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Microwave Assisted Hydrodistillation – An Overview of Mechanism and Heating Properties

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ABSTRACT

The efficiency of heating mechanism of microwave assisted hydrodistillation (MAHD) in the extraction of essential oil is presented. The theoretical study on the microwave heating mechanism is also reported in terms of dielectric properties, volume rate of heat generation and penetration depth. MAHD extraction method has the advantages of being rapid, less solvent consuming, environmental friendly and green technology. Detailed information on the heating mechanism of MAHD is therefore presented herein alongside the potential of MAHD.

INTRODUCTION

Microwave assisted hydrodistillation (MAHD) is an emerging and innovative advanced technique which has been used for extraction of volatile oils from plant materials. There are a lot researches which have been conducted using MAHD to extract essential oil from various types of plants. Some of the extracted plant materials includes *Thymus vulgaris L.* (Golmakani and Rezaei, 2008), *Schisandra chinensis* Baill fruits (Ma *et al.*, 2012), aromatic herbs (Filly *et al.*, 2014) and etc. Extraction through MAHD has been observed to hold numerous potentials for producing good quality and high yield of essential oils. The mechanism of microwave is associated with the irradiation of microwave energy which then turns into heat energy. This is then used for heating the solvents and plant materials during extraction thereby accelerating the extraction kinetics. The advantages of MAHD includes high quality of products, shorter extraction period, usage of less amount of solvent, faster extraction rate, cheap and environmental friendly (Delazar *et al.*, 2012). The application of microwave in extraction of essential oil began around the 1980s. Following the development of the technology of microwave assisted extraction method, it has recently become one of the most famous extraction techniques which have been regarded as fast and economically feasible. This paper presents an overview of the mechanism of microwave in extraction process, properties of its heating mechanism as well as its potential for essential oil extraction.

Theory Of Microwave:

Microwave refers to non-ionizing radiation waves with frequency range of 300 MHz to 300 GHz and wavelengths from 1 cm to 1 m. It is situated between X-ray and infrared rays in the electromagnetic spectrum (Letellier and Budzinski, 1999a). Initially, the two main applications of microwaves are as energy vectors and

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for communication purposes. However, it was later developed to be use in applications of food technology especially for the purpose of heating. Microwave is formed through two perpendicular fields which are magnetic field and electric field (responsible for heating) (Letellier and Budzinski, 1999a). Generally, conventional heating depends on convection and conduction processes in which case most of the energy is being lost or release to the environment. However, in the case MAHD, heating specifically takes places in selective and targeted materials. There is therefore no significant heat loss to the environment as the process occurs in a closed system. This excellent mechanism may practically shorten the extraction period (Huie, 2002).

The basic scientific principle of heating using MAHD depends on its direct interaction with polar solvents. During microwave heating, two phenomena are called to play which are ionic migration and dipole rotation. Most often, these phenomena occurs simultaneously (Letellier and Budzinski, 1999a; Letellier *et al*., 1999b). Ionic migration is the electrophoretic conduction of ions by the influence of changing electric field. This phenomenon occurs when a solution offers resistance to the migration of ions thereby producing friction which may bring about the heating up of the solution. On the other hand, dipole rotation stands for the realignment of dipoles of a molecule to an oscillating electric field. Heating is generally effective at a microwave frequency of 2450 MHz with wavelength of 12.2 cm and the domestic microwave is operated at this conditions. The electric component of the wave changes rapidly at 4.9×10^4 times per sec at this frequency (Mirza *et al*., 2003). At this frequency, the solvent molecule tries to orientate itself with the electric field in order to maintain itself in the same phase. Unfortunately, rapidly changing electric component of the wave causes the molecules to fail its reorientation thereby causing it to vibrate vigorously and produce heat through frictional force.

On the other hand, when the microwave frequency is higher than 2450 MHz, the electrical component of the waves increases its speed. Due to this, the solvent molecules do not get the opportunity or enough time to initiate orientation with the external electric field. Thus there is no occurrence of friction for the creation of any heating process. In another vein, if the frequency is lower than 2450 MHz, the electrical component of the waves decreases its speed and as a result, the molecules get excessive time to align itself with the electric field and therefore become slowed down. Thus there would be no occurrence of heat at all. This indicates that only the dielectric solvents or materials that have permanent dipoles can be heated up via microwave. The heating efficiency of the solvents in microwave is based on the dissipation factor (tan δ). Dissipation factor refers to the capability of the solvent to absorb microwave energy and convert it to heat energy which is transferred to the surrounding molecules (Mirza *et al*., 2003). The dissipation factor can be obtained as illustrated in Eq. 1.

