Growth and Characterization of Glycine single crystal by addition of Aluminum Chloride

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Abstract

In this work, growth of single glycine crystal with the addition of aluminium chloride, using a slow evaporation process. In this study, glycine is doped into 0.8M% of aluminium chloride. The semi-organic single crystal glycine doped with aluminium chloride has been produced at a constant temperature of 36°C. Powder X-ray diffraction (XRD) is used for verifying the crystalline nature and alpha phase of doped glycine that contains aluminium chloride. The thermal stability and thermal decomposition of the sample was studied by thermo gravimetric/differential thermo analytical method. Using the UV-absorption spectrum, the optical transmittance window and lower cutoff wavelength have been examined.

Keywords: UV-VIS, FTIR, XRD, solution growth technique, single crystal.
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Introduction

Amino acids belong to family of organic materials. The molecular chirality, presence of week Van der walls, hydrogen bond and zwitterionic nature, due to this feature of amino acid shows NLO properties [1]. Applications for NLO single crystals are numerous and include frequency mixing, second harmonic generation, electro-optic modulation, optical data storage, and communication. For this use, numerous researchers have attempted to create a variety of new NLO materials in recent years.

Organic crystals have high second harmonic conversion efficiency but haven't mechanical and thermal stability. Hence, semi organic crystals have both properties of organic and inorganic [2]. Because the organic material contains inorganic impurities, it has mechanical and thermal strength, which makes it appropriate for use [3]. Glycine have gained more attention of researchers for their NLO properties.

Glycine (NH_2CH_2COOH) is also known as amino acetic acid. It's the most basic of all amino acids. With various inorganic salts, it can take on a variety of forms. Alpha, beta, and gamma glycine are its three polymorphic crystal forms. Glycine is a key component of the structure of proteins, enzymes, hormones and plays a role as a neurotransmitter.

It has no centre of chirality, making it the only amino acid that forms proteins [4]. M. Lenin examined the growth of a novel semi-organic crystal of glycine lithium chloride from a water solution that contained glycine and lithium chloride in a 1:1 ratio [5].

The semi organic crystal of diglycine sulphate and ammonium sulphate were studied by Drozdowski et al [6]. Michel studied the crystal structures of grown crystal glycine nickel dichloride and glycine zinc sulphate [7]. Shanmugavadivu reported a nonlinear optical crystal of glycine potassium sulphate [8].

Glycine single crystals were grown using solvent evaporation and submerged seed solution methods from glycine and ammonium sulphate taken in equimolar ratio [9].

In this work, synthesis of glycine single crystal doped with aluminium chloride with 0.8%M by slow evaporation technique first time and studied the various characterization and result were discussed.

Experimental

Materials used

The molecular formula of Glycine is ($C_2H_5NO_2$). Molar mass: 75.07 g/mol, Aluminium chloride, ($Alcl_3$), AR (99%), M. Wt.: 133.33 g/mol. For the crystal formation, distilled water was utilized as solvent.

Crystal growth Method

Glycine single crystals were grown by the slow evaporation method. It is the method of solution growth at low temperatures. If the solution is supersaturated that is, contains more solute than can be in equilibrium with the solid. Bulk crystals with high solubility and temperature-dependent solubility can be grown using this technique [10].

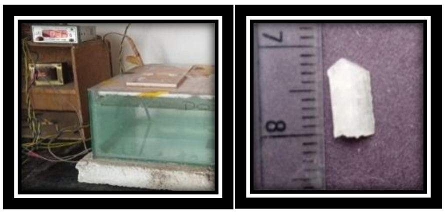

Figure 1: (a) Experimental apparatus and grown Glycine crystal.

In order to prepare the solution, glycine and $Alcl_3$ were taken. The aluminium chloride 0.8 M% was mixed with saturated glycine solution in a vessel, and the mixture was kept on a magnetic stirrer at room temperature and stirred thoroughly for approximately 4 hours. When a clear solution was seen, the solution was poured into a beaker. The saturated solution was filtered using Whatman filter paper in a clean vessel, which was covered with a perforated polythene sheet and placed in a temperature bath that was controlled by a microprocessor.

