MICROWAVE ASSISTED EXTRACTION OF ESSENTIAL OIL FROM LEMON LEAVES

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ABSTRACT

Microwave-assisted extraction (MAE) has been received increasing attention as an alternative technique to extract active ingredient from plant because of several advantages over other extraction methods, such as reduction of costs, shorter extraction time, less solvent consumption, higher extraction rate, better product quality, less energy consumption. In this study, MAE was performed to obtain essential oils from lemon leaves (Citrus Limon). Result show that hexane is an optimal solvent and the extraction was affected mainly by concentration of solvent, extraction Time, Ratio of solvent to material and Microwave power. The optimal conditions for extraction of essential oil were 60% hexane with the ratio of solvent to material at 20:1 under microwave power of 500 W for 10 min. The yield of final product essential oil was 2.2 %. The yield percentage of essential oils from MAE was compared with essential oils obtained by Direct heating Method (DHE). Compared with direct heating extraction (DHE), MAE reduced the extraction time and had less energy consumption.

INTRODUCTION

Microwaves are a form of non-ionizing electromagnetic radiation with frequencies ranging from 0.3 GHz to 300 GHz. This energy is transmitted as waves, which can penetrate in biomaterials and interact with polar molecules into materials, such as water to generate heat [1]. Fast heating is the main advantage of microwaves; the application in foods is performed at frequencies of 915 MHz at industrial scale and 2450 MHz in domestic ovens [2]. Due to economics and environmental issues, food and chemical industries are facing the challenge of using new technologies in order to reduce energy consumption and CO2 emissions [3]. Separation technologies, such as extraction, distillation and crystallization are promising areas of innovation which can promote the growth of sustainable processes in the chemical and food industries [4].

Application of microwaves in separation and extraction processes has shown to reduce both extraction time and volume of solvent required, minimizing environmental impact by emitting less CO2 in atmosphere and consuming only a fraction of the energy used in conventional extraction methods such as direct heating extraction or steam distillation [6]. Advances in microwave-assisted extraction have led in the development of various techniques such as compressed air microwave distillation, vacuum microwave hydro distillation, microwave hydro distillation, solvent-free microwave extraction, microwave accelerated steam distillation, and microwave by hydro diffusion and gravity [7]. MAE is a current technology to extract biological materials and has been regarded as an important alternative in extraction techniques because of its advantages which mainly are: reduction of extraction time and solvents, selectivity, volumetric heating and controllable heating process. Various researches has shown the efficiency of MAE in the extraction of different compounds such as essential oils, fragrances, pigments, antioxidants and other organic compounds as animal tissues, food and plants. In addition of the reduction of time, solvent usage and energy consumption, this process shows even more benefits like a more effective heating, faster energy transfer, and size reduced equipment, rapid onset of warming and increased yields [6].

The use of MAE in the isolation of herbal essential oils is an interesting alternative that provides more effectiveness than other processes as the conventional extraction of essential oils of herbs and spices by steam distillation. Several authors have used microwave extraction techniques to obtain essential oils from herbs. Early reports of MAE application to extract essential oils were recorded in the 80’s [8]. Lemon (Citrus Limon) is a local Indian plant. Lemon leaves contains abundant essential oil which can be used in many fields such as medicine, foodstuff, beverage and cosmetics. The main ingredients are limonene, myrene, α-Pinene,β-Pinene, camphene, p-cymene etc. Lemon essential oil can be very beneficial to the circulatory system and aids with blood flow, reducing blood pressure and helping with nosebleeds. It can help bring down fever; helps relieve throat infections, bronchitis, asthma and flu. It boosts the immune system and cleanses the body, improves the functions of the digestive system, and it is helpful with constipation, dyspepsia and cellulitis. Lemon oil soothes and relieves headaches and migraines and is helpful for rheumatism and arthritis. It is also used for clearing acne, cleaning greasy skin and hair, as well as removing dead skin cells, easing painful cold sores, mouth ulcers, herpes and insect bites. Lemon oil helps to fight against infections, aids the digestive system, soothes headaches, migraines and muscular problems and clears greasy skin and hair. Lemon oil helps to fight against infections, aids the digestive system, soothes headaches, migraines and muscular problems and clears greasy skin and hair.

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Currently essential oil is extracted with solvent by heating the material directly for several hours. The direct heating extraction method has the disadvantage of high energy consumption and poor quality.

**MATERIALS AND METHODS**

Fresh leaves of lemon tree were obtained from a village near Pune city (M.S) India. It was cut into pieces less than 2 X 2 cm within half a day after collection. The solvents used namely distilled water, ethanol, hexane, petroleum ether are analytical grade.

**Microwave assisted extraction (MAE)**

100 g of sample (lemon leaves) were placed in 1000 ml flat bottom flask. After adding proper volume of solvent, the flask containing the sample was introduced to the microwave oven (700W, Koryo) and adjusted to a condenser connected to a cold water recirculation system; the microwave oven was turned on and the desired conditions of time (10 or 20 min) and power (500w) were set to allow heating and consequent generation of vapors. No stirring or rotation was possible within the flask. Vapors began to rise into the flask’s neck until reach the condenser where they were cooled, the extracted liquid was received into a trap; the hexane was removed in a rotary evaporator at 35°C. The essential oil was stored under refrigeration until its analysis.

**Direct Heating extraction**

100g lemon leaves and hexane were put into three neck flask out fitted with stirrer and condenser. Extraction was carried out with duration of 1 hr. Generated vapors began to rise toward the condenser where they cools. The extracted liquid was received into a trap. Volume of oil obtained was determined and stored as explained in the previous section.

