
Solventless Extraction of Essential Oil

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<http://dx.doi.org/10.5772/intechopen.72401>

Abstract

Essential oil is one of an important concentrated liquid that possesses many physical, chemical and pharmacological properties. Extraction of essential is one of the main issues in the last decade. Conventional treatment consisting of hydrodistillation and steam distillation has many disadvantages and finds difficult to purify essential oil. Now, it is much easier to extract essential oil with the invention of new greener technologies that reduce the involvement of solvent, decrease the extraction time, energy and descent the interaction of the concentrated volatile liquid with atmospheric oxygen through the application of vacuum.

Keywords: solventless extraction, solvent-free extraction, essential oil, extraction technology, green extraction

1. Introduction

Essential oils are complex concentrated liquids comprise of volatile compounds. They have been extracted from numerous plant [1]. They have been widely used as a food preservative (eucalyptus essential oil, thyme), cosmetic preparation (lavender oil), antimicrobial (lemon grass, cumin, fennel), and anticancer agent (lemon grass, *Croton flavens*). Hydrodistillation and steam distillation is the common conventional method of extracting the essential oil [2–9]. These methods have some disadvantages Preservation of essential oil from its environment can be possible through a number of technologies such as nanospheres, liposome, microcapsules and nanoemulsions [10]. A lot of research is currently underway for extracting essential oil through new green methods. This review chapter documents the updated information about novel solvent-less extraction of essential oil.

2. Solventless extraction in a closed system

Solventless extraction of essential oil was designed in a closed system under reduced pressure using a vacuum and compared the results with the conventional methods such as hydrodistillation. The quality and quantity of essential oil extracted from *Chromolaena odorata*, *Citronella*, *Baekea frutescens*, Orange peel was found better than traditional method [11, 12]. The novel closed system was also optimized using central composite design (CCD) on *Chromolaena odorata* and maximum extraction yield was found to be under an ideal condition at 80°C temperature and 8 h of time [13].

3. Solvent-free microwave extraction

Solvent-free microwave extraction (SFME) of volatile natural substances was the first to patent in 2004. Farid Chemat et al. invented a method of extraction of essential oil consisting of a microwave oven with a microwave chamber for receiving the biological material and a condensation chamber. It was first tested on different spices such as ajowan (*Carum ajowan*, Apiaceae), cumin (*Cuminum cyminum*, Umbelliferae), star anise (*Illicium anisatum*, Illiciaceae) and the result had published on 4 February 2004. This technique is more fast, without solvent and effective when compared to hydrodistillation [14]. It was also evaluated on three aromatic herbs basil (*Ocimum basilicum*), garden mint (*Mentha crispa*), and thyme (*Thymus vulgaris*) where extraction time was significantly decreased from 4.5 h (hydro-distillation) to 30 min (SFME) [15]. This method was modified by many scientists. SFME method was modified by Wang et al. on dried *Cuminum cyminum* L. and *Zanthoxylum bungeanum* Maxim. by adding carbonyl iron powders (CIP) and mixed with the sample. CIP helps to reduce time (30 min) and microwave power (85 W) [16]. An attempt had been made to improve solvent-free extraction of essential oil using graphite powder, activated carbon powder. The effect was studied on *Illicium verum* Hook. f. and *Zingiber officinale* Rosc. [17]. Pressurized solvent-free microwave assisted extraction was used for extraction of phenolic compounds. The best extraction conditions were obtained, in a laboratory scale extractor of 50 mL filled with 4 g fresh berries, using a 1000 W microwave power applied during 50 s and repeated 5 cycles [18]. Solvent-free microwave extraction was modified by changing the flow of product toward the gravitation force. It is also known as Microwave dry-diffusion and gravity. It was better than hydrodistillation. The extraction performed in just 45 minutes with less energy and a clean process [19]. The extraction condition of SFME was optimized on *Elettaria cardamomum* (L.) using central composite design (CCD). The conditions such as time (min), power (W), humidity (%) was optimized by (CCD) and percentage yield (%) of was compared [20]. Optimization of SFME of pigeon pea leaves performed on an aliquot of 200 g plant materials that were wetted before extraction by soaking in water for 1 h [21]. Optimum parameters of SFME was performed on *S. chinensis* fruits and found ideal extraction time of 30 min, irradiation power 385 W and the moisture content of 68% respectively [22]. The quality of essential oil was also evaluated using Solvent-free microwave extraction method and compared with conventional method. It was found to be more effective than conventional method [23]. The effect of solvent-free microwave extraction was performed on several medicinal plants such as *Calamintha nepeta* [24], Basil leaves [25], *Dryopteris fragrans* [26], *Schisandra chinensis* [27], *Cymbopogon winterianus* [28].

