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Analysis of essential oil components of *Thymus ciliates* ssp. *coloratus* from Annaba, Algeria by FTIR

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Abstract

The 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. Infrared Spectroscopy (IR), Mass Spectrometry (MS) and Nuclear Magnetic Resonance Spectroscopy (NMR).The aim of this work was to determine the chemical composition of the essential oils of the aerial parts of *Thymus ciliates* ssp. *coloratus* collected from Annaba city (Algeria). The essential oils were detected and identified by Fourier Transformed Infrared Spectroscopy (FTIR) analysis. FTIR allowed us to identify 28 volatile compounds and indicated than the functional groups and the presence vibrational modes of the essential oils are CHx deformation (1500-1400 cm-1), C-H (Aromatic) (3150-3050 cm-1) and Alcohol/Phenol O-H Stretch (3550 - 3200 cm-1). The obtained results have shown that the essential oils can be fully utilized forpharmacy, cosmetology and industry.

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Introduction

The Algerian flora is characterized by its floral diversity: Mediterranean, Saharan and a tropical Palaeo flora, estimated at more than 3000 species belonging to several botanical families.

These species are mostly spontaneous with a significant number (15%) of endemic species. This has given the traditional pharmacopoeia an inestimable richness (Quézel and Santa, 1962).

Thyme is an endemic species of Algeria and the Iberian Peninsula. It's a perennial plant with a short inflorescence or extended to *verticilla* stres strongly interrupted and distant.

The bracts and the bracteols minimized and the corolla may overstep or not the calyx, the flowering period extends from spring to summer. Due to the application of *Thymus* species growing wild in Algeria as a culinary herb and in folk medicine (Amarti *et al*., 2011; Favarger, 1961; Hartvig, 1987).

Therefore, several essential oils of the Thymus species have been studied to investigate their chemical composition and antimicrobial, antioxidant activities (Hazzit *et al*., 2009; Amarti *et al*., 2013).

These products are of particular interest, because no bacterial resistance or adaptation has been described, and low or insignificant side effects have been found both in the essential oils and whole extracts (Nezhadali *et al*., 2014). He most species of Thymus contain phenolic monoterpenes, thymol and/or carvacrol (Amarti *et al*., 2011).

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.When you analyze essential oils with a chromatograph various organic components are found and the primary ones are as follows:

Terpenehydrocarbons: Monoterpenehydrocarbons, Sesquiterpenes, Oxygenatedcompounds: Phenols, Alcohols, Monoterpene alcohols, Sesquiterpenealcohols: Aldehydes, Ketones, Esters, Lactones, Coumarins, Ethers, Oxides (Boughendjioua, 2014).

Fourier Transformed Infrared Spectroscopy (FTIR) is based on the absorption of infrared radiation by the analyzed 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 (Meskine and Ouardi, 2010).

The aim of this work was to clearly describe the functional groups of Thyme (*Thymus ciliatus)* essential oils in order to better define the field of investigation of this natural product.

Material and methods

Plant material

The aerial part (leaves, stems) of *Thymus ciliatus* was harvested at vegetative, flowering, and after flowering stage of development from Annaba, Algeria.

The plants collected were identified by the Biological Vegetable Laboratory. Plant samples were dried in the share and conserved for future use.

Isolation of the essential oil

The100g of the air-dried leaves, stems were submitted for 03 hours to hydrodistillation using a Clevenger-type apparatus (Clevenger, 1928) according to the method recommended in the European Pharmacopoeia (European Pharmacopoeia, 1996). The essential oils were driedover anhydrous sodium sulphate and then stored at 4 °C (Kaloustrian and Hadji-Minaglou, 2012).

FTIR analysis

Fourier transform infrared spectroscopy is an analytical testing technique used to identify organic and some inorganic materials through the application of infrared radiation (IR).

As the sample absorbs the infrared light, the absorbance of energy at the various wavelengths is measured to determine the material's molecular composition and structure.

The patterns of absorption bands at the various wavelengths throughout the infrared region (or the FTIR spectrum) are unique to each material. Once the spectrum is produced, computer searches of reference libraries assist in the material's identification.

