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Encapsulation of essential oils in cyclodextrins: A review of extraction, inclusion-complex characterization and food applications

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Abstract
Essential oils (EOs) are complex mixtures of volatile plant metabolites with antimicrobial, antioxidant and anti-inflammatory properties, which explains their use in the food, pharmaceutical, cosmetic, agrochemical and hygiene sectors. Their high volatility, poor water solubility and sensitivity to light, heat and oxygen, however, limit their practical use. This review examines how encapsulation, and in particular complexation with cyclodextrins (CDs), can overcome these limitations. It first describes the composition and bioactivity of EOs, using black pepper, guava leaf and yarrow oils as examples, and compares conventional and innovative extraction techniques. General micro- and Nano encapsulation methods (spray drying, solvent evaporation, extrusion, freeze drying and supercritical-fluid processing) are then summarized, followed by the structure and properties of CDs. The formation of EO–hydroxypropyl-β-cyclodextrin (HPβCD) inclusion complexes is discussed on the basis of scanning electron microscopy, FT-IR and UV–Vis evidence, which consistently shows that the oils are accommodated within the CD cavity, although encapsulation efficiency is lower for whole oils than for isolated constituents. Finally, CD-based food systems (inclusion complexes, nanosponges and biopolymer composites) are compared with other nanocarriers. The literature indicates that CD complexation protects EO constituents, masks their sensory impact and permits controlled release, whereas safety assessment, regulatory clarity and scalable production remain priorities for future research.

Keywords: Essential oils, cyclodextrins, hydroxypropyl-β-cyclodextrin, encapsulation efficiency, nanoencapsulation, food technology applications

Introduction

Science plays a significant role in education, society and the economy, and it can improve quality of life while helping to address pollution, social tension and inequality. One of its main objective is to reduce dependence on hazardous synthetic pesticides and to replace them with safer alternatives. This aim is consistent which call for a global shift towards healthier and more sustainable food production and consumption. Modern agriculture relies heavily on pesticides because they raise yields and profits. As part of the Green Deal, the European Union's Farm to Fork Strategy therefore aims to halve pesticide use and the associated risks by 2030 [1]. At the same time, the EU remains the world's largest exporter of pesticides, including substances that are prohibited within its own member states [2]. In effect, compounds banned at home are shipped to regions with weaker regulation, and a considerable share of the agricultural produce that the EU imports originates from those same regions.

Despite all initiatives, pesticide residues are still found in food, animal feed, soil and water, with consequences for the health of humans, pets, and non-target beneficial organisms. Exposure to pesticides has been linked to higher rates of chronic disease. Depending on the compound, residues may persist from a few hours to several years, and wind and water can carry them far beyond the site of application. Prolonged use can disrupt habitats and food chains and ultimately, cause lasting, large-scale damage to biodiversity [3].

Essential oils (EOs) obtained from medicinal and aromatic plants are attractive natural alternatives to synthetic pesticides [4]. These complex mixtures of volatile compounds are recovered from different plant parts by a variety of extraction techniques, and their chemical diversity focuses on their use in the food, textile, pharmaceutical, medicinal, agrochemical, cosmetic and hygiene industries. Interest in EOs now extends well beyond fragrance. In foods they serve as preservatives and flavor enhancers, because their antimicrobial activity helps to maintain freshness while contributing taste [5, 6], and they are also found in many pharmaceutical products. Continuing research into their biological and molecular modes of action as shown in Fig. 1 is expected to support the development of more targeted drugs.

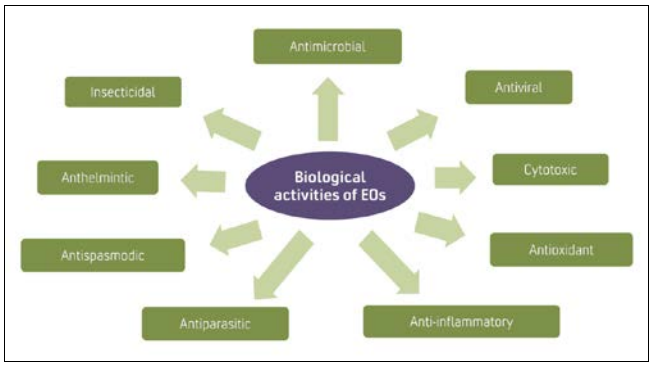


Fig 1: Principal biological activities attributed to essential oils (EOs).

In spite of this strong bioactivity, the practical use of essential-oil constituents (EOCs) is restricted by their poor bioavailability [7]. EOs also degrade readily on exposure to light, heat, oxygen and humidity [8], and many EOCs suffer from low water solubility, high volatility or an unpleasant odor. Encapsulation addresses most of these shortcomings [9,

[10], and microencapsulation in particular is widely adopted [11] because it protects the oil, preserves its properties and allows its release to be controlled.

Cyclodextrins (CDs) are among the most popular molecular hosts in supramolecular chemistry. They are cyclic oligosaccharides whose hydrophobic cavity can accommodate a guest molecule, provided that the guest fits the cavity; in solution this process is driven by non-covalent forces such as hydrogen bonding, electrostatic and van der Waals interactions [12]. Once an inclusion complex has formed, the physical and chemical properties of the guest are favorably modified.

This review surveys the encapsulation of EOs in CDs, with emphasis on hydroxypropyl-β-cyclodextrin (HPβCD). Section 2 outlines the composition and limitations of EOs, using black pepper, guava leaf and yarrow oils as examples, and Section 3 compares techniques for their extraction. Section 4 reviews general encapsulation principles and methods, and Section 5 introduces the structure and types of CDs. Section 6 discusses how EO–HPβCD inclusion complexes are characterized by electron microscopy and spectroscopy, Section 7 considers CD-based nanoencapsulation for food applications, and Section 8 summarizes the conclusions and future perspectives.

Essential Oils

1. Composition, bioactivity and stability limitations

In vitro and *in vivo* studies have shown that EOs possess antioxidant and antibacterial activity and show promise as antitumor and anti-inflammatory agents [13, 14]. Compounds such as eugenol, carvacrol and thymol are largely responsible for these effects, since they provide strong antioxidant and antimicrobial action (Fig. 2).

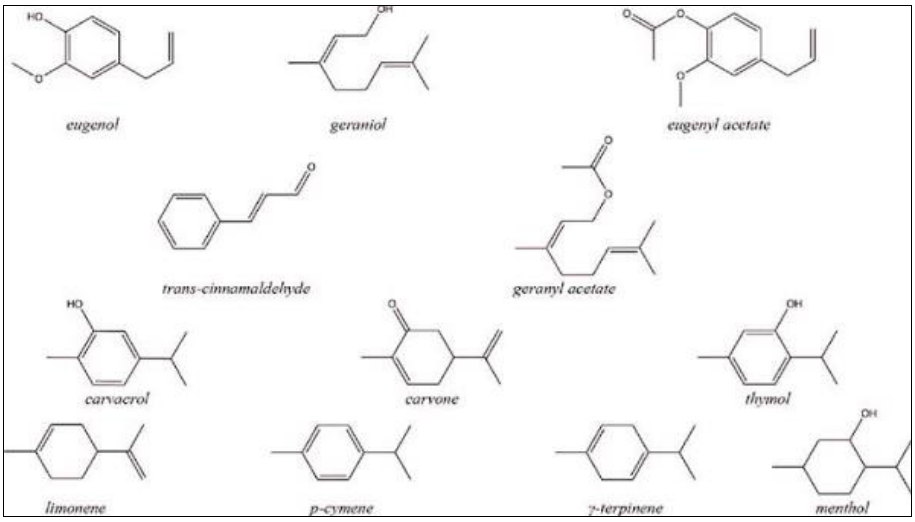


Fig 2: Chemical structures of representative essential-oil constituents: eugenol, geraniol, eugenyl acetate, trans-cinnamaldehyde, geranyl acetate, carvacrol, carvone, thymol, limonene, p-cymene, γ-terpinene and menthol.

