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Physicochemical study essential oils of *Thymus fontanesii* according to its conservation

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ABSTRACT

Our work aims to study a very aromatic and medicinal plant known by the locals called Thymus fontanesii (Thyme). The extraction of essential oil of the plant was carried by a drive to steam. The yield is attractive even in terms of industrial exploitation. A number of physicochemical characteristics were determined on freshly extracted oils. The study of the degradation of these oils as a function of time showed a high instability when stored at room temperature and in the presence of light and rather good preservation of the low temperature and protected from light.

Keywords: *Thymus fontanesi*i, Essential Oils, physicochemical characteristics, chemical and physical indices.

INTRODUCTION

The present work aims to study the physicochemical properties of essential oil of the plant *Thymus fontanesii* of the family Labiaceae. The essential oil is used extensively reviewed by humans at least since antiquity for their medicinal, culinary and fragrant. Their use however has always been practiced empirically. We chose to study these two plants for the following reasons:

- It is a wild plant, very abundant in western Algeria;
- It is widely used in herbal medicine especially as antiseptic, antispasmodic and expectorant;
- they are used as a condiment;
- They are rich in essential oils that have antioxidant, antibacterial, antifungal, anti-inflammatory and insecticide [1,2].

MATERIALS AND METHODS

Extraction of essential oil

- Sampling: once the plant picked the month of March, the leaves were dried for 10-15 days in darkness and at room temperature.
- Method of production: distillation by steam distillation of water. For 2h 30mn.
- Organoleptic properties of essential oils extracted:
- Color: Reddish yellow
- Odor: Aromatic, pungent
- Taste: Very spicy
- Calculation of Performance: The essential oil yield is defined as the ratio between the mass of essential oil obtained from the mass of dry vegetation.

The physicochemical indices of essential oils [3]

1. Chemical characteristics

1.1. Acid indices (AI)

The acid expresses the number of milligrams of potassium hydroxide (KOH) required to neutralize the free acids contained in one gram of essential oil.

It weighs 2 grams of essential oil, and is introduced into a glass flask. 5 ml of 95% ethanol and 5 drops of phenolphthalein (PP) at 0.2%. Neutralized by adding a burette through the ethanol solution of KOH (0.1 mol / l) until a pink color. We denote the volume of the ethanolic solution of KOH added. The calculation of AI is given by the formula:

$$\mathbf{AI = 5.61\ xV / M}$$

5.61: Corresponds to 0.1 mol / L KOH
M: mass in grams of the essential oil
V: Volume in milliliters of ethanol solution of KOH (0.1 mol / l) used for titration.

1. 2. Ester indices (IE):

The ester value is the number of milligrams of KOH needed to neutralize the free acids by hydrolysis of esters contained in one gram of essential oil.

It weighs 2 grams of essential oil, and is introduced into a glass flask. Was added through a burette 25ml of ethanol solution of KOH (0.5 mol / l). It adapts the condenser and placed the ball on the heating mantle and allowed to heat for one hour. Allowed to cool then add 20ml of distilled water and 5 drops of 0.2% PP. Finally, as the excess of KOH solution with hydrochloric acid 0.5 mol / l. alongside the operation cited, it makes a blank under the same conditions and with the same reagents. The calculation of EI is given by the formula:

$$\mathbf{IE = (28.05\ x\ (V0-V1) / M)-IA}$$

28.05 g / l corresponding to 0.5 mol / L KOH.
M: mass in grams of the test.
V0: Volume in cl ml solution (0.5 mol / l) used for the blank.
V1: volume in ml of the solution cl (0.5 mol / l) used to determine the IE of the essential oil.

1.3. Peroxide indices (PI):

The peroxide is the number of micrograms of active peroxide content in one gram of products and oxidizing potassium iodide to release iodine under the conditions of the method described.

Weigh 1g of the oil in a microwave tube you put in an Erlenmeyer flask, add 10 ml of chloroform and shake. Add 15 ml of acetic acid CHCOOH, then 1 ml of saturated aqueous KI, stopper immediately, shake the bottle and leave for 5 min in the dark. 75 ml of distilled water. Titrate carefully in the presence of starch, iodine released with Na2S2O3 solution (0.01 N) until complete discoloration of the solution.

