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# **Review: A Comparison of Conditions for The Extraction of**

# Vegetable and Essential Oils Via Microwave-Assisted Extraction

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Abstract. Microwave Assisted Extraction (MAE) has been utilized in extracting a myriad of phytochemicals, lipids, carbohydrates, and proteins. This extraction method essentially takes advantage of water present inside cells by exciting the molecules hence increasing the internal temperature and pressure till the cells bursts. MAE is studied extensively due to its advantages over other typical extraction methods; especially in essential oil extraction. However, there is not many researches on using MAE to extract vegetable oil. Essential and vegetable oils are both derived from plants, but the characteristics of both oils differs. Therefore, the proper conditions for MAE extraction of the oils are different. This paper reviews the MAE conditions such as extraction time, power of microwave, solvent to feed ratio, and the set-up of microwave equipment. This paper attempts to show the differences between the MAE conditions between vegetable and essential oil extraction. The challenges faced by MAE is also discussed briefly with suggestions to overcome them. The Internet of Things is also discussed for its implementation in a scaled up MAE reactor.

Keywords: Microwave assisted extraction; lipid; phytochemicals; essential oil; vegetable oil

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#### 1. Introduction

Microwave-assisted extraction (MAE) has been utilised in extracting both essential oil (EO) and vegetable oil (VO). However, due to the different characteristics of EO and VO, the parameters of the extraction between the two are different. VO is defined as fats extracted from either the seeds of a fruit or a vegetable or from other parts of plants [1]. EO is defined as volatile substances which is extracted via hydrodistillation or mechanical method that is mainly made up of terpenoid which is responsible for the scent, odour, or smell in plants [2][3]. Some important parameters that need to be considered for which are the solvent, solvent volume, extraction time, and microwave power [4]. Therefore, these parameters are discussed in this paper.

#### 2. Properties of VO and EO

#### 2.1 Composition of VO and EO

Generally, the major composition of vegetable oils are fats whereas the composition of essential oils are terpene/terpenoids [2]. VO is mainly comprised of glycerides with 97 % triacylglycerides [1]. The different type of fatty acids that are attached to the triacylglycerides generally determines the properties of the VO [1]. VO liquid state in room temperature is attributed to the high density of unsaturated fatty acids [1]. A lower melting point VO would usually contain C<sub>6</sub>-C<sub>12</sub> fatty acids [1]. For example, coconut oil extracted from coconut meat contains saturated fatty acids which are made up of mostly medium chain fatty acid [5]. A large portion of it is made up of lauric acid, a 12 carbon long fatty acid [5]. EO on the other hand, comprised of mainly the phytochemical terpenoid with only 1-10 wt% of the composition identified to be non-volatile substances [2]. The composition of EO includes terpenes derivatives, alcohols, esters, and ketones [2]. Interestingly, essential oil could be extracted from different parts of a plant like the extraction of flavonoids and tannins from coconut leaves [6]. For example, orange oil, an EO deriving from the peels of oranges, has a composition dominated by limonene; a terpene [7].

#### 2.2 Volatility and viscosity

Volatility is affected by the vapour pressure of the liquid where a high vapour pressure is an indication of high volatility [8] Vapour pressure is determined by the intermolecular force (IMF) of the liquid and also its molecular mass [8] Liquids with large IMF and molecular mass tend to have low vapour pressure hence lower volatility [8]. Essential oils are also known as volatile oils [9]. This is due to the fact that terpenoids or terpene derivates, the majority of the constituents of EO, is volatile [2]. Vegetable oils like olive, coconut, and palm oil are also known as non-volatile oil as those oils do not evaporate at room temperature and stay in liquid form due to IMF between fatty acids [1] [10]. If the oil is non-volatile, more often than not, it is viscous. For example, palm oil, a type of non-volatile oil, has a viscosity of 0.46 x10-1 Pa.s at 30 °C [11]

#### 2. Comparison of parameters in MAE

## 3.1 Extraction time

Table 1 shows the data for the extraction of several EO and Table 2 shows the data for the extraction of several VO done by several studies. Unless stated, the unit of the yield from the studies as shown in equation 1.

(1)

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 $\frac{\text{Oil extracted } (g)}{\text{Dry weight of sample } (g)} = \text{yield } (\%)$ 

Most of the studies of essential oil extraction via MAE are more than 30 minutes and VO extraction, less than 30 minutes. The long time for EO extraction via MAE is may be due the extraction is kept on going until the volume of EO extracted was constant [12] [13] [14].

