Study of Eucalyptus Essential Oil Acquired by Microwave Extraction

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Abstract
Classical extraction of essential oil such as Soxhlet extraction and steam distillation is still a formidable and time-solvent consuming. Microwave assisted process (MAP) is used to accelerate the extraction process of target compounds from a variety of sources. It can be used for the extraction of compounds from various plants and animal tissues, or the extraction of undesirable components from raw materials.

Eucalyptus leaves were used in this study to investigate the applicability of microwave irradiation for essential oil extraction. The influence of microwave power and time of exposure on extraction efficiency were studied. A comparison between microwave extraction and Soxhlet extraction (using same solvent) in terms of time and yield has been carried out.

INTRODUCTION

Essential oils can be broadly defined as volatile oils which differ fundamentally from the fixed fatty oils such as linseed, coconut and olive in being more mobile and volatile. Many essential oils, apart from being used for first aid or for treatment of common complaints, are also ideal as bath oils, perfumes or room fresheners. Even when they are used purely for esthetic purposes, they are still fulfilling a positive preventive and therapeutic role (Julia Lawless, 1997). The term “volatile oils” includes not only those substances derived from plant and animal materials such as essential oils, but also substances such as lipids, fatty acids, etc. which not having the same degree of volatility as essential oils, are expressed or volatilized from the glandular of such plant and animal materials, by the microwave extraction process (Pare, 1994).

Conventional extraction of essential oils includes: tumbling and shaking, oil infusion, cold expression, steam distillation and Soxhlet extraction can take from hours to days, and requires significant amounts of solvent. Following the results of the various experiments carried out it becomes obvious that extraction using microwave technology is a good alternative to conventional extraction techniques. The extraction by microwave as a new extraction technique has its own specific parameters that need to be characterized for every plant that contains essential oil. Microwaves can heat the material of polar molecules, when a material is exposed to microwaves, the polar molecules within the matrix are excited then orient themselves in the direction opposite to the field. The frequency of the change of polarity of the electric field determines the frequency at which the molecules will rotate within the field. As a result of this rotation, temperatures in the vicinity of the rotating molecule increase. The applied field prevents the molecules from reaching an equilibrium position and thus the polar molecules store potential energy while they are within the field. When the field is removed, the molecules relax to an equilibrium position and release additional kinetic energy in the form of heat.

The applicability of microwave irradiation for the extraction of various types of compounds from plant material, food and soil was investigated by Ganzler (1986). The production of volatile material from plant exposed to microwave energy in an air stream has been discussed by Cravciro (1989). Chen (1994) studied the microwave heating characteristics of extraction of rosemary and peppermint leaves suspended in hexane, ethanol and mixture of hexane and ethanol. Pare (1995) patented a general extraction
method for biological matter using microwave energy and a microwave transparent solvent. Weseler et al. (2002) has studied the antibacterial activity of Australian tea tree oil against several strains of *Malassezia pachydermatis*. Different solvents such as ethanol, trichloromethane, cyclohexane, n-hexane, petroleum ether (30-60°C), petroleum ether (60-90°C), No. 120 solvent oil and No. 6 extraction solvent oil were used to study the effect of solvency on the extraction of artemisinin from *Artemisia annua* using microwave assisted process by Jin-yu Hao et al. (2002). Eucalyptus has been chosen for this study based on: low price, immense usage and well identified constituents.

**EXPERIMENTAL PROCEDURES**

**Materials and Chemicals**

Standard samples (cineol, α-pinene, camphene, cymene, limonene and γ-terpinene) of high purity (purchased from Sigma-Aldrich) were used in this study for GC calibration. The calibration was done by preparing known concentrations of each component in ethanol solution. Plants leaves (Fig. 1 and 2) of eucalyptus were supplied by Nasuha Enterprise plantation (Malaysia). Ethanol of 96 v/v % was supplied by Fluka-Switzerland.

**Equipments**

In this study, gas chromatography (GC, Perkin Elmer- Auto system XL Gas Chromatograph) was utilized for the extracts analysis. Computerized microwave solvent extraction system (Milestone-Ethos Sel microwave lab station) was used for microwave extraction (Fig. 3 and 4). Total condensate solvent Soxhlet extraction unit has been used for eucalyptus essential oil extraction (Fig. 5).

**Procedure**

1. **Microwave Extraction.** In order to measure the optimal power and time, the following procedure was pursued: samples of eucalyptus leaves were placed in the sealed containers of 270 ml of each. Same volume (100 ml) of solvent (ethanol) was used throughout the tests. The power was ranging from 200 to 1000 W. The radiation dose was ranging from 60 to 300 second. The solution was agitated by magnetic stirrer. In between each dose of radiation, the solutions were allowed to cool back to room temperature (28°C). Sampling of 2 ml was carried out at the end of each dose of radiation, thus, the solute concentrations were corrected taking into account the change in solution volume. The extraction was done in an enclosed vessel, no evaporation was observed. All solutes concentrations are reported in mg/L.

