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Original Research Article

Chemical Composition of Moroccan Commercial Essential Oils of Mint: Mentha Spicata, Mentha Piperita, and Mentha Pulegium

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ABSTRACT

Mentha is a plant native to the Mediterranean region and is cultivated throughout the world due to its use in different areas. Mentha Spicata, Mentha Piperita, and Mentha Pulegium are among the most important aromatic plants of this genus. Recent studies have shown that the essential oils of these plants contain components that are active in the oral cavity due to their antibacterial, antifungal, and anti-inflammatory activities. However, the composition of these essential oils can be modified by environmental factors such as light, precipitation, the growing site, and soil, which might directly influence their activities. The purpose of this study is to physically and chemically characterize the different commercial essential oils of mint: spearmint, peppermint, and pennyroyal in order to determine their quality. These essential oils were collected from different regions in Morocco. The quality of the essential oils was determined by organoleptic properties and analysis of their constituents using thin layer chromatographic (TLC) technique. The results showed variations in the composition of some major components of these essential oils.

***Keywords*:** Essentials oils, Mentha spicata, Mentha Piperita, Mentha Pulegium, Thin layer chromatography

Introduction

Essential oils (EO) are volatile hydrophobic liquids extracted from plants, which often have strong aromas. They are extracted by various processes, such as hydrodistillation or solvent extraction, from the oil ducts, resin ducts, glands, or trichomes of plants. Essential oils have various therapeutic properties such as antiseptic, anti-inflammatory, diuretic, tonic, antispasmodic, and antibacterial activities. They are applied in different fields such as health, agriculture, cosmetics, and food processing.[1]

The quality of an EO is determined by physical tests (density, refractive index), and chemical tests (Thin layer chromatography). The methods of analysis are standardized in accordance to official compendia such as monograph in the European Pharmacopoeia, 7th edition, and in the AFNOR standards NF T 75-400 and 401, equivalent to ISO 7. An EOBBD (Essential Oils Botanically and Biochemically Defined) is considered to be 100% pure, 100% natural, and 100% total in terms of physical and chemical tests. It is qualified as pure marked by the absence of contamination by any other EO component and the fact that it was extracted from a homogeneous set of plants. It is qualified as natural by the absence of chemical solvent or synthetic molecule. It is qualified as total based on the collection of fractions by the technique of hydrodistillation with no removal of essential oil components.[2]

The quality and chemical composition of EOs depend on various factors, such as the geographical location of cultivation, the time of harvest, the part of the plant used, stage of the vegetative cycle, production methods, storage conditions, and storage duration.[1,3,4]

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In this study, the essential oils of three Mentha species were chosen because of their effects in the oral cavity. These include essential oils of spearmint (Mentha spicata), peppermint (Mentha pipereta), and pennyroyal (Mentha pulegium). The Mentha genus is indigenous to the Mediterranean region and is cultivated worldwide. These plants' extracts are mainly used for their antibacterial, antifungal, and antioxidant properties in the oral environment.[4-6]

Studies have shown differences in the chemical composition of essential oils extracted from pennyroyal, spearmint and peppermint.[7,8]

Therefore, the objective of the present research is to determine the chemical composition of spearmint, peppermint, and pennyroyal EOs collected from different localities and the assessment of their qualities.

Materials and Methods

Collection and organoleptic characteristics of the essential oils

The essential oils used in this study were collected from different sources and localities (drugstore, cooperative, herbalist) in Morocco within the months of March to June, 2021. The organoleptic characteristics such as flavour, colour and taste of the different essential oils were noted.

Preparation of the essential oil samples

The essential oils (22 µL each) were taken, supplemented with 2 mL of toluene. This solution was then stored in Eppendorf tubes to be used for TLC analysis.

Preparation of TLC reference standards

Aliquots (20 µL) of Thymol, Carvone, Menthol, Menthyl acetate, Pulegone, Limonene, Cineole, Menthofuran, Piperitenone, and Menthone were added to 1 mL of toluene. Thymol and menthol (0.01 g each) were added to 1 mL of toluene.

