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Extraction, production and analysis techniques for menthol: A review

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Abstract

Menthol (also known as mint camphor) is a cyclic monoterpene alcohol which is found as a major constituent in the essential oils of *Mentha* species and responsible for the distinctive smell and flavour of the plant. Menthol is a major essential oil constituent of a very limited number of aromatic plants, known to exhibit various biological properties. Extraction of *Mentha arvensis* essential oils under different extraction conditions causes the change in essential oil components. In the present review different extraction conditions have been discussed in order to get the maximum yield of mint essential oil as well as menthol. Almost 81% menthol is extracted by hydrodistillation method at temperature condition of 30º C. Steam distillation and microwave extraction have also been used to extract menthol under temperature conditions of 35 to 40 ºC and below 70 ºC. Isolation of menthol from extracted essential oils can be done by various methods such as fractional distillation and chromatographic adsorption. This isolated menthol has wide range applications in many industries including pharmaceutical and beverages. It also has excellent biological activities such as analgesic, antibacterial, antifungal, anaesthetic as well as chemopreventing and immunomodulating actions.

Key words: Essential oil, Extraction, Menthol, Analysis techniques

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1. Introduction

An essential oil is a concentrated hydrophobic liquid which consists of volatile (easily evaporated at normal temperatures) compounds present in plants. Other names of essential oils are volatile oils, ethereal oils, aetherolea, or simply as the oil of plants from which they are extracted. The name of essential oil is derived from essence because essential oils are responsible for fragrance or smell of plants from which it is derived. Plants produce essential oils for their needs. Generally, these oils are complicated mixture of those compounds which are organic in nature [1-2], these organic compounds are necessary for plants because they give smell and flavour to the plants [3]. Essential oils are terpene hydrocarbons as well as their oxygenated derivatives such as alcohols, aldehydes, ketones, acids and esters. Terpenes have very complicated chemical nature. Essential oils consist of rather shorter sequences known as monoterpenes and sesquiterpenes or ring-like structures. Besides, they contain also derivatives of phenylpropanes and simple aliphatic compounds [4]. However composition of each specific essential oil varies with the different seasons [2]. A large variety of methods are used for extraction of the oil from plant materials such as steam distillation, hydrodistillation and solvent extraction etc. A most famous method which is used for extraction of oil from plant materials is steam distillation method. In this process, volatile compound of plant material are vapourized by steam, which eventually go through a condensation and collection process [5]. In the method of solvent extraction, the plant material is treated with the solvent, as a result a sticky aromatic compound which is named as a "concrete" is produced. After that alcohol is added to this concrete substance and both are mixed well, the particles of oil are separated respectively [6].

For production of purified and natural ingredients in different fields, essential oils are extensively using in all over the world and their demand is wide-ranging day by day due to their limitless benefits. Thus, a large amount of essential oils are manufactured all over the world to run the factories and industries of flavouring and fragrances, beauty products and the health industry with phytomedicine [7]. Essential oils have also many uses in aromatherapy, which is a kind of substitute remedy in which healing effects are attributed to redolent compounds. Many essential oils have strong and powerful anti-inflammatory and antibacterial characteristics [8-9]. This property makes them a very beneficial and natural process in treatment of skin problems of all types [10]. The very conventional method which is employed in the chemical characterisation of essential oils is gas chromatography coupled with mass spectrometry (GC-MS) [11-13]. The GC technology is used to isolate the components and the MS is used to collect data from those individual components. Due to large and unique isolation and identification power, it is a very important instrument to recognize the quantities of compounds which are present in the essential oil [14].

Mentha arvensis L. (Lamiaceae), generally known as Japanese mint or menthol mint, is very famous and important for having menthol in large quantity in its essential oil. Menthol is universally applied in food, pharmaceutical, beauty products, and scent and fragrance industries in all over the world. The most common components of mint oil is menthol that is also called a monoterpene alcohol. Menthol is a unique and valuable component of plant, which gives the typical minty smell to *mentha* plants. Menthol has many refreshing and chilling characteristics or sensation when it is breath in, munched, swallowed or directly applied to the skin because of its function to chemically activate the cold-sensitive transient receptor potential cation channel. Menthol has been identified as a chief antifungal principle in peppermint oil. Thus in present review, optimizing conditions for getting maximum menthol yield are discussed in detail [15].