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \qquad (1)$$

where, tan δ is the dissipation energy, ε'' is the dielectric loss and ε' is the dielectric constant.

The dielectric loss refers to the ability to convert microwave energy to heat energy whereas dielectric constant stands for measure of the ability to absorb microwave energy. The dielectric properties of some commonly used solvents in extraction are tabulated in Table 1. From the table, it can be seen that solvents like ethanol and methanol exhibit lower ability to absorb microwave energy compared to water as depicted by their lower value of ε'. However, the overall heating efficiency for both solvents are higher than for water as depicted by their greater tan δ values. On the other hand, hexane and other less polar solvents such as chloroform are transparent to microwave and therefore do not generate heat.

Table 1: Dielectric properties of commonly used solvents in extraction (Desai *et al*., 2010)

Solvents	Dissipation factor at 2.45 GHz (tan δ)	Dielectric constant[a] (ε')	Dipole moment[a] (debye)
Acetone	-	21.4	-
Ethyl acetate	-	6.02	1.88
1-Butanol	0.571	-	1.66
Ethanol	0.941	25.7	1.69
Methanol	0.659	33.7	1.70
Diethyl ether	-	4.389*	-
Hexane	-	1.88	<0.1
Chloroform	-	4.8	-
Water	0.123	80.4	1.84

[a] represents determined at 20 ^{0}C and * at 18 ^{0}C

Principle of Mahd Extraction:

When heat is applied via microwave to the moisture (water) inside the cells of plant materials, there is evaporation, expansion and subsequently generation of high pressure on the oil gland cell wall (Wang *et al*., 2006). The generated internal pressure pushes out the oil gland cell wall, and intensively stretches out the wall to the extent that it might become ruptured. This would ease the process of release or leach out of essential oil from the plant material to the surrounding solvent (water). This phenomenon can be enhanced if the plant material is soaked in solvents like water with high microwave heating efficiency (higher dissipation factor value). Cellulose which is the active constituents in most of the plants may therefore be transformed into soluble fractions within few minutes. The high temperature absorbed by the plant cell wall may enhance the dehydration process of cellulose and decrease the mechanical strength of the plant. As a result, the solvent from the surrounding can easily enter into the cell wall (Latha, 2007).

A number of researches have been conducted to investigate the morphology of oil gland cells of some plants before and after MAHD extraction. These glands were observed with the help of scanning electron microscopy (SEM) techniques. In a particular research changes to the oil glands of *Thymus vulgaris* L. was studied (Golmakani and Rezaei, 2008). Similarly, surface of orange peel oil gland after MAHD and HD extraction was investigated (Ferhat *et al*., 2006). These studies shows that the oil cell glands after MAHD extraction shows mild disruption of the oil cell gland which was associated with the nature of heat distribution during MAHD extraction.

During MAHD extraction, the microwave energy is being highly absorbed by water (high dielectric properties). This energy is then converted into heat energy which is thereafter transferred to the plant material (Desai *et al*., 2010). Transfer of this heat energy can bring about a localization of heat at desired parts of the plant material especially the portion containing the oil gland. The oil glands can therefore conveniently release its oil without experiencing much damaging rupture as shown in Figure 1. This is not the case with conventional methods such as HD in which case higher levels of damaging rupture are often experienced by the glands of the plant material as can be seen in Figure 2. This have been attributed to the nature of heat distribution by HD in which case heat energy first reaches the solvent surface before heating the targeted plant material (Desai *et al*., 2010). This can therefore lead to explosive rupture of the glands in order to retrieve the oil (Ferhat *et al*., 2006; Golmakani and Rezaei, 2008).

Generally, the effect of microwave irradiation is strongly influence by the dielectric properties of both the plant matrix and solvent (Zuloaga *et al*., 1999). In most cases, the plant material is soaked into single solvent or a mixture of solvents that have high dielectric properties prior to the extraction in order to strongly absorb the microwave energy (Zuloaga *et al*., 1999). As the microwave irradiation intensity increases, the temperature rises alongside with it thereby easing the process of penetration of the solvent into the plant matrix. Essential oils are therefore released to the surrounding solvent. However, in some special cases, where the crude extract contains highly thermolabile components, the plant material is usually soaked into solvents which are transparent to microwave. These solvents such as hexane and chloroform can therefore help to prevent degradation of the bioactive constituents (Letellier and Budzinki, 1999a; Letellier *et al*., 1999b; Wang, *et al*., 2006; Zuloaga *et al*., 1999).

