

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

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## 14 Abstract

15 Mint essential oil (MEO) is an economically appreciated natural product with significant importance in  
16 the cosmetics, pharmaceuticals, foods, and healthcare products due to its biological activities. Extraction  
17 is a critical step in the production of essential oils from aromatic plants, such as mint. Conventional  
18 extraction approaches, such as steam distillation and solvent extraction are commonly used for the  
19 extraction of MEO. However, they are energy and time-consuming processes with relatively low  
20 extraction yields. Consequently, emerging techniques, such as microwave-assisted, ohmic-assisted,  
21 ultrasound-assisted, pulsed electric field, and super and sub-critical fluid extraction methods have been  
22 developed to overcome these shortcomings. This review aims to investigate the influence of different  
23 extraction methods and conditions on the extraction yield, composition, physicochemical properties,  
24 and bioactivity of MEO. Overall, selection of appropriate extraction method and conditions is a crucial

25 step in the extraction of MEO that can have significant effects on the quality and quantity of the isolated  
26 EOs. Inappropriate extraction of MEO may reduce the extraction yield, prolong the extraction duration,  
27 and increase the energy consumption. Additionally, it may deteriorate the physicochemical properties  
28 and bioactivity of the extracted MEO.

29

30 **Keywords:** Mint essential oil; Traditional Persian; Medicine Medicinal plants; Extraction Distillation;  
31 Green extraction technologies; Ultrasound-assisted extraction; Microwave-assisted extraction.

32

### 33 **Introduction**

34 The biologically active compounds extracted from the roots and aerial parts of medicinal plants contain  
35 primary and secondary metabolites. Plant secondary metabolites, also known as phytochemicals,  
36 include a wide range of bioactive compounds that play an essential role in protecting plants against  
37 biotic and abiotic stresses (Soleimani et al. 2022). The widespread use of phytochemicals in traditional  
38 cultures for health promotion and disease treatment has led to their incorporation into contemporary  
39 products. These plant-derived compounds are often preferred over synthetic chemicals. The World  
40 Health Organization (WHO) has estimated nearly 80% of the world's population relies on herbal  
41 medicines for health purposes (Sadowska et al. 2016). Essential oils (EOs) are complex mixtures of  
42 small organic molecules, including terpenic (especially mono- and sesquiterpenoids and mono- and  
43 sesquiterpenes), aromatic, and aliphatic components extracted from roots, stems, leaves, flowers, fruits,  
44 or seeds of many herbal plants (Ebadollahi et al. 2020). They are widely used in the food, cosmetic,  
45 perfumery, and pharmaceutical industries due to their flavor and fragrance properties, as well as their  
46 anti-inflammatory, antimicrobial, antioxidant benefits, and their demand is increasing day by day all  
47 over the world (Zhang et al. 2022).

48 Mint (*Mentha* spp.) is one of the most common medicinal plant worldwide, which belongs to the  
49 Lamiaceae family with 25–30 species and about 10 hybrids; and the species and hybrids of *Mentha*  
50 genus are produced from both inter and intra-specific crosses, which make mint cultivars highly

51 heterozygous. For instance, peppermint (*M. piperita*) is a first-generation hybrid obtained from  
52 interspecific crossing of spearmint (*M. spicata*) and water mint (*M. aquatica*). In addition, *M. arvensis*,  
53 *M. rotundifolia*, *M. longifolia*, *M. pulegium*, *M. citrata*, *M. canadensis*, *M. suaveolens*, and *M. viridis*  
54 are other common species/hybrids of *Mentha* genus (Moetamedipoor et al. 2021). Traditionally, leaves,  
55 stems, and flowers of mint have been used in herbal teas or as an additive in foods to provide aroma  
56 and flavor. Today, however, there is an increasing demand in mint cultivation because of the commercial  
57 profit of its EO, which is among the 10 most used commercial EOs in the world (Chrysargyris et al.  
58 2017).

59 Several mint species/hybrids are well-known for their high EO yields, especially *M. piperita*, *M.*  
60 *arvensis*, and *M. spicata*, which is synthesized and stored in glandular trichomes on the adaxial surface  
61 of their leaves. Mint essential oil (MEO) is known as a multi-purpose ingredient in various industries  
62 and its market is projected to experience an annual growth rate of 3 to 5% (Soleimani et al. 2022). It  
63 contains diverse monoterpenes (the C<sub>10</sub> class of isoprenoids) in its structure, mainly C<sub>2</sub>-oxygenated  
64 monoterpenes (e.g., carvone), C<sub>3</sub>-oxygenated monoterpenes (e.g., menthol), and acyclic monoterpenes  
65 (e.g., linalool) (Soleimani et al. 2022). It has been used in many medications to treat oral mucositis,  
66 enteritis, gastritis, irritable bowel syndrome (IBS), and respiratory tract disorders since ancient times.  
67 In addition, it can be added to the formulation of various foods, drugs, healthcare items, and agro-  
68 biological products as a cooling, flavor enhancing, antioxidant, antimicrobial, and insecticidal agent  
69 (Hedayati et al. 2023). However, these multiple biological properties and health-related behaviors of  
70 MEO are highly influenced by various endogenous and exogenous factors. The plant organ, genotype  
71 (variation within different or same species), environment (e.g., climatic, topographic variables, and soil  
72 properties), plant phenology (developmental stage at the time of sampling), and extraction method are  
73 the most important factors that can affect the levels of *Mentha* EO (Salehi et al. 2018). Therefore, it is  
74 necessary to optimize each of the factors for the specific use of MEO in various food and non-food  
75 industries.

76 MEO can be isolated by various extraction techniques from several parts of mint plants such as stems,  
77 leaves, and flowers. These methods are generally divided into conventional (e.g., steam distillation

78 (SD), solvent extraction (SE), hydro-diffusion, and hydro-distillation) and emerging (e.g., ultrasound-  
79 assisted extraction (UAE), microwave-assisted extraction (MAE), ohmic-assisted extraction (OAE),  
80 pulsed electric field-assisted extraction (PEFAE), supercritical fluid extraction (SFE), and subcritical  
81 water extraction (SWE) extraction techniques. SD is the most common technique for the extraction of  
82 MEO. In spite of the simplicity of this method, it has a great downside, which is very time-consuming  
83 and requires a huge amount of energy. Most importantly, the high temperature and moisture during the  
84 process may change the flavor and reduce the overall quality of the MEO (Batoool et al. 2018). These  
85 disadvantages have attracted the attention of many researchers and stimulated the intensification,  
86 improvement, and optimization of “emerging” or “green” extraction techniques with higher efficiency,  
87 lower cost, and shorter extraction time. Therefore, the main aim of this study is to provide a  
88 comprehensive review of various conventional and emerging methods for the extraction of MEO and  
89 evaluate their advantages and disadvantages in order to meet the manufacturers and consumers’  
90 demands.

91

## 92 **An overview of MEO**

### 93 *Botanical features*

94 The genus *Mentha* belongs to the Lamiaceae (Labiatae) family, which consists of approximately 30  
95 species and 10 hybrids. It is believed that this genus originated from the Mediterranean basin, and from  
96 there, it spread to other parts of the world. Currently, the genus *Mentha* can be found in temperate and  
97 sub-temperate regions across North America, Africa, Europe, Asia, and Australia (Moetamedipoor et al.  
98 2021). The taxonomists have assigned various names to the mint plants over the past two centuries,  
99 indicating a significant morphological diversity. Also, it should be mentioned that the systematics of the  
100 genus *Mentha* are complex and still not fully understood, primarily due to variations in chromosome  
101 number, cytotoxicity, morphological polymorphism, frequent hybridization between different species,  
102 and propagation of colonial mutants, as well as the occurrence of polyploidy, aneuploidy, and  
103 nothomorphs (Gholamipourfard et al. 2021; Salehi et al. 2018).