Many of the active compounds in EOs are, however, degraded by heat, light or oxygen. When added to food, even minute amounts of EOs can produce an overpowering flavor, and their poor miscibility with water limits contact with microorganisms in aqueous food matrices, which reduces antimicrobial efficacy [15, 16]. The following three oils illustrate these points and are used as model systems in the characterization studies discussed in Section 6.

2. Model essential oils

Black pepper. Black pepper oil is obtained from *Piper nigrum* L., often called the "king of spices" because of its pungent piperine [17]. The plant is used in medicine, food, preservation and even as a biocontrol agent [18], and its oil has demonstrated antioxidant and antimicrobial properties [19]. Chemically, the oil consists mainly of terpenes, notably β-caryophyllene, limonene, δ-3-carene and pinene [20] (Fig. 3).

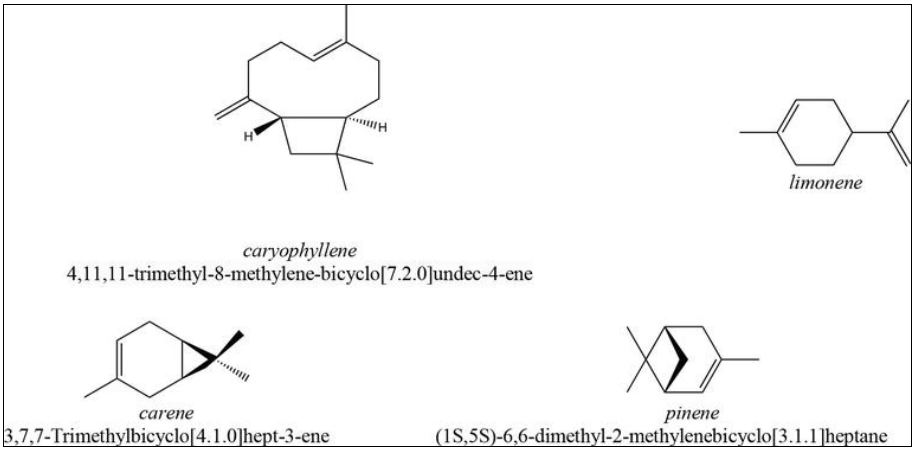


Fig 3: Major constituents of black pepper essential oil: β-caryophyllene, limonene, δ-3-carene and pinene.

Guava leaf. Guava (*Psidium guajava* L.) has a long history of use in traditional medicine. Bioactive compounds in guava leaf oil inhibit cell growth, act as antioxidants and kill microorganisms. Its main constituents are limonene, β-caryophyllene, 1,8-cineole and α-pinene.

Yarrow. Yarrow (*Achillea millefolium* L. s.l.) is notable for its wide range of pharmacological effects and has long been used in folk medicine [21, 22]. In Europe it has been applied to digestive complaints, diabetes, liver and biliary disorders and menstrual problems, and its anti-inflammatory and anticancer properties are also valued. Its essential oil contains many important compounds, including carvacrol, linalool, 1,8-cineole, camphor and a considerable amount of thymol [23] (Fig. 4). Carvacrol and thymol, however, are poorly stable towards light, oxygen and heat and decompose rapidly when unprotected. Encapsulating the yarrow oil is an effective way of shielding these fragile components.

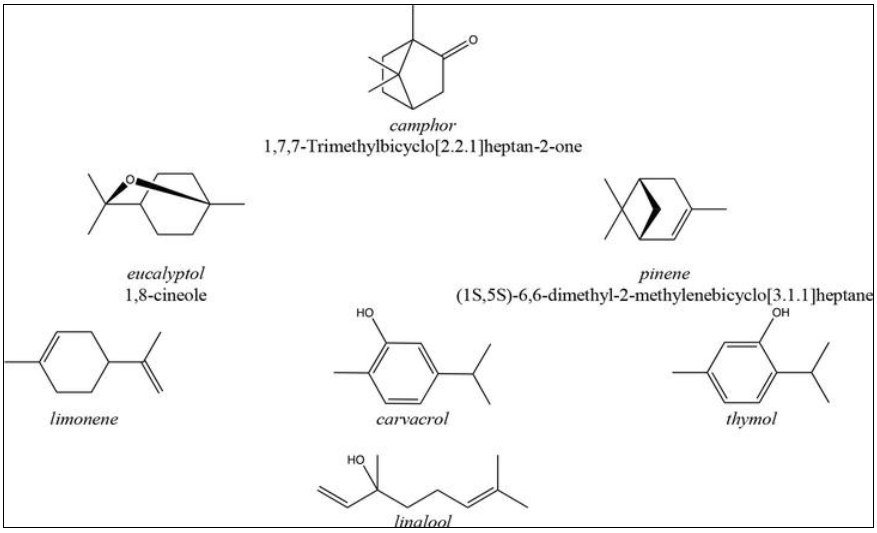


Fig 4: Principal constituents of yarrow essential oil: camphor, 1,8-cineole (eucalyptol), pinene, limonene, carvacrol, thymol and linalool.

Extraction Techniques

Aromatic plant material (leaves, flowers, bark, seeds or fruits) is harvested at maturity and stored under controlled light, temperature and humidity before the oil is recovered. The available extraction methods fall into two groups. Conventional methods include distillation, in which heated water releases the oil from the plant matrix, as well as cold pressing and organic-solvent extraction. Innovative methods have been introduced to overcome the drawbacks of the conventional ones: microwave-assisted extraction and supercritical fluid extraction (SCFE), for example, are more environmentally friendly and energy-efficient [24], and SCFE and subcritical liquid extraction are suitable for non-polar constituents. The advantages and limitations of the individual techniques are summarized in Table 1.

1. Steam distillation

Steam distillation is one of the most widely used techniques for obtaining EOs. It rests on the same principles as hydrodistillation, but the extraction time is shorter because the plant material does not come into direct contact with the water. The sample is packed into a column that is connected at its base to a flask of heated water, and the steam produced passes through the sample and carries the volatile oil to a condenser at the top of the column (Fig. 5). The condensed water–oil mixture can then be separated by liquid–liquid extraction [25]. The technique is applied extensively at commercial scale to oils such as lavender and patchouli, and to hops [26]. A Clevenger-type apparatus, which collects the separated oil layer directly, is shown in Fig. 6.