2. Essential oil of *Mentha arvensis*

The genus *Mentha* is the part of the Lamiaceae family and consists of a wide range variety of species, one of them is *M. arvensis*. This specie is herbaceous; the essential oil composition depends on the type of verities at different stages of its production and extraction conditions, however the major component is menthol (70% - 90%). The vital components found in *Mentha arvensis* oil other than menthol are menthone, isomenthone, limonene, neomenthol, methyl acetate, beta-caryopyllene, piperitone, alpha- and beta-pipene, tannins, flavonoids, esters, menthyl acetate, ketones, phellandrene, cineol piperitone and sesquiterpene [16]. The components of *Mentha arvensis* oils especially menthol and menthone are valuable due to their refreshing minty flavor and odour which they give to plants. Among all of the components of *Mentha* genus, menthol plays a very important and vital role due to its large number of activities related to health such as antioxidant, antimicrobial, anticancer and anti-inflammatory activities. Menthol has a significant role in medicine for treatment of stomach problem and also in relief of headache. Because of its wide range antiseptic and anti-bacterial properties, it plays important role in treatment of puffiness of gums, breathe freshers, oral herpes and odontalgia [17].

3. Methods of extraction

Various methods are used to extract the essential oils from plant materials such as hydrodistillation, steam distillation, solvent free microwave extraction and supercritical fluid extraction etc. Selection of method is very important, that method would select which give high content of Menthol.

3.1. Steam Distillation

Steam distillation is one of the major methods that are used for the extraction of essential oils from their sources, because of its economic value it is very preferred method. This distillation method is to be performed at low temperature which makes the feasible separation of non-volatile substances and the substances that are not mixable in water underneath the boiling points of every discrete component. Method of steam distillation was used to extract mint oil. The conventional steam distillation in 2-L steam distillation was used to withdraw the essential oils of *Mentha arvensis*. When the first drop of essential oil was deposited in the Florentine (a separator vessel), that was the moment from which the start of each & every distillation was measured. The mint oil was collected directly at the end of every distillation after turning off the electric power. These samples of corn mint oils were measured on analytical standard balance; Content of oil (the yield) was calculated as grams of oil per 100 g of dried Japanese corn mint shoots. The terminology “content” is ordinary used to indicate the amount of essential oil in biomass of 100 g. The word “concentration” is used to indicate the amount of constituents individually which present in mint oil as percentage of the total oil. The total output of different components was calculated from the yield of the essential oil and the concentration of every component in the oil. The yield of menthol was 74 to 79% and the temperature for this extraction was 35 to 40 ºC [18].

3.2. Hydrodistillation

Hydrodisttillation is most important method used for the restoration of essential oils. The herb material is kept in a still and steam is used directly for heating, on the other hand it can also be soaked in warm water or a perforated plate is used for the placement of the herb material above the lowest part of still that is partially filled with water and then heated. After this, the finally obtained vapour mixture goes through the condenser and the condensate is deposited in a Florentine flask where the water and the essential oil are separated from each other; this water can be sent back to the still or, in some cases, used as fragrant water. Some researchers used the method of hydrodistillation for the extraction of mint oil. The leaf samples were dried at 30 ºC in a hot air-oven (IM-30 m, IRMECO, Geesthacht, Germany) to constant weight. The dried leaves of *M. arvensis* were crushed prior to the operation and then 100 g of this dried leaves crushed sample were subjected to hydro-distillation for 3 h using a Clevenger-type apparatus. The distilled *Mentha arvensis* essential oil was desicated over anhydrous sodium sulfate, after dessication, filteration was done the obtained essential oil was stored at −4 ºC until it reached at its analysis. The yield of essential oil of *M. arvensis* was 17.0 g kg^{-1} during summer. The foremost components in the essential oils of *M. arvensis* during summer and winter were found to be menthol (78.90% and 81.30%) and isomenthone (6.35% and 6.19%), respectively [19].

