



(REVIEW ARTICLE)



Eco-friendly microwave-assisted methods for essential oils extraction and their way to industrial application: A comprehensive review

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World Journal of Advanced Research and Reviews, 2026, 30(01), 2567–2578

Publication history: Received on 28 March 2026; revised on 29 April 2026; accepted on 30 April 2026

Article DOI: <https://doi.org/10.30574/wjarr.2026.30.1.0782>

Abstract

Essential oils are of great interest in various industries, including food, cosmetics, perfumery, and pharmaceuticals, due to their flavoring properties and biological activities. As forecasts predict an increase in global demand for essential oils and, the industrial sector increasingly focuses on green and sustainable production methods; microwave heating has revolutionized essential oil extraction. The development of environmentally friendly, solvent-free extraction methods such as SFME (solvent-free microwave extraction) and MHG (microwave hydrodiffusion and gravity), as well as improvements to existing extraction techniques like hydrodistillation and steam distillation, illustrate this revolution. Reduced processing time, low energy consumption, and improved quality of the resulting essential oils are the main advantages of the application of microwave heating. However, adapting these methods to an industrial scale presents challenges. This article reviews the operating principles of various microwave-assisted essential oil extraction methods, as well as the advances made and challenges encountered in scaling them up to industrial production.

Keywords: Microwave Heating; Essential Oils; Distillation; Extraction

1. Introduction

Essential oils are hydrophobic and volatile secondary metabolites with a distinctive odor, extracted from various organs of aromatic plants, including buds, flowers, fruits, stems, branches, leaves, seeds, roots, etc [1,2] Essential oils have numerous and varied applications. Their fragrant nature makes them a choice raw material in perfume manufacturing. In cosmetics and in food industry, they are used to scent products. Essential oils often exhibit biological activities, including antioxidant, antimicrobial, anti-inflammatory, antidiabetic, antitumor, insecticidal, antiviral, analgesic, and sedative properties, which promotes their use in medicine and pharmacy but, also in aromatherapy [3,4]

Essential oils are playing an increasingly important role in modern society, and numerous studies predict a sharp rise in global demand in the coming years. This creates a crucial need to ensure that the supply can meet this growing demand while adhering to the principles of sustainable development. Traditional methods for direct extraction of essential oils include hydrodistillation, steam distillation, and cold pressing. They can also be extracted indirectly using solvents or absorbants and then later recovered by vacuum distillation [5,6]. These methods used for a long time, have shown their limitations, particularly in terms of execution time, energy consumption, and the use of organic solvents. Considering the ecological crisis and current trends of green chemistry, new unconventional methods have been developed. They are aiming to address the shortcomings of traditional methods, particularly in terms of reducing extraction time and energy consumption, and also promoting the use of green or eco-friendly solvents to produce higher-quality essential oils [7-9]. Unconventional methods include notably microwave-assisted extraction, ultrasound-assisted extraction, supercritical fluid extraction, pulsed electric field-assisted extraction, and enzyme-assisted

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extraction [10-12]. This work focuses on various microwave-assisted essential oil extraction techniques as potential more sustainable alternatives to traditional production methods. These techniques include solvent-free microwave extraction, microwave hydrodiffusion and gravity, microwave-assisted hydrodistillation, and microwave-assisted steam distillation. Indeed, they have shown promising results, as reported in several scientific studies [13]. However, their scaling up to industrial production presents numerous challenges, which are also discussed here.