Medicinal plants	Identified essential oil	Pharmacological activities	Reference
<i>Cuminum cyminum</i> L. and <i>Zanthoxylum bungeanum</i> Maxim	2-Methyl-5-(1-methylethyl)-bicyclo[3.1.0]hex-2-ene, 1-Methylethylidene-cyclohexane, α -Pinene, Camphene, β -Phellandrene, β -Pinene, β -Myrcene, β -Phellandrene 3-Carene, 4-Carene, 1-Methyl-2-(1-methylethyl) benzene, d-Limonene, Eucalyptol, 1-Methyl-4-(1-methylethyl)-1,4-cyclohexadiene, 1-Methyl-4-(1-methylethylidene)-cyclohexene, β -Terpineol, 6,6-Dimethyl-2-methylene-bicyclo[2.2.1]heptan-3-one, 4-Methyl-1-(1-methylethyl)-3-cyclohexen-1-ol, Pulegone, Cuminal 4-(1-Methylethyl)-1-cyclohexene-1-carboxaldehyde 2-Ethylidene-6-methyl-3,5-heptadienal, α -Proyl-benzenemethanol, 4-(1-Methylethyl)-1,4-cyclohexadiene-1-methanol, 6-Isopropylidene-1-methyl-bicyclo[3.1.0]hexane, Caryophyllene, 2,6-Dimethyl-6-(4-methyl-3-pentenyl)-bicyclo[3.1.1]hept-2-ene, 7,11-Dimethyl-3-methylene-1,6,10-dodecatriene, 2-Isopropyl-5- methyl-9-methylene-bicyclo[4.4.0]dec-1-ene, Octahydro-3,8,8- trimethyl-6-methylene-1H-3a,7-methanoazulene, Thujopsene, 1-(1,5-Dimethyl-4-hexenyl)-4-methylbenzene 5-(1,5-Dimethyl-4- hexenyl)-2-methyl-1,3-cyclohexadiene, Copaene, 1-Methyl-4-(5- methyl-1-methylene-4-hexenyl)-cyclohexene, β -Sesquiphellandrene, Caryophyllene oxide, Carotol	N.F	[16]
<i>Rosmarinus officinalis</i> L.	a-Pinene, Camphene, b-Pinene, Myrcene, a-Phellanderene, a-Terpinene c-Terpinene, Linalool, 1,1,8-Cineole, Camphor, Borneol, b-Caryophyllene, Trans b-ocimene, cis-Sabinene hydrate, Verbinone, Terpene-4-ol, Myrtenol, Bornyl acetate, Cis-jasmone, a-Humulene, Pentasiloxane Caryophyllene, 1,5-Diphenyl 2H-1,2,4 triazoline, 1-Methyl-2,4- nitrophenyl benzimid, 2-Methoxy-3,8-dioxocephalotax-1-ene, 1,2-Benzenedicarboxylic acid, 9-Octadecenoic acid, Docosanoic acid	Anti-bacterial	[31]
<i>Dryopteris fragrans</i>	Cedrene [1S-(1a,4a,7a)]-1,2,3,4,5,6,7,8-octahydro-1,4,9,9-tetramethyl- 4,7-methanoazulene Caryophyllene, 10S,11S-himachala-3(12),4-diene, 4-(2,6,6-Trimethyl-2-cyclohexen-1-yl)-2-butanone, (E)-4-(2,6,6- trimethyl-2-cyclohexen-1-yl)-3-buten-2-one, 1,2,3,4,4a,5,6,8a- Octahydro-7-methyl-4-methylene-1-(1-methylethyl)naphthalene, (R)-c-cadinene 4-(2,6,6-Trimethyl-1-cyclohexen-1-yl)-3-buten-2-one, (1a,4ab,8aa)-1,2,3,4,4a,5,6,8a-Octahydro-7-methyl-4-methylene-1-(1- methylethyl)naphthalene, Albicanol, [1R-(1a,4ab,8aa)]-decahydro- 1,4a-dimethyl-7-(1-methylethylidene)-1-naphthalenol, Calarene epoxide, (1R,4S,11R)-4,6,6,11-tetramethyltricyclo[5.4.0.0(4,8)] undecan-1-ol, 1,4,4a,5,6,7,8,8a-Octahydro-2,5,5,8a-tetramethyl-1- naphthalenemethanol, (-)-Isolongifolol, acetate, 1R,4S,7S,11R-2,2,4,8 tetramethyltricyclo[5.3.1.0(4,11)]undec-8-ene, [3S-(3a,5a,8a)]- 1,2,3,4,5,6,7,8-octahydro-a,a,3,8-tetramethyl-5-azulenemethanol acetate,	Anti-oxidant	[26]
<i>Schisandra chinensis</i> (Turcz.) Baill	a-Pinene, Camphene, 2-Carene, D-Limonene, o-Cymene, gamma-Terpinene, Thymol methyl ether, L-Bornyl acetate, Cyclocopacamphene, Ylangene, beta-bourbonene, (+)-Sativene,(-)-beta- Elemene, Germacrene-D, (E)-(b)-Farnesene, (E)-a-bergamotene, Elixene, alpha-amorphene,(-)-beta-Chamigrene, Beta-bisabolene, b-maaliene, c-cadinene, b-himachalene, a-Chamigrene, (+)-a-Longipinene, (+)-Cuparene, b-Caryophyllene, Guaiene, (+)-Ledene, L-calamenene	Anti-oxidant	[22]
<i>Cajanus cajan</i> (L.) Millsp.	3,6-Dimethyl-octane, Naphthalene, Dodecane, 6-Ethyl-undecane, 4-Methyl-dodecane, 4-Ethyl-undecane, 4,6-Dimethyl-dodecane, 1-Methyl-naphthalene, 2,6,11-Trimethyl-dodecane, α -Longipinene, 2-Methyl-tridecane, (+)-Cyclosativene, Ylangene, α -Copaene, Tetradecane, Longifolene, Caryophyllene, β -Selinene, α -Bergamotene, α -Himachalene, Humulene, Alloaromadendrene, α -Bisabolene, 2,4-Bis(1,1-dimethylethyl)-phenol, δ -Cadinene, Hexadecane, Norphytane	Anti-microbial activities	[21]