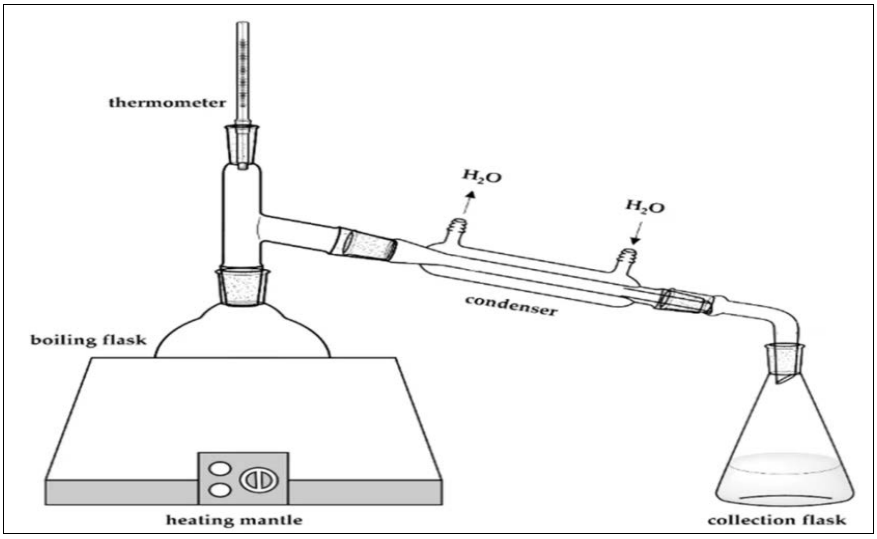


Fig 5: Laboratory distillation set-up for the isolation of essential oils (boiling flask with heating mantle, condenser and collection flask).

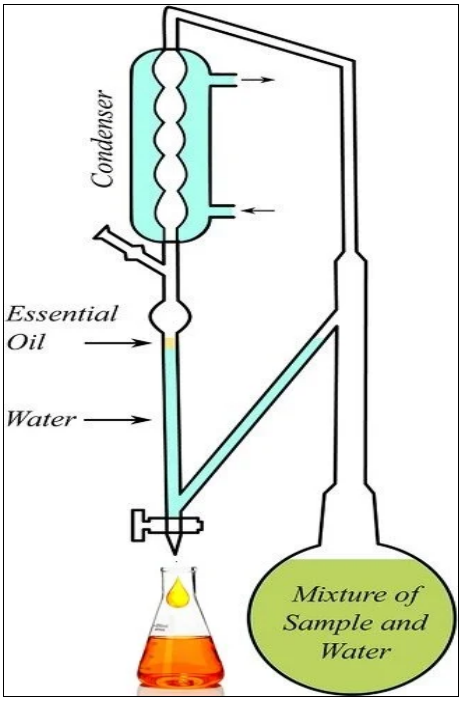


Fig 6: Clevenger-type apparatus for hydrodistillation, showing the condenser and the separation of essential oil from water.

2. Organic-solvent extraction

Some oils, such as jasmine and rose, have low thermal stability and cannot tolerate the high temperatures of distillation. For these, inexpensive, chemically inert organic solvents with a low boiling point can be used. In organic-solvent extraction the sample is kept in contact with a solvent such as petroleum ether, hexane, benzene or toluene for a sufficient period so that the soluble material passes into the solvent. The solvent is subsequently evaporated to concentrate the extract. When yields are high, this approach removes the need for a final filtration step and keeps the sample in continuous contact with fresh solvent, as in Soxhlet extraction [27]. Solvent extraction is the conventional technique used most often in the cosmetics industry [28].

3. Microwave-assisted extraction (MAE)

The demand for more environmentally friendly and energy-efficient processes has made microwave-assisted extraction an alternative to conventional methods. The sample is placed in a solvent-free microwave reactor, where electromagnetic energy is converted into heat, the internal temperature of the plant cells rises and the moisture inside them evaporates [29] (Fig. 7). The resulting increase in internal pressure ruptures the oil glands and releases the essential oil [30]. MAE has been used to extract EOs from a number of plant matrices, including orange, laurel, lemon, mint, rosemary and basil.

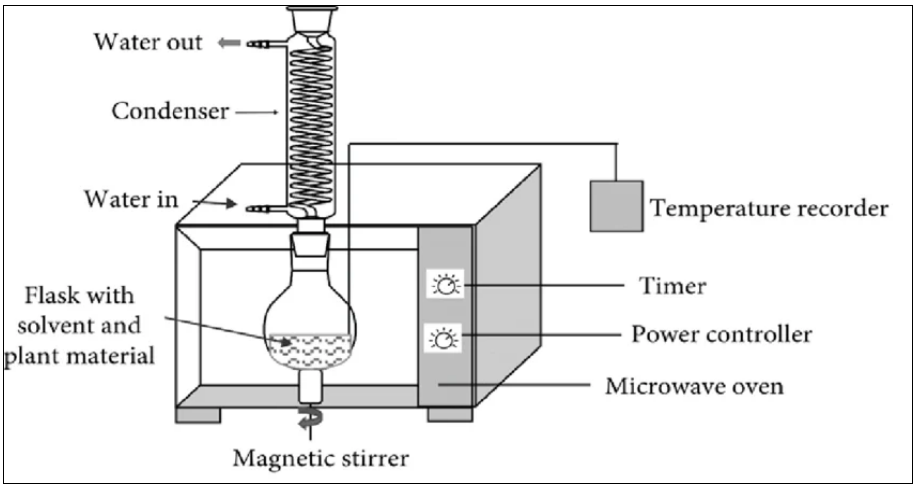


Fig 7: Schematic diagram of microwave-assisted extraction (MAE) of essential oils.

4. Supercritical fluid extraction (SCFE)

Supercritical fluid extraction is a clean, efficient and environmentally responsible way of separating EOs. Supercritical fluids are used as extractants because their properties suit the process: low viscosity, high diffusivity (and therefore high penetrating power) and a density close to that of a liquid. Water, carbon dioxide (CO_2), methane, ethylene and ethane can all be used as supercritical solvents, but CO_2 is the most common choice because of its low cost, non-flammability, low toxicity and reactivity, and easily reached critical point (31.2 °C and 72.9 atm). Once the optimal extraction temperature and pressure have been set,

the supercritical fluid dissolves and extracts the oil from the sample, and the extract solution is then held at a pressure below the critical point [31]. On depressurization the fluid turns into a gas that no longer has any solvating power [32], so the extract separates from the solvent. Commercial use of this technique is growing for EOs from the leaves of flowering plants, the horseradish tree, laurel, rosemary and sage.

Table 1: Advantages and limitations of conventional and innovative techniques for essential-oil extraction.

Method type	Technique	Advantages	Limitations
Conventional	Steam distillation	Reduced loss of polar constituents; shorter extraction time than hydrodistillation	Long extraction times; higher cost and formation of undesirable compounds because of the prolonged processing time
	Organic-solvent extraction	1. Simple, inexpensive and fairly effective 2. Suitable for small-scale operations	1. Time-consuming 2. Excessive solvent consumption 3. Limited scope for accelerating the process by agitation 4. Solvents may alter the chemical composition of the final product or have harmful effects
Innovative	Supercritical fluid extraction (SCFE)	1. Short extraction time 2. Low-toxicity solvents 3. Solvent-free extract	High installation, maintenance and equipment costs
	Microwave-assisted extraction (MAE)	1. High repeatability 2. Easy handling 3. Minimal solvent use 4. Reduced energy input 5. Enhanced extraction yield	Filtration or centrifugation is required as a final step

Encapsulation of Essential Oils

1. Principles

Encapsulation establishes a functional barrier between a core material and its coating (wall) material. The barrier preserves the biological, functional and physicochemical characteristics of the core and prevents unwanted chemical and physical interactions. The most suitable method and wall material depend on the final application of the product and on the processing conditions, and the wall material determines particle stability, process efficiency and the degree of core protection [33]. Because bioactive compounds have inherent limitations, a barrier that restricts exchange with the surroundings is particularly attractive: during handling, production and storage, the encapsulated material is shielded from temperature, moisture, light, oxidation, undesirable interactions with other ingredients and mechanical stress [34]. For essential oils, encapsulation therefore extends shelf life and maintains biological and functional properties over long storage periods.