2. **Soxhlet Extraction.** Total condensate solvent (ethanol) Soxhlet process was used, with the material/solvent ratio of 4 gm leaves/100 ml ethanol. The samples were analyzed every 60 minutes for the period of 300 minutes. All solutes concentrations are reported in mg/L. Eq. (1) was used to correct the concentration due to sampling:

\[
C = C_n (V-2 [n-1])/V
\]

\(C\) = corrected concentration (mg/L)
\(C_n\) = concentration obtained directly from chromatogram (mg/L)
\(N\) = number of sampling
\(V\) = initial volume of solvent (100 ml)

**RESULTS AND DISCUSSION**

The major constituents of eucalyptus essential oil (cineol, α-pinene, camphene, cymene, limonene and γ-terpinene) were chosen to inspect the influence of microwave power and time of exposure on extraction efficiency.
Influence of Microwave Power on Essential Oil Extraction

Power ranges from 200 W to 1000 W was applied and real (absorbed) power was calculated using Eq. (2). The irradiation time was 1 minute; sample weight was 6 gm/100 ml ethanol. Temperature increase (\(\Delta T\)) corresponding to the power of 200, 400, 600, 800, 1000, was 6, 11, 16, 20, and 25\(^\circ\)C respectively.

\[
P = m \frac{C_p \Delta T}{t} \quad (2)
\]

\(P\) = real power (W)
\(m\) = material and solvent weight (gm)
\(C_p\) = mean heat capacity (material + solvent), J gm\(^{-1}\) K\(^{-1}\)
\(\Delta T\) = temperature increase (\(^\circ\)C)
\(t\) = time (second)

\[
\therefore P = 30.125 \Delta T
\]

Microwave power output is assumed to be same of the power delivered to the microwave oven for materials of high dielectric constant (such as water 77.5) due to sufficient absorption of microwave. For different materials of lower dielectric constant (such as ethanol 22.3), the microwave absorption will not be as high as water. The real power represent 90% of the applied power at 200 W then start to decrease to be 75% at 1000 W (Fig. 6). The solution can absorb microwave at low applied power and its ability of absorption decreases with increasing the applied power due to its low dielectric constant. This phenomenon could be attributed to the change in the efficiency of the magnetron. It has been reported that the efficiency of the magnetron decreases as it gets hotter. This heat represents the principal efficiency loss in the magnetron. The cooling time in between each dose has considered this fact. It should be borne in mind that the power output of the magnetron alters with the variations in the main voltage, \(+6\%\) change in supply voltage produced an average deviation of \(+16.5\%\) in power output (Swain, 1993). Fig. 7 shows the constituents concentration of eucalyptus at different applied power, the results of extraction was not linear with power increasing. It can be seen at 800 W, the concentration of all constituents (except cineole) was close to that of 1000 W. As a result, for highest obtained essential oil, microwave power of 1000 W is recommended.

Influence of Microwave Irradiation Time on Essential Oil Extraction

To study the influence of radiation time on the essential oil extraction, time range of 1 to 5 minute microwave radiation was tested. From Fig. 8 it could be seen that the concentration increased for irradiation time 1 to 3 minute, but, for 4 and 5 minute, the obtained constituents concentration decreased. Although of the fact of higher exposure time, higher temperature increase, but, at the same time, moisture content will be decreased due to evaporation. Microwave heating is affected mainly by moisture content, the decrease of water in plant leads to decrease in extraction although higher temperature can be obtained due to moisture content plays important role in microwave heating.

Soxhlet extraction: Following same procedure of analysis for the six constituents (cineol, \(\alpha\)-pinene, camphene, cymene, limonene and \(\gamma\)-terpinene), Soxhlet extraction with material/solvent ratio of 4 gm/100 ml was used to extract eucalyptus essential oil. Table 1 shows that the extraction equilibrium has been achieved after 240 minutes. The concentrations of the constituents obviously are less than that of microwave extraction (Fig. 9). Only four constituents (\(\gamma\)-terpinene, cineol, \(\alpha\)-pinene and limonene) have been extracted by Soxhlet, indicating that Soxhlet performance feeble compare with Microwave extraction.

CONCLUSION

The influence of microwave power and time of exposure on microwave extraction of eucalyptus essential oil was studied. Applied microwave power of 1000 W was found
to give optimal extraction, the study shows that the real power corresponds to 1000 W is 750 W. Microwave irradiation time of 3 minute was enough for microwave extraction process, giving more time, led to reduce moisture content and subsequently, reduced extraction. Soxhlet the traditional extraction technique, shows much less obtained essential oil concentration (about 40% of that of microwave) as well as, only four constituents were extracted. Soxhlet the traditional extraction technique, shows much less obtained essential oil concentration (about 40% of that of microwave).

**Literature Cited**


**Tables**

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<thead>
<tr>
<th>Constituents</th>
<th>Time (minute)</th>
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<tbody>
<tr>
<td></td>
<td>60</td>
</tr>
<tr>
<td>γ-terpinene</td>
<td>-</td>
</tr>
<tr>
<td>cineol</td>
<td>4.38</td>
</tr>
<tr>
<td>α-pinene</td>
<td>-</td>
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<tr>
<td>limonene</td>
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Figures

Fig. 1. Eucalyptus leaves.  

Fig. 2. Eucalyptus tree.  

Fig. 3. Microwave oven.  

Fig. 4. Lab terminal controller.  

Fig. 5. Soxhlet unit.
Fig. 6. Plot of applied and real power.

Fig. 7. Constituent’s concentrations at different applied microwave power.
Fig. 8. Constituent’s concentrations at different microwave time exposure.

Fig. 9. Constituents cumulative concentration – material/ethanol ratios plot of Soxhlet extraction.