Effect of Microwave Power on the Yield of Essential Oil from Lavender

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ABSTRACT

Currently, microwave-assisted extraction (MAE) is the most widely used method to extract essential oil from several types of plants. MAE is considered to provide a shorter period for extraction and needs less amount of solvent compared to conventional methods. Hence, it should be an environment-friendly approach. In this particular study, the extraction of lavender oil was undertaken using the MAE method. This study aimed to investigate the effect of microwave power on the yield of essential oil from lavender. The experiment was conducted using a Miniflow 200SS instrument at microwave power ranging from 50 W to 200 W. The extraction temperature and ratio of material to solvent were constant at 100°C and 1:2 respectively. The maximum weight of lavender was approximately 15g. The highest yield of 1.8673%±0.054 was obtained with 175 W for 595 sec of microwave heating. The optimum microwave power was 150 W where the sample consumed 25.24 Wh and presented a yield of about 1.8346 ±0.049%. The obtained results have shown that microwave power has a slight
improvement in the yield at higher microwave power if the same amount of energy is consumed by the materials.

**Keywords:** Extraction, Microwave-assisted Extraction, Essential Oil.

1. INTRODUCTION

Lavender, a fragrant herb is a genus of 39 species of flowering plants in the Mint family (scientifically known as Lamiaceae) [1]. Lavender oil is one of the most useful medicinal essential oils for its applications in the food and fragrance industries. The typical pain-reducing effect of the application of lavender oil is believed to be due to its main compounds such as linalyl acetate and linalool which have analgesic properties [2]. Commercially, the lavender provides many important essential oils especially to the fragrance industry, in terms of shampoo, lotions, perfumes, and other cosmetics. In food industries, lavender oil is used as one of the major ingredients of ice cream and flavoring beverages [3, 4]. Essential oils have wide industrial applications which make them a great market for farmers due to the growing demand in the UK. The world growth in industrialization has increased the global market for essential oils to 2.6 billion dollars, with an annual growth rate of 7.5% [5].

Separation technologies, such as distillation, membrane, and extraction are known as the promise methods in the areas of innovation which can improve the development of sustainable processes in the chemical industry [6]. Several studies have been carried out on the extraction of essential oil from different materials including solvent extraction, ultrasound-assisted extraction, and Soxhlet. However, most such methods indicate lower extraction efficiency, higher extraction period, and large capital [7]. However, MAE is carried out within less time and with a reduced solvent ratio, which is considered as advantages of MAE over its alternative [6-11].
Extraction processes conducted under the action of microwave radiation are supposed to be affected to a certain extent by the main benefits of microwave extraction volumetric and selective heating [12]. Additionally, other advantages in microwave extraction are effective heating without any heat contact, shorter time for the dissipation of energy, smaller equipment, better yield, quicker response to the process heating control, and simplicity of the process [3]. Although microwave technology showed great advantage, they are still mainly limited to laboratory applications. Due to economic and environmental issues, there are difficulties in using the latest technologies to produce oil from plant materials. This is because of the continuing demand for reducing energy consumption and CO\textsubscript{2} emissions [7, 13].

Over the past decade, various studies have been undertaken in separation processes using microwave extraction to isolate biological compounds. Some of these applications are extraction of essential oil from lavender [3], extraction of the flavonoids from Radix Astragali [14], extraction of phenolic compounds from olive leaves with several kinds of solvents [15], isolating the volatile compounds from Nigella Sativa seeds [16], extraction of pectin from waste Citrullus lanatus fruit rinds [17], extraction of polysaccharides from mulberry leave [18] and extraction of herbicides in soybean samples [19].

Advances in MAE have led to the development of various techniques such as nitrogen-protected microwave-assisted extraction (NPMAE), vacuum microwave-assisted extraction (VMAE), microwave accelerated steam distillation (MASD), solvent-free microwave extraction (SFME), microwave by hydro diffusion and gravity (MHG) microwave hydrodistillation (MWHD) [9, 11]. VMAE is preferable to be conducted at low temperatures and vacuum conditions. In this method, extraction takes a longer time which attributable to the mild condition. Also, It has been observed that the development of the solvent-free microwave extraction method indicates a higher yield from plant materials and dried materials before moistened [20]. However, SFME is more preferable to extract the essential oil owing to its efficiency compared to the conventional method.
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[21]. This method could not be conducted in this study as the solvent was needed to be used to avoid burning the raw materials.

