

# Extraction of essential oil by Microwave-assisted extraction: A review

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## ABSTRACT

*Essential oil is concentrated hydrophobic liquid which containing volatile aromatic compounds extracted from flowers, leaves, stems, roots, seeds, barks, resins, or fruit rinds. Extraction of essential oils is one of the most time and effort consuming processes. In this work, the volatile compounds of different plant material were extracted by Microwave-assisted extraction (MAE) techniques. Gas chromatography/mass spectrometry was used to identify and quantify the volatile compound composition. MAE has been recognized as a technique with several advantages over other extraction methods, such as reduction of costs, extraction time, energy consumption, and CO<sub>2</sub> emissions. MAE technique is a green technique which is highlighted by increased extraction yield, decreased time and solvent consumption, moreover the reproducibility is better. For this purpose, various process parameters such as solid loading, water volume, microwave power and extraction time were studied in detail.*

**Keyword :** - *Hydrophobic liquid, Green technique, Solid loading*

## 1. INTRODUCTION

Essential oils are concentrated volatile aromatic compounds produced by plants, the easily evaporated essences that give plants their wonderful scents. Each of these complex precious liquids is extracted from a particular species of plant life. Each plant species originates in certain regions of the world, with particular environmental conditions and neighboring fauna and flora [1].

Essential oils are frequently referred to as the "life force" of plants. Unlike fatty oils, these "essential" oils are volatile, highly concentrated, substances extracted from flowers, leaves, stems, roots, seeds, bark, resin or fruit rinds. The amount of essential oils found in these plants can be anywhere from 0.01 % to 10 % of the total. That's why tons of plant material are required for just a few hundred pounds of oil. These oils have potent antimicrobial factors, having wide range of therapeutic constituents. These oils are often used for their flavor and their therapeutic or odoriferous properties, in a wide selection of products such as foods, medicines, and cosmetics. Essential oils cannot be substituted with synthetics. Only pure oils contain a full spectrum of compounds that cheap imitations simply cannot duplicate [2].

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. The traditional technologies pertaining to essential oil processing are of great significance and are still being used in many parts of the globe. Water distillation, water and steam distillation, steam distillation, cohobation, maceration and effleurage are the most traditional and commonly used methods. Maceration is adaptable when oil yield from distillation is poor. Distillation methods are good for powdered almonds, rose petals and rose blossoms, whereas solvent extraction is suitable for expensive, delicate and thermally unstable materials like jasmine, tuberose, and hyacinth. Water distillation is the most favored method of production of citronella oil from plant material [2].

### 1.1 Essential oil sources

Plant organs containing natural essential oils. Essential oils are generally derived from one or more plant parts, such as flowers (e.g. rose, jasmine, carnation, clove, mimosa, rosemary, lavender), leaves (e.g. mint, *Ocimum* spp., lemongrass, jamrosa), leaves and stems (e.g. geranium, patchouli, petitgrain, verbena, cinnamon), bark (e.g. cinnamon, cassia, canella), wood (e.g. cedar, sandal, pine), roots (e.g. angelica, saffras, vetiver, saussurea, valerian), seeds (e.g. fennel, coriander, caraway, dill, nutmeg), fruits (bergamot, orange, lemon, juniper), rhizomes (e.g. ginger, calamus, curcuma, orris) and gums or oleoresin exudations (e.g. balsam of Peru, balsam of Tolu, storax, myrrh, benzoin)[3].

Depending upon the plant family, essential oils may occur in specialized secretary structures such as glandular hairs (Labiatae, Verbenaceae, Geraniaceae), modified parenchymal cells (Piperaceae), resin canals (conifers), oil tubes called vittae (Umbelliferae), lysigenous cavities (Rutaceae), schizogenous passages (Myrtaceae, Graminae, Compositae) or gum canals (Cistaceae, Burseraceae). It is well known that when a geranium leaf is lightly touched, an odor is emitted because the long stalked oil glands are fragile. Similarly, the application of slight pressure on a peppermint leaf will rupture the oil gland and release oil. In contrast, pine needles and eucalyptus leaves do not release their oils until the epidermis of the leaf is broken. Hence, the types of structures in which oil is contained differ depending on the plant type and are plant family specific. Unfortunately, not enough is known even today about these oil secretary structures to carefully categorize them. Based on the currently available information, it may be inferred that oils of the Labiatae, Verbenaceae and Geraniaceae families are the only superficial oils known; consequently, the others are considered subcutaneous oils [2].