Thin Layer Chromatographic (TLC) analysis

The TLC analyses of the essential oils of the test samples and that of the reference standard compounds were performed on precoated

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aluminium-backed silica gel plates. The mobile phase consisted of a mixture of solvents (toluene, 95%, and ethyl acetate, 5%). The elution was carried out for one hour. The plates were dried in a hot air oven at 100°C for 2 min, and thereafter sprayed with vannilin sulphuric acid spray reagents and then dried in the oven for 8 to 12 min.

Results and Discussion

Organoleptic properties

The organoleptic properties in terms of colour, odour and taste of the essential oils of the three Mint (Spearmint, Peppermint, and Pennyroyal) are presented in Tables 1, 2 and 3.

Thin layer Chromatography (TLC) analysis

Essential Oil samples of the three mints (Spearmint, Peppermint, and Pennyroyal) obtained from different sources were subjected to TLC analysis with the aim of detecting the chemical composition using thymol, carvone, menthol, menthyl acetate, pulegone, limonene, 1,8-cineole, menthofuran, piperitone, and menthone as reference standards. Eleven (11) samples of spearmint essential oils were analyzed. The results show the appearance of six (6) distinct spots altogether (S1 – S6) with their Rf values calculated (Table 4). The first spot (S1) corresponding to menthol, which was present in the majority of the samples but with different intensities, except for samples "1", "2", and "3", in which the spot was totally absent. The second spot (S2) (a yellowish-brown spot) corresponding to piperitenone was present in the majority of the samples, but absent in samples "1", "2", and "3" in addition to samples "3*" and "9". The third spot (S3) corresponding to the compound carvone, was found only in four of the samples: "4", "5", "6," and "8". On the other hand, the fourth spot (S4), which corresponds to 1,8-cineole, appeared in most of the samples, while it was absent in samples "2*", "3", "3*", and "6". Menthone designated as spot number 5 (S5) was only present in samples "2", "3", "4", "5", and "7", while a single spot of menthofuran (S6) was noticed in only one sample (sample "9"). There was no thymol, menthyl acetate, pulegone, and limonene in any of the samples.

The TLC of samples of the same brand of essential oil purchased from different places were included. These samples were designated as "2", "2*", "3", and ‘‘3*" as shown in the table. Samples "2*" and "3*" consisted of a single component, which is menthol with low intensity.

The study, conducted in different regions of Morocco (Settat and SAIS valley), revealed that in the Settat region the main components of spearmint essential oil were carvone (57.00%), limonene (9.14%), and germacrene (8.12%), and in the SAIS valley region, the components were carvone (44.94%), dihydrocarvyl acetate (15.40%), and limonene (8.42%). The study conducted in 2010 in the same region revealed carvone (73.01%), limonene (8.54%), and 1,8-cineole (6.76%).[7]

The TLC profile of essential oil of peppermint is shown in Table 5, the values represent the Rf of the different spots seen in the TLC chromatogram (Figure 1). A total of four distinct spots were identified (S1 – S4). Spot S1 which corresponds to menthol was present in all the samples except in samples "13" and "19".

Table 1: General and organoleptic characteristics of commercial spearmint Eos

Brand	Color	Odor	Savor	Place of Purchase
1	Colourless	++	Minty	Cooperative
2	Colourless	+	Minty	Herbalist
2*	Colourless	+	Minty	Herbalist
3	Colourless	++	Minty	Cooperative
3*	Colourless	+	Minty	Herbalist
4	Colourless	++	Minty	Cooperative
5	Colourless	++	Minty	Cooperative
6	Colourless	++	Minty	Cooperative
7	Colourless	++	Minty	Cooperative
8	Colourless	+	Minty	Herbalist
9	Colourless	++	Minty	Herbalist

*: same brand

Table 2: General and organoleptic characteristics of commercial peppermint Eos

Brand	Color	Odor	Savor	Place of Purchase
10	Colorless	++	Minty	Cooperative
11	Pale yellow	++	Minty	Parapharmacy
12	Pale yellow	++	Minty	Cooperative
13	Pale yellow	+++	Minty	Parapharmacy
14	Pale yellow	++	Minty	Cooperative
15	Colorless	++	Minty	Parapharmacy
16	Pale yellow	+++	Minty	Herbalist
17	Pale yellow	+++	Minty	Herbalist
18	Colourless	++	Minty	Herbalist
18*	Pale yellow	+++	Minty	Herbalist
19	Pale yellow	+	Minty	Herbalist

*: same brand

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The intensity of the spots differed among the samples. The S2 spot corresponding to piperitenone was present in six samples with low intensity but absent in samples "10", "13", "14", "17", and "19". An S3 spot corresponding to 1,8-cineole appears in all samples except sample "19", and has a high intensity in sample "13". The S4 spot corresponding to menthone was observed in all samples except sample "19". The components thymol, carvone, menthyl acetate, limonene, menthofuran, and pulegone were absent in all samples. The spots for all the reference standards were absent in sample "19".

