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Synthesis, Growth and Characterization of Glycine Zinc Acetate, a New Nonlinear Optical Material

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ABSTRACT: *Crystals of Glycine Zinc Acetate (GZA), an Organic nonlinear optical material, was obtained by slow evaporation solution growth method using water as a solvent. Crystal purity was increased by the method of recrystallization. The grown crystals of GZA were subjected to characterization studies like powder XRD, FTIR, UV-Vis, TGA and DTA.The structure, lattice parameters and cell volume of GZA crystals were obtained by powder X-Ray diffraction studies. The presence of various functional groups of GZA and their vibrational modes were identified from Fourier Transform Infrared (FTIR). The optical transmission range of the GZA crystals was determined by UV-Visible Spectroscopy. The crystals of GZA were found to have wide range of transparency in the visible region. Thermal stability of the grown crystal GZA was studied using TGA and DTA.*

Keywords: *Crystal growth; X-ray diffraction; FTIR; NLO property; UV; TGA and DTA.*

I. Introduction

A close literature survey shows that the various amino acids exhibit more Non-linear optical properties[1]. The research of suitable nonlinear material is an important task because of their potential application in telecommunication for efficient signal processing and optical information storage devices [2]. Inorganic materials are widely used in signal processing fields like signal frequency shifting, optical logic and optical memory areas, but the nonlinearity of the inorganic material is very poor. Organic compounds possess high nonlinearity because they provide high degree of delocalization due to weak Vander Waal's and hydrogen bonds. Hence, they are optically more nonlinear than inorganic materials.

Amino acids are the suitable materials for NLO applications because they contain a proton donor Carboxylic acid(-COO) group and proton acceptor amino($-NH_2$) group in them. Therefore amino acid exhibits dipolar nature which enhances the physical and chemical properties in amino acid.

In this present paper, Glycine is combined with zinc acetate for the very first time and a new NLO material is grown by slow evaporation method at room temperature using water as a solvent. The grown crystals of Glycine zinc acetate (GZA) are subjected to various characterization studies like Powder X-Ray diffraction, Fourier Transform Infrared spectral analysis, FT-Raman IR analysis, UV-Visible and thermal studies TGA/DTA.

II. Raw material preparation of Glycine Zinc Acetate

2.1 Experimental procedure

Glycine Zinc Acetate was prepared from Glycine and zinc acetate, taken in equimolar ratio (1:1). The molecule glycine has two groups (carboxylic and amino acid) that can be protonated. The starting material for the synthesis of GZA was calculated according to the following reaction.

$H_2N\text{-}H_2C\text{-}COOH + Zn(CH_3COO)_2 \longrightarrow H_2N\text{-}H_2C\text{-}COOH.Zn(CH_3COO)_2$

Glycine — Zinc Acetate — Glycine Zinc Acetate

Initially Zinc acetate (1M) was first dissolved in deionized water. Glycine was then added to the solution slowly by continuous stirring for one hour to form a homogeneous mixture. The solution was placed in undisturbed position. After few days, GZA raw material was collected from the bottom of the beaker.Glycine Zinc Acetate raw material was recrystallized twice which improves the purity of the grown crystals. Small crystals of GZA were obtained after a period of one week.

III. Characterization Studies

The crystals obtained from recrystallization process were subjected to the following studies. The crystal structure, cell parameters and volume of the grown crystal have been determined powder X-Ray diffraction analysis. The presence of various functional groups and modes of vibration were identified by FTIR and Raman IR analysis. The optical transmission range of the grown crystal GZA was determined by UV-

Visible Spectroscopic analysis. The NLO property of the grown crystal were confirmed by second harmonic generation using Nd:YAG laser. TGA and DTA were carried out to study about the thermal stability of the grown crystal.

3.1 Powder X-Ray diffraction analysis

Powder X-Ray diffraction analysis has been carried out to confirm the crystallinity and also used to determine the lattice parameter [3]. X-Ray diffraction studies of crystals of Glycine zinc acetate was carried out using Rigaku Corporation, Japan make D/max Ultima III X-ray diffractometer with CuKα (λ = 1.5418Å) radiation. The instrument was operated in theta-theta vertical mode at a scan rate of 4°/min and the sample was scanned over the range from 10° to 80°. The detector used to be a scintillation counter. The resultant powder X-ray diffraction pattern of grown crystal GZA is shown in the fig.1

The X-Ray Diffraction pattern of GZA crystal was indexed using Powder X-VB VERSION BY CHENG DONG BASED ON TREOR90. The lattice constants and lattice parameter were calculated from the data obtained from XRD pattern and the values confirms that the grown crystal belongs to "Hexagonal system" with lattice parameter a = 7.0675, b = 7.0675, c = 11.0290, α = β = 90°and γ = 120°.