**RESULT AND DISCUSSION**

**Selection of Solvent**

Distilled water, ethanol, hexane and petroleum ether were used respectively to extract essential oil from lemon leaves under same conditions. Each was extracted twice at total solvent/mass ratio 100:1 and microwave power (500W) for 10min. It can be seen in table 1 that hexane is the optimal solvent among all used solvent, concerning yield of essential oil, Petroleum ether may also be a good solvent. However distilled water and ethanol are not suitable for extraction because of low yield.

<table>
<thead>
<tr>
<th>Sr.No.</th>
<th>Solvent</th>
<th>Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Distilled Water</td>
<td>1.3</td>
</tr>
<tr>
<td>2</td>
<td>Ethanol</td>
<td>1.45</td>
</tr>
<tr>
<td>3</td>
<td>Hexane</td>
<td>1.95</td>
</tr>
<tr>
<td>4</td>
<td>Petroleum Ether</td>
<td>1.8</td>
</tr>
</tbody>
</table>

**Table 1 Selection of Solvents**

**Effect of Concentration of hexane on extraction of essential oil**

In the present study, hexane-water solutions at different ratios were tested as extraction solvent for extraction of essential oil from lemon leaves under microwave irradiation. Other experimental parameters were set as follows: the ratio of solvent to material, 20:1 (mL/g); microwave power 500 W; and extracting time, 10 min. The results revealed that the extraction yield was the highest when 60% hexane was used as extracting solvent (Figure 1). Seen from Figure 1, the yields increased when the concentration of hexane increased from 30% to 60%. Then, the yields declined from 60% to 90% hexane. These results supported previous findings that the concentration of organic solvent played an important role in the extraction of bioactive components from plant materials [9,10]. The results indicated that 60% hexane was suitable for the microwave-assisted extraction of essential oil from the plant, which was chosen as extracting solvent in the further experiments.

**Effect of ratio of solvent to material extraction of essential oil**

In order to obtain the maximum extraction yield, effects of the ratio of solvent to material on the extracted yield of essential oil were studied. The amount of solvent was changed to examine different solvent/mass ratios. Other experimental parameters were 60% hexane, and 10 min of microwave power with 500 W. The results were displayed in Figure 2. The extraction yield increased with the increased ratio of solvent to material from 5:1 to 20:1 and then falls down slightly when ratio exceeds 20:1. The extraction yield increased about 50% when the ratio of solvent to material increased from 5:1 to 20:1, which indicated that the ratio of solvent to material has a significant effect on the extraction efficiency of essential oil. The ratio of 20:1 was chosen as the optimal ratio of solvent to material.

**Effect of Microwave power on extraction of essential oil**

Effects of microwave power on the yield of essential oil were investigated under the conditions of 60% hexane, the ratio of solvent to material at 20:1, the extraction time of 10 min. The results are shown in Figure 3. The yield of essential oil increased slightly from 200 to 300 W. The yield of essential oil decreased with increasing microwave power from 500 to 700 W. The highest yield was obtained at 500 W (Figure 3). The microwave can accelerate the extraction process for desorption of the targeted compounds from matrix at low power, and may induce
The yield of essential oil - diffusion of effective ingredients from the cell wall and cell membrane easily. Therefore, 500 W of microwave power was used in the further experiments.

**Comparison of MAE with DHE**

Experiment was carried out under the optimal condition of MAE and the reported optimal condition of DHE using the same batch of lemon leaves. As shown in Table 2, 10 min total extraction time of MAE is much shorter than 120 min for DHE. In addition, total solvent-material ratio required by MAE is 20:1, less than the 40:1 of DHE.

**Table 2 Comparison of MAE with DHE**

<table>
<thead>
<tr>
<th>Extraction methods</th>
<th>Total Extraction time (min)</th>
<th>Total solvent material ratio</th>
<th>% Yield</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAE</td>
<td>10</td>
<td>20:1</td>
<td>2.2</td>
</tr>
<tr>
<td>DHE</td>
<td>120</td>
<td>40:1</td>
<td>1.98</td>
</tr>
</tbody>
</table>

The yield of essential oil is more in MAE than DHE. The greater extraction efficiency of MAE found in this work may be traced to the following reasons. (1) Cell wall and cell membrane of lemon leaves were broken by microwave irradiation; essential oil may get across the cell wall and cell membrane easily. (2) Electromagnetic field produced by microwave may accelerate diffusion of effective ingredients from material to solvent.

**Conclusion**

The factors affecting the yield during the extraction of essential oil from lemon leaves are concentration of solvent (hexane), extraction time, solvent-material ratio and microwave power. The optimal conditions of MAE of essential oil from lemon leaves are concluded that the extraction is performed with microwave power 500 W for 10 min, total solvent-material ratio is 20:1. Hexane is the optimal solvent for extraction. The yield of essential oil obtained under the above condition is 2.2 %. Pharmacologically, lemon essential oil has great importance. The essential oil from Lemon can be used in a cream or lotion to clear congested skin. The antiseptic effect of lemon oil on the other hand, helps to treat any cuts, boils and minor wounds. In vapor therapy, the essential oil can be used for colds, voice loss, flu, depression, stress, lack of energy and fatigue. It can be used in blended massage oils or diluted in the bath to assist with digestive problems, lack of energy, fatigue, infections, flu, obesity, overweight, rheumatism, depression, stress and as a general tonic. Compared with DHE, MAE has the advantage of shorter time and less solvent consumption, making it as a better alternative method to extract essential oil from lemon leaves.

**References**


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