104

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

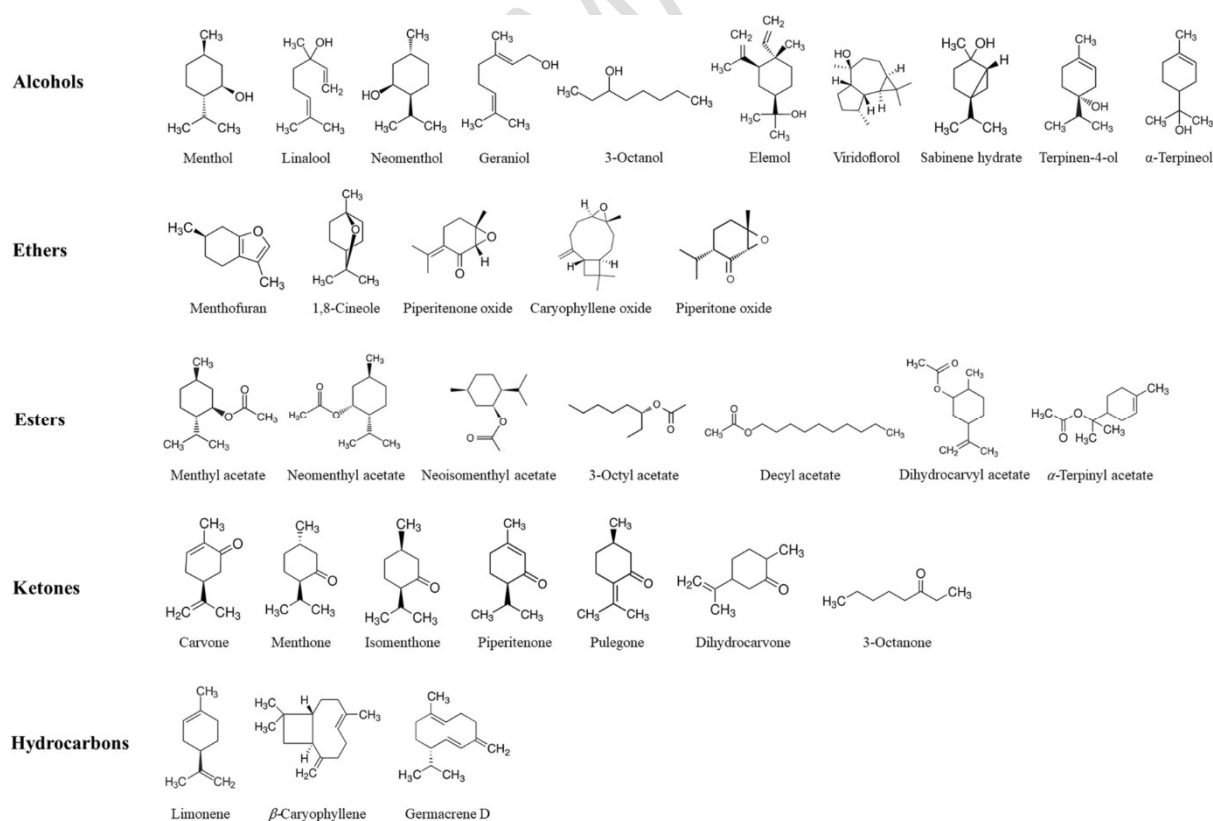
### 112 *Chemical composition*

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

122 The chemical compounds in MEO are susceptible to changes depending on the variety, climate, soil  
123 composition, agronomic conditions, and harvest time, as well as drying and extraction methods. As  
124 shown in Fig. 1, MEO contains a wide range of functional groups that are responsible for its biological  
125 characteristics, including alcohols (e.g., menthol, linalool, neomenthol, geraniol, 3-octanol, elemol,  
126 viridoflorol, cis/trans-sabinene hydrate, terpinen-4-ol, and  $\alpha$ -terpineol), ethers (e.g., menthofuran, 1,8-  
127 cineole, piperitenone oxide, caryophyllene oxide, and piperitone oxide), esters (e.g., menthyl acetate,  
128 neomenthyl acetate, neoisomenthyl acetate, 3-octyl acetate, decyl acetate, dihydrocarvyl acetate, and  $\alpha$ -  
129 terpinyl acetate), ketones (e.g., carvone, menthone, isomenthone, piperitenone, pulegone, and trans-

130 /cis-dihydrocarvone, 3-octanone), and hydrocarbons (e.g., limonene,  $\beta$ -caryophyllene, and germacrene  
 131 D) (Gholamipourfard et al. 2021; Salehi et al. 2018). Different species of *Mentha* can be categorized  
 132 into three groups based on their biosynthetic pathway and geographical conditions: those that produce  
 133 EOs rich in menthol, carvone, or pulegone/piperitone. For instance, the dementholized EO obtained  
 134 through gas chromatography-mass spectrometry (GC-MS) analysis reveals the presence of ten key  
 135 components in peppermint (*M. piperita* L.) EO, which includes menthol (30.0 to 55.0%), menthone  
 136 (14.0 to 32.0%), menthylacetate (2.8 to 10.0%), isomenthone (1.5 to 10.0%), menthofuran (1.0 to 9.0%),  
 137 limonene (1.0 to 5.0%), 1,8-cineole (3.5 to 4.0%), pulegone (max. 4.0%), isopulegol (max. 0.2%), and  
 138 carvone (max. 1.0%) On the other hand, the cornmint (*M. arvensis* L.) EO consists of nine primary  
 139 compounds, which are menthol (30 to 50%), menthone (17 to 35%), isomenthone (5.0 to 13.0%),  
 140 limonene (1.5 to 7.0%), menthyl acetate (1.5 to 7.0%), isopulegol (1 to 3%), pulegone (max. 2.0%),  
 141 carvone (max. 2.0%), and 1,8-cineole (max. 1.5%) (Kumar et al. 2011; Soleimani et al. 2022).

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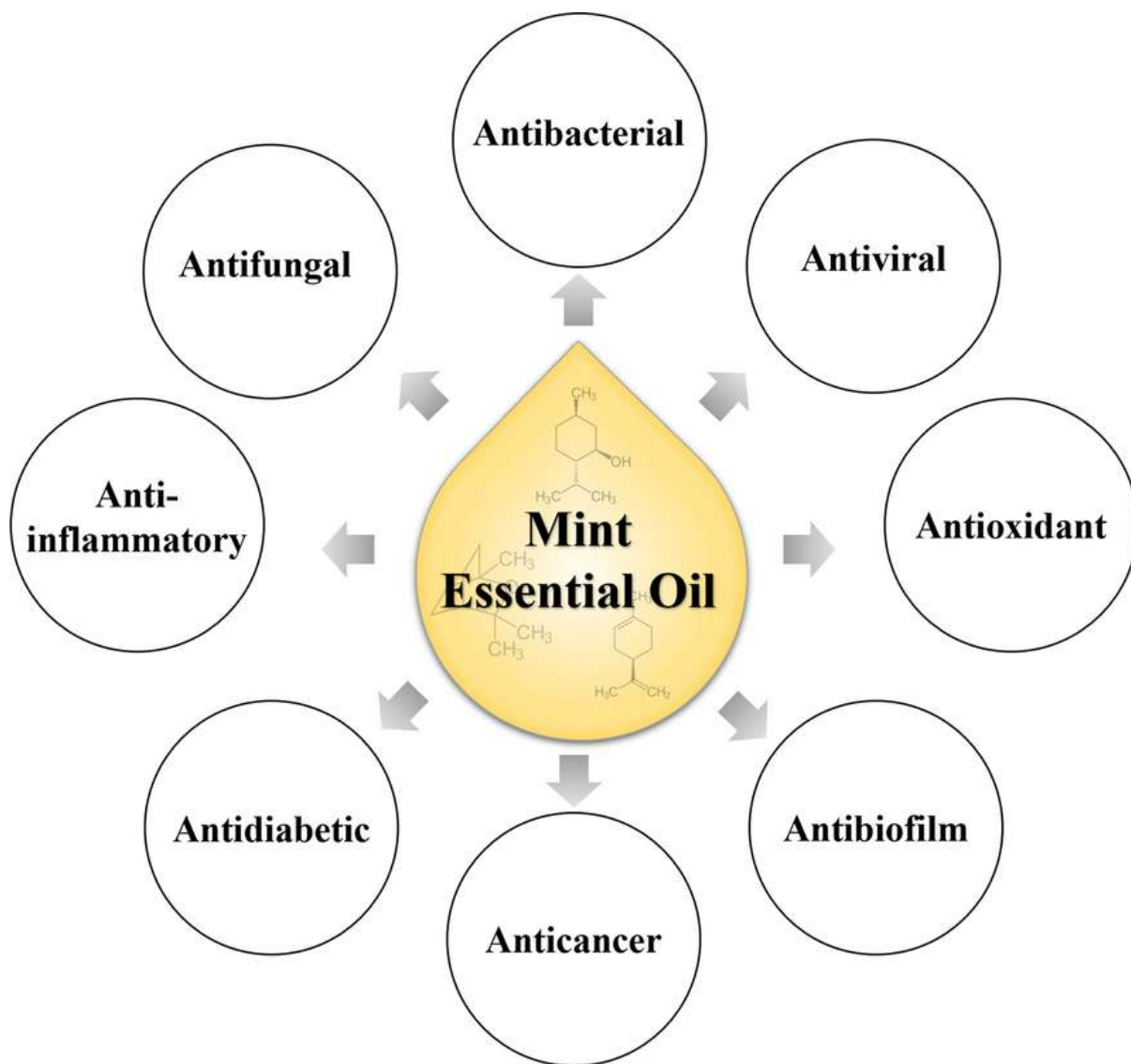
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**Fig. 1.** Common functional groups in mint essential oil (MEO)

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

#### 150 *Biological properties*

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



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

167

168

169 There has been a growing interest in the utilization of EOs as a natural antioxidant in recent years. It is  
 170 widely recognized that reactive oxygen species (ROS) cause damage upon cellular macromolecules  
 171 (e.g., proteins, lipids, and nucleic acids) and are implicated in the development of numerous human  
 172 diseases (e.g., cancer, cardiovascular diseases, metabolic disorders, diabetes, etc.). In various  
 173 pathological circumstances, the imbalance in oxidation-reduction (redox) potential fails to eliminate  
 174 excessive quantities of ROS. Consequently, oxidant species like superoxide ( $O_2^-$ ) and hydrogen  
 175 peroxide ( $H_2O_2$ ) are generated as a result of the pathogen phagocytosis by these cells, serving as a  
 176 defense mechanism against harmful insults. To counteract this response, antioxidant pathways are  
 177 activated to protect against ROS-induced cellular damage (Pizzino et al. 2017). In previous studies,

178 MEO has shown significant in vitro and in vivo antioxidant activities. For instance, Wu et al. (2019)  
179 evaluated the antioxidant activity of EOs obtained from three mint species, including native spearmint  
180 (*M. spicata*), Scotch spearmint (*M. gracilis*), and peppermint (*M. piperita*). All of these mint-derived  
181 EOs showed significant antioxidant activity in chemical-based assays. However, *M. piperita* EO showed  
182 higher antioxidant capacity compared to *M. spicata* and *M. gracilis* EOs. Furthermore, the maximal  
183 cellular antioxidant activity (CAA) was observed at a much lower dose for *M. piperita* EO (5 µg/mL)  
184 than for native or Scotch mint EOs (100 µg/mL). Alsaraf et al. (2021), Aimad et al. (2021), and Almeida  
185 et al. (2022) have also reported the effective antioxidant activity of various MEOs cultivated in Oman,  
186 Morocco, and Brazil, respectively.