The main active components of peppermint (menthol and menthone) were present in all the EOs analysed. Menthol is of therapeutic value due to its anti-inflammatory, antiviral, and anesthetic properties.[19] The constituent 1,8-cineole which was found to be present in all the EOs analysed is a compound with anti-inflammatory and anti-anxiety properties.[20]

The study of the chemical composition of peppermint essential oil carried out in Taounate city, Morocco revealed the main constituents as follows: menthol (46.32%), menthofuran (13.18%), menthyl acetate (12.10%), menthone (7.42%), 1,8-cineole (6.06%), neomenthol (4.79%), limonene (3.01%), and carvone (1.02%).[8] Al-Mijalli et al highlighted the chemical composition of peppermint from the El Gharb region in Morocco, with the main components being menthol (38.73%), menthone (29.24%), and 1,8-cineole (6.75%).[10] In Algeria, peppermint is mainly composed of trans-carveol (58.98%), D-limonene (19.94%), carvone (2.07%), and 4-terpineol (3.01%).[11]

The essential oils of pennyroyal were analyzed by TLC using the same reference standards as mentioned above. The TLC chromatogram showed four distinct spots with their Rf values presented in Table 6. The fourth spot (S4) which corresponds to pulegone, was identified as the major component of this variety in Morocco , and it is well reported in various articles due to its anti-inflammatory, analgesic, antifungal, and antibacterial properties.[17,21,22] This compound was most intense in sample "27". The appearance of a spot (S3) was noticed in all the samples, and this spot corresponds to the compound piperitenone. Unfortunately, the other two spots (S2 and S1) remain unknown because there were neither reference standards nor bibliographic data that could be used to match them. The spot S2 was observed only in three of the samples (samples "20", "21", and ‘‘22’’), while spot S1 was present in the majority of the samples, but absent only in two of the samples (samples "23" and "24").

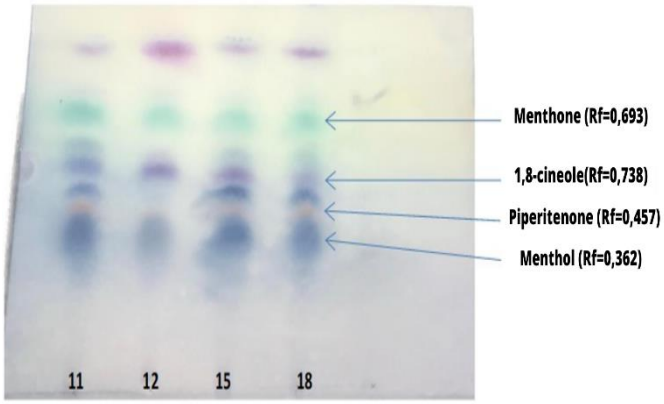

Figure 1: TLC Chromatogram of peppermint essential oil samples (11,12,15 and 18) from different brands.

Table 3: General and organoleptic characteristics of commercial pennyroyal Eos

Brand	Color	Odor	Savor	Place of Purchase
20	Pale yellow	+++	Minty ++	Cooperative
21	Pale yellow	+++	Minty ++	Cooperative
22	Pale yellow	+++	Minty ++	Cooperative
23	Colorless	++	Minty +	Cooperative
24	Colorless	++	Minty +	Herbalist
25	Colorless	+++	Minty +	Herbalist
26	Colorless	++	Minty +	Herbalist
27	Colorless	+++	Minty +	Herbalist
28	Colorless	+++	Minty +	Herbalist

