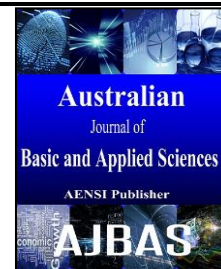




AUSTRALIAN JOURNAL OF BASIC AND APPLIED SCIENCES

ISSN:1991-8178 EISSN: 2309-8414
Journal home page: www.ajbasweb.com



Techniques For Extraction of Essential Oils From Plants: A Review

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ARTICLE INFO

Article history:

Received 11 September 2016

Accepted 10 November 2016

Published 28 November 2016

Keywords:

Essential oil; Extraction method;
Conventional methods; Non-
Traditional extraction

ABSTRACT

Background: Pure essential oils are derived from various part of the plants. These essential oils have a very high commercial value due to its properties. They are widely used in the various fields of industries, such as perfumery industries and pharmaceuticals. The essential oils for are primarily used in the perfumery industry and have a very high commercial value due to its therapeutic properties. Objective: study the techniques available to extract the essential oils from different plant and study the efficiency and the selectivity for this techniques. Results: The essential oils of the plant are composed of heat-sensitive chemical compounds, the use of steam distillation and Microwave-assisted extraction (MAE) techniques would inevitably inflict thermal degradation to the natural fragrance. In this experimental work, solvent extraction method was employed due to its mild extracting condition and lower operating cost. Ethanol was used as the solvent due to its high availability in the market. The extract compositions were compared using gas chromatography analysis. Conclusion: Extraction of essential oils out by different techniques and Innovative methods avoid shortcomings of content optional techniques friendly to environmental to avoid chemical risk, extraction time, consumption the solvents and obtain yield quality of essential oils.

INTRODUCTION

Essential oils are plant-based volatile oils with strong aromatic components that are made up of different chemical compounds. For example, alcohols, hydrocarbons, phenols, aldehydes, esters and ketones are some of the major components of essential oil (Younis *et al.*, 2008). Oils from hundreds of plant species are available commercially (Formaceck and Kubeckza, 1982). Numerous studies have demonstrated the efficiency of Essential Oils in low doses in the fight against bacterial pathogens (Oussalah *et al.*, 2006, 2007) even against multi-resistant bacteria (Mayaud *et al.*, 2008; Burt *et al.*, 2004). The effectiveness of these procedures has been attributed mainly to the presence of active phytochemicals or bioactive compounds in plants. Given the scope of searching new antimicrobial agents (Seoussen *et al.*, 2016), antimicrobials derived from plant materials are often regarded as natural and safe compared to industrial chemicals. Of late, plant-based medicine has become more popular due to the increasing concern of consumers with regard to the use of synthetic chemical preparations and use of artificial antimicrobial preservatives, especially in modern food protection practices. The worldwide market for essential oil growth rapidly and nowadays a lot of scientific research presently focused on the industrial development together with environmental preservation by used different techniques as Hydrodistillation (HD), Supercritical Fluid Extraction (SFE), Microwave-Assisted Hydrodistillation (MAHD) and Ultrasound-assisted extraction (UAE).

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To Cite This Article: Hesham H. A. Rassem, Abdurahman H. Nour, Rosli M. Yunus., Techniques For Extraction of Essential Oils From Plants: A Review. *Aust. J. Basic & Appl. Sci.*, 10(16): 117-127, 2016

1. Essential Oils:

An essential oil is a concentrated hydrophobic liquid containing volatile aroma compounds from the plant. They are also known as aromatic oils, fragrant oils, steam volatile oils, ethereal oils, or simply as the "oil of" the plant material from which they were extracted, such as oil of clove. The advantages of essential oils are their flavor concentrations and their similarity to their corresponding sources. The majority of them are fairly stable and contain natural antioxidants and natural antimicrobial agent as on citrus fruits (Somesh *et al.*, 2015). Essential oils are usually colorless, particularly when fresh. Nevertheless, with age essential oil may oxidize which resulting the color becomes darker. Therefore, essential oil needs to be stored in a cool, dry place tightly stoppered and preferably full in amber glass containers.

