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Mint (*Mentha* spp.) essential oil extraction: from conventional to emerging technologies

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Abstract Mint essential oil (MEO) is an economically appreciated natural product with significant importance in the cosmetics, pharmaceutics, foods, and healthcare products due to its biological activities. Extraction is a critical step in the production of essential oils from aromatic plants, such as mint. Conventional extraction approaches, such as steam distillation and solvent extraction are commonly used for the extraction of MEO. However, they are energy and time-consuming processes with relatively low extraction yields. Consequently, emerging techniques, such as microwave-assisted, ohmic-assisted, ultrasound-assisted, pulsed electric field, and super and sub-critical fluid extraction methods have been developed to overcome these shortcomings. This review aims to investigate the influence of different extraction methods and conditions on the extraction yield, composition, physicochemical properties, and bioactivity of MEO. Overall, selection of appropriate extraction method and conditions is a crucial step in the extraction of MEO that can have significant effects on the quality and quantity of the isolated EOs. Inappropriate extraction of MEO may reduce the extraction yield, prolong the extraction duration, and increase the energy consumption. Additionally, it may deteriorate the physicochemical properties and bioactivity of the extracted MEO.

Keywords Mint essential oil · Traditional Persian medicine · Medicinal plants · Extraction · Distillation · Green extraction technologies · Ultrasound-assisted extraction · Microwave-assisted extraction

Introduction

The biologically active compounds extracted from the roots and aerial parts of medicinal plants contain primary and secondary metabolites. Plant secondary metabolites, also known as phytochemicals, include a wide range of bioactive compounds that play an

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essential role in protecting plants against biotic and abiotic stresses (Soleimani et al. 2022). The widespread use of phytochemicals in traditional cultures for health promotion and disease treatment has led to their incorporation into contemporary products. These plant-derived compounds are often preferred over synthetic chemicals. The World Health Organization (WHO) has estimated nearly 80% of the world's population relies on herbal medicines for health purposes (Sadowska et al. 2016). Essential oils (EOs) are complex mixtures of small organic molecules, including terpenic (especially mono- and sesquiterpenoids and mono- and sesquiterpenes), aromatic, and aliphatic components extracted from roots, stems, leaves, flowers, fruits, or seeds of many herbal plants (Ebadollahi et al. 2020). They are widely used in the food, cosmetic, perfumery, and pharmaceutical industries due to their flavor and fragrance properties, as well as their anti-inflammatory, antimicrobial, antioxidant benefits, and their demand is increasing day by day all over the world (Zhang et al. 2022).

Mint (*Mentha* spp.) is one of the most common medicinal plant worldwide, which belongs to the Lamiaceae family with 25–30 species and about 10 hybrids; and the species and hybrids of *Mentha* genus are produced from both inter and intra-specific crosses, which make mint cultivars highly heterozygous. For instance, peppermint (*M. piperita*) is a first-generation hybrid obtained from interspecific crossing of spearmint (*M. spicata*) and water mint (*M. aquatica*). In addition, *M. arvensis*, *M. rotundifolia*, *M. longifolia*, *M. pulegium*, *M. citrata*, *M. canadensis*., *M. suaveolens*, and *M. viridis* are other common species/hybrids of *Mentha* genus (Moetamedipoor et al. 2021). Traditionally, leaves, stems, and flowers of mint have been used in herbal teas or as an additive in foods to provide aroma and flavor. Today, however, there is an increasing demand in mint cultivation because of the commercial profit of its EO, which is among the 10 most used commercial EOs in the world (Chrysargyris et al. 2017).

Several mint species/hybrids are well-known for their high EO yields, especially *M. piperita*, *M. arvensis*, and *M. spicata*, which is synthesized and stored in glandular trichomes on the adaxial surface of their leaves. Mint essential oil (MEO) is known as a multi-purpose ingredient in various industries and its market is projected to experience an annual growth rate of 3 to 5% (Soleimani et al. 2022). It contains diverse monoterpenes (the C10 class of isoprenoids) in its structure, mainly C2-oxygenated monoterpenes (e.g., carvone), C3-oxygenated monoterpenes (e.g., menthol), and acyclic monoterpenes (e.g., linalool) (Soleimani et al. 2022). It has been used in many medications to treat oral mucositis, enteritis, gastritis, irritable bowel syndrome (IBS), and respiratory tract disorders since ancient times. In addition, it can be added to the formulation of various foods, drugs, healthcare items, and agro-biological products as a cooling, flavor enhancing, antioxidant, antimicrobial, and insecticidal agent (Hedayati et al. 2023). However, these multiple biological properties and health-related behaviors of MEO are highly influenced by various endogenous and exogenous factors. The plant organ, genotype (variation within different or same species), environment (e.g., climatic, topographic variables, and soil properties), plant phenology (developmental stage at the time of sampling), and extraction method are the most important factors that can affect the levels of *Mentha* EO (Salehi et al. 2018). Therefore, it is necessary to optimize each of the factors for the specific use of MEO in various food and non-food industries.

MEO can be isolated by various extraction techniques from several parts of mint plants such as stems, leaves, and flowers. These methods are generally divided into conventional (e.g., steam distillation (SD), solvent extraction (SE), hydro-diffusion, and hydro-distillation) and emerging (e.g., ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), ohmic-assisted extraction (OAE), pulsed electric field-assisted extraction (PEFAE), supercritical fluid extraction (SFE), and subcritical water extraction (SWE) extraction techniques. SD is the most common technique for the extraction of MEO. In spite of the simplicity of this method, it has a great downside, which is very time-consuming and requires a huge amount of energy. Most importantly, the high temperature and moisture during the process may change the flavor and reduce the overall quality of the MEO (Batool et al. 2018). These disadvantages have attracted the attention of many researchers and stimulated the intensification, improvement, and optimization of "emerging" or "green" extraction techniques with higher efficiency, lower cost, and shorter extraction time. Therefore, the main aim of this study is to provide a comprehensive review of various conventional and emerging methods for the extraction of MEO and evaluate their advantages and disadvantages in order to meet the manufacturers and consumers' demands.

An overview of MEO

Botanical features

The genus *Mentha* belongs to the Lamiaceae (Labiatae) family, which consists of approximately 30 species and 10 hybrids. It is believed that this genus originated from the Mediterranean basin, and from there, it spread to other parts of the world. Currently, the genus *Mentha* can be found in temperate and subtemperate regions across North America, Africa, Europe, Asia, and Australia (Moetamedipoor et al. 2021). The taxonomists have assigned various names to the mint plants over the past two centuries, indicating a significant morphological diversity. Also, it should be mentioned that the systematics of the genus *Mentha* are complex and still not fully understood, primarily due to variations in chromosome number, cytomixis, morphological polymorphism, frequent hybridization between different species, and propagation of colonial mutants, as well as the occurrence of polyploidy, aneuploidy, and nothomorphs (Gholamipourfard et al. 2021; Salehi et al. 2018).

Mint species are fragrant and herbaceous perennial plants that are known for their wide-spreading underground and aboveground stolons. The stems of these plants are branched and square-shaped in cross-sections. The leaves are aromatic and arranged in opposite pairs, with either serrated or smooth edges, and are lanceolate to oblong-elliptical in shape. Another distinguishing feature of mint species is the presence of purple or white, bilaterally zygomorphic flowers, which are densely clustered in structures called verticillasters at the nodes of the stem. In addition, the fruits of these plants are small, dry and schizocarpic, containing 1 to 4 seeds (Yousefian et al. 2023).

Chemical composition

Plants produce a variety of chemical compounds, some of which are always present, while others are secreted in response to environmental stresses, such as climate change, injury, infection, and predators. EOs are a complex mixture of volatile compounds derived from a small portion of the plant's overall composition (less than 5% of the plant's dry matter), consisting mainly of terpene hydrocarbons (isoprenes) and terpenoids (isoprenoids). The main compounds of terpenes are monoterpenes (comprising over 80% of the EO's composition and containing 10 carbon atoms, C10) and sesquiterpenes (containing 15 carbon atoms, C15). Whereas, terpenoids are oxygenated derivatives of terpene hydrocarbons, such as alcohols, ethers, esters, ketones, aldehydes, acids, and phenols (Angane et al. 2022).