#### 3.2 Microwave power

Generally, the higher the power, the larger the yield of oil [15]. Some studies utilised irradiation power that are not optimised like the maximum power was used to extract lemon essential oil [16]; which is unnecessary even if the objective of the study was to compare the heat and mass transfer of different MAE mode of operations. Whereas, optimised conditions like that was done, the studies showed that high power is not needed and the extraction duration is lower than conventional extraction duration [17] [18] [13]. A high microwave power would incur solvent losses the longer the extraction due to solvent boiling [19].

#### 3.3 Solvent and solvent to feed ratio (SF)

There is a glaring difference between the solvent used in VO and EO MAE extraction where EO extraction utilised only water as solvent and some studies do not even use solvents for the extraction [20] [21]. This method is known as Solvent Free MAE (SFME). From the Table 1, it can be observed that EO extraction usually uses a lot of solvent when compared to VO extraction. It is true that the more solvent used, the greater the mass transfer of oil to solvent hence increasing yield. However, if the feed or samples are fully immersed and there is sufficient contact between the feed and solvent, the volume would be adequate [15]. Other than that, a higher amount of solvent would also be economically inefficient due to a higher energy requirement to heat up, cool down, and separating the solvent from the extracted compound [22]. Thus, optimisation of SF ratio is important as the amount of solvent to be used and its recovery is a limitation in terms of cost [23].

It can be observed that there is a difference between the utilised solvents between EO and VO. VO extraction via microwave used organic or non-polar solvents whereas EO essentially used only either water or steam. So, it is clear that EO extraction is much safer, cheaper, and environmentally friendlier than VO extraction. It is interesting to note that EO, like VO, is also non-polar; i.e. it will not dissolve in water. EO's composition is made up of terpenoid which is volatile and therefore contributes to EO's volatility [24]. Due to this, and also the fact that terpenoids are also thermolabile, hydrodisillation is used in most of EO's extraction methods which would preserve the components of EO that would otherwise decompose at high temperatures [4].

#### 3.4 Microwave setup

The setup of the extraction equipment is an important factor for the effectiveness of MAE. Many setups have been devised such as gravity MAE, solvent free MAE, and steam diffusion MAE [25]. Commercial MAE equipment serve advantages in controlling other parameters that may be effective in increasing yield like RotoSYNTH's rotating vessel, higher power (Mars 6, CEM), and controlling pressure (Discover SP).

It can be seen from the table EO MAE are ex-situ extraction where EO is extracted outside the vessel in which the feed or material was in. Figure 1 shows the typical setup of MAE for EO where sometimes the collector would be replaced by a clavenger apparatus. A modification of this set up was done where the setup was in an inverted position where instead of the water vapour and EO going up, the extraction was assisted by gravity and went down [25]. This is known as the gravity assisted MAE. It is also possible for EO extraction to not include solvents where this setup is known as solvent free MAE [26] [20] [27]. Modifications play a part in yield. This was proved by extracting orange essential oil by 4 different MAE methods where it was found out that 92% of the total oil could be extracted by using the gravity assisted

Table 1 Essential oil extraction via MAE

IOP Conf. Series: Materials Science and Engineering 778 (2020) 012172 doi:10.1088/1757-899X/778/1/012172

hydrodistillation MAE. Other method had lowered yield due to excessive heating and also the increased solubility of EO in water [25].

On the other hand, VO MAE are in-situ where VO extracted stays in the vessel in which the feed or material was in. All VO extraction from the table are in-situ with the exception of hazelnut oil extraction where the oil was obtained by pressing after microwave treatment [28]. As for the other studies, the solvent would then be evaporated usually by rotary evaporator [29]. It would be unlikely for VO extraction MAE setup to mirror the setup for EO unless it is to extract the EO present in VO. This is due to the volatility of the oil. At room temperature, it can be observed that a typical VO like coconut oil, soybean oil, and olive oil would not evaporate as opposed to EO like lavender oil or lemongrass oil where these two would evaporate. Therefore, the setup of the MAE should be taken into account and prior to that, what type of oil is to be extracted. A good setup would be one that would aid in the dispersal of solvent and microwave throughout the feed.

e	it to Micro	wa 1	Microwave set up		Yield (%,	Reference
ttio ve r (W)	2	wer	-		(m/m)	
/w) 500		H	Ex-situ (Co-axial MV nydrodistillation)	, ,	0.75	[30]
g of 100		I	Ex-situ (Dry-diffusion ar gravity assisted)	pr	2.59	[13]
f feed 200 25 g steam		I s	Ex-situ (steam distillatic and gravity assisted)	ų	4.42	[31]
/v) 600			Ex-situ (hydrodistillation	$\overline{}$	1.31-1.39	[12]
500		щ	Ex-situ (Milestone Di list, gravity assisted)	5	$0.33 \pm 0.09$	[14]
ut 400 up till ices ple		<u> </u>	Ex-Situ (hydrodiffusic ınd gravity)	ų	92 (oil/max oil)	[25]
up till ices ple		<b>H</b>	3x-Situ (steam diffusion)	-	67 (oil/max oil)	[25]
600		H O	5x-situ (hydr listillation)	6	1.9437 ± 0.0888 (70.1456 % oil/max oil)	[18]
600		H O	Ex-situ (air hydr listillation)	6	$\begin{array}{c} 2.7678 & \pm \\ 0.1993 \\ (99.8863 \ \% \end{array}$	[18]