2. Capsule size, structure and release

Depending on size, polymer coatings used to enclose solid, liquid or gaseous material yield macrocapsules (>5000 μm), microcapsules (0.2–5000 μm) or nanocapsules (<0.2 μm) [35]. Nanoencapsulation refers to the nanoscale enclosure of one or more substances within another material. Microencapsulation is a well-established approach that works with larger particles than nanoencapsulation, and macroencapsulation involves larger particles still [36]. In a typical procedure, the material to be encapsulated is suspended in a solution and coated by dissolving and precipitating the encapsulating agent. Alternatively, the encapsulating agent and the core molecule are dissolved in the same solvent and precipitate simultaneously (co-precipitation), so that the encapsulating layer contains the chemical particles in the form of microspheres or microcapsules [37]. By isolating the core, the encapsulating agent protects it and allows its release at the desired site and time in response to a specific stimulus.

Microspheres and microcapsules are shown schematically in Fig. 8. Microspheres are matrix systems in which the core is uniformly distributed or dissolved in a polymer network, whereas microcapsules have a large inner core containing the active ingredient, surrounded by a layer of encapsulating agent that forms the capsule membrane. Depending on whether the core is present as particles (suspended) or in a molecular state (dissolved), microspheres can be homogeneous or heterogeneous [38]. The encapsulating agent must not react with the core and must be able to seal it inside the capsule and protect it from adverse conditions.

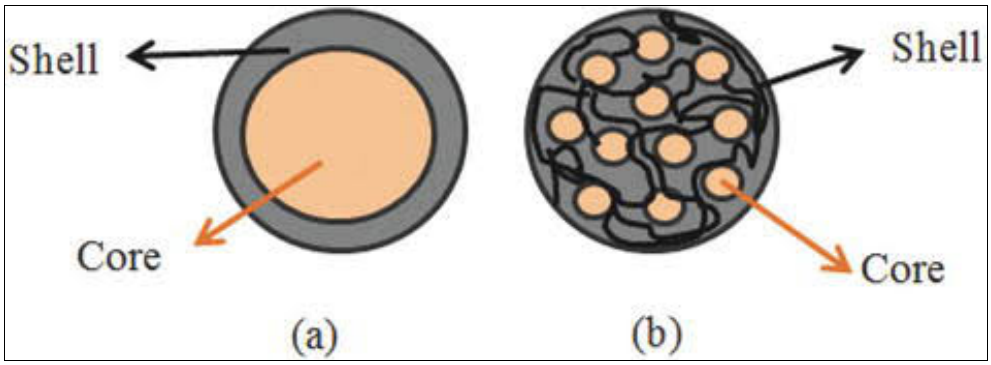


Fig 8: Types of microparticles: (a) microcapsule, with a distinct core surrounded by a shell, and (b) microsphere, with the core distributed within a polymer matrix.

Interactions between the wall material and the core can affect particle size, core volatility and release rate [39]. Two groups of mechanisms govern core release. In diffusion, the active compound is released gradually by passing through the coating wall without damaging its physical integrity. In triggered release, a change in pH, mechanical stress, temperature, enzymatic activity, time or osmotic force breaks down the capsule and releases the active compound immediately.

3. Encapsulation techniques

The final application of the product determines the choice of coating material and encapsulation technique. The decision takes into account the chemical and physical stability of the product, the concentration of the compounds to be encapsulated, the required particle size, the release mechanism and the manufacturing cost [40]. Several factors determine how effective an encapsulation process is. The molecular weight, volatility, polarity and chemical functionality of the core, together with the properties of the wall material and the chosen method, all influence how well the active agent is retained within the shell. The essential stages of any encapsulation process are incorporation of the bioactive component, droplet formation, solvent removal, capsule collection and drying. A range of techniques has been developed to suit different core and shell materials and to produce particles of varying size, thickness and shell permeability [41], which in turn allows the release rate of the active principle to be adjusted. The most common techniques are spray drying, solvent evaporation, extrusion, freeze drying and encapsulation with supercritical fluids, which are described below.

3.1. Spray drying

Spray drying comprises four steps: (i) preparation, (ii) homogenization, (iii) atomization of the suspension and (iv) drying of the atomized particles. The encapsulation efficiency of the process depends on first producing a stable oil-in-water emulsion or suspension and then spraying it as fine droplets into a drying chamber. The ratio of the hydrophilic and lipophilic phases, the oil-droplet size distribution, the dry-matter concentration and the emulsion viscosity must therefore be optimized beforehand. The emulsions need to be viscous enough to be pumped and sprayed, yet must not become sticky or hygroscopic after drying, so that the particles remain stable during storage [42]. The method requires both a suitable composition of the solution that contains the bioactive compounds and well-adjusted operating conditions; the relevant parameters include solids concentration, aspirator rate, atomizing-air flow, liquid feed rate and inlet-air temperature [43]. The choice of polymer and the droplet-size distribution of the emulsion are two further factors that affect particle formation.

The main advantages of spray drying are that particle formation and drying are combined in a single step, large-scale production is possible, the equipment is simple, operating costs are low and continuous operation is feasible. It yields good quantities of high-quality capsules that are small, dissolve rapidly and are highly stable [43]. Its disadvantages are the high temperatures, the rapid drying rates and the variable distribution of particle size and shape, which generally rule out the encapsulation of thermosensitive materials. Gallo *et al.* investigated how the operating parameters of spray drying influence the physical properties of powdered *Rhamnus purshiana* extract, and Roccia *et al.* assessed the effect of spray-drying conditions on the characteristics of powdered sunflower oil. Fernandes *et al.*, Goñi *et al.* and Oliveira *et al.* microencapsulated passion fruit seed oil, rosemary oil and eugenol, respectively, by spray drying, and Gallardo *et al.* [44] spray-dried linseed oil for use in functional foods.

3.2. Solvent evaporation

This technique relies on agitating an emulsion so that its internal phase evaporates. The coating material is usually dissolved in a volatile organic solvent, and the core material is then dissolved or dispersed in this solution to give a suspension, emulsion or solution. The organic phase is emulsified under stirring in a dispersion medium that contains a non-solvent for the encapsulant together with a suitable emulsifier and that is immiscible with the organic solvent. When the emulsion has stabilized, stirring is continued while the solvent diffuses through the continuous phase and evaporates, leaving solid microspheres. These are collected by centrifugation or filtration, washed and dried [45]. Patil *et al.* [46] applied the solvent-evaporation method to encapsulate clove oil in methylcellulose microcapsules.

3.3. Extrusion

Oil encapsulation by extrusion proceeds in three main steps: (i) injection, (ii) melt extrusion and (iii) centrifugal (co-)extrusion. Its main advantages for EOs are protection against oxidation, a longer shelf life and a lower rate of evaporation. On the other hand, the particles are unevenly distributed and the process is costly. Soliman *et al.* [47, 48] used extrusion to microencapsulate the essential oils of thyme (*Thymus vulgaris*), clove (*Eugenia caryophyllata*) and cinnamon (*Cinnamomum zeylanicum*).