187 In recent studies, the anticarcinogenic effects of MEO against prostate, colon, bladder, cervical,  
188 leukemic, skin, liver, and lung cancers have been comprehensively reviewed by Zhao et al. (2023) and  
189 Kazemi et al. (2023). In addition, the anti-inflammatory (Hejna et al. 2021), antidiabetic (Gülçin et al.  
190 2022), and antibiofilm (Kang et al. 2019) activities of MEO have been also investigated. Moreover,  
191 previous studies showed the insecticidal properties of MEO against several insects, such as *Tribolium*  
192 *castaneum*, *Toxoptera aurantii*, *Mayetiola destructor*, *Sitophilus oryzae*, *Sitophilus zeamais*, *Sitophilus*  
193 *granarius*, and *Rhyzopertha dominica* (Eftekhari et al. 2021). MEO is also known for its analgesic,  
194 antispasmodic, antipruritic, antidepressant, and anti-anxiety effects, which highlights the potential  
195 application of MEO as a natural therapeutic agent for various health conditions (Kazemi et al. 2023).

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### 197 **Extraction of MEO**

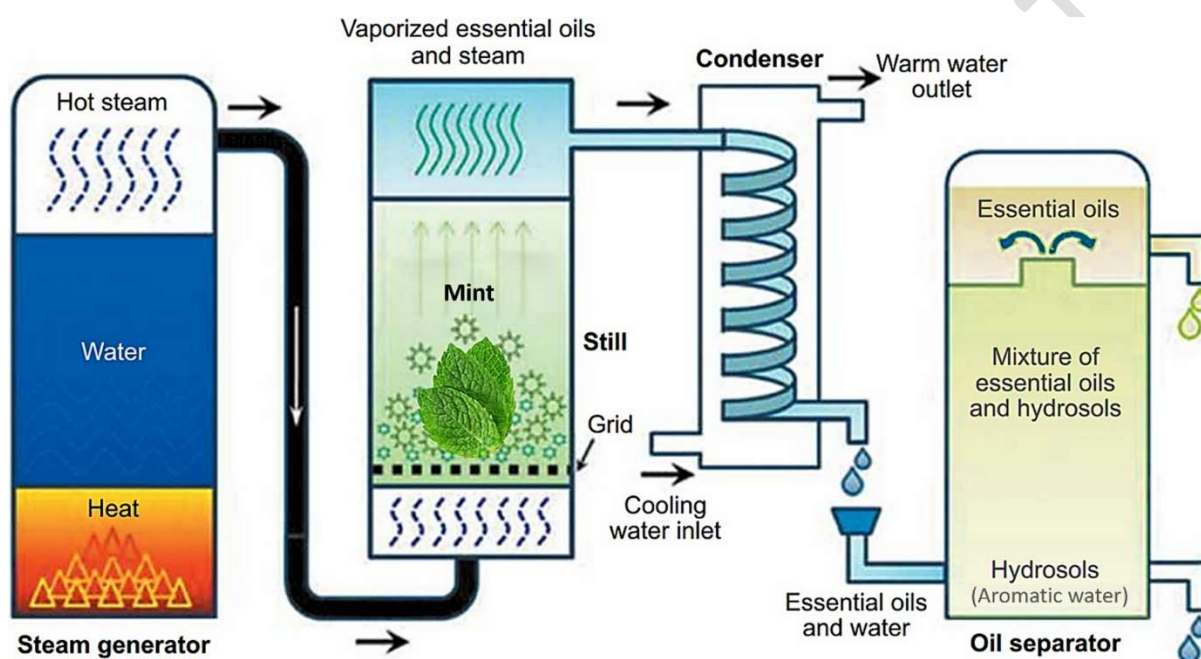
198 The extraction methods and conditions have significant effects on the production yield, bioactivity,  
199 and physicochemical properties of MEO. Inappropriate extraction may destroy the phytochemicals  
200 and result in loss of pharmacological and flavor compounds, and physical changes to MEO.

201 Generally, the extraction methods are divided into two groups: conventional or classical methods and  
202 emerging or green methods, each method has its advantages and disadvantages which are discussed in  
203 this section.

204 *Conventional methods*

205 The conventional methods for the extraction of EOs include hydro-diffusion, solvent extraction (SE),  
206 hydro-distillation, and steam distillation (SD). Hydro-diffusion is a classical extraction method that is  
207 only used for dried plants, which may be damaged at boiling temperature. In this method, steam is  
208 applied from the top of a generator to the container that holds the dried plants. Hydro-diffusion is  
209 performed under vacuum or at low pressure and the temperature of steam is below 100 °C (Kant and  
210 Kumar 2022). On the other hand, SE is used to extract EOs from delicate plant materials, such as  
211 flowers, which are sensitive and cannot be subjected to heat or steam. Different solvents, such as  
212 ethanol, methanol, hexane, acetone, and petroleum ether are added to the plant materials and mildly  
213 heated, subsequently the mixture is filtered and the solvent is evaporated (Tongnuanchan and Benjakul  
214 2014). The filtrate is a mixture of EO, wax, resin, and fragrance; to purify the EOs, alcohol is added to  
215 the filtrate and then distilled at low temperatures. Alcohol absorbs fragrance and then evaporated  
216 whereas the EOs remain in the flask residue. SE is more complicated, expensive, and time-consuming,  
217 than other EOs extraction methods, therefore it is not widely used for the extraction of EOs (Li et al.  
218 2009). In addition, hydro-distillation, attributed to the discovery by Avicenna, stands as an ancient and  
219 most straightforward extraction methods (Khan et al. 2023). Hydro-distillation is a commonly used  
220 method to extract EOs from plants. It can be carried out using two main methods: The Clevenger method  
221 and simple SD. In the Clevenger method, a mixture of the plant material and water is heated and causes  
222 the EOs to evaporate. The steam and EOs are then cooled and condensed back into a liquid. The EOs  
223 are then separated from the water using a separating funnel. In simple SD, steam is passed through a  
224 bed of the plant material and carries the EOs. The steam and EOs are then cooled and condensed back  
225 into a liquid. The EOs are then separated from the water using a separating funnel. Both methods  
226 produce two layers: a water layer and an oil (EOs) layer (Ashraf et al. 2020). A typical SD system  
227 consists of essential components, such as a steam generator, extraction unit, connecting pipes,  
228 condenser, and oil separator (Fig. 3). The heater acts as the power source, heating water within the boiler  
229 to produce the necessary steam for extraction. The cylindrical extraction unit accommodates batches of  
230 plants with a mesh positioned at a specific height above its base. It features two openings: one at the

231 bottom for delivering boiler-generated steam and another at the top for conveying steam laden with  
 232 extracted volatile oil to the condenser. The condenser, comprising two tubes with a continuous water  
 233 flow system, efficiently cools and condenses the steam. Connected to the extraction unit, it receives  
 234 both steam and oil. The collected volatile oil is funneled and gathered. Approximately, 93% of EOs can  
 235 be extracted by SD and the remaining 7% can be isolated by other approaches (Babu and Kaul 2005;  
 236 Masango 2005).



237  
 238 **Fig. 3.** Schematic diagram of steam distillation (SD) system for mint essential oil (MEO) extraction;  
 239 adapted from Machado et al. (2021)

240 In 2020, Radwan et al. (2020) conducted a study to establish a straightforward SD system for  
 241 peppermint volatile oil extraction. Their experiments, involving varying boiler inlet water flow rates  
 242 and different batch sizes of peppermint plants, revealed that with a 1.5 l/h boiler inlet water flow rate  
 243 and a 700 g batch size, the system achieved optimal conditions, yielding maximum productivity, an  
 244 88.6% extraction efficiency, and an energy requirement of 0.48 kWh/ml EO. In another study, the  
 245 chemical composition of *Mentha aquatica* L. EOs was investigated by Andro et al. (2013) at various  
 246 stages of its ontogenetic cycle. The primary objective of their research was to scrutinize the chemical  
 247 constituents present in these EOs. Aerial parts of the *Mentha aquatica* L. plant were systematically

248 collected during three distinct phases: the vegetative, flowering, and senescence. The EOs were  
249 meticulously extracted using the hydro-distillation method, employing a Neo-Clevenger device.  
250 Subsequently, the acquired EO samples underwent a rigorous analysis through Agilent GC-MS. The  
251 quality of the EOs exhibited variations contingent on the specific timing of the investigations. In total,  
252 a notable forty-one different substances were successfully identified, with a noteworthy twenty of them  
253 appearing consistently across all samples gathered during the three distinct phenophases. Among the  
254 primary substances identified were menthofuran, limonene, trans- $\beta$ -ocimene, ledol, and  $\beta$ -  
255 caryophyllene. The extraction process for obtaining these EOs involved hydro-distillation employing  
256 water vapors and a Neo-Clevenger type device. The method entailed the placement of crushed plant  
257 material into the device's flask, followed by the addition of 500 ml of water, which was then adapted  
258 for SD. The distillation procedure unfolded over a span of three to four hours, maintaining a moderate  
259 temperature throughout.

