

Characterization of a crystal grown from *Ocimum Basilicum* leaves and branches

R. J. C. Lima¹, A. J. D. Moreno*¹, E. M. Diniz¹, R. S. G. Oléa², J. M. Sasaki³, J. Mendes Filho³, P. T. C. Freire³, F. M. Pontes⁴, E. R. Leite⁴, and E. Longo⁴

¹ Laboratório de Crescimento e Caracterização de Cristais, Departamento de Física, Universidade Federal do Maranhão, Av. dos Portugueses s/n, CEP 65080-040, São Luís, Maranhão, Brazil

² Laboratório de Produtos Naturais, Departamento de Química, Universidade Federal do Maranhão, Av. dos Portugueses s/n, CEP 65080-040, São Luís, Maranhão, Brazil

³ Laboratório de Crescimento e Caracterização de Cristais, Departamento de Física, Universidade Federal do Ceará, C.P. 6030, Fortaleza, Ceará, 60455-970, Brazil

⁴ LIEC-CMDMC, Departamento de Química, Universidade Federal de São Carlos, CP 676, São Carlos, São Paulo, 13565-905, Brazil

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Leaves and branches of *Ocimum Basilicum* were dried, reduced to coarse powder and extracted in aqueous metanol and aqueous hexane. This paper discusses the X-ray diffraction and FT-Raman spectra measured at room temperature. The data obtained have been used to propose a general assignment of the vibrational fundamentals on the base of frequencies assigned in other related single crystals. From the results we can conclude that the crystal has a high symmetry.

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1 Introduction

In the course of a continuing search on plants found in the flora of Maranhão, a state from northwest of Brazil, we have grown crystals during EtOH extract of the dried leaves and branches of *Ocimum Basilicum*. The *Ocimum Basilicum* is known in Brasil as Manjerição and in countries of spanish language as albahaca. It is largely used in traditional medicine to bronchitis, coughs, sore throat, headache, stomach cramps, nausea, general pain and antiseptic. It can also be used in foods and flavorings [1]. It is among the plants that contain high concentrations of the defense compounds of the phenylpropene class (eugenol, chavicol, and their derivatives) [2-5]. It has been recognized as important spices for human consumption (e.g. cloves) and has high economic value.

Raman spectroscopy and x-ray scattering studies can be useful tool to understand configurations of new structures. Studies of the vibrational spectra of new crystals are invariably difficult, particularly when precise information about their structure is not yet possible. In such cases, the information that can be obtained from the Raman spectra may be insufficient to assign precisely all the bands observed. However, useful information can be obtained from crystal growth procedures that allow assigning the fundamentals satisfactorily. The objectives of the present study are two-fold. Firstly, we report the growth of crystals from leaves and branches of *Ocimum Basilicum*, and secondly we discuss their characterization by x-ray diffraction and FT-Raman spectroscopy.

* Corresponding author: e-mail: jeferson@ufma.br

2 Experimental

2.1 Extraction and crystal growth

Powdered leaves and branches of *Ocimum Basilicum* were soaked with EtOH. The material was left to extract for two weeks at room temperature. After filtered and solvent evaporation in vacuum (60°C) a dark green viscous extract was obtained. Chromatography over silica gel with solvent of increasing polarity afforded a fraction eluted with hexane- EtOAc (7-3). The vessels were covered with perforated closure and kept at room temperature (300K) in a dust free atmosphere. After a definite period of evaporation the solution became supersaturated and tiny transparent and good quality colorless crystals nucleated (Fig. 1).

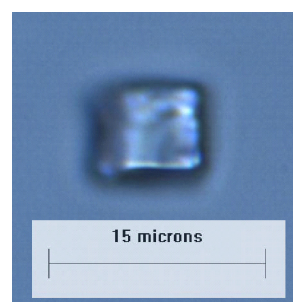


Fig. 1 Crystal grew from *Ocimum Basilicum* leaves and branches.

2.2 Characterization

The crystals obtained have been analyzed by Fourier transform Raman spectroscopy and X-ray diffraction (XRD) analysis. FT-Raman spectra were recorded in a IR FT Raman, RFS 100/S Bruker, excited by a Nd-YAG laser at 1064 nm. The spectra were recorded in the wave number range of 4000–40 cm^{-1} . Powder X-ray diffraction analysis was carried out in the 2θ ranges from 5° to 32° using a Rigaku DMAX X-ray powder diffractometer. X-ray source was $\text{CuK}\alpha$, with an accelerating voltage of 40 kV and a current of 25 mA.

3 Results and discussion

3.1 X-ray study

The x-ray diffraction pattern shows only four peaks, indicating a high symmetrical crystal (Fig. 2). A search in the literature for x-rays diffraction data comparison did not allow the identification of the crystal structure.

