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Studies On The Growth And Characteristics Of Structural And Photoconductivity Of Bis Glycine Maleate Single Crystal

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Abstract:A single crystal of Bis glycine maleate has been grown from aqueous solution by slow evaporation method by optimizing the growth parameters within a period of 45 days. From the x-ray diffraction the crystal was found to be crystallize in hexagonal structure with a=7.402 Å, b=7.04 Å, c= 5.48 Å and α=β=90°, γ =120°. The functional group was confirmed in FTIR analysis. The UV-Vis spectral analysis has revealed the absence of high absorption region between 200 to 800nm. The optical band gap was calculated and found to be 4.06 eV. The photoconductivity study confirmed that it is a positive photoconductivity

Keyword:X-Ray Diffraction, Growth from solution, Single Crystal Growth, Optical Properties, FTIR,Photoconductivity.

INTRODUCTION:

Since 1961, when the nonlinear optical (NLO) phenomenon was observed for the first time, NLO frequency conversion materials have played more and more important role in many fields, such as laser technology, optical communication, optical data storage etc. [1]. In the context of NLO, organic materials have advantages such as large NLO coefficients and structural diversity or flexibility, compared to the inorganic counterparts [2]. They also have some inherent drawbacks, for example, poor physico-chemical stability and low mechanical strength. As a result, the quest for new frequency conversion materials is presently concentrated on semi-organic crystals due to their large nonlinearity, high resistance to laser induced damage, low angular sensitivity and good mechanical hardness [3–5].

A survey of literature shows that the three polymorphs of α, β and γglycine can be formed under different solution conditions. γ-Glycine can be formed by spontaneous nucleation from pure aqueous glycine. γ-Glycine, the least table polymorph, can be formed from mixed solvents such as methanol or ethanol and water. γ-Glycine can be formed from aqueous solutions of glycine made acidic with acetic acid or basic with ammonium hydroxide [6]. Zaccaro et al. [7] reported that the no photochemical laser-induced nucleation of solutions of the amino acid glycine in water reduced the γ-polymorph. Garetz and Matic [8] reported a fascinating work on polymorph of glycine.

In the present investigation, a new organic compound of glycine with maleic acid, a dicarboxyclic acid with relatively large π- conjugation was synthesized in the alkaline medium of 10% ammonium hydroxide solution. The bulk single crystals of BGM have been grown by slow evaporation method and the grown crystals were subjected to structural, Optical and mechanical property studies.

1.Experimental procedure

The analytical-grade glycine and maleic acid in the molar ratio 2:1 were taken as the starting materials for synthesizing BGM. The calculated amount of glycine was first dissolved in the alkaline medium of 10%ammonium hydroxide solution. Then, maleic acid was added to the solution slowly with continuous stirring at a temperature slightly more than the room temperature. The prepared solution was left to dry at room temperature. The BGM salt was obtained after two days. The purity of the synthesized salt was improved by successive recrystallization process. The reaction mechanism of the chemical synthesis is as follows.

$$2(NH_2CH_2COOH) + COOH - CH = CH - COOH \rightarrow HOOC - H_2C - NH_3^+COO^- - C$$

Bis Glycine Maleic acid

$$= CH - COO^- NH_3^+ - CH_2 - COOH$$

Bis Glycine maleate

Ammonium hydroxide was found to be a better solvent for growing single crystals of BGM because of its high solubility and low evaporation rate. Good quality crystals were obtained within 45 days as shown in Fig:1

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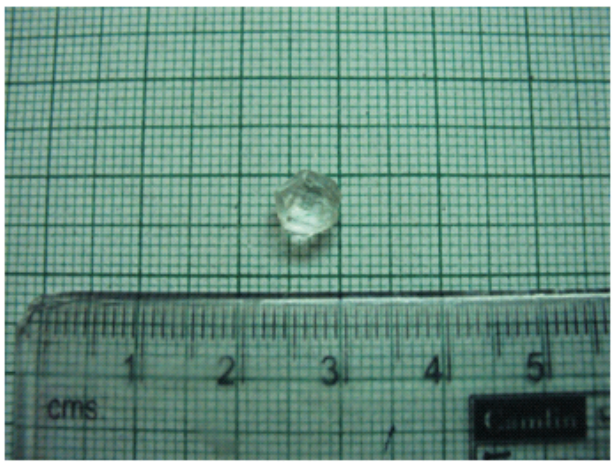