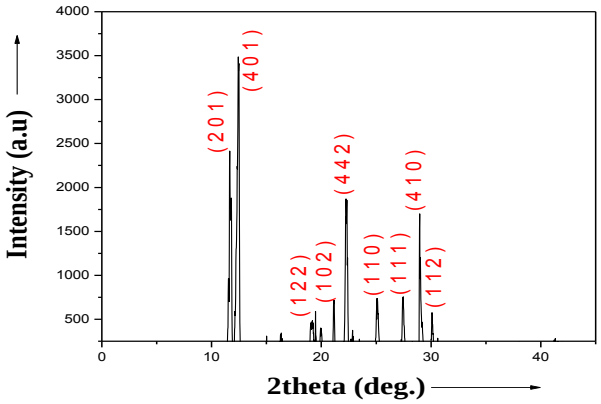

Fig 1- Powder X-ray diffraction pattern of GZA crystal

3.2 Fourier Transform Infrared spectral analysis

Fourier Transform Infra-Red (FTIR) spectroscopy was used to identify the functional groups present in the grown crystals of GZA [4]. The FTIR spectrum of the grown crystal GZA was recorded with a BRUKER-Fourier Transform Infrared spectrometer in the range 4000cm^{-1} to 400cm^{-1} using KBr pellet technique. The observed spectrum of GZA crystal is shown in fig.2. The vibrational frequency of various functional groups of GZA and the assignments corresponding to the peaks are presented in table.2.

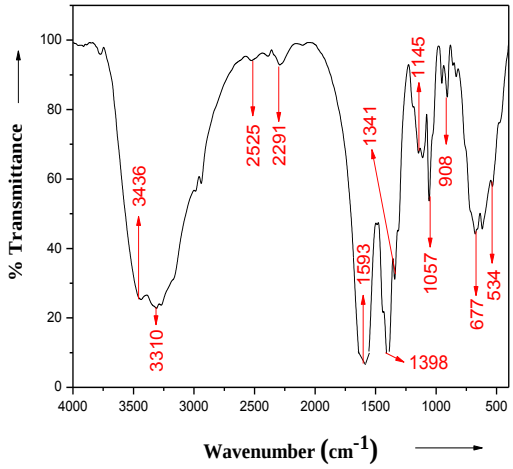

Fig 2 - FTIR spectrum of Glycine Zinc Acetate crystals

In GZA, the broad peak at 3436cm^{-1} is assigned to symmetric stretching vibration of H_2O molecule of Glycine. The peak at 3310cm^{-1} is due to symmetric stretching vibration of NH_2. The peak at 2940cm^{-1} is due to asymmetric stretching vibration of CH_2.The peak at 1398cm^{-1} and 677cm^{-1} corresponds to COO^- symmetric stretching and scissoring vibration. The peak at 952cm^{-1} and 908cm^{-1} is due to CH_2 rocking. CH_3 rocking vibration is observed at 1341cm^{-1}. NH_3^+ degenerative deformation is observed at 1341cm^{-1}. The rocking and scissoring vibrations of COO^- are observed at 677cm^{-1} and 534cm^{-1}. Out of plane vibration of CO_2 is observed at 834cm^{-1}.

Table 1 - Vibrational band assignments of Glycine zinc acetate

Wavenumber (cm^{-1})	**Assignments**
3436	Symmetric stretching vibration of H_2O
3310	Symmetric stretch of NH_2
2940	CH2 asymmetric stretching bands
1593	NH3+ degenerative deformation
1398	COO- symmetric stretching vibration
1341	CH3 rocking
1145	ND3* symmetric deformation
1111	NH3+ parallel rocking modes
952	CH2 rocking
908	CH2 rocking
834	CO2- out of plane vibration
677	COO- Scissoring vibration
534	COO- rocking

3.3 UV-Visible spectral analysis

The Ultraviolet Visible spectrum gives information about transmission range of the grown crystal. UV-Visible spectrum was recorded using Perkin Elmer make Lambda 35 UV –Visible Spectrometer in the range from 190nm to 1100nm and the observed spectrum GZA crystal is shown in following figure. The observed spectrum of GZA was covering the region near UV, Visible region and NIR region respectively. Transparency in the visible region is a desirous property for any NLO material[5].