The constant temperature bath 35 °C the solution was allowed for slow evaporation and grown crystal obtained within 30 days. The figure 1 shows crystal growth experiment and grown crystal.

Characterisation Studies

XRD Analysis

The X- ray diffraction graph of a grown crystal gets into peak position and intensities. The position of the lines on graph are shows spacing between planes in the lattice and the intensities depends on the atoms position on particulars sites. In order to analyze the crystal's structure, the generated glycine was subjected to a powder x-ray diffraction examination using a Lynx Eye X-ray diffractometer with Kα radiation (λ = 1.54060 Å) at room temperature (25 °C).

For the analysis, the growing crystal was ground into a fine powder. The powder sample was measured between 10^0 and 80^0.

The diffracted beam's intensity was measured as a function of 2Ɵ, and the peaks were indexed in accordance with the literature report [9,11].

Alpha glycine data from JCPDS CARD NO:32-1702 and the measured values agree well. Glycine crystal doped with aluminium chloride belongs to monoclinic system with space group $P2_1/n$. The lattice parameter a = 5.4305 Å, b = 11.901 Å, c = 5.1971 Å, α = γ = 90, β = 111.92^0. The characteristics peak at 2Ɵ =29.56 for $Alcl_3$ doped glycine and prominent planes are (040), (021), (-110), (044). The Strong, pointed peaks show up, confirming the grown sample's good crystallinity. Figure 2 displays the index XRD plane for the synthesized crystal.

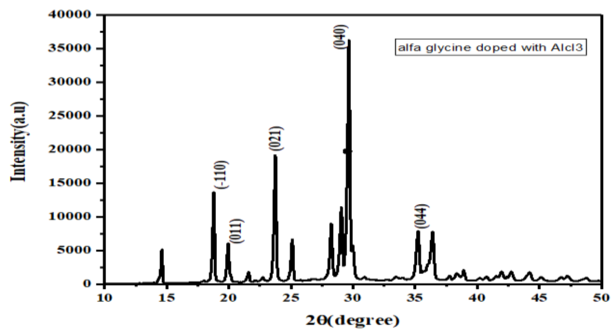

Figure 2: XRD studies of α -glycine crystal

FTIR Studies

Figure 3 displays the powdered doped glycine's acquired FTIR spectra. The functional groups of the generated α single crystal are in good agreement with the results that have been reported [9-11].

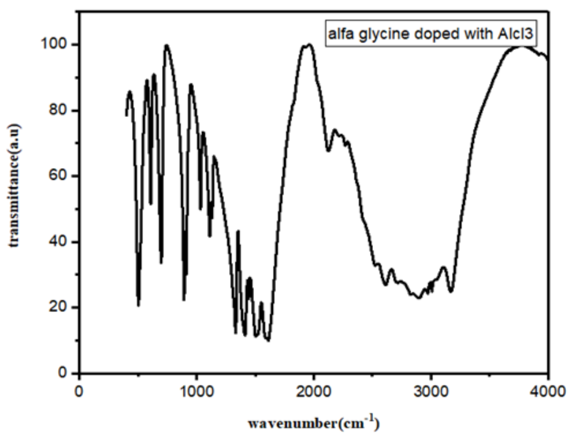

Figure 3: FTIR Analysis of α glycine single crystal.

Table 1: FTIR Analysis.

