



Organoleptic and Physicochemical Properties of Algerian Lemon Essential Oil

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Abstract: The objective of this work is to study the organoleptic and physicochemical properties of the essential oil of Limon (*Citrus limon*) grown in Collo, Skikda city (Algeria). The evaluation of the yield of essential oil extracted by cold expression is: 1.0%. The qualitative and quantitative analysis by (GC / MS) of the essential oil allowed to identify 53 compounds which represent: 99.938%, the main ones being: Limonene (61.647%), β -Pinene (13.852%), γ -Terpinene (9.959%) followed by other low-molecules: α -Pinene (2.279%), Myrcene (1.888%), α -Citral (1.702%), β -Citral (1.046%), β -Bisabolene (1.026%) totaling approximately: 93.399%. The density is: 0.855 ± 0.005 . The measurement of the refractive index calculated and brought to 20°C is of low light refraction: 1.4700 ± 0.005 . The boiling and evaporation index are values: (180-188°C).

Keywords: Lemon, *Citrus Limon*, Essential Oil, Chemical Composition, Density, Refractive Index, Boiling and Evaporation Index

1. Introduction

Essential oils possess many biological activities. They have organoleptic properties: (appearance, color, odor, flavor) and physicochemical properties: (chromatographic profile, density, refractive index and melt index). The methods used to determine the physicochemical indices are those indicated by the French Standards Association (AFNOR) Code of Standards. The purpose of the present work was to determinate the organoleptic and physicochemical properties of Algerian Lemon essential oil. And relate them with their chemical composition, for further application in food and pharmaceutical industries as natural valuable products.

2. Materials and Methods

2.1. Selection of Plant Material

Our samples come from a private farm, located in the region of Collo, Skikda city. The harvest was carried out

during the period from November 2012 to March 2013. The plant material thus harvested undergoes certain treatments given the various locations of the secreting sites of the essential oil in the plant.

The upper part of the pericarp or zest (the exocarp) with a test sample of 400 g is harvested on fresh fruit, it is presented in thin ribbon is narrow, rough appearance, green or yellow outside, white and spongy inside (mesocarp). This choice is justified by: the richness of the zest in essential oil compared to the other parts of the fruit [1].

2.2. Extraction of the Essential Oil (Cold Expression)

It is the simplest of the processes, but it applies only to citrus fruit, the fruit-peel of which contains pockets secreting essences [2]. Cold expression is reserved for the extraction of volatile compounds in pericarps. It is a mechanical treatment which consists in tearing the pericarps rich in secretory cells [3]. The product thus obtained is called gasoline because it has not undergone any chemical modification [2].

2.3. Measurement of Relative Density at 20°C (Standard NF T 75 - 111) [4]

The relative density of the essential oil is defined as the ratio of the mass of a certain volume of oil at 20°C. and the equal mass of volume of distilled water at 20°C. This size is dimensionless and its symbol is d_{20}^{20} . The density is measured using a pycnometer of volume: 5 ml at the temperature of 20°C.

2.4. Measurement of the Refractive Index (Standard NF T 75 - 112) [5]

The measurements were carried out using a Prisma-CETI convex refractometer. When the determination is carried out at a temperature other than 20°C, the correction is carried out at 20°C. using the formula:

$$I_{20} = I_t + 0,00045 (T - 20^{\circ}\text{C})$$

I_{20} = Index at 20°C.

I_t = Temperature or ambient temperature.

T = Ambient temperature.

2.5. Melting Point Determination (AOCS, 1997. in Soares et al., 2009) [6]

The principle is based on the heating of a capillary tube containing a test portion of the essential oil in a heating and the notation of the melting temperature.