The calculation of PI is given by the formula: **IP = 8000 V / m.**

m: is the mass of the test.

V is the volume of N/100 thiosulphate solution.

1.4. Iodine indices (II)

Iodine is a measure of the unsaturated constituents of the essential oil. Is the mass of iodine that can be set in the double bonds.

Weigh 1g of the oil in a microwave tube you put in an Erlenmeyer flask, add 10 ml of chloroform and 25 ml of HANÜCH. Stopper and shake. Place the solution in the dark for 1 hour. Add 20 mL of saturated aqueous solution of KI to 10% fresh and 150 ml of distilled water, shake to remove the iodine content in the I2 CH2O. Titrate carefully with the solution Na2S2O3 (0.1 N) until the color changes to yellow brown initial. At this point, add 2 drops of starch and fresh color is blue violet. Continue to titrate with Na2S2O3 solution (0.1 N) until discoloration. For the witness, we will follow the same steps using 1g ED. The calculation of PI is given by the formula:

$$II = [AB / m] * f * 12.69$$

m: is the mass of the test.

A: is the volume of N/10 thiosulphate solution of the witness.

B: is the volume of N/10 thiosulphate solution of the sample.

f: factor Na2S2O3 f € [0.99-1.05]

RESULTS AND DISCUSSION

Levels of essential oils

The yield of essential oil of bay is 2%. This result is greater than that given by Dob by T. & al (2006) [4] (0.9%) and Fesneau M. (2005) [1] for *Thymus zygis* (0.4 to 0.7%). On the other hand, it is within the ranges given by Fesneau M. (2005) [1] for *Thymus vulgaris* (1.7 to 2.5%).

The quality of essential oils depends on many causes, including the process for obtaining the state of maturation and storage of the substance, its origin.

The conservation of essential oils requires well-stoppered bottles, keeping them away from air and light. It is indeed important, to prevent their oxidation, polymerization and gumming them that everyone was able to observe when these precautions were not observed [5].

Physicochemical measures are among the factors used in determining the quality of essential oils.

Quality control of essential oil of *Thymus fontanesii*
From the physical point of view
The results of the physico-chemical analysis obtained are summarized in the following table 1:

Table 1. The physicochemical indices of essential oil extracted from freshly.

Property	**values**
	Essential oil of Thyme
density	0.9219
Refractive index at 20 °	1.4999
Rotatory power	+3.4313 (Dextrogyre)
Miscibility with ethanol	1ml/0.6ml up to 20ml of ethanol
Freezing point <-20 °	< -20°
Acid Indice	1.458
Ester Indice	16.83
Iode Indice	502.524
Peroxyde Indice	8000

The value of the density of our essential oil is 0.9219. Referring to the following table 2, we note that our oils complex.

	D < 0.9	**0.9 < D < 1**	**D > 1**
Essential oils	rich in terpenes	Have a complex composition	Products still contain the aromatic series, sulfides, nitrites

-The density value of our oil is in the range given by Garnero (1991) [|6] and Fesneau M. (2005) [1] for the essential oil of *Thymus vulgaris* (0.9 to 0.955). It is slightly larger than the values obtained with those of essential oils of *Thymus serpylloides* and *Thymus longiflorus* (0.9) [7].
- The index of refraction is 1.499, this value is within the range given by Fesneau M. (2005) [1]. (1.491 to 1.51) for the essential oil of *Thymus vulgaris* and is close to value given for the essential oil of *Thymus serpylloides* (1.496) [7].

- The polarization of our essential oil is +3 °, 4313 '. It allowed us to infer that our essential oil is dextrorotatory.
- The freezing point is below - 20 ° C.
- Our essential oil is miscible with 0.6 volumes of ethanol at 95 °, this result is significantly lower than those obtained in previous work.
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From the chemical point of view

- The value of the acid of the essential oil of Thyme is of 1.458. This value is much lower than those given by Naves (1974) [8] for *Thymus vulgaris* (8.4)
- The ester value of our essential oil of 16.83. This value is close to that of *Thymus vulgaris* (18.2) naves 1974, and is far below that of *Thymus hymalis* (59.2) [8].
- The peroxide of our essential oil is 8000.
- The iodine value of our essential oil is 502,52.

