

Fourier Transformed Infrared Spectroscopy Analysis of Constituents of Lemon Essential Oils from Algeria

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To cite this article:

Hicham Boughendjioua, Samah Djeddi. Fourier Transformed Infrared Spectroscopy Analysis of Constituents of Lemon Essential Oils from Algeria. *American Journal of Optics and Photonics*. Vol. 5, No. 3, 2017, pp. 30-35. doi: 10.11648/j.ajop.20170503.12

Received: June 9, 2017; **Accepted:** June 26, 2017; **Published:** October 30, 2017

Abstract: Three main tools are used to determine the structures of organic molecules. These tools are infrared (IR) spectroscopy, mass spectrometry (MS) and nuclear magnetic resonance (NMR) spectroscopy. Organic molecules absorb light (infrared, ultraviolet, etc.) at particular wavelengths based on different vibrational modes unique to the specific functional groups and structural features. In the present study, the volatile compounds of Lemon (*Citrus limon* L.) collected from Collo, Skikda city (Algeria), were detected and identified by Fourier Transformed Infrared Spectroscopy (FTIR) analysis (qualitative identification). FTIR allowed us to identify 10 volatile compounds and indicated that the functional groups of the essential oils are C-H (Alkene), C-H (aromatic) and C=C. The obtained results have shown that the essential oils can be fully utilized for pharmacy, cosmetology and industry.

Keywords: Lemon, *Citrus Limon* L., Essential Oil, Fourier Transformed Infrared Spectroscopy (FTIR), Functional Groups

1. Introduction

Citrus plants are well-known crops all over the world with potential socio-economic influence. They are well-known for their flavor, nutritional value and medicinal features. The medicinal activities for this genus are attributed to the presence of many medicinally active secondary metabolites such as essential oils [1].

Citrus essential oils have been applied in many products, such as cosmetics, medical formulation, beverages and foods, as flavoring agents as well as for aromatherapy. They are also used for their germicidal, antioxidant and anticarcinogenic properties [2].

Citrus essential oils contain 85-99% volatile and 1-15% non-volatile components [3]. The volatile constituents are a mixture of monoterpenes hydrocarbons, sesquiterpene hydrocarbons and their oxygenated compounds including [4]: aldehydes (citral), ketones, acids, alcohols (linalool) and esters [5, 6]. The main volatile compounds presenting in the citrus essential oils are pinene (there are two isomers: alpha-pinene and beta-pinene), limonene, citral, myrcene, linalool,

α-terpineol (there are five isomers: alpha-terpineol, beta-terpineol, gamma-terpineol, delta-terpineol and 4-terpineol) present in different percentages.

Terpineol is usually a mixture of these isomers having alpha-terpineol as major constituent. The chemical structures of the compounds listed above are shown in Figure 1. The major chemical component of the citrus oils is limonene and it varies between 68-98% in sweet orange, 45-76% in lemon and 32-45% in bergamot [3, 7].

Most people either use essential oils for their therapeutic effect or for the fragrance alone but it is also interesting to take note of the chemistry, of which the oils are made up from.

Essential oils, like all organic compounds, are made up of hydrocarbon molecules and can further be classified as terpenes, alcohols, esters, aldehydes, ketones and phenols etc.

Every single oil normally has more than a hundred components, but this figure can also run into thousands, depending on the oil in question.

(a). The various organic components found when analyzing essential oils with a chromatograph are:

- (b). Terpene hydrocarbons: Monoterpene hydrocarbons, Sesquiterpenes.
- (c). Oxygenated compounds: Phenols, Alcohols.
- (d). Monoterpene alcohols.
- (e). Sesquiterpene alcohols: Aldehydes, Ketones, Esters, Lactones, Coumarins, Ethers, Oxides [8].

Fourier Transformed Infrared Spectroscopy (FTIR) is based on the absorption of infrared radiation by the analysed

material. Through the detection of the characteristic vibrations of the chemical bonds, it makes it possible to carry out the analysis of the chemical functions present in the material [9].