2.6. Chromatographic Analysis

The GC-MS analysis was performed using a Hewlett Packard 5973-6800 system operating in EI mode (70 eV) equipped with a split/splitless injector (250°C), a split ratio 1/50, using a fused silica HP-5 MS capillary column (30 m × 0.25 mm (i.d.), film thickness: 0.25 μm . The temperature

program for the HP-5 MS column was from 60°C to 280°C at a rate of 2°C/min. Helium was used as a carrier gas at a flow rate of 0.5 ml/min. Injection volume of the sample was 0.2 μl . The identification of the components was conducted in an IS system managing a library of spectrum wiley7n.l. The GC-MS analysis was performed at the Scientific and Technological Scientific Research Center on Physico Chemical Analysis (CRAPC), Bab ezzouar (Algiers, Algeria).

3. Results and Discussion

3.1. Rendement

The results obtained indicate that the extraction yield of the essential oil by cold expression is 1.0%. From the results quoted in the literature, it is quite clear that citrus fruits contain little essential oil. The results obtained in this work are almost similar to the other results cited. Indeed, Jeannot et al., 2005 [7] and Fuselli et al., 2008 [8] observed yields ranging from 0.7 to 0.9% for the essential oil of Citrus silt. However, Rega et al., (2003) [9] reported that yields of essential oil in Citrus differed by species and unexpectedly reported yields of 1-3%. This difference could be explained according to Kelen and Tepe, 2008 [10] by choosing the harvest period because it is essential in terms of yield and quality of the essential oil. The climate, the geographical area, the genetics of the plant, the organ of the plant used, the degree of freshness, the drying period, the method of extraction used, etc. These factors can have a direct impact on yields of essential oil Vekiari et al., 2002 [11].

3.2. Organoleptic Properties

The following table (Table 1) shows the characteristics of the essential oil obtained (color, odor, flavor, solubility) in the species studied.

Table 1. Organoleptic characteristics of Lemon essential oil.

Plant	Color	Odour	Flavor	Solubility
Lemon	Transparent liquid, Pale yellow or greenish yellow	Aromatic, typical of citral	Aromatic and bitter	Fat-soluble, miscible with other solvents

3.3. Physico-chemical Properties

3.3.1. Chemical Composition

The qualitative and quantitative analysis by (GC / SM) of essence identified 53 compounds which represent a total of 99,986%. The essence of Citrus silt consists mainly of compounds of which: γ -terpinene (9,959%) are major compounds, followed by other low-molecular-weight molecules such as α -pinene (2.279%), Myrcene (1.888%), α -Citral (1.702%), β -Citral (1.046%) β -Bisabolene (1.026%), totaling about 93.399% (Table 2 and Figure 1).

Our results for the chemical profile of the essential oil of Citrus limon agree with those of;

Vekiari et al., 2002 [11], which showed that the Lemon species are characterized by a high concentration of β -Pinene (21.2%), γ -terpinene (17.4%), α -pinene (9.8%).

Vasudeva and Sharma, 2012 [12] GC-MS profile depicted that a total of 14 components were present. The monoterpene hydrocarbons constituted the most dominant chemical group and among the monoterpene hydrocarbons dl-limonene (89.098%) is the largely predominant and specific component followed by β -myrcene (2.933%). dl-limonene contributes to the aromatic odour of the oil and hence the plants belong to dl-limonene chemotype. Other monoterpene hydrocarbons were α -pinene (0.865%), sabinene (0.161%), Δ -3-carene (0.544%). Oxygenated monoterpenes were identified as second main group components, among these major ones were (\pm)-linalool (2.927%), α -terpineol (0.597%), (E)-citral (0.749%) and 4-terpineol (0.169%), Citronellol (0.304%) and cis-citral (0.304%). No sesquiterpene hydrocarbon and oxygenated sesquiterpene were reported. The other oxygenated components were identified as aldehydes components which includes octanal (0.746%), decanal

(0.335%) and alcohol component 1-octanol (0.211%).

Kamaliroosta et al., 2016 [13], eighteen compounds were identified in the essential oil of sweet Lemon peel where limonene (49.79%), xanthotoxin (12.62%), 6,6-dideuteronon-1-ol-3 (10.20%) and 4-vinyl- 2-methoxy-phenol (5.20%) were the major components.

Studies carried out in Algeria, on the other hand, have produced almost identical results; Hellal, 2011[14]: Limonene (51.39%), β -Pinene (17.04%), γ -terpinene (13.46%), α -pinene (3.07%).

