INVESTIGASTION OF SPECTRAL AND DIELECTRIC PROPERTIES OF GLYCINIUM OXALATE CRYSTAL

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Abstract

An organic material of GOX crystal has been synthesized by slow evaporation technique. The lattice dimensions were determined from the single crystal XRD technique. The presence of functional groups was identified by FT-IR spectroscopic study. UV-Visible spectrum analysis was determined linear optical properties and the band gap energy is 5.18 eV. The luminescence spectrum was measured in the GOX crystal. The dielectric studies reveals that the dielectric constant, dielectric loss and ac conductivity of the material.

Keyword: crystal growth; FT-IR; band gap; mechanical; electrical properties

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1. Introduction

Amino Acid attract lots of interest from chemists, optical physicists and material scientists because of their excellent performance with respect to opto-electronics and NLO properties [1].Amino acid salts exhibit structural phase Transitions of various types and they also exhibit ferroelectric, antiferroelectric or ferroelastic behavior[2]. Glycine is the simplest and the only non-chiral amino acid nature. It can exist in cationic, Zwitter ionic or anionic forms. Oxalic acid is the simplest dicarboxilic acid and it can exist in the oxalate. Glycine exists in the cationic forms with a positively charged amino group, while oxalic acid exists as the anion [3].

Hence, in this present study an attempt has been made to growth of glycine oxalate is slow evaporation technique at room temperature. The grown crystal are subjected to characterization studies like single crystal XRD, FT-IR,UV-Vis-NIR, electrical, mechanical, fluorescence and thermal stability.

2. Experimental Method

Glycine Oxalate single crystal was prepared by slow evaporation technique. Glycine and Oxalic acid were taken in equimolar ratio (1:1) in double distilled water to get saturated aqueous solution and was then allowed to evaporate at room temperature. Colourless transparent crystal of glycine oxalate were obtained with of glycine oxalate were obtained within 18 days. Repeated recrystallisation yielded good quality crystal. The grown crystal of GOX is shown in fig.1.

Fig.1. as- Shown in grown crystal of GOX

3. Result and Discussion

3.1 Single Crystal XRD

The X-ray data were collected using the Bruker Kappa Apex II X-ray diffractometer. The structure was solved by direct method and refined by full matrix least square technique using the SHEL XL program. The calculated lattice parameter values are a = 10.65Å, b = 5.69Å, c = 12.14Å, $\alpha = \gamma = 90^{o}$, $\beta = 113.95^{o}$, and Cell Volume (V) = 673Å^3. The X-ray data prove that the GOX crystal is monoclinic structure and space group of$P2_1/C$. The XRD data of the sample are in good agreement with the reported values and thus confirm the grown crystal [4].

3.2 FT-IR Spectral Study

The functional groups were identified with Perkin Elmer RX I spectrometer with KBr pellet technique used to recorded in the range of 400-4000 cm^{-1}. The resulting spectrum is shown in fig.2. In the high-energy region, there is a broad band between 2100 and 3500 cm^{-1}. There are intense sharp peaks in this band at 3402, 3208, due to N-H and O-H vibrations. The involvement of NH_3^+ hydrogen bonding in evident of the fine structure of the band in lower-energy region. The C-H stretching vibration of the methylene group is observed at 2856 cm^{-1}. The C=O stretch of COOH seems to have an intense sharp peak at 1725 cm^{-1}.

The peaks at 1594 cm^{-1} are due to asymmetrical NH_3^+ bending mode. The well sharp peak at 1511 cm^{-1} is due to asymmetrical N-O bend. The CH_2 bending modes are observed at 702 cm^{-1} [5]. These vibrations clearly demonstrate the existence of glycine in it is salt form with Oxalic acid.