The aim of this work was to clearly describe the functional groups of Lemon essential oils in order to better define the field of investigation of this natural product.

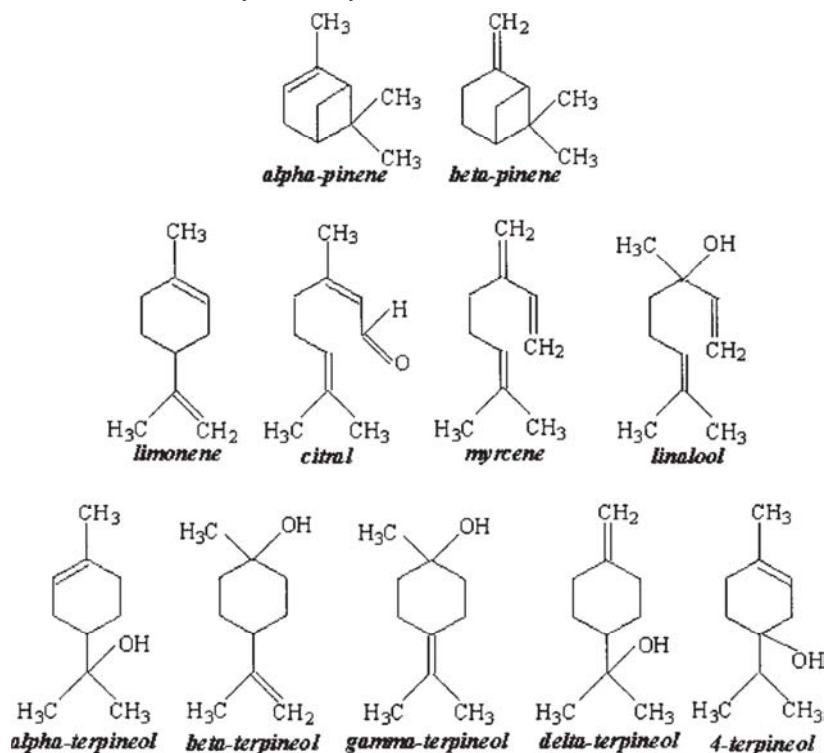


Figure 1. The Chemical Structures of the Main Volatile Compounds Present in the Citrus Essentials Oil.

2. Material and Methods

2.1. Plant Material

400 g of the tested sample was obtained from a private farm located in the region in Collo (Skikda city, North-East of Algeria). Plant harvesting was carried in March 2013. The upper part of the pericarp was harvested from fresh fruit, this choice was justified by the richness of zest in essential oils compared to other parts of the fruit [10].

2.2. Isolation of the Essential Oil

Obtaining essential oils was carried out by cold expression (physical process), it's the simplest processes applied only to citrus fruit [11], this extraction does not change the composition of the oil [12]. The product obtained is called gasoline, because it does not undergo any chemical modification [11, 13]. The obtained essential oil was stocked at 4°C until further analyses.

2.3. FTIR Analysis

Spectroscopy is based on the study of the interactions between matter and electromagnetic radiation. This radiation

consists of a particle beam having an undulating motion. All electromagnetic radiation forms the electromagnetic spectrum. In the spectrum, four regions are discernible: X-rays, ultraviolet (UV), visible and infra-red (IR). The emphasis will be on infrared (IR) spectroscopy because spectrum analysis allows us to follow a reaction process, determine the dosage of a compound, check the purity of a product and identify an unknown. Moreover, it is an inexpensive and easy to use process which makes IR spectroscopy the most widely used spectral method used by chemists. The principle is based on molecular vibrations. The energy emitted as photons can be absorbed by the material causing vibration in the molecules. This vibration changes the angle and the distance between the atoms.

When the molecule returns to its original form, energy will be released as heat. The absorption and release of energy by the molecule will be recorded by the apparatus and translated into a band spectrum. The analysis of this spectrum makes it possible to obtain the necessary information on the material analyzed.

Qualitative information: The wavelengths to which the sample absorbs are characteristic of the chemical groups present in the analyzed material.