1.1. Microwave heating

Before discussing microwave-assisted essential oil extraction methods, it is important to understand how microwave heating works. Microwaves are electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz, that is, within the wavelength range of 1 mm to 1 m [14]. Microwaves are located on the right side of the electromagnetic spectrum, just after infrared waves. Microwave heating relies on the ability of these waves to interact with polar or polarized molecules such as water [15-17]. It is worth noting that an electromagnetic wave is a coupling of an electric field and a magnetic field perpendicular to each other propagating through space at a certain speed [18-20]. When faced with matter, microwaves can be reflected (by metals), transmitted (by electrically neutral materials such as paper, glass, plastic, and ceramics), or absorbed (by polar materials) [21-23]. Interaction between microwaves and matter can lead to various phenomena, primarily dielectric losses, conductive losses, and magnetic losses. Microwave heating in essential oil extraction is essentially linked to dielectric loss, or dielectric heating, which involves the interaction of a dielectric material with the electrical component of the electromagnetic wave. This type of heating is carried out via two main mechanisms: dipolar polarization and ionic conduction [24,25]. In the first case, electric dipoles (polar molecules such as water, for example) sensitive to the electric field of the wave attempt to align themselves with the field through rotational movement. Dipoles that struggle to keep up with the high-frequency oscillation of this external field collide with each other, resulting in energy conversion into heat [26,27]. Within the ionic conduction mechanism, mobile charges move through the material under the influence of the oscillating electric field of the microwave, creating electric currents. The electrical resistance resulting from collisions of these charges with neighboring atoms generates heat [17,28,29].

In traditional heating, energy is transferred to the material through convection, conduction, and radiation, from the surface to the interior, whereas in microwave heating, energy is delivered directly to the target through interactions between molecules and electromagnetic waves. The latter is known as volumetric heating [30,31]. The microwave field used is characterized by two main parameters that significantly influence the heating process: frequency and power. The frequency of the generated microwaves is the number of times per second that the electromagnetic field reverses its direction, thus forcing the water molecules to align with each oscillation, creating the friction that generates heat [16,32]. In general, lower frequencies tend to penetrate food more deeply but are absorbed less efficiently, while higher frequencies are absorbed more easily at the surface. This often leads to uneven heating, overheating the exterior while leaving the interior less heated. Taking all these constraints into account, the optimal frequency generally used in domestic and industrial commercial ovens is 2.45 GHz. This frequency is chosen for its balance between penetration depth and energy absorption, particularly by water molecules [33]. The frequency of 915 MHz is also used in industry when a deep penetration depth is required [23]. Microwave power refers to the amount of electromagnetic energy delivered by the oven per unit of time and is expressed in Watts (Joule/s). Increasing microwave power leads to a faster rate of temperature rise and therefore a shorter heating time [34,35]. For domestic microwave ovens, power typically reaches 700 W to 1200 W, while industrial ovens can reach several kilowatts. The efficiency of microwave heating also depends heavily on the characteristics of the material, including its size, shape and composition, but also on dielectric and thermal properties [36,37]. In general, polar materials are considered more susceptible to microwave heating than nonpolar [38,39]. Microwave heating offers significant advantages over conventional heating, including a high heating rate, low thermal degradation, shorter processing time, low maintenance, and low energy consumption [16,40,41]. Microwaves can be generated in various ways, but the most common is the use of magnetrons, which convert electrical energy into microwave radiation [23,42,43]. Having reviewed the principles of microwave heating, microwave-assisted essential oil extraction, methods are discussed below.

2. Microwave-assisted essential oil extraction methods

2.1. Solvent-Free Microwave Extraction (SFME)

The solvent-free microwave extraction (SFME) is a microwave-assisted extraction method performed at atmospheric pressure, without the addition of solvents, and can be considered a type of dry distillation using microwave heating instead of conventional heating [44]. The extraction relies on the ability of water molecules (dipoles) in the fresh sample to interact with microwaves, causing the water to heat up. The evaporation of this water leads to plant tissues rupture

and, the release of their contents [45]. Essential oils are then carried out of the material by the steam. The setup of a solvent-free microwave extraction is illustrated in figure 1.

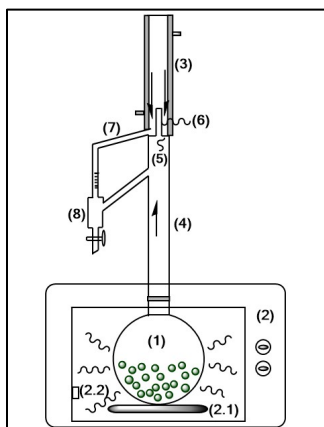


Figure 1 SFME setup

The round flask containing the fresh material (1) is placed in a microwave oven (2) on a rotating platform (2.1) and connected to a condensing chamber equipped with a cooling system (3) by a connecting channel (4), located above the microwave oven. This channel has a separating wall (made of glass or plastic) at its upper end (5) containing an upward-facing tube-shaped opening (6). The condensing chamber has a drain channel (7) leading to a receiver (8). When assembled, the flask, the connecting channel, the condensing chamber, and the receiver form a closed system, which prevents the entry of undesirable substances into the system.