The chemical compounds in MEO are susceptible to changes depending on the variety, climate, soil composition, agronomic conditions, and harvest time, as well as drying and extraction methods. As shown in Fig. 1, MEO contains a wide range of functional groups that are responsible for its biological characteristics, including alcohols (e.g., menthol, linalool, neomenthol, geraniol, 3-octanol, elemol, viridoflorol, *cis/trans*-sabinene hydrate, terpinen-4-ol, and α-terpineol), ethers (e.g., menthofuran, 1,8-cineole, piperitenone oxide, caryophyllene oxide, and piperitone oxide), esters (e.g., menthyl acetate, neomenthyl acetate, neoisomenthyl acetate, 3-octyl acetate, decyl acetate, dihydrocarvyl acetate, and α-terpinyl acetate), ketones (e.g., carvone, menthone, isomenthone, piperitenone, pulegone, and *trans-/cis*-dihydrocarvone, 3-octanone), and hydrocarbons (e.g., limonene, *β*-caryophyllene, and germacrene D) (Gholamipourfard et al. 2021; Salehi et al. 2018). Different species of *Mentha* can be categorized into three groups based on their biosynthetic pathway and geographical conditions: those that produce EOs rich in menthol, carvone, or pulegone/piperitone. For instance, the dementholized EO obtained through gas chromatography-mass spectrometry (GC-MS) analysis reveals the presence of ten key components in peppermint (*M. piperita* L.) EO, which includes menthol (30.0 to 55.0%), menthone (14.0 to 32.0%), menthylacetate (2.8 to 10.0%), isomenthone (1.5 to 10.0%), menthofuran (1.0 to 9.0%), limonene (1.0 to 5.0%), 1,8-cineole (3.5 to 4.0%), pulegone (max. 4.0%), isopulegol (max. 0.2%), and carvone (max. 1.0%) On the other hand, the cornmint (*M. arvensis* L.) EO consists of nine primary compounds, which are menthol (30 to 50%), menthone (17 to 35%), isomenthone (5.0 to

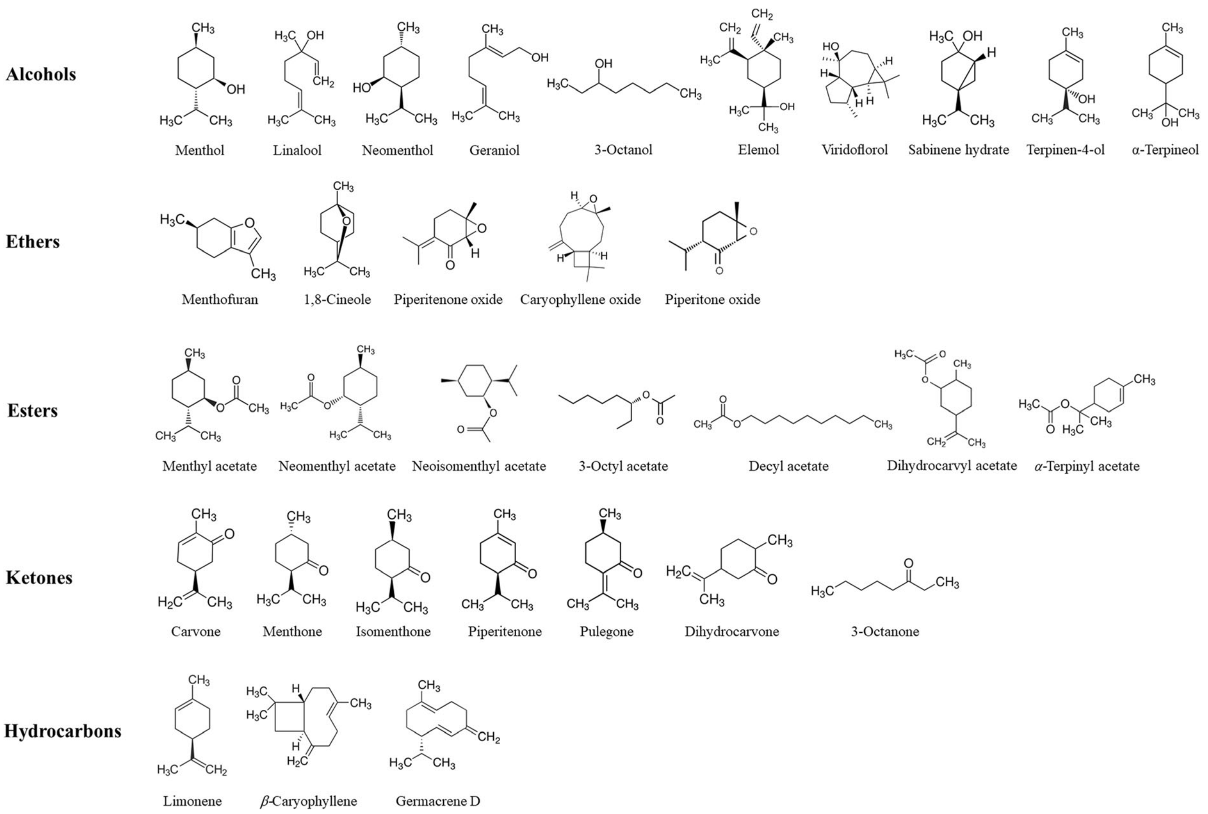

Fig. 1 Common functional groups in mint essential oil (MEO)

13.0%), limonene (1.5 to 7.0%), menthyl acetate (1.5 to 7.0%), isopulegol (1 to 3%), pulegone (max. 2.0%), carvone (max. 2.0%), and 1,8-cineole (max. 1.5%) (Kumar et al. 2011; Soleimani et al. 2022).

Furthermore, a wide range of other chemical compounds, such as phenolic acids, flavonoids, tannins, coumarins, stilbenes, and lignans, are also present in mint tissues. In this regard, caffeic acid, chlorogenic acid, rosmarinic acid, salvianolic acid, luteolin-7-O-glucoside, eriocitrin, luteolin-7-O-rutinoside, and hesperidin are recognized as the primary non-volatile components in mint species (Yousefian et al. 2023).

Biological properties

MEO has shown numerous biological and health-related effects in several aspects, such as antibacterial, antifungal, antiviral, antioxidant, anti-inflammatory, anticancer, and so on (Fig. 2). Considering the wide variety of chemical compounds found in MEO, it is possible that the biological activities are not attributed to a specific mechanism of action. Also, the exogenous factors may have a significant effect on the biological activity of MEO. For instance, the antimicrobial efficacy of MEO can be affected by its chemical compositions, environmental conditions, and the type of pathogenic and spoilage microorganisms (e.g., bacteria, fungi, or viruses) (Reyes-Jurado et al. 2015). The growth of both Gram-positive and Gram-negative bacteria, such as *Bacillus subtilis*, *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Escherichia coli*, *Serratia marcescens*, *Klebsiella pneumoniae*, and *Proteus vulgaris aureus* can be inhibited by MEO (Eftekhari et al. 2021). The antifungal and antiviral activities of MEO are also reported in several studies against various fungal diseases and human immunodeficiency virus (HIV) infections (Hu et al. 2019; Nadjib 2020). Generally, the antimicrobial properties of MEO are due to the presence of bioactive compounds (e.g., menthol, menthone, isomenthone, rosmarinic acid, caffeic acid, and luteolin), which disrupt the microbial membrane, leading to subsequent damage to the cell organelles (Saqib et al. 2022).

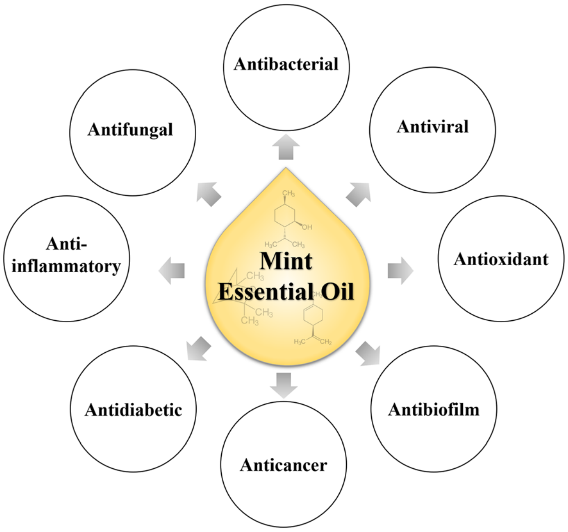

Fig. 2 Some of the biological activities of mint essential oil (MEO)

These results indicate the potential application of MEO as a natural preservative in the food industry.

There has been a growing interest in the utilization of EOs as a natural antioxidant in recent years. It is widely recognized that reactive oxygen species (ROS) cause damage upon cellular macromolecules (e.g., proteins, lipids, and nucleic acids) and are implicated in the development of numerous human diseases (e.g., cancer, cardiovascular diseases, metabolic disorders, diabetes, etc.). In various pathological circumstances, the imbalance in oxidation-reduction (redox) potential fails to eliminate excessive quantities of ROS. Consequently, oxidant species like superoxide (O_2^-) and hydrogen peroxide (H_2O_2) are generated as a result of the pathogen phagocytosis by these cells, serving as a defense mechanism against harmful insults. To counteract this response, antioxidant pathways are activated to protect against ROS-induced cellular damage (Pizzino et al. 2017). In previous studies, MEO has shown significant in vitro and in vivo antioxidant activities. For instance, Wu et al. (2019) evaluated the antioxidant activity of EOs obtained from three mint species, including native spearmint (*M. spicata*), Scotch spearmint (*M. gracilis*), and peppermint (*M. piperita*). All of these mint-derived EOs showed significant antioxidant activity in chemical-based assays. However, *M. piperita* EO showed higher antioxidant capacity compared to *M. spicata* and *M. gracilis* EOs. Furthermore, the maximal cellular antioxidant activity (CAA) was observed at a much lower dose for *M. piperita* EO (5 µg/mL) than for native or Scotch mint EOs (100 µg/mL). Alsaraf et al. (2021), Aimad et al. (2021), and Almeida et al. (2022) have also reported the effective antioxidant activity of various MEOs cultivated in Oman, Morocco, and Brazil, respectively.