Quantitative Information: The intensity of absorption at

the characteristic wavelength is related to the concentration of the chemical group responsible for absorption.

This method of analysis is simple to implement and not destructive. It allows the analysis of both organic and inorganic materials [9].

FTIR is performed with a PERKIN ELMER (universal ATR Sampling Accessory) apparatus, the operating conditions are as follows: technique: ATR, analysis range: 4000- 600 cm^{-1} . The results are directly compared with those of the internal bibliography of the apparatus; 01. Euclidean, 02. PSU / peak, 03. MIX PSU, 04. Peak Match, 05. PEAK / psu, 06. MIX PEAK. In the present work the Euclidean library has been used. The FTIR analysis was

performed at the Regional Police Scientific Laboratory (Constantine, Algeria).

3. Results and Discussion

Determination of the Functional Groups Present Using FTIR

Fourier transformed infrared spectroscopy is one of the most widely employed techniques for functional groups identification. Figures 2 to 3 and Table 1 showed the infrared spectra and the characteristic bands observed in Lemon essential oil in the range of 4000-600 cm^{-1} :

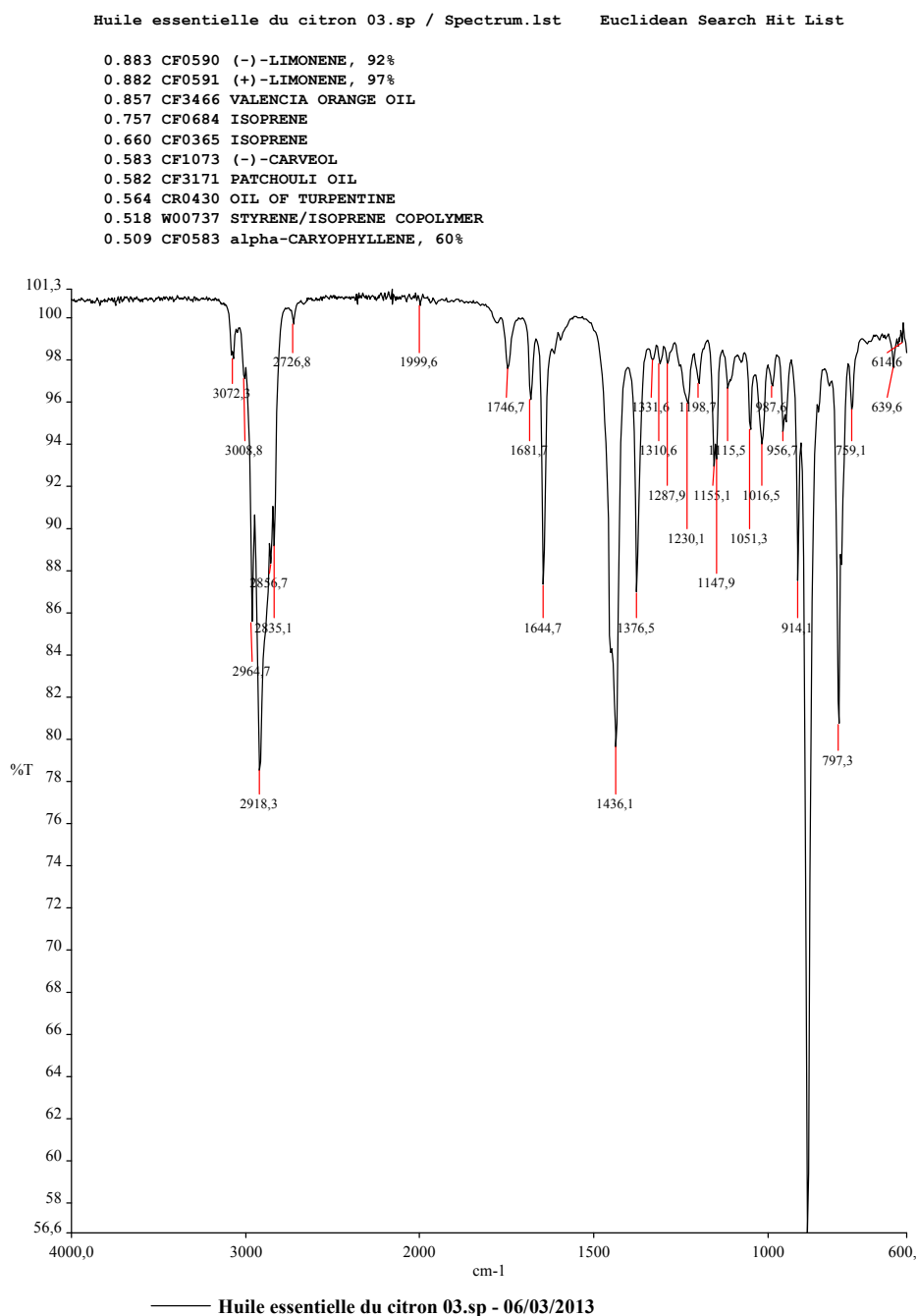


Figure 2. FTIR of Lemon Essential Oil.

