Structural, Optical, Mechanical studies on Glycine Lithium Sulphate (GLS) single crystal

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Abstract: Glycine Lithium Sulphate (GLS) is one of the potential materials for non-linear optical property applications. Single crystals of glycine lithium sulphate (GLS) with very high degree of transparency were grown from aqueous solution by slow evaporation technique. Single crystal X-ray diffraction analysis reveals that the crystal belongs to orthorhombic system with the space group Pna2₁. The presence of functional groups was identified from FTIR spectrum. The optical absorption study depicts the transparency of the crystal in the entire visible region and the cut off wavelength has been found to be 300 nm. The microhardness studies were also reported for grown crystals.

1. Introduction

The search for new organo metallic complexes possessing high optical non linearity is an important task because of their practical applications in harmonic generation, switching and other optical signal processing devices. [1-3]. Glycine is a simplest amino acid and it forms number of complexes with various inorganic salts [4]. Organic and non organic materials with aminoacid group were combined for the production of excellent material and considerable efforts were taken by material researchers to achieve it. Over the decades, several researchers were extensively investigating on amino acid family of crystals due to their non-linear optical properties [5]. Some complexes of glycine with HCl [6], lithium sulfate [7], LiNO₃ [8], sodium nitrate [9] form single crystals but some of these are reported to have NLO property and some are reported not having NLO property. In the present exploration, slow evaporation method is utilized and good quality glycine lithium sulphate crystal is harvested. The obtained crystal was further characterized using XRD, FTIR, UV, Microhardness studies to understand the various properties.

2. Material Synthesis

Single crystal of glycine lithium sulphate (GLS) was grown by slow evaporation solution growth technique at room temperature. Glycine lithium sulphate is synthesized by dissipating glycine and lithium sulphate in the molar ratio 1:1 in double distilled water. The complete volume of solution was continuously stirred vigorously by using magnetic stirrer for 6 hours to yield a homogeneous mixture of solution. Then the solution was filtered and allowed to evaporate at room temperature. The good transparent defect free crystal with dimension 13 x 8.5 x 5.7 mm³ was harvested in a period of 3 weeks. The reaction is given below

\[ C_2H_5NO_2 + Li_2SO_4 \rightarrow [Li_2(SO_4)(C_2H_3NO_2)] \]  \hspace{1cm} (1)

The grown crystal is shown in figure 1.

Fig 1: Photograph of GLS single crystal
3. Results and Discussion

3.1. Single crystal X-ray analysis

Single crystal X-ray diffraction analysis for the GLS crystals was carried out to identify the cell parameters using ENRAF NONIUS CAD-4 automatic X-ray diffractometer. The X-ray diffraction study reveals that the crystal belongs to orthorhombic system with space group Pna21 and the lattice parameters are $a = 4.9704$ Å, $b = 7.874$ Å, $c = 16.2873$ Å, $\alpha = \beta = 90^\circ$ and $\gamma = 90^\circ$ and $V = 615.9$ Å$^3$ [10].

3.2. FTIR Spectral Study

The FTIR analysis of GLS was recorded in KBR phase in the frequency region 450-4000 cm$^{-1}$ using PERKIN ELMER SPECTROMETER. The FT-IR spectral analysis was shown in Figure 2. In the range of 2954-3451 cm$^{-1}$ the broad and strong intense peak occurs due to hydrogen bond interactions involving NH$_3^+$ stretching vibrations. The sharp absorption bands at 1339 cm$^{-1}$ and 1639 cm$^{-1}$ correspond to the asymmetric S = O stretching vibrations and C = O asymmetric vibrations respectively. The oxygen atom of the carbonyl and sulphate group therefore acts as hydrogen bond acceptor. The Li-N stretching vibration is observed at 505 cm$^{-1}$ and the absorption band at 1445 cm$^{-1}$ corresponds to CH$_2$ stretching vibration. The stretching vibration due to SO$_4$ group appeared at 906 and 703 [11-12]. The absorption band at 551 cm$^{-1}$ is assigned to the out of plane bending vibration of carboxylate group. The characteristic absorption band at 1042 cm$^{-1}$ is attributed to C-N Stretching vibration.

![Fig 2: FT-IR spectrum of GLS crystal](image)

Uv-Vis spectroscopy

Optical absorption spectrum was recorded for grown Glycine lithium sulphate single crystal samples using SHIMADZU MODEL UV 1650 PC Spectrophotometer in the wavelength range 200 - 1200 nm. Figure 3 shows excellent optical transparency of the material. The UV cut off wavelength for the grown crystals were found to be 300 nm. This makes it a potential material for optical device fabrication [10].

![Fig 3: UV Visible spectrum of GLS](image)
Microhardness study

Vicker’s microhardness indentations were made on the grown GLS crystal with room temperature by varying the load range from 25 g to 100 g using Vicker’s microhardness tester. Three trials were made for each indentation. Vicker’s diamond pyramidal number $H_v$ is calculated using the equation

$$H_v = 1.8544 \frac{P}{d^2}$$  \hspace{1cm} (2)

where $P$ is the applied load in g, $d$ is the diagonal length in mm and $H_v$ in kg/mm$^2$. The variation of $H_v$ with the applied load of TGZC crystal as shown in Fig 4. A plot of log $P$ vs log $d$ as shown in Fig 5. The plot of log $d$ vs log $P$ yields a straight line and the slope gives work hardening coefficient ‘n’. The value of $n$ is 3.75 and it is more than 2 so it is categorized as soft material [13-14].

![Fig 4: Variation of Load P Vs Hardness](image1)

![Fig 5: Variation of log d Vs log P](image2)

The elastic stiffness constant ($C_{11}$) is calculated using Wooster’s empirical relation $C_{11} = (H_v)^{7/4}$ [15]. The high value of $C_{11}$ indicates the binding forces between the ions are quite strong. The stiffness constant increases with increase of load as shown in Figure 6.

![Fig 6: Stiffness constant vs Load P](image3)

4. Conclusion

Glycine lithium sulphate (GLS) has been grown by slow evaporation technique and have achieved good quality crystal. The XRD data for the grown crystal reveals that the GLS crystal belongs to orthorhombic system with space group Pna21. The presence of functional group is confirmed by FTIR spectral analysis. The microhardness study shows that the GLS crystal belongs to soft material category. UV –Vis spectrum shows that it is suitable for NLO applications and the lower cutoff wavelength is around 300nm.

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References

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