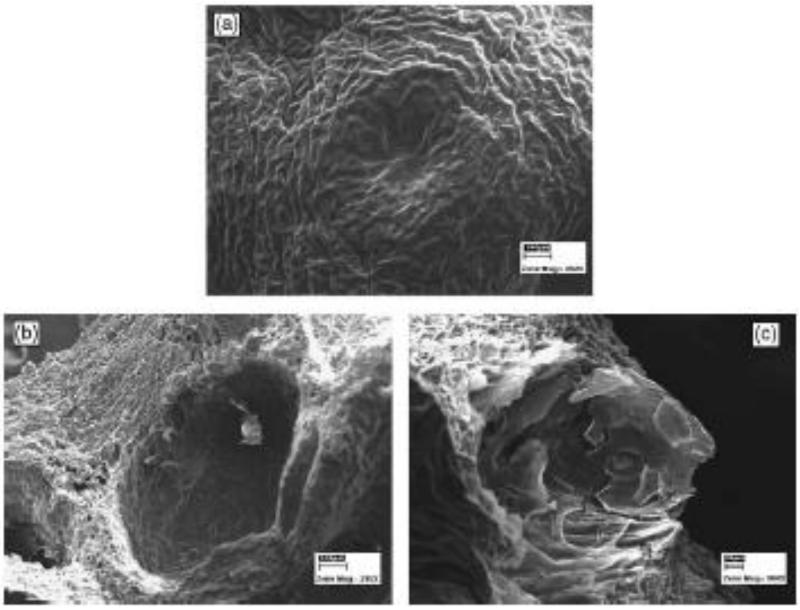

Fig. 1: Scanning electron micrographs of orange peel (a) untreated, (b) after MAD for 30 min and (c) after HD for 3h (Ferhat *et al*., 2006).

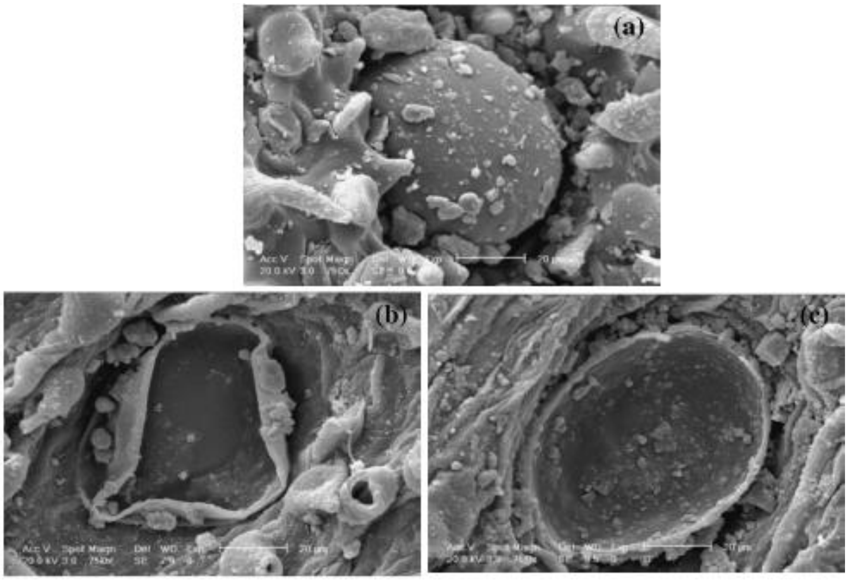

Fig. 2: Scanning electron micrographs of thyme leaves for (a) untreated, (b) after HD (60 min) and (c) after MAHD (30 min) (Golmakani and Rezaei, 2008).

Principle Sketch Of Microwave Assisted Hydrodistillation:

The schematic diagram of lab scale microwave assisted hydrodistillation was illustrated in Figure 3. The choosen plant materials will be soaked in particular amount of water for a particualar period of time. According to Jeyaratnam *et al*. (2016c), it was reported that pre-treatment process like soaking process may affect the yield of essential oil. The reason provided is that soaked plant materials absorbed water in certain time and becomes swollen. Therefore, the expanded and swollen plant materials may ease the process of realising essential oils from the plant materials to the water during extraction process (Jeyaratnam *et al*., 2016c). After the plant matrix (plant material + water) reach the boling point, it will start to evaporate through the clevenger. The mixture of essential oil and water which is in vapour state pass through the condenser where it is converted from vapour to aqueous phase. The aqueous phase then accumulates at the end of the clevenger tube. The excess water may return into the plant matrix through water reflux tubing which ensure the plant matric has sufficient water for the extraction process.