In recent studies, the anticarcinogenic effects of MEO against prostate, colon, bladder, cervical, leukemic, skin, liver, and lung cancers have been comprehensively reviewed by Zhao et al. (2023) and Kazemi et al. (2023). In addition, the anti-inflammatory (Hejna et al. 2021), antidiabetic (Gülçin et al. 2022), and antibiofilm (Kang et al. 2019) activities of MEO have been also investigated. Moreover, previous studies showed the insecticidal properties of MEO against several insects, such as *Tribolium castaneum*, *Toxoptera aurantii*, *Mayetiola destructor*, *Sitophilus oryzae*, *Sitophilus zeamais*, *Sitophilus granarius*, and *Rhyzopertha dominica* (Eftekhari et al. 2021). MEO is also known for its analgesic, antispasmodic, antipruritic, antidepressant, and anti-anxiety effects, which highlights the potential application of MEO as a natural therapeutic agent for various health conditions (Kazemi et al. 2023).

Extraction of MEO

The extraction methods and conditions have significant effects on the production yield, bioactivity, and physicochemical properties of MEO. Inappropriate extraction may destroy the phytochemicals and result in loss of pharmacological and flavor compounds, and physical changes to MEO. Generally, the extraction methods are divided into two groups: conventional or classical methods and emerging or green methods, each method has its advantages and disadvantages which are discussed in this section.

Conventional methods

The conventional methods for the extraction of EOs include hydro-diffusion, solvent extraction (SE), hydro-distillation, and steam distillation (SD). Hydro-diffusion is a classical extraction method that is only used for dried plants, which may be damaged at boiling temperature. In this method, steam is applied from the top of a generator to the container that holds the dried plants. Hydro-diffusion is performed under vacuum or at low pressure and the temperature of steam is below 100 °C (Kant and Kumar 2022). On the other hand, SE is used to extract EOs from delicate plant materials, such as flowers, which are sensitive and cannot be subjected to heat or steam. Different solvents, such as ethanol, methanol, hexane, acetone, and petroleum ether are added to the plant materials and mildly heated, subsequently the mixture is filtered and the solvent is evaporated (Tongnuanchan and Benjakul 2014). The filtrate is a mixture of EO, wax, resin, and fragrance; to purify the EOs, alcohol is added to the filtrate and then distilled at low temperatures. Alcohol absorbs fragrance and then evaporated whereas the EOs remain in the flask residue. SE is more complicated, expensive, and time-consuming, than other EOs extraction methods, therefore it is not widely used for the extraction of EOs (Li et al. 2009). In addition, hydro-distillation, attributed to the discovery by Avicenna, stands as an ancient and most straightforward extraction methods (Khan et al. 2023). Hydro-distillation is a commonly used method to extract EOs from plants. It can be carried out using two main methods: The Clevenger method and simple SD. In the Clevenger method, a mixture of the plant material and water is heated and causes the EOs to evaporate. The steam and EOs are then cooled and condensed back into a liquid. The EOs are then separated from the water using a separating funnel. In simple SD, steam is passed through a bed of the plant material and carries the EOs. The steam and EOs are then cooled and condensed back into a liquid. The EOs are then separated from the water using a separating funnel. Both methods produce two layers: a water layer and an oil (EOs) layer (Ashraf et al. 2020). A typical SD system consists of essential components, such as a steam generator, extraction unit, connecting pipes, condenser, and oil separator (Fig. 3). The heater acts as the power source, heating water within the boiler to produce the necessary steam for extraction. The cylindrical extraction unit accommodates batches of plants with a mesh positioned at a specific height above its base. It features two openings: one at the bottom for delivering boiler-generated steam and another at the top for conveying steam laden with extracted volatile oil to the condenser. The condenser, comprising two tubes with a continuous water flow system, efficiently cools and condenses the steam. Connected to the extraction unit, it receives both steam and oil. The collected volatile oil is funneled and gathered. Approximately, 93% of EOs can be extracted by SD and the remaining 7% can be isolated by other approaches (Babu and Kaul 2005; Masango 2005).

In 2020, Radwan et al. (2020) conducted a study to establish a straightforward SD system for peppermint volatile oil extraction. Their experiments, involving varying boiler inlet water flow rates and different batch sizes of peppermint plants, revealed that with a 1.5 l/h boiler inlet water flow rate and a 700 g batch size, the system achieved optimal conditions, yielding maximum productivity, an 88.6% extraction efficiency, and an energy requirement of 0.48 kWh/ml EO. In another study, the chemical composition of *Mentha aquatica* L. EOs was investigated by Andro et al. (2013) at various stages of its ontogenetic cycle. The primary objective of their research was to scrutinize the chemical constituents present in these EOs. Aerial parts of the *Mentha aquatica* L. plant were systematically collected during three distinct phases: the vegetative, flowering, and senescence. The EOs were meticulously extracted using the hydro-distillation method, employing a Neo-Clevenger device. Subsequently, the acquired EO samples underwent a rigorous analysis through Agilent GC-MS. The quality of the EOs exhibited variations contingent on the specific timing of the investigations. In total, a notable forty-one different substances were successfully identified, with a noteworthy twenty of them appearing consistently across all samples gathered during the three distinct phenophases. Among the primary substances identified were menthofuran, limonene, trans-β-ocimene, ledol, and β-caryophyllene. The extraction process for obtaining these EOs involved hydro-distillation employing water vapors and a Neo-Clevenger type device. The method entailed the placement of crushed plant material into the device's flask, followed by the addition of 500 ml of water, which was then adapted for SD. The distillation procedure unfolded over a span of three to four hours, maintaining a moderate temperature throughout.

Furthermore, Beigi et al. (2018) investigated the composition and amount of EO extracted by hydro-distillation from the leaves of peppermint (*Mentha piperita* L.), employing various drying methods. The research aimed to provide a comprehensive analysis of the EO from peppermint leaves under different drying

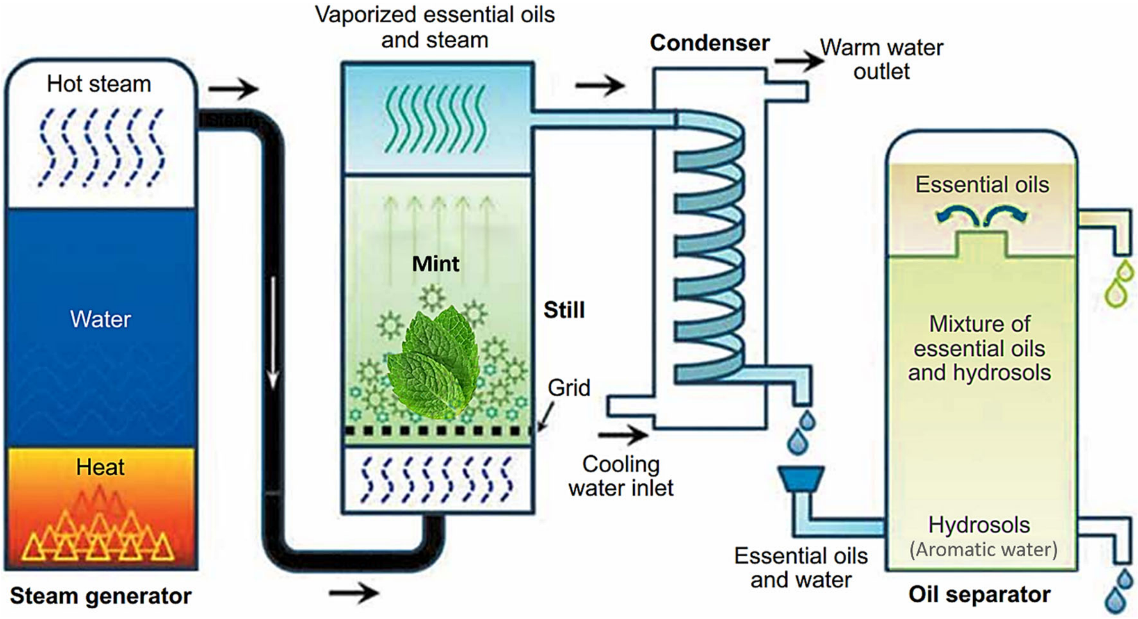

Fig. 3 Schematic diagram of steam distillation (SD) system for mint essential oil (MEO) extraction; adapted from Machado et al. (2021)

conditions. Thin-layer drying experiments were conducted using methods such as shade drying, hot air drying, and microwave drying. EOs were subsequently extracted from both fresh and dried samples using hydro-distillation, with GC-MS analysis revealing valuable insights. The study found that the highest oil yield (22.2 g/kg dry matter) was obtained from leaves dried using hot air at 50 °C, while the lowest yield (1.3 g/kg dry matter) occurred with microwave drying at its highest power. Drying temperature inversely correlated with EO content. Major compounds in the EO included menthol, menthone, menthofuran, 1,8-cineole, and menthyl acetate, with their concentrations varying significantly across different drying methods. For oil extraction, both dried and fresh leaves underwent hydro-distillation in a Clevenger-type apparatus for 3 h. In another study, EO components of Egyptian *Mentha pulegium* L. were extracted through hydro-distillation and analyzed via GC-MS. Pulegone was the predominant compound, classifying the oil as a pulegone chemotype (43.5% pulegone content). The oil displayed antioxidant properties in both hexanal/hexanoic acid and DPPH assays, particularly in dichloromethane extracts. To obtain the oil, dried leaves were subjected to SD for 4 h, followed by 6-hour extraction with dichloromethane. Non-volatile compounds were isolated by successive extracting using methanol and dichloromethane. The study highlights *Mentha pulegium* L. as a potential source for cost-effective flavorings and antioxidants, valuable for preventing oxidation in various food products (El-Ghorab 2006).