Essential oil is used in perfumery, aromatherapy, cosmetics, incense, medicine, household cleaning products and for flavoring food and drink. They are valuable commodities in the fragrance and food industries. More than 250 types of essential oils. A number of countries produce different kinds of essential oils. India ranks second in the world trade of essential oils (Rao *et al.*, 2005). Essential oils are derived from various sections of plants. An essential oil is usually separated from the aqueous phase by a physical method that does not lead to significant change in its chemical composition (Figure 1). Essential oils could be then subjected to an appropriate further treatment. Essential oils are oily aromatic liquids extracted from aromatic plant materials. They could be biosynthesized in different plant organs as secondary metabolites (A. El Asbhanni *et al.*, 2015).

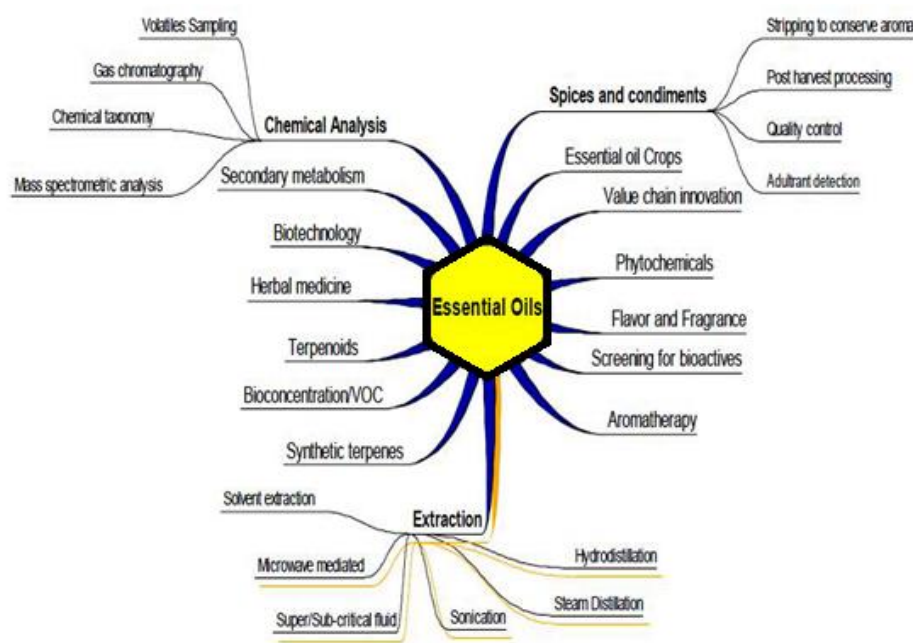


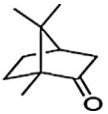
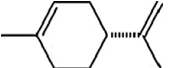
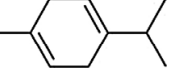
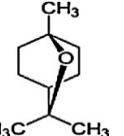
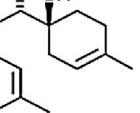
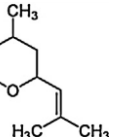
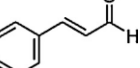
Fig. 1: Show the copious branching of specializations in the work of consequential oils.

2. Chemical constituents of Essential Oils:

They are complex mixtures of volatile compounds such as terpenes (mostly monoterpenes and sesquiterpenes), phenolics and alcohols (Lucchesi *et al.*, 2004). However, the essential oils are highly complex and may include oxygenated compounds. The essential oil is so called because they were believed to represent the quintessence of odor and flavor from the flower kingdom – differ in composition properties from fatty or fixed oils, which consist for the most part of glycerides and from mineral or hydrocarbon oils. A scientific definition of the term essential or volatile oils is not possible, although several practical definitions exist. The most common one defines an essential oil as a more or less volatile material isolated from an odorous plant of a single botanical species by a physical process (see Table 1).

They are oxygenated derivatives of hydrocarbon terpenes such as aldehydes, ketones, alcohols, phenols, acids, ethers and esters (Bakkali *et al.*, 2008). Some terpenes are potent drugs against diseases such as heart disease (Liebgott *et al.*, 2000), malaria (Parshikov and Netrusov, 2012) and cancer (Ebada *et al.*, 2010).

Table 1: Some compounds of essential oils with physical properties and biological applications.