260 Furthermore, Beigi et al. (2018) investigated the composition and amount of EO extracted by hydro-  
261 distillation from the leaves of peppermint (*Mentha piperita* L.), employing various drying methods. The  
262 research aimed to provide a comprehensive analysis of the EO from peppermint leaves under different  
263 drying conditions. Thin-layer drying experiments were conducted using methods such as shade drying,  
264 hot air drying, and microwave drying. EOs were subsequently extracted from both fresh and dried  
265 samples using hydro-distillation, with GC-MS analysis revealing valuable insights. The study found  
266 that the highest oil yield (22.2 g/kg dry matter) was obtained from leaves dried using hot air at 50 °C,  
267 while the lowest yield (1.3 g/kg dry matter) occurred with microwave drying at its highest power.  
268 Drying temperature inversely correlated with EO content. Major compounds in the EO included  
269 menthol, menthone, menthofuran, 1,8-cineole, and menthyl acetate, with their concentrations varying  
270 significantly across different drying methods. For oil extraction, both dried and fresh leaves underwent  
271 hydro-distillation in a Clevenger-type apparatus for 3 h. In another study, EO components of Egyptian  
272 *Mentha pulegium* L. were extracted through hydro-distillation and analyzed via GC-MS. Pulegone was  
273 the predominant compound, classifying the oil as a pulegone chemotype (43.5% pulegone content). The  
274 oil displayed antioxidant properties in both hexanal/hexanoic acid and DPPH assays, particularly in

275 dichloromethane extracts. To obtain the oil, dried leaves were subjected to SD for 4 h, followed by 6-  
276 hour extraction with dichloromethane. Non-volatile compounds were isolated by successive extracting  
277 using methanol and dichloromethane. The study highlights *Mentha pulegium* L. as a potential source  
278 for cost-effective flavorings and antioxidants, valuable for preventing oxidation in various food  
279 products (El-Ghorab 2006).

280 To determine the duration and best timing for extraction of EO by hydro-distillation from *Mentha*  
281 *suaveolens* L., Garzoli et al. (2015) used a multidisciplinary approach. Their study involved a thorough  
282 examination of EO extraction, particularly focusing on the influence of harvesting time and extraction  
283 duration. The analysis, performed via GC-MS, identified piperitenone oxide as a key component  
284 contributing to the oil's antifungal activity. Notably, EOs with lower piperitenone oxide content still  
285 exhibited antifungal properties, possibly due to compounds like para-cymenene. Importantly, a bacterial  
286 reverse mutation assay indicated the safety of these EOs. The findings provided specific  
287 recommendations: for enhanced antifungal activity, limit hydro-distillation to a maximum of 3 h,  
288 regardless of the harvest time, while for maximum oil yield, harvest in August. July was identified as  
289 the period with the highest piperitenone oxide content. This multidisciplinary approach sheds light on  
290 optimizing EO production from *Mentha suaveolens* L., considering both chemical composition and  
291 biological activity. Another study focused on predicting the EO yield extracted by hydro-distillation  
292 from *Mentha arvensis* L., also known as corn mint, which is extensively used in flavor, fragrance, and  
293 pharmaceutical industries. The study developed a predictive model based on solid diffusion principles,  
294 particularly focusing on the geometry of flake particles. The model's accuracy was validated by  
295 comparing it with experimental data on oil yield over time. This experimental data was collected from  
296 both bench-scale and pilot-scale distillation setups, providing a comprehensive assessment of the  
297 model's performance. This research enhances our understanding and ability to predict EO yields during  
298 the hydro-distillation process for corn mint, benefiting various industries reliant on these EOs (Katiyar  
299 2017).

300 Emerging technologies

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

### 308 *Supercritical fluid extraction (SFE)*

309 Supercritical fluid extraction (SFE) stands out as an eco-friendly and efficient method for extracting  
310 EOs from plants and herbs. Using supercritical CO<sub>2</sub>, known for its selectivity and absence of harmful  
311 solvents, ensures high-quality extracts in moderate timeframes. This process is widely used to isolate  
312 EOs, involving pure CO<sub>2</sub> or a co-solvent. Fractionation is a common step to separate volatile oil  
313 compounds from other extracted substances. Parameters like pressure, temperature, and co-solvent  
314 presence impact the extraction and fractionation process. Compared to traditional methods like hydro-  
315 distillation, SFE often yields superior-quality extracts (Fornari et al. 2012).

316 SFE is emerging as a highly promising technique for isolating volatile compounds from natural sources,  
317 challenging traditional methods like SE, SD, and hydro-distillation. SFE's selectivity can be tailored by  
318 factors such as solvent choice, temperature, pressure, extraction times, flow rate, and modifiers. The  
319 solvent's strength in supercritical fluids correlates with its density, primarily regulated by adjusting  
320 extraction pressure. The low viscosity and high diffusibility of solutes in supercritical fluids promote  
321 efficient mass transfer during extraction. A study on *Mentha pulegium* L. EO composition, extracted  
322 using supercritical carbon dioxide, examined various parameters like pressure, temperature, extraction  
323 time, and modifier usage. Results showed that optimal SFE conditions considerably yielded around  
324 30% menthone and 52% pulegone, while hydro-distillation favored 38% pulegone and 20% menthone.  
325 This study showed SFE's potential and efficiency in extracting volatile compounds while offering  
326 selectivity adjustments through parameter control (Aghel et al. 2004). In addition, a comparative study  
327 was conducted to assess the efficiency and quality of EO extraction from spearmint (*Mentha spicata* L.)

328 using both SFE and SD methods. The primary goals were to optimize SFE conditions for producing  
329 high-quality MEO and to evaluate the impact of different drying methods (oven drying and freeze  
330 drying) on EO quality. Additionally, the study compared the flavor compounds in the SFE-extracted  
331 MEO with those obtained through traditional SD. The research identified the highest extraction yield  
332 under specific SFE conditions, notably at 50 °C and 350 bar. However, for superior MEO quality, the  
333 optimal SFE parameters were 30 °C and 150 bar. Moreover, the composition of the EO obtained via  
334 SFE outperformed that from SD. The study also provided insights into extraction yields and EO quality  
335 from spearmint samples collected in various regions, contributing valuable information on EO  
336 extraction efficiency and quality, particularly for spearmint (Al-Marzouqi et al. 2007). In a recent study,  
337 MEO was obtained through diverse extraction methods, including sub-/supercritical extraction with or  
338 without a modifier, Soxhlet extraction, and hydro-distillation using various solvents. The results  
339 demonstrated that the highest MEO yield over 2%, which was achieved with SFE at 300 bar and 50 °C,  
340 with a yield isotherm crossover occurring between 140 and 170 bar. When a co-solvent was employed  
341 in SFE, ethanol outperformed ethyl acetate in terms of oil yield. The extracted MEO was rich in  
342 compounds with therapeutic and industrial significance, such as pulegone, cineol, and carvone, and  
343 exhibited commendable antioxidant activity, enhancing its suitability for various applications. This  
344 research offers valuable insights into MEO extraction, considering different methods and operational  
345 conditions (Almeida et al. 2012).

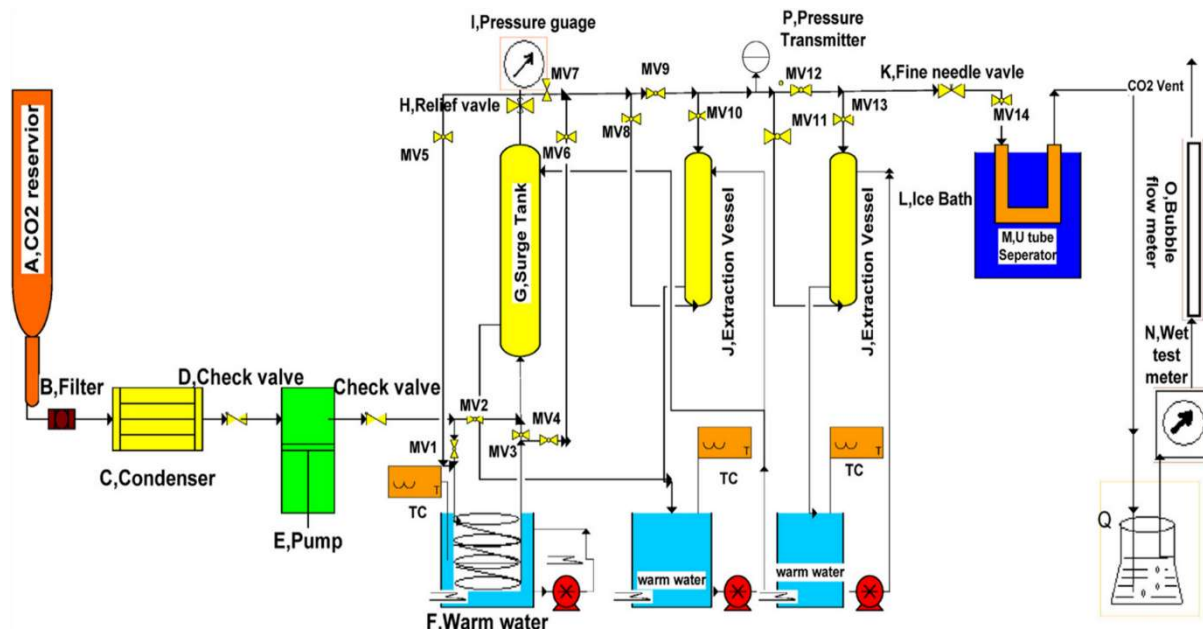
346 A study investigated the chemical composition and biological properties of *Mentha longifolia* L. EO  
347 employing different extraction techniques. In this regards, three different methods, including  
348 supercritical CO<sub>2</sub> fluid extraction, lipophilic SE (n-hexane), and SD were compared. Lipophilic SE  
349 yielded the highest EO content at 1.2% (w/w). Gas chromatography-flame ionization detector (GC-  
350 FID) and GC-MS analyses identified a total of 39 compounds in the extracted EOs. Major components  
351 in EOs from SD and lipophilic SE included carvone, limonene, trans-caryophyllene, and  $\alpha$ -terpineol. In  
352 contrast, the supercritical CO<sub>2</sub>-extracted oil contained germacrene D, trans- $\beta$ -farnesene, trans-  
353 caryophyllene, and carvone, as major constituents. All EOs exhibited varying antioxidant activities,  
354 with the supercritical CO<sub>2</sub>-extracted oil demonstrating the highest scavenging activity in both DPPH

