

Extraction of rosemary essential oil by steam distillation and hydrodistillation

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ABSTRACT: Rosemary oil was extracted by both steam and hydrodistillations then analysed by gas chromatography and gas chromatography–mass spectrometry. The effect of time of extraction enabled us to follow the evolution of the yield and oil composition obtained by both processes. Copyright © 2003 John Wiley & Sons, Ltd.

KEY WORDS: *Rosmarinus officinalis* L; rosemary; Labiatae; essential oil composition; extraction; steam distillation; hydrodistillation

Introduction

Rosemary (*Rosmarinus officinalis* L) is an aromatic, medicinal and condiment plant that belongs to the Family Labiatae. It is widely spread in Algeria and broadly used in traditional medicine. Rosemary is selected because it is of interest as a preservative due to its antioxidative characteristics and it is used in the pharmaceutical, food and cosmetic industries. Therefore this work was undertaken in order to contribute to a better knowledge of the essences of rosemary coming from the area of Bibans, located approximately 200 km east of Algiers, and, more specifically, to compare rosemary essential oils obtained by steam and hydrodistillations from a qualitative and quantitative point of view.

Experimental

The vegetal matter used comes from the 'Portes de Fer' in the Bibans area, located 200 km east of Algiers and identified according to the *Flora of Algeria*.¹ A sample specimen was deposited in the Herbarium of the Botany Department at the Algeria National Institute of Agronomy. Identification of the plant was confirmed by Montpellier University botanists and the Biology Institute of Algiers University of Sciences and Technology. Extractions were realized on a laboratory scale.

To extract the essential oil by steam distillation, the vegetable matter was placed in a glass column, of which the lower and higher parts were connected to a water flask and a condenser, respectively. The water vapour produced in the flask crosses the plant, charged with essential oil then to the condenser, where it is condensed. After condensation, the oil is separated from water by decantation. The extraction of essential oil by hydrodistillation was carried out under the same conditions as the steam distillation. The only difference is that in this case the vegetable matter is laid out in the flask containing water and the unit is carried to boiling. The vapour mixture of water–oil produced in the flask then passes to the condenser, where it is condensed. The oil is recovered after decantation.

The essential oils obtained by both extractions were analysed by gas chromatography (GC) and gas chromatography–mass spectrometry (GC–MS) under previously established operating conditions.^{2,3} A 25 m × 0.23 mm fused silica capillary column coated with polydimethylsiloxane (DBP-1) of 1.5 µm film thickness was the column fitted in the GC and GC–MS systems. The column temperature was computed to start at 60 °C, increasing to 200 °C at 3 °C/min. The carrier gas at 1 ml/min was nitrogen for GC and helium for GC–MS. In addition, the oil was recovered at regular time intervals and analysed by GC in order to follow yield and oil composition. The essential oil yield was estimated according to the dry vegetal matter by using the following equation:

$$R_{\text{HE}}(\%) = \frac{m_{\text{HE}}}{m_{\text{S}}} \times 100 \quad (1)$$

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where m_{HE} = essential oil mass (g), m_S = dry vegetal matter mass (g) and R_{HE} = essential oil yield (%).

Results and Discussion

Identification of the Components of the Essential Oil

The identification of the components of the essential oil extracted by both steam and hydrodistillations was carried out by comparison of their mass spectra and retention times to those of reference standards.^{4,5} The results are presented in Table 1.

From this table, it can be seen that these oils were characterized by the presence of monoterpene hydrocarbons, oxygenated monoterpenes and sesquiterpenes, but the quantitative differences were observed in the contents of these components. The steam distillation oil composition is comparable to that reported in the

Table 1. Chemical composition of rosemary essential oil extracted by steam distillation and hydrodistillation

Components	Relative content (%)	
	Steam distillation	Hydrodistillation
α -Pinene	5.2	0.4
Camphene	3.0	0.3
β -Pinene	5.7	0.3
Myrcene	1.7	tr
β -Phellandrene	tr	tr
<i>p</i> -Cymene	2.2	tr
1,8-Cineol	52.4	31.9
γ -Terpinene	0.5	tr
Sabinene hydrate	0.3	0.4
Terpinolene	0.2	tr
Linalol	1.1	3.9
Camphor	12.6	19.7
Borneol	3.4	12.1
Terpinene-4-ol	0.7	4.0
α -Terpineol	2.1	12.8
Bornyl acetate	1.1	3.1
Thymol	tr	—
Carvacrol	tr	—
Eugenol	tr	—
α -Copaene	0.2	0.6
β -Caryophyllene	4.2	3.0
α -Humulene	0.4	tr
Germacrene D	0.3	tr
α -Murolene	0.2	tr
α -Farnesene	0.1	tr
γ -Cadinene	0.4	tr
Calamenene	tr	tr
δ -Cadinene	0.3	tr
Calacorene	0.2	tr
α -Cadinene	tr	tr
Caryophyllene oxide I	0.1	tr
Caryophyllene oxide II	0.1	tr
Humuladienol	tr	—
Humulene oxide	tr	—
Palmitic acid	0.1	tr
Miristic acid	tr	tr

tr, trace < 0.05%.

