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Chemical Profile and Extraction Technique of Oil of *Mentha Arvensis*

M.A.Nadeem[1], B.K.Saxena[2], N.Akbar[3]
[1]Institute of Technology and Management, Gwalior, India.
[2]DAV-PG Collage, Kanpur, India.
[3]Fragrance & Flavour Development Centre, Kannauj, India.

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ABSTRACT

Menthol mint oil is distilled by water steam distillation from leaves of Mentha arvensis and is the most importance source of L-menthol. It contains L-menthol 68.3%, menthone 8.2%, isomenthone 4.4%, menthyl acetate 4.3%, mixture of isomers of menthol 4.5%, cis-3- hexanal 0.2-% and limonene 1.2%, However percentage of components depends on the genetic and ecological conditions. Major component L-Menthol is isolated by freezing at low temperature with the recovery of around 65% in form of menthol flakes and the remaining material is known as DMO or dementholised oil (30%). During the process 1% loss is generally found. All the components are being used in Flavours, Pharmaceuticals, Tobacco and other cosmetic Industries.

Keywords: Crystallization**;** Dementholised Oil; Essential oil; Mentha arvensis; Menthol bold crystals; Menthol flakes; Odour evaluation; Terpenes.

Introduction

Mint oil comes under the category of essential oils also known as Mentha. It's by product is dementholised oil (DMO) and it a source of terpenes, menthyl acetate and liquid menthol. Mentha is basically divided into various categories like *Mentha arvensis, Mentha piperita, Mentha citrate, Mentha spicata, Mentha gracilis, Mentha Longifolia* and various others, thanks to agriculture science. Menthol mint is the backbone of livelihood for thousands of farmers across the northern plains of India. At present, India is global leader in mint oil production covering nearly 90% of total world production out of which around 85% is spread out in Uttar Pradesh such as Barabanki, Sambhal, Moradabad, Budaun, Rampur and Bareilly. The rest 15% is in Punjab, Madhya Pradesh and Bihar. This was all done through a systematic but economically driven total value chain right from development of variety to extension, survey of status of mint and there after introduction of multi commodity exchange for forward trading. Lot of MSMEs are involved in the distillation and trade related to menthol mint in India.

In India it was introduced in 1954 by Regional Research Laboratory Jammu (now Indian Institute of Integrative Medicine). In 1964 it all started in tarai region of Uttar Pradesh by M/s Hindustan Richardson Ltd., Mumbai. Subsequently it started increasing and reached to a level of 250 M tonnes in 1976. In 1980 the existing clone of Menthol mint started deteriorating, then variety development programme was taken up by Central Institute of Medicinal & Aromatic Plants (CIMAP), Lucknow. As a result CIMAP/MAS-1 was developed in 1983 and later Shivalik (1990), Gomti (1995), Himalaya (1996) Kosi (1998) and recently released CIM- Kranti were also developed. In spite of development of many improved verities like Kosi and recently released CIM- Kranti, most of the farmers are cultivating old varieties like Shivalik and Himalaya developed during 1990 and 1996 due to lack of awareness.

There is need to create awareness among farmers to adopt cultivation of recently developed high quality varieties for high yield in terms of quality and quantity. In this paper we were emphasized on routine industrially accepted and viable technology used for the extraction of essential oil of menthol mint and production of crystals of different grade and quality as flakes and bold crystals. The physico chemical and instrumental testing is also mentioned hereby compare with the International and National standards.

Materials and Methods

The mint oil is mainly produced in the region of Uttar Pradesh, Bihar, Madhya Pradesh & Himalayan region, which is extracted by the Mentha leaves by the help of water steam distillation method and the yield obtain is about 0.3 to 0.5% .The major demand is the Menthol crystals which are mainly

used by tobacco, pan masala, and pharmaceutical industries. These crystals are prepared by the two methods.

Convectional Method

This method practices into the deep freezer which is at -45°C about 48 hours. Further the freeze oil is transferred into basket centrifuge in which the menthol crystals are separated in the form of flakes which is also known as DMO (dementholised oil).

Improved Method

This process is basically used for the separation of undesirable products like monoterpenes & menthone. The oil which is remaining is further rectified and known as terpeneless oil.

The *Mentha arvensis* is tested for its physical and chemical properties like viz optical rotation, refractive index, specific gravity, solubility, congealing points and flash point and compared with National standard as shown in Table-III. Some important tests as alkaline test, paper test, water test and solid sedimentation test were also performed to check the quality as well as any adulteration or foreign materials involvement. Further, the oil of *mint* was also analyzed for determination of valuable and major components using Hewlett Packard 5890 series II gas chromatograph equipped with flame ionization detector (FID) and Carbowax 20mm polar fused silica capillary column (30m x 0.32mm.). The injector and detector temperature were maintained 210^0C and 220^0C, respectively. Nitrogen was used as carrier gas with the flow rate of 1.5 ml/min. Sample volume of 0.1 µL with the split less mode of injection was used (split ratio 60:1). . Initial Oven temperature was programmed at 130°C, held for 5 minutes, then raised to 185°C at the rate of 2°C/min and held for 5 minutes, finally raised to 230°C at the rate of 8°C/min and was held for 20 minutes. Temperature of the Injector and detector was maintained at 250°C and 270°C respectively. The components were identified by comparing the retention time with those reported in the literatures. Percentage of individual component was calculated based on the GC peak areas. The oil was also compared with International Standard as shown in Table-IV .