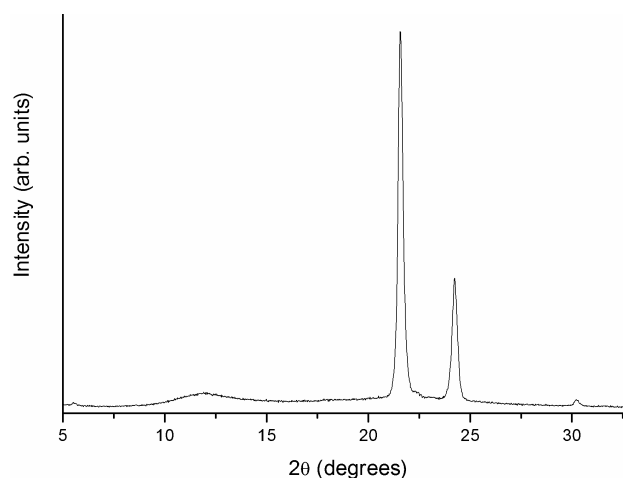


Fig. 2 X-ray diffraction pattern of crystal of *Ocimum Basilicum*.

3.2 Vibrational studies at room temperature

FT-Raman spectra of the *Ocimum Basilicum* crystal measured in the 3600–100 cm^{-1} frequencies region are shown in Fig. 3. The wavenumbers, relative intensities of the bands and proposed assignment are summarized in Table 1. The assignments labeled in Table 1 is a description of the modes involved rather than a corrected quantum-mechanical description of individual precise molecular motion, so, it is a tentative assignment. The bands observed in the above spectra are discussed in close relation to other organic compounds [6-13]. Although there is no precise information about the crystal structure, some feature should be expected. Among the several possible distinct structures we can suppose a simple aliphatic structure.

Table 1 Experimental data for the vibrational spectra of crystal grew from *Ocimum Basilicum* leaves and branches.

Raman Position	Assignments
2957 sh	$\nu_a(\text{CH}_2)$
2.930 w	$\nu_a(\text{CH}_3)$
2880 vs	$\nu(\text{CH})$
2846s	$\nu(\text{CH})$
2720 w	
1485 sh	$\delta(\text{CH}_2)$
1462 s	$\delta(\text{CH}_2)$ sciss
1440 s	$\delta(\text{CH}_3)$ bending
1414 s	$\delta(\text{CH}_3)$ bending
1370 vw	$\delta(\text{CH}_3)$ sciss
1294 s	$\delta(\text{CH})$ bending
1170 vw	
1130 s	
1118 sh	
1060 s	$\nu(\text{CO})$
889 vw	$\nu(\text{CC})$
874 vw	$\nu(\text{CC})$
236 vw	$\delta(\text{skel.})$
220 w	$\delta(\text{skel.})$
110 w	Translacional mode

A careful inspection of the Raman spectra in the entire frequency region reveals some interesting points:

1. Their FT-Raman spectra did not show the presence of stretching of hydroxyl and amide groups (3500-3000 cm^{-1});
2. Also, there are no bands in the region of carbonyl stretching, aliphatic unsaturated stretching, aliphatic ring compounds and aromatic components, and deformations of amide (1750-1500 cm^{-1});
3. The C-C stretchings occurs in the same regions of similar vibrations in aminoacids [10-14].
4. There are no fundamental modes near 1205 and 1145 cm^{-1} , characteristics of either δCCH of ring structure and νCC ring breathing modes.
5. There are no peaks between 600 and 800 cm^{-1} indicating short polymethylenic chains.

4 Conclusion

Based on comparative Raman spectroscopic analysis it was possible to provide a plausible assignment of Raman fundamentals observed in crystals grown from leaves and branches of *Ocimum Basilicum*. The absence of bands characteristics of unsaturated aliphatic, aromatic rings and esters suggests that the crystal is

predominantly composed of saturated chain, probably bonded by weak van der Waals forces between the chains. All above features could be accommodated only in a symmetrical structure, probably composed of fragments such as $\text{-CH}_2\text{CH}_2\text{CH}_2$, $\text{-CHCH}_2\text{CH}_2$, etc.

It is interesting to notice that the *Ocimum Basilicum* crystal has high symmetry because there are few peaks in their Raman spectra. The x-ray diffraction pattern confirms this conclusion.

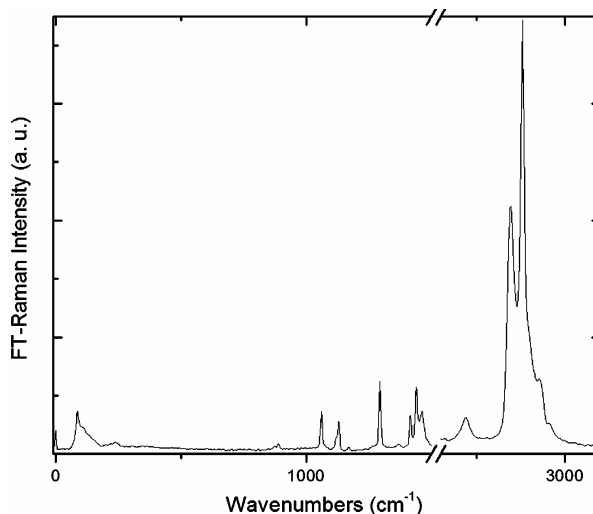


Fig. 3 FT-Raman spectra of the crystal grew from leaves and branches of *Ocimum Basilicum*.

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