literature,^{6–13} more specifically to those of the Tunisian and Greek oils^{14–15} and to commercial standards.¹⁶ This oil has high monoterpene hydrocarbons and ether contents, even though the water-distilled oil is characterized by high ketone, alcohol and ester contents. The contents of the sesquiterpenes were practically the same.

The monoterpene hydrocarbons compounds are in small proportions in the hydrodistilled oil, due to chemical conversions in the presence of water, resulting from hydrolysis reactions of these components in monoterpene alcohol's components. The quantitative differences in the rosemary oils composition may be due to extraction processes, as previously reported by Boelens.⁶

Yield and Essential Oil Composition as Function of Time

The yield of essential oil obtained by the steam distillation is 1.2%. This value is comparable to that given by Fournier *et al.*¹³ for Tunisian rosemary. The yield of the hydrodistillation is much lower at only 0.44%. The essential oil yield as a function of time is shown in Figure 1, and all oils are recovered after 30 min. In addition, the yield increases quickly at the beginning of the extraction, its evolution becoming slower thereafter. Moreover, after 10 min steam distillation more than 80% of essential oil are recovered; whereas for the hydrodistillation it takes at least 30 min to extract 88% of the oil.

Analysis by gas chromatography of the oil recovered with regular time intervals enabled us to follow the evolution by time of the relative content of some major components of oil belonging to various known chemical families: 1,8-cineol, camphor, borneol, α -terpineol, bornyl acetate, β -caryophyllene and δ -cadinene, as shown in Figures 2 and 3. Figure 2 shows a rapid evolution of the content of the considered components, and those recovered in the ascending order of their boiling points. Indeed, in the steam distillation, the 1,8-cineol is mostly recovered in the first 5 min of the process, followed by camphor, borneol and α -terpineol, which reach their maximum contents after 10 min extraction;

