

Investigations on Mechanical and Dielectric Properties of NLO material: 4-methoxybenzoin

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Abstract

Organic nonlinear optical single crystals of 4-methoxy benzoin were grown by slow evaporation technique. The XRD studies confirm the crystalline nature of the grown crystals. The mechanical strength of the grown crystal was found by using Vickers microhardness test. The dielectric behavior of the crystal was determined at different temperatures in the frequency range of 50 Hz to 5 MHz was investigated.

Key words: Solution growth, Single crystal XRD, Microhardness, Dielectric studies.

1. Introduction

The Nonlinear optics (NLO) is now established as an alternative field to electronics for future photonic technologies. The fast growing development in the optical fiber communication systems have stimulated the search for new NLO materials that are capable of fast and efficient processing of optical signals and integrated optics [1]. Many optically active amino acids show high efficient optical second harmonic generation and are promising candidates for laser and optical communication technology [2]. The crystals capable of generating second harmonics must have a unit cell with no centre of inversion and this requirement is met by the crystals of pure amino acids since these molecules themselves are dissymmetric. The growths, structural, vibrational, optical and second harmonic generation (SHG) properties of single crystals of 4-methoxy benzoin were reported earlier [3]. The present investigation, deals with the growth of 4-methoxy benzoin single crystals by slow evaporation technique. The grown single crystals were subjected to single crystal X-ray diffraction, microhardness and dielectric studies. The results of these investigations are discussed.

2. Materials and Methods

4-methoxy benzoin has been synthesized using slow evaporation technique at room temperature yielded many small crystals. The defect-free, optically clear, and perfectly shaped tiny crystals were chosen as seeds for the growth experiment. From XRD studies, it is understood that 4-methoxy benzoin belongs to orthorhombic in structure with space group $Pca2_1$. The single crystalline nature is thus confirmed and the space group reveals that the material shows no inversion symmetry (non-centrosymmetry). The mechanical characterization of the 4-methoxy benzoin crystals have been examined using Vickers microhardness studies. The single crystals with flat and smooth faces were chosen for the static indentation tests and the same crystal was mounted on the base of the microscope. The indentations were made gently by varying the loads from 10 to 50 g for a dwell period of 10 s using both Vickers diamond pyramid indenter attached to an incident ray research microscope (Mitutoyo MH112, Japan). The intended impression of Vickers was approximately square in shape. The shape of the impression is dependent on the structure, face and materials used. After unloading, the length of the two diagonals was measured by a calibrated micrometer attached to the eyepiece of microscope. For each load, at least five well-defined indentations were considered and the average is taken as d . The dielectric measurements for 4-methoxy benzoin single crystals have been carried out using HIOKI 3532-50 LCR HITESTER. Carefully selected samples were cut using a diamond saw and polished using paraffin oil and fine grade alumina powder to obtain a good surface finish, and coated with conducting silver paste in order to increase the ohmic contact. The dielectric permittivity measurements were carried out with the sample placed inside a dielectric cell whose frequencies could be varied in a controlled manner for different temperatures 35 -95°C, respectively.

3. Results and discussion

3.1 Single Crystal XRD

The study revealed that 4-methoxy benzoin single crystals belong to orthorhombic crystal system with a non-centrosymmetric space group $Pca2_1$. The cell parameters are $a=14.47$ Å, $b=14.09$ Å $c=5.84$ Å and $V=1192.64$ Å³. The XRD data of the present work is in good agreement with the reported values [3].