To determine the duration and best timing for extraction of EO by hydro-distillation from *Mentha suaveolens* L., Garzoli et al. (2015) used a multidisciplinary approach. Their study involved a thorough examination of EO extraction, particularly focusing on the influence of harvesting time and extraction duration. The analysis, performed via GC-MS, identified piperitenone oxide as a key component contributing to the oil's antifungal activity. Notably, EOs with lower piperitenone oxide content still exhibited antifungal properties, possibly due to compounds like para-cymenene. Importantly, a bacterial reverse mutation assay indicated the safety of these EOs. The findings provided specific recommendations: for enhanced antifungal activity, limit hydro-distillation to a maximum of 3 h, regardless of the harvest time, while for maximum oil yield, harvest in August. July was identified as the period with the highest piperitenone oxide content. This multidisciplinary approach sheds light on optimizing EO production from *Mentha* *suaveolens* L., considering both chemical composition and biological activity. Another study focused on predicting the EO yield extracted by hydro-distillation from *Mentha arvensis* L., also known as corn mint, which is extensively used in flavor, fragrance, and pharmaceutical industries. The study developed a predictive model based on solid diffusion principles, particularly focusing on the geometry of flake particles. The model's accuracy was validated by comparing it with experimental data on oil yield over time. This experimental data was collected from both bench-scale and pilot-scale distillation setups, providing a comprehensive assessment of the model's performance. This research enhances our understanding and ability to predict EO yields during the hydro-distillation process for corn mint, benefiting various industries reliant on these EOs (Katiyar 2017).

Emerging technologies

Conventional extraction methods have several disadvantages, such as long extraction period and high energy consumption. Also, the conventionally extracted EOs undergo chemical alterations, such as oxidation, hydrolysis, and isomerization due to the application of high temperatures in these methods. Therefore, emerging extraction approaches have been developed to address the shortcomings of conventional methods. Several innovative techniques such as microwave-assisted, ohmic-assisted, ultrasound-assisted, pulsed electric field, and super and sub-critical extraction have been used for the extraction of MEO that will be discussed in this section.

Supercritical fluid extraction (SFE)

Supercritical fluid extraction (SFE) stands out as an eco-friendly and efficient method for extracting EOs from plants and herbs. Using supercritical CO_2, known for its selectivity and absence of harmful solvents, ensures high-quality extracts in moderate timeframes. This process is widely used to isolate EOs, involving pure CO_2 or a co-solvent. Fractionation is a common step to separate volatile oil compounds from other extracted substances. Parameters like pressure, temperature, and co-solvent presence impact the extraction and fractionation process. Compared to traditional methods like hydro-distillation, SFE often yields superior-quality extracts (Fornari et al. 2012).

SFE is emerging as a highly promising technique for isolating volatile compounds from natural sources, challenging traditional methods like SE, SD, and hydro-distillation. SFE's selectivity can be tailored by factors such as solvent choice, temperature, pressure, extraction times, flow rate, and modifiers. The solvent's strength in supercritical fluids correlates with its density, primarily regulated by adjusting extraction pressure. The low viscosity and high diffusibility of solutes in supercritical fluids promote efficient mass transfer during extraction. A study on *Mentha pulegium* L. EO composition, extracted using supercritical carbon dioxide, examined various parameters like pressure, temperature, extraction time, and modifier usage. Results showed that optimal SFE conditions considerably yielded around 30% menthone and 52% pulegone, while hydro-distillation favored 38% pulegone and 20% menthone. This study showed SFE's potential and efficiency in extracting volatile compounds while offering selectivity adjustments through parameter control (Aghel et al. 2004). In addition, a comparative study was conducted to assess the efficiency and quality of EO extraction from spearmint (*Mentha spicata* L.) using both SFE and SD methods. The primary goals were to optimize SFE conditions for producing high-quality MEO and to evaluate the impact of different drying methods (oven drying and freeze drying) on EO quality. Additionally, the study compared the flavor compounds in the SFE-extracted MEO with those obtained through traditional SD. The research identified the highest extraction yield under specific SFE conditions, notably at 50 °C and 350 bar. However, for superior MEO quality, the optimal SFE parameters were 30 °C and 150 bar. Moreover, the composition of the EO obtained via SFE outperformed that from SD. The study also provided insights into extraction yields and EO quality from spearmint samples collected in various regions, contributing valuable information on EO extraction efficiency and quality, particularly for spearmint (Al-Marzouqi et al. 2007). In a recent study, MEO was obtained through diverse extraction methods, including sub-/supercritical extraction with or without a modifier, Soxhlet extraction, and hydro-distillation using various solvents. The results demonstrated that the highest MEO yield over 2%, which was achieved with SFE at 300 bar and 50 °C, with a yield isotherm crossover occurring between 140 and 170 bar. When a co-solvent was employed in SFE, ethanol outperformed ethyl acetate in terms of oil yield. The extracted MEO was rich in compounds with therapeutic and industrial significance, such as pulegone, cineol, and carvone, and exhibited commendable antioxidant activity, enhancing its suitability for various applications. This research offers valuable insights into MEO extraction, considering different methods and operational conditions (Almeida et al. 2012).

A study investigated the chemical composition and biological properties of *Mentha longifolia* L. EO employing different extraction techniques. In this regards, three different methods, including supercritical CO_2 fluid extraction, lipophilic SE (n-hexane), and SD were compared. Lipophilic SE yielded the highest EO content at 1.2% (w/w). Gas chromatography-flame ionization detector (GC-FID) and GC-MS analyses identified a total of 39 compounds in the extracted EOs. Major components in EOs from SD and lipophilic SE included carvone, limonene, trans-caryophyllene, and α-terpineol. In contrast, the supercritical CO_2-extracted oil contained germacrene D, trans-β-farnesene, trans-caryophyllene, and carvone, as major constituents. All EOs exhibited varying antioxidant activities, with the supercritical CO_2-extracted oil demonstrating the highest scavenging activity in both DPPH and ABTS assays. This study sheds light on the chemical composition and antioxidant potential of *Mentha longifolia* L. EOs obtained through different methods, emphasizing the superior antioxidant properties of supercritical CO_2 extraction (Bai et al. 2020). Besides, in a study focusing on supercritical CO_2 extraction of MEO, various important aspects were explored. The research involved the preparation of dried leaves through milling and sieving to obtain particles of different sizes. These particles were then subjected to supercritical CO_2 treatment under specific conditions (40 °C and 10 MPa) for MEO extraction. An innovative aspect of this study was the introduction of a novel modeling approach called the "pseudo kinetic model". This model was proposed to better describe and predict the extraction process and its kinetics. The study compared the performance of the pseudo kinetic model with Sovova's model, a widely used model in supercritical CO_2 extraction literature. The primary goal was to assess how particle size influences extraction yield and establish an optimal methodology for determining the parameters of the pseudo kinetic model, potentially providing a more accurate representation of the extraction process. Results showed that both the pseudo kinetic model and Sovova's model effectively described the supercritical CO_2 extraction process, suggesting the potential utility of the pseudo kinetic model for understanding and predicting extraction dynamics. Overall, this study advances our understanding of how particle size affects extraction yields and introduces an innovative modeling approach that could enhance the efficiency and accuracy of supercritical CO_2 extraction processes (Maksimovic et al. 2012).

In another study conducted on SFE by Pop and Barth (2001), the focus was on Romanian *Mentha* hybrids as the source of EOs high in Z-sabinene hydrate. The aim of the research was to extract the volatile components, particularly Z-sabinene hydrate and its acetate, from a mint hybrid found in Romania. This was achieved using supercritical CO_2 extraction. The study determined that the highest yield, amounting over 2%, was achieved within a pressure range of approximately 93 bar and at a temperature of 50 °C. Additionally, a kinetic study was carried out to evaluate the supercritical CO_2 extraction process, and the results were compared to those obtained through hydro-distillation. The monitoring of the extraction process was conducted using and GC-MS analysis. The research established that the optimal conditions for supercritical CO_2 extraction resulted in extracts with a high content of Z-sabinene hydrate and its acetate, while still retaining a flavor profile similar to the original plant material. These optimal conditions were identified as a pressure range around 95 bar and a temperature of 50 °C. The study also highlighted an interesting finding regarding hydro-distillation: it was noted that only at the beginning of the process was it possible to obtain a high content of Z-sabinene hydrate. However, achieving a high content of its acetate was found to be challenging through this method. In conclusion, this study not only provides an effective method for obtaining Z-sabinene hydrate from a natural source but also emphasizes the importance of using mild extraction conditions to preserve valuable natural compounds. This underscores the potential of supercritical CO_2 extraction as a valuable tool in this context. In a separate study, EO extraction from spearmint leaves was conducted using supercritical CO2 extraction (Fig. 4). The primary aim of this research was to explore how various operational

