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Growth and Characterization of Triglycine Sulphate(TGS) Single Crystals

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ABSTRACT

Triglycine Sulphate (TGS) is an important material used in the fabrication of high sensitivity infrared detectors at room temperature. Single crystals of TGS were grown by solution method with slow evaporation and water as solvent at room temperature. The grown crystals were characterized by XRD analysis. It is found to exhibit monoclinic structure with the space group $P2_1$. The chemical composition of triglycine sulphate crystal was confirmed by Energy Dispersive X-ray Analysis (EDAX). The Refractive index was calculated as 1.534 by using Brewster's angle method. UV-Visible spectra showed that the grown crystals have wide optical transparency in the entire visible region.

Introduction

Triglycine sulphate (TGS) is a ferroelectric crystals with chemical formula $(NH_2CH_2COOH)_3$. (H_2SO_4) has found many applications in ferroelectric detection on a large spectral bandwidth. The ferroelectric crystals find important applications in optoelectronics such as capacitors, non-volatile memory devices[1], actuators, high-performance gate insulators, etc. All ferroelectric materials are pyroelectric, however, not all pyroelectric materials are ferroelectric. Below Curie temperature, ferroelectric and pyroelectric materials are polar and possess a spontaneous Polarization or electric dipole moment [2-5]. However, this polarity can be reoriented or reversed fully or partially through the application of an electric field with ferroelectric materials.

The important phase for device fabrication is the (010) face which is perpendicular to the ferroelectric axis of the TGS crystal therefore, it is essential to know crystal orientation and habit. Hydrogen bonded ferroelectric crystals form a subclass of ferroelectrics in which hydrogen bonds plays an important role in determining the properties. Non linear optical (NLO) organic materials plays a vital role for optical second harmonic generation (SHG) due to their applications in the domain of optoelectronics and photonics.

Amino acids are strong candidates for optical second harmonic generation (SHG) because they contain chiral carbon atom and crystallizes in non-centro symmetric space groups. Among twenty amino acids glycine is in centro symmetric space group, and hence to find the NLO property, pure glycine with sulphuric acid, hydrochloric acid and nitric acid combinations has been taken. This paper defines the crystal structure of triglycine sulfate (TGS) and the grown crystal has been investigated by the XRD, EDAX, Refractive index and UV-Visible spectrum.

Experimental

A. Material Synthesis

Commercially purchased Analar Reagent (AR) grade glycine and concentrated sulphuric acid (H_2SO_4) were used for synthesis and growth. Glycine and H_2SO_4 in the molar ratio of 3: 1 were dissolved in de-ionized water. The solution is filtered and transferred to a Petri dish for crystallization. Fully transparent and good quality crystals were obtained in a period of 30 days. The purity of the synthesized salts of TGS was improved by successive re-crystallization.

The chemical reaction for obtaining TGS salt is as follows:

$3(NH_2CH_2COOH) + H_2SO_4 \rightarrow NH_2CH_2COOH)_3.(H_2SO_4)$

B. Determination of Solubility

The re-crystallized salt of TGS was added to 50 ml of de-ionized water in an air tight container for the measurement of solubility in the temperature range 30-50°C. Solubility study was carried out using a hot plate magnetic stirrer and a digital thermometer (accuracy is ±0.1° C). The temperature was controlled using a voltage regulator attached to the magnetic stirrer. Initially, the solution was kept at 30 °C and stirred continuously for about 2 hours. After attaining the saturation, the solubility was determined gravimetrically [6]. The same procedure was followed for other temperatures of TGS salt. The solubility curve for TGS salt is shown in fig.1.

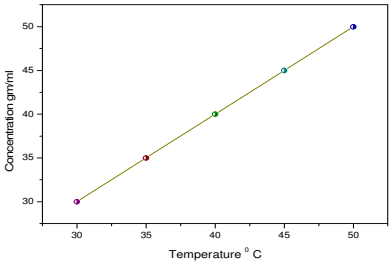

Fig 1 Solubility curve of TGS crystal

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C. Growth Of TGS

TGS single crystals were grown using slow solvent evaporation technique. Glycine and H_2SO_4 is in the molar ratio of 3: 1 were dissolved in de-ionized water. The supersaturated solution was prepared according to the solubility data. The solution is filtered and transferred to a Petri dish for crystallization. Fully transparent and good quality crystals were obtained within 30 days. The purity of the synthesized salts of TGS was improved by successive re-crystallization. The grown TGS is as shown in figure 2.