## 2. METHOD FOR EXTRACTION OF AN ESSENTIAL OIL

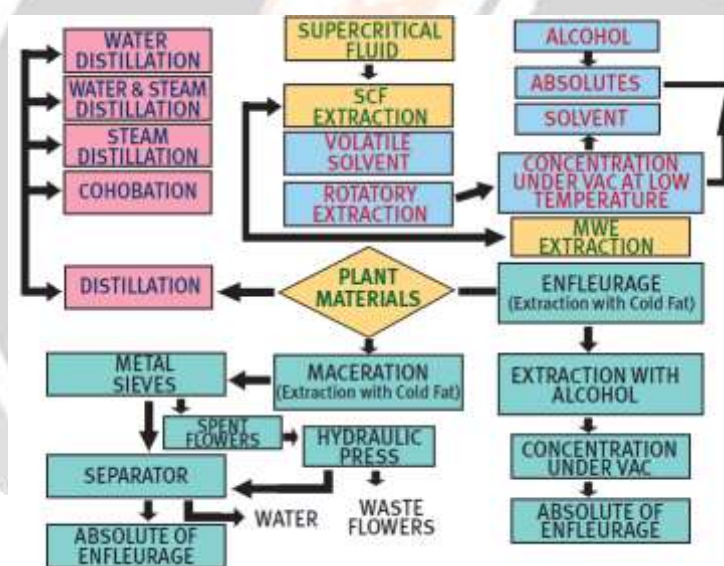


Fig-1: Methods of producing essential oils from plant materials [1]

Methods for producing essential oils from plant materials are summarized in Fig-1. Regarding hydrodistillation, the essential oils industry has developed terminology to distinguish three types: water distillation; water and steam distillation; and direct steam distillation. Originally introduced by Von Rechenberg, these terms have become established in the essential oil industry. All three methods are subject to the same theoretical considerations which deal with distillation of two-phase systems. The differences lie mainly in the methods of handling the material. Some volatile oils cannot be distilled without decomposition and thus are usually obtained by expression (lemon oil, orange oil) or by other mechanical means. In certain countries, the general method for obtaining citrus oil involves puncturing the oil glands by rolling the fruit over a trough lined with sharp projections that are long enough to penetrate the epidermis and pierce the oil glands located within outer portion of the peel (ecuelle method). A pressing action on the fruit removes the oil from the glands, and a fine spray of water washes the oil from the mashed peel while the juice is extracted through a central tube that cores the fruit. The resulting oil-water emulsion is separated by centrifugation. A variation of this process is to remove the peel from the fruit before the oil is extracted. Often, the volatile oil content of fresh plant parts (flower petals) is so small that oil removal is not

commercially feasible by the above mentioned methods. In such instances, an odorless, bland, fixed oil or fat is spread in a thin layer on glass plates. The flower petals are placed on the fat for a few hours; then repeatedly, the oil petals are removed, and a new layer of petals is introduced. After the fat has absorbed as much fragrance as possible, the oil may be removed by extraction with alcohol. This process, known as enfleurage, was formerly used extensively in the production of perfumes and pomades [1].

### 2.1 Modern (Non-traditional) Methods for Extraction of an Essential Oils

Traditional methods of extraction of essential oils have been discussed and these are the methods most widely used on 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 [2].

These techniques are as follows:

- Headspace trapping techniques
  - Static headspace technique
  - Vacuum headspace technique
  - Dynamic headspace technique
- Solid phase micro-extraction (SPME)
- Phytosol (phytol) extraction
- Protoplast technique
- Simultaneous distillation extraction (SDE)
- Controlled instantaneous decomposition (CID)
- Thermomicrodistillation
- Molecular spinning band distillation
- Membrane extraction
- Accelerated solvent extraction (ASE)
- Ultrasound assisted extraction (UAE)