Medicinal plants	Identified essential oil	Pharmacological activities	Reference
<i>Ocimum basilicum</i> L.	Sabinene, Octen 3 ol, β -Pinene, Heptanol, β -Myrcene, p-Cymene, Limonene, 1,8-Cineole, β -Ocimene, γ -Terpinene, Fenchone, Linalool, Camphor, Menthol, α -Terpineol, Methyl chavicol, Nerol, Neral, Geraniol, Geranial, α -Terpinenyl acetate, Neryl acetate, α -Copaene, Geranyl acetate, β -Bourbonene, β -Cubebene, β -Elemene, Methyl eugenol, β -Caryophyllene, α -Bergamotene, α -Humulene, β -Farnesene, Germacrene-D, γ -Cadinene, Δ -Cadinene, α -Bisabolene, β -Bisabolene, Spathulenol, Caryophyllene oxide, α -Cadinol	N.A	[25]
<i>Hippophae rhamnoides</i>	Isorhamnetin, isorhamnetin 3-O-glucoside, isorhamnetin 3-O-rutinoside and quercetin 3-O-glucoside	Anti-oxidant	[32]
<i>Lavandula angustifolia</i>	1, 8-cineole, Camphor, Borneol, p-cymene, Limonene, Cryptone, isobornyl formate, cuminaldehyde, Valerianol, α -pinene	Anti-bacterial activity	[33]
<i>Calamintha nepeta</i>	a-Thujene, a-Pinene, Sabinene, b-Pinene, D2-Carene, a-Terpinene, Camphene, (Z)-b-Ocimene, allo-Ocimene, Myrcene, Limonene, c-Terpinene, p-Cymene, Octan-3-ol, Eugenol, Geranyl acetone, Hexahydrofarnesylacetone, Octacosane, Phytol, Caryophyllene oxide, T-Cadinol, a-Cadinol, T-Muurolol, a-Copaene, b-Elemene, b-Cubebene, b-Bourbonene, a-Humulene, Caryophyllene, Germacrene-D, c-Cadinene, epi-Sesquiphellandrene, d-Cadinene, Menthyl acetate, Bornyl acetate, Menthol, Chrysanthenone, Piperitenone, Piperitenone oxide, Isopulegonen, Pulegone, Piperitone, cis-Sabinene hydrate, 1,8-Cineole, Dihydrocarveol, trans-Sabinene hydrate, Menthone, Isomenthone, Terpinen-4-ol, a-Terpineol	N.A	[24]

Table 1. List of medicinal plants and their identified essential oil evaluated under microwave assisted solvent-free extraction.