EO components	Molecular structure	Chemical Formula	Molecular Weight	Boiling point C°	Refractive index (20 C°)	Plant source	Some biological application	References
Ketones alcohols Camphor		C ₁₀ H ₁₆ O	152.23	204	-	Lavendula stoechas	Antispasmodic, sedative, diuretic, antirheumatic, anti-inflammatory, anti-anxiety	(Braden <i>et al.</i> , 2009)
Monoterpenes D-Limonène		C ₁₀ H ₁₆	136.23	175.4	1.473	Citrus limon	Antifungal, Antioxidant	(Singh <i>et al.</i> , 2010)
g-Terpinène		C ₁₀ H ₁₆	136.23	183	1.474	Origanum vulgare	Antioxidant	(Ruben Olmedo, 2014)
Terpenic oxides 1,8-Cineole		C ₁₀ H ₁₈ O	154.25	176	1.457	Eucalyptus poly bractea	Antiinflammatory activity (asthma)	(Juergens <i>et al.</i> , 2003)
Oxygenated sesquiterpenes α-Bisabolol		C ₁₅ H ₂₆ O	222.37	153	1.496	Matricaria recutita	Anti-irritant, anti-inflammatory, antimicrobial	(Joseph M. Mwaniki <i>et al.</i> , 2015)
Terpenic oxides Cis-Rose oxide		C ₁₀ H ₁₈ O	154.25	70–71	1.454	Rosa damascena	Antiinflammatory, Relaxant	(Nonato <i>et al.</i> , 2012; Boskabad y <i>et al.</i> , 2006)
Cinnamaldehyde		C ₉ H ₈ O	132.16	248–250	1.621	Cinnamomum, Zeylanicum	Bactericide, fungicide, insecticide	(Ye <i>et al.</i> , 2013)

3. Essential oils extraction methods:

Essential oils are used in a wide variety of consumer goods such as detergents, soaps, toilet products, cosmetics, pharmaceuticals, perfumes, confectionery food products, soft drinks, distilled alcoholic beverages (hard drinks) and insecticides. The world production and consumption of essential oils and perfumes are increasing very fast. Production technology is an essential element to improve the overall yield and quality of essential oil. Essential oils are obtained from plant raw material by several extraction methods (Wang and Weller, 2006)(Dick and Starmans,1996).

3.1 Classical and Conventional Methods:

There are several by the numbers methods of extraction behavior of essential oils. The timid technologies about essential oils processing are of abundant significance and are still overused in copious parts of the globe. Hydrodistillation (HD), Steam distillation (SD), Solvent extraction, Enfleurage, Cohobation, and Maceration are the roughly traditional and generally used methods.

3.1.1 Hydrodistillation (HD):

Hydrodistillation is a traditional method for removal of essential oils. Water or hydrodistillation is one of the oldest and easiest methods (Meyer-Warnod *et al.*, 1984). Being used for the extraction of essential oils. Hydrodistillation normally used to isolation essential oils from the aromatic and medicinal plant. The conventional method for the extraction of essential oils is hydrodistillation (HD), in which the essential oils are evaporated by heating a mixture of water or other solvent and plant materials followed by the liquefaction of the vapors in a condenser. The setup comprises also a condenser and a decanter to collect the condensate and to separate essential oils from water, respectively (Figure 2). The principle of extraction is based on the isotropic distillation. In fact, at atmospheric pressure and during extraction process (heating), water or other solvent and oils molecules. Hydro-distillation (HD) is a variant of steam distillation, which is bespoke by the French

Pharmacopoeia for the extraction of Essential oils from dried plants and the quality control of essential oils in the lab. There are three types of hydrodistillation: with water immersion, with direct vapor injection and with water immersion and vapor injection. It is a multilateral process that can be utilized for large or small industries. The distillation time depends on the plant material being processed. Prolonged distillation produces only a small amount of essential oil, but does add unwanted high boiling point compounds and oxidation products.