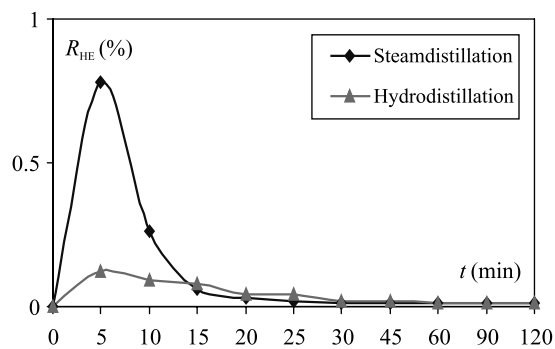


Figure 1. Evolution of the essential oil yield by time

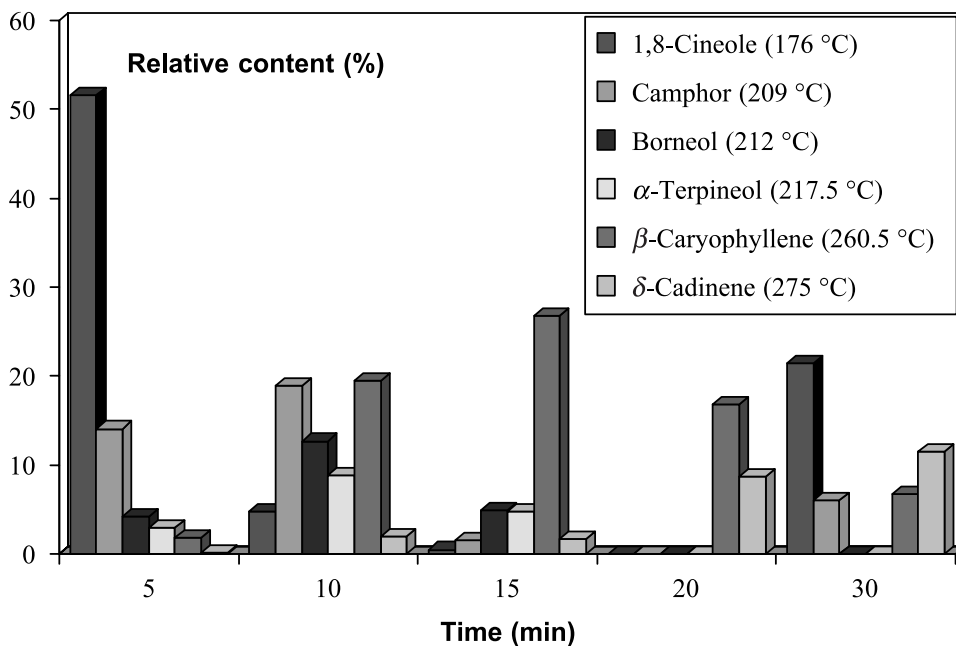


Figure 2. Evolution of the content of some components of rosemary essential oil during steam distillation

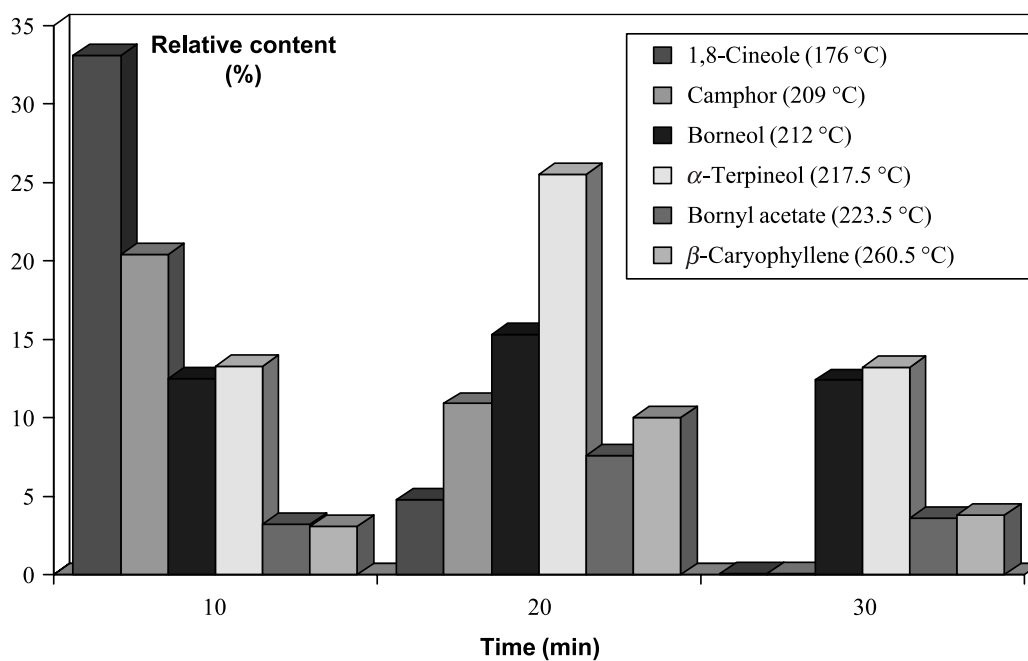


Figure 3. Evolution of the content of some components of rosemary essential oil during hydrodistillation

then the β -caryophyllene follows them with a maximum content after 15 min and finally the δ -cadinene with an optimal content after 20 min of steam distillation. The fact that the components considered are recovered in the ascending order of their boiling points allows us to suppose that the steam distillation depends on phenomena of transfer of the oil located at the surface of the plant and of its evaporation.

The 1,8-cineol and camphor are recovered in greater proportion after 10 min hydrodistillation, as shown in Figure 3. We observe thereafter the simultaneous extraction of borneol, α -terpineol, bornyl acetate and β -caryophyllene, which reach their optimal contents after 20 min of the process. After 30 min, only traces of these components are recovered. In addition, the considered components are not recovered in the order of their boiling

points. Indeed, 1,8-cineol is collected at the same time as camphor, whereas it is more volatile. The same happens for other components which have different boiling points. Similar observations were reported by Morin *et al.*¹⁷ for the hydrodistillation of lavender. These authors suggest that the order of exit of the components is dictated by their polarity and not by their volatility. It is thus probable that phenomena of diffusion and polarity due to the action of water intervene simultaneously during the hydrodistillation of essential oil.

Conclusion

This study made it possible to identify the components of the essential oils of rosemary obtained by two extraction processes. Steam distillation seems to be the more suitable process, since it gave better yield and an oil composition comparable to those reported in the literature and that recognized by the commercial standards. In addition, the results obtained show that the duration of extraction could be limited to 10 min, since more than 80% of oil is extracted at the end of this time.

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