Once the mixture of water vapor and essential oils has been extracted from the biological material, it is evacuated by natural convection from the top of the microwave oven through the connecting channel (4) to the condensation chamber via the tube-shaped opening (6). The connecting channel can be surrounded by a wall-mounted electric heater to prevent premature condensation of the vapors. Due to the widening of the space at the inlet of the condensation chamber, the vapor flow is preferentially dispersed towards the cold walls where condensation occurs. The distillate falls back onto the wall of the connecting channel (5) and is directed towards the receiver (8) via the discharge channel (7). The receiver is an overflow device connected to the connecting channel and equipped with a valve. This device acts as a separator and also prevents premature drying of the biological material by redirecting the condensed water it contains back into the flask. Thus, the water in the system is kept circulating while the extracted product remains in the receiver. At the end of the extraction, the tap is opened to collect the essential oils. The microwave oven temperature is measured using an infrared sensor (2.2), which allows the regulation of the microwave radiation to maintain the temperature below 100°C, but ideally between 80 and 90°C. The material can be stirred to improve its exposure to microwave radiation [46-48]. The advantages of SFME compared to conventional methods such as hydrodistillation are its rapidity, the absence of solvent use and low energy costs. The purity and quality of the extract are also improved [49-51]. Qiu et al. (2022) extracted essential oils from 100 g of *Litsea cubeba* fruit using SFME and hydrodistillation. A yield of 1.8% was obtained after 24 min of SFME, while the yield after 60 min of hydrodistillation was only 1.6% [51]. Similar observations were made by Araujo et al. (2021), who obtained a yield of 2.4% after 30 min of SFME and 3.16% after 120 min of hydrodistillation of 500 g of the aerial parts of *Thymus mastichina* [44].

2.2. Microwave Hydrodiffusion and Gravity (MHG)

Microwave Hydrodiffusion and Gravity (MHG) was developed by Vian et al. in 2008 for the extraction of essential oils and other bioactive compounds from aromatic plants. This method combines microwave heating and the action of earth's gravity at atmospheric pressure [52-54]. Just like SFME, MHG is a sustainable extraction method that does not involve the use of solvents [55,56]. Figure 2 illustrates the setup of an MHG.

MHG setup consists of an inverted flask (1) containing a perforated Pyrex disc (2) on which fresh biological material rests without the addition of any solvent. The flask is placed in a microwave oven (3) where microwaves heat the water inside the plant cells. The resulting thermal agitation ruptures the cells and, releases the essential oils from the biological material via hydrodiffusion [57]. The extract, a mixture of water and essential oil, flows from the flask under the influence of gravity through the perforated disc and is cooled in the condenser (4) before being collected in a separate flask (5). The peculiarity of MHG is that it does not involve evaporation of essential oil and water (a distillation) but, hydrodiffusion of the extract which remains liquid throughout the extraction process and goes through the condenser

under the action of gravity [58-60]. MHG is a simple, rapid, and environmentally friendly technique that uses no solvents and consumes little energy compared to conventional methods. MHG is also used in the extraction of other bioactive compounds [61-63].