Also, a study by Sahraoui et al. [14] concluded that the yield of essential oil from lavender, extracted by microwave steam distillation (MSD) was considerably higher than the oil extracted by the SD method. Another study was carried out to extract the essential oil from Nigella Sativa by MAE; reported that this process is a promising technology to isolate the essential oils in terms of saving energy and time as well as less environmental impacts [19]. In this study, MAE was selected based on the advantages explained above; such as, reducing the energy consumptions and extraction time which results in a smaller footprint needed for separation units using this technology.

There are many important factors in microwave extraction which have been being investigated such as the volume of solvent, power level, temperature, heating period, and pretreatment of feedstock. Higher microwave power will generally improve productivity in a shorter time and lower energy consumption, result in smaller extraction equipment [10, 22]. Based on these hypotheses, this study will focus on the effect of microwave power on the yield of lavender oil. Several reports on applications of MAE have shown that when the microwave power increases, the yield increases until a certain level of microwave power. However, the chemical composition of essential oils might be affected by microwave power based on the plant materials [16, 17, 19, 23-25]. Nevertheless, MAE is based on the localized heating which is provided by the microwave power to destroy the plant matrices result in allowing the volatile compounds to diffuse out to the solvent.

In MAE the process is fast and has high productivity. This is due to the consequences of a combination of both phenomena, mass and heat gradients cooperating in the same direction. In contrast, the normal extraction heat moves into the substrate whereas the mass comes out of it. In conventional methods, the heat transfers as a result of heating medium.
to the interior of the material. On the other hand, in MAE the heat is distributed volumetrically in the irradiated medium [12]. Microwave energy breaks hydrogen bonds, because of the dipole rotation of molecules induced by microwave and movement of ions in the solution [20].

2. AIMS AND OBJECTIVES

This study aimed to investigate the effect of microwave power in the microwave-assisted extraction method which was used as an alternative to the conventional method (steam distillation) to extract the essential oil from lavender flowers.

The objective was to examine the effects of microwave power ranging from 50 to 200 W, on the yield of essential oil with different extraction times, to consume the same energy for each sample.

3. MATERIAL AND METHODOLOGY

3.1. Sample preparation

Experiments were conducted using lavender collected from local gardens in Nottingham. Upon deliverance to the laboratory, the flowers were stripped, weighed, and stored at a temperature of 10°C in order to keep it fresh for the next experiments. A 30 mL of pure lavender oil was used for the calibration curve.

3.2. MAE apparatus and procedure

The experiment was conducted with the Miniflow 200SS microwave equipment which allowed the measurement of reflected power to calculate the total power observed by the sample and it is presented in figure 1. This equipment is capable of producing a very low controlled power output (0 to 200 W with 1 W power increments) together with the use of a precisely defined frequency range from 2,425 to 2,475 MHz.

Based on the aim of this study it was important to determine the quantity of energy transferred to the sample and it could be determined by calculating the difference between the emitted and the reflected power observed when irradiating a sample. This is possible due to the use of a
solid-state microwave generator as opposed to a magnetron. The equipment used a fiber optic probe to measure the temperature.

![Figure 1: Miniflow 200SS microwave instrument [26]](image)

At first, approximately 15 g of fresh lavender and 30 mL of water (the solvent) were added to an extractor (Figure 2, A), which was transferred to the microwave instrument’s cavity (figure 2, B and C). The extractor was connected to a cold trap which was covered by ice (figure 2, D, and E). The cold trap was used in order to prevent vapors from being evacuated from the experiment. Before turning the Miniflow 200SS ON, the extraction conditions such as temperature, power, and extraction time were set-up for each experiment. Microwave power was varied ranging from 50 to 200 W. The extraction time was decreased from 2081 and 521 sec respectively in accordance with the power levels mentioned above. Throughout the study, the extraction temperature was fixed at 105°C and the solvent to lavender flower ratio was fixed at 2:1. For each experiment, the microwave leakage was checked after the microwave was turned ON using the HI-1600 microwave oven survey meter as a safety check. At the end of each cycle, instruments were left to cool down. Lastly, the laboratory apparatus, through which the liquid was flowing, were washed by 10 mL of n-hexane. This was to get all the essential oil that might be sticking to the inner surfaces. The extracted liquid (water, lavender oil, and 10mL hexane) was kept in the fridge (10°C) for further analysis.
4. RESULTS

In this study, different microwave power has been used to evaluate the impact of microwave power on the productivity of lavender oil. The extraction time was varied to input approximately the same energy into the samples. The influences of different microwave power levels, from 50 W to 200 W, were examined in terms of microwave power effect on the yield. Figure 4 shows such an effect. At lower power levels of 50, 75, 100, and 125 W, there were no discernible changes in the yield. It is worth mentioning that there was a slight increment in productivity in the range of 150 to 200 W.