FTIR analysis is used to: Identify unknown materials, identify, and in some cases quantify, surface contamination present on a material, identify additives in a polymer; some need to be chemically removed, e. g. solvent extraction. FTIR analysis testing can provide: precise measurements, analysis with little or no harm to the sample, sample information collection at high speed (Boughendjioua, 2014).

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; Euclidean, 02. PSU/peak, 03. MIX PSU, 04. Peak Match, 05.PEAK/psu, 06. MIX PEAK. In our study we used the Euclidean library.

The FTIR analysis was performed at the Central Police Scientific Laboratory (Constantine, Algeria).

Results and discussion

Determination of the functional groups present using FTIR

Fourier transform infrared spectroscopy is one of the most widely employed techniques for functional groups identification. Fig. 1to3 and Table 1 showed the infrared spectra and the characteristic bands observed in Thymus oil in the range of 4000-600 cm^{-1}.

Table 1. Links present in the molecule of essential oil.

Presentlinks in essential oil	Theoretical frequency of the band (cm^{-1})	Presence of the bandin the spectrum
CHx deformation	1500-1400	Yes
C-H (aromatic)	3150-3050	Yes
Alcohol/Phenol O-H Stretch	3550-3200	Yes

In an earlier work developed by Sadou *et al.* (2016), the chemical composition of the essential oil of *Thymus ciliates* obtained at vegetative, flowering, after flowering stage was analyzedby GC–MS allowed us to identify 60 compounds and indicated that the main compounds constituting thymol (33.39%), carvacrol (30.85%) and carvacrol (31.37%).

Among the 28compounds revealed by FTIR: 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 1500-1400 cm^{-1} corresponding to CHx deformation, the signals which appeared between 3150-3050 cm^{-1} corresponding to C-H (aromatic) stretching, groups. For Alcohol/Phenol O-H with characteristic absorption 3550-3200cm^{-1}(broad, s), is one of the most distinct and easily recognizable peaks in an IR spectrum is the broad O-H absorption of alcohols and phenols (Dias, 2005) Table 1 and Fig. 4).