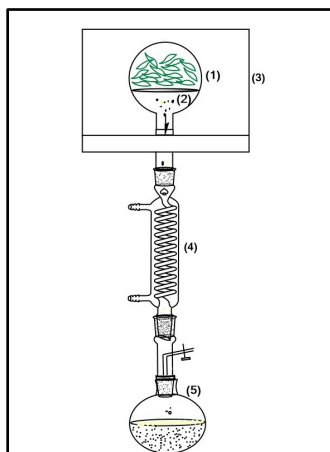


Figure 2 MHG setup

Vian et al. (2008) extracted essential oils from 500 g of *Mentha spicata L.* and *Mentha pulegium L.* using MHG without solvent addition and by hydrodistillation with the addition of 3 L of water. In 15 min of MHG, yields of 0.60% and 0.95% were obtained, compared to 0.59% and 0.90% in 3 h of hydrodistillation for *Mentha spicata L.* and *Mentha pulegium L.*, respectively. The essential oils obtained were of similar composition. The energy cost of 0.2 kWh for MHG was also significantly lower than the 3 kWh required for hydrodistillation [54]. Villa et al. (2022) extracted volatile compounds from 30 g of *Rosa x damascena* petals by MHG and SFME, and from 50 g of the same material by hydrodistillation and steam distillation. A yield of 0.28% was obtained in 5 minutes of MHG, against yields of 0.22% and 0.17% for 3 hours of hydrodistillation and steam distillation, respectively. SFME, on the other hand, yielded 0.40% in 12 minutes of extraction [59].

2.3. Microwave-assisted hydrodistillation (MAHD)

Microwave-assisted hydrodistillation (MAHD) is a modified form of hydrodistillation in which the traditional heating of biological material immersed in water is replaced by microwave irradiation [64]. A typical MAHD setup consists of three elements: a microwave oven, a flask, and a Clevenger apparatus, as illustrated in Figure 3 [65,66]. A flask (1) containing the material immersed in a certain amount of water is placed in a microwave oven (2) for heating. This flask is connected to a Clevenger apparatus (3) equipped with a condenser (4). The resulting distillate falls into the apparatus's receiver, allowing for the separation of water and oil. It is important to note that there are various variants of the Clevenger apparatus but the general operating principle remains the same [67,68].

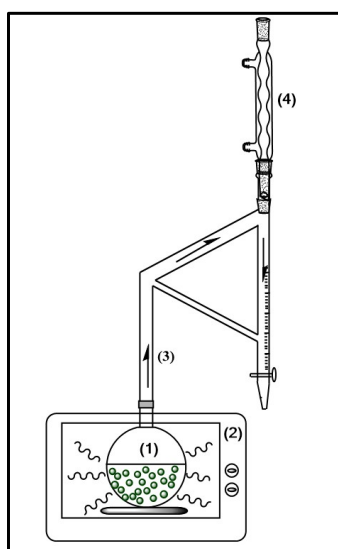


Figure 3 Microwave-Assisted Hydrodistillation Setup

Compared to conventional hydrodistillation, MAHD offers the advantage of a faster hydrodistillation process and reduces the amount of solvent and energy used. The quality of the oils obtained is also better than that of conventional hydrodistillation, in which the extracted substances often undergo alterations due to prolonged heating [69-71]. Numerous studies report MAHD experiments for essential oil extraction using a modified domestic microwave oven (a hole is drilled in the top to create the setup) [72]. The use of classic simple distillation setups without Clevenger apparatus is also reported [73,74].

Ghazanfari et al. (2020) extracted essential oils from 200 g of *Coriandrum sativum L.* seeds with 1.5 L of water using MAHD and conventional hydrodistillation. A yield of 0.325% was obtained after 1 hour of MAHD extraction, compared to a yield of 0.31% after 4 hours of hydrodistillation [70]. Boudraa et al. (2021) extracted essential oils from 30 g of *Foeniculum vulgare* seeds using MAHD and hydrodistillation with 300 mL of water. After 20 minutes of MAHD, a yield of 0.89% was obtained, compared to a yield of 0.77% after 3 hours of hydrodistillation [68]. Moradi et al. (2018) extracted essential oils from 100 g of *Rosmarinus officinalis L.* leaves using MAHD and hydrodistillation with 300 mL of water, respectively, for 30 min and 1.5 h extraction times. MAHD yielded approximately 1.5 mL of essential oils, compared to 1.3 mL for hydrodistillation [71].

2.4. Microwave steam distillation (MSD)

The application of microwave heating to steam distillation of essential oils has already been proposed. Microwave steam distillation (MSD) is a version of steam distillation in which microwave heating of the biological material is added to the heat provided by hot water vapor [37]. Figure 4 illustrates the setup of an MSD.

