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Growth and characterization of glycine sodium nitrate (GSN) single crystal

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Good optical quality single crystals of glycine sodium nitrate (GSN) were grown by slow evaporation technique from its aqueous solution at room temperature. Single crystal X-ray diffraction (XRD) studies revealed the presence of monoclinic structure with space group Cc, and Fourier transform infrared transmission (FTIR) has confirmed the presence of the functional groups present in the single crystal system. The thermal stability of the crystal was studied by Thermogravimetric/Differential Thermal Analyzer (TG/DTA). Ultraviolet (UV) spectrum showed no significant absorption in the region 200-2000 nm suggesting the optical transparency of the grown crystals.

Key words: Single crystal, solution growth, X-ray diffraction (XRD), thermogravimetric/differential thermal analyzer (TG/DTA).

INTRODUCTION

Among the organic materials, amino acids constitute a family in which glycine is the simplest of all the amino acids. It has been reported that some complexes of amino acids with simple inorganic salts may exhibit ferroelectric properties (Pepinsky et al., 1975, 1958; Deepthy et al., 2001). Hoshino et al. (1959), reported about the dielectric properties of triglycine fluroberyllate. Some complexes of glycine with H_2SO_4 (Hoshino et al., 1957), CaCl₂ (Natarajan et al., 1984), CaNO₃ (Natarajan et al., 1984) BaCl₂ (Narayanan et al., 1975), SrCl₂ (Ravikumar et al., 1985), COBr₂ (Baran et al., 2003) and LiNO₃ (Subha et al., 2001) form single crystals but none of these are reported to have nonlinear optical property. Glycine sodium nitrate is one of the complexes of glycine and it possesses both Non-linear Optical (NLO) and ferroelectric properties. This material can find useful applications in the field of opto-electronics. This material

is characterized in order to find the suitability of the required applications. Hence, in the present investigation, the growth aspects of the Glycine Sodium Nitrate (GSN) crystal have been carried out using solvent evaporation technique. The grown crystal is subjected to different characterizations such as single X-ray diffraction (XRD), Fourier transform infrared transmission (FTIR), Ultraviolet (UV), and thermogravimetric Analyzers /Differential Thermal Analyzer (TGA/DTA) studies were carried out for the grown crystals.

EXPERIMENTAL PROCEDURE

Colourless single crystals of GSN were grown, from aqueous solution by slow evaporation technique. The starting materials were analytical grade reagents glycine and sodium nitrate. The solution was prepared by dissolving equimolar amounts of γ -glycine and sodium nitrate in deionized water by slow evaporation at ambient temperature. The reaction taking place is given as follows:

$$C_2H_5NO_2 + Na (NO_3) \rightarrow Na (NO_3). C_2H_5NO_2$$
 (1)

The solution was allowed to evaporate slowly. Small transparent single crystals were obtained through spontaneous nucleation. Selecting macro defect free crystals as seeds, single crystals were grown to a dimension of 22x20x10 mm³ at ambient temperature as

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Abbreviations: FTIR, Fourier transform infrared transmission; **XRD,** X-ray diffraction; **GSN,** Glycine Sodium Nitrate; **TG/DTA,** Thermogravimetric/Differential Thermal Analyzer.



Figure 1. Grown single crystals of GSN.

shown in Figure 1, by slow evaporation technique. During the growth of GSN, several funguses like organism were formed in the solution. These organisms initially started on the surface and gradually after a few days, sinked into the solution, thereby contaminating it, and the growth of the crystals to larger dimension is prohibited. The growth of these microbes is prevented by adding a few drops of hydrogen peroxide (H_2O_2).

RESULTS AND DISCUSSION

Single crystal X-ray diffraction (XRD)

The single crystal XRD analysis for the grown crystals has been carried out to identify the lattice parameters. The calculated lattice parameters are; a=14.323 Å, b=5.2573 Å, c=9.1156 Å. The crystal belongs to monoclinic structure with space group Cc. The reported values are in agreement with the previous work (Suresh et al., 2010).

Fourier transform infrared transmission (FTIR) spectral analysis

The FTIR analysis of GSN crystal was carried out between 400 - 4000 cm⁻¹ using IFS BRUKKER 66v spectrometer and the resulting spectrum is shown in Figure 2. The spectrum shows that the glycine molecule exists as a dipolar ion in which the carboxyl group is present as a carboxylate ion and amino group exists as ammonium ions. The peaks at 1116, 1136 and 1507 cm⁻¹ are due to NH_3^+ group of glycine. The sharp peak at 829 cm⁻¹ corresponds to NO_3^- group which confirms the presence of nitrate in the grown crystal. The CH_2 group is found to be near 2890 cm⁻¹. The peaks at 507, 891, and 1615 cm⁻¹ are attributed to the carboxylate group of glycine molecule.

Thermal analysis

The thermo gravimetric analysis of GSN crystal was carried out on the sample weight of 34.120 mg between 50 to 1400 °C at a heating rate of 5 K/min in nitrogen atmosphere and the resulting thermo gram is shown in the Figure 3. There is a sharp weight loss at 250.4, 374.5, 526.6 and 1121.0 °C. There is no loss of water below 225°C, illustrating the absence of absorbed water in the crystal. There is a sharp weight loss at 225 °C, which is assigned as the melting point of the crystal. This indicates that crystal could be used for any optical application below its melting point. The sharpness of the thermogram reveals the purity of the crystal.

DRS – UV-visible spectral studies

The optical absorption spectrum of GSN crystal was recorded in the range 200 - 2000 nm using Varian Cary 5E spectrophotometer and the spectral result is depicted in Figure 4. As the crystal is colorless, there is a very low absorption in the entire visible region which is the most



Figure 2. FTIR spectrum of glycine sodium nitrate single crystal.



Figure 3. The TG-DTA curve of GSN crystal.

desirable property of the crystals used for NLO applications. The UV cut off frequency for the grown crystal was found to be at 310 nm.

Conclusion

Transparent single crystals of GSN have been grown



Figure 4. Optical absorption spectrum of GSN single crystal.

successfully using slow solvent evaporation technique. Xray analysis reveals that GSN crystallizes into the monoclinic system with space group Cc. UV-Visible absorption spectrum shows excellent transmission in the entire visible region. The FTIR spectrum shows that glycine molecule exists as a dipolar ion in which the carboxyl group is present as carboxylate ion and amino group is present as ammonium ion. TG and DTA thermograms reveal that the sample is highly stable up to 225°C.

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