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Enhancement of Pure and Dye Doped Glycine Related Single Crystal by Slow Evaporation Method

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ABSTRACT

Single crystals of Violet dye doped Glycine Hydro Bromide (VGHB) has been successfully grown by the slow evaporation technique from the aqueous solution. Good optical quality Single crystal of VGHB with well dimension is obtained. The structural characterization of the grown crystals was confirmed by Powder X-Ray diffraction. The modes of vibration of the molecules and the presence of functional groups were identified using FT-IR technique. The UV – visible spectrum proves that the grown crystals have wide optical transparency in the entire visible region. Its mechanical hardness were estimated by Vickers micro hardness tester. The Meyer index value (n) indicates the grown crystal belongs to soft-material category. The stiffness constant (C11) was computed using the Wooster's empirical relation.

Key Words: Powder XRD, FT-IR, UV-visible spectrum, Micro Hardness, Non Linear Optics

INTRODUCTION

The synthesis of novel and efficient frequency conversion materials has resulted in the development of semi-organic materials, which have high resistance to laser induced damage, low angular sensitivity, large nonlinearity and good mechanical stability [1–3]. Amino acids are exciting organic materials for NLO applications as they contain donor carboxylic acid (COOH) group and the proton acceptor amino (NH_2) group in them, known as zwitterions. These Amino acids hydrogen bonds, in the form of N–H^+—O–C, which are very strong in nature. Hydrogen bonds have also been used in the possible generation of non-centrosymmetric structures, which is a requirement for an effective NLO crystal. Amino acids have two or more types of coordination atoms and can operate as various bridging ligands [4-6]. Glycine is the simplest form of amino acid which reacts with other inorganic compounds to give a good mechanical and thermal stability. It has no asymmetric carbon and is optically inactive [7]. The growth of Glycine with Hydro Bromide were already reported. In thispaper we report the growth, optical,mechanical and dielectric properties of VGHB single crystals.

MATERIALS & METHODS

A well cleaned beaker was filled with certain amount of water which was doubly distilled. The water was chosen as solvent. VGHB single crystals were grown from aqueous solution by slow evaporation technique by dissolving a stoichiometric ratio (1:1) of high-purity Glycine (AR grade), Hydro Bromide(AR grade) in doubly deionized water at room temperature (303 K). After preparing a clear solution of glycine, the proportional amount of Hydro Bromide mixed progressively with continuous stirring of the solution to bring a homogenous mixture. The prepared solution was filtered and kept in the dust- free atmosphere. As days go on crystallization takes place, and the seed crystal keeps on growing and attain the maximum size till the solvent get evaporated. Defect-free seed crystal was suspended in the mother solution which was allowed to evaporate at ambient temperature. Purity of the crystal was improved by successive re-crystallization process. Colorless good optical single crystal of GZAS of dimensions 0.6×0.8×0.1 mm^3 was harvested in a period of 10 days. The photograph of the as grown single crystal is shown in figure 1 and growth conditions are given in table 1.

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RESULTS & DISCUSSIONS

Single crystal X-Ray diffraction analysis

Single crystal BGHB was subjected to single-crystal X-ray diffraction using a Brukers axe diffractometer. The observed cell dimensions of the grown crystal are a= 12.23A°, b = 4.78A° and c = 5.28 A° with unit cell volume= 308A°^{3} and α=γ= 90° and β=308°. The single crystal X-ray diffraction analysis reveals that the compound crystallizes in the monoclinic P system [8].

Powder X-Ray diffraction analysis

Powder X-ray diffraction pattern was recorded for VGHB crystal using Bruker D8 advance diffractometer with Cukα (λ= 1.5406 A^0) radiation. The sample was scanned in the range 50 to 700 at a scan rate of 10 / min^{-1}. From the X-ray diffraction data the various planes of reflections were indexed using the software XPERT-PRO. When X-rays interact with a crystalline substance, diffraction pattern is obtained. The techniques based on observing the scattered intensity when X-ray beam is incident on a sample. It is a function of incident and scattered angle, polarization and wavelength or energy. Indexing is the process of determining the unit cell dimensions from the peak positions. It is the first step in diffraction pattern analysis. The indexed pattern of the grown crystal is shown in figure 2. The peaks observed from the powder XRD spectrum are given in Table 2. From the powder X-ray diffraction pattern, the presence of prominent Bragg's peak 2θ angle confirms the perfect crystalline structure.

