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Valorization of *Myrtus communis* Essential Oil Obtained by Steam Driving Distillation

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Abstract: This study consists of the valorization of the essential oil of dry leaves of this plant (*Myrtus communis* L.) obtained by steam driving. This research was undertaken in order to characterize the essential oil during spring season of the Myrtle, coming from a mountainous area, located roughly at 100 km in the east of Algiers. The analysis of this oil was carried out by gas chromatography and gas chromatography coupled with mass spectroscopy. This analysis revealed that the 1, 8-cineole and limonene represent the major compound of this oil. Also, the mode of exit versus time of the various compounds of the oil was studied. The fraction collected between 15 and 30 min revealed to contain 47. 33% in 1, 8-cineole and limonene.

Key words: Essential oil, *Myrtus communis* L., extraction, steam distillation, 1, 8-cineole, limonene

INTRODUCTION

Essential oils are oily, volatiles and odorous substances that one can extract from plants which are called aromatic plant. The essential oil is known as a product obtained from a vegetable raw material either by steam drive or by mechanical processes from the epicure of citrus. Essential oil is then separated from the aqueous phase by physical processes (Roatbi *et al*., 2007).

The main goal of this study is the extraction of essential oil from dry Myrtle, plants largely available in the mountainous and rocky areas which surround the Mediterranean. The Myrtle *Myrtus communis* L. is widespread in Algeria especially in the littoral tell Algéro-Constantinois. It is commonly known under the name of Riehen. Another species of the Myrtle can also be found in the central Sahara (Hoggar) called locally Tefeldest (Pandolfi, 1998).

This plants, for a long time marginalized by researchers, has a great importance in popnlar medicine. In Morocco, it is used as stimulant, disinfectant and for local applications on the abscesses, furuncles, ulcerations and burns. The Myrtle is also consumed in the form of infusion against the diarrheas (Gauthier *et al.,* 1988). In Tunisia, the decoction of the fruits is recommended to relieve the gastric pains and ulcer (Messaoud *et al*., 2005). According to a popnlar tradition in Algeria, the branches of the Myrtle are deposited on the graves (the essential oil of Myrtle has a very powerfnl insecticidal virtue recommended in the fight against parasites (Özcan *et al*., 2001)). The Touaregs use it for its properties, they cure the wounds and ulcers; On the other hand, the dried fruits are used as condiments (Pandolfi, 1998). The leaves of Myrtle contain tannins, resinous substances and essential oil. The disinfectants properties of the Myrtle are due to its essential oil, containing especially the terpenes, 1, 8-cineole (Eucalyptol) and others components like esters and alcohols (Ciccarelli *et al*., 2008).

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The Myrtle is small tree, all year long green, aromatic and quite present in the Mediterranean area. The branches are very bulky with a shining green color. Odorous white flowers are visible from May to July. The fruit is Black blue at maturity.

The essential oil from leaves, flowers and fruits of the *Myrtus communis* vary in the composition (Ciccarelli *et al*., 2008). The aim of this study consists of the valorization and characterization of the essential oil of dry leaves of Algerian *Myrtus communis*.

MATERIALS AND METHODS

This study is carried out on dry leaves of Myrtle. The gathering of the samples was made in the north of Algeria, in a homogeneous area (between 15 and 20 trees having the same nature of the ground). The leaves of the Myrtle were dried under protection of heat and light during 30 days, where humidity becomes almost constant. These conditions of drying are those permitting minimum losses of essential oil (Gauthier *et al*., 1988; Sefidkon *et al*., 2006).

For all the extractions, the plants are from the same source and the treatment of the samples is identical.

The equipment used in this study includes:

- **For the Extraction of Essential Oil:** An extractor, a condenser and a heat insulation system. The extractor is a glass column of 10 liters capacity with an external diameter of 22 cm in which the vegetable matter is put.
- **Analysis Equipments:** Gas Chromatograph (GC) and a gas chromatograph coupled to a mass spectroscopy (GC/MS).

The plants are introduced into the packed column. The packing, heat resistant, improves the heat transfer between vegetable and steam. The packing of the plants inside the column improves the residence time of the steam. Condensed steam is recovered in a decanter.