Fig:1 Grown BGM crystal

Characterization

3.1 X-ray diffraction studies

Single crystal X-ray diffraction study was carried out on the BGM single crystal with ENRAF NONIUS CAD4 diffractometer. From this study, the lattice parameter values of BGM crystal were found to be a=7.402 Å, b=7.04 Å, c= 5.48 Å and α=β=90°, γ =120° .This indicates that BGM crystallizes in hexagonal system. X-Ray diffractogram of the crystal was recorded on the XPERT-PRO diffractometer using CuKα radiation(λ=1.540598).The particle size , dislocation density , strain values were calculated for the FWHM values in Table:1.It reveals that when the FWHM value increases dislocation density and strain value increases as the particle size decreases. Hence, the single crystal of BGM satisfies the prime requisite for the material to possess nonlinear optical properties.

Table: 1 Structural Parameters

S.no	FWHM(β)	Particle size(D)	Dislocation Density(δ)	Strain Value(ε)
1	0.04	36.9285	0.7332	0.0098
2	0.08	19.723	2.5821	0.01835
3	0.12	12.8272	6.1186	0.0282
4	0.16	9.349	11.4655	0.03873

3.2 FT-IR Spectral analysis

The infrared spectral analysis is effectively used to understand the chemical bonding and it provides information about molecular structure of the synthesized compound. Each and every one chemical compounds have their own typical IR spectrum [9,10].The FTIR Spectrum of the title material was recorded using the PEFTIR Spectrometer in the wave number range 600-4000 cm-1 by KBr pellet method and is shown in the Fig: 2 The intense absorption band at 3107 cm-1 is difficult to assign for the overlapping frequencies of (O –H)and (N –H).Because O –H vibrations of water molecule and hydrogen bonds extend to the region near 3200 cm -1 .The broad band in the higher energy region between 2169 and 1594cm-1 is due to NH3+ Bending vibrations. The peaks at 1527 cm-1 corresponds to NH3 +symmetric stretching. These results confirm that the glycine exists in zwitter ionic form in the grown crystal. The involvement of NH3 + in hydrogen bonding is evident from the fine structure of the band in the lower energy region. The absorption bands occur at 885 cm -1corresponding to C –C stretching.On investigation of the absorption bands below 1000 cm-1 , COO bending COO rocking are identified at 685 and 502 cm-1 ,respectively [11].

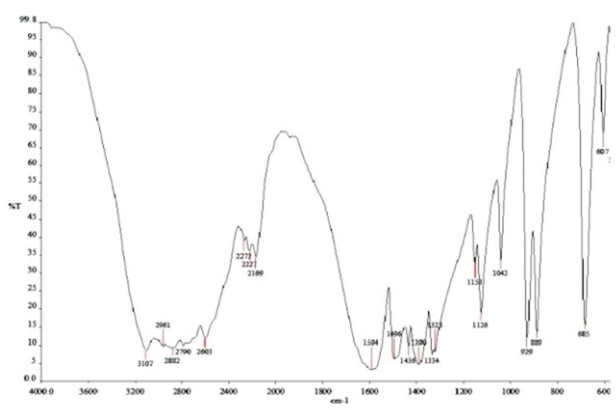

Fig: 2 FT-IR Spectrum Of BGM crystal

3.3 UV-VIS-IR analysis

Optically transparent BGM single crystal recorded the absorption spectrum by employing Varian UV-Vis-NIR spectrometer in therange200 to 800 nm. The recorded UV–Vis–NIR absorptionspectrum isshownin theFig: 3. Thereisno absorptionin theentireVis–NIR rangewithalowercut-offat 206nm,the wide range of transparency in UV, entire visible and IR region makes it a very potential material for blue light emission. It can be seen from absorption spectra that there is no absorption in range 400 nm to 800nm enables the use of this material for second harmonic generation (SHG) applications. The threshold at which the absorption data showing an abrupt rise is determined graphically and it can be the band gap of the material [12]. According to Tauc relation, [13-15] a graph (Fig. 4) has been plotted between hν and (αhν)2 to estimate the direct band gap value, where α is absorption coefficient and hν is the energy of the incident photon (E = hν).The energy gap (Eg) is determined by extrapolating the straight line portion of the curve to (αhν)2 = 0. From the plot, the band gap of BGM is found to be 4.06