Instrumentation	Extraction conditions	Results	Reference
Reactor (500 mL), microwave oven, agitator, shielded non-invasive thermometry system, transformer with maximum output power is 800 W with 2450 MHz of microwave irradiation frequency (MIF).	100 g of sample and 20 g of carbonyl iron powder (CIP) were added inside the reactor, stirred, heated (85 W) for 30 min at 100°C with speed of rotation (200 rpm), concentrated outside the microwave oven by a cooling system	CIP helps to improve the microwave absorption capacity than water and is faster (30 min) than conventional method	[16]
The multi-mode reactor (2 × 800 W, 2450 MHz), rotating microwave diffuser, plasma coated PTFE cavity, circulating cooling system at 5°C	250 g of Rosmarinus leaves were placed into the reactor without the addition of water or any solvent	Higher amounts of oxygenated monoterpenes were found as compared to conventional method	[31]
Microwave-accelerated reaction system (1000 W, 2450 MHz) multimode microwave reactor armed with a TFT multicolour liquid crystal screen, a power sensor (power range 0–1000 W), an infrared temperature sensor, a temperature controller and electromagnetic stirrer	200 g plant material was moistened prior to extraction by soaking in certain proportions of water (weight basis) for 1 h and then draining off the excess water. After that, the moistened materials were subjected to the microwave oven cavity and a condenser was used to collect the extracted essential oils in a pre-setting procedure.	A maximal extraction yield of 0.33% was achieved under optimal conditions of extraction time 34 min, irradiation power 520 W and humidity 51% 16	[26]

Instrumentation	Extraction conditions	Results	Reference
Multimode microwave reactor, temperature Infrared sensor with maximum output power is 700 W, microwave rotating diffuser that ensures homogeneous microwave distribution	A 100 g of <i>S. chinensis</i> fruits were moistened prior extraction by soaking in water then draining the excess of water. The extraction was continued until no distillate was obtained. The essential oil was collected, dried over anhydrous sodium sulfate and stored at 0°C until analyzed	Identification of optimum parameters was as follows; extraction time 30 min, irradiation power 385 W and the moisture content of <i>S. chinensis</i> fruits 68%, respectively	[22]
The microwave-accelerated reaction system with multimode microwave reactor (2.45 GHz), IR temperature sensor, an electromagnetic stirrer, a time calculator controller, circulating water-cooling system	200 g plant materials were wetted before extraction by soaking in a certain proportion of water for 1 h, and then removal the excess water. The wetted material was placed in the reaction flask and connected to a glass reaction flask	The optimal parameters were extraction time 44 min, irradiation power 660 W, and humidity 68%, with extraction yield of 0.330 (% w/w)	[21]
Microwave oven (EMM-2007X, Electrolux, 20 l, the maximum delivered the power of 800 W) with a wave frequency of 2450 MHz. A round bottom flask with a capacity of 1000 ml was placed inside the oven and was connected to the three-way adapter and Liebig condenser through the hole. Then, the hole was closed with PTFE to prevent any loss of the heat inside	150 g of fresh plant materials were placed in the reaction flask and heated by microwave irradiation with 400 W (50% power) for 30 min without adding any solvent or water. During the process, the vapor passed through the condenser outside the microwave cavity where it was condensed. Essential oil and water were simply separated by decantation. The essential oil was collected in amber vials, dried over anhydrous sodium sulfate and stored at 277 K	SFME exhibit shorter extraction times as compared to conventional method (30 min vs. 4.5 h) and better yields (0.13% vs. 0.11%)	[25]
Milestone EOS-G microwave laboratory oven having a multimode microwave reactor (2.45 GHz) with a maximum delivered the power of 900 W. The extraction vessels are made from Pyrex and have a capacity of 1000 mL with a temperature sensor optic fiber which was inserted in the center of embedded plant material and also in the reactor above the matrix	400 g of sea buckthorn press cake was heated using a fix power density 1 W·g ⁻¹ without the addition of solvents or water. The crude extract was collected continuously in a graduated cylinder. The extraction was continued until no more extract was obtained or overheating was detected	This method exhibit shorter extraction time (15 min), cleaner feature (no solvent or water used) and extraction of valuable flavonoids (Isorhamnetin, isorhamnetin 3-O-glucoside, isorhamnetin 3-O-rutinoside and quercetin 3-O-glucoside) at optimized power (400 W)	[32]
Microwave apparatus, 2450 MHz with maximum power 1000 W and ACTE0 sensor for temperature monitoring. The power of the oven was 500 W for 10 min. The temperature was achieved at 95°C, and the extraction was carried out for 25 min	30 g of dried <i>Lavandula angustifolia</i> was soaked in 20 mL distilled water at room temperature (25°C) for 1 h in order to hydrate the external layers of the plant material. The moistened plant material was placed in a flat-bottom flask combined with a Clevenger apparatus. The SFME process was performed for 35 min. The essential oils were collected in amber colored vials, dehydrated with anhydrous sodium sulfate, capped under nitrogen and kept at 4°C	It helps to extract more oxygenated compounds	[33]