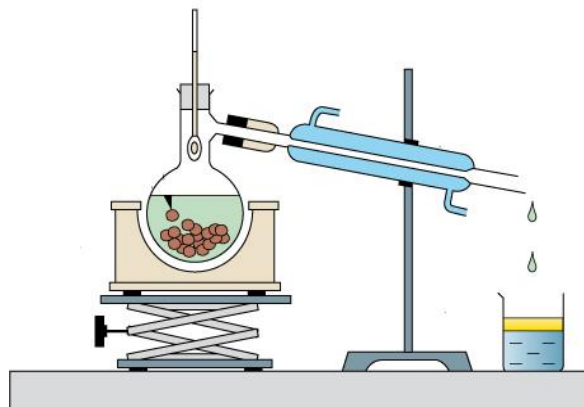


Fig. 2: The schematic subsidize apparatus for hydrodistillation.

3.1.2 Steam Distillation:

Steam distillation is a type of distillation (a separation or extraction process) for a temperature-sensitive plant such as natural aromatic compounds. It once was a popular laboratory method for purification of organic compounds but has become obsolete by vacuuty distillation. Steam distillation still important in certain industrial sectors (Fahlbusch *et al.*, 2003). Steam distillation is one of ancient and official approved methods for isolation of essential oils from plant materials. The plant materials charged in the alembic are subjected to the steam without maceration in water. The injected steam passes through the plants from the base of the alembic to the top. Steam distillation is a method where steam flows through the material as shown in Figure 3. This steam functions as agents that break up the pores of the raw material and release the essential oil from it. The system yields a mixture of a vapor and desired essential oil. This vapor is then condensed further and the essential oil is collected (Rai R. and Suresh B., 2004). The principle of this technique is that the combined vapor pressure equals the ambient pressure at about 100 °C so that the volatile components with the boiling points ranging from 150 to 300 °C can be evaporated at a temperature close to that of water. Furthermore, this technique can be also carried out under pressure depending on the essential oils extraction difficulty.

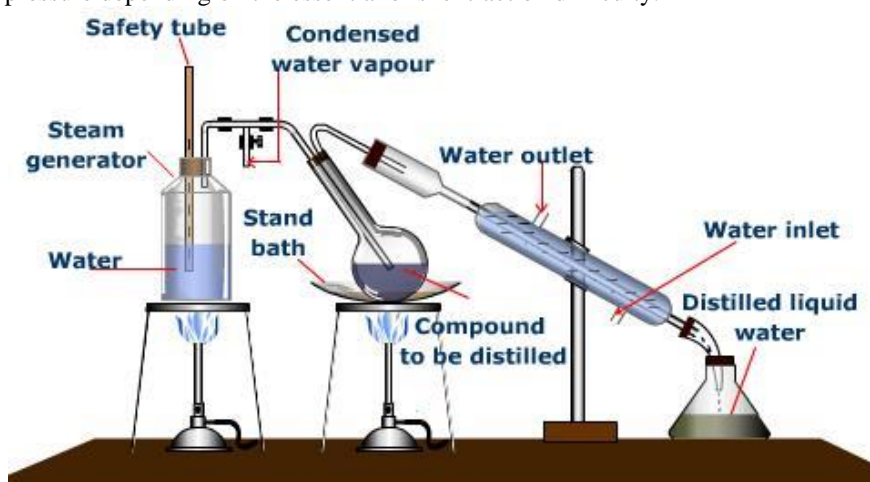


Fig. 3: The schematic subsidize apparatus for Steam distillation

3.1.3 Solvent extraction:

Solvent extraction, also known as Liquid-liquid extraction or partitioning, is a method to separate a compound based on the solubility of its parts. This is done using two liquids that don't mix, for example, water and an organic solvent (Figure 4). In the Solvent-Extraction method of Essential Oils recovery, an extracting unit is loaded with perforated trays of essential oil plant material and repeatedly washed with the

solvent. Solvent extraction is used in the processing of perfumes, vegetable oil, or biodiesel. Solvent extraction is used on delicate plants to produce higher amounts of essential oils at a lower cost (Chrissie *et al.*, 1996). The most frequently applied sample preparation procedure in plant material analysis. The quality and quantity of extracted mixture are determined by the type of extra heat applied because of the method is limited by the compound solubility in the specific solvent used. Although the method is relatively simple and quite efficient, it suffers from such disadvantages as long extraction time, relatively high solvent consumption and often unsatisfactory reproducibility (Dawidowicz *et al.*, 2008).