355 and ABTS assays. This study sheds light on the chemical composition and antioxidant potential of  
356 *Mentha longifolia* L. EOs obtained through different methods, emphasizing the superior antioxidant  
357 properties of supercritical CO<sub>2</sub> extraction (Bai et al. 2020). Besides, in a study focusing on supercritical  
358 CO<sub>2</sub> extraction of MEO, various important aspects were explored. The research involved the  
359 preparation of dried leaves through milling and sieving to obtain particles of different sizes. These  
360 particles were then subjected to supercritical CO<sub>2</sub> treatment under specific conditions (40 °C and 10  
361 MPa) for MEO extraction. An innovative aspect of this study was the introduction of a novel modeling  
362 approach called the “pseudo kinetic model”. This model was proposed to better describe and predict the  
363 extraction process and its kinetics. The study compared the performance of the pseudo kinetic model  
364 with Sovova’s model, a widely used model in supercritical CO<sub>2</sub> extraction literature. The primary goal  
365 was to assess how particle size influences extraction yield and establish an optimal methodology for  
366 determining the parameters of the pseudo kinetic model, potentially providing a more accurate  
367 representation of the extraction process. Results showed both the pseudo kinetic model and  
368 Sovova’s model effectively described the supercritical CO<sub>2</sub> extraction process, suggesting the potential  
369 utility of the pseudo kinetic model for understanding and predicting extraction dynamics. Overall, this  
370 study advances our understanding of how particle size affects extraction yields and introduces an  
371 innovative modeling approach that could enhance the efficiency and accuracy of supercritical CO<sub>2</sub>  
372 extraction processes (Maksimovic et al. 2012).

373 In another study conducted on SFE by Pop and Barth (2001), the focus was on Romanian *Mentha*  
374 hybrids as the source of EOs high in *Z*-sabinene hydrate. The aim of the research was to extract the  
375 volatile components, particularly *Z*-sabinene hydrate and its acetate, from a mint hybrid found in  
376 Romania. This was achieved using supercritical CO<sub>2</sub> extraction. The study determined that the highest  
377 yield, amounting over 2%, was achieved within a pressure range of approximately 93 bar and at a  
378 temperature of 50 °C. Additionally, a kinetic study was carried out to evaluate the supercritical CO<sub>2</sub>  
379 extraction process, and the results were compared to those obtained through hydro-distillation. The  
380 monitoring of the extraction process was conducted using and GC-MS analysis. The research  
381 established that the optimal conditions for supercritical CO<sub>2</sub> extraction resulted in extracts with a high

382 content of Z-sabinene hydrate and its acetate, while still retaining a flavor profile similar to the original  
383 plant material. These optimal conditions were identified as a pressure range around 95 bar and a  
384 temperature of 50 °C. The study also highlighted an interesting finding regarding hydro-distillation: it  
385 was noted that only at the beginning of the process was it possible to obtain a high content of Z-sabinene  
386 hydrate. However, achieving a high content of its acetate was found to be challenging through this  
387 method. In conclusion, this study not only provides an effective method for obtaining Z-sabinene  
388 hydrate from a natural source but also emphasizes the importance of using mild extraction conditions  
389 to preserve valuable natural compounds. This underscores the potential of supercritical CO<sub>2</sub> extraction  
390 as a valuable tool in this context. In a separate study, EO extraction from spearmint leaves was  
391 conducted using supercritical CO<sub>2</sub> extraction (Fig. 4). The primary aim of this research was to explore  
392 how various operational parameters affect the efficiency of EO extraction from spearmint leaves. The  
393 study encompassed a series of well-planned extraction experiments to assess the influence of different  
394 variables on the extraction process. These variables included dynamic extraction time (i.e., spanning  
395 from 20 to 120 min), mean particle size (i.e., ranging from 0.18 to 2 mm), CO<sub>2</sub> flow rate (i.e., varying  
396 from 0.06 to 0.35 g/min), extraction temperature (i.e., ranging from 38 to 50 °C), and extraction pressure  
397 (i.e., ranging from 85 to 120 bar). In the concluding phase of the investigation, the researchers developed  
398 a second-order polynomial equation to pinpoint the optimal parameters for supercritical CO<sub>2</sub> extraction  
399 of EOs from spearmint leaves. Additionally, the composition of the EO extracted under these optimal  
400 conditions was analyzed using GC-MS. This research contributes valuable insights into optimizing the  
401 parameters of supercritical CO<sub>2</sub> extraction for spearmint leaf EOs, shedding light on the composition  
402 of the extracted oil under optimal conditions, which holds significance for various applications and  
403 further research endeavors in this domain (Shahsavarpour et al. 2017). In a further study, Shrigod et al.  
404 (2017) investigated SFE of MEO to optimize EO yield and carvone content. Using a central composite  
405 rotatable design, they explored temperature (i.e., 35–55 °C), pressure (i.e., 100–300 bar), dynamic time  
406 (i.e., 20–90 min), and particle size (i.e., 0.2–1 mm) as key parameters. Particle size had the most  
407 significant impact on oil yield, followed by pressure, temperature, and dynamic time. For carvone  
408 content, pressure was most influential, followed by particle size and dynamic time. The study  
409 determined optimal conditions as follows: 48 °C temperature, 151 bar pressure, 0.4 mm particle size,

410 and 37.5 min dynamic time, resulting in 1.4% oil yield and 998 mg/100 g carvone content. The oil  
 411 showed potent antimicrobial activity, particularly against E. coli. These findings offer valuable insights  
 412 for MEO extraction using supercritical CO<sub>2</sub>.



413  
 414 Fig. 4. Schematic diagram of the extraction of mint essential oil (MEO) with supercritical carbon  
 415 dioxide (CO<sub>2</sub>); adapted from Shamsavarpour et al. (2017)

416 *Subcritical water extraction (SWE)*

417 Subcritical/superheated water extraction (SWE), also known as pressurized low-polarity water  
 418 extraction (PLPWE), or pressurized hot-water extraction (PHWE) is an emerging and green technology  
 419 for the extraction of bioactive compounds. The subcritical state of water refers to the liquid water at  
 420 temperatures between 100 and 374.15 °C and pressures below the critical pressure ( $P_c = 22.1$  MPa) and  
 421 higher than the vapor pressure. Under these extreme conditions, the polarity of water is much lower  
 422 than at room temperature. Therefore, the organic compounds are more soluble and the extraction process  
 423 can be accomplished in a shorter time. The subcritical state of fluid offers several superior  
 424 characteristics, such as lower viscosity, density, and surface tension, as well as enhanced diffusivity  
 425 between gas and liquids. The use of subcritical state of fluids has been reported by many researchers  
 426 and found that this is a better and powerful alternative of EOs extraction technique (Özel et al. 2006).

427 This extraction technique is considered the best alternative approach as it enables a fast EO isolation  
428 process. Moreover, it is a cost-efficient extraction, simple and environmentally friendly process due to  
429 the application of non-toxic and non-flammable solvents. EOs with more valuable properties and higher  
430 amount of oxygenated components with no significant presence of terpenes can be obtained and allow  
431 substantial cost saving in terms of both energy and plant materials (Tongnuanchan and Benjakul 2014).

432 In this respect, Chiou et al. (2019) used subcritical water with pressure-releasing distillation to extract  
433 EOs and volatile fractions from Japanese mint (*Mentha arvensis* L. var. *piperascens* Malinv. cv.) and  
434 compared the results with the EOs obtained by SD. The process was performed at 100–220 °C, during  
435 5–60 min. GC was used to quantitatively analyze the extracted compounds. The results revealed that  
436 increasing the extraction temperature from 100 to 180 °C, increased the yields of l-menthol, l-menthone,  
437 l-limonene, and piperitone, but at higher temperatures the extraction yield was decreased. On the other  
438 hand, the yields of l-menthyl acetate, iso-menthone, and 3-octanol increased up to 220 °C. Extending  
439 the extraction time, negatively affected the extraction of MEO compounds and the highest yield was  
440 obtained after 5 min treatment at 180 °C. While, the extraction by team distillation required more than  
441 1 h to reach a similar yield. In a similar study, Ammann et al. (1999) compared the effects of SWE,  
442 SFE, and SD on the extraction of peppermint (*Mentha piperita* L.) EOs. The extraction yield and  
443 composition of MEO isolated by SD were more appropriate than other EOs. However, the extraction  
444 by SWE was faster than other methods and the extraction efficiency was higher than SFE. During the  
445 first few minutes, the speed of all extraction methods was comparable. However, shortly thereafter the  
446 SFE speed decreased and it presented the lowest yield. They stated that SFE takes 3–10 times longer  
447 than SD to achieve a comparable yield and is the least suitable method for MEO extraction. In another  
448 study, Kubátová et al. (2001) compared the extraction yields of oxygenated and non-oxygenated  
449 compounds in peppermint by SWE, SFE, and hydro-distillation. The results indicated that, increasing  
450 the temperature of SWE from 100 to 175 °C increased the extraction rate; nevertheless, some of the  
451 desired components such as terpinene and linalool exhibited considerable degradation at temperatures  
452 above 150°C. Conversely, components such as thymoquinone were not degraded in SWE as the  
453 extracted flavor compound was not exposed to atmospheric oxygen. The amounts of menthol,

454 menthone, carvone, piperitone, pulegone, eucalyptol, and neomenthol obtained from peppermint after  
455 4 h of hydro-distillation were comparable to those extracted by SWE processing for 30 min at 150 °C  
456 or 12 min at 175 °C and SFE for 1 h. However, the recovery of less polar compounds such as menthyl  
457 acetate in SWE was only 40% of hydro-distillation. Therefore, SWE selectively extracts the flavor  
458 compounds and can extract more polar (oxygenated) compounds in a short time (as little as 12 min). In  
459 contrast, SFE and hydro-distillation do not act selectively and the extracted samples contain flavor  
460 compounds as well as plant alkane waxes. In a further study, Çam et al. (2019) optimized the extraction  
461 conditions of MEO by SWE. They investigated the effects of different extraction times (i.e., 1, 5, 10, or  
462 30 min), temperatures (i.e., 40, 70, 100, 130, or 160 °C), and extraction cycles (1–5) on the composition  
463 and extraction yield of MEO. The results revealed that the best extraction conditions were as follows:  
464 temperature of 130 °C and extraction time of 10 min after 3 extraction cycles. They reported that the  
465 MEO content of samples obtained at 130 and 160 °C did not show statistically significant differences.  
466 The dominant component of MEO was determined as menthol by GC-FID. The extraction yield under  
467 optimized conditions was approximately 2-fold higher than the conventionally extracted sample.