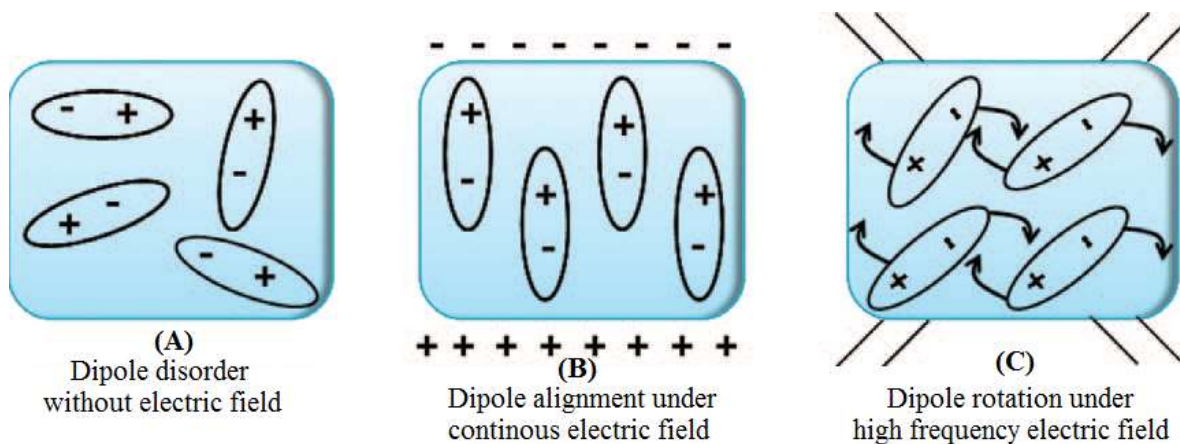
## 3. MICROWAVE-ASSISTED EXTRACTION (MAE)

### 3.1 Principle of Microwave Heating

Microwave radiation interacts with dipoles of polar and polarizable materials. The coupled forces of electric and magnetic components change direction rapidly (2450 MHz). Polar molecules try to orient in the changing field direction and hence get heated. In non-polar solvents without polarizable groups, the heating is poor (dielectric absorption only because of atomic and electronic polarizations). This thermal effect is practically instantaneous at the molecular level but limited to a small area and depth near the surface of the material. The rest of the material is heated by conduction [1].

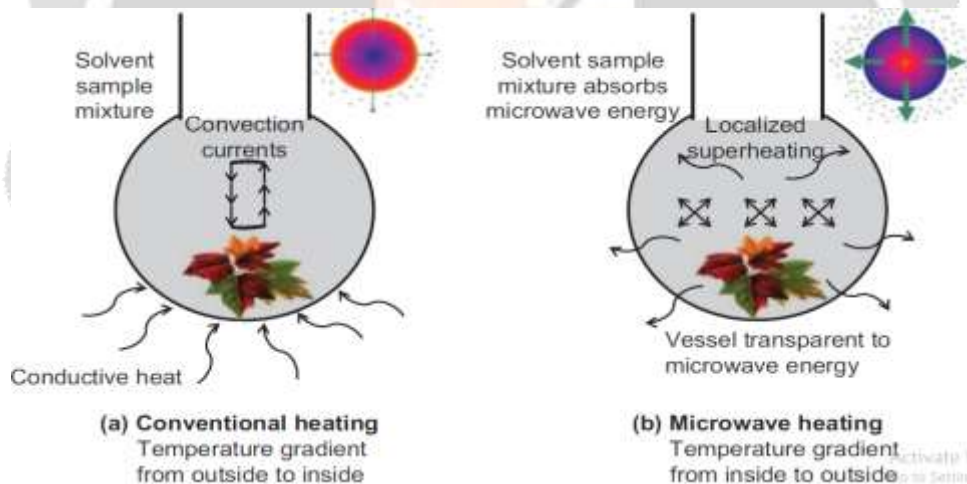
Thus, large particles or agglomerates of small particles cannot be heated uniformly, which is a major drawback of microwave heating. It may be possible to use high power sources to increase the depth of penetration. However, microwave radiation exhibits an exponential decay once inside a microwave absorbing solid [2].

In the microwave heating process, energy transfer occurs by two mechanisms: dipole rotation and ionic conduction through reversals of dipoles and displacement of charged ions present in the solute and the solvent. In many applications these two mechanisms occur simultaneously. Ionic conduction is the electrophoretic migration of ions when an electromagnetic field is applied, and the resistance of the solution to this flow of ions results in friction that heats the solution. Dipole rotation means realignment of dipoles with the applied field. At 2450 MHz, which is the frequency used in commercial systems, the dipoles align and randomize  $4.93 \times 10^9$  times per second and this forced molecular movement results in heating [2].



