

Mechanical Properties of L-Lysine Doped Triglycine Sulfate (TGS) Single Crystals

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Abstract: Single Crystals of L-Lysine doped Triglycine Sulfate (TGS) were grown by conventional method. The grown crystals were characterized by single crystal XRD, Powder XRD, FTIR, UV-Vis Spectroscopy and Microhardness. The single crystal and powder XRD was to study the morphology and structure of the grown crystal. The presence of functional group of the materials was qualitatively estimated by FTIR. The optical nature of the crystal was studied by UV-Vis spectroscopy. Mechanical properties were determined by Microhardness studies. The results were discussed.

Keywords: TGS, conventional method, optical transmittance, mechanical properties

1. Introduction

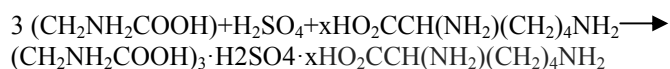
Triglycine Single Crystals are well known for ferroelectric material. It many potential applications in the field of optoelectronics especially in Infrared detectors, optical transmitters, etc. It has second order phase transition at Curie temperature (49°C). Ferroelectric materials are polar and possess spontaneous polarization below Curie temperature.

TGS has some disadvantages such that it is depolarized by thermal, mechanical and electrical means. Several dopants were added to reduce these types of difficulty to increase the property of the crystal for practical applications. It undergoes the second order phase transition at Curie temperature from ferroelectric to paraelectric [1]. It belongs to the monoclinic system. Many authors have investigated the effect of doping various amino acids like Alanine, Valine, Sacrosine, Tyrosine, etc., on TGS [2, 3]. The space group transform from non-centrosymmetric P_{21} in the ferroelectric phase to centrosymmetric $P_{21/m}$ in the paraelectric phase [1].

In this present work, we report the repercussion of L-Lysine in Triglycine Sulfate (TGS) single crystals. The method used for the growth of L-Lysine doped TGS was Conventional method which can also be called as slow evaporation solution growth method. The structural studies were made on the grown crystal by single crystal XRD. The powder XRD confirms the crystallinity of the grown crystal. The presence of functional groups confirm by Fourier Transform Infrared Spectroscopy. The mechanical property was estimated by microhardness studies. The optical transmittance was determined by UV-Vis spectroscopy.

2. Experimental work

AR grade of Glycine and sulfuric acid were taken for the preparation of the solution 3:1 molar ratio respectively and dissolved in 100ml of de-ionized water. L-Lysine of 8mol% was taken as dopant and added with the saturated solution of TGS. The following reaction taken place:



The prepared solution was stirred from 8-12 hrs to attain homogeneous solution and it was maintained at 50 °C to avoid oxidation throughout the preparation of the solution.

Then, it was filtered by Whatmann filter paper to reduce the impurity of the solution in undisturbed condition by controlled evaporation. After 2 weeks, nucleation was taken place and the seed crystals were formed. The transparent good quality crystals were select to grow bulk single crystals for optical applications. After 20days later, bulk single crystals were harvest. The grown crystals were shown in fig.1

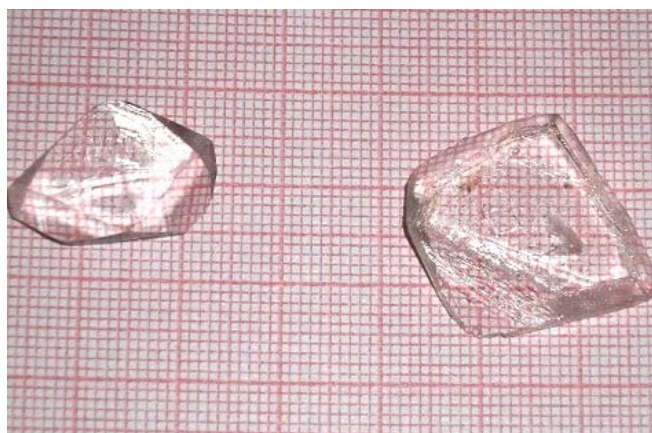


Figure 1

3. Characterization

3.1 Single Crystal X-ray Diffraction

The Single crystal XRD analysis was taken in the Bruker Single crystal X-ray diffractometer with Mo $K\alpha$ radiation to identify crystal structure and lattice parameters of the grown crystals.

3.2 Powder X-ray Diffraction

The powder XRD pattern was recorded by the Reich Scieft Instrument. The scan time was set as 2min and the angle range from 10°-70°. The recorded pattern is used to analysis the nature of the crystal.