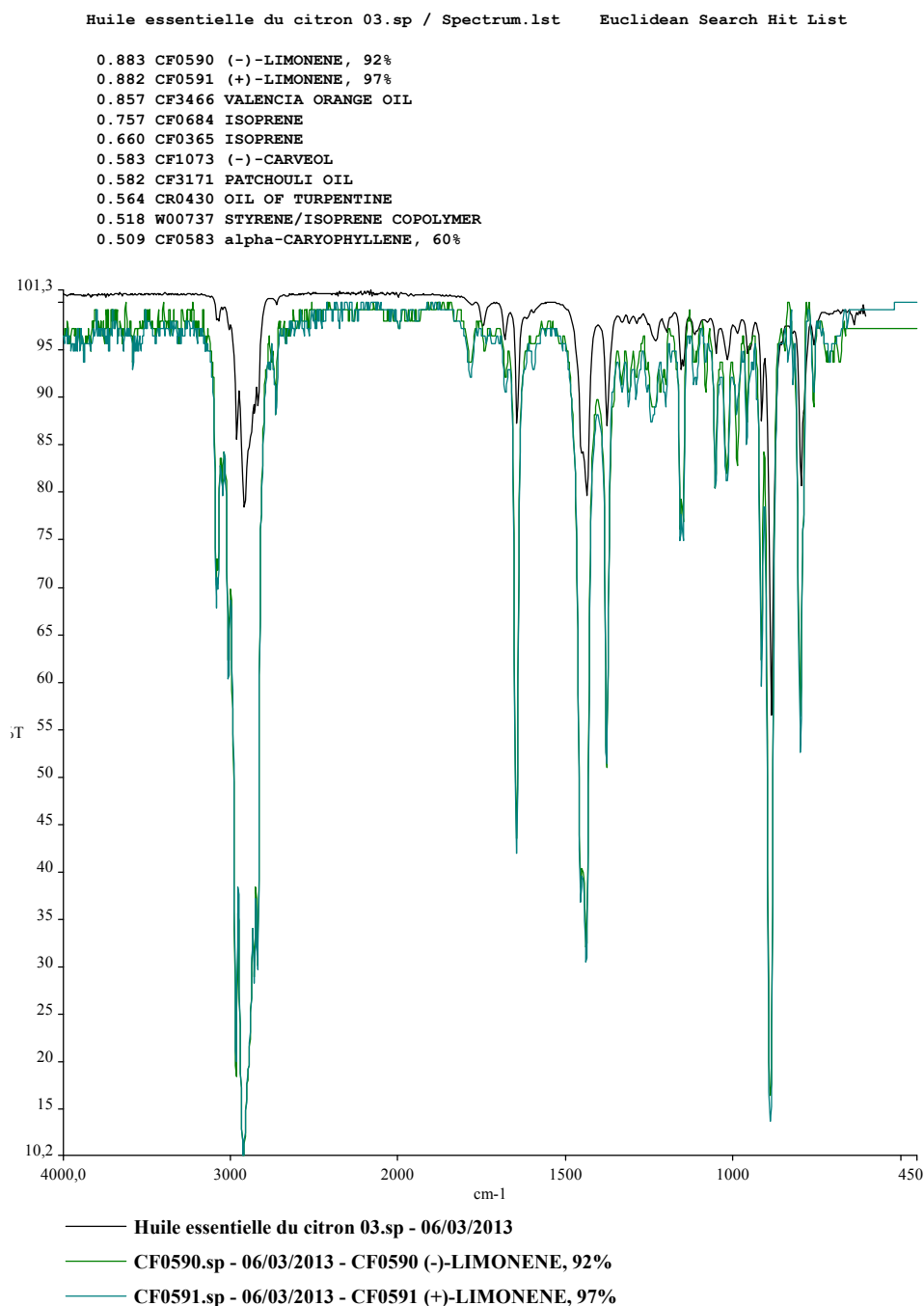


Figure 3. FTIR of Lemon Essential Oil Compared to the Bibliography.

Table 1. Links Present in the Molecule of Limonene.

Present links in Limonene	Theoretical frequency of the band (cm ⁻¹)	Presence of the band in the spectrum
C-H (Alkene)	3100-3000	Yes
C-H (aromatic)	3150-3050	Yes
C=C	1600-1680	Yes

In an earlier work developed by Boughendjioua and Djeddi (2014) [14], the chemical composition of the essential oil of *Citrus limon* detected by GC-MS allowed us to identify 53 compounds and indicated that the main compounds constituting the volatile oil were mainly Limonene (61.64%), β -pinene (13.85%) and γ -terpinene (9.95%).

The spectrum of Limonene as well as that of pure essential

oils had several similarities (Figure 1 and 2) Unfortunately, no "fingerprint" of the extracted oil coincided with that of pure essential oils or Limonene.

Among the ten compounds revealed by FTIR: (1). (-) - limonene, 92%, (2). (+) - limonene, 97%, (3). Valencia orange oil, (4). Isoprene, (5). Isoprene, (6). (-) - carveol, (7). Patchouli oil, (8). Oil of turpentine, (9). Styrene / isoprene

copolymer, (10). Alpha-caryophyllene, 60%.

On the other hand, the complete and detailed study of a spectrum is an operation rarely practiced in current interpretation because of the complexity of the analysis. It is therefore often limited to the identification of functional groups through the location of the different bands on the spectrum.