**Fig-2:** Dipolar molecules behavior: (A) without electric field, (B) under continuous electric field, and (C) under high frequency electric field [1]

Energy transfer is the main characteristic of microwave heating. Traditionally, in heat transfer of the conventional process, the energy is transferred to the material by convection, conduction, and radiation phenomena through the external material surface in the presence of thermal gradients. In contrast, in MAE, the microwave energy is delivered directly to materials through molecular interactions with the electromagnetic field via conversions of electromagnetic energy into thermal energy [2].



**Fig-3:** Representation of two heating modes: (1) by convection, (2) by microwave energy [1]

### 3.2 Advantages of Microwave heating

- Improved “existing” products
- Increased marker recovery
- Increased purity of the extract
- Reduced heat degradation
- Reduced processing costs
- Significantly faster extraction
- Much lower energy usage
- Much lower (order of magnitude) solvent usage
- Potential for “new” products

### 3.3 Drawbacks of Microwave-assisted extraction

- Extraction solvent must be able to absorb microwaves
- Clean-up step needed
- Waiting time for the vessels to cool down

### 3.4 Parameters in Microwave Assisted Extraction

- Selection of Solvent and Solvent-to-Feed Ratio (S/F)
- Effect of Extraction Time and Cycle
- Effect of Microwave Power and Extraction Temperature
- Effect of Contact Surface Area and Water Content
- Effect of Stirring

## 4. COMPARISON OF MICROWAVE-ASSISTED EXTRACTION WITH OTHER EXTRACTION TECHNIQUES

To introduce bioactive plant extracts in pharmaceutical and cosmetic formulations, industries are looking for green and efficient extraction processes free of toxic solvents. Methodologies using biodegradable and nontoxic solvents such as water and ethanol are being developed. The traditional techniques of solvent extraction of plant materials are based on the correct choice of solvents and the use of heat or/and agitation to increase the solubility of the desired compounds and improve the mass transfer. Soxhlet extraction is the most common and is still used as a standard in all cases. As a result of several secondary metabolites, the development of high performance and rapid extraction methods is an absolute necessity.

The new extraction techniques with shortened extraction time, reduced solvent consumption, increased pollution prevention, and with special care for thermolabile constituents have gained attention. In the many published papers comparing MAE with other advanced and conventional extraction methods, MAE has been accepted as a potential and powerful alternative for the extraction of organic compounds from plant materials. The ideal extraction technology depends on the type of compound to be extracted, whereas the extraction method efficiency is based on the highest recovery, especially of the effective constituents, the shortest processing time, the lowest production cost, and use of minimum organic solvent. There have been numerous reviews and research on the advances of different extraction techniques, comparing their results.

In the extraction of bioactive compounds from plants, MAE was reported to be more efficient compared to conventional techniques such as Soxhlet and advanced methods of extraction including ultrasound-assisted extraction (UAE), pressurized liquid extractions (PLE), and supercritical fluid extraction (SFE), which have emerged as energy-saving technologies. Over the years the procedures based on MAE have replaced some conventional extraction methods and have been adopted over decades in laboratories and industry.

In addition, the progress in microwave extraction gave rise to other categories of techniques to improve its performance: (1) microwave-assisted distillation (MAD) for the isolation of essential oils from herbs and spices; (2) microwave hydrodiffusion and gravity (MHG), a combination of microwave heating and distillation at atmospheric pressure that requires less energy and no solvent and simply combines microwaves and earth gravity at atmospheric pressure; (3) vacuum microwave hydrodistillation (VMHD), which uses pressures between 100 and 200 mbar to evaporate the azeotropic mixture of water–oil from the biological matrix; (4) microwave-integrated Soxhlet extraction (MIS), a combination of microwave heating and Soxhlet; and (5) solvent-free microwave extraction (SFME), based on the combination of microwave heating and distillation, which is performed at atmospheric pressure. If these techniques are explored scientifically, they can be proven to be efficient extraction technologies for ensuring the quality of herbal medicines worldwide.

As already mentioned, MAE is increasingly employed in the extraction of natural products as an alternative to traditional techniques of extraction for several reasons: reduced extraction time, reduced solvent consumption, and less environmental pollution as a result of increased efficiency and clean transfer of energy to the matrix, improved extraction yield and product quality, because materials can be rapidly heated, and often processed at lower temperatures; up to 70% energy saving compared to conventional energy forms from the high energy densities and the direct absorption of energy by the materials; compact systems, as small as 20% of the size of conventional

systems; and selective energy absorption resulting from the dielectric properties of the material and applicator design.