During the crystallization of menthol mint oil, the menthol flakes and menthol bold crystals were also tested for purity and odour evaluation as shown in table-II.

Results and Discussion

Table I shows the *Mentha arvensis* oil parameters according to which the sample is tested. The researches shows that the menthol content of Uttar Pradesh and Himalayan is good that any other region. Where in the case of Himalayan oil, it is slightly superior in its appearance and the melting point. Based on chromatographic analysis of tested sample, major components as L-Menthol 68.3%, menthone 8.2%, Isomenthone 4.4%, menthol isomers 4.5%, menthyl acetate 4.3%, limonene 1.2%, cis-3-hexanol 0.2% , mixture of various terpenes 7.7% and total menthol content 76.9% were found. The same sample was processed for production of menthol flakes by conventional method, the recovery of flakes was found 65% with process lose of 1%. The dementholised oil was also produced as byproduct with recovery of 30%. The high value of congealing point indicates the maximum purity of L- Menthol and it was found 99.0% by GLC analysis. In second improve method, the purity of menthol flakes was found 99.5 % or above with no traces of monoterpenes in final product. It means terpeneless menthol flakes are superior in odour evaluation and widely used in flavour, Tobacco and pharmaceutical industries.

Table-I Quality characteristics of *Mentha Arvensis*

S.No.	Parameters	Values/ Results
1.	Optical Rotation	-33.5°C
2.	Specific Gravity at 27°C	0.8988
3.	Solubility in 70% Ethyl alcohol at 27°C	Up to 3 volume
4.	Refractive Index at 27°C	1.4590
5.	Congealing Point	21°C
6.	Alkaline Test	Transparent or negative
7.	Solid Sedimentation test	0.02%
8.	Paper test	Negative or pure
9.	Water test	0.63%
10.	Flash Point	75°C
11.	L-Menthol	68.3%
12.	Total Menthol Content(TMC)	76.9%
13.	Menthone	8.2%
13.	Isomenthone	4.4%
14.	Menthyl acetate	4.3%
15.	Menthol isomers	4.5%
16.	Limonene	1.2%

Table -II Quality characteristics of flakes

S.No.	Parameters	Values
1.	Congealing point	44°C
2.	Optical rotation	-52
3.	L-Menthol	99.5%

Table -III National standard (BIS) of oil of *Mentha Arvensis*

S. No.	Characteristic	Requirement	Method of Test
1.	Colour and Appearance	Colourless and pale yellow or greenish yellow liquid	IS 326(Part 2)
2.	Odour	Characteristic strong minty, herbal followed by cooling sensation	IS 2284
3.	Relative density at 27 °C	0.877 to 0.912	IS 326(Part 3)
4.	Refractive index at 27 °C	1.4560 to 1.4640	IS 326(Part 5)
5.	Optical rotation at 27 °C	-35° to -45°	IS 326(Part 4)
6.	Solubility in ethanol (volume fraction 70%) at 27 °C	2 to 4 volumes of ethanol	IS 326 (Part 6)
7.	Esters as menthyl acetate percent by mass,	3 to 15	IS 326(Part 8)
8.	Free alcohol as l-menthol percent by mass, *Min*	60	
9.	Total alcohol as l-menthol percent by mass, *Min*	65	IS 326(Part 9)
10.	Ketone as menthone percent by mass (using free hydroxylamine method	5 to 20	IS 326(Part 11)

Table -IV International standards (ISO) of oil of *Mentha Arvensis*

S. No.	Characteristic	Requirement	Method of Test
1	Appearance	Clear, mobile liquid	
2	Colour	Almost colourless to amber yellow	
3	Odour	Characteristic of mint, menthol-like	
4	Relative density at 20 °C	0.890 to 0.910	ISO 279
5	Refractive index at 20 °C	1.4570 to 1.4650	ISO 280
6	Optical rotation at 20 °C	-22° to -13°	ISO 592
7	Miscibility in ethanol (volume fraction 70%) at 20 °C	1 volume of oil shall require a maximum of 4 volumes of ethanol to obtain a clear solution.	ISO 875
8	Acid value	Maximum: 1	ISO 1242
9	Ester value	8 to 25 Corresponding to an ester content, expressed as menthyl acetate, of 3% to 9%	ISO 709
10	Menthol content by determination of ester value after acetylation	40% to 60% (expressed as free menthol)	ISO 1241
11	Carbonyl value	91 to 146 Corresponding to a menthone content of 25% to 40%	ISO 1271
12	Flash point	Mean value is +75 °C (of partially dementholised oil)	

Conclusion

It is clearly indicated from the analytical data that the impact of main components on the odour intensity of menthol mint oil is high. Based on chemical profile, percentage of major components is around 90%. Total recovery of menthol flakes depends upon the percentage of L- Menthol content of the oil. The purity of menthol fakes is very near to 100 % because it is process at low temperature around -45^0 C.

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