

Effect of Hydro Distillation Process on Extraction Time and Oil Recovery at Various Moisture Contents From Mentha Leaves

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ABSTRACT : The experiments were carried out to study the kinetics of mentha oil extraction from mentha leaves (*Mentha arvensis* L.) and the quality analysis was carried out for the oil extracted. The oil was extracted from mentha leaves at three different moisture contents of 74.30 per cent (fresh leaves), 42.30 per cent (shade dried) and 19.35 per cent (sun dried) using hydro distillation method. Various physicochemical tests were carried out on the oil extracted. The results revealed that the hydro distillation process took more time for oil extraction and oil recovery was less. Various physicochemical properties such as acid value, refractive index, specific gravity, saponification values and solubility in water did not show significant variations with respect to oil extracted by differently pretreated mentha leaves.

KEYWORDS : Hydro distillation, Mentha, Oil recovery

I. INTRODUCTION

Essential oils by definition are the liquid products of steam distillation of plant parts. An essential oil may contain tens or hundreds of volatile and non-volatile compounds, the cause of their characteristic fragrance or flavour. Essential oil are present in plants in specialised cells/glands (subcuticular spaces of glandular cells, organelles.), these glands may be at anywhere on plant body depending upon the morphology and physiology of the plant. Some time it may be on leaves, flowers, stems, roots, bark or wood. The world's total production of essential oils is estimated at about 1,00,000-1,10,000 tons and India stands at number three, contributed with a share of 16-17 per cent. In value terms India's position is number two with the share of 21-22 per cent, thanks to mint revolution in North India. Of the total world production of essential oils, 15 products constitute 90 per cent of the total, including lavandin, *Mentha arvensis* and *Mentha piperita* (peppermint) (Hay and Waterman 1993). *Mentha arvensis* is cultivated in India in the semi- temperate regions in the foothills of Himalayas in Punjab, Himachal Pradesh, Uttar Pradesh and Bihar. In 1997 the area under mentha in U.P. went up to 40,000 hectares from 20,000 hectares in 1996. Even in 1998, the area under mentha is reported to have gone up. The all-India area under mentha in the country is estimated at about 1,00,000 hectares (Sharma 2009).

Various constituents of mint oil as per monographs of Internation Pharmacopoeia are limonene (1.0-5.0 per cent), cineole (3.5-14.0 per cent), menthone (14.0-32.0 per cent), menthofuran (1.0 -9.0 per cent), isomenthone (1.5-10.0per cent), menthyl acetate (2.8-10.0 per cent), isopulegol (max. 0.2 per cent), menthol (30.0-55.0 per cent), pulegone (max. 4.0 per cent) and carvone (max. 1.0 per cent). The ratio of cineole content to limonene content should be minimum two (Alankar 2009). The natural oil yields on an average 40-50 per cent menthol and 50-60 per cent dementholised oil (Anonymous 2009). The dementholised oil has been found to contain menthyl acetate (24.4 per cent), free menthol (44.8 per cent), menthone (24.6 per cent) and hydrocarbons (6.2 per cent) (Board 2010). Mint is also used for flavouring meat, fish, sauces, soups, stews, vinegar, teas, tobacco and cordials. The main use of mint is the extraction of volatile oil which contains menthol and is used in medicine for stomach disorders, in cough drops, inhalation, mouthwashes, toothpastes, etc. and also for flavoring in cigarettes. In hydro-distillation method the material is completely immersed in water and then boiled. In this procedure the surrounding water act as a barrier in preventing the material from overheating, so here oil can be extracted to certain degree only. The apparatus used is Clevenger-type hydro-distillation apparatus. Upon cooling, the water and the essential oil separate in the collector. This hydro-distillation process can be done at a reduced pressure (under vacuum) to decrease the temperature to less than 100^oC. This can be beneficial in protecting heat sensitive chemical compounds from rearrangement or complete decomposition which will affect the essential oil quality. Although a considerable amount of work has been done and data generated on the extraction of mentha oils by hydro distillation however, to the best of our knowledge there are no standard set of pre treatments or process conditions for its efficient extraction. There was a need to optimize the pre conditions and process parameters for getting the maximum yield of oil. The present project was

therefore undertaken with the main objective to get the maximum yield of oil under different moisture conditions.

MATERIALS AND METHOD

The experiments to accomplish the desired objectives were performed in the laboratories of the Department of Processing and Food Engineering, Punjab Agricultural University, Ludhiana. The experiments were conducted focusing on the achievement of the conceptual study, laboratory experimental work, analyzing and completion of the project.