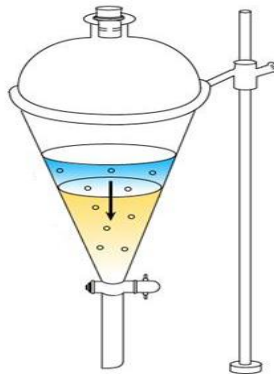


Fig. 4: Solvent Extraction

3.1.4 Soxhlet Extraction:

A Soxhlet extractor is a piece of laboratory apparatus (Harwood, Laurence M. and Moody, Christopher J, 1989) invented in 1879 by Franz von Soxhlet. (Soxhlet *et al.*, 1879) It was originally designed for the extraction of a lipid from a solid material (Figure 5). Typically, a Soxhlet extraction is used when the desired compound has a limited solubility in a solvent, and the impurity is insoluble in that solvent. It allows for unmonitored and unmanaged operation while efficiently recycling a small amount of solvent to dissolve a larger amount of material. Soxhlet extraction involves solid-liquid contact for the removal of one or several compounds from a solid by dissolution into a refluxing liquid phase. In a conventional soxhlet device, the solid matrix is placed in a cavity that is gradually filled with the extracting liquid phase by condensation of vapors from a distillation flask. When the liquid reaches a preset level, a siphon pulls the contents of the cavity back into the distillation flask, thus carrying the extracted analytes into the bulk liquid (Schantz *et al.*, 1998). This procedure is repeated until virtually complete extraction is achieved. There are several advantages of Soxhlet extraction. The most important are that the sample is repeatedly brought into contact with fresh portions of the solvent. This procedure prevents the possibility of the solvent becoming saturated with extractable material and enhances the removal of the analyte from the matrix. Moreover, the temperature of the system is close to the boiling point of the solvent. This excess energy in the form of heat helps to increase the extraction kinetics of the system. Soxhlet extraction has several disadvantages, including it requires several hours or days to perform; the sample is diluted in large volumes of solvent, and due to the heating of the distillation flask losses due to thermal degradation and volatilization have been observed.

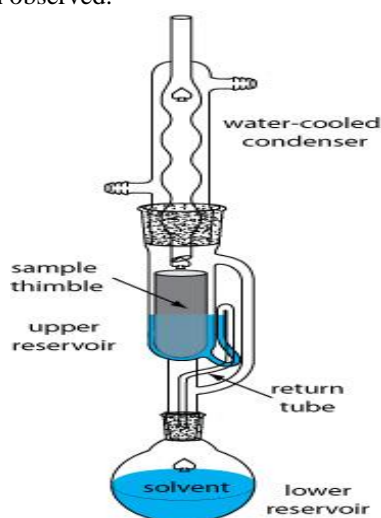


Fig. 5: Soxhlet Extraction

3.1.5 Cold Pressing method:

The term cold pressed theoretically means that the oil is expeller-pressed at low temperatures and pressure. Cold pressed method is one of the best methods to extract essential oils. This process is used for most carrier oils and many essential oils. This process ensures that the resulting oil is 100% pure and retains all the properties of the plant (Figure 6). It is a method of mechanical extraction where heat is reduced and minimized throughout the batching of the raw material. The cold pressed method is also known as scarification method. Cold pressed method is mainly used for extracting essential oils from plants, flower, seeds, lemon, tangerine oils (Arnould *et al.*, 1981). In this process, the outer layer of the plants contains the oil are removed by scrubbing. Then the whole plant is pressed to squeeze the material from the pulp and to release the essential oil from the pouches. The essential oil rises to the surface of the material and is separated from the material by centrifugation.