#### 468 *Microwave-assisted extraction (MAE)*

469 Microwaves (MW) are electromagnetic waves between infrared (IR) and radio frequency (RF) waves  
470 with frequencies ranging from 300 MHz to 300 GHz. MW processing is a heat treatment that plays an  
471 important role in the safety and quality of food products. It is used in many fields of the food industry,  
472 such as drying, preheating, cooking, thawing, tempering, blanching, roasting, pasteurization,  
473 sterilization, enzyme inhibition and inactivation, microbial inactivation, and extraction (Hashemi et al.  
474 2019). In this method, heat is generated inside the food matrix based on two mechanisms: (1)  
475 electrophoretic transport of electrons and ions, which creates an electric field responsible for particle  
476 movements, and (2) dipole rotation, which is caused by the alternating movement of polar molecules.  
477 These two phenomena promote the release of energy, resulting in rapid heating, which accelerates tissue  
478 damage and increases the diffusion of targeted compounds (Marić et al. 2018). The increasing demand  
479 for environmental protection has prioritized research efforts to expand the use of MW technology,  
480 especially in practices that require purification and extraction.

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

493 Numerous studies have demonstrated that MAE process requires shorter time, lower energy, and solvent  
494 consumption compared to conventional extraction methods. Also, the enhanced extraction kinetics  
495 increase the extraction yield of MAE (Ripoll and Hincapié-Llanos 2022). Consequently, MAE is an  
496 acceptable and feasible technique for the extraction of EOs from aromatic plants such as mint and  
497 several researchers have investigated the MAE of MEO (Table 1). In this regard, Abdel-Hameed et al.  
498 (2018) evaluated the efficiency of different extraction techniques, such as hydro-distillation, MWAHD,  
499 and SFMWE to isolate EOs from *Mentha piperita* L.; and also, investigated the antimicrobial and  
500 anticancer properties of EOs obtained by these extraction approaches. The chemical composition and  
501 the extraction yield of EOs obtained by different extraction methods showed significant differences.  
502 However, no significant differences were observed among the MEO yields extracted via the three  
503 different methods. In addition, the methods based on MW were more energy-saving compared to  
504 conventional hydro-distillation methods. The chemical properties of EOs were qualitatively similar to  
505 each other; however, they were not much quantitatively different. Also, all of the MEO samples showed  
506 moderate anticancer and high antimicrobial activities in vitro (Abdel-Hameed et al. 2018). Moreover,  
507 Wang et al. (2006) used SFMWE and MWAHD to extract MEO with no solvents or pre-treatments. The

508 SFMWE method was accomplished in 30 min, while MWAHD took 90 min and conventional hydro-  
 509 distillation was completed in 180 min. The EOs extracted by different techniques had similar chemical  
 510 compositions. Nonetheless, the SFMWE method was more feasible for the extraction of MEO. In  
 511 another study, Figueredo et al. (2012) reported similar results for *Mentha spicata* L. EOs extracted by  
 512 MWAHD or hydro-distillation methods and stated that the major components of MEO were consistently  
 513 present, regardless of the extraction technique (Fig. 5). These findings also confirmed the results of a  
 514 previous research by Kohari et al. (2020) who reported that the composition of on Japanese peppermint  
 515 (*Mentha arvensis* L.) extracted via SFMWA and hydro-distillation were similar to each other. In a further  
 516 study, Shankar and Natarajan (2022) led relevant research on the extraction of *Mentha spicata* L. EO.  
 517 The oil yield, chemical composition, and antioxidant activity of EOs extracted by MWAD and hydro-  
 518 distillation methods were analyzed. The MWAD method resulted in the maximum oil yield because of  
 519 its efficient heat-transfer capacity. The qualitative differences between the EOs extracted via MWAD  
 520 and hydro-distillation were identified in the chemical profile of the EOs when analyzed by GC-MS. The  
 521 DPPH assay showed that the EOs extracted by MAE presented better radical scavenging activity and  
 522 greater antioxidant activity, compared to the hydro-distillation.

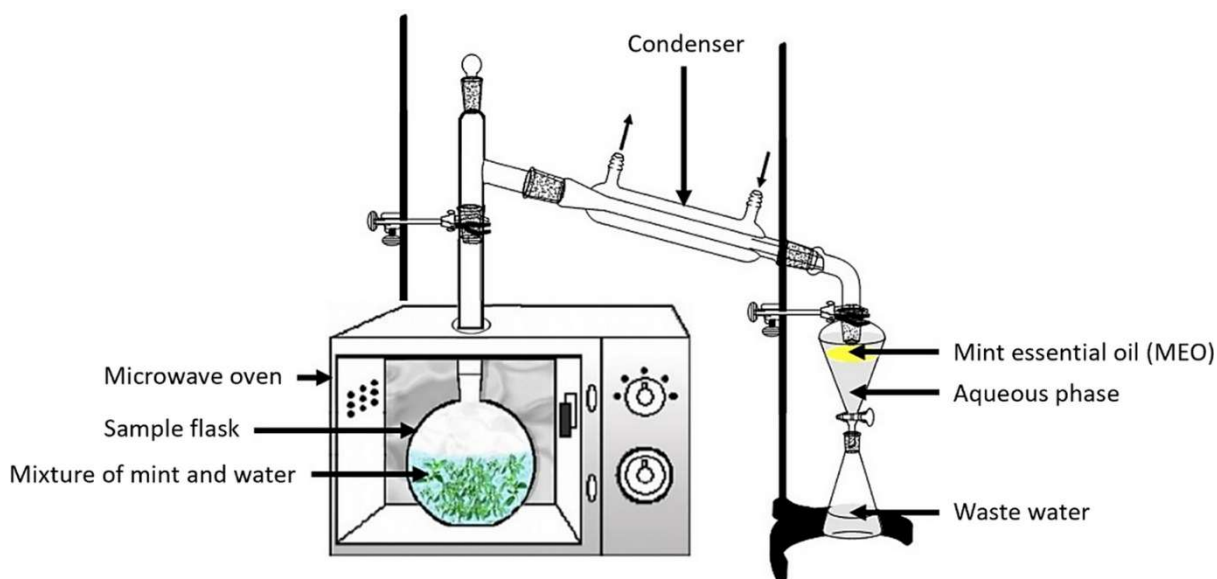
523 **Table 1.** Effects of species, cultivation area, and microwave-assisted extraction conditions on the  
 524 yield and main component of MEO

Species	Cultivation area	Extraction conditions			MEO yield (%)	Main component	Reference
		Power (W)	Time (min)	Solid: liquid ratio			
<i>Mentha piperita</i> L.	Taif, Saudi Arabia	500	40	1:3	0.36	Carvone	Abdel-Hameed et al. (2018)
<i>Mentha longifolia</i> L.	Taif, Saudi Arabia	500	40	1:3	0.60	Pulegone	Abdel-Hameed et al. (2018)
<i>Mentha arvensis</i> L.	Lam Dong, Vietnam	540	20	1:2	0.8	Menthol	Bui-Phuc et al. (2020)

<i>Mentha spicata</i> L.	Konya, Turkey	800	10	2:1	ND	Carvone	Figueredo et al. (2012)
<i>Mentha rotundifolia</i> L.	Zaragoza, Spain	900	5	1.25:10	1.6	Piperitenone oxide	García-Sarrió et al. (2018)
<i>Mentha arvensis</i> L.	Hokkaido, Japan	150	60	1:10	0.93	l-Menthol	Kohari et al. (2020)
<i>Mentha piperita</i> L.	East Java, Indonesia	450	60	1:10	0.69	ND	Kunhermanti and Mahfud (2023)
<i>Mentha crispa</i> L.	Reunion Island, France	500	30	1:20	0.095	Carvone	Lucchesi et al. (2004)
<i>Mentha pulegium</i> L.	El-Guetfa, Algeria	800	30	ND	0.175	Pulegone	Messaoudi et al. (2021)
<i>Mentha piperita</i> L.	Novi Sad, Serbia	800	120	1:10	0.80	Menthol	Radivojac et al. (2021)
<i>Mentha spicata</i> L.	Thanjavur, India	630	3	1:2	2.53	Carvone	Shankar and Natarajan (2022)

525

526



527

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

530 In a recent study, Kunhermanti and Mahfud (2023) optimized the SFMWE of EOs from *M. piperita* L.  
 531 using response-surface-methodology (RSM). The variables included the feed-to-distiller volume (F/D)  
 532 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  
 533 optimized MEO extraction yield was 0.6937% that was obtained by applying MW power of 450 W,  
 534 particle size of 1 cm, 60 min of extraction time, and 0.1 g/mL of F/D ratio. Furthermore, Lucchesi et al.  
 535 (2004) employed the SFMWE and hydro-distillation techniques to extract EOs from *M. crispa* L. The  
 536 extraction by SFMWE was completed in half an hour; while, the classical hydro-distillation took 4.5 h.  
 537 Additionally, the SFMWE yielded a larger amount of MEO, as well as more oxygenated compounds  
 538 yield. Therefore, they confirmed that SFMWE can be employed as a suitable alternative to traditional  
 539 extractions of MEO. In another study, Messaoudi et al. (2021) extracted EOs from *M. pulegium* L. by  
 540 hydro-distillation, SD, and MWAD, and investigated their chemical composition, antibacterial,  
 541 antifungal, and anti-inflammatory activities. The results revealed that MEOs had significant anti-  
 542 inflammatory activity and antimicrobial properties against pathogenic bacteria and fungi. The  
 543 differences were particularly observed in the extraction yield and the components of EOs obtained by  
 544 different extraction methods. Besides, the MWAD was performed in 30 min, while conventional  
 545 extraction was completed in 180 min. However, the extraction yield of MEO isolated by hydro-

546 distillation and SD was higher than that of MWAD extracted MEO, and the worst chemical composition  
547 was obtained by MWAD.