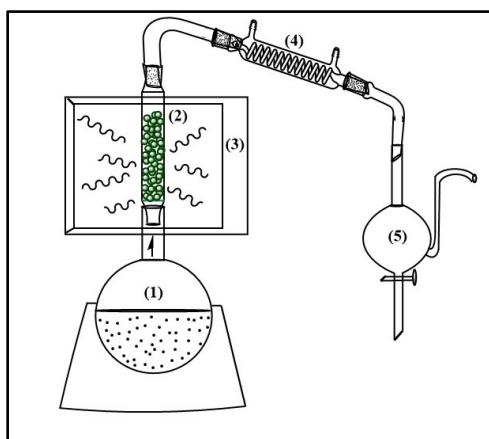


Figure 4 MSD setup

A cartridge containing the biological material, loaded into a cylindrical Pyrex tube (2), is placed in a microwave oven (3). Heating begins as soon as steam from a steam generator (1) outside the oven comes into contact with the material. Microwaves facilitate the rupture of the steam-hydrated cells, and the released oil is carried to the condenser (4). After condensation, the distillate is collected in the florentine flask (5), where the oil is separated from the water [75]. MSD offers advantages over steam distillation in that it is faster, consumes less energy, and is also said to yield a higher-quality essential oil [76]. The setup shown in Figure 4 has been adapted for the production of a device specifically designed for MSD. However, it is worth noting that other scientific studies report a different setup but with the same idea of combining traditional steam distillation with microwave heating [77,78]. Sahraoui et al. (2008) compared the extraction of essential oils from 20 g of dried lavender flowers using MSD and conventional steam distillation. Essential oils of similar quality, with identical yields of 2.7%, were obtained after 6 minutes with MSD and 30 minutes with conventional steam distillation. Because the extraction time was shorter, steam consumption, and therefore energy costs, were also lower for MSD. Energy consumptions of 0.05 kWh/g and 0.3 kWh/g were reported for MSD and conventional steam distillation, respectively [75]. Sahraoui et al. (2015) reported the extraction of essential oils from 20 g of dried *Thymus palleseus* leaves using MSD and conventional steam distillation. In the case of MSD, a yield of 2.45% was obtained after 5 minutes, whereas after 20 minutes of conventional steam distillation a yield of 2.35% was obtained [76].

2.5. Other microwave-assisted essential oil extraction techniques

Other microwave-assisted techniques for obtaining essential oils have been proposed but, have not gained traction within the scientific community. This is the case with Mengal et al. (2006), who filed a patent for the development of a

solvent-free, microwave-assisted method for extracting natural products. Unlike the SFME described above, the significantly different setup includes a pressure-reducing device to facilitate the destruction of plant cells in the biological material [79]. Following the same pressure-reduction logic, a patent for a microwave-assisted vacuum distillation (VMHD) system was published [80]. However, the complexity of the setup makes the implementation of such a device difficult. Craveiro et al. (1989) also published a work presenting a solvent-free essential oil extraction device in which fresh biological material is heated in a microwave oven. Unlike SFME, the vapors generated are carried away by an airflow generated by an air pump, and the water is not redirected into the biological material to sustain the distillation [81].

3. Application of Microwave-Assisted Essential Oil Extraction in Industry

Although the production of microwave-assisted essential oil extraction equipment for laboratories is advanced, scaling it up for industrial applications is still under development. Indeed, scaling up processes using microwave heating encounters difficulties related to the ununiformity of the heating process. This phenomenon is due to various factors, the most frequently cited being the shape, size, dielectric and thermal properties of the material being processed, as well as the non-uniform distribution of electromagnetic waves within the processing cavity. Ununiform heating implies that some areas of the material heat up more quickly than others. These areas called hot spots result from an uneven distribution of electromagnetic waves within the microwave oven cavity, as well as from the heterogeneity of the material, which means that some areas absorb more microwaves than others—for example, in areas of the material with higher humidity. These hot spots can lead to burns and even explosions of the heated material, raising safety concerns [82-85].

Irregularly shaped and thick materials experience much less uniform heating compared to thin, regularly shaped materials [33]. The lack of control over the electromagnetic field within the cavity is also a major problem. Indeed, once generated, microwaves can overlap at certain points, leading to localized concentrations of electromagnetic energy. Moreover, due to the cavity design, this energy may be concentrated in a certain place, generally in the center of the oven, limiting the usable volume [86,87]. Two different approaches are generally exploited to try improving the uniformity of microwave heating: improving and controlling the distribution of electromagnetic magnetic energy within the cavity and, adjusting the position of the heated material to make microwave absorption as uniform as possible (sample agitation or rotation) [88-90].