Several investigations by Moufida and Merzouk, 2003 [15], Belleti et al., 2004 [16], Rehman et al., 2004 [17], have shown that typically Lemon essences consist mainly of monoterpene compounds (97%). Whereas other compounds such as alcohols, aldehydes, esters are only represented with low contents ranging from (1.8% to 2.2%).

By studying the chemical composition of Lemon essences, Nogata et al., 2006 [18] concluded that in addition to monoterpene, these species contain fairly low fatty acids (0.8%). Linolenic acid is the main fatty acid of *Citrus sinensis*; Linolic acid is found in *Citrus limon* and oleic acid in *Citrus aurantium*. Flavonoids are found in citrus essences and make up the non-volatile part of oils and are useful in differentiating citrus species.

According to Senatore et al., 2002 [19], variations in the chemical composition of Lemon gasoline, both qualitatively and quantitatively, may be influenced by one or a combination of factors: genetic make-up, age, the environment of the plant and the presence of chemotypes.

(Wolford et al., 1971, Shaw, 1979, Boelens, 1991, Smith et al., 2001) [20, 21, 22, 23] distinguished that essential oils of *Citrus silt Limon* could be chemotypes: β -pinene, limonene, linalool, acetate Linalyl, citral, and citronellal; *Citrus aurantium* of chemotypes: limonene, linalool, decanal, and *Citrus sinensis* of chemotypes: D-limonene, geraniol, linalol, citral, citronellal, terpineol and decanal. It should also be noted that according to Minh et al., 2002 [24], the maturity of the fruit has a significant impact on the chemical composition of the essential oil. Thus, terpenes are exclusively present in the essential oil of the unripe fruit, as the fruit ripens the concentrations of aldehydes, oxygenated terpenes and aliphatic sesquiterpenes increase. And finally, Kim et al., 2014 [25] they found the chemical constituents of the *Citrus* essential oil, analyzed by gas chromatography-mass spectrometry (GC-MS), included γ -terpinene (24.7%), 2-beta-pinene (16.6%), 1-methyl-2-isopropylbenzene (11.5%), L-limonene (5.7%), beta3-ocimene (5.6%), and alpha-pinene (4.7%).

Table 2. Chemical composition of Lemon essential oil (Principal constituents).

N°	Compound	Retention time	%
01	β -Pipene	13.091	13.852
02	Myrcene	13.856	1.888
03	Limonene	17.422	61.647
04	γ -Terpinen	18.923	9.959
05	β -Citral	31.511	1.046
06	α -Citral	33.645	1.702
07	β -Bisabolene	48.606	1.026

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Sample Name: Citrus limon
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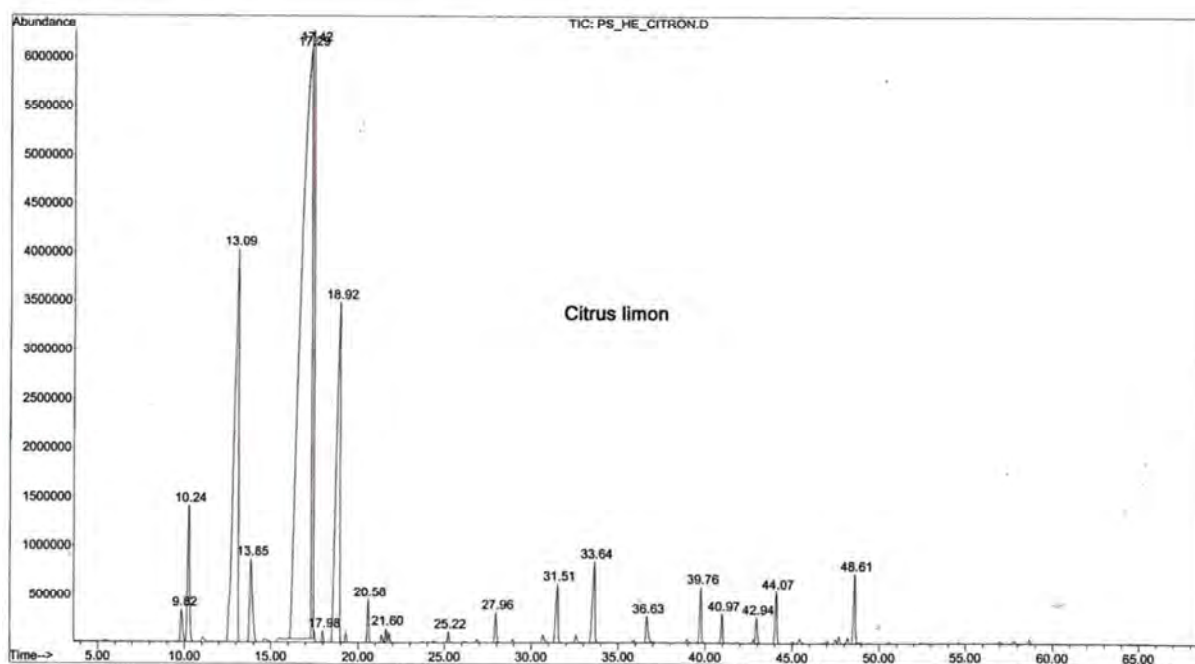