The most noticeable points of yield increase were from 1.4134±0.037% to 1.8346 ±0.049% for a microwave power increase from 125 to 150 W. The maximum yield of essential oil was about 1.8673%±0.054 at a microwave power of 175 W with a total elapsed time of 694 sec (11.567 min). However, at higher microwave power (200 W), there was a small drop in the yield to 1.6816±0.032%. Nevertheless, these yields were conducted in a short time, that is more energy-consuming compared to productivity and such drawbacks will be explained in section 5.
Although the results did not show a high yield, these results should be interpreted with extreme care. To achieve this, figure 5 was created to compare the influence of microwave power against the yield and the total energy absorbed. The most effective microwave power range was between 150 and 175 W, which shows that the highest yield was obtained at 28.02 Wh of energy consumption. The power of 150 W indicated a similar yield (1.8364±0.049%) as of 175 W (1.8673±0.054%) at lower energy consumptions (about 25.24 Wh). However, at microwave power (200 W), 26.98 Wh was absorbed by the sample, the yield had a small decrease of 0.29%. From figure 5 it can be seen 150 W was the optimum microwave power. Noticeably, lower energy of 25.11Wh was consumed by the sample at 100 W compared to 26.37 Wh at 50 W with a similar yield of 1.3715±0.028% and 1.3515±0.039% respectively. The reasons attributed to such results will be argued in section 5.
Figure 5: Yield and energy absorbed at different microwave power

The data illustrated in Figure 6 were obtained from the Miniflow 200SS instrument where it was clear that the temperature rise was more rapid at higher microwave power. The temperature of the sample reached 100°C in a shorter time of approximately one minute at a microwave power of 200 W. In contrast, at a low microwave power of 50 W, the mixture spent more time of about 5 minutes to reach the same temperature. The rapid increase in temperature increases the initial dissolution compared to conventional extraction. Nevertheless, there was a rise in temperature of the system during microwave extraction that enhanced the hydrolysis reaction and the diffusion. This might not be sufficient to enhance the diffusion of the hydrolyzed components to the solution during the early stages. These data supported that microwave extraction needed a much shorter time compared to the conventional methods.
This particular study's results agree with others in supporting the notion that MAE is a time saver technology but failed to identify any significant differences in the yield at various power levels. The next section thus moves on to discuss possible reasons for such results.

5. DISCUSSION

5.1. Optimal working conditions for MAE

This study was undertaken to investigate the effect of microwave power in improving the yield and minimizing the extraction time. To achieve this goal, experiments were conducted for a maximum time of 2081 sec (34.683 min) at a power of 50 W. Based on energy balance, the minimum microwave power required to evaporate approximately 54.45 g of water in 15 min was determined as 105 W, while the moisture in the lavender flower was around 63%. The preliminary tests have shown that solvent-free microwave extraction resulted in lower extraction time, despite that the lavender flowers slightly burned which attributed to the small amount of sample. Therefore, the solvent was needed to be used to conduct these experiments. Water was selected as a solvent because it is sufficiently polar and it would be easily heated by microwave irradiation. A ratio of 2:1 water to lavender was used as the optimum mixture.
Undoubtedly, the important benefits of MAE were rapidity and energy savings. The amount of water used was twice the weight of lavender which indicated that probably most of the microwave energy was consumed by water to be heated and vaporized. Consequently, only an insufficient fraction of energy might have been absorbed by the essential oils inside the lavender flowers resulting in poor yield and high energy consumptions.

5.2. Relation of yield with density
In this work, a calibration curve for the precise density measurement was used to obtain the yield of a tiny amount of isolated oil in the samples. The curve covered a wide range of lavender oil concentrations, ranging from 0.1 to 10%v in 10 mL hexane, it was accepted based on R squared value which was equal to 0.9921 as it is shown in Figure 7. The densities of samples were determined from the average of three repeated measurements by the Densito PX30 instrument. The maximum uncertainty of density measurement was calculated as ±0.0005 g/cm3. The small yield might be attributed to the small amount of the sample of fresh lavender of about 15 g contained by the small extractor.