3.4. Supercritical-fluid technology

Encapsulation with supercritical fluids was developed to reduce the drawbacks of conventional techniques and offers considerable benefits for the pharmaceutical, cosmetic and food industries. The method is inherently non-toxic, allows simple solvent removal without product deterioration, and accepts a wide variety of materials while giving controlled particle sizes and morphologies. It is generally preferred for EOs that are sensitive to air, chemicals and high temperatures. Because supercritical CO_2 is the usual medium, the technology is regarded as green. The reduced viscosity, increased diffusivity, lower surface tension, faster processing and high solubility of active compounds in supercritical CO_2 make it well suited to enclosing bioactive compounds [49, 50].

The supercritical equipment consists of a temperature-controlled water bath, a high-pressure CO_2 pump, a magnetic stirrer plate, a high-pressure stainless-steel impregnation cell and a pressure transducer. A mesh divides

the impregnation cell into two chambers: the essential oil is placed in the lower chamber and the microparticles or matrices to be impregnated in the upper one. This approach has been used to encapsulate lavender, oregano, canola and passion fruit seed oils [51]. Selection of the encapsulating material is a crucial step and should be based on the intended use, non-toxicity, bioactivity and the particle-production process [52]. Biodegradable polymers such as PLA, PLGA and PCL have been applied mainly as coatings in the medical field, particularly in drug release and tissue engineering. Inorganic materials such as silicates, clays and polyphosphates can be used to a lesser extent, and proteins can also serve as wall materials.

Cyclodextrins

CDs are simple, low-cost nanomaterials that can entrap gases, liquids and solids by forming host–guest complexes [53]. Uptake of the guest is energetically favorable, and free CDs coexist in equilibrium with CDs that contain a guest. CDs are particularly effective at capturing volatile organic compounds (VOCs) in both liquid and solid systems [54]. In doing so, they mask undesirable flavors and odors, increase bioavailability and improve the physical and chemical stability of the system. VOC/CD inclusion complexes that deliver VOCs safely and selectively to biological targets are currently of great interest as novel treatments for a range of diseases (Fig. 9).

1. Physicochemical characteristics

CDs are hollow, truncated-cone-shaped molecules of nano- to micrometer dimensions, composed of D-glucose units linked by α-1,4 glycosidic bonds [55]. The wider rim of the cone carries the secondary hydroxyl groups of carbons 2 and 3, whereas the narrow rim carries the primary hydroxyl groups of carbon 6. These hydroxyl groups make the outer surface hydrophilic [56]. The interior of the cavity, in contrast, is lined by non-polar hydrogens (on carbons 3 and 5), ether-type oxygen atoms and the carbon skeleton of the glucose units, which together create a hydrophobic pocket. Hydrophobic molecules, including VOCs, are accommodated in this cavity, and because the exterior remains hydrophilic, the inclusion complex stays water-soluble.

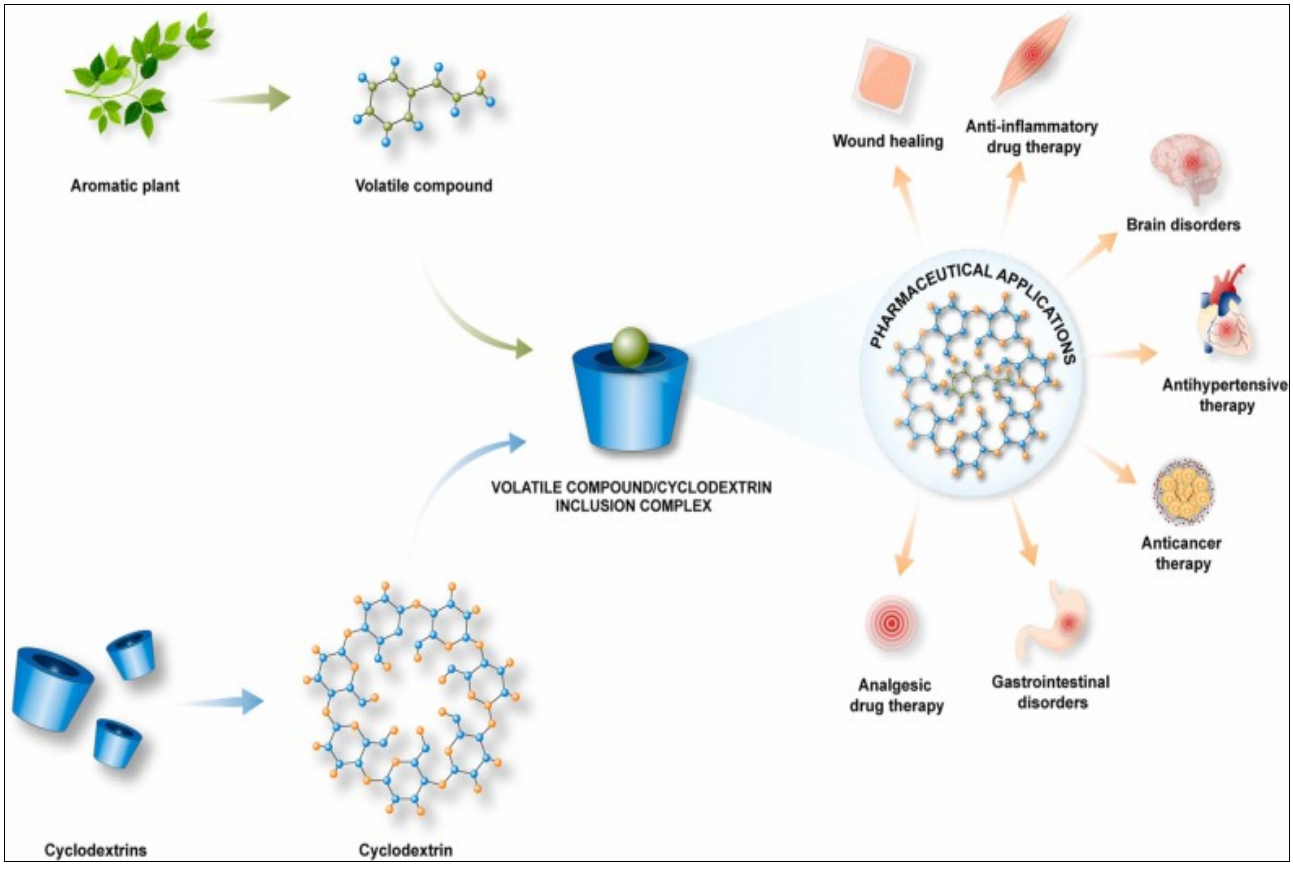


Fig 9: Pharmaceutical applications of volatile-compound/cyclodextrin (VOC/CD) inclusion complexes, including wound healing, anti-inflammatory, analgesic, anticancer and antihypertensive therapy, and treatment of brain and gastrointestinal disorders.

2. Types of cyclodextrins and their prevalence

CDs are classified according to the number of D-glucose units they contain: α-CD (six), β-CD (seven) and γ-CD (eight). β-CD is the most widely used, accounting for about 70% of the CDs produced annually, mainly because its cavity accommodates a broad range of guest molecules and it is cheaper to purify than the other types [57]. γ-CD, with its larger cavity, is selected for bulkier guests, whereas α-CD suits small molecules or long alkyl chains and is used less often. CDs with more than eight glucose units are of little use in pharmaceuticals [58].