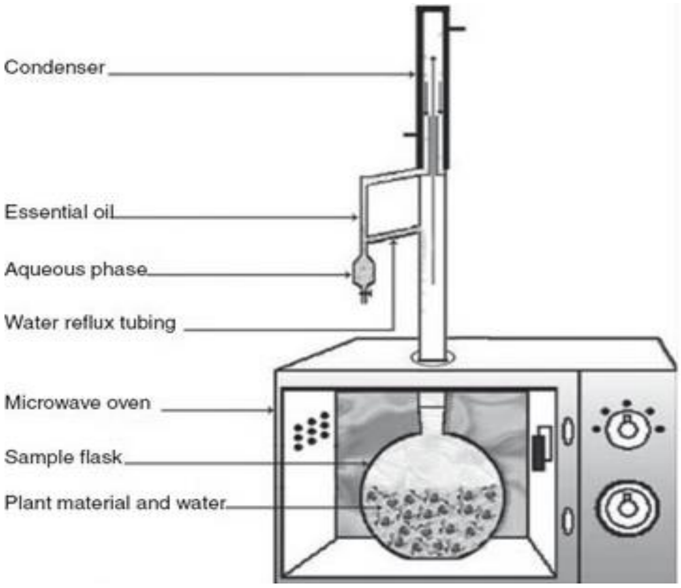

Fig. 3: Schematic diagram of lab scale microwave assisted hydrodistillation (Jeyaratnam *et al*., 2016a)

Mechanism of Microwave Assisted Hydrodistillation:

The basic scientific mechanism of the MAHD method is different from those of conventional methods. Extraction in MAHD takes places with the aid of electromagnetic waves which changes the structure of oil gland cells. There is a synergistic effect of two transport phenomena such as heat and mass transfer which are acting in the same direction. This may therefore result in rapid production of extracts from the plant material (Chemat *et al*., 2006). In the case of conventional method, both mass and heat moves in opposite direction. Thus, the mass transfer takes place from inside to outside whereas heat is transferred from the surrounding (outside) to inside of the substrate as illustrated in Figure 3. Furthermore, in conventional extraction heat is being supplied from the heating source to the interior of plant material sample, whereas, in MAHD heat is volumetrically distributed inside the irradiated medium.

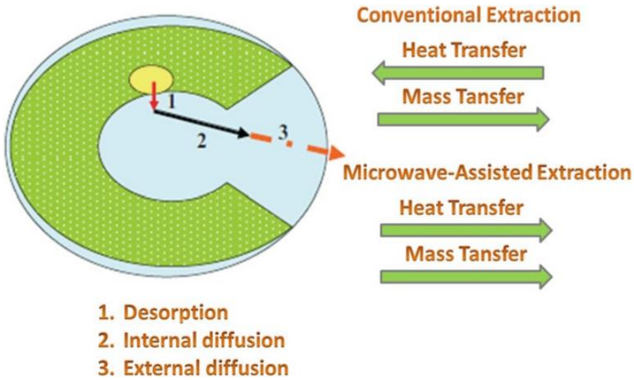

Fig. 3: Fundamental mechanism of mass and heat transfer in conventional and microwave extraction (Veggi *et al*., 2012)

Heat Transfer In Microwave Assisted Hydrodistillation:

Generally, due to the water content in plant material, they may be termed as poor electrical insulators. In essence, they can store and distribute energy when microwave is irradiated on them. However, the microwave energy radiated is in itself not a thermal energy. Rather, the heating is a combination of electromagnetic radiation formed with the dielectric properties of the materials when it is subjected to electromagnetic field. Dielectric properties therefore act as an essential parameter in determining the interaction between plant matrix and the electric field (Wang *et al*., 2006). The rate of conversion of electrical energy into heat energy in plant materials is described in the following Eq. 2. (Latha, 2007).

$$P = K.f\varepsilon' E^2 tan\delta \qquad (2)$$

where, P is microwave power distribution per volume unit, K is a constant, f is the frequency applied, ε' is the dielectric constant of material used, E is the electric field strength and $tan\delta$ is the dielectric loss tangent.