Fig.2 Photograph of as Grown TGS crystal

Characterization

A. X-ray Diffraction

The grown crystals have been characterized by powder X-ray diffractometer. Fig.3 represents the powder X-ray pattern of the grown TGS. The system is found to exhibit monoclinic structure with the space group $P2_1$ and the lattice parameters of pure TGS crystal are a = 9.601 Å, b = 12.560 Å and c = 5.450 Å which are in good agreement with the literature [7].

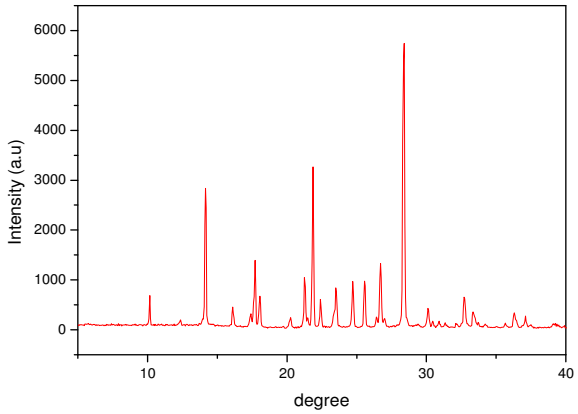

Fig.3 Powder XRD Pattern of TGS

Energy Dispersive X-Ray Analysis (EDAX)

The EDAX has been performed to identify the elements present in the crystals. The EDAX spectrum of pure TGS is shown in Figure 4. The atomic and weight percentages of the elements are shown in Table 1.

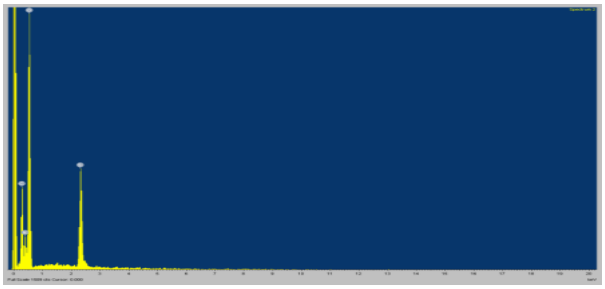

Fig.4 EDAX Spectra of pure TGS crystal

Table 1

Data for atomic and weight percentage of the elements

TRI GLYCINE SULPHATE

ELEMENT	ATOMIC %	WEIGHT %
C	25.51	20.43
N	17.02	15.90
O	55.23	58.90
S	02.24	04.78
TOTAL	100	100

Refractive Index Measurement

The refractive index of the TGS crystal was determined by Brewster's angle method using He-Ne laser of wavelength 632.8 nm. A polished flattened single crystal of TGS was mounted on a rotating mount at an angle varied from 0 to 90 degrees. The angular reading on the rotary stage was observed, when the crystal is perfectly perpendicular to the intra-cavity beam. The crystal was rotated until the laser oscillates and the angle has been set for maximum power output. Brewster's angle (θ_p) for TGS is measured to be 56.9 ± 0.5 degree. The refractive index has been calculated using the equation $n = \tan \theta_p$; where θ_p is the polarizing angle and it is found to be 1.534 which is good agreement with the literature [8].

UV-Visible Spectroscopy

Good optical transmittance and lower cut off wavelength are very essential properties for nonlinear optical (NLO) crystals [9]. The transmission spectrum of pure TGS is shown in Figure 5. The optical spectra of TGS were recorded in the range 200-800 nm using Spectrophotometer. It is seen from that absorption spectrum the crystal is transparent in the range 295-800 nm without any absorption peak, which is an important parameter of NLO crystals.

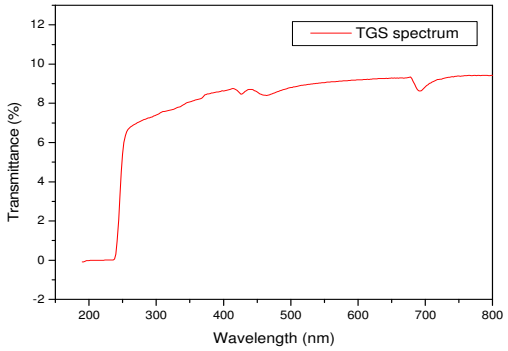

Fig.6 UV-Vis Spectra of TGS

Conclusion

Transparent single crystals of TGS have been grown successfully using slow solvent evaporation technique. X-ray analysis reveals that TGS crystal belongs to monoclinic structure with space group $P2_1$. The chemical composition of triglycine sulphate was confirmed by Energy Dispersive X-ray Analysis (EDAX). The refractive index was calculated as 1.534 by using Brewster's angle method. A UV-Visible spectrum shows that the grown crystals have wide optical transparency in the entire visible region.

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