In view of the high number of compounds contained in an essential oil and their chemicals diversity, the CPG on a capillary tube with programming temperature is the best adapted for such analysis (Roatbi *et al*., 2007).

Identification of the chemical composition of the essential oil of the Myrtle was carried out by GC.

Essential oil was dissolved in 5 mL ether solvent and 5 µL of this diluted oil was used under the following GC conditions:

Carbowac stationary phase (20 m*0.25 mm i.d., film thickness 0.25 mm) 80°C for 2 min, then 7°C min^{-1} up to 220°C; detector: FID, detector temperature: 250°C for 4 min, split injection temperature: 250°C, carrying gas: N_2 (1 mL min^{-1}).

We have also analyzed this essential oil by CG/SM on an apolar capillary tube according to the following operating conditions:

Essential oil was dissolved in 5 mL of Hexane solvent and 1 mL of this diluted oil was used under the following GC/MS conditions:

Five MS stationary phase (5% phenyl and 95% methyl-polysiloxane) (15 m*0.25 mm i.d., film thickness 0.25 mm) 100°C for 8 min, then 7°C min^{-1} up to 250°C for 25 min; detector: mass spectrometer, detector temperature: 300°C, Split injection temperature: 250°C, carrying gas: He (1 mL min^{-1}).

RESULTS AND DISCUSSION

Determination of the essential oil yield; The yield in essential oil is given by the following equation:

$$R = (Mh \times 100)/[Mc\,(1-Y)] \quad (1)$$

Where:

R = Yield in essential oil (%)
Mh = Mass extracted oil (g)
Mc = Mass load (g)
Y = Humidity of the vegetable matter

We studied the evolution of drying according to the time (Fig. 1), the Humidity of the plant is determined by the equation:

$$Y = (Mt - Ms)/Mf \quad (2)$$

where, Mt (g) is the mass of the load at the time t, Mf (g) is the mass of the final load after drying, Ms is given by:

$$Ms = Mf \times (1 - Yf) \quad (3)$$

Yf is the humidity of the final load determined by the method of DEAN and STARK (Busman *et al*., 2005).

The yield obtained is 0.33% for the essential oil of the dry leaves collected in the month of March. This result is obtained for optimal experimental conditions (Fig. 2). This value is variable, depending on the load and the collected period (Table 1). The yield obtained for the dry leaves collected in June and under the same operating conditions is 0.39%.