548 In a different study, Mohammadhosseini et al. (2023) extracted *M. pulegium* L. EO, by MWAHD and  
549 headspace-solid phase micro-extraction (HS-SPME) combined with GC-MS. The MEOs isolated by  
550 HSSPME-GC-MS and MWAHD-GC-MS techniques revealed 28 and 30 components, respectively.  
551 Also, the MEO obtained by both extraction methods contained large amounts of oxygenated  
552 monoterpenes and non-terpenoid hydrocarbons; however, the concentration of their individual  
553 compounds was significantly different. Carvone comprised 56.0% of the MEO extracted by MWAHD,  
554 whereas the content of carvone in MEO from SPME technique was 17.7%. In a further study, Bui-Phuc  
555 et al. (2020) evaluated the effect of hydro-distillation and MAE on the extraction of EOs from Japanese  
556 mint (*Mentha arvensis* L.) and found that the MEO components and yield were influenced by the  
557 extraction technique. The extraction yield was 0.0832 mL g<sup>-1</sup> per dry weight and the menthol  
558 comprised 70.60% of the MEO when extracted by MAE. While, using the hydro-distillation technique  
559 yielded 0.0510 mL g<sup>-1</sup> MEO per dry weight and generated a smaller amount of menthol (62.80%).  
560 Moreover, the trans- and cis-menthone, occurred abundantly in the EO when using either MAE or  
561 hydro-distillation (4.62–5.01% and 20.19–16.30, respectively). In comparison with the hydro-  
562 distillation, MAE is reportedly capable of extracting higher EO yields and more menthol. The MAE is  
563 particularly the base of an advantageous approach due to its shorter distillation time and more efficient  
564 extraction. Similarly, Pavlić et al. (2021) reported that MWAHD is a good alternative to traditional EO  
565 recovery methods. The results showed that the MEO obtained by MWAHD provided higher content of  
566 menthone and similar amount of menthol compared to the EOs extracted by hydro-distillation. Also,  
567 the enzyme-inhibitory and antioxidant activities did not show significant differences in EOs obtained  
568 by MWAHD and hydro-distillation.

569 Since the efficiency of the MAE process is controlled by parameters, such as temperature, power, and  
570 extraction time, some researchers have investigated the influence of these parameters on the  
571 extraction efficiency of MEO. For instance, Costa et al. (2014) used a calorimetric technique to  
572 calibrate a MW reactor for MAE under different extraction times and temperatures. MEO was

573 extracted from the dried mint leaves by MAE at different temperatures (i.e., 40 °C, 50 °C, and 60 °C)  
574 and times (i.e., 10, 20, and 30 min). The GC-MS analysis results revealed that carvone was the major  
575 compound in the extracted MEOs. A face-centered central composite design (2<sup>2</sup> + 2 center points) was  
576 applied and the highest percentage of the carvone (0.091) was obtained for temperature and time at 40  
577 °C and 30 min, respectively. While, the lowest percentage of the carvone (0.011) was determined for  
578 temperature and time of 50 °C and 10 min, respectively.

#### 579 *Ultrasound-assisted extraction (UAE)*

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

590 The use of ultrasound treatment for EO extraction has several advantages over conventional methods.  
591 For instance, it significantly reduces the processing time and increases the extraction yield at lower  
592 temperatures. Additionally, the energy consumption and maintenance costs of ultrasound processing is  
593 relatively low; therefore, it is considered as an economic technology (Giacometti et al. 2018). However,  
594 conflicting results have been also reported regarding the impact of ultrasound on EOs. For instance,  
595 some studies have demonstrated that the UAE of EOs can significantly improve the bioactivity and  
596 physicochemical properties of herbal compounds (Damyeh et al. 2016). While others have stated that  
597 sonication do not have considerable effects of EOs (Kowalski et al. 2019), and some studies reported  
598 that UAE have negative effects on EOs (Tekin et al. 2015; Yu et al. 2021). They have stated that the  
599 sudden collapse of microbubbles in sonication process generates high temperature and pressure, free

600 radicals, and superoxide, which may adversely affect the quality of phytochemicals. In addition, the  
601 operational conditions, such as intensity, frequency, and sonication time have significant effects on the  
602 properties of phytochemicals. Therefore, the optimization of UAE can provide EOs with higher  
603 bioactivity compared to conventionally extracted EOs.

604 The optimization of MEO extraction by UAE technique has been addressed by several researchers in  
605 recent years. For instance, Sierra et al. (2022) optimized the UAE of *Mentha spicata* L. EO and  
606 determined the chemical composition of the extracted EO by GC-MS and high-performance liquid  
607 chromatography with diode-array detection (HPLC-DAD) analysis. Furthermore, Peleg's equation and  
608 polynomial regression analysis were used to define the extraction process. The modeling results  
609 revealed that time, solvent, and amplitude have significant effects in the recovery of MEO. The  
610 optimized extraction conditions were as follows: ethanol/water ratio (80:20), and 90% amplitude for 5  
611 min, which resulted in the highest amounts of terpenoid compounds. In another study, Mansoori et al.  
612 (2022) utilized UAE to extract limonene and carvone from *Mentha spicata* L. Box-Behnken design  
613 (BBD) was used to optimize the extraction parameters, such as extraction time (20–60 min), amplitude  
614 (20–100%), and the solvent: solid particles ratio (10–30 mL/g). The correlations obtained by different  
615 models revealed that the second-order polynomial model can be successfully applied to optimize the  
616 extraction variables. Also, the optimal conditions for the extraction of both limonene and carvone were  
617 the extraction time of 51 min, amplitude of 95% (380 W), and the solvent: solid particles ratio of 24  
618 mL/g. Under these optimized conditions, the extraction yield was 19.958 for carvone and 5.957 mg/g  
619 for limonene. These values are in good agreement with the predicted values of 20.237 and 6.024 mg/g  
620 obtained by BBD. Also, the results of this study revealed that UAE reduced the extraction time and  
621 temperature; while, increased the extraction yield by 54% compared to conventional hydro-distillation.  
622 Furthermore, Shotipruk et al. (2001) studied the feasibility of UAE for the extraction of menthol from  
623 peppermint. They exposed the plant materials to sonication for 1 h at 22 °C in an ultrasound bath of 40  
624 kHz and found that increasing the treatment time and temperature significantly increased the amount of  
625 menthol release. The extended sonication time increases the diffusion of menthol in the cuticle of  
626 glandular trichomes of peppermint and its exudation from the damaged trichomes. Whereas, increased

627 temperature increases the solubility of menthol and cause the phase transition of menthol crystals to a  
628 liquid state, and thus, facilitates its release from cells. In addition, Kowalski et al. (2015) studied the  
629 effects of ultrasound-assisted preliminary maceration on the chemical composition and extraction yield  
630 of EOs from peppermint, marjoram, and chamomile. The plant materials (20 g) were added to distilled  
631 water (1000 mL) and treated with an ultrasound bath at 30 °C with a frequency of 40 kHz and power of  
632 240 W. The results demonstrated a significant enhancement in the amount of extracted MEO and  
633 marjoram EO. However, sonication did not have a significant effect on the extraction yield of  
634 chamomile EO. Additionally, the composition of EOs was similar to conventionally extracted EOs and  
635 sonication did not destroy the components of EOs.