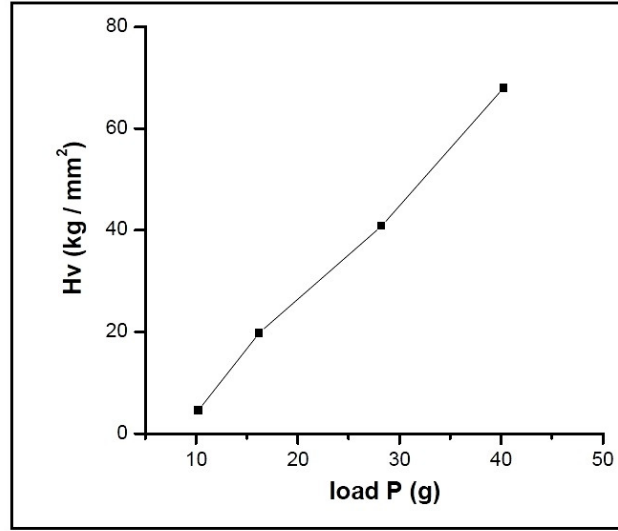
3.2 Microhardness study

The mechanical properties of crystals are evaluated by mechanical testing which reveals certain mechanical characteristics. The fastest and simplest type of mechanical testing is hardness measurement. Among the different testing methods, the Vickers hardness test is more commonly used. Microhardness study was conducted using a Vickers microhardness tester fitted

68 with a diamond pyramidal indenter. The applied load was varied from 10 to 50 g for a constant
69 indentation period of 10 s. The Vicker's hardness number H_V is calculated using the relation

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71
$$H_V = 1.8544P / d^2 \text{ kg} / \text{mm}^2$$
 (1)
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73 where P is the indenter load in kg and d is the diagonal length of with applied load is shown in
74 Fig. 1. It is evident from the plot that the microhardness of the crystal increases with increasing
75 load. According to Onitsch [4], the work hardening coefficient (n) has been found to be 1.62
76 (Fig.2).
77



78
79 Fig. 1. Vickers hardness of 4MB as a function of applied load
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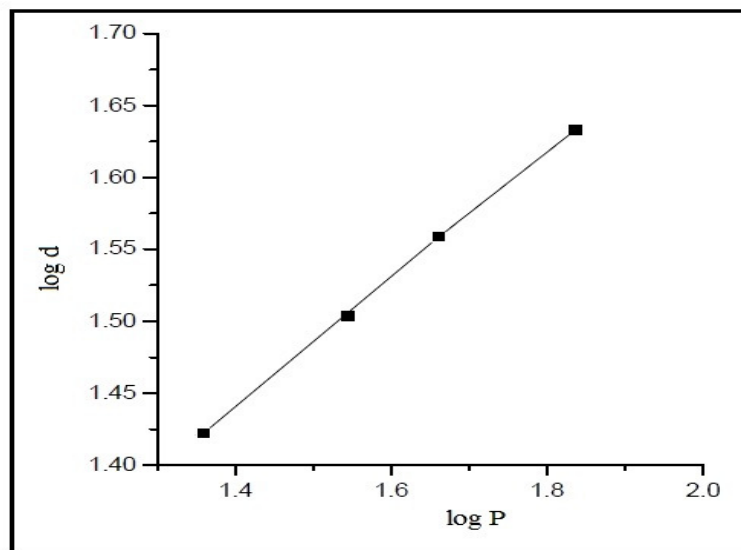


Fig. 2. Plot of log d Vs log p

3.3 Dielectric Studies

The dielectric analysis is an important characteristic that can be used to bring knowledge based on the electrical properties of a material medium as a function of temperature and frequency. Based on this analysis, the capability of storing electric charges by the material and capability of transferring the electric charge can be measured. The dielectric properties are correlated with electro optic property of the single crystals, particularly when they are non-conducting materials [5]. Microelectronics industry needs low dielectric constant (ϵ_r) materials as an interlayer dielectric. The dielectric constant is calculated using the formula

$$\epsilon_r = \frac{Ct}{\epsilon_0 A} \quad (2)$$

where C is capacitance (F), t the thickness (m), A the area (m^2), and ϵ_0 the absolute permittivity in the free space having a value of $8.854 \times 10^{-12} \text{ Fm}^{-1}$. The frequency dependence of the dielectric constant at different temperatures is shown in Fig.3. It is found that the dielectric constant of 4-methoxy benzoin is high at low frequencies and it decreases with increasing frequency. The high value of dielectric constant is attributed to high ionic conductivity. Most of the solid electrolytes have high dielectric constant [6]. The dielectric constant of a material is composed of four contributions such as electronic, ionic, orientational and space charge polarizations. All these may be active at low frequencies. In fact, the nature of the variation of dielectric constant