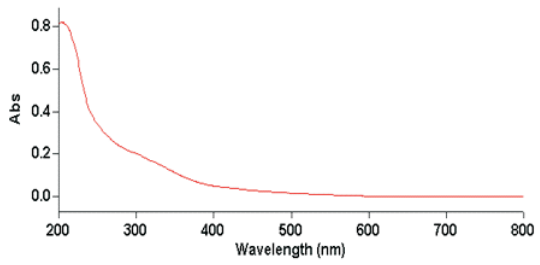

Fig: 3 Optical Absorption Spectrum of BGM Crystal

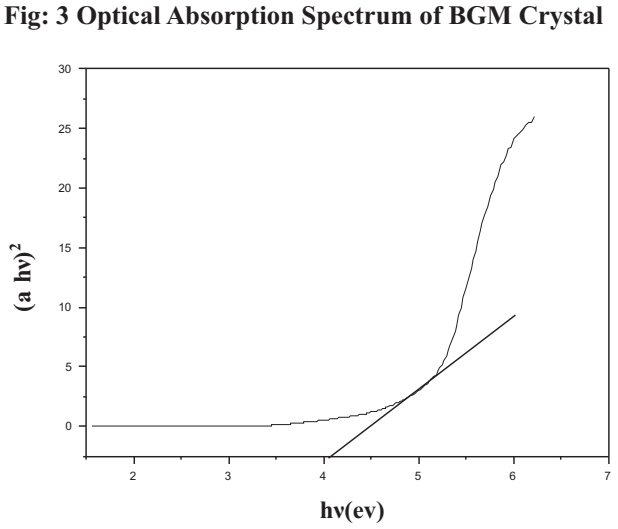

Fig: 4 Plot of (αhν)2versus photon energy hν

3.4 Photoconductivity

The field dependent photoconductivity of the BGM crystal is shown in Fig. (5). It is observed that the dark current (d) and the photocurrent (p) shows linear response with respect to the applied field and at any instant, the photocurrent is found to be greater than the dark current. Hence, the crystal exhibits positive photoconductivity. As the crystal is possessing high band gap for photon absorption, the applications are extended for soliton wave communication, where the optical pulses propagate within the photonic band gap for large distances without any distortion [16].

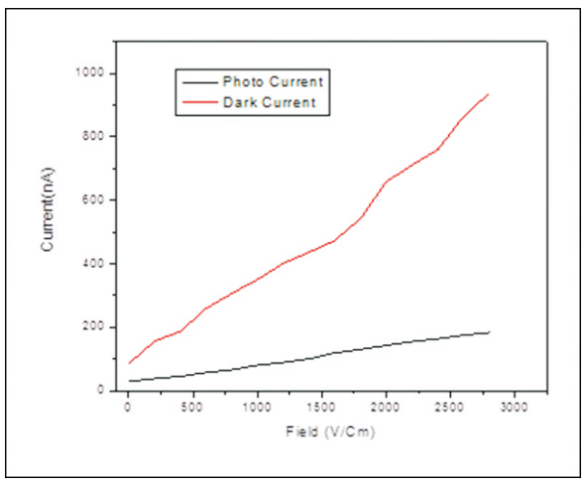

Fig: 5 Photoconductivity of BGM crystals

CONCLUSION

Optically transparent nonlinear optical BGM single crystalwasgrown by temperature lowering method from aqueous solution containing glycine, maleicacid and ammonium hydroxide.The singlecry stalX-raydiffr action studiesrevealedthatthegrowncrystalofBGMbelongsto a hexagonal system. FTIR spectrum confirms the presence of various functiona lgroupsof Bisglycinema leateand the existenceofglycineinzwitterionicformin thegrowncrystalhas alsobeen identified.Theoptical behaviour was studie dusingUV – Vis – NIRanalysisandfoundthatthereisno absorptionbetween400 to800nm. The band gap was found to be 4.06eV.The photoconductivity study confirms that it is a positive photoconductivity. Since photocurrent is greater than the dark current.

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