MEO, and carry MEO to the targeted sites. The physicochemical, morphological, and release properties of MEO micro/nanoparticles obtained by different wall materials and encapsulation methods have significant differences. Thus, a suitable methodology and wall material are needed to prepare stable and economically feasible MEO micro/nanoparticles. Choosing an appropriate method depends on the desired properties and application of the encapsulates.

Generally, the current studies focus on the process optimization and properties of encapsulated MEO and less on practical applications. Therefore, the production feasibility of encapsulated MEO on a large scale should be considered in future studies. On the other hand, promising results are reported in in vitro studies; but, a limited number of studies addressed the application of encapsulated MEO in real systems. Further studies are needed to investigate the application of different types of MEO encapsulates in various products, animals and human body.

Declaration of Competing Interest

The authors declare no conflict of interest.

Data availability

The article includes any data that support the findings of this study. No new data were created or analyzed in this study.

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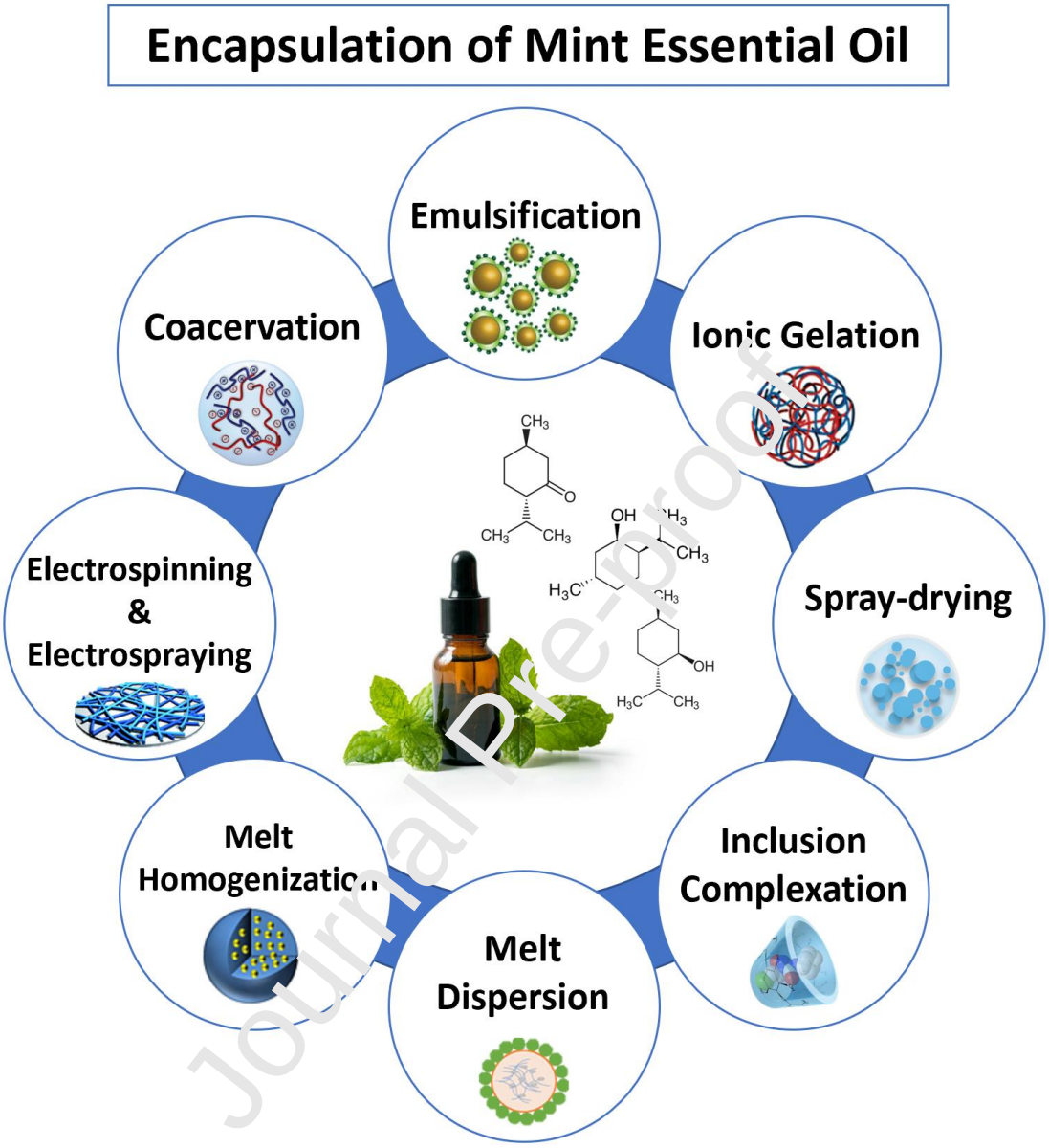
Graphical abstract



Highlights

- Encapsulation improves the stability and solubility of mint essential oil (MEO)
- Encapsulation extends the industrial applications of MEO
- Several methods have been emerged for the encapsulation of MEO
- Encapsulated MEO can be used in food, bioagricultural, pharmaceutical, and health care products