Table 4: TLC Profile (Rf values) of spearmint essential oil samples

S	1	2	2*	3	3*	4	5	6	7	8	9
S1	-	-	0.445	-	0.445	0.376	0.376	0.376	0.382	0.354	0.354
S2	-	-	-	-	-	0.462	0.462	0.462	0.471	0.412	-
S3	-	-	-	-	-	0.5	0.5	0.5	-	0.45	-
S4	0.586	0.586	-	-	-	0.617	0.617	-	0.675	0.522	0.693
S5	-	0.771	-	0.771	-	0.703	0.703	-	0.719	-	-
S6	-	-	-	-	-	-	-	-	-	-	0.806

Table 5: TLC Profile (Rf values) of peppermint essential oil sample

Sample	10	11	12	13	14	15	16	17	18	18*	19
S 1	0.362	0.362	0.362	-	0.362	0.362	0.393	0.393	0.393	0.393	-
S2	-	0.456	0.456	-	-	0.456	0.460	-	0.460	0.460	-
S3	0.738	0.738	0.738	0.738	0.738	0.738	0.690	0.690	0.690	0.690	-
S4	0.693	0.693	0.693	0.693	0.693	0.693	0.733	0.733	0.733	0.733	-

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Table 6: TLC Profile (Rf values) of pennyroyal essential oil samples

S	20	21	22	23	24	25	26	27	28
S1	0.141	0.141	0.141	-	-	0.109	0.158	0.158	0.158
S2	0.187	0.187	0.187	-	-	-	-	-	-
S3	0.393	0.4	0.4	0.382	0.322	0.335	0.329	0.329	0.329
S4	0.593	0.593	0.606	0.579	0.509	0.509	0.548	0.548	0.548

A study conducted in Morocco in the region of Ouazzane revealed that the main component of pennyroyal essential oil is pulegone (40.98%), followed by menthone (21.16%), and α-terpineol (7.98%).[12] In the Middle Atlas regions, a study was conducted in several districts. In the first district, Khénifra, the essential oil components were pulegone (81.49%), humulene (2.89%), and caryophyllene (1.7%). In the Mrirt district, pulegone (71.97%), peperitenone (26.07%), and chrysanthenol (0.80%) were the major components. In the Azrou region, it was found that pulegone (68.86%), piperitenone (24.81%), and chrysanthenol (1.03%) were the major components.[13] In the northern region, the components identified in the essential oil of pennyroyal extracted by the hydrodistillation technique were pulegone (77.16%), piperitenone (6.54%), and piperitenone oxide (1.82%),[14] while that extracted by the steam distillation technique, the main components were pulegone (33.65%), α-terpinenyl acetate (24.29%), and 1,8-cienole (1.05%).[15]

Ait-Ouazzou et al highlighted the main components of pennyroyal essential oil in the Taourit region as pulegone (69.80%), piperitenone (3.10%), and isopulegone (1.80%),[16] whereas the study conducted in the regions of Marrakech and OuedLaou highlighted the major components to be pulegone (75.48%), carvone (6.66%), and dihydro-carvone (4.64%).[17]

The chemical composition of essential oils can vary according to climate, soil composition, plant organ, age, and stage of the vegetation cycle of a plant, as well as differences in species and chemotypes and the extraction procedure of the essential oil.[4,7]

The absence of major components in some commercial essential oils and their presence in others give an indication of the quality of these oils, which directly influences the activity of this product. According to a study conducted on peppermint, researchers found that the main components, menthol, menthone, and 1,8-cineole, have antimicrobial activity against gram-positive and gram-negative bacteria, and that the absence of any of these components reduces or abolishes this activity.[17,18]

In addition, it can also be said that the quality of essential oil may be dependent on the place of purchase since we have analyzed essential oils of the same brand but purchased from different places and it was found that there were differences in the results of the components. These differences may arise from the fact that these oils could have undergone modifications, such as dilutions by other products or water.

Conclusion

Several factors, such as the place of purchase, the geographical location of cultivation, the time of harvest, the part of the plant, production methods, storage conditions, and duration of storage can combine to modify the quality of essential oils and consequently diminish or abolish the therapeutic activity it possesses. To avoid the side effects caused by deteriorated or adultrated products, the use of essential oils must be preceded by the quality assessment (label composition).

Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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