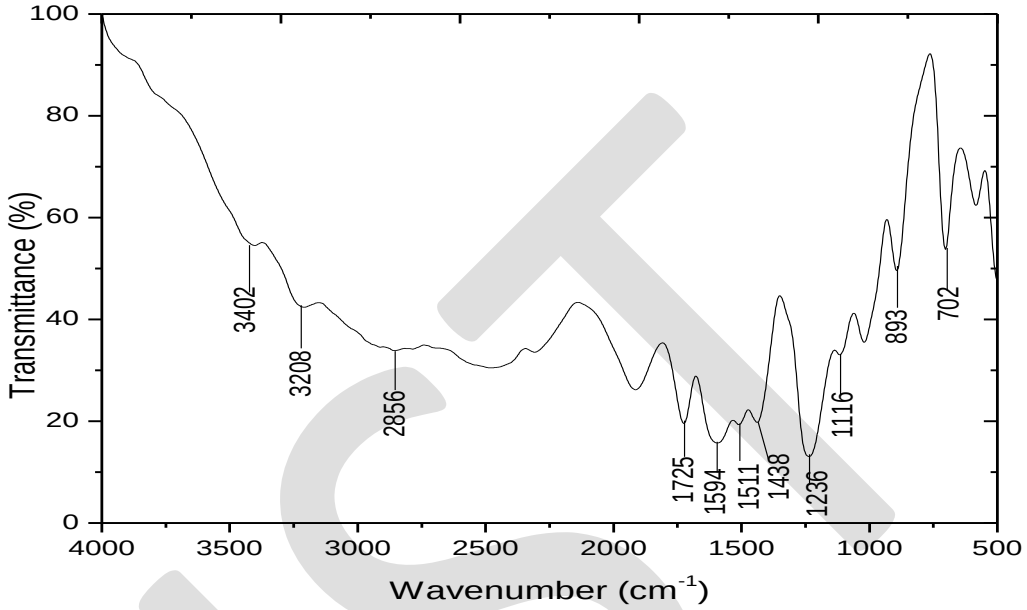

Fig.2. the FT-IR spectrum of GOX crystal

3.3 UV-Vis-NIR Studies

The UV-Vis spectrum studied by Perkin-Elmer Lambda 35 spectrometer the range of 200-800 nm. The recorded absorbance spectrum is shown in fig.3. It reveals that grown crystal has widely absorbance in the visible region. The lower cut off wavelength is at 239 nm. A peak occurred at 239 nm in the spectrum may be due to the π-π* transition. A feature that promotes possible optical application in device operating at the visible-NIR wavelength range [6,7]. The optical band gap measure for the following relation,

$$\propto h\nu = A(E_g - h\nu)^n$$

Where A is a constant, E_g is the optical band gap, h is the Planck's constant and ν is the frequency of the incident photons. The band gap of GEC crystal value was found to be 5.18 eV.

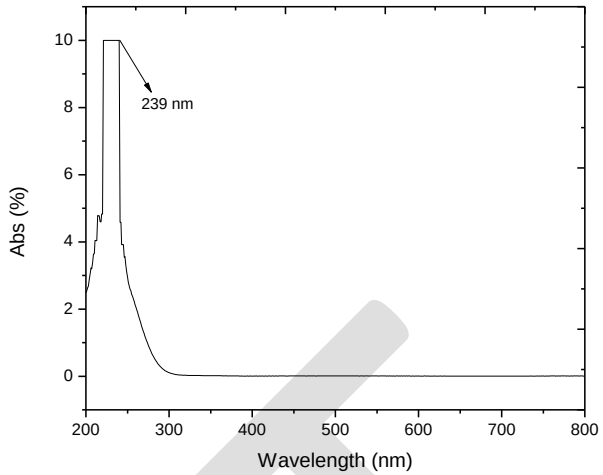

Fig.3. UV-Visible absorbance spectrum of GOX crystal

3.4 Fluorescence Study

Fluorescence is the phenomenon in which electronic states of solids are excited by light of particular energy and the excitation energy is released as light [8,9].The emission spectra of the complexes were studied in water solutions. The spectra of the complexes are similar and the spectrum of GOX was shown in Fig.4.The excitation wavelengths match the absorption spectra of the ligand. It is clear that the excitation of these complexes is mostly ligand based [10]. The spectrum shows the strong emission at 492 nm and emission spectrum the spectrum strong emits in green color.

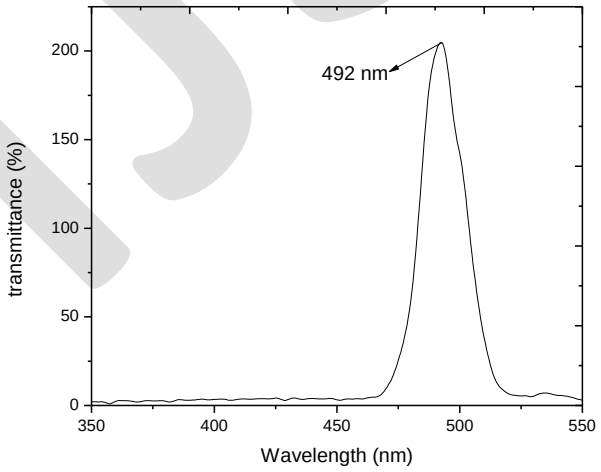

Fig.4. Luminescence spectrum of GOX crystal

3.5 Dielectric study

The dielectric constant (ε) of the material under study was calculated by using the following equation:

$$\varepsilon = \frac{Ct}{\varepsilon_o A}$$

Where C is the capacitance (F), ε_o is the free space dielectric constant value (8.854 x10-^{12} F/m), A is the capacitor area (m^2) and t is the thickness (m) of the material. The imaginary part of the dielectric constant was calculated using the following relation

$$\varepsilon_i = \varepsilon \tan\delta$$

Where tan δ is the dielectric loss. From the dielectric constant and dielectric loss, the ac conductivity (σac) of the present sample was calculated using the following relation

$$\sigma_{ac} = \omega\varepsilon\varepsilon_0 \tan\delta$$

Where ω is the angular frequency and σac is a temperature and frequency dependent function and is attributed to the dielectric relaxation caused by localize dielectric charge carriers, which obey the power law [11].