PREPARATION OF SAMPLE

The mentha variety '*Mentha arvensis* L.' was procured from farms of village Rakba, Ludhiana and was stored at 5°C and 85 percent relative humidity in cold stores of pilot plant in the Department of Processing and Food Engineering, Punjab Agricultural University, Ludhiana. The technological feasibility of both fresh and dry mentha leaves was assessed to obtain essential oil.

PRE TREATMENT OF MENTHA LEAVES

Mentha was subjected to pre treatment in terms of separation of leaves and partial drying of leaves. Mentha leaves were separated from their bushes and stored. These leaves were taken at three different moisture contents. The mentha leaves were dried under shade as well as sun so that two different moisture contents of leaves could be obtained. So, the mentha leaves were used as fresh, shade dried, and sun dried samples for the purpose of oil extraction and subsequently quality evaluation.

HYDRO DISTILLATION

The Clevenger-type apparatus was used for this purpose. For distillation, a mixture of mentha leaves (200 g) and 1000 ml of water was put into a 2000 ml round bottomed flask. The temperature was set at 80°C for the extraction of essential oil. The process in Clevenger-type apparatus was run for the time till no further oil could be extracted. The essential oil was vaporized with the steam. Condensation occurred as the vapours of essential oil and steam mixture passed through a condenser. The condensate, a mixture of oil and water, was then separated. Essential oil being lighter settled above water and it was collected. To study the kinetics of extraction of oil, essential oil was collected at regular intervals during the extraction process.

PHYSIO-CHEMICAL ANALYSIS OF MENTHA OIL

MOISTURE CONTENT

Ten grams of leaves were accurately weighed into a petri- dish previously weighed. The dish containing the leaves was heated in an oven at 105 °C for 24 hours. The %age moisture was calculated from the loss of mass after 24 hour drying on wet basis (Chegini and Ghobadian 2007).

$$\text{Moisture Content (\%)} = \frac{W_1 - W_2}{W_1} \times 100$$

Where,

W₁ = initial weight of the sample (g)

W₂ = final weight of the sample (g)

This moisture content was converted into moisture content on dry weight basis by using the following formula. The main purpose of expressing moisture content on dry weight basis is that it gives the percent moisture on the basis of dried weight, which remains constant and acts as reference level.

SAPONIFICATION VALUE

Saponification value is defined as the weight of potassium hydroxide expressed in milligrams required to saponify 1 g of fat or oil. 2 g of the oil was weighed into a 200 ml conical flask, to which 50 ml 0.5 M alcoholic KOH was added. This was refluxed for 30 min, followed by the addition of 3 drops of phenolphthaleine indicator and was titrated with 0.5 M HCl until the pink coloration disappeared. This was repeated without the oil and the titre value was determined as the blank (AOAC 2000).

$$\text{Saponification value} = \frac{(t_1 - t_2) \times 28.1}{W}$$

Where, t₁ = blank value, t₂ = sample value and w = weight of sample.

ACID VALUE

The acid value is the number of milligrams of potassium hydroxide necessary to neutralize the free acids in 1 gram of sample. With samples that contain virtually no free acids other than fatty acids, the acid value may be directly converted by means of a suitable factor to percent free fatty acids.

2 g of the sample was weighed into a conical flask containing 50 ml of isopropyl alcohol. To the mixture, 3 drops of phenolphthalein indicator was added. This was titrated with 0.1 M NaOH from the burette (Paudyal et al., 2012).

$$\text{Acid value} = \frac{5.61 \times \text{titre value}}{\text{Weight of Sample}}$$

SPECIFIC GRAVITY

Specific gravity is the ratio of the density (mass of a unit volume) of a substance to the density (mass of the same unit volume) of a reference substance. The specific gravity of the extracted oil was determined using a hydrometer. The oil was poured into a cylindrical long tube, and the hydrometer was immersed into it and allowed to float (AOAC 2000).

REFRACTIVE INDEX

A property of a material that changes the speed of light, computed as the ratio of the speed of light in a vacuum to the speed of light through the material. Refractometer was used to determine the refractive index of the extracted oil. One drop of the oil was dropped on the cell compartments of the instrument. The necessary adjustments were made and the result was recorded when the lower part became darker (Hand Refractometer Abbas).