3.3 Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy analysis is used to find the functional groups of the grown crystal. It was taken perkinelmer lambda 35 equipment.

3.4 Optical Studies

The transmittance of the crystal has been observed by perkelmer lambda 35 UV-VIS-NIR spectrophotometer. The spectrum recorded from 100-1200 nm region.

3.5 Microhardness

Vickers hardness was taken to measure the mechanical strength and the resistance at which the localized deformation takes place of the crystal. It was carried out by Leitz-Wetzler hardness tester. Vickers diamond pyramidal indenter was used for indentation and it was attached with Leitz indent light microscope for the analysis.

4. Results and Discussion

4.1 Single Crystal XRD

Single crystal XRD reveals that the grown crystal belongs to monoclinic crystal system. Lattice parameters of L-Lysine doped TGS were tabulated in table.1 It was compared with pure TGS single crystals which shows the slight changes by the dopant of L-Lysine. It reveals that the crystal belongs to P_{21} space group. The unit cell volume has been calculated as $v=360.84$ by the formula for monoclinic system

$$v = abc \sin(\beta)$$

Table.1

Sample	a(Å)	b(Å)	c(Å)	β (°)
Pure TGS[6]	9.417	12.640	5.735	110.23
L-Ly TGS	9.148	12.247	5.829	107.39

4.2 Powder XRD

The diffraction pattern of the grown crystal shown fig.2. The sharp peaks show the good crystallinity of the grown crystal. The grain size was calculated by the following Scherrer's formula

$$t = \frac{K * \lambda}{B * \cos \theta_B} \quad (1)$$

Where t is the crystallite size of the crystal, K is the constant depend on crystalline shape (0.89), λ is the wavelength of X-ray, B is the Full Width Half Max. and θ_B is the Bragg angle. The grain size was calculated as 87.77nm.

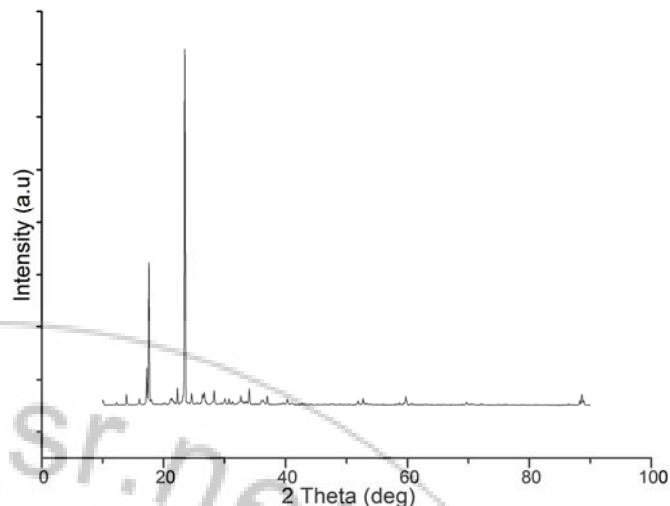


Figure 2

4.3 Fourier Transform Infrared Spectroscopy

The spectrum of the grown crystals was shown in figure.2. It was scanned from 400 – 4000nm. The presence of amine group was found out by NH_2 stretching at 3171.20 cm^{-1} . The peaks of CH_3 stretching vibrations were obtained at 2893.94 cm^{-1} , 1867.74 cm^{-1} and 1711.16 cm^{-1} . NH_2 peak was observed at 1623.54 cm^{-1} . The peak position of 1503 cm^{-1} reveals the deformation and stretching of the grown crystals. CH_3 deformation was identified at 1424 cm^{-1} . A plane bending of C-H was at 1128.86 cm^{-1} . The presence of NH_2 rocking was observed at 1019 cm^{-1} . The peak at 901.54 cm^{-1} represents the wagging. The presence of C-H in plane was identified at 859.61 cm^{-1} . The C-S stretching peaks are attributed at 616.51 cm^{-1} and 570.45 cm^{-1} . The inclusion of CCC out of plane bending was obtained at 499.71 cm^{-1} .