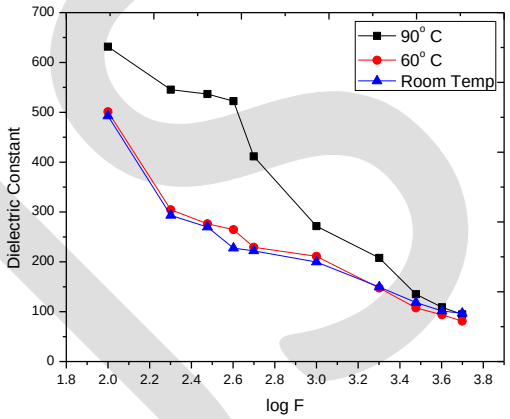

Fig.5.Variation of dielectric constant versus log F for GOX crystal

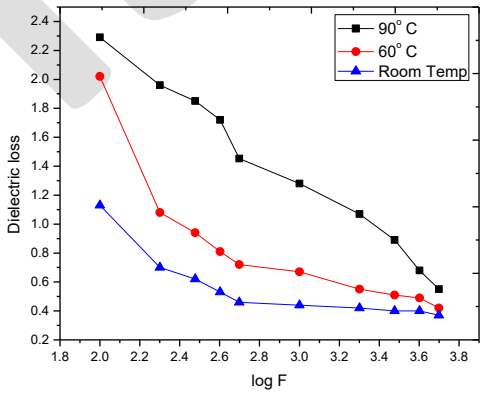

Fig.6. Variation of dielectric loss versus log F for GOX crystal

The variation of dielectric constant (ε) and loss as a function of frequency of the ac field is shown in fig.5 and fig.6. It is clear from fig.5 that dielectric constant of GMC decreases gradually with increasing frequency. The decrease of dielectric constant with increase of frequency is a normal dielectric behavior and can be explained on the basis of various polarization mechanisms. There are four primary mechanisms of polarization in materials, that is, electronic, ionic or atomic, dipolar or orientation and space charge or interfacial polarization. At low frequencies, all the mechanisms of polarization contribute to the dielectric constant and with the further enhancement in the frequency of the applied field, the contributions from different polarizations start reducing. For example, at very large frequencies, it is only electronic polarization which contributes to the dielectric constant, where as ionic polarization takes place at IR frequencies. In the present material, the high increase of dielectric constant at lower frequencies may be due to the space charge polarization because of the existing crystal lattice defects. The gradual decrease in dielectric constant with frequency suggests that the material under consideration has ferroelectric domains of different sizes and hence varying relaxation times fig.7 shows variation of σac with log(f) in the frequency range of 20 Hz to 1MHz

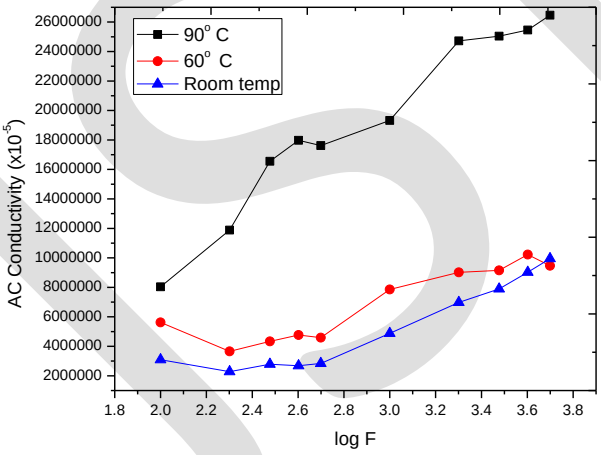

Fig.7. Variation of AC conductivity versus log F for GOX crystal

Conductivity of the crystal at room temperature region is determined by intrinsic defects caused by thermal fluctuations in the crystal. For any substance, more and more defects are produced as the temperature increases which, in turn, increase the conductivity [12].

4. Conclusion

An organic material of GOX crystal has been synthesized and good quality single crystal have been grown by slow evaporation technique. The lattice dimensions were determined from the single crystal XRD technique and found that GOX crystal belongs to monoclinic crystal with$P2_1/C$ space group. The presence of functional groups was confirmed by FT-IR spectroscopic study. In this work, the band gap energy was found to be 5.18 eV using UV-

Visible transmittance data. The luminescence spectra of the GOX crystal show UV light, green light emission which indicated their high structural and optical quality. The dielectric studies reveals that the behavior of space-charge polarization of the material. Finally, we concluded that the material may be utilized in the NLO device applications.

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