SOLUBILITY TEST

2 ml of the oil was poured into a test tube, containing 4 ml of water, and the content of the test tube were vigorously shaken to mix them and allowed to stand for 5 min. At the end of the 5th minute, the solubility of the oil in water was noted (AOAC 2000).

RESULTS AND DISCUSSIONS

EFFECT OF HYDRO DISTILLATION PROCESS ON OIL RECOVERY OF FRESH MENTHA LEAVES

In first 10 minutes 3 ml of oil was recovered resulting in 2.62 percentage recovery on dry basis. During next 10 minutes there was an increase of 1.05 ml in yield of oil resulting in 3.54 per cent oil recovery at the end of 20 minutes. At the end of 30 and 40 minutes percentage oil recovery was found to be 3.79 and 3.89 per cent respectively. After 120 minutes total 4.6 ml of oil was recovered giving 4.02 per cent oil recovery. In the next hours there was no oil recovery in the process. Hence, the total oil recovery from 200 gram sample was found to be 4.02 per cent. It was clear that most of the oil was extracted in the first 20 minutes. It might be mainly due to the fact that diffusion of oil was fast due to high initial oil content. This diffusion rate decreased significantly when the oil content of leaves decreased. Asekun *et al* 2007 studied the effect of different drying on oil extraction of *Mentha longifolia L. subsp. Capensis*. They found that the oil yield of fresh mentha leaves (*Mentha longifolia L. subsp. Capensis*) was 0.79 (%v/w).

EFFECT OF HYDRO DISTILLATION PROCESS ON PERCENTAGE OIL RECOVERY OF SHADE DRIED MENTHA LEAVES

In first 5 minutes 1.5 ml of oil was recovered. During next two intervals of 5 minutes each there was increase of 0.7 and 0.5 ml in yield of oil resulting in 1.52 and 1.87 per cent oil recovery respectively. At the end of one hour, yield was noted to be 2.08 per cent. Hence, the total recovery from 150 gram of shade dried leaves yielded 2.08 per cent of oil. Zinni *et al* (2011) extracted the essential oil from shade dried peppermint leaves with a yield of 0.53 per cent. The most of the oil was extracted in first 15 minutes and thereafter extraction process was quite slow. It might be due to the fact that diffusion of oil was fast due to high initial oil content. This diffusion rate decreased significantly when the oil content of leaves decreased.

EFFECT OF HYDRO DISTILLATION PROCESS ON OIL RECOVERY OF SUN DRIED MENTHA LEAVES

During first 5 minutes 1.3 ml of oil was recovered. During next two intervals of 5 minutes each an increase in yield of 0.8 ml and 0.7 ml was noted. After half an hour total oil yield noted was 3.0 ml. After another 30 minutes only 0.1 ml of oil was recovered resulting in total yield of 3.1 ml and 3.45 per cent oil recovery. Here, most of the oil was extracted in first 15 minutes and thereafter extraction process was quite slow. It might be due to the fact that diffusion of oil was fast due to high initial oil content. This diffusion rate decreased significantly when the oil content of leaves decreased. Asekun *et al* 2007 studied the effect of different drying on oil extraction of *Mentha longifolia L. subsp. Capensis* and found that the oil yield of sun dried mentha leaves (*Mentha longifolia L. subsp. Capensis*) was 2.61%.

EFFECT ON EXTRACTION TIME OF OIL FROM MENTHA LEAVES AT VARIOUS MOISTURE CONTENT BY HYDRO DISTILLATION PROCESS

The extraction time was maximum for the mentha leaves at 74.30 per cent moisture content whereas leaves at 19.35 per cent and 42.30 per cent moisture content take 60 min less time for the extraction. So, although maximum oil was extracted from fresh samples it took maximum time of 120 minutes to get that yield.

QUALITY PARAMETERS

SENSORY ANALYSIS OF THE MENTHA OIL

According to the sensory analysis done, the oil was found to be homogenous, opaque, light yellowish in colour with characteristic pungent odour of mentha. The colour of mentha oil obtained at various moisture contents been given in Table 4

SAPONIFICATION VALUE

The saponification value for hydro distillation method at different moisture content varied from 205.56 to 213.56. The saponification values of oil extracted by hydro distillation process were almost half than those of steam distillation process. The possible reason could be that during the steam distillation process, only the oil molecules were extracted where as in hydro distillation, liquid water extracted other saponifiable material also.

ACID VALUE, REFRACTIVE INDEX, SPECIFIC GRAVITY AND SOLUBILITY TEST

The results for the acid Value, refractive index, specific gravity and solubility test remained same irrespective of the moisture content. The figures are shown in the table 5.