4





Figure 1 Typical MAE setup for EO extraction



Figure 2 Typical MAE setup for VO extraction

Reference	[28]	[17]	[32]	[33]	[29]
Yield (%, w/w)	45.3 (g oil/ max oil g)	$36.89 \pm 0.29$	52	36	62.97
Microwave set up	Ex-situ (Hydraulic press post treatment)	In-situ (5 times interval heating, 30 seconds rest for each interval)	In-situ (Milestone RotoSYNTH)	In-situ (condenser on top connected to vessel inside the modified microwave)	In-situ (modified with mechanical stirrer)

Table 2 Vegetable oil extraction via MAE



#### 3. Problems faced by MAE

Some of the problems faced by this type of extraction are (1) solvent must be able to absorb microwave [34]. One should mix non-polar solvent with polar solvents to increase the effectivity of the solvents to absorb microwave [33]. (2) Thermolabile substances extraction [34]. Interestingly, it was suggested that transparent solvents are recommended for the extraction of thermolabile substances, which would explain why most studies for EO extraction via MAE used water [4]. (3) Other solvents other than water may be hazardous although there are studies from the table which used solvents such as n-hexane without any problems [35]. (4) Overpressure and leakage [4]. There are commercial microwaves that can control pressure and detect leakage [4]. However, modifying household microwaves do not have this luxury unless they are fitted with pressure and leak sensors. Other sensors that could be fitted are thermocouples (to monitor the temperature of the microwave) and thermographic camera (to observe heat distribution). (5) Non-polar solvents are unable to absorb microwave effectively [19]. This is akin to running the microwave empty; if the vessel used to contain the solvent does not absorb the microwave effectively [36]. This would damage the magnetron beyond repair and would cause arcing in the cavity or along the waveguide [35]. To detect a weak magnetron, household microwave manufacturers would provide a benchmark on how long would it take for water to boil. This method however, is painstakingly slow [37]. Another way to detect failing magnetron is via connecting it to an ohm meter between the terminals of a magnetron where a good magnetron would read less than 1  $\Omega$  when the ohm meter is set to the lowest resistance [37]. Other than that, by connecting a magnetron's terminal and metal casing to an ohm meter at the highest resistance scale, a reading of infinity would indicate a good magnetron [37]. (6) arcing. To prevent arcing, the lining of the cavity and the waveguide must be shielded. This should also be applied to sensors that are exposed directly to the microwave [38]. Recommended metals to prevent arcing are such as molybdenum, stainless steel, and

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platinum [38]. Problems 4 and 5 may not be so important in a lab scale setup, but to scale MAE up and for it to be viable for the industry, these problems are important to be addressed.

#### 4. Advanced analytics and Internet of Things as a solution

Previously, it was mentioned that many sensors are needed to maintain a setup of a scaled up MAE. Studies of MAE pilot scale [39] [40]. The MAE reactor used by [39] was equipped with a rotatable drum which the feed is inserted in, a sensor that sense microwave and sends signals to adjust the microwave power if the power is too low, a resistance temperature detector, and an interlock to prevent accidental openings. The MAE reactor used by [40] was a continuous process (fitted with a pump) instead of a batch. At the end of the reactor, a thermocouple was fitted unto and at the start of the reactor, an infrared probe was added [40]. For safety, a vent was added in the case of over-boiling and a release valve was added in the case of blockage [40]. Other than the sensor installed in the reactor by [39] to adjust the microwave power, there is no mention of both reactors being automated and there are no sensors that monitor the electrical part of the reactor which would make maintenance a difficult task.

Internet of Things (IoT) is the utilisation of the internet to control and monitor devices remotely by having the devices send and receive data [41]. Advanced analytics is used in IoT by gathering data from sensors to be utilised for predictive analytics [42]. The implementation of predictive analytics would aid in automation and the implementation of new hardware [42]. This would also make predictive maintenance possible which helps in the optimisation of maintenance whether it be predicting when a part is going to malfunction, optimal time for maintenance, process optimisation, and to design new facilities [43]. By implementing IoT with predictive analytics in scaled up MAE reactors, it would be possible to optimise the process effectively with less time consumed and monitor the parts of the MAE reactors for any loss of performance; without being overwhelmed by large amounts of data.

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