Chemical Study by GC-MS of the Essential Oils of Certain Mints Grown In the Region of Settat (Morocco): Mentha Piperita, Mentha Pulegium and Mentha Spicata

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Abstract

This work aims to evaluate the composition of the essential oils of certain mints grown in the region of Settat Morocco and to determine their chemical compositions. The essential oils obtained by hydrodistillation have an average yield of: 2.93% for Mentha pulegium, 1.23% for Mentha piperita and 0.91% for Mentha spicata. Analyzes of oils by GC-MS, showed the presence of several components including Menthone (42.97%) and Menthol (27.64%) are the major components of Mentha piperita, piperitone (31.27%) and piperitenone (22.98%) were obtained as the major compounds of Mentha pulegium, and so the main essential oil compounds of Mentha spicata are carvone (33.14%) and trans- Carveol (20.06%).

Keywords: Essential oils; Chemical composition; GC-MS; Mentha piperita; Mentha pulegium e; Mentha spicata.

Introduction

Medicinal plants are important for pharmacological research and drug development for thousands of years, man used plants found in nature, to treat and cure diseases. The use of plants in herbal medicine is very old and is currently a region of interest to the public [1]. Recently, researchers have estimated that there are about 400,000 plant species worldwide, of which about one-quarter or one-third have been used by societies for medicinal purposes [2,3]. Mint has a privileged place in herbal medicine; it was cultivated since ancient times for its medicinal properties. The importance of mints in the world and the interest of the Moroccan population in their use. Tradition attributes it aromatic properties (tonic, fortifying) and digestive properties (fight heaviness, bloating, etc.), used to relieve, colic, nausea, diarrhoea, and Crohn’s disease [4].

Material and Methods

Plant Material

Samples of the aerial part (stems, leaves and flowers) of Mentha pulegium, Mentha spicata and Mentha piperita were collected in March (2018) respectively in the region of Settat (Morocco). These three species have been verified by a botanist Professor Adnane EL-YAACOUBI in EST-Khenifra (University of Sultan Moulay Slimane).

Extraction of the Essential Oil

A vegetal mass which represents a proportion of about 40% is completely immersed in water (proportion of 60%), a few grains of pumice are added, and all is then brought to a boil. The heating is maintained at a gentle temperature for 4 hours, the vapours are
condensed in a refrigerant and the essential oil and water separate by difference in density. The HE was extracted with ethyl acetate and then dried with Na2SO4. The solvent is evaporated using a rotary evaporator; the oil is recovered, placed in opaque samples which are stored in the refrigerator analysis.

**Calculation of Yield**

The yield of essential oil (expressed as a percentage) is calculated by the ratio between the weight of the oil extracted and the weight of the plant material used [5-8]. The essential oil yield was determined with respect to the dry matter, evaluated from 100g dried in an oven for 48 h at 60°C. The essential oil obtained is stored at a temperature of 4°C in the dark.

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Y(\%) = \left( \frac{w_{\text{oil}}}{w_{\text{ms}}} \right) \times \left( \frac{m_{\text{ms}}}{100} \right)
\]

**Gas Chromatography (GC) / Mass Spectrometry (MS)**

The analysis of the essential oils was carried out by gas chromatography coupled with mass spectrometry (GC-MS). Coupling was performed on a Hewlett-Packard model 5970 (quadrupole detection system) equipped with a fused silica capillary column of 2mm x 0.23mm DB1 type; temperature programming from 50°C to 200°C, with a gradient of 3°C min⁻¹. The retention indices were determined by gas chromatography on two fused silica capillary columns (25m x 0.25mm) of the type OV-101 and Cabowax 20 M, with temperature programming identical to that used for the coupling. (Shimatzu GC-14A equipped with a flame ionization detector and a C-R4A model integrator).

**Results and Discussion**

**Yield in Essential Oil**

The average yield of essential oil of each species was calculated according to the dry plant material obtained from the aerial parts (stems, leaves and flowers) of the plants studied. The yield of essential oil obtained is given in Table 1. The average yields of essential oils were calculated according to the dry plant material of the aerial part of the plant. *Mentha pulegium* samples were about 2.93% higher than *Mentha piperita* at 1.23%, and *Mentha spicata* at 0.91%. The *Mentha piperita* rate is almost identical to that obtained by Adjou, E.S. et al. [5] which is (1.17%), whereas the *Mentha pulegium* is lower than that obtained by Hmiri, S. et al. [6], and which is 3.30%. for *Mentha spicata* the rate of return obtained by Hmiri, S. et al. [6] is almost identical which is 0.96%, therefore it can be concluded that several factors could be at the origin of these variations: the age of the plants [7-8], that of the tree [9], the nature of the soil and the climate [10-11], the part of the plant subjected to extraction and the period of harvest.