with frequency indicates which type of contributions present in the sample. The decrease in dielectric constant with increase in frequency may be attributed to the dependence on the electronic, ionic, orientational and space charge polarizations. Fig. 4 shows the decrease of dielectric loss of 4-methoxy benzoin as a function of frequency. It is also observed that as the temperature increases, the value of dielectric constant also increases to a considerable value (Fig. 5). The same trend is observed in the case of dielectric loss versus temperature as well (Fig. 6). The low value of dielectric loss indicates that the grown crystals are good in quality [7]. The characteristic of low dielectric constant and dielectric loss with high frequency for a given sample suggests that the sample possesses enhanced optical quality with lesser defects and this parameter is of vital importance for various nonlinear optical materials and their applications.

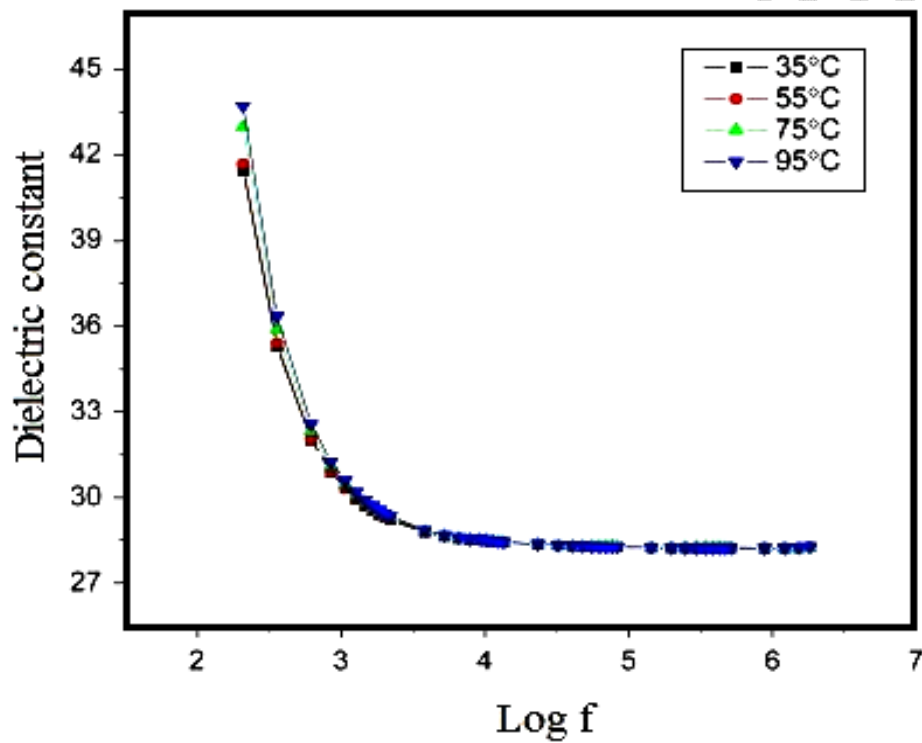