The spectrum presents characteristic bands at 1600-1680 cm^{-1} corresponding to $\text{C}=\text{C}$, the signals which appeared between 3100-3000 cm^{-1} and 3150-3050 cm^{-1} are caused by the asymmetrical and symmetrical stretching vibrations of $\text{C}-\text{H}$ groups (Figure 4 and 5).

It should be noted that Alkane $\text{C}-\text{H}$ bonds are fairly

ubiquitous and therefore usually less useful in determining structure. For $\text{C}-\text{H}$ Stretch with characteristic absorption (cm^{-1}); 3100 - 3010 (m), absorption peaks above 3000 cm^{-1} are frequently diagnostic of unsaturation [15].

According to Elzey et al., (2016) [16] the FTIR spectrum of the pure essential oil of Lemon, showing the expected characteristic $\text{C}-\text{H}$ stretch ($\sim 2900 \text{ cm}^{-1}$), $\text{C}=\text{O}$ stretch ($\sim 1700 \text{ cm}^{-1}$), broad $\text{O}-\text{H}$ stretch ($\sim 3400 \text{ cm}^{-1}$), and $\text{C}-\text{O}$ stretch ($\sim 1100 \text{ cm}^{-1}$) of terpenoid components. The compositions and constituents of essential oils may vary and highly depend on the geochemistry of the soil where it is cultivated. In general, essential oils are made up of terpenes such as terpineol, cineole, citronellal, and others.