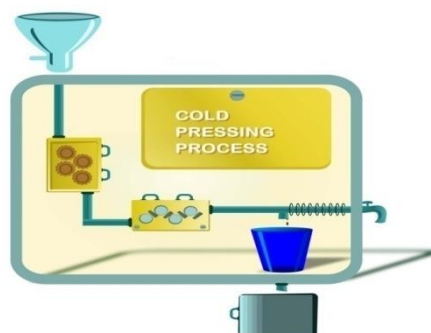


Fig. 6: Cold Pressing Method

3.2 Innovative Techniques Of Essential Oils Extraction (Non-Traditional):

One of the disadvantages of conventional techniques is related with the thermolability of Essential oils components which undergo chemical alterations (hydrolyse, isomerization, oxidation) due to the high applied temperatures. The quality of extracted Essential oils is therefore extremely damaged particularly if the extraction time is long. It is important that extraction methods could maintain Essential oils chemical composition and natural proportion at its original state. Since economy, competitiveness, eco-friendly, sustainability, high efficiency and good quality become keywords of the modern industrial production, the development of essential oils extraction techniques has never been interrupted. Strictly speaking, conventional techniques are not the only way for the extraction of essential oils. Novel techniques, for example, abide by green extraction concept and principles have constantly emerged in recent years for obtaining natural extracts with a similar or better quality to that of official methods. New extraction techniques must also reduce extraction times, energy consumption, solvent use and CO₂ emissions.

Traditional methods of extraction of essential oils have been discussed and these are the methods most widely used on a commercial scale. However, with technological advancement, new techniques have been developed which may not necessarily be widely used for commercial production of essential oils but are considered valuable in certain situations, such as the production of costly essential oils in a natural state without any alteration of their thermosensitive components or the extraction of essential oils for micro-analysis.

3.2.1 Supercritical Fluid Extraction (SFE):

Supercritical Fluid Extraction (SFE) is the process of separating one component (the extractant) from another (the matrix) using supercritical fluids as the extracting solvent. Extraction is usually from a solid matrix, but can also be from liquids. Supercritical fluids have been used as solvents for a wide variety of applications such as essential oil extraction and metal cation extraction (Figure 7). In practice, more than 90% of all analytical supercritical fluid extraction (SFE) is performed with carbon dioxide (CO₂) for several practice reasons. Apart from having relatively low critical pressure (74 bars) and temperature (32°C), CO₂ is relatively non-toxic, nonflammable, noncorrosive, safe, available in high purity at relatively low cost and is easily removed from the extract (Rozzi *et al.*, 2002). The main drawback of CO₂ is its lack of polarity for the extraction of polar analytes (Pourmortazavi *et al.*, 2007). These essential oils can include limonene and other straight solvents. Carbon dioxide (CO₂) is the most used supercritical fluid, sometimes modified by co-solvents such as ethanol or methanol. It was found that extracts prepared by SFE yielded a higher antioxidant activity than extract prepared by other methods (Fadel *et al.*, 1999). This extraction method produces higher yield, higher diffusion coefficient, and lower viscosity. Many essential oils that cannot be extracted by steam distillation can be obtainable with carbon dioxide extraction. Nevertheless, this technique is very expensive because of the price of this equipment for this process is very expensive and it is not easily handled. Supercritical extracts proved to be of superior quality, with better functional and biological activities (Capuzzo

et al., 2013). Furthermore, some studies showed better antibacterial and antifungal properties for the supercritical product.

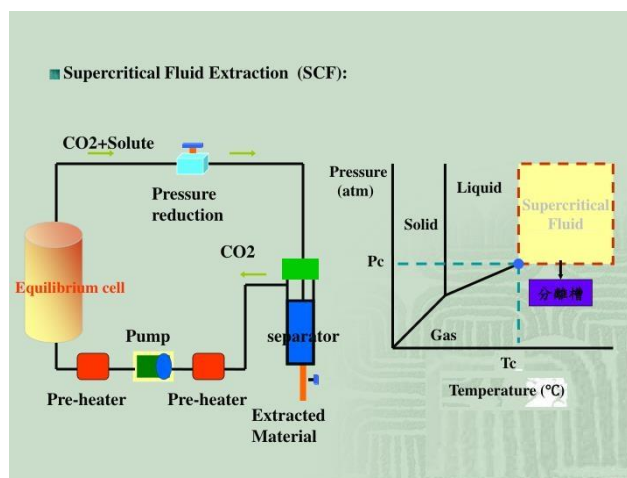


Fig. 7: Supercritical Fluid Extraction (SFE)

3.2.2 Microwave-Assisted Hydrodistillation (MAHD):

Microwave-assisted hydrodistillation is an advanced hydrodistillation technique utilizing a microwave oven in the extraction process. (Golmakani *et al.*, 2008) reported some recently published studies have successfully utilized a microwave oven for the extraction of active components from plants. The efficiency of Microwave-assisted hydrodistillation is strongly dependent on the dielectric constant of water and the sample (Brachet *et al.*, 2002).