Evolution of chemical characteristics over time

In this section, we followed the chemical quality of essential oil for 150 days. To this end, we kept both samples in glass tubes wrapped at two different temperatures, 4 ° C and at room temperature, and a sample in a glass tube unwrapped for 150 days at room temperature. The indices were made each 15 days for three samples. The results are illustrated in Figures 1, 2,3 and 4.

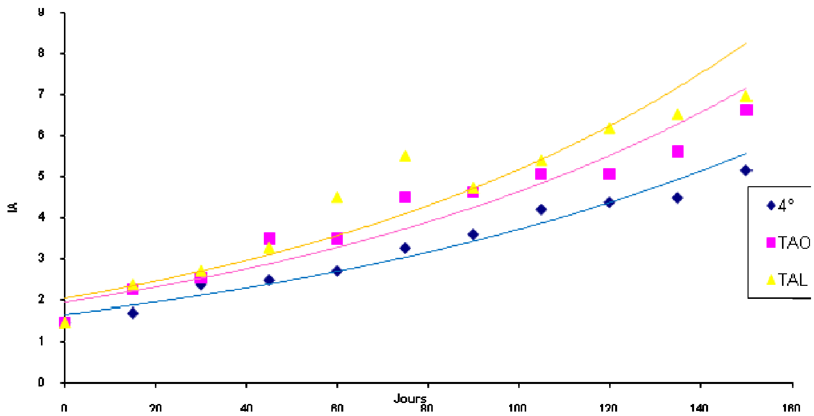

Figure 1: Curve of variation of the AI of the essential oil of *Thymus fontanesii*

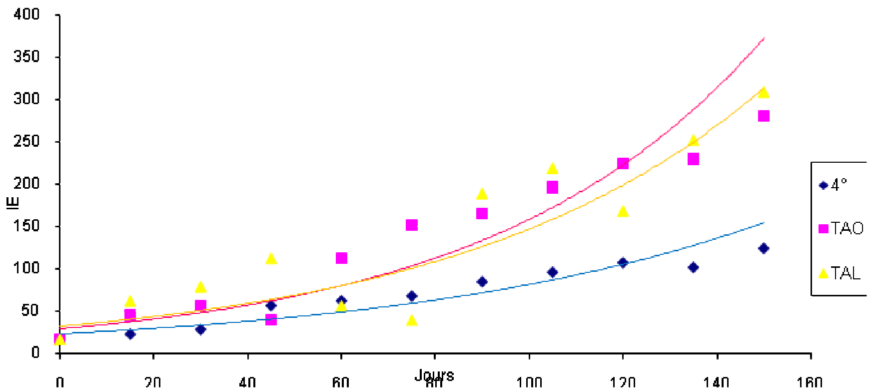

Figure 2: Curve of variation of the EI of the essential oil of *Thymus fontanesii*