![Figure 7: Theoretical densities against experimental densities](image)

These results of poor yield were related to the small density of samples which is likely to attribute to the evaporation of light components.
from flowers. The solution density was expected to be in between the densities of lavender oil and pure n-hexane which are 0.885 and 0.6548 g/cm³ respectively. However, this reason might not be valid since the yield was obtained from sample density. This can be explained by the major components of lavender oil including Linalool, linalyl acetate, camphor, and germacrene, etc., have a higher density than n-hexane [4]. Moreover, the lightest compound of lavender oil such as germacrene has a density of 0.793 g/cm³ which is higher than the density of pure hexane [27]. Therefore, a tiny amount of oil might have been isolated and a longer time should have been applied to achieve a higher yield.

5.3. Microwave power and energy consumption effects
During the experiments energy consumption increased up to 28.061 Wh, which could be sufficient enough to diffuse the essential oil out from the plant material, consequently increasing the yield [3, 6, 7, 9]. Most reports indicate that the yield of essential oil from lavender was above 9% [3, 11, 12]. In contrast, in this study, the maximum yield was just 1.8673±0.054%. There are several possible explanations for these results. First, it may be due to the use of a small amount of lavender leading to a tiny amount of oil extracted. Another possible explanation can be the lack of adequate time and microwave power to move the volatile oil out from the plant material. Thus higher microwave power might have been required to isolate the essential oil. At high microwave power, the sample would have reached the boiling point in a shorter time resulting in a sudden increase in the pressure inside the flowers which would have increased the mass transfer [17].

Chemat and Cravotto [12] argued that high microwave power might be the reason for poor productivity as a result of the degradation of thermally sensitive compounds. They stated that the power of 500 W and higher did not influence the productivity of flavonoids.

Another study to extract the essential oil from the aromatic herbs by Filly et al. [21] found that at microwave power between 250 to 500 W there was a loss of oxygenated compounds and the matrices were carbonized before the oil was isolated. Similarly, Song et al. [28] reported that high
microwave power of 350 W leads to degradation of phenols compounds. Furthermore, at higher microwave power and high temperature, impurities can also move into the solvent at fast rupture [12]. Sahraoui et al. [26], reported that at a temperature higher than 110°C the yield decreased. This was due to the stability of the flavonoids and degradation. In contrast, Chemat et al. [3] obtained a yield of 8.86% from 50 g of dry lavender by MASD at much higher power (1000 W) and extraction time of 900 sec. Thus, a longer time should have been applied in order to induce more energy at a microwave power of 200 W. It seems that the microwave extraction power needs to be moderated to reduce the extraction time and to reach the required temperature, hence preventing the samples to be burnt during the extraction process.

The negative result can be due to the fast heating that caused the loss of the volatile compounds. A study to extract volatiles from Nigella Sativa seeds by Liu et al. [16] supported this argument, they found that some oil was lost due to the fast heating. Ma et al. [24] examined the effects of power on the extraction of essential oil from Schisandra Chinensis Baill fruits, they found that when microwave power was increased there was a significant increase in reaction temperature which may result in carbonization of the raw materials due to internal overheating [16, 24]. Also, Farhat et al. [11] suggested that microwave power needs to be moderated otherwise the volatile compounds will be burned. There was an improvement in the production when higher microwave power was used at constant energy consumption. This can be explained by the faster rise in temperature at higher microwave power which increased the pressure inside the plant and caused the destruction of the sample's surface, consequently allowing the transfer of volatile compounds to the solvent and increasing the yield. Even though more energy was consumed at 200 W than at 150 W, the yield was slightly decreased from 1.8673±0.054% to 1.6816±0.032% (figure 4). It is believed that at a maximum microwave power of 200 W more time is required to dissipate the energy sufficiently, resulting in diffusing the volatile compound to the surface of plant material. Additionally, Sahraoui et al. [14], asserted that MAE of essential oil from plant materials with improved microwave
irradiation was connected to the direct impacts of the energy consumption by biomolecules through ionic conduction and dipole rotations. Hence, this results in power dissipation inside the solvent and plant material and thus producing heating and mass transfer. Another possible explanation of low yield obtained with the variation of microwave power might be the loss of chemical structure as a result of overexposure to microwave radiation. This reason might not be valid because based on energy balance adequate time was allowed as presented in figure 5. Liu et al. [16] suggest that microwave power alone does not have a clear effect on the energy consumption by the samples. According to Li et al. [19], the density of energy should be taken into account as a parameter of power level alone. Even at various power levels, the yield was small which might be a consequence of insufficient energy consumption by the samples. Therefore increasing extraction time to increase the energy consumption might result in higher yield.