It is assumed that heat transferred from the plant material to the surrounding solvent (water) is a rapid process which considers water and the plant matrix to have the same temperature (Latha, 2007). Hence, the volume rate of heat generation of plant matrix is calculated using energy balance equation illustrated in Eq. 3.

$$q_{MW} = \frac{hA}{V}(T - T_a) + \frac{\varepsilon\sigma A}{V}\left[(T + 273.16)^4 - (T_a + 273.16)^4\right] + pC_p\left(\frac{dT}{dt}\right) \qquad (3)$$

where, $\frac{hA}{V}(T - T_a)$ is heat loss by convective heat transfer to the surrounding, $\frac{\varepsilon\sigma A}{V}\left[(T + 273.16)^4 - (T_a + 273.16)^4\right]$ is radiative heat loss from the wall surface and $pC_p\left(\frac{dT}{dt}\right)$ is heat accumulation in the cinnamon matrix.

The contribution of the first and second terms from the Eq. 3 is very much smaller compared to the third term. Besides that, the heat produced by the glass reactor is assumed to be negligible as it has only minimum mass at the same low dielectric constant. Therefore, in calculating the volume of heat generation, q_{mw}, the density (ρ) and heat capacity (C_p) of the plant matrix were calculated based on the mixing rule.

After eliminating the heat loss by convective and radiative heat loss, the volume rate of heat generation can be derived. Volume rate of heat generation is a critical term for analyzing the efficiency of microwave heating properties. This term interrelates temperature rise and dielectric properties of the plant matrix. Volume rate of heat generation of the matrix can therefore be calculated using the following formula.

$$Q_{mw} = \rho_{mix}\, x\, C_{p\,mix}\, x\, \frac{dT}{dt} \qquad (4)$$

where, Q_{MW} is volume rate of heat generation, cal/ cm^3.sec, ρ_{mix} is the density of plant matrix mixture, g/cm^3, $C_{p\,mix}$ is heat capacity of plant matrix, cal/ ^{0}C.g and $\frac{dT}{dt}$ is the rate of temperature increase, ^{0}C/ sec.

To apply Eq. 4, the density (ρ) and specific heat capacity (C_p) of the matrix mixture can be calculated by the following simple mixing rules:

$$P_{mix} = \rho_W \emptyset + \rho_s (1 - \emptyset) \quad (5)$$
$$C_{p\,mix} = C_{p,W} \emptyset + C_{p,s} (1 - \emptyset) \quad (6)$$

Where, Φ is the volume fraction.

Generally, the rate of heat generation is influenced by the dielectric properties. Three dielectric properties that are predominantly used in microwave heating are tangent loss, dielectric constant and dielectric loss. Tangent loss can be measured by dividing dielectric loss by dielectric constant. The dielectric constant and dielectric loss of a solvent such as water is calculated by using the following models illustrated in Eq. 8 and 9 respectively. The value of dielectric constant and dielectric loss of the plant material (cinnamon bark powder) was reported as 2.8 and 0.38 respectively by (Khan and Chandel, 2011) at 2450 MHz.

Simple mixing rule was then applied to calculate the total dielectric properties.

$$\varepsilon'_w = 85.215 - 0.33583T \quad (7)$$
$$\varepsilon''_w = 320.685T^{-1.0268} \quad (8)$$

where, ε'_w is the dielectric constant of water, ε''_w is the dielectric loss of water and T is the temperature (^{0}C).

On the other hand, penetration depth, D_p at a particular frequency is influenced by the dielectric properties of material matrix in an inverse manner. The depth of penetration is defined as the depth into the plant matrix at which the power density has decrease to 1/e (about 37 %) of its surface value. Eq. 10 illustrates the penetration depth:

$$D_p = \frac{\lambda_\circ\, x \sqrt{\varepsilon'}}{2\pi\varepsilon''} \quad (9)$$

where, $\lambda_\circ$ is the wavelength of microwave energy at 12.24cm for 2450 MHz.

