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Comparative Study of Solvent Free Microwave Extraction and Hydrodistillation for the Extraction of Essential Oil from *Menthapiperita*

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Abstract—*The Essential oils are blend of various aromatic hydrocarbon compounds, valuable gift of nature extracted from different parts of plants. Due to various undesirable effects imposed by chemical products, human's interest is rising in the exploitation of natural and herbal products for various purposes. This laid down growing Industrial attention in demand of essential oil. Although the role played by essential oil is very vast and some of the major application include their use as a medicinal purpose, aromatherapy, pharmaceutical product, an insect repellent, and many more. Menthapiperita member of the Lamiaceae (Labiatae) family, a native of Europe is widely cultivated for peppermint oil extraction in north India.*

Peppermint oil is distinct from other oils as a result of its powerful aromatic characteristic that when it dispersed into working and living area it clarifies the air, cleanses the air by removing metallic particles and toxins from the air and enhances atmospheric oxygen. When it is rubbed on the feet, its penetrating quality is so effective that within a minute it circulate to each cell in the body. There are various conventional methods for example steam distillation, hydrodistillation, solvent extraction method for extracting peppermint oil from plant material. In this study, essential oil of Mentha piperita was extracted by solvent free microwave extraction and scrutinize the operating parameters for the maximum yield of oil. SFME is green technology for oil extraction, which uses electromagnetic waves to rupture oil gland at atmospheric pressure from aerial parts of the plant.

Solvent free extraction method was operated in the absence of solvent. In this, extraction was performed at 50, 100, and 150gram of raw material for different extraction time (30, 60, 90 and 120 min). It was obtained as a green technique to the environment as there was no residue of waste water.

From the results the maximum yield of peppermint oil was achieved from solvent free microwave extraction method with 1.02% yield at 250W in 90 min. Testing of volatile compounds present in extracted peppermint oil was investigated by Gas Chromatography and mass spectrometry.

1. INTRODUCTION

Essential oils are often extracted from seeds, flowers, leaves, barks, roots similarly other numerous segment of a plant which carries unique flavor, essence and scent. Essential oils are usually described as concentrated liquid, ample in aromatic volatile hydrocarbons. Essential oils are well known with having hydrophobic and liophilic property. Extract of essential oil are abstruse mixture of volatile chemical compounds with alcohol, aldehydes and ketone which are blended in various structure of plant part and predominance of terpenes is associated in oil. In industrial scale, essential oils are typically extracted from number of extraction methods such as hydrodistillation the most common conventional method. Industry extracts their oil from fresh and partially dried leaves as a raw material. The extract that is extracted from medicinal plant comes in competition in the market due to their uses in pharmaceuticals, cosmetics, foods and flavoring and in perfumery market which generally use their active substance. The continents of Asian due to its diversity of climate appears as a the most important originator of essential oil. India and china involved in a major role followed by Sri Lanka, Indonesia and Vietnam. [Ranitha M. et. al. 2014]

*Menthapiperita L.*also known by peppermint leaves is the member of mint which comes in Lamiaceae family. It is a perennial, a plant lasting for many seasons and more and glabrous herb.This plant is extensively cultivated for medicinal and aromatic purpose for various applications. [Gavahian et. al. 2015]The parts of plant above the ground mainly contain essential oil which is a mixture of large number of aroma chemicals such as menthol, isomenthone, menthone, menthofuran etc. peppermint oil could be extracted from whole plant above the ground just before flowering. Colour less to pale yellow liquid with strong aromatic taste, pepper-like pungent odor, are the appearance of peppermint oil. There are several methods of extraction like solvent extraction, distillation hydrodistillation and steam distillation),

supercritical fluid extraction which is used to extract the concentrate and volatile compounds.

Solvent free microwave extraction is one of developing technique for the extraction of essential oil without addition of water or any solvent. SFME appears to be particularly attractive for the isolation of essential oil from *Mentha Piperita L.* This technique is based on the combination of low microwave heating and distillation is performed at atmospheric pressure. Some of advantage of this method over other method includes rapidity in attaining the extraction temperature of 100^o C for the first essential oil droplet, high yield of essential oil, lower energy requirement and high purity of the oil extracted using this method.

The main present research objectives are to examine the effectiveness of this technique on the yield of peppermint oil extraction and parametric study of this technique in order to obtain the higher yield and identify all volatile compounds present in the extracted oil by Gas chromatography and mass spectrometry technique.