Conventional techniques for the extraction of active constituents are time and solvent consuming, thermally unsafe and the analysis of numerous constituents in plant material is limited by the extraction step (Mandal *et al.*, 2007). High and fast extraction performance ability with less solvent consumption and protection offered to thermolabile constituents are some of the attractive features of this new promising microwave-assisted hydrodistillation technique (Figure 8). Application of Microwave-assisted hydrodistillation in separation and extraction processes has shown to reduce both extraction time and volume of solvent required, minimizing environmental impact by emitting less CO₂ in atmosphere (Lucchesi *et al.*, 2004; Ferhat *et al.*, 2006) and consuming only a fraction of the energy used in conventional extraction methods (Farhat *et al.*, 2009). The use of Microwave-assisted hydrodistillation in industrial materials processing can provide a versatile tool to process many types of materials under a wide range of conditions.

Microwave-assisted hydrodistillation is a current technology to extract biological materials and has been regarded as an important alternative in extraction techniques because of its advantages which mainly are a reduction of extraction time, solvents, selectivity, volumetric heating and controllable heating process.

The principle of heating using Microwave-assisted hydrodistillation is based upon its direct impact with polar materials/solvents and is governed by two phenomenon's: ionic conduction and dipole rotation, which in most cases occurs simultaneously (Letellier *et al.*, 1999).

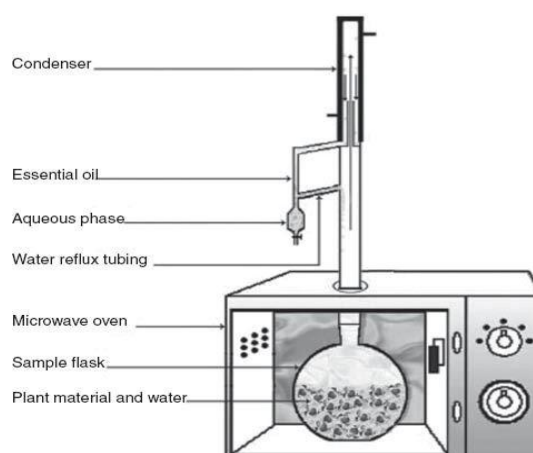


Fig. 8: Microwave-assisted hydrodistillation

3.2.3 Ultrasound-assisted extraction (UAE):

Ultrasound-assisted extraction (UAE) is a good process to achieve high valuable compounds and could be involved in the increase in the estimate of some food by-products when used as sources of natural compounds or plant material (Bhaskaracharya *et al.*, 2009). The major importance will be a more effective extraction, so saving energy, and also the use of mean temperatures, which is beneficial for heat-sensitive combinations. This technique was developed in 1950 at laboratory apparatus (Vinatoru *et al.*, 2001). Ultrasound allows selective and intensification of essential oils extraction by release from plant material when used in combination with other techniques for example solvent extraction and hydrodistillation (Figure 9). Ultrasound technology has been featured as a valuable method in food engineering processes and plants (Bhaskaracharya *et al.*, 2009), and become this field from the techniques active. In these applications the power ultrasound increases the surface wetness evaporation average and causes oscillating velocities at the interfaces, which may affect the diffusion boundary layer and generate rapid series of alternative expansions of the material, affecting cluster transfer (García-Pérez *et al.*, 2006).

The plants raw material is immersed in water or another solvent (Methanol or ethanol or anyone from the solvents) and at the same time, it is subjected to the work of ultrasound (Karim *et al.*, 2012). This technique has been used for the extraction of many essential oils especially from the flower, leaves or seeds (Sereshi *et al.*, 2012).