Potential of Microwave Assisted Hydrodistillation:

Before this time, the microwave energy has been employed in many fields such as in chemical synthesis (Budarin *et al*., 2015), biodiesel production (Gude *et al*., 2013), communication (Harel *et al*., 2015) and food processing **(**Puligundla *et al*., 2013). Recently, laboratory scale microwave has shown some good potential over conventional extraction methods such as hydrodistillation (Filly *et al*., 2014). Incorporating microwave energy into hydrodistillation is however thought to be a great idea for obtaining synergistic benefits from combination of the two approaches. Therefore, a number of researches have been carried out to explore the potential of microwave assisted hydrodistillation in extraction processes. The potential of microwave assisted hydrodistillation was explored in extraction applications such as extraction of essential oil from *Cinnamomum cassia* (cinnamon) (Nitthiyah *et al*., 2016a; Nitthiyah *et al*., 2016b), Patchouli (*Pogostemon Cablin*) leave (Kusuma and Mahfud, 2015), *Rosemarinus offcinalis* (rosemary) (Karakaya *et al*., 2014) and ginger (*Zingiber Officinale Roscoe*) and lemongrass (*Cymbopogon Citratus*) (Ranitha, 2012). Results from these researches indicated that microwave assisted hydrodistillation shows better performance especially in terms of quality of essential oil obtained compared to those obtained from other extraction techniques. Other features that were reportedly derivable from microwave assisted hydrodistillation technique include rapid extraction, cost effectiveness, energy saving, and possibility for obtaining higher oxygenated compounds. Specifically, the higher percentage of oxygenated compounds obtained through microwave assisted hydrodistillation extraction technique is believed to possess great medicinal benefits. This is because it aids the production of natural aroma in essential oil from the plants materials (Kusuma and Mahfud 2015; Geng *et al*., 2011; Nitthiyah *et al*., 2016c; Shan et. al., 2006). In another vein, Nitthiyah *et al*. (2016b) reported that cinnamon oil obtained at optimum conditions through microwave assisted hydrodistillation produces higher active compound compared to conventional hydrodistillation method. This large quantity of active compo4und can therefore help to ease the process of isolation of the active compound (Nitthiyah et. al., 2016b). Similarly, Kusuma and Mahfud (2015) stated that extraction of Patchouli leave through microwave assisted hydrodistillation produces higher chemical compounds which is about 19 components compared to hydrodistillation method just around 14 components. The report further stated that although both extraction techniques did preserve the volatile active compounds, microwave assisted hydrodistillation technique produced additional new four compounds (Kusuma and Mahfud 2015).

Based on these reports, it can be inferred that microwave assisted hydrodistillation technique can be helpful in discovering new chemical compounds that may facilitate the discovery of new drugs. Furthermore, technological revolution towards green extraction concept and the demands for new energy saving, cheap and technically feasible technique makes microwave assisted hydrodistillation a highly desirable method. Among the several important benefits which may be derived from the use of MAHD, environmental friendliness due to non-consumption of organic solvents makes it the method of preference.

Future Trends of Microwave Assisted Hydrodistillation:

Hydrodistillation (HD) had been commonly used conventional extraction technique which has also been used to obtain essential oils. However, HD has been reported to alter the chemical compounds especially the monoterpene compounds present in the extracted oil. Aside from this, longer extraction time and the need for high energy makes HD less desirable.

Therefore, microwave extraction was invented by incorporating microwave and hydrodistillation method, which has been perceived to possess several potential benefits. This method is therefore being constantly improved to meet industrial demands. Improvement to this method brought led to the discovery of microwave assisted hydrodistillation (MAHD). Technology revolution towards green extraction concept and the demand for energy saving, cost effective and technically feasible techniques makes this method highly desirable. Apart from other important potential advantages associated with MAHD, it is recently the method of preference because it is environmental benign due to non-consumption of organic solvents.

Similarly solvent free extraction (SFE) is another important advanced technique which may be projected to replace MAHD in the near future. However, the high operating cost and need to optimize many operating parameters often limit the use of this mehod. In addition, the high pressure and temperature which accompanies SFE often hampers full recovery of the essential oil. Although the extracts obtained via SFE usually possesses high yield with good oil quality, economical issues and complex operating process are usually the limiting factors.

The use of MAHD is fast becoming a household due to its many advantages. It has also been employed for obtaining essential oil.

Conclusion:

This paper describes the heating mechanism of MAHD in terms of dissipation factor, dielectric properties, rate of conversion and volume rate of heat generation. This advanced extraction technique has great potentiality compared to other conventional methods due to its' excellent heating properties which helps to accelerate the essential oil production. In short, the benefits which may be derived from MAHD includes but not limited to time and energy saving which leads to overall cheap cost. It also has the advantage of better quality and higher yield of essential oils. Besides that, if other factors such as extraction time, irradiation energy, volume and type of solvent are properly controlled, there is a greater prospect for MAHD technique.

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