Native CDs have shortcomings, notably limited aqueous solubility, and chemically modified derivatives have therefore been developed to improve solubility. The best-

known are 2-hydroxypropyl-β-cyclodextrin (HPβCD; Fig. 10), hydroxypropyl-γ-cyclodextrin (HP-γ-CD), randomly methylated-β-cyclodextrin (RAMEB) and sulfobutylether-β-cyclodextrin (SBE-β-CD) [59]. Together, these derivatives account for roughly 10% of annual CD production.

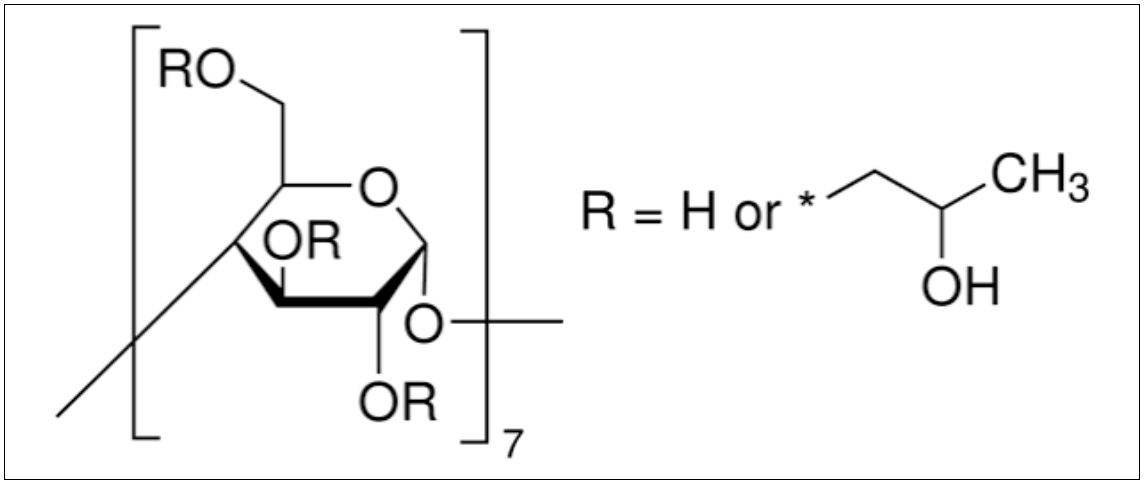


Fig 10: Chemical structure of 2-hydroxypropyl-β-cyclodextrin (HPβCD), a seven-unit β-CD derivative in which R = H or a 2-hydroxypropyl group.

Characterization of Essential Oil–HPβCD Inclusion Complexes

Formation of inclusion complexes between EOs and HPβCD can be confirmed by comparing the morphology and the spectral properties of the free components with those of the complexes. This section summarizes evidence from studies of guava leaf, yarrow and black pepper oils encapsulated in HPβCD, obtained by scanning electron microscopy (SEM), Fourier-transform infrared (FT-IR) spectroscopy and UV–visible spectrophotometry.

1. Morphological analysis

Formation of an inclusion complex clearly alters the morphology of CDs [60].

Fig. 11 shows SEM micrographs of free HPβCD, and Fig. 12 those of the HPβCD complexes of guava, black pepper and yarrow oils at the same magnification. In overall shape, the encapsulated samples closely resemble free HPβCD. This similarity suggests that free HPβCD molecules associate through hydrogen bonding in solution and therefore tend to aggregate [61]. When an inclusion complex forms, however, the morphology of the CD changes, which prevents the molecules from agglomerating into large clusters. The same behavior has been reported for carvacrol with β-CD: the inclusion complex was dispersed more evenly, whereas free β-CD formed clumps [60].

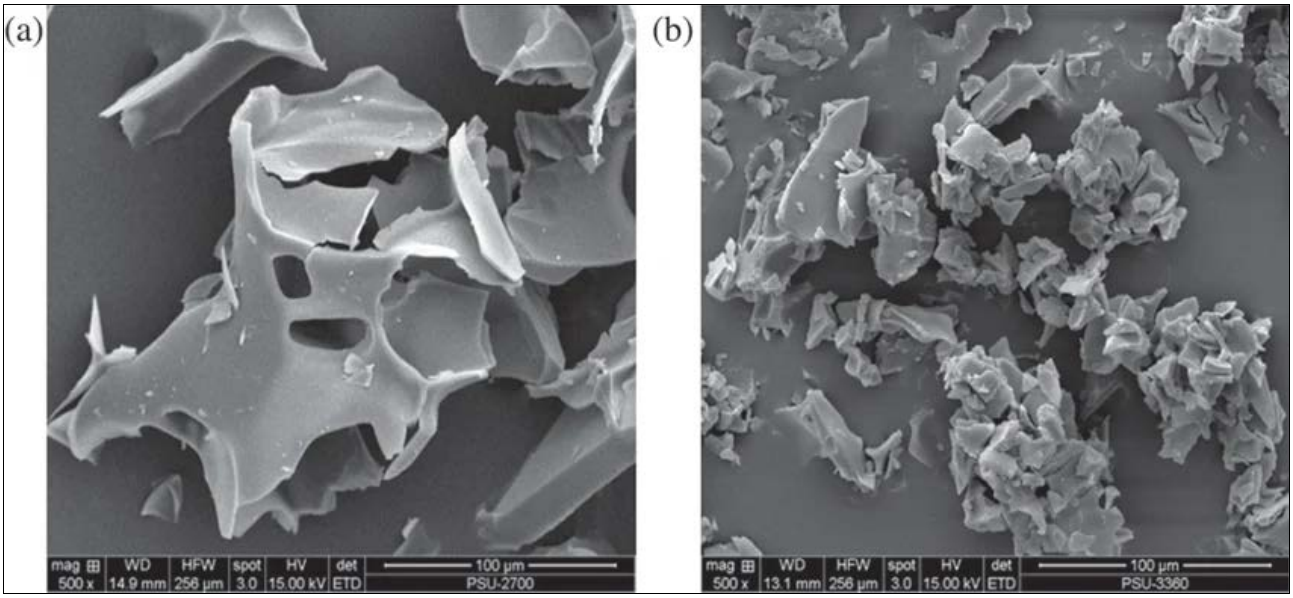


Fig 11: Scanning electron micrographs of free HPβCD at 500× magnification (a, b).

Free HPβCD particles are considerably larger than those obtained after encapsulation, in agreement with the findings of Guimaraes and co-workers for carvacrol encapsulated in β-CD.

HPβCD is known to form clusters in solution through intermolecular hydrogen bonds, but these bonds are disrupted when certain essential oils are added [62]. The clusters therefore break up and the particle size decreases.