Instrumentation	Extraction conditions	Results	Reference
Vacuum and nitrogen gas was applied on and off to remove air and replacing it with nitrogen in a closed system. At the end of the extraction process water and oil was separated and anhydrous sodium sulfate was used to dry the excess water	Fresh leaves of aromatic plants were grinded and to break them into smaller pieces and increasing the area of contact. Then, the grind leaves were put in a flask which was connected to another flask as a receiving flask. Firstly, the raw material was cooled down to a very low temperature to prevent decomposition and to avoid premature oil evaporation	Essential oil produced is lighter in color, higher yield, contains a cleaner, better purity and produced a stronger aroma compared to the essential oil produced from hydro-distillation	[12]

Table 2. Instrumentation and extraction conditions of solvent-free extraction.

This modern method was transformed from laboratory scale to pilot and industrial scale [29]. List of Medicinal plants, their identified essential oil evaluated under Microwave assisted solvent-free extraction were represented in **Table 1**. Instrumentation and extraction conditions of solvent-free extraction were mentioned in **Table 2** (**Figures 1–3**).

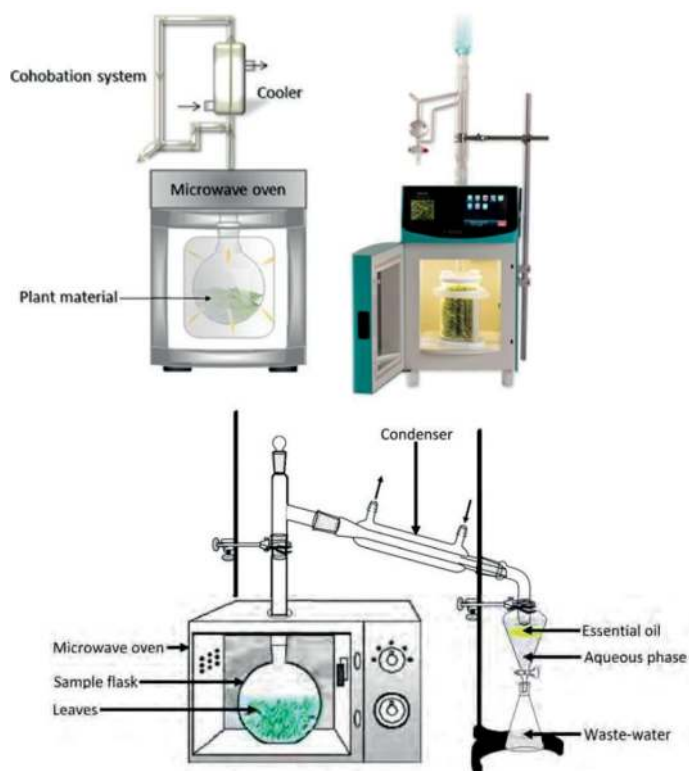


Figure 1. Solvent-free microwave extraction (SFME) [25, 30].



Figure 2. Microwave hydro-diffusion and gravity (MHG) [30].

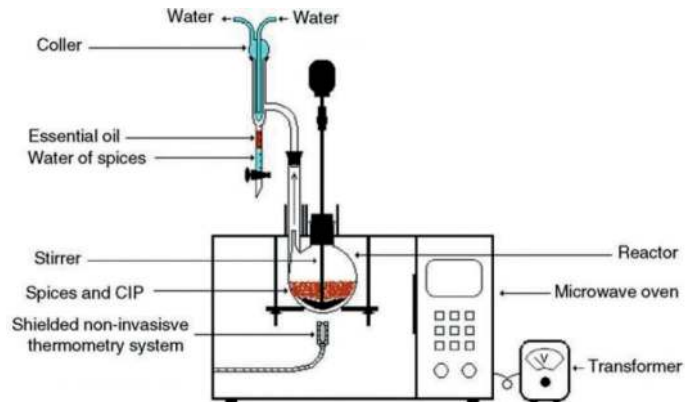


Figure 3. Improved SFME [16].

4. Conclusion

The term Solvent-less and solvent-free extraction have been used as synonymous with each other. Extraction of essential oil using these methods has a number of advantages such as fast action, cleanliness, green method, low energy output as compared to traditional extraction method. However, microwave extraction needs extra care before use as it may cause some negative effect on human health. There are many opportunities and modification possible in term of purification of essential oil by applying in combination with other extraction technique.

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