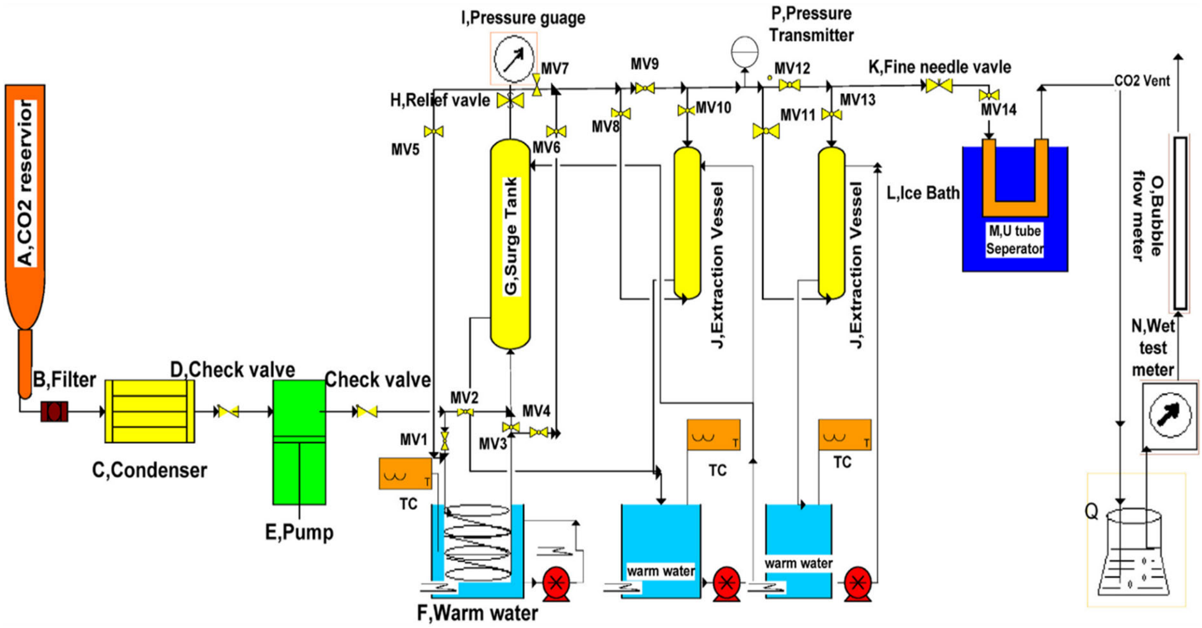

Fig. 4 Schematic diagram of the extraction of mint essential oil (MEO) with supercritical carbon dioxide (CO_2); adapted from Shahsavarpour et al. (2017)

parameters affect the efficiency of EO extraction from spearmint leaves. The study encompassed a series of well-planned extraction experiments to assess the influence of different variables on the extraction process. These variables included dynamic extraction time (i.e., spanning from 20 to 120 min), mean particle size (i.e., ranging from 0.18 to 2 mm), CO_2 flow rate (i.e., varying from 0.06 to 0.35 g/min), extraction temperature (i.e., ranging from 38 to 50 °C), and extraction pressure (i.e., ranging from 85 to 120 bar). In the concluding phase of the investigation, the researchers developed a second-order polynomial equation to pinpoint the optimal parameters for supercritical CO_2 extraction of EOs from spearmint leaves. Additionally, the composition of the EO extracted under these optimal conditions was analyzed using GC-MS. This research contributes valuable insights into optimizing the parameters of supercritical CO_2 extraction for spearmint leaf EOs, shedding light on the composition of the extracted oil under optimal conditions, which holds significance for various applications and further research endeavors in this domain (Shahsavarpour et al. 2017). In a further study, Shrigod et al. (2017) investigated SFE of MEO to optimize EO yield and carvone content. Using a central composite rotatable design, they explored temperature (i.e., 35–55 °C), pressure (i.e., 100–300 bar), dynamic time (i.e., 20–90 min), and particle size (i.e., 0.2–1 mm) as key parameters. Particle size had the most significant impact on oil yield, followed by pressure, temperature, and dynamic time. For carvone content, pressure was most influential, followed by particle size and dynamic time. The study determined optimal conditions as follows: 48 °C temperature, 151 bar pressure, 0.4 mm particle size, and 37.5 min dynamic time, resulting in 1.4% oil yield and 998 mg/100 g carvone content. The oil showed potent antimicrobial activity, particularly against E. coli. These findings offer valuable insights for MEO extraction using supercritical CO_2.

Subcritical water extraction (SWE)

Subcritical/superheated water extraction (SWE), also known as pressurized low-polarity water extraction (PLPWE), or pressurized hot-water extraction (PHWE) is an emerging and green technology for the extraction of bioactive compounds. The subcritical state of water refers to the liquid water at temperatures between 100 and 374.15 °C and pressures below the critical pressure ($P_c = 22.1$ MPa) and higher than the vapor pressure. Under these extreme conditions, the polarity of water is much lower than at room temperature. Therefore, the organic compounds are more soluble and the extraction process can be accomplished in a shorter time. The subcritical state of fluid offers several superior characteristics, such as lower viscosity, density, and surface tension, as well as enhanced diffusivity between gas and liquids. The use of subcritical state of fluids has been reported by many researchers and found that this is a better and powerful alternative of EOs extraction technique (Özel et al. 2006). This extraction technique is considered the best alternative approach as it enables a fast EO isolation process. Moreover, it is a cost-efficient extraction, simple and environmentally friendly process due to the application of non-toxic and non-flammable solvents. EOs with more valuable properties and higher amount of oxygenated components with no significant presence of terpenes can be obtained and allow substantial cost saving in terms of both energy and plant materials (Tongnuanchan and Benjakul 2014).

In this respect, Chiou et al. (2019) used subcritical water with pressure-releasing distillation to extract EOs and volatile fractions from Japanese mint (*Mentha arvensis* L. var. *piperascens* Malinv. cv.) and compared the results with the EOs obtained by SD. The process was performed at 100–220 °C, during 5–60 min. GC was used to quantitatively analyze the extracted compounds. The results revealed that increasing the extraction temperature from 100 to 180 °C, increased the yields of l-menthol, l-menthone, l-limonene, and piperitone, but at higher temperatures the extraction yield was decreased. On the other hand, the yields of l-menthyl acetate, iso-menthone, and 3-octanol increased up to 220 °C. Extending the extraction time, negatively affected the extraction of MEO compounds and the highest yield was obtained after 5 min treatment at 180 °C. While, the extraction by team distillation required more than 1 h to reach a similar yield. In a similar study, Ammann et al. (1999) compared the effects of SWE, SFE, and SD on the extraction of peppermint (*Mentha piperita* L.) EOs. The extraction yield and composition of MEO isolated by SD were more appropriate than other EOs. However, the extraction by SWE was faster than other methods and the extraction efficiency was higher than SFE. During the first few minutes, the speed of all extraction methods was comparable. However, shortly thereafter the SFE speed decreased and it presented the lowest yield. They stated that SFE takes 3–10 times longer than SD to achieve a comparable yield and is the least suitable method for MEO extraction. In another study, Kubátová et al. (2001) compared the extraction yields of oxygenated and non-oxygenated compounds in peppermint by SWE, SFE, and hydro-distillation. The results indicated that, increasing the temperature of SWE from 100 to 175 °C increased the extraction rate; nevertheless, some of the desired components such as terpinene and linalool exhibited considerable degradation at temperatures above150°C. Conversely, components such as thymoquinone were not degraded in SWE as the extracted flavor compound was not exposed to atmospheric oxygen. The amounts of menthol, menthone, carvone, piperitone, pulegone, eucalyptol, and neomenthol obtained from peppermint after 4 h of hydro-distillation were comparable to those extracted by SWE processing for 30 min at 150 °C or 12 min at 175 °C and SFE for 1 h. However, the recovery of less polar compounds such as menthyl acetate in SWE was only 40% of hydro-distillation. Therefore, SWE selectively extracts the flavor compounds and can extract more polar (oxygenated) compounds in a short time (as little as 12 min). In contrast, SFE and hydro-distillation do not act selectively and the extracted samples contain flavor compounds as well as plant alkane waxes. In a further study, Çam et al. (2019) optimized the extraction conditions of MEO by SWE. They investigated the effects of different extraction times (i.e., 1, 5, 10, or 30 min), temperatures (i.e., 40, 70, 100, 130, or 160 °C), and extraction cycles (1–5) on the composition and extraction yield of MEO. The results revealed that the best extraction conditions were as follows: temperature of 130 °C and extraction time of 10 min after 3 extraction cycles. They reported that the MEO content of samples obtained at 130 and 160 °C did not show statistically significant differences. The dominant component of MEO was determined as menthol by GC-FID. The extraction yield under optimized conditions was approximately 2-fold higher than the conventionally extracted sample.