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

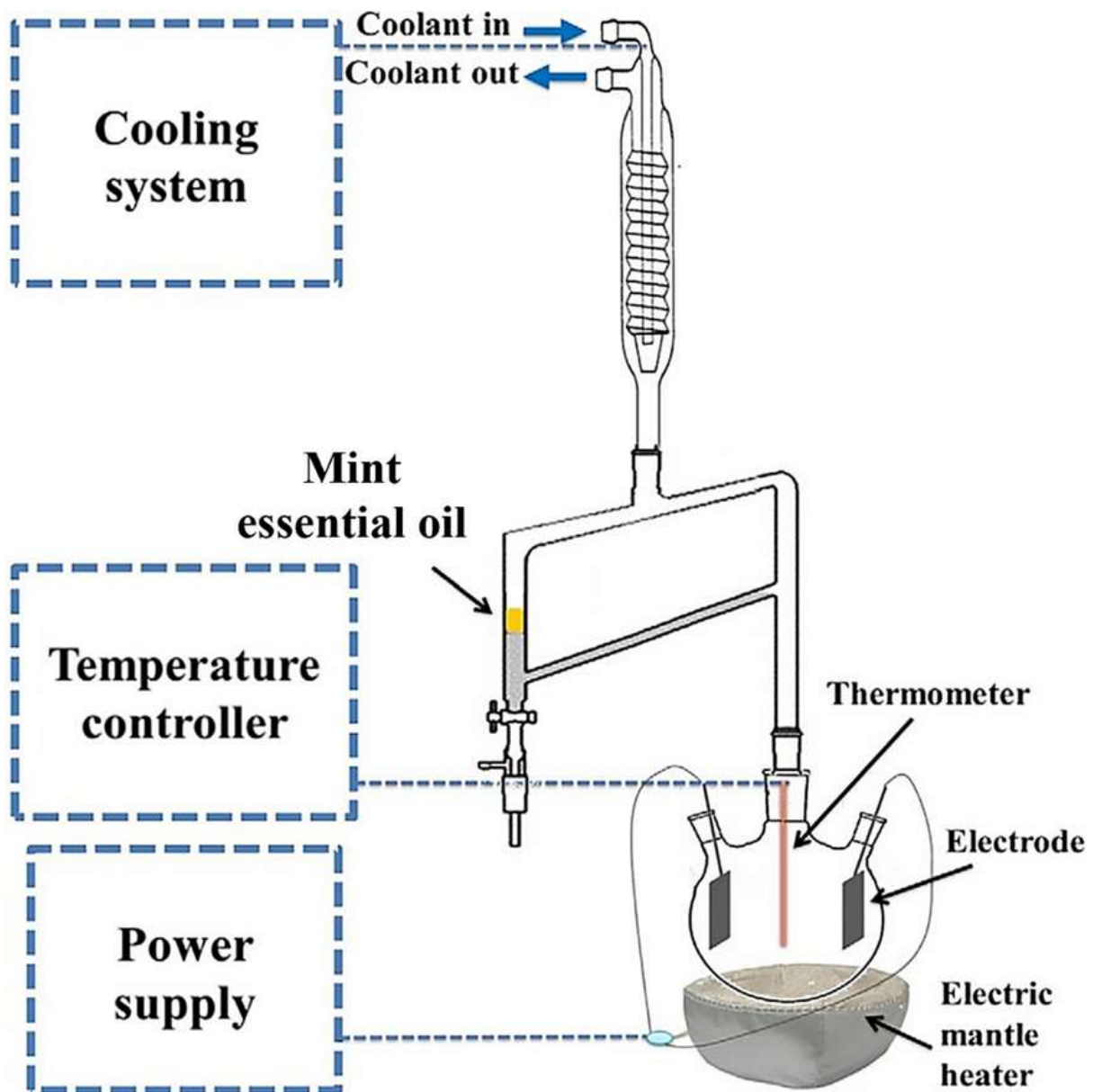
649 Overall, UAE of MEO represents a higher yield compared to the conventional methods in a much  
650 shorter processing time, less solvent usage, and lower energy consumption. All these factors  
651 demonstrated the application of ultrasound as a “green” and “high-efficiency” method for the extraction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

653 *Ohmic-assisted extraction (OAE)*

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

665 Application of OH for the extraction of EOs is a relatively new concept and several studies have  
666 reported the successful extraction of EOs by OH. In this respect, Gavahian et al. (2015a) compared the  
667 efficiency of ohmic assisted hydro-distillation (OAHD) and traditional hydro-distillation for the  
668 extraction of peppermint EOs (Fig. 6). The dried aerial parts of peppermint (30 g) were added to 500  
669 mL salted water (1% NaCl, w/v) and treated by different heating methods for 2 h. OAHD was performed  
670 by an ohmic distillator with platinum electrodes operating at 220 V, 50 Hz. While, conventional hydro-  
671 distillation was performed by a Clevenger-type apparatus on a laboratory heater. To remove water from  
672 the extracted MEO, it was dried over anhydrous sodium sulfate; and then, refrigerated in amber  
673 containers (4 °C). The results indicated that OAHD was an environmentally friendly extraction method  
674 as it significantly reduced the extraction time and energy consumption. OAHD was accomplished in  
675 less than half an hour while hydro-distillation process took about 1 h. Besides, scanning electron  
676 microscopy (SEM) images of peppermint leaves that were exposed to OAHD revealed a quick rupture  
677 of EO glands that accelerated the extraction process. Whereas, those subjected hydro-distillation did  
678 not show a sudden rupture of EO glands. Based on the GC-MS analysis results, the individual  
679 compounds of MEOs extracted by OAHD and hydro-distillation did not show obvious changes.  
680 Furthermore, the results indicate that using 1% NaCl did not have any adverse effects on the extractions

681 parameters and quality of obtained EOs from mint. In a similar study, they investigated the influence of  
682 different extraction parameters such as frequency (i.e., 25, 50 and 100 Hz) and voltage (i.e., 220 and  
683 380 V) on the quality and quantity of peppermint EOs extracted by OAHD, and compared the results  
684 with the EOs extracted by conventional hydro-distillation. The results showed that the extraction time  
685 reduced from 19.71 to 13.54 min by increasing the voltage of OAHD. While the extraction time in  
686 hydro-distillation was about one hour. However, the frequency did not have any significant effects on  
687 the extraction time and yield. Also, the GC-MS analysis of EOs extracted under different conditions did  
688 not show evident changes in their individual compounds (Gavahian et al. 2015b). In another study, they  
689 investigated the impact of NaCl concentration (i.e., 1, 3, and 6% w/v) and pre-treatment with ultrasound  
690 (i.e., 5, 10, and 15 min) on the MEO extracted by OAHD. The results indicated that the concentration  
691 of NaCl solution had significant effects on the extraction parameters of OAHD and the increase of  
692 electrolyte concentration increased the speed of extraction process with no adverse effects on the quality  
693 of extracted MEO. Therefore, the extraction time can be reduced by the optimization of NaCl  
694 concentration. Whereas the effects of sonication pre-treatment on extraction rate and duration were  
695 negligible. GC-MS analysis did not indicate any noticeable change in the compounds of all the extracted  
696 EOs (Gavahian et al. 2017).



697

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

700 *Pulsed electric field-assisted extraction (PEFAE)*

701 Pulsed electric field (PEF) is a non-thermal emerging method that involves applying high voltage  
 702 electric fields for extremely short (milliseconds to microseconds) periods to a material positioned  
 703 between two electrodes. This technique is mainly used for food preservation by the inactivation of  
 704 microorganisms and enzymes. PEF inactivates microorganisms by producing pores in their cell  
 705 membrane. As a result, the intracellular contents leak out of the cell and the cell membrane functions

706 interrupt (Abenoza et al. 2013; De Silva et al. 2018). The pore formation in PEF processing can occur  
707 in plant tissues, increases the permeability of plant cells and facilitates the release of intracellular  
708 compounds (Fincan and Dejmek 2002). Therefore, in recent years, PEF processing has been applied to  
709 increase the extraction yield of EOs, such as MEO.

710 Generally, PEF is applied to plant materials as a pre-treatment before the conventional extraction  
711 methods in order to increase the extraction yield of EOs and intensify the concentration of valuable  
712 compounds. In this regard, Miloudi et al. (2022) investigated the effects of PEF processing on the  
713 quantity and quality of extracted *Mentha spicata* L. EOs. PEF with a pulse frequency of 1 Hz was used  
714 as a pre-treatment and then MEO was extracted by hydro-distillation with a Clevenger-type apparatus.  
715 The samples without PEF pre-treatment were considered as the control. Dried Mint (20 g) was  
716 immersed in distilled water (200 ml) and heated by a balloon heater. The vapors were cooled to condense  
717 and accumulated in the collector of Clevenger apparatus, where MEO and water were separate by  
718 differences in their densities. The results showed that the antioxidant and anti-inflammatory properties  
719 of MEO were comparable in control and PEF-treated samples. The extraction yield of the control was  
720 0.94% in 120 min of hydro-distillation while a similar yield was obtained after 200 pulses of PEF in 60  
721 min of hydro-distillation. The GC-MS analysis showed that the composition of MEO was almost the  
722 same in PEF and conventionally extracted MEO, indicating that MEO and its individual compounds  
723 were not affected by PEF treatment. These findings are in agreement with the results reported by Cherrat  
724 et al. (2014) who stated that the chemical composition and antioxidant properties of *Mentha pulegium*  
725 L. EO were not affected by PEF. However, contradictory results have been reported by Sadowska et al.  
726 (2019) who studied the effect of the number of PEF pulses applied to peppermint on the EO content.  
727 They subjected the plant materials to 0, 150, 250, or 350 pulses with 25  $\mu$ s duration and the time interval  
728 of 10 s. Subsequently, the EOs were extracted by hydro-distillation in a Clevenger apparatus and the  
729 content and composition of EOs were analyzed. The highest extraction yield was observed in the control  
730 sample and PEF treatment decreased the extraction yield of MEO. Nevertheless, the number of pulses  
731 did not affect the MEO content. Therefore, the processing conditions, such as number, intensity, and  
732 duration of pulses are key factor for the successful extraction of MEO by PEF technique.

733

#### 734 **Research gaps and future trends**

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

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

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

757

758

## 759 **Conclusion**

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

772

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777 Sara Hedayati: supervision, conceptualization, methodology, paper collection and classification,  
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779 methodology, paper collection and classification, preparing figures, writing - original draft. Vahid  
780 Baeghbali: conceptualization, methodology, writing - original draft. Zahra Tahsiri: conceptualization,  
781 methodology, writing - original draft. Mohammad Hashem Hashempur: supervision, writing - original  
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783

784 **Conflict of interest**

785 The authors declare no conflict of interest.

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