2. MATERIALS AND METHODS

2.1. Sample preparation

The whole plants of peppermint were collected from one farm of Daliyanpur situated near Lucknow in U.P, India. The plant was freshly cut, 90-120 cm from root in the morning of April 29, 2017 they were collected. After harvesting, the plants were immediately packed in sealed plastic bag, which were stored in a refrigerator at 4.7^o C for further isolation of essential oil.

Figure 1: Fresh leaves of menthe piperita.

2.2. Moisture content

Leaves of 10.12 gram were placed into petri-dish. Then loaded dish was heated in hot air oven at 50^o C for 8 hours. The moisture content percentage in leaves was calculated from following formula:

$$(\text{Moisture content})\% = \frac{Wi - Wf}{Wi} * 100$$

Where,

W_i = Initial weight of the sample (g)

W_f = Final oven dried weight of the sample (g)

2.3. Solvent Free Microwave Extraction

Solvent free extraction method was operated in the absence of solvent. The process was differing as change in cell structure because of electromagnetic waves causes the extraction which allows the essential oil to run out from the leave. Extraction from leaves was done by placing 100 grams of leaves in 1000mL of round bottom flask with no solvent and kept in oven for providing heat. The oven was operated at 200W, 250W and 300W for different extraction time (30, 60, 90 and 120 min). It was obtained as a green technique to the environment as there was no residue of waste water. The excess water was removed by drying over anhydrous magnesium sulfate in extracted oil. Pure oil was collected in the vials and then stored at 4.7o C for GC-MS analysis.

Figure 2: Solvent Free Microwave Attraction.

2.4. Traditional hydro-distillation

Hydro-distillation process was conducted by Clevenger apparatus of 34/35 size with heating mantle as heating source of 1 L capacity. In this procedure, the experiment was performed at 60^o C for 6:1, 8:1, 10:1 and 12:1 ratio of water to leaves in the different period of time interval i.e. 30, 60, 90, 120, 150, 180, 210, 240, 270 min. Experiment was operated at atmospheric pressure. The extracted oil was collected in the separating funnel and after collecting, water in peppermint oil was removed by drying over anhydrous magnesium sulfate. Pure oil then stored in the vials and put in refrigerator at 4.7^o C.

2.5. Yield of extracted oil

In order to examine the performance of SFME, yield of oil was calculated from following equation:

$$\text{Yield \%} = \frac{Amount of \boldsymbol{essential oil}(g) obtained}{Amount of raw material(g) used} * 100$$

2.6. Analysis by GS-MS (Gas Chromatography and Mass Spectrometry)

This analytical technique is a combination of two major blocks: gas chromatography and mass spectrometry in order to find the various volatile compounds with in the oil injected. This technique used the capillary column that can be polar and non-polar which usually depends on the diameter, length and film thickness of the column. The time taken by the molecules to travel through the column is the retention time that is different for each molecule. The molecules eluted from the column at different time and entered into spectrometry block where molecules braked into the ionized fragment and then detect through mass to charge ratio. The recognition of components in the extracted oil was performed with an Agilent Technologies- 5975B GC coupled with MS system. There are following specification that was used by system in examining the volatile compounds:

Capillary column: HP-5MS fused silica non polar capillary column

Dimension of column: 30 m x 0.25 mm x 0.25µm.

Carrier gas: Helium gas with flow rate of ±1.5ml/min.

Injector and detector temperature: 240^{o} C and 230^{o} C respectively.

The 1 µL was injected into the split ratio of 120:1. The electron ionization system with ionization energy of 70ev detected the data or spectra of components. Oven temperature was set at 55 ^{o} C (hold for 5 min) then increased to 185^{o} C with flow rate of 4.5^{o} C/min (hold for 2 min) then finally increased to 230^{o} C at 8^{o} C/min and then set as isothermal for 20 min.

2.7. Energy consumption calculation

The energy required to achieve the complete extraction was calculated with the given below equation. Power consumption by heating source was basis to calculate the energy required from all three method.

$$\text{E (KWh/day)} = \frac{P(W) \times t(\frac{h}{day})}{1000\ (\frac{W}{KW})}$$

E= Energy Consumption.

P= Microwave power

3. RESULTS AND DISCUSSION

Solvent free microwave extraction of aerial part of Mentha Piperita gave colorless liquid with a yield of 1.02% (w/w). GS-MS testing of extracted oil revealed that there are 34 components present in the oil.