On the other hand, some disadvantages can also be mentioned: additional filtration or centrifugation is necessary to remove the solid residue after the process; the efficiency of microwaves can be poor when the target compounds or solvents are nonpolar, or when they are volatile; and the use of high temperatures that can lead to degradation of heat-sensitive bioactive compounds.

#### 4.1 MAE Versus Soxhlet Extraction

Soxhlet is the typical technique and the main reference for evaluating the performance of other solid–liquid extraction methods as it has long been one of the most used solid–liquid extraction techniques. In Soxhlet extraction the solid material containing the solutes is placed inside a thimble holder, which is connected to a flask containing the extraction solvent, and submitted to reflux. After this process, the extract is concentrated by evaporation of the solvent. This method has a large dependence on plant characteristics and particle size, as the internal diffusion may be the limiting step during extraction, and extraction and evaporation temperatures affect the quality of the final products.

It is a general and well-established technique, which surpasses in performance other conventional extraction techniques except, in a limited field of applications, the extraction of thermolabile compounds. Furthermore, it presents other disadvantages such as poor extraction of lipids, long operation time, high solvent consumption, and operation at the solvent's boiling point. The advantages of this method include no requirement of a filtration step after leaching and the displacement of transfer equilibrium by repeatedly bringing fresh solvent into contact with solid matrix.

Studies show that MAE allows the reduction of time and solvent consumption, as well as improvement in global yield. Kapas A. et al. extracting whitanolides from *Lochroma gesneroides*, showed a drastic reduction in solvent usage (5 vs. 100 ml) and in extraction time (40 min vs. 6 h). Another study concluded that the same quantity and quality of tanshiones from *Salvia miltiorrhiza* Bunge was obtained with 2 min of MAE and 90 min of Soxhlet. Higher yield was obtained when extracting artemisinin from *Artemisia annua* L. by MAE; in 12 min, 92.1% of artemisinin was recuperated by MAE whereas several hours were needed by Soxhlet to reach only about 60% extraction efficiency.

#### 4.2 MAE Versus Supercritical Fluid Extraction (SFE)

For green extraction, the use of SFE is very attractive because the solute is easily recovered and the solvent can be recycled by the simple manipulation of parameters such temperature and/or pressure. Supercritical fluids present liquid-like densities, whereas their viscosity is near that of normal gases and their diffusivity is about two orders of magnitude higher than in typical liquids.

Carbon dioxide CO<sub>2</sub> is the most used solvent in SFE because it is safe, nontoxic, and generally available at a reasonable cost. However, even at high densities, CO<sub>2</sub> has a limited ability to dissolve highly polar compounds. The addition of modifiers to CO<sub>2</sub> can improve the extraction efficiency by increasing the solubility of the solute in the solvent.

The ease of tuning the operating conditions to increase the solvation power makes this technology a good option for the selective recovery of several types of substances. This combination of properties makes SFE an important process in the food, pharmaceutical, and cosmetic industries because it is possible to fabricate products without toxic residues, with no degradation of active principles, and with high purity. Thus, SFE can be a fast, efficient, and clean method for the extraction of natural products from vegetable matrices.

Compared to SFE, MAE has a disadvantage, because cleanup is usually needed for this relatively selective technique. However, method development is often more complex in SFE and additionally sample throughput is not as high as in MAE. Furthermore, the efficiency of MAE can be poor when either the target compounds or solvents are nonpolar, or when they are volatile. According to Raghavan S., drying of the samples can be avoided for sample preparation with MAE, whereas samples are usually dried before SFE.

From the economic point of view, MAE is feasible as it requires moderate cost for equipment setup and is much cheaper as compared to SFE. Moreover, MAE has low risks and no major safety issues as most extractions are generally carried out under atmospheric condition. Several studies compared SFE and MAE. Thostenson et al. extracted artemisin from *Artemisia annua* L. by MAE, Soxhlet, and SFE. They found that MAE saves much time (12 min) and gives a high extraction rate (92.1%); SFE gives the lightest extract color but the lowest extraction yield while several hours were needed for Soxhlet. The same results were found by Chan C. et al. comparing MAE with SFE and Soxhlet.