Microwave-assisted extraction (MAE)

Microwaves (MW) are electromagnetic waves between infrared (IR) and radio frequency (RF) waves with frequencies ranging from 300 MHz to 300 GHz. MW processing is a heat treatment that plays an important role in the safety and quality of food products. It is used in many fields of the food industry, such as drying, preheating, cooking, thawing, tempering, blanching, roasting, pasteurization, sterilization, enzyme inhibition and inactivation, microbial inactivation, and extraction (Hashemi et al. 2019). In this method, heat is generated inside the food matrix based on two mechanisms: (1) electrophoretic transport of electrons and ions, which creates an electric field responsible for particle movements, and (2) dipole rotation, which is caused by the alternating movement of polar molecules. These two phenomena promote the release of energy, resulting in rapid heating, which accelerates tissue damage and increases the diffusion of targeted compounds (Marić et al. 2018). The increasing demand for environmental protection has prioritized research efforts to expand the use of MW technology, especially in practices that require purification and extraction.

Microwave-assisted extraction (MAE) usually functions through different modes of extraction, i.e., MW polarization, hydro-diffusion, or a combination of these modes. The polarity of plant compounds and their structure mainly determine the type of extraction mechanism. During MAE, the moisture inside the cell walls interacts with the oscillating MW field (from positive to negative) due to the dielectric properties of water, leading to ionic rotations that create extreme heat and pressure inside the cell. Accordingly, the plant tissue is ruptured and its content is released into the surrounding solvent (Liew et al. 2016; Picot-Allain et al. 2022). The processing conditions for the MAE of EOs is varied for plant sources; and parameters such as power, pH, processing time, and the plant-solvent ratio should be optimized in this process. Also, MAE can be performed by different approaches, such as microwave-assisted distillation (MWAD), microwave-assisted hydro-distillation (MWAHD), microwave steam distillation (MWSD), microwave hydro-diffusion and gravity (MHG), as well as solvent-free microwave extraction (SFMWE) (Kokolakis and Golfinopoulos 2013).

Numerous studies have demonstrated that MAE process requires shorter time, lower energy, and solvent consumption compared to conventional extraction methods. Also, the enhanced extraction kinetics increase the extraction yield of MAE (Ripoll and Hincapié-Llanos 2022). Consequently, MAE is an acceptable and feasible technique for the extraction of EOs from aromatic plants such as mint and several researchers have investigated the MAE of MEO (Table 1). In this regard, Abdel-Hameed et al. (2018) evaluated the efficiency of different extraction techniques, such as hydro-distillation, MWAHD, and SFMWE to isolate EOs from *Mentha piperita* L.; and also, investigated the antimicrobial and anticancer properties of EOs obtained by these extraction approaches. The chemical composition and the extraction yield of EOs obtained by different extraction methods showed significant differences. However, no significant differences were observed among the MEO yields extracted via the three different methods. In addition, the methods based on MW were more energy-saving compared to conventional hydro-distillation methods. The chemical properties of EOs were qualitatively similar to each other; however, they were not much quantitatively different. Also, all of the MEO samples showed moderate anticancer and high antimicrobial activities in vitro (Abdel-Hameed et al. 2018). Moreover, Wang et al. (2006) used SFMWE and MWAHD to extract MEO with no solvents or pre-treatments. The SFMWE method was accomplished in 30 min, while MWAHD took 90 min and conventional hydro-distillation was completed in 180 min. The EOs extracted by different techniques had similar chemical compositions. Nonetheless, the SFMWE method was more feasible for the extraction of MEO. In another study, Figueredo et al. (2012) reported similar results for *Mentha spicata* L. EOs extracted by MWAHD or hydro-distillation methods and stated that the major components of MEO were consistently present, regardless of the extraction technique (Fig. 5). These findings also confirmed the results of a previous research by Kohari et al. (2020) who reported that the composition of on Japanese peppermint (*Mentha arvensis L.*) extracted via SFMWA and hydro-distillation were similar to each other. In a further study, Shankar and Natarajan (2022) led relevant research on the extraction of *Mentha spicata* L. EO. The oil yield, chemical composition, and antioxidant activity of EOs extracted by MWAD and hydro-distillation methods were analyzed. The MWAD method resulted in the maximum oil yield because of its efficient heat-transfer capacity. The qualitative differences between the EOs extracted via MWAD and hydro-distillation were identified in the chemical profile of the EOs when analyzed by GC-MS. The DPPH assay showed that the EOs extracted by MAE presented better radical scavenging activity and greater antioxidant activity, compared to the hydro-distillation.

Table 1 Effects of species, cultivation area, and microwave-assisted extraction conditions on the yield and main component of MEO

Species	Cultivation area	Extraction conditions			MEO yield (%)	Main component	Reference
		Power (W)	Time (min)	Solid: liquid ratio			
Mentha piperita L.	Taif, Saudi Arabia	500	40	1:3	0.36	Carvone	Abdel-Hameed et al. (2018)
Mentha longifolia L.	Taif, Saudi Arabia	500	40	1:3	0.60	Pulegone	Abdel-Hameed et al. (2018)
Mentha arvensis L.	Lam Dong, Vietnam	540	20	1:2	0.8	Menthol	Bui-Phuc et al.(2020)
Mentha spicata L.	Konya, Turkey	800	10	2:1	ND	Carvone	Figueredo et al. (2012)
Mentha rotundifolia L.	Zaragoza, Spain	900	5	1.25:10	1.6	Piperitenone oxide	García-Sarrió et al. (2018)
Mentha arvensis L.	Hokkaido, Japan	150	60	1:10	0.93	l-Menthol	Kohari et al. (2020)
Mentha piperita L.	East Java, Indonesia	450	60	1:10	0.69	ND	Kunhermanti and Mahfud 2023)
Mentha crispa L.	Reunion Island, France	500	30	1:20	0.095	Carvone	Lucchesi et al. (2004)
Mentha pulegium L	El-Guetfa, Algeria	800	30	ND	0.175	Pulegone	Messaoudi et al. (2021)
Mentha piperita L.	Novi Sad, Serbia	800	120	1:10	0.80	Menthol	Radivojac et al. (2021)
Mentha spicata L.	Thanjavur, India	630	3	1:2	2.53	Carvone	Shankar and Natarajan 2022)

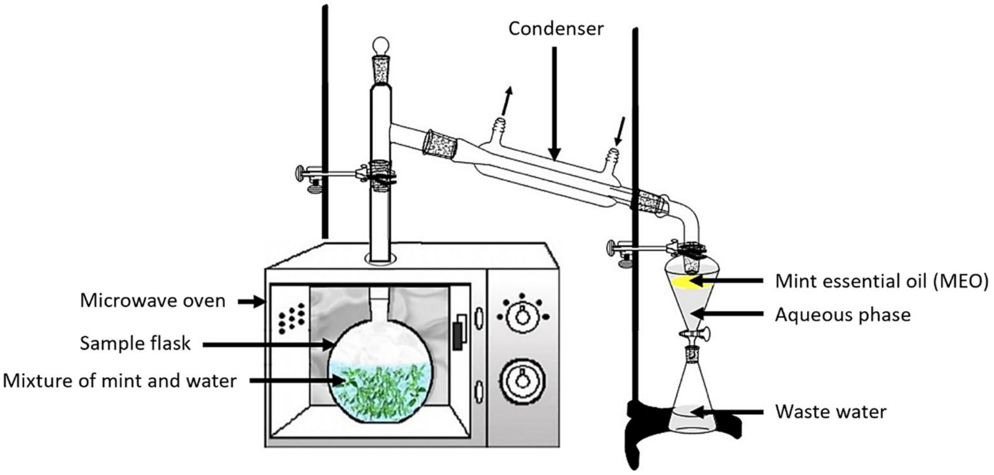

Fig. 5 Schematic diagram of microwave-assisted hydro-distillation (MWAHD) for mint essential oil (MEO) extraction; adapted from Kusuma et al. (2017)

In a recent study, Kunhermanti and Mahfud (2023) optimized the SFMWE of EOs from *M. piperita* L. using response-surface-methodology (RSM). The variables included the feed-to-distiller volume (F/D) ratio (i.e., 0.08–0.12 g/mL), MW power (i.e., 150–450 W), and particle size (i.e., 0.5–1.5 cm).

The optimized MEO extraction yield was 0.6937% that was obtained by applying MW power of 450 W, particle size of 1 cm, 60 min of extraction time, and 0.1 g/mL of F/D ratio. Furthermore, Lucchesi et al. (2004) employed the SFMWE and hydro-distillation techniques to extract EOs from *M. crispa* L. The extraction by SFMWE was completed in half an hour; while, the classical hydro-distillation took 4.5 h. Additionally, the SFMWE yielded a larger amount of MEO, as well as more oxygenated compounds yield. Therefore, they confirmed that SFMWE can be employed as a suitable alternative to traditional extractions of MEO. In another study, Messaoudi et al. (2021) extracted EOs from *M. pulegium* L. by hydro-distillation, SD, and MWAD, and investigated their chemical composition, antibacterial, antifungal, and anti-inflammatory activities. The results revealed that MEOs had significant anti-inflammatory activity and antimicrobial properties against pathogenic bacteria and fungi. The differences were particularly observed in the extraction yield and the components of EOs obtained by different extraction methods. Besides, the MWAD was performed in 30 min, while conventional extraction was completed in 180 min. However, the extraction yield of MEO isolated by hydro-distillation and SD was higher than that of MWAD extracted MEO, and the worst chemical composition was obtained by MWAD.