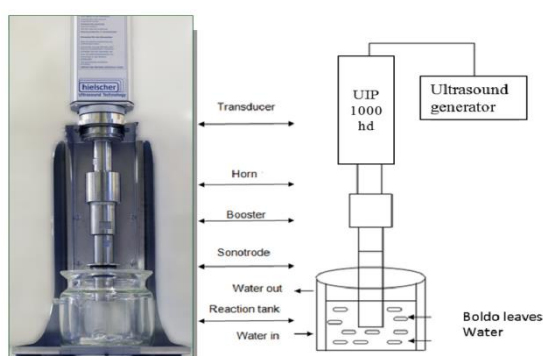


Fig. 9: Ultrasound-assisted extraction (UAE)

3.2.4 Solvent-free microwave extraction (SFME):

Solvent-free microwave extraction (SFME) is in the extraction procedure of essential oil which is cloaca by the in site water of the plant material without added any solvent (Lucchesi *et al.*, 2007). Developed this method by Cheat and co-workers (Lucchesi *et al.*, 2004a,b). Based on the integration of dry distillation and microwave heating energy, (Figure 10). It consists on the microwave dry-distillation at atmospheric pressure of plant without adding water or any organic solvent (Filly *et al.*, 2014). In a model SFME procedure, the plant material was moistened before to extraction by soaking in a certain amount of water for 1 to 2 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 presetting procedure. The irradiation power, temperature, and extraction time were controlled by the panel in the instrument. The separated essential oil was dried over anhydrous sodium sulfate and stored at 4 °C in the dark. The extraction yield of essential oil was calculated as follows:

$$\text{Extraction yield (ml/kg)} = V/M$$

where V is the volume of essential oil in herb samples (ml), and M is the mass of the herb samples (kg).

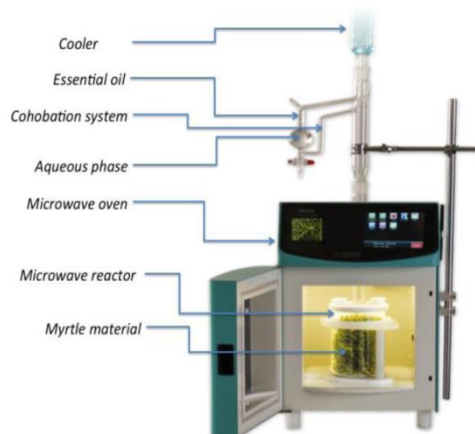


Fig. 10: Solvent-free microwave extraction (SFME)

3.2.5 Microwave hydro diffusion and gravity (MHG):

Is a new green technique for the extraction of essential oils. This green extraction technique is an original microwave blend microwave heating and earth attraction at atmospheric pressure. MHG was conceived for experimenter and processing scale applications for the extraction of essential oils from different kind of material plants (Figure 11) (Abert *et al.*, 2008).

Microwave hydro diffusion and gravity (MHG) become clear not only as economic and efficient but also as environment-friendly, not require solvent or water and as it does require less energy (Chemat *et al.*, 2004). The performances and advantages of this technique are a reduction of extraction time (in the case of hydro-distillation it takes 90min or more but in this technique only 20min) and reducing environmental impact and power saving (Vian *et al.*, 2008).

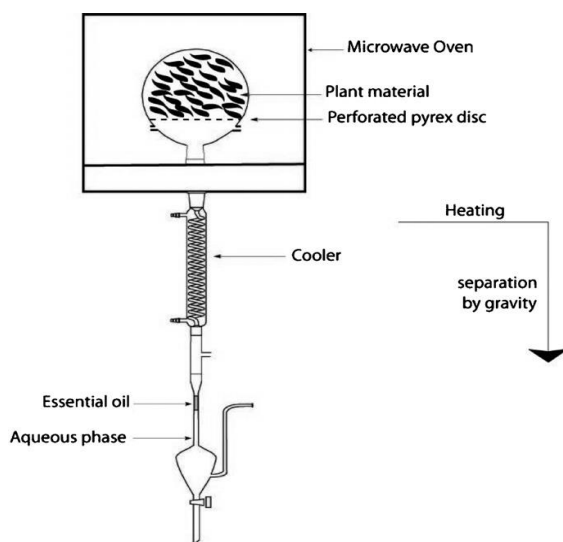


Fig. 11: Microwave hydro diffusion and gravity (MHG)

Conclusion:

Essential oils are natural products which consist of many volatile molecules. They have been used for several applications in pharmaceutical, cosmetic, agricultural and bioactivity example flowers. Extraction of essential oils could be carried out by various techniques. Have Innovative methods avoid shortcomings of content optional techniques to reduced chemical risk, extraction time and high energy input and obtain yield quality of essential oils. Despite their numerous application, except if essential oils are very sensitive to environmental factors used as such.

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