FTIR Spectral analysis

Identify the elements and the functional groups present in the grown crystal qualitatively, the FTIR spectra were obtained using Perkin-Elmer spectrometer with KBR beam splitter and global source. The intense sharp peaks in this band at 3105cm^{-1} may be assigned to N–H stretching vibrations. 2887 cm^{-1} may be assigned to NH_3^+ stretching band[7]. The peaks 2601 cm^{-1} are attributed to the C-H stretching mode vibration[9]. The symmetric and asymmetric stretching modes of COO- are seen at 1599, 1496, 1437 and 1393 cm^{-1}[8]. The intense peaks at 1154 and 1125 cm^{-1} indicate the C= O stretching of the COO- group[9]. The O–H out of plane deformation peaks observed at 980 and 926 cm^{-1}, C –C stretching seen at 890cm^{-1}.The peak at 685 cm^{-1} is due to N–H out-of-plane bending vibrations were also observed[8]. The torsion oscillation of NH_3^+ is revealed by the intense sharp peak at 503 cm^{-1} and 557cm^{-1}[9].

Optical analysis

To determine the transmission range and hence to know the suitability of VGHB single crystals for optical applications, the UV–vis spectrum was recorded in the range of 190 to 1100 nm using Shimadzu UV 1600 photo spectrometer. The lower cut off of the VGHB crystal is at 380 nm, 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 to 1000nm enables the use of this material for second harmonic generation (SHG) applications[8].

Micro hardness study

The mechanical strength of the grown crystal was studied using LEITZ WETZLER Vickers pyramidal indenter. Micro hardness measurement is commonly used to determine the mechanical strength of the material which is related to bond strength and defect structure. Optically clear and defect free crystal plate taken perpendicular to the growth direction was subjected to indentation tests at room temperature. The diagonal length of the indentation (d) in μm for various applied load (P) in g was measured for a constant indentation period of 15 s.

Where H_v is Vickers hardness number, P is the indenter load in kg and d is the diagonal length of the impression in mm. According to the indentation size effect (ISE), micro hardness of crystals decreases with increasing load and in reverse indentation size effect (RISE) hardness increases with increasing load. In our case, H_v increases with load up to 100 g and becomes load independent for P≥100 g.

In figure 5(a), the load can be increases and also the hardness also increases. In figure 5(b), the plot of straight line gives the Meyer's index value "n" can be calculated which is above 2. It confirms the soft nature of the material.

Antimicrobial activity

Bacterial cultures such as, Staphylococcus aureus, E.coli, Candida albicans and A. niger were obtained from Eumic analytical Lab.

PREPARATION OF CULTURE MEDIA

NUTRIENT AGAR MEDIUM

Nutrient agar medium is one of the most commonly used medium for several routine bacteriological purposes:

Ingredients		Grams/Litre
Peptone	:	5gm
Beef extract	:	3gm
Agar	:	15gm
Sodium chloride	:	5gm
Yeast extract	:	1.5gm
pH	:	7.0

After adding all the ingredients into the distilled water it is boiled to dissolve the medium completely and sterilized by autoclaving at 15 Ib psi pressure (121˚C)for 15 minutes.

The nutrient agar medium was prepared and sterilized by autoclaving at 121˚C 15 lbs pressure for 15 minutes then aseptically poured the medium into the sterile petriplates and allowed to solidify the Bacterial broth culture was swabbed on each petriplates using a sterile buds. Then wells were made by well cutter. The organic solvent extracts of leaves were added to each well aseptically.

This procedure was repeated for each Petri plates then the petriplates were incubated at 37˚C for 24 hrs. After incubation the plates were observed for the zone of inhibition.

CONCLUSION

Single crystals of Violet dye doped Glycine Hydro Bromide (VGHB) has been successfully grown by the slow evaporation technique from the aqueous solution. Good quality transparent single crystal of VGHB with well dimension is obtained. The single crystal X-ray diffraction studies confirm the monoclinic structure of the grown crystal. FTIR confirms the presence of all functional groups in the grown crystal. UV-Vis absorption study confirms low cutoff at 380 nm wavelength. The grown crystal is transparent in UV, entire visible and IR region. From the mechanical measurements, Vickers hardness number H_v increases with increase in load and it belongs to soft material category as Meyer's index number 'n' is greater than 2. The stiffness constant C_{11} is quite high, revealing that the binding forces between the ions are quite strong. This confirms the higher stability of the crystal. The antimicrobial activity can be measured the VGHB crystal which indicates that the E.coli is more effective compared with other organism. From all the above discussion, the VGHB crystal is a prominent material for the opto-electronic application and can be used in EO modulators.

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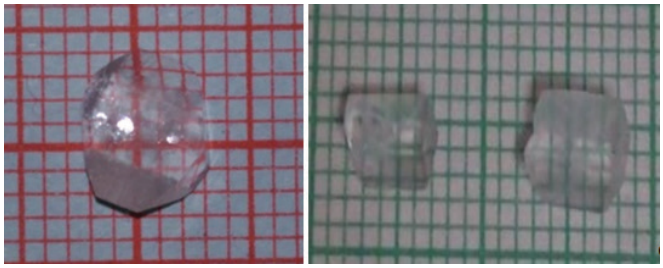

Figure 1: Photograph of theas grown pure and violet dye doped Glycine Hydro Bromide crystals.