The optical spectrum of GZA shows a good transmittance in the visible region. From the spectrum of GZA crystal, there is no absorption in the range from 300nm to 1400nm. From the graph, it is clear that the grown GZA crystal has a UV cutoff wavelength around 193nm. This makes these crystals suitable for UV tunable laser and SHG device applications [2].

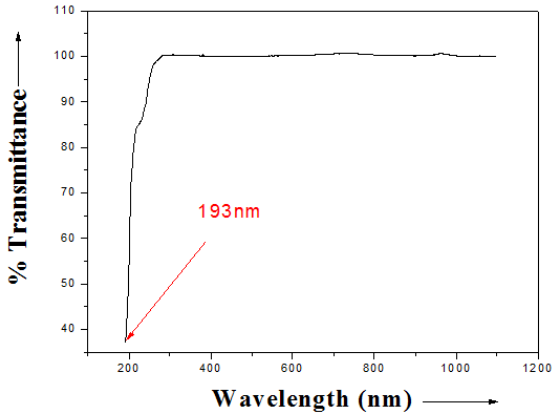

Fig 3 - UV-Visible spectrum of Glycine zinc acetate crystals

3.4 TGA and DTA analysis

The Thermogravimetric analysis and Differential thermogravimetric analysis of Glycine Zinc Acetate have been recorded using a CNST thermal analyser [4]. Ceramic crucible was used for heating the sample and the analysis were carried out in an atmosphere of Nitrogen at a heating rate of 20°C per minute for the temperature 30°C-500°C. The TGA and DTA traces are shown in the following fig.

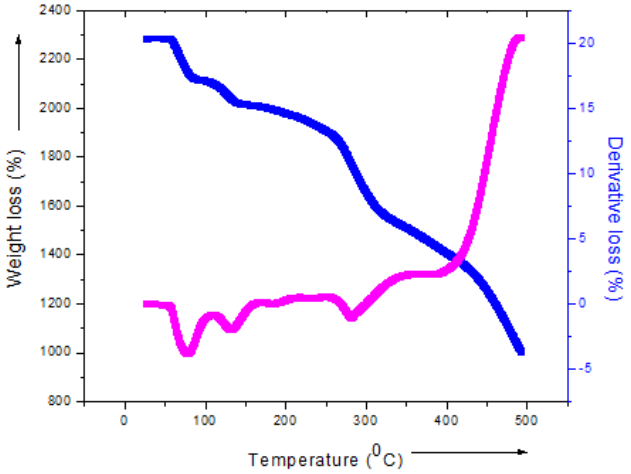

Fig 4 TGA and DTA Analysis of GZA crystals

In TGA, the compound starts to lose water of crystallization at about 57.90°C and the loss continues upto 83.70°C. The weight loss in the temperature rangefrom 83.70°C to 491.59°Cis due to the decomposition of Glycine zinc acetate crystal.

In DTA, the peak at 78**°C,** which is correspondsto the evaporation of water molecule. The second and third endothermic peaks were observed at 133**°C** and 281**°C** due to the mother compound decomposition of acidic and nitrogen. The melting point of glycine is 233**°C** but the endothermic peak was observed at 281**°C** which confirms the addition of Zinc Acetate.

IV. Conclusion

Glycine Zinc Acetate (GZA) crystals were prepared by slow evaporation method using water as a solvent for the first time. The lattice parameters were found by Powder X-ray diffraction analysis and the grown crystals belong to Hexagonal system. The presence of functional groups and their modes of vibration were confirmed by FTIR analysis. The UV-Vis spectra showed that the grown crystals were highly transparent in the entire visible region with a lower cutoff wavelength at 193nm, making it a potential candidate for NLO applications. Thermal stability of the grown crystal was studied using TGA/DTA analysis and the result shows that the grown crystal was stableupto 134°C.

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