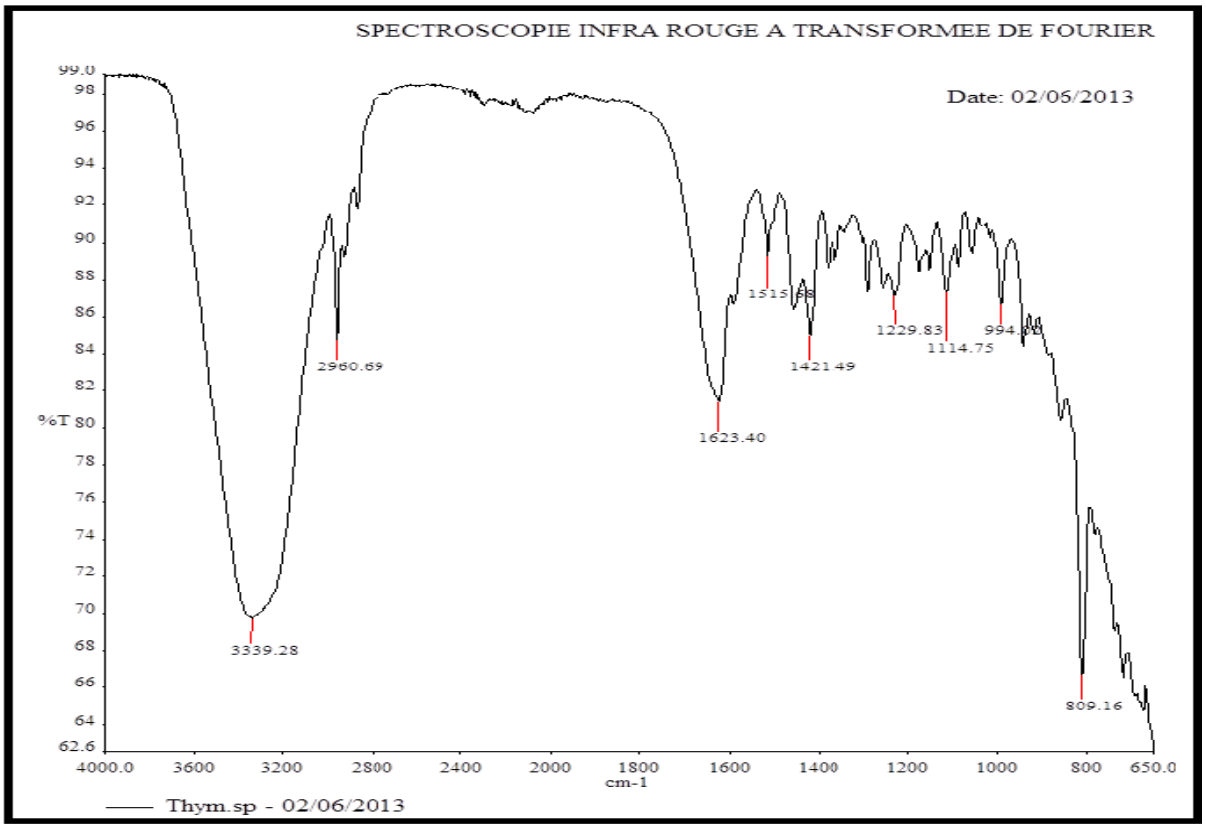

Fig. 1. FTIR of *Thymus ciliates* essential oil.