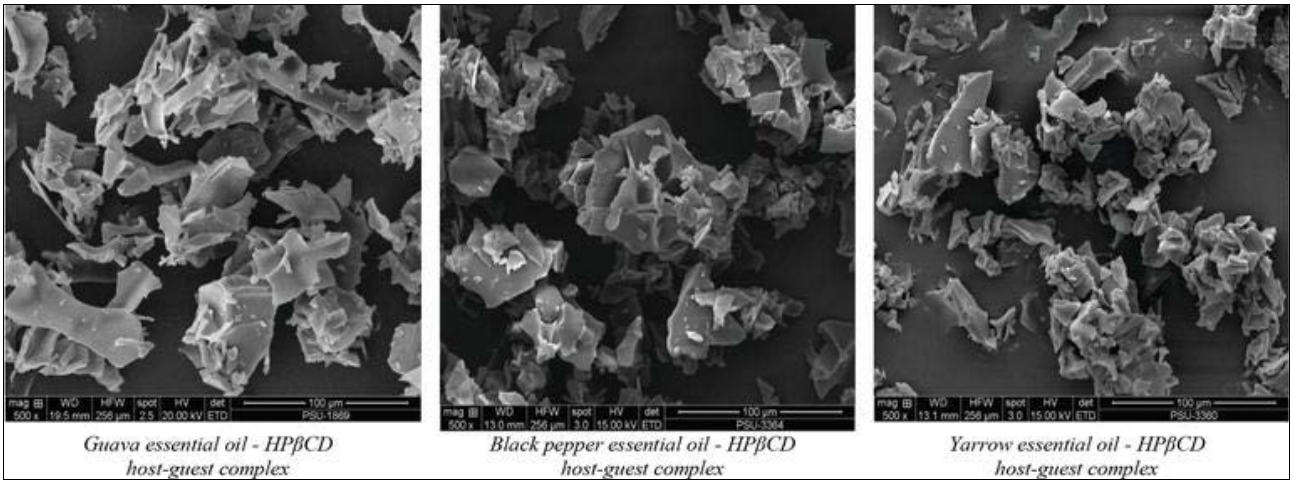


Fig 12: Scanning electron micrographs of the HPβCD inclusion complexes of guava, black pepper and yarrow essential oils at 500× magnification.

2. Fourier-transform infrared spectroscopy (FT-IR)

FT-IR spectroscopy can be used to follow changes in band position, shape and intensity. The spectrum of black pepper oil shows strong absorptions at 2954, 2923 and 2865 cm^{-1}, all attributed to C–H stretching of methylene groups [63]. Further bands occur at 1638 cm^{-1} (H–O–H bending), 1447 cm^{-1} (C–H scissoring), 1369 cm^{-1} (symmetric CH_3 deformation), 886 cm^{-1} (C–H deformation) and 789 cm^{-1} (S–C absorption). Once the black pepper oil has been encapsulated, however, its spectrum changes completely: no bands of the free oil remain, because the HPβCD bands entirely mask them. This indicates that the oil is located inside the HPβCD cavity as an inclusion complex.

The FT-IR spectrum of yarrow oil displays several distinct absorption bands. A prominent peak at 2956 cm^{-1} arises from symmetric $=CH_2$ stretching, and the peak at 2926 cm^{-1} from C–H stretching of methylene groups, while a further band at 2869 cm^{-1} indicates –CH stretching. The peak at 1626 cm^{-1} is assigned to C–C stretching in allyl groups and the band at 1652 cm^{-1} to H–O–H bending. Nearly overlapping bands at 1380 and 1366 cm^{-1} correspond to symmetric deformation of CH_3 groups, and the band at 1446 cm^{-1} to C–H scissoring. Bands attributed to P–O bonds appear at 1240 and 1103 cm^{-1}, C–O–C stretching gives a peak at 1022 cm^{-1}, and C–S–C stretching a prominent peak at 916 cm^{-1}. Finally, the bands at 875 and 865 cm^{-1} result from C–H bending in aromatic rings. HPβCD itself shows clear bands at 3406 cm^{-1} (O–H stretching), 2970 cm^{-1} (symmetric $=CH_2$ stretching), 2930 cm^{-1} (C–H stretching), 1646 cm^{-1} (H–O–H bending), 1157 cm^{-1} (asymmetric C–O–C stretching) and 1083 and 1033 cm^{-1} (symmetric C–O–C stretching) [64]. The FT-IR spectrum of the inclusion complex is almost identical to that of HPβCD, with no features of yarrow oil. HPβCD masks essentially all of the oil's absorption bands in the complex, which indicates that the yarrow oil is enclosed in the HPβCD cavity and that an inclusion complex has formed.

In the FT-IR spectrum of guava leaf oil, the visible bands are C–H stretching of methylene groups at 2921 cm^{-1}, H–O–H bending at 1642 cm^{-1}, C–H scissoring at 1447 cm^{-1} and symmetric CH_3 deformation at 1376 cm^{-1} (which appears as a doublet), together with S–C absorption at 789 cm^{-1} and C–H deformation at 886 cm^{-1}. The spectrum of the encapsulated guava leaf oil is markedly different: none of the features of the free oil are evident, since the intense, broad HPβCD band masks the oil's absorptions. Notably, the HPβCD band at 608 cm^{-1} disappears in the encapsulated sample. This change most likely reflects an interaction between guava leaf oil and HPβCD and thus the formation of an inclusion complex.

3. UV–visible spectrophotometry

The bioactive substances in EOs absorb UV or visible light at characteristic wavelengths, and formation of a CD host–guest complex alters the UV–Vis spectrum. In the absence of complexation, the spectrum of the guest simply follows that of the CD mixture. UV–Vis spectrophotometry can therefore be used to verify whether HPβCD forms inclusion complexes with the three essential oils. Guava leaf oil and limonene provide a useful example: comparison of their UV absorption spectra with those of their inclusion complexes reveals clear differences. Guava leaf oil absorbs most strongly at 214.5 nm, mainly because of limonene. A further peak near 205 nm originates from β-caryophyllene or pinene, and the peak at 275 nm from 1,8-cineole.

Physical mixtures of HPβCD with guava leaf oil or limonene gave spectra identical to those of the oil or limonene alone; before complexation, the absorption was the same as that of the original compounds. Once the active compounds were genuinely enclosed in the HPβCD cavity, however, their characteristic absorption peaks disappeared from the spectra of the inclusion complexes. To recover the encapsulated compounds, the complexes were dissolved in 95% acetonitrile, which released the active compounds while the HPβCD remained as a separate phase. After centrifugation, the HPβCD could be separated from the limonene or guava leaf oil solution [65]. The solution was diluted 100-fold with acetonitrile before its absorbance was measured with a UV spectrophotometer.

In the compounds recovered from the complexes, the absorption peaks of guava leaf oil reappear: besides limonene, clear peaks at 205 and 275 nm are seen, which are assigned to β-caryophyllene and 1,8-cineole. The main active compounds of guava leaf oil were therefore incorporated into the HPβCD complex. For yarrow oil, the UV spectrum shows peaks in the 270–275 nm range,

indicating carvacrol, 1,8-cineole, thymol and camphor, and a smaller band at 243 nm attributable to linalool. Physical mixtures of HPβCD with yarrow oil or with a pure compound such as carvacrol give spectra consistent with those of the oil or of carvacrol alone. Once the active compounds of yarrow oil, or carvacrol alone, are entrapped in HPβCD, however, their characteristic absorption peaks vanish, which demonstrates that they are firmly encapsulated.

When the compounds were released from the yarrow-oil complex, peaks at about 270–275 nm again showed the presence of carvacrol together with 1,8-cineole, thymol, camphor and linalool. The components of yarrow oil were thus entrapped in HPβCD, in agreement with earlier work. The encapsulation efficiency of yarrow oil was, however, much lower than that of its pure constituents. During inclusion, the main active compounds probably compete with the other components of the essential oil, which reduces efficiency. For black pepper oil, the absorption spectrum shows distinct peaks at 200, 205 and 214.5 nm, corresponding to δ-3-carene, β-caryophyllene and limonene [66]; the largest peak, at 205 nm, directly indicates β-caryophyllene.