CONCLUSION

It was noted that the maximum oil recovery was from fresh mentha leaves at 74.30% moisture content as compared to shade dried and sun dried mentha leaves. Most of the oil was recovered in first 20 min which is little more time consuming than shade dried and sun dried leaves. The saponification value of fresh mentha leaves at 74.30% moisture content was noted to be higher whereas not much change in other properties was visible.

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FIGURES AND TABLES

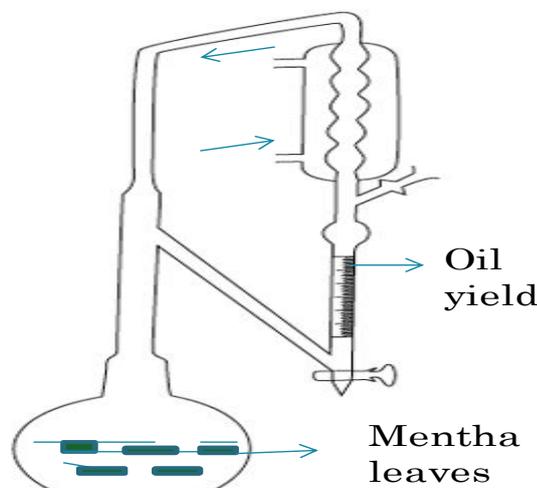


Fig 1: Clevenger-type apparatus – Appareil de type Clevenger

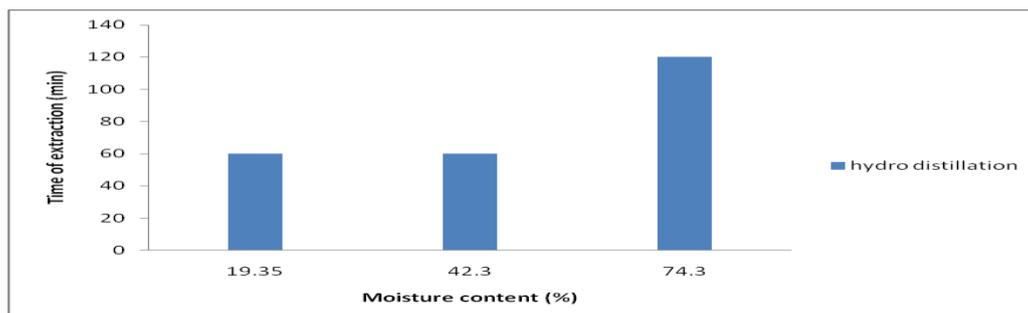


Fig 2: Effect on extraction time of oil from mentha leaves at various moisture content by hydro distillation process

Table 1: Effect of hydro distillation process on percentage oil recovery of fresh mentha leaves having moisture content of 74.30 per cent

Time (min)	Oil yield(ml)	%age oil recovery(w.b)	Oil recovery % (d.b)
10	3.00	1.35	2.62
20	4.05	1.82	3.54
30	4.35	1.95	3.79
40	4.45	2.00	3.89
120	4.60	2.07	4.02

Table 2: Effect of hydro distillation process on percentage oil recovery of shade dried mentha leaves having moisture content of 42.30 per cent.

Time(min)	Oil yield(ml)	Oil recovery % (w.b)	Oil recovery % (d.b)
5	1.50	0.90	1.04
10	2.20	1.32	1.52
15	2.70	1.62	1.87
60	3.00	1.80	2.08

Table 3: Effect of hydro distillation process on per centage oil recovery of sun dried mentha leaves having moisture content of 19.35 per cent.

Time(min)	Oil yield(ml)	Oil recovery %(w.b)	Oil recovery % (d.b)
5	1.30	1.17	1.45
10	2.10	1.89	2.34
15	2.80	2.52	3.12
30	3.00	2.70	3.34
60	3.10	2.79	3.45

Table 4: Colour of mentha oil at various moisture content

Process	Colour
Moisture Content	74.30% 42.30% 19.35%
Hydro distillation	Light yellowish Light yellowish Yellowish with little ting of green

Table 5

Test Name	Fresh Leaves (74.30 %)	Shade Dried (42.30%)	Sun Dried (19.35%)
Saponification Value	213.56	207.94	205.56
Acid Value	2.24	2.24	2.24
Refractive Index	1.46	1.47	1.46
Specific Gravity	0.92	0.92	0.89
Solubility Test	Non Soluble	Non Soluble	Non Soluble