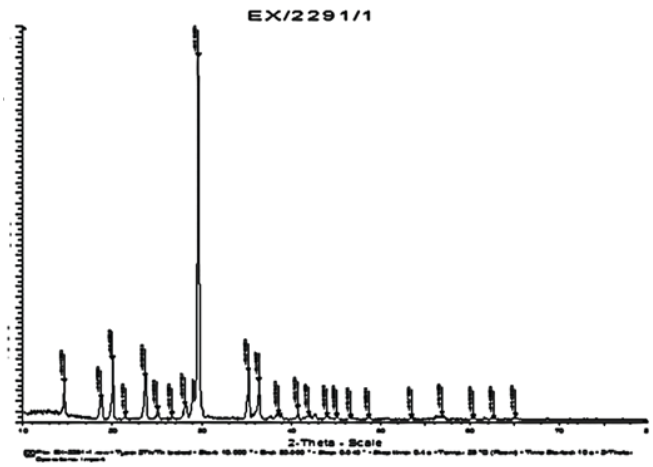

Figure 2: XRD patterns of VGHB crystal.

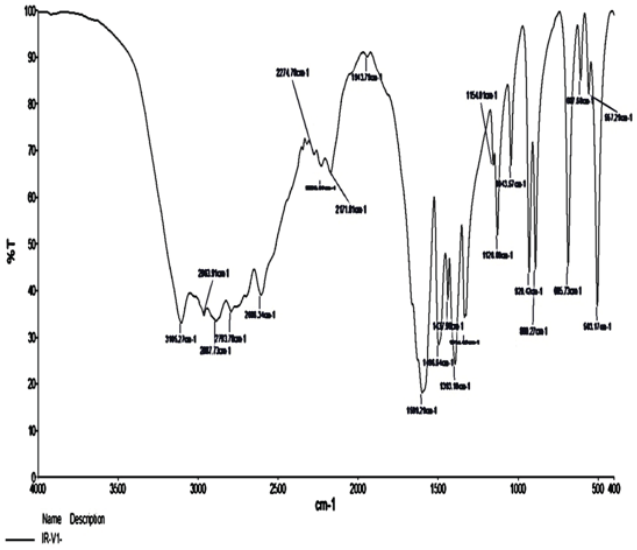

Figure 3: FTIR spectrum of VGHB.

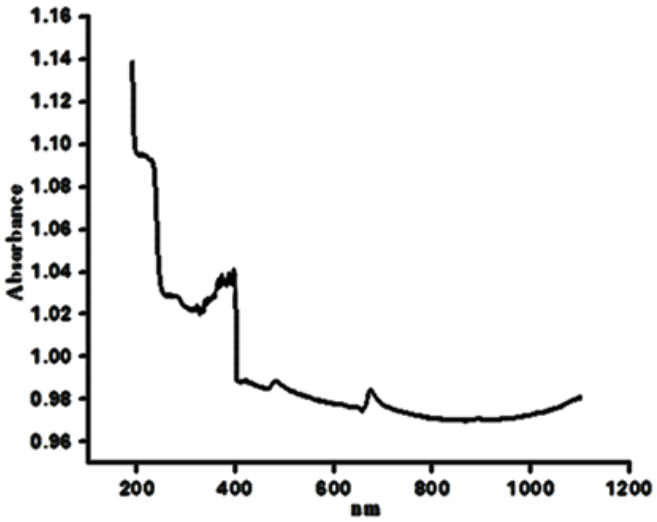

Figure 4: Optical absorbance spectrum of VGHB.