6. CONCLUSIONS AND RECOMMENDATIONS

The present study was undertaken to investigate the effect of microwave power on the extraction yield of essential oil from lavender. The obtained results have shown that microwave power has a small effect on the yield of essential oil at higher microwave power levels at the same amount of energy consumption by the materials. Samples absorbed an unequal amount of energy at various microwave powers due to the reflection of power by the sample. The range of energy consumed was between 25 to 28 Wh at a different range of microwave powers. Therefore, it seems that an insufficient fraction of energy was absorbed by the lavender flower resulting in poor yield.

In this study, the yield of essential oil was found to be (1.8673%±0.054). The yield was approximately constant at low microwave power (below 125 W). There was a small improvement in the yield at higher microwave power (above 150 W), by about 0.3%. In regards to the environmental aspect, these results failed to support that microwave extraction is a green
technology. This is due to the high consumption of energy for low production.

Special attention is needed to be given to the extraction by MAE including the influences of many important factors such as temperature, solvent ratio, microwave power, and extraction time. These parameters should be studied to explore their interaction effects on the extraction of the essential oils, in order to select the optimum conditions. More experiments are needed to be carried out in order to evaluate the effect of microwave power by varying other parameters such as temperature and solvent type. The sample was extremely limited (15 g) by the size of the microwave cavity. Therefore, another possible area of future research would be to explore with a larger amount of sample using another instrument such as MiniLabotron 2000 for which the amount could be increased as much as 100 g. The increase in the volume of extracted oil could allow more analysis to be conducted, such as determining physical properties and gas chromatography (GC). This could show the effect of microwave power on the chemical composition of the essential oil. More research is needed to examine the links between energy absorbed by the plant materials and the yield using different approaches like microwave steam distillation and microwave by hydro diffusion and gravity.

REFERENCES


Effect of Microwave Power on the Yield of Essential Oil from Lavender


peels: Microwave hydrodiffusion and gravity. Journal of food Engineering, 90, 409-413.


Effect of Microwave Power on the Yield of Essential Oil from Lavender

saikosaponins from Radix Bupleuri. Separation and Purification Technology, 61, 266-275.


تأثير قدرة الموجات الدقيقة على إنتاجية الزيوت العطرية من وردة الخزامي

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الملخص

تعتبر تقنية الاستخلاص باستخدام الموجات الدقيقة من العمليات الأكثر استعمالًا في استخلاص الزيوت العطرية من مختلف الأزهار والنباتات. حيث أنها تحتاج لوقت أقل أي أنها تستهلك أقل طاقة. كما تحتاج إلى كمية قليلة من المذيب مقارنة بالعمليات الأخرى مثل التقطير بالبخار، ولذا تعتبر من الطرق الصديقة للبيئة. في هذا البحث سيتم دراسة تأثير قدرة الموجات الدقيقة على Miniflow 200SS إنتاجية زيت الخزامي. حيث أجريت هذه التجربة باستخدام جهاز يدعى Miniflow 200SS في مدى موجات دقيقة من 50 واط إلى 200 واط. والظروف التشغيلية المثلى هي 100°C ونسبة المذيب إلى المذاب هي 1:2 ووزن العينة من ورد الخزامي حوالي 15 جرام. حيث وصلت الإنتاجية إلى 0.054±0.018673% عند قدرة 175 واط ولمرة تسمى 596. أفضل قدرة إنتاجية كانت عند 150 واط حيث أصمت العينة مايقارب 25.2 واط ساعة وانتاجية 0.049±0.0346ه. تستنتج من هذه الدراسة أنه يوجد تأثير لقدر الموجات الدقيقة في زيادة إنتاجية الزيوت النباتية كلما زادت القدرة عندما تمت نسبة كمية الطاقة من قبل العينة.

الكلمات المفتاحية: الاستخلاص، الاستخلاص بالموجات الدقيقة، زيت أساسي.