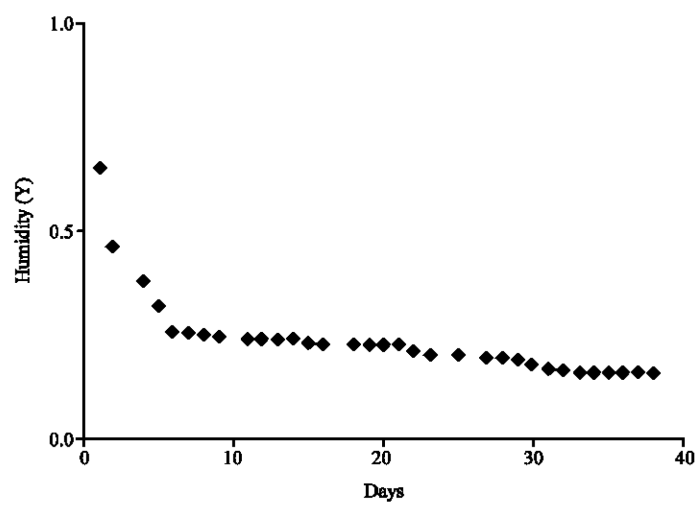

Fig. 1: Evolution of the humidity of the vegetable matter according drying time

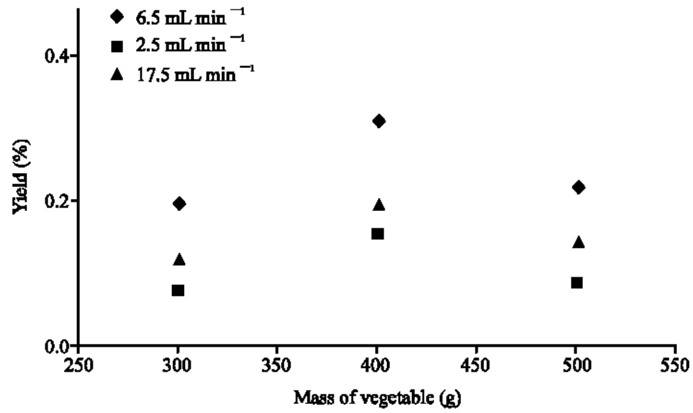

Fig. 2: Evolution of the yield *Myrtus communis* essential oil according to mass of vegetable and various steam flow; steam flow 12.5 mL min^{-1}, mass of the vegetable matter of 400 g, height of the plant bed 32 cm and humidity of the plant: Y = 0.158

Table 1: Evolution of the yield *Myrtus communis* essential oil according to the period and the state of the matter

Period	State of the matte Fresh leaves	Dry leaves	Fresh leaves and flowers	Dry leaves and flowers
March	0.35	0.33	-	-
June	0.41	0.39	0.35	0.32

Fresh leaves; Y = 0.580, Dry leaves; Y = 0.158

This value has been found close to the one found for previous work done in the Maghreb countries. The yield of essential oil for Tunisian myrtle reaches 0.41% in August (Naceurim *et al*., 2006) and for the Moroccan myrtle between 0.29 and 0.36% (Gauthier *et al*., 1988; Aidi Wannes *et al*., 2006). The losses of essential oil during drying does not exceed 10% similar observations were reported by Gauthier *et al*. (1988).

These results showed that the drying of the plant decreases the volume of the vegetable matter and thus allow us to treat greater quantities while preserving the qualities of the essential oils.

According to these results, the quantity of oil essential obtained with a load of 400 g would be the same with 800 g of fresh load. For economic considerations, it would be interesting to treat dry loads, especially for plants with poor yield.

Composition of Essential Oil Myrtle

The analysis by CG of essential oils revealed that the major peak is that of 1,8-cineole and limonene, it reaches a value of 26.4% in March period (Table 2), this value characterizes the essential oils of the Myrtle in other countries, Tunisia 22% (Messaoud *et al*., 2005), Morocco 21% (Aidi Wannes *et al*., 2006) and 12.7-19.6% for the Myrtle of Greece (Gardeli, 2008).

Analysis of essential oil obtained by GC and GC/MS revealed a multitude of components. Twenty one were identified; they represent about 70% of this oil. The analysis by CG/MS gave two peaks, one for 1,8- cineole and the other for Limonene and revealed that the major compound is 1.8-cineol with 15.79% (Table 3).

Evolution of the Essential Oils Versus the Extraction Time

The evolution of the yield versus time has led to a cumulated yield of 0.2% in regard to the relatives losses for each sample (Fig. 3).

Table 2: Evolution of the 1.8-cineole and limonene according to the period and the state of the matter

	State of the matter	
Period	Fresh leaves	Dry leaves
March	26.40	-
June	12.36	10.28

Fresh leaves; Y = 0.580, Dry leaves; Y = 0.158

Table 3: Chemical composition of *Myrtus communis* oils dry leaves (March period)

	Relative contents (%)	
Components	1	2
α-pinene	1.08	2.99
β-pinene	-	2.99
Camphene	0.35	0.45
Pulegone	-	5.69
Limonene	26.40*	8.69
1,8-cineole	-	15.79
p-cymene	3.15	-
C8-aldehyde	-	3.60
Linalool	1.63	1.90
α-terpineol	1.31	1.29
γ-terpineol	1.36	-
Trans-sabinene hydraté	-	2.40
β-phellandrene	-	4.19
Myrtenol	-	1.29
Geraniol	0.33	0.14
Citronellyl acetate	1.26	-
Linalyl acetate	0.60	3.14
Terpenyl acetate	0.49	2.14
Neryl acetate	1.65	1.14
Geranyl acetate	0.60	1.14
Bornyl acetate	-	2.99