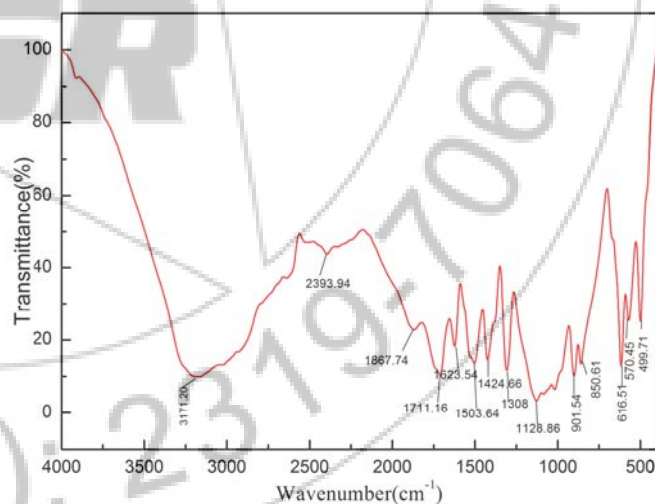


Figure 3

4.4 UV-Vis Spectroscopy

The optical transmission and absorption spectra were shown in figure.4 and figure.5. The spectrum was observed in the entire visible and IR region. The cut off wavelength was found to be at 463nm. The transmittance was found at 79.1%. The energy band gap was calculated as 2.68eV which was found by the following relation

$$E_g = hc/\lambda$$

Where h is the Planck's constant, C is the speed of light and λ is the cut off wavelength. The absorption spectrum reveals the absorbance of the grown crystal observed from 200nm and remains same till 1100nm.

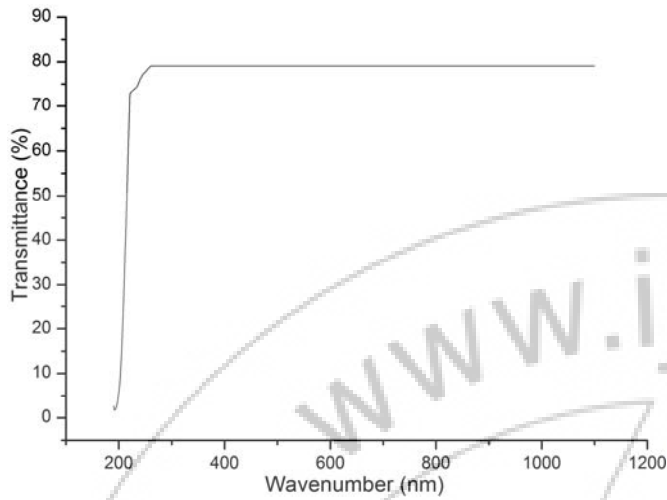


Figure 4

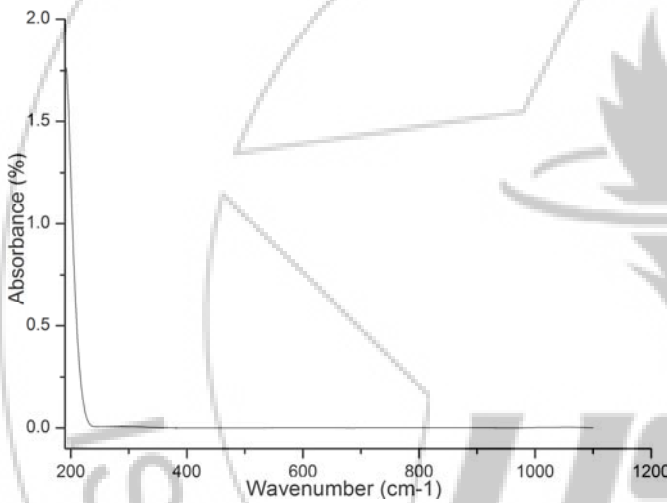


Figure 5

4.5 Microhardness

For different applied loads (P) from 5-100g, the hardness values (H_v) was determined using the relation

$$H_v = 1.8544 P / d^2 \quad (2)$$

where P is the applied load in gram and d is the diagonal length in mm. It was shown in figure.6. It indicates that the hardness value increases with increase in applied load which depict the mechanical strength is good for the grown crystal.