**Table 1**: Yield in essential oils.

| Species         | Average |
|-----------------|---------|
| Mentha piperita | 1.23%   |

**Chemical Composition Analysis by GC-MS**

The gas chromatographic analysis results coupled with the mass spectrometry of the essential oils of the studied plants are represented in (Tables 2). Chromatographic analyzes of essential oils made it possible to identify 26 compounds which represent approximately (99.53%) for *Mentha piperita*, for *Mentha pulegium* 29 compounds which represent approximately (99.84%) and for *Mentha spicata* 30 compounds (99.93%) (Table 2). The analysis of the results given in Table 2 showed all of the following results: Menthone, Menthol was obtained as the majority compounds, with a percentage of (70.61%) in the essential oil of *Mentha piperita*. *Mentha pulegium* essential is characterized by the presence of piperitone and piperitenone as major chemical constituents with a percentage of (54.25%), and as well as the main essential oil compounds of *Mentha spicata* are: carvone and trans-Cardveol with a percentage of (53.20%). Analyzes carried out in Serbia on the essential oil of *Mentha piperita* by Sokovic MD [12], showed the presence of the following main component: menthol (37.4%), menthyl acetate (17.4%) and menthone (12.7%), thus in Italy the results of Ashok, K. S [13] presented the main following compounds: menthanol (36.24%) and menthone (32.42%).

**Table 2**: Chemical composition of the essential oils of *Mentha piperita*, *Mentha pulegium* and *Mentha spicata*.

| Identification | Mentha piperita | Mentha pulegium | Mentha spicata |
|---------------|-----------------|-----------------|---------------|
| Sabinene      | 1.24            | 0.67            | 0.13          |
| γ-Terpinene   | 0.25            | 0.56            | 0.98          |
| Oct-1-en-3-ol | -               | -               | 0.25          |
| Tumerone      | 4.01            | -               | -             |
| Civulene      | 1.99            | 0.98            | -             |
| piperitenone  | -               | 22.98           | -             |
| piperitone    | -               | 31.27           | -             |
| Menthone      | 42.97           | 6.58            | 2.19          |
| Menthol       | 27.64           | 4.31            | 1.42          |
| 1,8-Cineole   | 2.85            | 1.31            | 3.99          |
| Isomenthone   | -               | 0.95            | -             |
| neomenthol    | -               | 3.54            | -             |
| Isomenthone   | 2.48            | -               | 3.33          |
| Limonene      | 1.22            | 1.89            | 2.16          |
| α-terpinol    | -               | 5.07            | -             |
| pulegone      | -               | 5.80            | -             |
| α-Pinene      | 1.57            | -               | 1.06          |
| β-Pinene      | 0.99            | -               | 0.45          |
| Germacrene-D  | 1.75            | -               | 3.14          |
| Camphene      | 0.58            | 0.02            | -             |
Another study in Iran by Behnam, S. M [14] presented the following results: menthon (44.1%), menthol (29.5%), menthylace-tate (3.8%) which are the main compounds of essential oil Mentha piperita. The study conducted in Morocco by Derwich, E [15] on the aerial parts of Mentha piperita showed the predominant major compounds of the following species: Menthone (29.01%), followed by menthol (5.58%), menthylace-tate (3.34%), menthofurran (3.01%), 1,8-cineole (2.40%), isomenthone (2.12%), limonene (2.10%), α-pinene (1.56%), germacrene-D (1.50%), B-pinene (1.25%), sabine (1.13%) and pulegone (1.12%). The essential oil composition of Mentha pulegium determined in our study showed a profile relatively similar to those published for other geographical regions: menthorn (44.1%), menthol (29.5%), menthylace-tate (3.8%), isomenthone (3.8-4.0%), neo-menthol (0.7-1.3%), pule-gol acetate (0.1-1.2%), γ-terpinene (0.9-1.2%) , β-caryophyllene (0.1-0.9%) and β-caryophyllene oxide (0.3-1.9%) in India [20]. On the other hand, this composition is different from that of the essential oil of Mentha pulegium leaves studied in Tunisia and Morocco, where the main components were respectively menthol (48.56%) [21] And pulegone (73%), 33% [22]. The essential oil composition of Mentha spicata in the plains of northern India, the carvone content varied between 45.9% and 77.1% [23]. The percentage of car-vone also varies according to the spearmint oil grown in different countries, eg Egypt (46.4% -68.55%) [24,25], Canada (59% -74%) [26], in Colombia (61.53%) [27]; Turkey (78.35% -82.2%) [28,29], China (55.45% -74.6% [31], Bangladesh (73.2%) [32], Algeria (59.4%) [33] and Morocco (29%) [34] Lesser amounts of carvone were reported in Iranian Spearmint essential oil (22.4%). A chemo-type rich in linalool (82.8%) has also been reported in Turkey [19] in another report on M. spicata essential Iranian oil, α-terpinene (19.7%), piperitone oxide (19.3%) isomenthone (10.3%) and β-caryophyllene (7.6%) have been reported as major components.

Conclusion
The essential oil yield of the plants studied Mentha piperita, Mentha pulegium and Mentha spicata is acceptable. The essential oil of Mentha piperita is rich in Menthone (42.97%) and Menthol (27.64%), the most abundant chemical compounds in the essential oil of Mentha pulegium are piperitone (31.27%) and piperitonenone (22.98%) and for Mentha spicata the presence of carvone (33.14%) and trans-carveol (20.06%). Several factors could be responsible for these variations: the age of the leaves, the nature of the soil and the climate, the part of the plant subjected to extraction and the period of harvest.

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