3.3. Microwave extraction

For the research and development purposes on extraction from the medicinal plants, microwave assisted extraction technique is playing a vital role. Dipole rotation and the ionic conduction are two main objectives for the heat generation on which this technique is based, while its output or its efficiency is dependent of the dielectric properties of the herb material. This extraction came into process when the water present internaly in the plant absorbs energy produced by the microwaves and this absorbance become a cause for the enhancement in the pressure inside the plant material, which become the reason cell structure to break and the solvent is then allowed to penetrate into the cell matrix. This method is used for the extraction of mint oil. This microwave assisted extraction was made according to a face-centered central composite experimental design using two stages and two repetitions at the central point. Every experiment for extraction via microwaves was carried out using 2 g of mint powder with 60 ml of ethanol–hexane solvent mixture. The two foremost reagents used for experiments of extraction are Ethanol and Hexane. Vacuum filtration of outcoming extracted material was done by using whatman 1 filter paper and also rinsed with the solvent mixture, which was separated from the oil by evaporation using a rotavapor with a heating bath set at 66 ºC and 55 rpm. When a mixture of two solvents ethanol and hexane was used, the yields of extracted components such as menthone, menthofuran and menthol were rise up [20].

Table.1 Yield of *M.arvensis* essential oil by various techniques.

Techniques	**Conditions**	**Yield of *Mentha Arvensis* essential Oil**	**Oil Contents**
Steam Distillation	35 to 40 ºC	0.43 to 1.06%	Alpha-pinene. Sabinene, beta pinene, myrcene, 3-octanal, limonene, eucalyptol, isopulegon, menthone, isomenthone, menthol, piperitone and beta bourbonene.
Hydrodistillation	30 ºC	1.77%	Menthol and isomenthone are major components.
Microwave extraction	Below 70 ºC	0.36 to 0.61%	Menthone, menthofuran and menthol are major components
Supercritical fluid extraction	Decreasing the pressure from 400 to 200 bar	1.63% to 2.38%	Menthol, carvon and isomenthone are major components.

4. Methods of Analysis

Techniques of chromatography are applicable in the separation and identification of compounds. Gas Chromatography-Mass Spectrometry is used mostly in the isolation and recognition of compounds which are present in the essential oils. The identification of the components of essential oil is usually happen with the assistance of gas chromatography equipped and together with flame ionization detector (FID) and MS detectors, a capillary column (30m×0.25mm, film thickness 0.25μm), and a split. Test conditions may differs according to sample of essential oil to be examined and the column used for its identification or analysis. For example, GC–MS experiments are often performed with injector (by which we inject sample of our own requirements whose identification are required) and detector while the temperatures of injector kept at 250 ºC and those of detector kept at 270°C, respectively, with the oven initially at 50 °C for 2–3min and then risen up of temperature by 3–10°C/min until it reaches 200–240°C, with a flow of 0.7–1.0μL/min. Regularly, the gas used as a carrier gas is Helium at a flow rate of 0.7–1.0mL/min, the samples are diluted (1/10, v/v), and the volume injected is 0.2μL in the split mode (split ratio 1:44). Results are usually prepared with a library mass spectral search program .The recognition and quantification of the various chemical components comprising essential oils can be detected in gas chromatography. The retention time of the peaks is the major factor in recognition of the individual compound while the area of the peak is responsible for the quantification of every single component present in essential oil. The compiled & gathered data can then be compared against reference standards to find out the purity. The short retention time for the resolution of component is one of the main objective in isolation. The suitable and proper parameters must be used to achieve this objective. Chiral stationary phases allow the isolation of components with their optical isomerism. As essential oils contain volatile compounds and less volatile compounds so the temperature must be varies during the identification of compounds in essential oils in gas chromatography. The temperature is necessarily to start off low and then rose every minute until it reaches 200℃ to find out the elution of heavy terpenoids. This makes possible the shortening in the elution times, separate and narrow peaks which brings us more and more near to accurate results.

5. **Methods of isolation of menthol**

Fractional distillation and Chromatographic adsorption methods can be used to isolate menthol from extracted essential oil of mint.