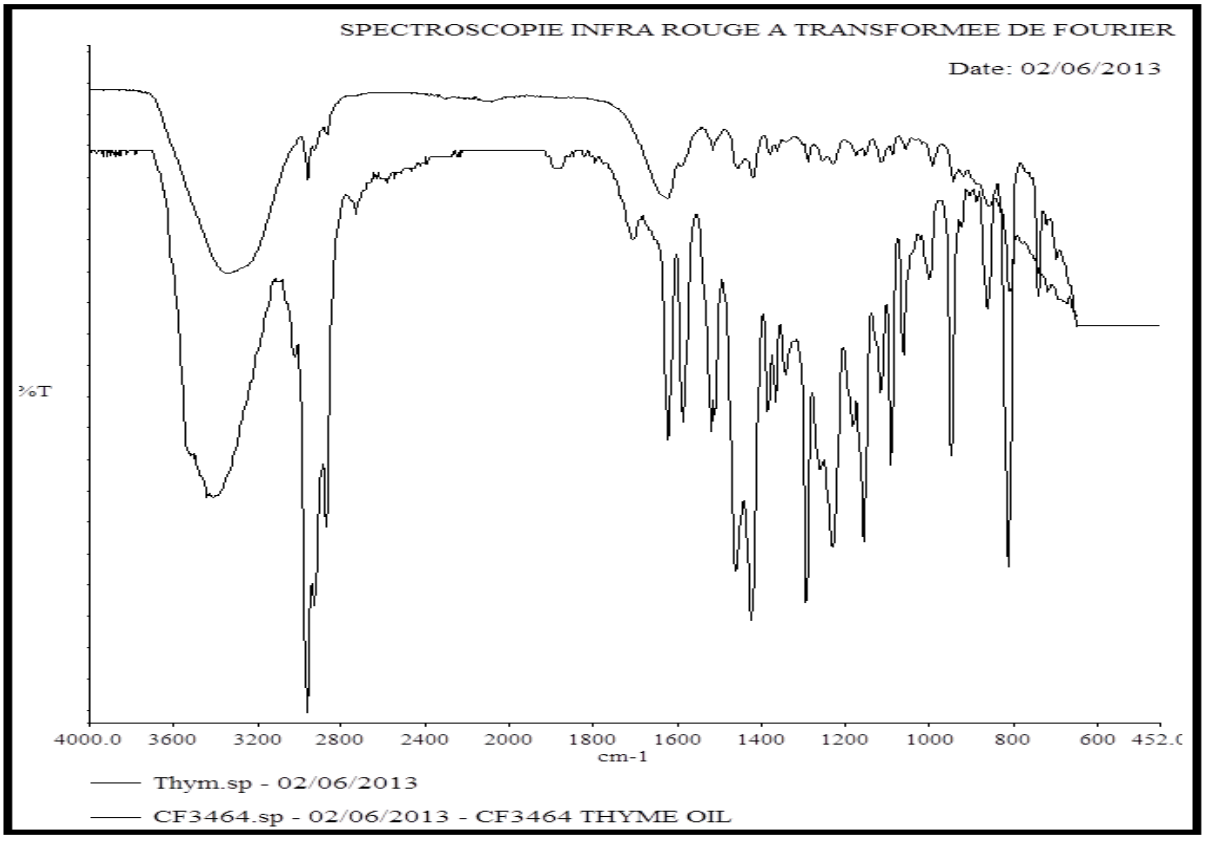

Fig. 2. FTIR of *Thymus ciliates* essential oil compared to the bibliography.