$AlCl_3$ doped glycine (α-glycine) cm^{-1}	Band assignment
2611	NH_3^+ stretching vibration
2133	Combinational bond
1612	asymmetric CO_2 stretching vibration
1505	NH_3^+
1421	Symmetric stretching vibration COO^-
1322	twisting vibration of CH_2
1121	Rocking vibration of NH_3^+
1023	Asymmetric stretching vibration of CCN
999	Rocking vibration of CH_2
890	symmetric stretching vibration of CCN
698	Bending vibration of COO^-
605	wagging vibration of COO^-

It can be shown from the report of α-glycine doped $AlCl_3$ that the carboxylic groups correspond to the functional groups 503, 607, and 697 cm^{-1}. According to the FTIR spectra, the detected at 1023 cm^{-1} and 890 cm^{-1} correspond to the C-C-N symmetric stretching vibration and the C-C-N asymmetric stretching vibration, respectively. Table 1 displays the detected frequencies along with the glycine single crystal's assignment.

Analysis of the UV-visible spectrum

Figure 4. displays UV-Vis transmission spectrum of the crystal-grown. The spectrum was obtained between 200 and 1100 nm. It can be seen from the spectrum that the lower cutoff wavelength is at 304 nm.

Throughout the visible spectrum, the crystal is clear. It implies that the crystal is appropriate for the process of frequency doubling.

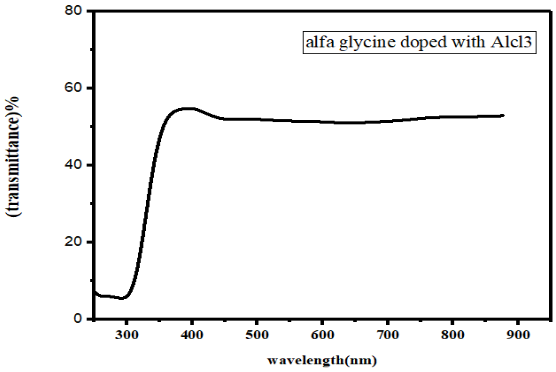

Figure 4: UV spectra of glycine single crystal

TGA and DTA Studies

Thermo-gravimetric analysis was carried out to examine the alpha glycine crystalline sample's thermal behaviour. In a nitrogen atmosphere, the thermal analyser was used with a heating rate of 20 °C/min. Temperatures between 20 and 1000 °C were used to record the TGA. Glycine's breakdown is represented by the peak in the α glycine DTA curve at 262.34 °C. The graph shows that the heat flow remained constant until it reached 262.34 °C. Until the melting phase, there is also no weight loss. The DTA curve shows endothermic process.

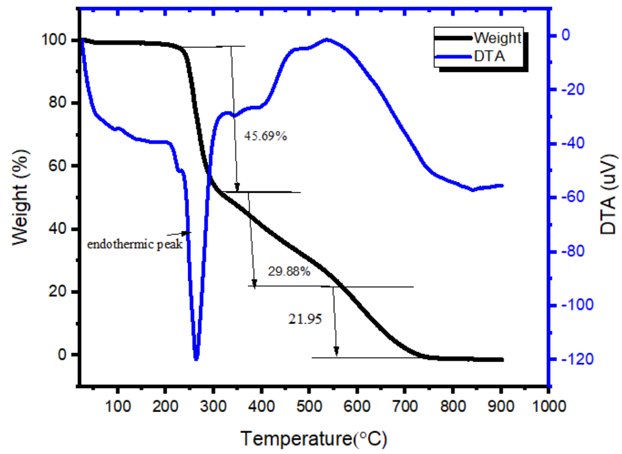

Figure 5: TGA and DTA graph.

Conclusion

We have generated and study a single crystal of alpha glycine using the slow evaporation approach and doping it with $alcl_3$ at a concentration of 0.8M % at first time. Aluminium chloride doped with glycine crystallizes in a monoclinic system, according to powder XRD examination. Good crystallinity of the developed crystal is confirmed by the emergence of a strong and sharp peak. Different functional groups are confirmed to be present by FTIR analysis. UV examination shows that the transmitters range from 300 to 900 nm, with the lower cut-off frequency at 304 nm. Up to 265 °C, alfa glycine single crystals are thermally stable.

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