Advances toward scaling up of microwave-assisted essential oil extraction technologies have been reported in recent years. The company Milestone (www.milestonesci.com) has developed a 75L capacity, multimode microwave-assisted essential oil extraction pilot reactor, the MAC-75. This reactor can be used for SFME, MHG, as well as for extracting herbs and spices with non-flammable solvents such as water, glycerin, and vegetable oils. To generate microwaves, the MAC-75 uses four 1500W magnetrons. It features a 150 L stainless steel cavity housing a removable, perforated PTFE rotating drum that can hold up to 75L of biological material. The perforations on the drum allow for the circulation of vapors and liquids. The cavity is also capable of operating under vacuum. The temperature inside the cavity is controlled by a resistance temperature sensor inserted within the cavity, which automatically adjusts the irradiated power. An industrial touchscreen terminal allows for device control, ideal for quality control processes [91]. The same company also produced the *Ethos X line* reactor, similar to the MAC-75, which has a 45 L capacity and is used for MAHD. It features a hybrid heating system that combines microwave heating using a 4 kW magnetron and conventional heating via a 1.8 kW resistor located inside the cavity to accelerate the extraction process. The material is first placed in appropriate bags before being loaded into the rotating drum. The cavity then automatically fills with water for hydrodistillation. After condensation in the cooling system connected to the reactor, the distillate is collected in a burette where separation takes place [69].

Filly et al. (2014) performed a pilot-scale extraction of rosemary essential oil from 3 kg of fresh biological material in a MAC-75 reactor. After 30 minutes of extraction at 100°C, a yield of 0.50% was obtained, with 80% of the final yield achieved after 24 minutes. The essential oil obtained was of good quality, although some minor compounds present in laboratory-produced essential oil were absent [92]. Araujo et al. (2021) also used the MAC-75 to extract essential oil from 4 kg of previously dried and rehydrated aerial parts of *Thymus mastichina* in 60 minutes [44]. Figalço et al. (2016) extracted 10 mL of essential oil from 20 kg of lemon peels by MAHD using the MAC-75 at 80 °C for one hour [93]. The MAC-75 has also been used for polyphenol extraction from waste [94,95]. Lamberti et al. (2021) used the ETHOS XL reactor to extract essential oils from 8,2 kg of fresh *Humulus lupulus* L at 100°C, a yield of 4,39 mL/kg was obtained within 70min of extraction [69].

Scientific works found in the literature report small quantities of processed material and can hardly be considered industrial productions that can compete with the quantities currently produced by traditional methods. Currently,

scientists are actively working on overcoming the challenges on the way of industrial application of microwave-assisted essential oils extraction methods. This will introduce new green and more sustainable ways of producing essential oils at large scale.

Abbreviations

- SFME: Solvent-free Microwave Extraction
- MHG: Microwave Hydrodiffusion and Gravity
- MAHD: Microwave-Assisted Hydrodistillation
- MSD: Microwave steam distillation
- VMHD: Vacuum Microwave Hydrodistillation

4. Conclusion

Microwave heating offers significant advantages over traditional heating methods in essential oil extraction through reduced processing time, lower energy consumption, and improved essential oil quality. The nature and operating principle of this heating method have enabled the development of solvent-free essential oil extraction methods such as SFME and MHG, as well as improvements in the performance of traditional methods like hydrodistillation and steam distillation. They are considered environmentally friendly and enhance the quality of the resulting essential oils. These technological advancements are crucial for meeting the requirements for green processes imposed by the ecological crisis. However, scaling up these technological processes presents technical challenges. If those challenges are overcome, these methods will help meet the growing global demand for essential oils following the popularization of aromatherapy and consumer demand for natural additives and active compounds in different industries.

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare no conflict of interest

Authors' contributions

RMS: Finding, analysis, organization of raw data and writing. KMT: revision and correction.

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