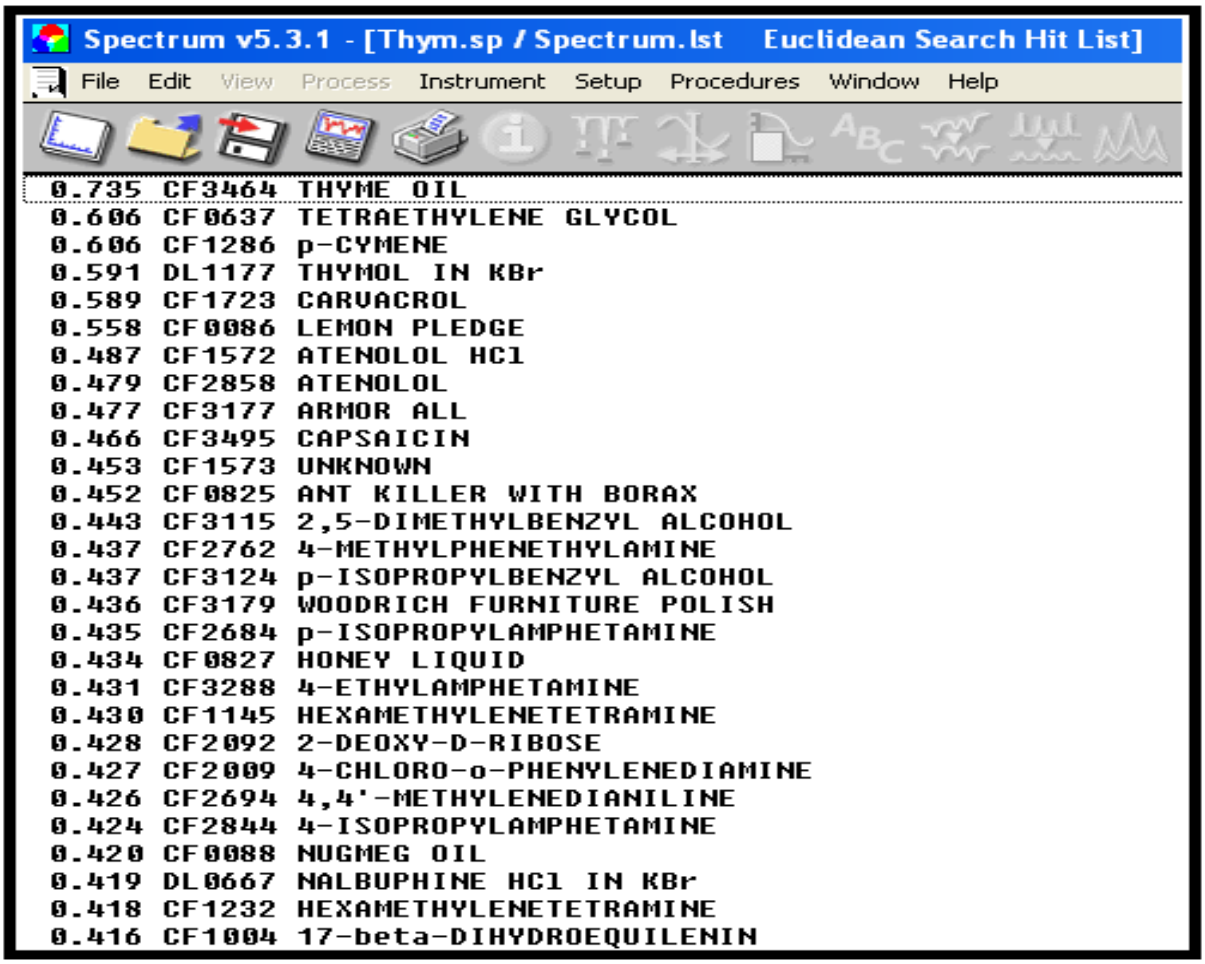

Fig. 3. Interpretation of the spectrum obtained with respect to the internal library Euclidean.

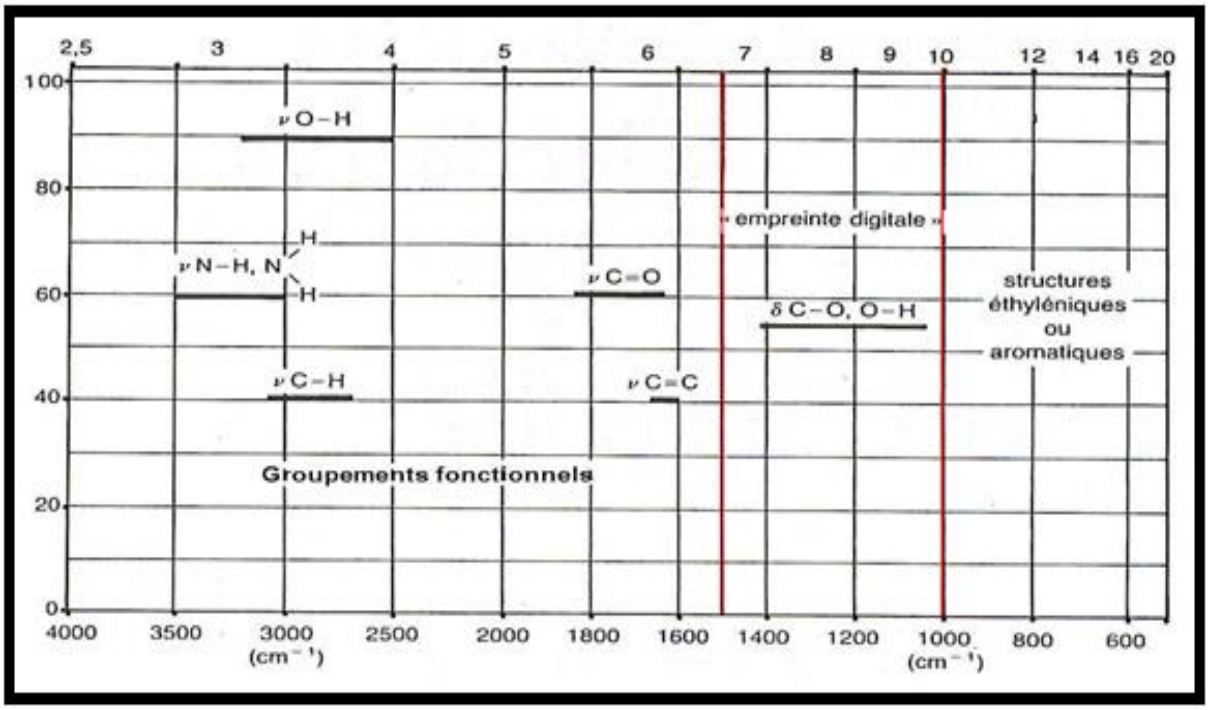

Fig. 4. Graphs of IR spectroscopy.

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 Thyme (*Thymus ciliatus*) can be fully utilized for the manufacture of perfumery products, antimicrobial and antiseptic agents or even in petrochemical industry or alkenes serve as a feedstock for the petrochemical industry because they can participate in a wide variety of reactions, prominently polymerization and alkylation.

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