1: Components of essential oil identified by GC. (Carbowac stationary phase), 2: Components of essential oil identified by GC/SM. (5MS stationary phase) *1,8 cineole+Limonene

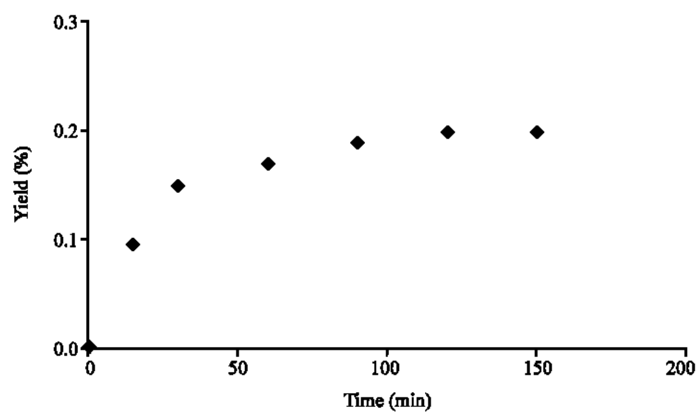

Fig. 3: Evolution of the essential oil yield according to time. Dry leaves (March period)

This Evolution is Characterized by Two Distinct Periods

During the first stage, the extracted amount of essential oils increases rapidly the first 30 min because of the entrainment of oil towards surface. This phenomenon is frequently observed in the case of the plants whose deposits are directed towards outside. The vapour easily destroys the envelope covering the deposit of essential oil. As result, the contact: vapour-oil is quickly established (Boutekedjiret, 2003).

Table 4: Evolution of the components of *Myrtus communis* oils according to the time of extraction. Dry leaves (March period)

Components	Relative contents (%) 0-15 min	15-30 min	30-60 min
α-pinene	1.09	1.002	3.21
Camphene	0.45	0.40	1.40
1,8-cineole+Limonene	13.13	47.33	29.17
p-Cymene	3.38	3.16	-
Linalool	1.89	3.12	1.74
α-terpineol	2.18	0.82	1.49
γ-terpineol	2.19	1.27	7.76
Geraniol	-	1.14	4.92
Terpenyl acétate	2.07	1.28	7.76
Citronellyl acetate	2.18	1.31	-
Linalyl acetate	4.06	-	1.89
Géranyl acetate	1.55	1.14	4.12

The second stage represents the stripping of the oil from the internal toward the surface by diffusion phenomenon, it is the slowest step and it ends with a plateau representing the end of the extraction. This step imposes the kinetics of the process.

Evolution of the Various Compounds According to the Time of Extraction

The first three fractions were analyzed by GC. The majority of the compounds appeared the first 15 min. The results obtained allow us to deduce the fraction of extraction time to consider for recovering one rich compound contained in the essential oil. The results are shown in Table 4.

Fraction 15-30 min is the richest in 1,8-cineole and Limonene, but relatively poor in others compounds. Fraction 30-60 min represents a multitude of compounds with 29.17% in 1,8-cineole and Limonene. This kinetics permits to define a time of extraction of 60 min in order to obtain an essential oil relatively rich in therapeutic components. In the same way, the evolution of the various compounds according to time revealed that 1.8-cineol and limonene are extracted before esters. Thus one can say that the compounds are extracted according to their boiling points.

The fact that the considered components are recovered in the ascending order of their boiling points, allows us to suppose that the steam distillation depends on the phenomena of transfer of the oil located at the surface of the plant.

CONCLUSION

This research is a contribution for characterising the essential oil of *Myrtus communis*. The extraction of this oil was carried out by steam stripping. Essential oils obtained were analyzed by means of two complementary methods, the GC and the GC/MS.

The yield and the composition of the oils obtained are comparable to those reported in the literature. The study of the kinetics of extraction of these essential oils allowed us to select the time of extraction according to the desired composition.

For economic considerations, it would be interesting to treat dry loads, especially for plants with poor yield.

Although, the Myrtle provides relatively poor yields of essential oil, its therapeutic properties makes that one should exploit it.

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