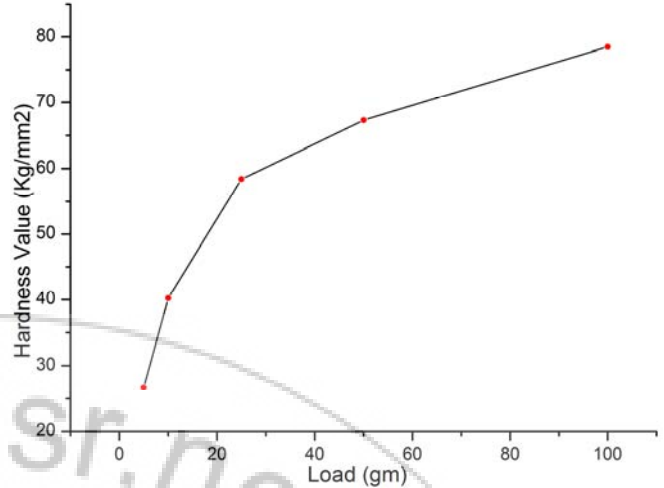


Figure 6

The stiffness constant was calculated for different applied load and hardness values by the Wooster's empirical relation

$$C_{11} = (H_v)^{7/4} \quad (3)$$

The graph was drawn between the hardness value (H_v) and stiffness constant (C_{11}) and shown in figure.7 which indicate as the hardness increases the stiffness constant also increases.

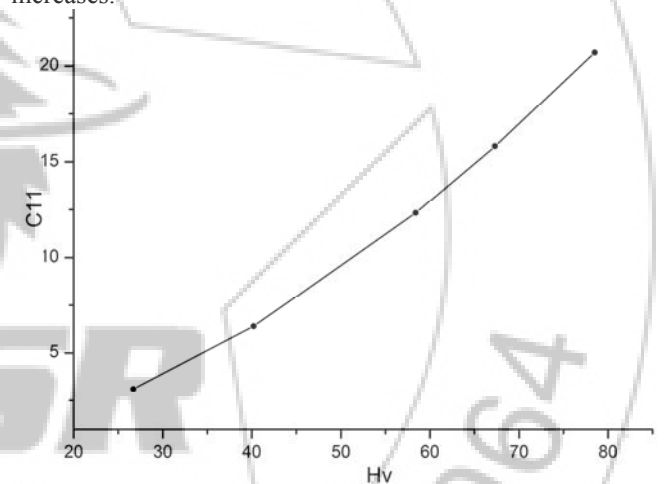


Figure 7

Meyer's law describes the empirical relation between the indentation size and applied load. The Meyer's law represented by the following equation

$$P = kd^n \quad (4)$$

Can also be written as

$$\log P = \log k + n \log d \quad (4)$$

Where P is the applied load in gram, k is the material constant. n is the Meyer's Index which can be determined from the graph plotted between $\log P$ and $\log d$. The graph between $\log P$ and $\log d$ was shown in figure.8.

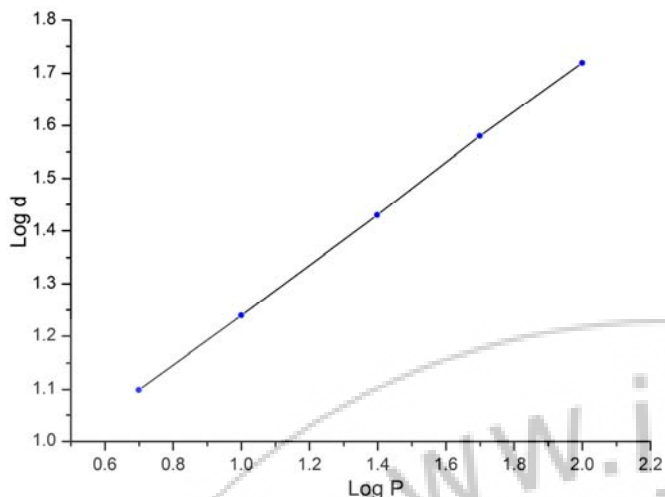


Figure 8

The Meyer's index of the grown crystal was calculated as 2.258 which indicate the grown crystal belongs to the soft category. The value of 'n' is above 1.6 means; it belongs to the soft materials. If the value of 'n' is below 1.6 means, it belongs to the hard materials. Hence, the grown single crystals can be used for the application in the magnetic field like sensors, detectors etc.

5. Conclusion

L-Lysine (8mol%) doped Triglycine Sulfate (TGS) single crystals were grown by conventional method. The lattice parameters were slightly changed by the inclusion of the doping material which was estimated by Single crystal and Powder X-ray diffraction. The functional groups were analyzed qualitatively by Fourier Transform Infrared (FTIR) Spectroscopy. The optical transmittance and absorption were observed by UV-Vis spectroscopy. The energy band gap was calculated as 2.68eV which is suitable for optoelectronic applications. The mechanical properties were analyzed by Micro hardness studies. From hardness value, the Meyer's index was calculated as 2.258 which depicts the grown crystals belongs to soft material and can be used for the applications of sensors, detectors etc.

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