3.1. Determination of operating parameters for SFME

3.1.1. Effect of irradiation time

The variation in yield of extracted peppermint oil on different extraction time at three microwave power level for 100 gram of plant matters was observed. There was an initial progressive increase in the yield of peppermint, as the extraction time increase from 30 min to till 90 min. However, when extraction time became extended beyond 90min to 120 minute, there was notably slightly variation in the yield of peppermint oil. The rate of extraction was high at the beginning of the extraction but get slow gradually by time. Highest 1.02% yield of oil was obtained at 90 min of extraction time.

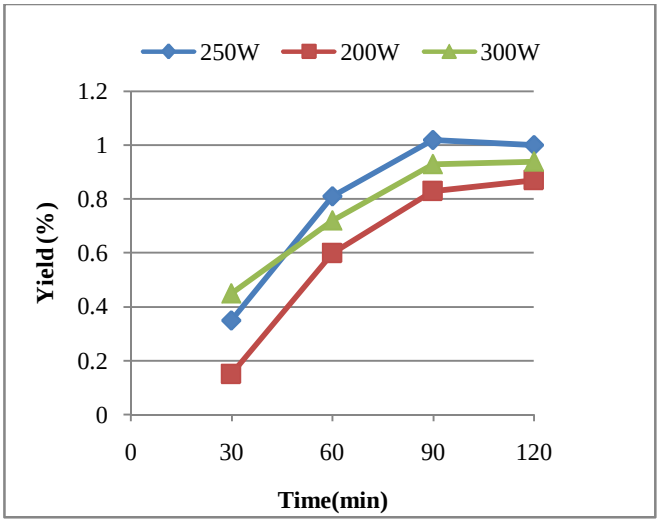

Figure 3: Effect of extraction time on oil's yield.

3.1.2. Effect of irradiation power

Three microwave power levels were used on 100 grams of plant sample and yield were investigated on different microwave power watt as shown in the figure. The extraction was improved by raising the microwave power from 200W to 250W with .83% and 1.02% respectively on 90 min. And observed decrease in yield when power increased from 250W to 300W with 1.02% with .93% respectively. Hence 250W and 90min was found operating conditions for extracting higher essential oil from plant matter. Furthermore, on parameters of 250W of microwave power and 90 min of extraction time, again experiment performed on 50 gram and 150 gram of leaves the yield obtained 0.86% in 60 min and 0.98% in 90 min respectively.

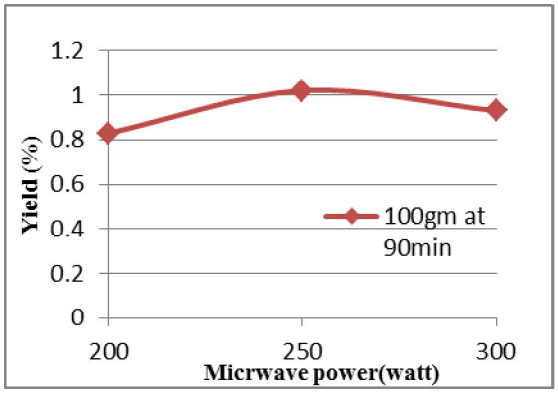

Figure 4: Effect of Microwave power on oil's yield.

3.2. Traditional HD method

In this method experiment was performed at different ratio- 1:06, 1:08, 1:10 and 1:12 at constant temperature of 60° C. In this study it was found that combination 1:10 provide maximum yield 0.96%. An increasing rate of oil yield was achived from 30-240 min, after this there was no significant change in yield.

3.3. GC-MS analysis of extracted oil

By using GC/MS techniques, total 34 compounds were identified in oil extracted from SFME method. Menthol, menthone, limonene, menthofuran, menthene, isopulegol, 3-paramenthene were recognized as the major constituents in the sample of oil extracted from SFME method. The oil was dominated by Oxygenated hydrocarbon compound such as menthol (61.66%) in SFME method. While mono terpenes and terponids were identified as a minor components such as α-pinene, β-pinene, 1,8 cineole, menthyl acetate, menthene, isomenthene, limonene and more.