In a different study, Mohammadhosseini et al. (2023) extracted *M. pulegium* L. EO, by MWAHD and headspace-solid phase micro-extraction (HS-SPME) combined with GC-MS. The MEOs isolated by HSSPME-GC-MS and MWAHD-GC-MS techniques revealed 28 and 30 components, respectively. Also, the MEO obtained by both extraction methods contained large amounts of oxygenated monoterpenes and non-terpenoid hydrocarbons; however, the concentration of their individual compounds was significantly different. Carvone comprised 56.0% of the MEO extracted by MWAHD, whereas the content of carvone in MEO from SPME technique was17.7%. In a further study, Bui-Phuc et al. (2020) evaluated the effect of hydro-distillation and MAE on the extraction of EOs from Japanese mint (*Mentha arvensis* L.) and found that the MEO components and yield were influenced by the extraction technique. The extraction yield was 0.0832 mL g^{-1} per dry weight and the menthol comprised 70.60% of the MEO when extracted by MAE. While, using the hydro-distillation technique yielded 0.0510 mL g^{-1} MEO per dry weight and generated a smaller amount of menthol (62.80%). Moreover, the trans- and cis-menthone, occurred abundantly in the EO when using either MAE or hydro-distillation (4.62–5.01% and 20.19–16.30, respectively). In comparison with the hydro-distillation, MAE is reportedly capable of extracting higher EO yields and more menthol. The MAE is particularly the base of an advantageous approach due to its shorter distillation time and more efficient extraction. Similarly, Pavlić et al. (2021) reported that MWAHD is a good alternative to traditional EO recovery methods. The results showed that the MEO obtained by MWAHD provided higher content of menthone and similar amount of menthol compared to the EOs extracted by hydro-distillation. Also, the enzyme-inhibitory and antioxidant activities did not show significant differences in EOs obtained by MWAHD and hydro-distillation.

Since the efficiency of the MAE process is controlled by parameters, such as temperature, power, and extraction time, some researchers have investigated the influence of these parameters on the extraction efficiency of MEO. For instance, Costa et al. (2014) used a calorimetric technique to calibrate a MW reactor for MAE under different extraction times and temperatures. MEO was extracted from the dried mint leaves by MAE at different temperatures (i.e., 40 °C, 50 °C, and 60 °C) and times (i.e., 10, 20, and 30 min). The GC-MS analysis results revealed that carvone was the major compound in the extracted MEOs. A face-centered central composite design (2^2 +2 center points) was applied and the highest percentage of the carvone (0.091) was obtained for temperature and time at 40 °C and 30 min, respectively. While, the lowest percentage of the carvone (0.011) was determined for temperature and time of 50 °C and 10 min, respectively.

Ultrasound-assisted extraction (UAE)

Ultrasound is an emerging technology with several applications in detection, modification, and extraction of natural compounds, such as polyphenols, hydrocolloids, fibers, and EOs. It is an acoustic treatment using sound waves with frequencies higher than 20 kHz, that is beyond the human hearing threshold. Ultrasound waves create cavitation within the aqueous phase. The sudden collapse of the cavitation bubbles near the cell walls generates strong mechanical forces such as shear force, microscopic turbulence, and shock waves that cause cell disruption (Tarahi and Ahmed 2023). As a result, the solvent permeation into plant tissues is enhanced and the extraction efficiency of EOs is increased. In addition to the cell wall disintegration, the enhancement of extraction yield by ultrasound treatment is attributed to particle size reduction and mass transfer enhancement by the collapse of cavitation bubbles (Da Porto and Decorti 2009; Romdhane and Gourdon 2002).

The use of ultrasound treatment for EO extraction has several advantages over conventional methods. For instance, it significantly reduces the processing time and increases the extraction yield at lower temperatures. Additionally, the energy consumption and maintenance costs of ultrasound processing is relatively low; therefore, it is considered as an economic technology (Giacometti et al. 2018). However, conflicting results have been also reported regarding the impact of ultrasound on EOs. For instance, some studies have demonstrated that the UAE of EOs can significantly improve the bioactivity and physicochemical properties of herbal compounds (Damyeh et al. 2016). While others have stated that sonication do not have considerable effects of EOs (Kowalski et al. 2019), and some studies reported that UAE have negative effects on EOs (Tekin et al. 2015; Yu et al. 2021). They have stated that the sudden collapse of microbubbles in sonication process generates high temperature and pressure, free radicals, and superoxide, which may adversely affect the quality of phytochemicals. In addition, the operational conditions, such as intensity, frequency, and sonication time have significant effects on the properties of phytochemicals. Therefore, the optimization of UAE can provide EOs with higher bioactivity compared to conventionally extracted EOs.

The optimization of MEO extraction by UAE technique has been addressed by several researchers in recent years. For instance, Sierra et al. (2022) optimized the UAE of *Mentha spicata* L. EO and determined the chemical composition of the extracted EO by GC-MS and high-performance liquid chromatography with diode-array detection (HPLC-DAD) analysis. Furthermore, Peleg's equation and polynomial regression analysis were used to define the extraction process. The modeling results revealed that time, solvent, and amplitude have significant effects in the recovery of MEO. The optimized extraction conditions were as follows: ethanol/water ratio (80:20), and 90% amplitude for 5 min, which resulted in the highest amounts of terpenoid compounds. In another study, Mansoori et al. (2022) utilized UAE to extract limonene and carvone from *Mentha spicata* L. Box-Behnken design (BBD) was used to optimize the extraction parameters, such as extraction time (20–60 min), amplitude (20–100%), and the solvent: solid particles ratio (10–30 mL/g). The correlations obtained by different models revealed that the second-order polynomial model can be successfully applied to optimize the extraction variables. Also, the optimal conditions for the extraction of both limonene and carvone were the extraction time of 51 min, amplitude of 95% (380 W), and the solvent: solid particles ratio of 24 mL/g. Under these optimized conditions, the extraction yield was 19.958 for carvone and 5.957 mg/g for limonene. These values are in good agreement with the predicted values of 20.237 and 6.024 mg/g obtained by BBD. Also, the results of this study revealed that UAE reduced the extraction time and temperature; while, increased the extraction yield by 54% compared to conventional hydro-distillation. Furthermore, Shotipruk et al. (2001) studied the feasibility of UAE for the extraction of menthol from peppermint. They exposed the plant materials to sonication for 1 h at 22 °C in an ultrasound bath of 40 kHz and found that increasing the treatment time and temperature significantly increased the amount of menthol release. The extended sonication time increases the diffusion of menthol in the cuticle of glandular trichomes of peppermint and its exudation from the damaged trichomes. Whereas, increased temperature increases the solubility of menthol and cause the phase transition of menthol crystals to a liquid state, and thus, facilitates its release from cells. In addition, Kowalski et al. (2015) studied the effects of ultrasound-assisted preliminary maceration on the chemical composition and extraction yield of EOs from peppermint, marjoram, and chamomile. The plant materials (20 g) were added to distilled water (1000 mL) and treated with an ultrasound bath at 30 °C with a frequency of 40 kHz and power of 240 W. The results demonstrated a significant enhancement in the amount of extracted MEO and marjoram EO. However, sonication did not have a significant effect on the extraction yield of chamomile EO. Additionally, the composition of EOs was similar to conventionally extracted EOs and sonication did not destroy the components of EOs.

Some studies have investigated the effects of ultrasound in combination with other treatments on the extraction of MEO. For instance, Moradi-Sadr et al. (2023) studied the effects of ultrasonic and cold plasma (CP) pre-treatments alone and in combination, and also the ultrasound and water/plant ratio on the MEO extraction. The process was optimized by RSM with a central composite design (CCD). The variables included sonication temperature and time, CP power, and the ratio of water to plant material. The results indicated that the extraction yield of MEO in all of the treatments was higher than the control. The optimal conditions of ultrasound treatment caused a 119.7% increase in MEO compared to the control, while the enhancement of MEO yield in water to material ratio-ultrasonic and CP-ultrasonic pre-treatments were 206.6% and 155.7%, respectively. The extent of oxygenated monoterpenes under optimized conditions in all three treatments was higher than that of the control. Also, it was found that increasing the CP power and sonication time, increased the MEO extraction yield. While, increasing the sonication temperature decreased the extraction efficiency. Moreover, an inverse correlation was observed between the water to plant ratio and the MEO yield.

Overall, UAE of MEO represents a higher yield compared to the conventional methods in a much shorter processing time, less solvent usage, and lower energy consumption. All these factors demonstrated the application of ultrasound as a “green” and “high-efficiency” method for the extraction of MEO.

Ohmic-assisted extraction (OAE)

Ohmic heating (OH) is considered as an innovative thermal process that creates heat inside the material (Niakousari, Hedayati, Tahsiri, et al., 2019). This alternative heat treatment is considered as a high temperature short time (HTST) process. During ohmic processing an alternating electrical current pass through the food matrix and uniformly increases the temperature due to its electrical resistance. Therefore, the disadvantages of conventional heating such as fouling of the heat exchangers, overheating, and non-uniform heating of food products, would not occur in OH. This innovative technology has attracted considerable attention in different areas of food processing (e.g., extraction, sterilization, fermentation, blanching, dehydration, and evaporation) due to its effectiveness in inactivation of microorganisms, lower energy consumption and shorter operation time compared to conventional methods. Also, it has been stablished that the food products treated by OH have higher nutritional and sensory quality (Niakousari et al. 2019).