5.1. Fractional distillation

Ordinarily 60% to 85% menthol with 12% to 20% menthone is present in *Mentha arvensis* essential oil, but there is a drawback of presence of these two compounds together, as they match to a certain limit in their physical features, so the isolation of menthol from the essential oil of *mentha* become difficult. Fractional distillation is thought good technique for isolation of menthol from the essential oils of *mentha.* In a research fractionation was carried out under the pressure of 50mm Hg for the distilling off the Xenthone and lower boiling components, after this steam distillation processes the separation of menthol from non-volatile residue left behind, then the purification of menthol proceeds, ideally by their maceration in boiling water that is repeated many times until the melting point of product reached at desired U. S. P standard. Instead of the minor difference in the boiling points of these two components under atmospheric pressure, when pressure is reduced from atmospheric pressure it came into notice that this minor differences in boiling points and the relative volatility is enhanced, so precise fractional distillation proceeds the separation of menthol and menthone from mint oil. Menthol is produced from mint oil, there must be an efficient column that fractionates and an extraordinary reflux ratio, which is not ordinarily to be known on commercial operations. In spite of the fact that fractional distillation is normally utilized in examining the synthesis of essential oils, it has not been used for the creation of menthol from mint oils on a business Scale. The following steps are, consequently, proposed with the goal that less strict requirements on the exact fractionation can be endured: (l) By process of vacuum distillation in a column, menthone and other fractions are distilled off; (2) isolation of menthol from non-Volatile compounds by steam distillation; and (3) menthol purification by process of digestion in water, formation of crystals and drying. Fractional distillation of mint oil was done in a batch column which consisted of about 50 theoretical plates and having a ratio of about 50 at a pressure of 20mm. This fractionally distilled mint oil consisted of 65.2% menthol, 12.5% menthone and 1.8% menthyl esters. After that, distillation was stopped and product obtained which was known as menthone-free-oil, was solidified at normal temperature and weighed almost 70% of total charge. It was consisted of about 89% menthol and 6.5% unstable components. After that, under the atmospheric pressure, steam distillation of menthone free oil was done until all the unstable components disappeared. The condensate isolated into two layers and on cooling, the menthol layer Solidified to a crystalline mass. After drying process, the melting point of menthol was 29 ºC to 35 ºC which was not still reached the U.S. P. standard. The yield of this crude menthol was about 88% of the menthone-free-oil. This unrefined menthol was cleaned by absorption with water to evacuate the polluting influences. One hundred ml of water were warmed with around ten grams of crude menthol in a flask for about 10 minutes. Continuous shaking and stirring was important to keep the oily menthol layer in private contact with the water layer. The menthol layer was solidified after cooling and then it was isolated from solution. It was melted between 38° C to 40.5 °C on drying. The process of digestion using another 100ml of water was repeated once again. Enhancement in melting point of the product was within U.S.P specifications i.e. 41.6 °C to 42.7 °C. Long needle like crystals of pure product obtained was nearly 9.5g. Almost 92% USP standard menthol was obtained from crude menthol. There is small loss of menthol with impurities in solution of water so at room temperature; the solubility of menthol is only 0.42 g/liter of solution. We isolate about 85% of the total menthol comprised in the mint oil.

Table.2 Yield of menthol obtained by fractional distillation

Fractional distillation		
Conditions		Yield
Pressure	20mm	89% menthol
Reflux ratio	50	
Temperature	23^0C	
Melting point at 1st digestion process	38 to 40.5^0C	88% menthol
Meting point at 2nd digestion process	41.6 to 42.7^0C	92% menthol

4.2. Chromatographic adsorption

Despite the fact that the composition of mint oils is very confused, the main constituents might be named menthol, menthone, menthyl esters, and terpene hydrocarbons. Yield obtained from *Mentha Arvensis* has about 60% to 85% menthol in it. The two components which are present in mint oil in large quantities are menthol and menthone. Quantitative yields of both menthol and menthone can be acquired in a straightforward procedure operable on a commercial scale using adsorbents and specific eluants. The corn mint oil is dissolved in a generally non polar solvent and after that passed over an adsorbent;