The MAE gave the most concentrated extract with 8.15% of 5,8-dihydroxycoumarin (extract yield, 0.42%) from sweet grass. In addition, only 5 min gave the highest yield of triterpenoid saponins (0.968%), whereas SFE and UAE required several hours or even more than 10 h and gave a lower yield.

#### **4.3 MAE Versus Ultrasound-Assisted Extraction (UAE)**

Ultrasound-assisted extraction (UAE) in the food industry has been the subject of research and development; its emergence as a green novel technology has also attracted attention to its role in environmental sustainability. Ultrasound has been used in various processes of the chemical and food industries; it is a rapid technique, consumes small amounts of fossil energy, and allows reducing solvent consumption, thus resulting in a purer product and higher yields.

The principle of high-power ultrasound has been attributed to the acoustic cavitation phenomenon that appears when high-intensity acoustic waves are generated in a fluid. The extraction mechanism involves two types of physical phenomena: diffusion through the cell walls and washing out the cell content once the walls are broken. Ultrasound waves modify their physical and chemical properties after their interaction with subjected plant material, and their cavitation effects facilitate the release of extractable compounds and enhance mass transport by disrupting the plant cell walls. Developments in ultrasound technology and its potential benefits have triggered interest in the application of power ultrasound on a wider range of chemistry processing.

The combination of sonication and microwaves was studied for extraction of lipids from vegetables and microalgae sources. Ultra sonication alone, microwave irradiation alone, or a combination of both techniques gave excellent extraction efficiencies in term of yield and time, with a tenfold reduction in the time needed with conventional methods, and increase of yields from 50% to 500%. MAE possessed higher efficiency (11.62%) for the extraction of triterpene saponins from yellow horn (*Xanthoceras sorbifolia* Bunge.) compared with UAE (6.78%) and reflux extraction (10.82%).

#### **4.4 MAE Versus Pressurized Liquid Extraction (PLE)**

Pressurized liquid extraction (PLE), also referred to as pressurized solvent extraction (PSE) and accelerated solvent extraction (ASE), is now well accepted as an alternative to Soxhlet extraction and has been successfully used to isolate antioxidants from plants, such as thermolabile anthocyanins from jaboticaba (*Myrciaria cauliflora*).

The use of the PLE technique is an attractive alternative because it allows fast extraction and reduced solvent consumption. This technique allows the use of solvents or solvent mixtures with different polarities under high pressures (up to 20 MPa), keeping the extraction solvent in the liquid state, and temperatures ranging from room temperature up to 200°C.

The pressurized solvent at a determined temperature is pumped into an extraction vessel containing the sample matrix. Using high temperature accelerates the extraction process by increasing the solubility of the analyses in the solvent and thus increasing the kinetic rate of desorption of the solute from the sample matrix; this occurs because the pressurized solvent remains in the liquid state well above its boiling point, allowing high-temperature extraction. Considerable increase in the mass transfer rates results from the decrease of viscosity and superficial tension of the solvent.

Moreover, the use of high temperatures, which on the one hand increases extraction rates, on the other hand may lead to degradation of thermolabile compounds. PLE uses liquid solvents; therefore, its basic principle is considered

similar to those of classic extraction. Partly because these newer technologies are automated and the solvents are under “superheated” conditions (the effect of microwaves in MAE or elevated temperature or pressure in PLE), they are more user friendly, much quicker, and require significantly less organic solvent. Although in PLE the filtration step is “included” in the process, in MAE a cleanup step is often needed. MAE is considered an easy technique, and compared to SFE and PLE, it is less expensive. Although good recovery rates were obtained with both extraction methods, MAE provided advantages with regard to sample handling, cost, analysis time, and solvent consumption.

## 5. CONCLUSION

MAE has been recognized as a technique with several advantages over other extraction methods, such as reduction of costs, extraction time, energy consumption, and CO<sub>2</sub> emissions. MAE technique is a green technique which is highlighted by increased extraction yield, decreased time and solvent consumption, moreover the reproducibility is better. This extraction method is rapid compared to conventional method like hydrodistillation, soxhlet extraction. MAE technique produced more oxygenated compounds, is more cost effective and environmental friendly. These results indicate that cinnamon oil extracted through MAE method exhibit better properties especially in terms of quality when with conventional hydrodistillation technique. It also suggests that MAE is suitable for extracting volatile oils from different plant material without necessarily causing any adverse change to the chemical composition of the oil.

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