The UV spectra of HPβCD mixed with pure β-caryophyllene or with black pepper oil match those of the individual substances. These absorption peaks disappear from the spectrum of the inclusion complex once the active compounds of black pepper oil, or pure β-caryophyllene, are enclosed in the HPβCD cavity, and they reappear when the compounds are extracted from the complex: the compounds isolated from the black pepper complex absorb at 205 and 214.5 nm, corresponding to β-caryophyllene and limonene. The UV data therefore show that HPβCD entraps the chemical constituents of black pepper oil. As with yarrow oil, however, the encapsulation efficiency for the active components of black pepper oil is considerably lower than that obtained with pure β-caryophyllene, most likely because the principal components must compete with the other constituents during complex formation.

Overall, the successful encapsulation of EOs in HPβCD shows that formation of the host–guest complex gives the oils physicochemical properties that differ from those of the free oils.

Cyclodextrin-Based Nanoencapsulation for Food Applications

Cyclodextrin-based systems are among the most intensively studied nanoencapsulation approaches for food, particularly for the delivery of hydrophobic and volatile bioactive compounds such as essential oils [67]. CDs are important in food technology because they form stable inclusion complexes, that is, non-covalent host–guest associations between the CD molecule and the encapsulated compound. Beyond simple inclusion complexes, recent research has explored more sophisticated CD-based nano-formats, including nanosponges, polymeric networks and composites with biopolymers, which offer higher loading capacity and regulated release [68]. Table 2 compares different nanoencapsulation techniques and CD-based systems for essential oils in food applications.

Table 2: Comparison of nanoencapsulation systems for essential oils in food applications: cyclodextrin-based systems versus nanoemulsions, biopolymer nanoparticles and liposomes.

Encapsulation system	Basis of formation	Loading capacity	Release characteristics	Sensory effects	Regulatory status in food	Key advantages
CD inclusion complexes	Host–guest inclusion in a hydrophobic cavity	Low to moderate (1:1 molar ratio typical)	Controlled; often triggered by temperature or moisture	Low; efficient taste and aroma masking	GRAS status for α-, β- and γ-CD (FDA); authorized food additives (European Food Safety Authority, EFSA)	Simple, well established; efficient masking; improved solubility
CD nanosponges	Porous network of cross-linked CD polymers	High; multiple inclusion sites plus adsorption	Long-term, sustained release (days to weeks)	Low to moderate; controlled release reduces sensory burst	Novel; some derivatives may need case-by-case authorization	Extended release, high loading and versatility
Nanoemulsions	Surfactant-stabilized oil-in-water dispersions	High; depends on the volume of the oil phase	Rapid initial release followed by a prolonged phase	Moderate; high EO content at the interface may intensify aroma, and surfactants may affect taste	Depends on the surfactant; many food-grade emulsifiers (e.g. Tweens) are available	Improved bioavailability, high loading and ease of production
Biopolymer nanoparticles	Self-assembly or cross-linking of proteins and polysaccharides	Moderate; depends on the structure of the polymer network	Controlled by swelling or polymer degradation	Low to moderate; biopolymers are usually tasteless, but release can reveal the EO flavor	Generally GRAS (chitosan, alginate, whey protein, starch)	Edible and biodegradable; can provide additional functional properties (e.g. film formation)
Liposomes	Phospholipid bilayer vesicles incorporating hydrophilic and hydrophobic substances	Moderate (hydrophilic core plus bilayer)	Variable; can be triggered or sustained	Low to moderate; phospholipids may contribute flavor but can also mask aroma	Certain phospholipids are GRAS; assessed case by case	Biocompatible; can carry both hydrophilic and hydrophobic substances

1. Cyclodextrin nanosponges

Despite these advantages, conventional inclusion complexes have a very low loading capacity because of the limited size of the CD cavity. Cyclodextrin-based nanosponges were developed to overcome this limitation. Nanosponges are three-dimensional, cross-linked polymeric networks made from CDs, with a porous structure that combines multiple inclusion sites with additional adsorption regions within the polymer matrix. This architecture allows larger amounts of bioactive compounds to be encapsulated and gives better-regulated, prolonged release [69]. From the standpoint of food technology, these properties are particularly useful for applications that require extended antibacterial or antioxidant action, such as the long-term storage of fresh or minimally processed foods.

2. Composites with food-grade biopolymers

The development of composite systems with food-grade biopolymers is another significant advance in CD-based nanoencapsulation. Such composites combine the film-forming, mechanical and barrier properties of biopolymers such as chitosan, starch, cellulose derivatives and proteins with the encapsulating capacity of CDs. Chitosan-based composites are especially interesting because chitosan is inherently antibacterial and forms cohesive, flexible films. Incorporating CD inclusion complexes or essential-oil-loaded nanosponges into chitosan matrices frequently produces a synergistic antibacterial effect, which results from both contact-active inhibition and controlled release of volatile compounds [68]. Cellulose- and starch-based CD composites offer inexpensive and biodegradable alternatives to single-use food-packaging materials, since the CDs help to retain volatile bioactive compounds during film processing and storage and prevent their rapid evaporation. Protein-based films, such as those made from whey protein, offer good mechanical strength and oxygen-barrier properties, while the CDs regulate the release of the encapsulated compounds on moist food surfaces [69, 70]

Conclusions and Future Perspectives

Essential oils have attracted growing attention as potent, bio-sourced and environmentally acceptable alternatives to synthetic pesticides and sanitizers, owing to their distinctive physicochemical, biological and functional properties. Their direct use is nevertheless limited by volatility, poor water solubility and sensitivity to light, heat and oxygen. The extraction route also matters: conventional distillation and solvent methods are simple and well established but can be slow, solvent-intensive or capable of altering oil composition, whereas microwave-assisted and supercritical-fluid extraction are greener and more efficient, at the cost of additional equipment or downstream filtration steps (Table 1).

Micro- and nanoencapsulation provide a way around these limitations. How successfully an oil is encapsulated depends on the intended application, the carrier material, the particle morphology and the preparation technique. Of the processes reviewed here, spray drying and extrusion are scalable but may expose heat-sensitive oils to high temperatures or yield unevenly distributed particles; freeze drying gives high-quality products at the expense of cost and processing time; and supercritical-fluid processing is clean but demands specialized equipment.

Cyclodextrins, and HPβCD in particular, form inclusion complexes that protect essential-oil constituents, reduce their volatility and mask their odor and taste. The SEM, FT-IR and UV–Vis evidence for guava leaf, yarrow and black pepper oils consistently indicates that the oils are located within the HPβCD cavity: the complexes are less aggregated than free HPβCD, the bands of the oils vanish from the FT-IR spectra, and the characteristic UV peaks reappear only after the actives are released. Encapsulation efficiency was, however, lower for whole oils than for isolated constituents such as carvacrol or β-caryophyllene, presumably because the components compete for the cavity. For food applications, inclusion complexes are simple, scalable and compatible with food-grade regulation, but their loading capacity is limited. Nanosponges and CD–biopolymer composites extend loading capacity and release duration and are particularly well suited to active packaging and long-term preservation (Table 2).

Encapsulation also raises safety, environmental and regulatory questions concerning its effects on human health and the environment. Because no dedicated international regulation has been introduced, the safety aspects of these systems remain insufficiently understood and require further study. Future research should therefore concentrate on: (i) safety assessment and risk evaluation of micro- and nanoencapsulated essential oils in different applications; (ii) improvement of existing encapsulation methods and their industrial-scale production; (iii) optimization of the factors that control the release of bioactive compounds, in order to enhance their activity; and (iv) exploration of further direct applications of essential-oil particles, particularly in biological and food systems.

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