the adsorbed material is then washed with eluants to isolate the menthone and menthol into obvious divisions. The menthol and menthone might be then isolated from the solvents by any ideal methods, as by dissipating the solvents. The adsorbents might be those typically utilized in chromatographic adsorption, for example fuller's earth, magnesia in activated form, charcoal in its activated form, alumina in its initiated forms and so forth. The eluants are picked with the object of acquiring quick and complete freedom of one adsorbent at any given moment into obvious parts. At the point when absorption of mint oil was done at the top of column and afterward it was washed with appropriately chosen solvents then the adsorbed solutes are compelled to relocate through the column. Under these conditions, the components which are not strongly adsorbed move quickly and the strongly adsorbed components move at a low speed. Because of this differential relocation, the components which are adsorbed gradually isolated from each other. The adsorption affinity of main components in mint oil was in following order menthol, menthone, methyl esters and terpene hydrocarbons, the last being least adsorbed. In a research, corn mint oil consisted of almost 65.2% menthol, 12.5% menthone and 1.8% methyl esters were used as raw material. Carbon in the activated form was crushed and passed through 100 or 200 sieve and then it was used as an adsorbent. Internally 6.5cm glass column was squeezed with 1200 grams of the adsorbent described above to 50 cm tallness. Mint oil which was refined by steam distillation was 25 grams, was dissolved in a hydrocarbon solvent at a boiling point of 77-115 ºC to make solution of 150ml and then it was adsorbed at peak portion of the column. Progressively the column was developed with 1000ml by volume mixture of hydrocarbon solvent. The adsorbent proportion was 1:50 when the direct rate of stream of eluant kept at 10mm/min. The solvents were wiped off after gathering the fractions of percolate. Three obvious fractions were acquired: the terpenes and menthyl esters were cleaned out with water first; the menthone fraction turned out straightaway; and the last fraction contained just menthol. The menthol melted at 41 to 43 ºC. The yield of menthol and menthone were 95% and 90% individually.

Table.3. Yield of menthol obtained by chromatographic adsorption

Chromatographic adsorption		
Conditions		Yield
Melting point	41-43^{0}C	95%
Boiling point	77-115^{0}C	

5. Uses and economic importance

Essential oils generally have number of medicinal applications due to its antioxidant [21], antimicrobial [22], anticancer and anti-inflammatory activities. *Mentha arvensis* is monetarily vital because of great source of menthol. Oil from *Mentha Arvensis* contains 70% of menthol which is utilized for various purposes. Menthol has numerous curative uses, for example menthol is usually added to drops for curing cough and other medications of cough to help calm sore throats, hacks, and mouth and throat aggravations. Numerous menthol creams exist that are used for massaging directly on the skin to help soothe muscle throbs and spasms, sprains, skin aggravations, and migraines. Numerous objectives for dental use, for example toothpastes, floss picks, and mouthwashes contain menthol to help scrub and refresh the mouth and avert awful breath. It is a scent fixing utilized in decorative cosmetics, fine aromas, shampoos, different toiletries and furthermore in family unit items, in invigorating creams and moisturizers [23]. Menthol as a noteworthy dynamic constituent has been utilized for a considerable length of time as customary medicines for various illnesses including contaminations, sleep deprivation, and peevish inside disorder just as a creepy crawly repellent. Menthol is also added to topical and fluid definitions for its proposed antibacterial and antifungal properties notwithstanding its individual pharmacological activities, for example the cooling impact or for gustatory and olfactory upgrades of the detailing. One property of menthol that makes it a good definition vehicle is the expansion in dermal retention for some hydrophilic mixes as a perforation enhancer. World formation of menthol was assessed as 3500tons in most recent one and half decade (15years) and is likely significantly more today. Oil from peppermint sources contains 30-50% menthol and the world production of peppermint oil is about 8000 tonnes per year.

6. Conclusion

Essential oils are sweet-smelling slick fluids acquired from plant material, for example blooms, buds, seeds, leaves, twigs, bark, herbs, wood, fruits and roots. Essential oils are known as optional metabolism active components and they are compulsory for plant's immunity as they show antimicrobial properties. *Mentha arvensis* is a well-known *Mentha* specie which contains an adequate measure of menthol in its oil. Menthol is extricated from *Mentha arvensis* essential oil by various extraction techniques for example, hydrodistillation, steam distillation, microwave extraction and numerous different techniques however stream distillation and hydrodistillation are most imperative since they give high amount of menthol. Menthol has discovered applications in different areas including the pharmaceutical and industries those produce cosmetics and to the tobacco and nutrition industry where it is utilized as a source of increasing flavour and making products tasteful, additive and for its cooling qualities. Various biological properties have been attributed to menthol and the mint oils wealthy in menthol including antibacterial and pain stimulating impacts among others. One of the significant impacts of menthol is the impression of coolness delivered when it is to be eaten or chewed, breathed in or its application to the skin.

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