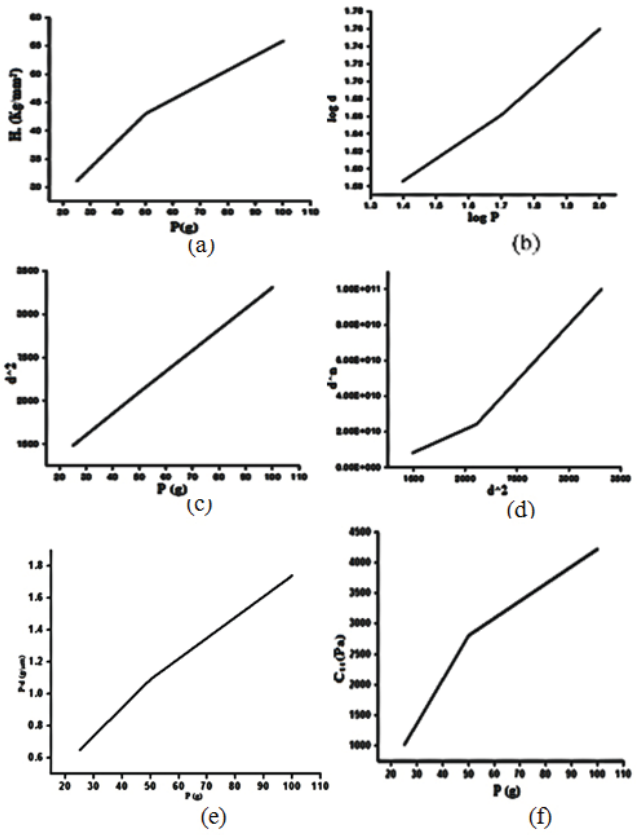

Figure 5: (a) P Vs. H_v

(b) log P Vs. log d

(c) P Vs. d^2

(d) d^2 Vs. d^n

(e) P Vs. p/d

(f) P Vs.Stiffness constant

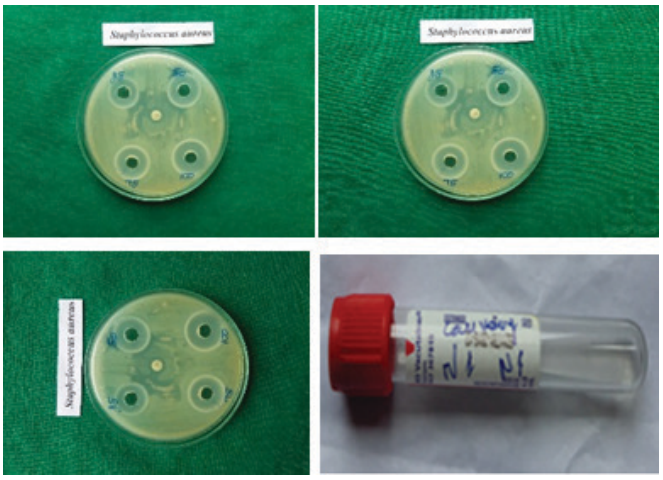

Figure 6: Photograph of Staphylococcus aureus of VGHB crystal.

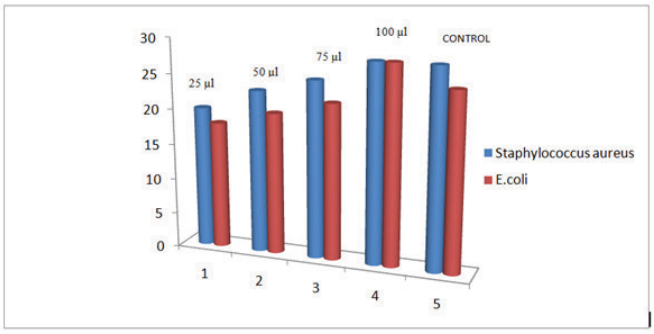

Figure 7: Comparision of Staphylococcus aureus and E.coli

Table 1: Growth conditions of VGHB crystal

Solute	Violet dye doped glycine with hydro bromide acid
Solvent	Double distilled water
Method	Slow evaporation method
Growth period	10 days
Purification	Recrystallization
Size of the crystal	0.6×0.8×0.1 mm^3

Table 2: X-Ray Diffraction of VGHB crystal

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
14.7114	21587.3	0.1476	6.02157	15.44
18.9070	416.63	0.1476	4.69376	0.30
20.0694	935.04	0.1476	4.42446	0.67
21.0625	156.56	0.1476	4.21804	0.11
23.8792	913.62	0.1476	3.72649	0.65
24.6229	157.43	0.1476	3.61559	0.11
26.8119	454.06	0.1476	3.32518	0.32
28.7567	688.56	0.1968	3.10456	0.49
29.1615	2079.59	0.1476	3.06238	1.49
29.7686	139801	0.1476	3.00130	100.00
32.1141	371.55	0.1476	2.78725	0.27
35.3500	243.96	0.1968	2.53917	0.17

Table 3: Vibrational assignments of FTIR of VGHB crystal

Present work	Standard value of parent compound	Vibrational assignments
3105	3100	O–H and NH_3+ stretching bands
2887	2897	NH_3+ stretching band
2601	2601	C-H stretching
1599, 1496, 1437, 1393	1576, 1497, 1447, 1446, 1335, 1386	NH_3+ or COO- groups or COOH group or C–O stretch of -COOH
1154,1125	1125	C= O stretching
980,926	980,915,926	-CH_2 rock or O–H out of plane
890	887,874,890	C –C stretching
685	671	N–H out-of-plane
503,557	504	NH_3+ Oscillations

Table 4: Theoretical Vicker's Hardness number and Stiffness constant of VGHB crystal

Load (g)	H_v (Kg/mm^2) (Exp.)	(Th.)	d^2 (mm)	$C_{11} \times 10^{14}$ (Pa)
25	31.15	31.19	1486	1.017
50	43.05	44.04	2104	2.811
100	55.9	55.98	3312	4.224