Figure 1. Chromatographic profile of Lemon essential oil analyzed by GC / MS.

3.3.2. Density

The density of an essential oil is a very important criterion for evaluating the quality of an essential oil in different areas of life (cosmetics, pharmacy, food industry, chemical, etc.). It can easily give insight into the naturalness of the product as well as attempts at fraud and alteration.

Table 3. Density value of Lemon essential oil (mean \pm standard deviation).

Plant	Density (found)	Density (AFNOR NF T. 75-202)
Lemon	0,855 \pm 0,005	0,850 à 0,870

From the result (Table 3) of the obtained density, it can be said that the oil conforms to international standards. (According to the French Association of Standardization).

On the other hand, according to Ferhat et al., 2007 [26] the density of the essential oil of the Lemon obtained by cold expression is: 0,856.

3.3.3. Refractive Index

The refractive indices were calculated and brought to 20°C. using an Abbot refractometer and are shown in the table below (Table 4).

Table 4. Value of the refractive index at 20°C. (mean \pm standard deviation) of Lemon essential oil.

Plant	Refractive index (found)	Refractive index (AFNOR)
Lemon	1,4700 \pm 0,005	1,474 à 1,475

The refractive index is the ratio between the celerity of light in the vacuum and the celerity of light in the medium under consideration. This report indicates the ability of the essential oil to reflect light. The refractive index of the sample corresponds to the AFNOR standards. It indicates its low refraction to light.

According to Ferhat et al., 2007 [26] the refractive index of the Lemon essential oil obtained by cold expression is: +1.475.

3.3.4. Melt Index

The melt index is usually found directly on the majority essential oil compounds after separation and not on the oil itself. Therefore, a separation of the major components of the oil is indispensable and indicated to determine this index (Table 5).

Table 5. Value of the melting index of Lemon essential oil.

Plant	Boiling temperature (found)	Evaporation T° (found)	Melting Index (literature)
Lemon	180	188	Limonene 175.4

4. Conclusion

Lemon (*Citrus Limon*) has received special attention over the past few years for its different properties, it is a source of essential oil or essence highly appreciated. Although vegetable biomass is a very promising source for the future, very little work has been done on the study of the

organoleptic and physicochemical properties of aromatic fractions of Lemon grown in Algeria. Because of its chromatographic profile, the essential oil extracted by cold expression of this plant possesses organoleptic and physicochemical properties very appreciated in perfumery and will be very coveted in the sector of the food, pharmaceutical and cosmetic industry. And in order to determine whether the essential oil of Lemon complies with the standards of the European Pharmacopoeia, a series of tests has been carried out following as far as possible the conditions described according to the French Standards Association (AFNOR) Code of Standards.

Competing Interests

The authors declare there that they have no competing interests.

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