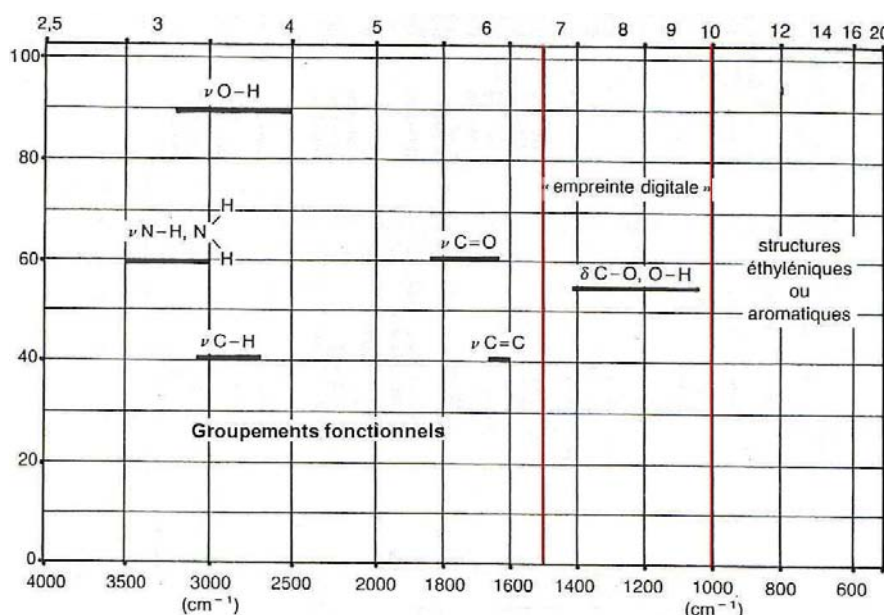


Figure 4. Graphs of IR Spectroscopy.

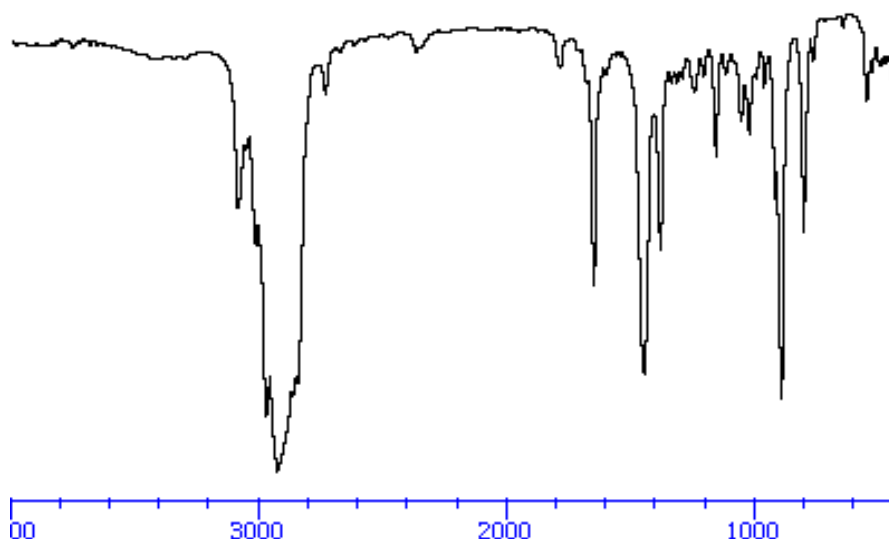


Figure 5. IR Diagram of d Limonene.

4. Conclusion

IR Spectroscopy is an extremely effective method for determining the presence or absence of a wide variety of

functional groups in a molecule; IR spectra can be used to identify molecules by recording the spectrum for an unknown and comparing this to a library or data base of spectra of known compounds. Computerized spectra data bases and

digitized spectra are used routinely in this way in research, medicine, criminology, and a number of other fields. The ingredients obtained from this study indicate that the essential oil of Lemon (*Citrus limon* L.) can be fully utilized for the manufacture of perfumery products, antimicrobial and antiseptic products.

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