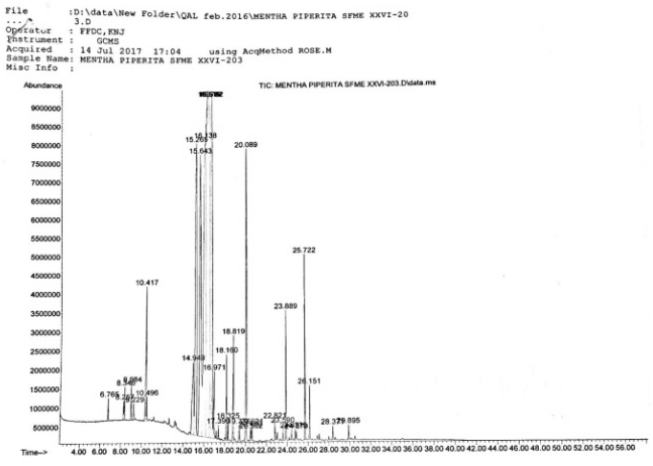

Figure 5: A characteristic Gas chromatogram and mass spectra of the constituents of essential oil from SFME.

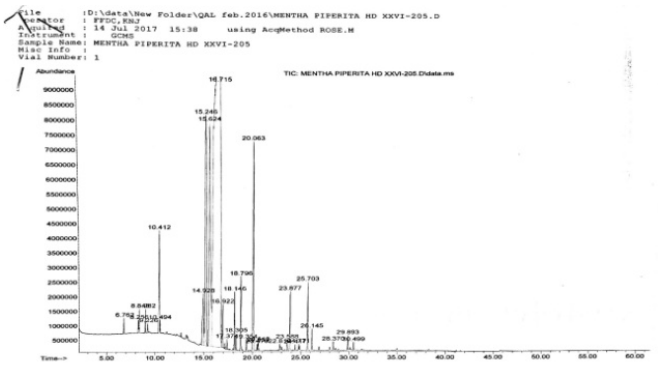

Figure 6: A characteristic Gas chromatogram and mass spectra of the constituents of essential oil from HD.

Table 1: Composition of constituents in essential oil from SFME.

Sr. no.	Peak no.	Compounds	RT	Area %	CAS
1	1	α-pinene	6.766	0.22	000080-56-8
2	2	β-Phellandrene	8.254	0.19	000555-10-2
3	3	β-pinene	8.349	0.34	000127-91-3
4	4	Myrcene	8.986	0.37	000123-35-3
5	5	3-Octanol	9.226	0.20	000589-98-0
6	6	Limonene	10.414	1.95	000138-86-3
7	7	1,8- cineole	10.500	0.20	000470-82-6
8	8	Isopulegol	14.947	2.35	121468-66-4
9	9	Para-Menthone	15.265	9.45	000089-80-5
10	10	Para-Menthone	15.644	9.33	000089-80-5
11	11	Menthol	16.134	14.75	000089-78-1
12	15	α-terpineol	16.968	0.80	000098-55-5
13	16	Octanol Acetate	17.898	0.14	000112-14-1
14	17	CyclohexylFormate	18.164	0.91	004351-54-6
15	18	Pulegone	18.327	0.22	000089-82-7
16	19	Piperitone	18.818	1.14	000089-81-6
17	20	N-Decanol	19.377	0.29	000112-30-1
18	21	3-Para menthene	20.091	5.06	000500-00-5
19	22	Carvomenthy Acetate	20.478	0.13	020777-43-9
20	23	Nonanyl Acetate	20.564	0.09	000143-13-5
21	24	Isopulegyl Acetate	20.624	0.14	109010-11-9
22	25	β-Bourbonene	22.818	0.25	005208-59-3
23	26	Decyl Acetate	23.592	0.14	000112-17-4
24	27	E-Caryophyllene	23.893	1.36	000087-44-5
25	28	Hexyl 2-menthyl Butanoate	24.418	0.09	010032-15-2
26	29	Piperitone	24.779	0.10	000089-81-6
27	30	α-Humulene	24.882	0.12	006753-98-6
28	31	D- Germacrene	25.725	1.96	023986-74-5
29	32	Bicyclogermacrene	26.155	0.51	024703-35-3
30	33	Trans-Muurola-3,5- Diene	28.374	0.13	189165-77-3
31	34	3z-Hexenyl phenyl Acetate	29.897	0.16	042436-07-7

Table 2: Comparison of energy consumption.

	SFME	HD
Extraction time(min)	90	270
Electric energy consumption(kwh)	0.375	1.0125

4. CONCLUSION

In this work, SFME was examined to extract the peppermint essential oil on the various parameters in order to get the higher yield. These parameters were mainly mass of raw material, time and microwave power. And from the results it was found that SFME was more auspicious as it required shorter extraction time and produced higher amount of quality yield. It was found that 1.02% of yield was obtained at 250W of power in 90 min extraction time from 100 gram of plant material. It was also found that it is eco-friendly to the nature because of no ejection of carbon dioxide.

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