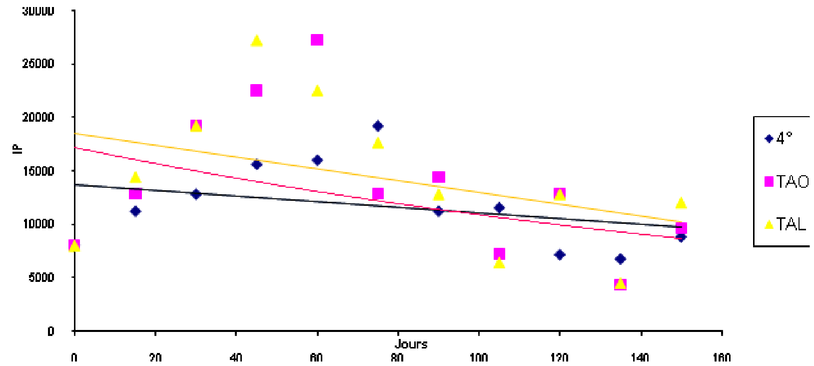

Figure 3: Curve of variation of PI of the essential oil of *Thymus fontanesii*

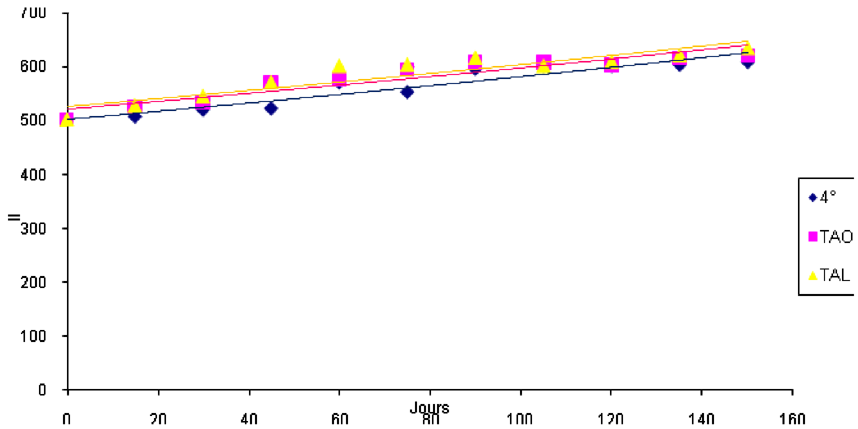

Figure 4: Curve of the variation II of the essential oil of Thymus fontanesii

4 °C: ET stored at 4 ° C
TAO: essential oil kept at room temperature in the shade
TAL: Essential oil stored at room temperature and exposed to light

- The acid oils stored at room temperature increases throughout the duration of the conservation of 1458 for oil freshly extracted until the value of 6.62 at the 150th day of oil stored at the Protect from light and 6.95 for the oil exposed to light. While the acid oil stored at 4 ° C undergoes an increase from 1,458 to 5.161 during the 150 days of storage.

- The Ester oil is extracted from freshly 16.83. This value has increased to a maximum of 280.5 for the oil stored at room temperature and away from light and 308.95 for the oil stored at room temperature and exposed to light . While the oil stored at 4 ° C, this index increased only slightly compared with other samples from 16.83 until reaching a level of 123.42 at 150th day.

Also in this case the increase is not due for two samples stored at room temperature. EI has declined 45th day for oil kept in the dark, and the 60th, 75th and 120th day for the woman who was exposed to light. This reduction always results in a remarkable increase in the acid.

- The peroxide of 3 samples, no increase in a remarkable way to the end of storage. It goes from 8000 to the freshly extracted oil to a peak of 27,200 in the 45 th and 60th day for oil kept in the dark and exposed to the light, respectively, and a maximum of 19200 at the 75th day at 4 ° C. Then it starts to decrease and increase to reach the 150th day following values: 8800 for the oil stored at 4 ° C, 9600 for oil kept in the dark and 12000 for the exposure to light.

- The iodine value increases over time from 502,524 to the freshly extracted oil to a value of 601.5 at the 60th day for the oil exposed to light, and a value of 596.43 on 75th day and 90th day to the oil stored at room temperature away from light and those stored at 4 ° C, respectively. These values start to stabilize and converge to values very close to the 150th day.

CONCLUSION

The quality control of our essential oils with the physicochemical characteristics helped to highlight the quality of these oils have a complex composition, high acidity and physicochemical indices comparable to those obtained in the literature.

Monitoring of chemical characteristics (AI, EI, PI, II) essential oils for 150 days under appropriate storage conditions (4 ° C in the shade, ambient temperature in the shade and in the presence of light) showed a slight variation in these indices when the oil is kept away from light and low temperature (4 ° C). These same oils are easily degraded when stored at room temperature and to a lesser degree for those kept in the dark. Result confirmed by several authors in particular Pibiri MC (2006) [9]: "Essential oils are stable at low temperatures if stored properly: to protect from oxidation and polymerization caused by air and by the light."

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