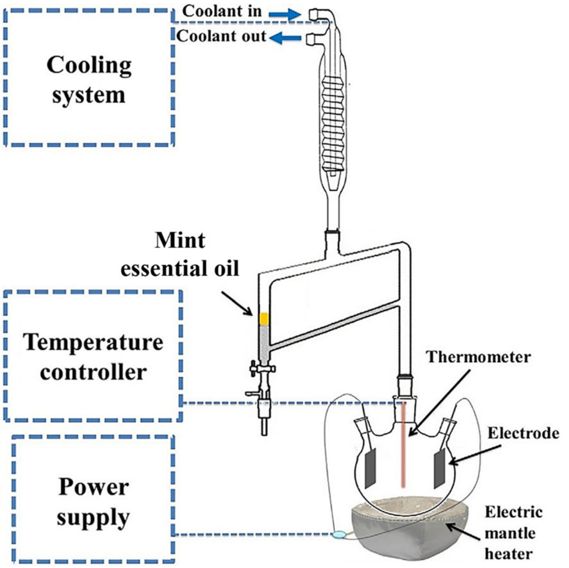

Fig. 6 Schematic diagram of ohmic assisted hydro-distillation (OAHD) system for mint essential oil (MEO) extraction; adapted from Gavahian et al. (2020)

Application of OH for the extraction of EOs is a relatively new concept and several studies have reported the successful extraction of EOs by OH. In this respect, Gavahian et al. (2015a) compared the efficiency of ohmic assisted hydro-distillation (OAHD) and traditional hydro-distillation for the extraction of peppermint EOs (Fig. 6). The dried aerial parts of peppermint (30 g) were added to 500 mL salted water (1% NaCl, w/v) and treated by different heating methods for 2 h. OAHD was performed by an ohmic distillator with platinum electrodes operating at 220 V, 50 Hz. While, conventional hydro-distillation was performed by a Clevenger-type apparatus on a laboratory heater. To remove water from the extracted MEO, it was dried over anhydrous sodium sulfate; and then, refrigerated in amber containers (4 °C). The results indicated that OAHD was an environmentally friendly extraction method as it significantly reduced the extraction time and energy consumption. OAHD was accomplished in less than half an hour while hydro-distillation process took about 1 h. Besides, scanning electron microscopy (SEM) images of peppermint leaves that were exposed to OAHD revealed a quick rupture of EO glands that accelerated the extraction process. Whereas, those subjected hydro-distillation did not show a sudden rupture of EO glands. Based on the GC-MS analysis results, the individual compounds of MEOs extracted by OAHD and hydro-distillation did not show obvious changes. Furthermore, the results indicate that using 1% NaCl did not have any adverse effects on the extractions parameters and quality of obtained EOs from mint. In a similar study, they investigated the influence of different extraction parameters such as frequency (i.e., 25, 50 and 100 Hz) and voltage (i.e., 220 and 380 V) on the quality and quantity of peppermint EOs extracted by OAHD, and compared the results with the EOs extracted by conventional hydro-distillation. The results showed that the extraction time reduced from 19.71 to 13.54 min by increasing the voltage of OAHD. While the extraction time in hydro-distillation was about one hour. However, the frequency did not have any significant effects on the extraction time and yield. Also, the GC-MS analysis of EOs extracted under different conditions did not show evident changes in their individual compounds (Gavahian et al. 2015b). In another study, they investigated the impact of NaCl concentration (i.e., 1, 3, and 6% w/v) and pre-treatment with ultrasound (i.e., 5, 10, and 15 min) on the MEO extracted by OAHD. The results indicated that the concentration of NaCl solution had significant effects on the extraction parameters of OAHD and the increase of electrolyte concentration increased the speed of extraction process with no adverse effects on the quality of extracted MEO. Therefore, the extraction time can be reduced by the optimization of NaCl concentration. Whereas the effects of sonication pre-treatment on extraction rate and duration were negligible. GC-MS analysis did not indicate any noticeable change in the compounds of all the extracted EOs (Gavahian et al. 2017).

Pulsed electric field-assisted extraction (PEFAE)

Pulsed electric field (PEF) is a non-thermal emerging method that involves applying high voltage electric fields for extremely short (milliseconds to microseconds) periods to a material positioned between two electrodes. This technique is mainly used for food preservation by the inactivation of microorganisms and enzymes. PEF inactivates microorganisms by producing pores in their cell membrane. As a result, the intracellular contents leak out of the cell and the cell membrane functions interrupt (Abenoza et al. 2013; De Silva et al. 2018). The pore formation in PEF processing can occur in plant tissues, increases the permeability of plant cells and facilitates the release of intracellular compounds (Fincan and Dejmek 2002). Therefore, in recent years, PEF processing has been applied to increase the extraction yield of EOs, such as MEO.

Generally, PEF is applied to plant materials as a pre-treatment before the conventional extraction methods in order to increase the extraction yield of EOs and intensify the concentration of valuable compounds. In this regard, Miloudi et al. (2022) investigated the effects of PEF processing on the quantity and quality of extracted *Mentha spicata* L. EOs. PEF with a pulse frequency of 1 Hz was used as a pre-treatment and then MEO was extracted by hydro-distillation with a Clevenger-type apparatus. The samples without PEF pre-treatment were considered as the control. Dried Mint (20 g) was immersed in distilled water (200 ml) and heated by a balloon heater. The vapors were cooled to condense and accumulated in the collector of Clevenger apparatus, where MEO and water were separate by differences in their densities. The results showed that the antioxidant and anti-inflammatory properties of MEO were comparable in control and PEF-treated samples. The extraction yield of the control was 0.94% in 120 min of hydro-distillation while a similar yield was obtained after 200 pulses of PEF in 60 min of hydro-distillation. The GC-MS analysis showed that the composition of MEO was almost the same in PEF and conventionally extracted MEO, indicating that MEO and its individual compounds were not affected by PEF treatment. These findings are in agreement with the results reported by Cherrat et al. (2014) who stated that the chemical composition and antioxidant properties of *Mentha pulegium* L. EO were not affected by PEF. However, contradictory results have been reported by Sadowska et al. (2019) who studied the effect of the number of PEF pulses applied to peppermint on the EO content. They subjected the plant materials to 0, 150, 250, or 350 pulses with 25 µs duration and the time interval of 10 s. Subsequently, the EOs were extracted by hydro-distillation in a Clevenger apparatus and the content and composition of EOs were analyzed. The highest extraction yield was observed in the control sample and PEF treatment decreased the extraction yield of MEO. Nevertheless, the number of pulses did not affect the MEO content. Therefore, the processing conditions, such as number, intensity, and duration of pulses are key factor for the successful extraction of MEO by PEF technique.

Research gaps and future trends

Currently, research on MEO extraction has made significant progress; however, several research gaps remain, offering opportunities for further investigation and innovation. Firstly, while conventional extraction methods, such as SD, are widely used, there is an urgent need to optimize and simplify these processes in order to improve their efficiency and sustainability. Investigating variations in extraction parameters, such as temperature, pressure, and extraction time, can provide valuable insights into maximizing oil yield and quality. Secondly, the use of emerging technologies for MEO extraction is an evolving area with significant potential. Different techniques, such as SFE, OAE, MAE, UAE, and so on, have shown several advantages, but their scalability, cost-effectiveness, and comparative efficacy with traditional methods require further in-depth investigation. Understanding the underlying mechanisms of these emerging technologies and their impact on the chemical composition of the extracted EOs is crucial for their successful integration into industrial practices.

Furthermore, there is a notable gap in research regarding the influence of mint species and varieties on EO composition. Different *Mentha* species and varieties may significantly affect oil yield and chemical profiles, influencing their applicability in various industries, such as food, pharmaceuticals, and cosmetics. Therefore, a comprehensive analysis of these variations can guide the selection of mint cultivars tailored to specific end-use requirements. Moreover, there is still little research on the post-extraction processing and storage of MEOs. Investigating the stability, shelf-life, and potential degradation pathways of these oils under different storage conditions is critical for maintaining their quality and bioactivity over time.

Overall, filling these research gaps in future studies can contribute to the advancement of MEO extraction technologies, strengthening sustainability, maximizing efficiency, and expanding the range of applications in various industries.

Conclusion

MEO is a valuable and widely used EO with numerous industrial applications. Extraction has a great role in the bioactivity and physicochemical properties of MEO. Despite the simplicity and availability of conventional EOs extraction apparatuses, high energy and time consumption have restricted the application of these extraction techniques. Application of emerging technologies, such as microwave-assisted, ohmic-assisted, ultrasound-assisted, pulsed electric field, and super and sub-critical fluid extraction methods can reduce the energy dissipation and extraction time and improve the extraction yield of MEO. However, the review of several researches revealed that not only the quality, but also the quantity, and composition of MEO can be altered by the processing conditions. Therefore, the extraction parameters should be optimized to obtain a high content of MEO with the best biological and physicochemical properties. Such advantages make emerging technologies excellent alternatives to conventional extraction approaches for MEO that can be considered as environmentally friendly extraction methods and can encourage the development of this emerging technology in industries.

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Declarations

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