Fig. 3. Variation of dielectric constant with log f

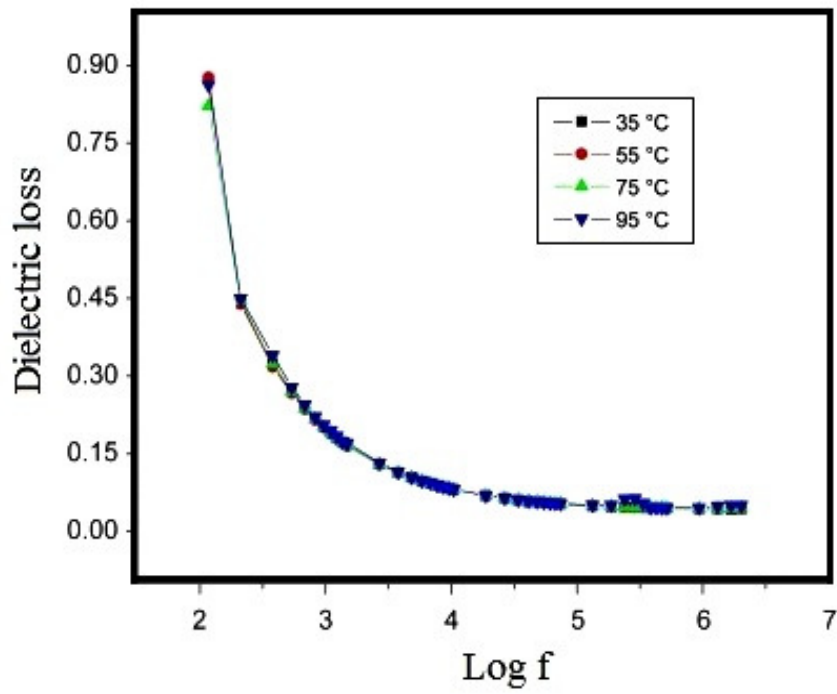


Fig.4. Variation of dielectric loss with log f

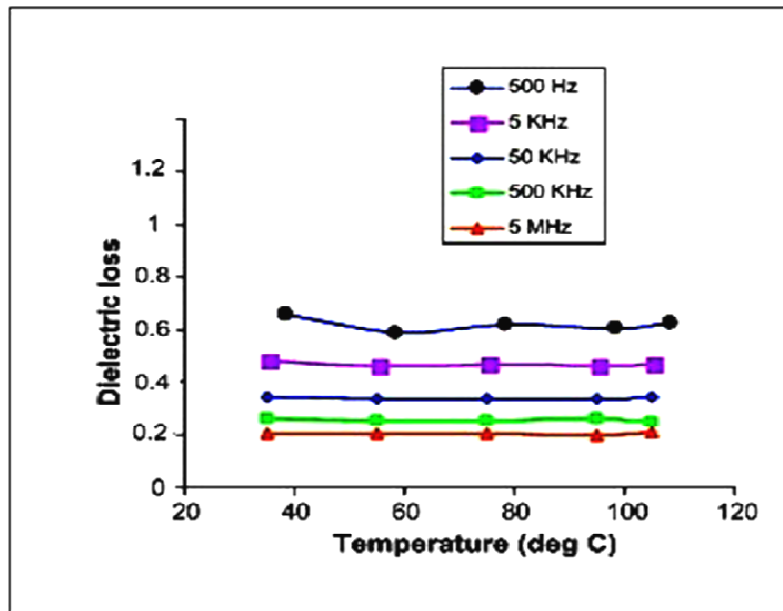


Fig.5. Temperature dependence of dielectric constant

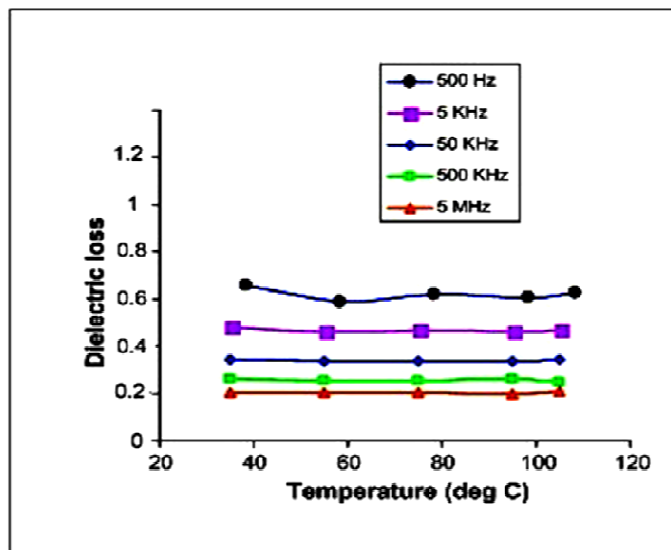


Fig.6.Temperature dependence of dielectric loss

Conclusion

The single crystals of 4-methoxy benzoin were grown by slow evaporation technique. The grown crystal is confirmed by X-ray diffraction analysis and it is found that the crystal belongs to the orthorhombic system with the space group $Pca2_1$. Mechanical hardness studies reveal that Vicker's hardness number increases as the load increases. The dielectric studies revealed that 4-methoxy benzoin has low dielectric loss with less power dissipation and hence this crystal can be used for optical applications. The dielectric characterization shows low value of dielectric constant at higher frequencies for these crystals.

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