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Growth and Characterisation of Glycine Sodium Nitrate and L-Proline Doped Glycine Sodium Nitrate NLO Crystal

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ABSTRACT

Using slow evaporation process the homogeneous solution of glycine sodium nitrate (GNS) and L-Proline doped glycine sodium nitrate (GSNLP) was prepared for equimolar ratio at room temperature. The crystalline nature is confirmed by powder XRD analysis for both GSN and GSNLP crystal. The cell parameters and structure of grown crystal were determined using single crystal XRD analysis. The result confirmed it belongs to monoclinic crystal system. Fourier transform infrared (FTIR) confirmed the functional groups which are present in the crystal. From UV-Visible analysis the cut off wavelengths has been found for GSN and GSNLP respectively. Comparison of the non-linear optical efficiency of grown GSN and GSNLP crystal, the Second harmonic generation (SHG) have been performed.

KEYWORDS: Slow evaporation process, Powder XRD, Single crystal XRD, FTIR, UV-Vis, SHG.

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INTRODUCTION

Optoelectronic and photonics are the field where NLO crystals can play major role. In recent years the amino acids plays a vital role in the NLO. Since the weak Vander walls and hydrogen bond provides good transparency in the UV-Vis-NIR range. Zwitter ionic nature favors physicochemical stability. Molecular chirality secures acentric crystallography structure¹. NLO provides many key functions like frequency conversion and optical switching. Organic materials exhibit higher nonlinear second order coefficients². NLO materials will be the key elements for future photonic technologies based on the fact that photons are capable of processing information with the speed light³. Glycine [NH₂CH₂COOH] is the simplest amino acid. Glycine remains as one of the most extensively studied amino acids, as it is known to form innumerable complexes with metals, inorganic salts and inorganic acids⁴. In the present study GSN and GSNLP crystal were grown by using slow evaporation method at room temperature⁵. The grown crystal was subjected to various characterizations such as powder XRD, single XRD, FTIR, UV-Vis, SHG.

EXPERIMENTAL PROCESS

The GSN were taken in equimolar ratio and GSNLP were also taken in equimolar ratio. The compound was dissolved in a double distilled water and stirred with magnetic stirrer (Remi-1 MLH) at room temperature for 6 hours to achieve the homogenous solution. The saturated solution was filtered with Whatmann filter paper. The crystal clear solution is allowed to slow evaporation and closed with pinhole perforated sheet⁶⁻⁹. The beaker was kept in the dust free atmosphere for slow evaporation. Lydia et al., grown the single crystals of dichlorobisL-proline (DBLPZ) by slow evaporation technique¹⁰. After a time span of 30 days and 4 month GSN and GSNLP crystal was harvested. The photograph of the grown crystal are shown in Fig 1.



Figure 1: GSN and GSNLP crystal

RESULT AND DISCUSSION

Powder x-ray diffraction analysis

The powder XRD pattern was recorded at room temperature with CuK α alpha radiation of wavelength $\lambda = 1.540 \text{ \AA}$. The sample was scanned over the range of 0° - 80° at a scan rate of 1° per minute. The powder XRD pattern of the grown GSNLP crystal is shown in Fig 2. The sharp peaks observed in the XRD pattern confirm the crystalline nature of the sample.

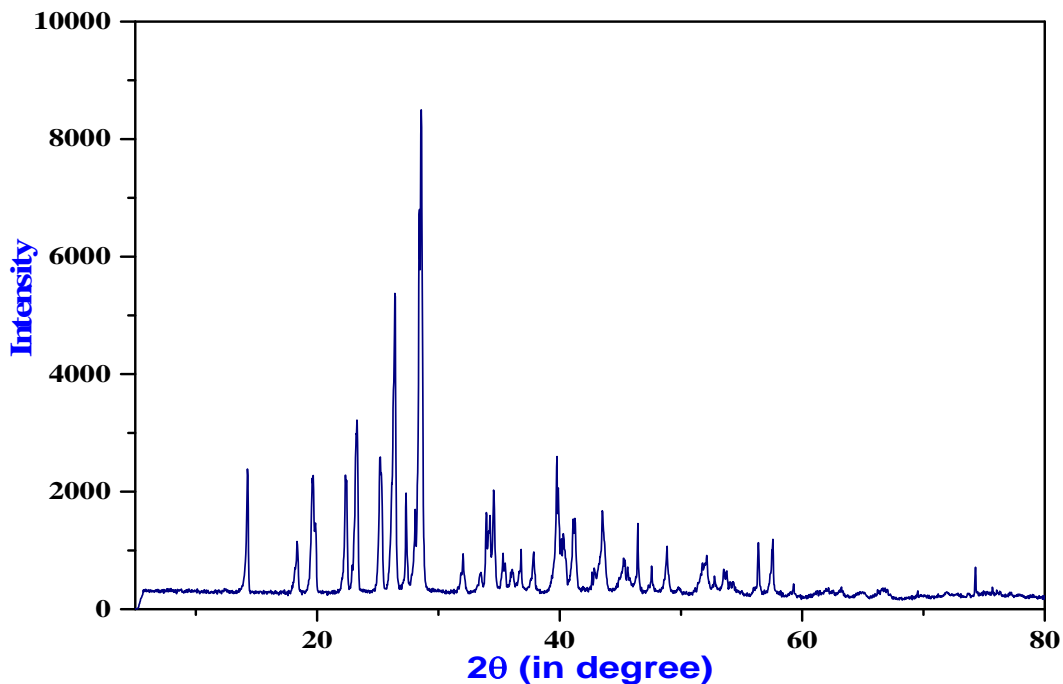


Figure 2: Powder XRD pattern for GSNLP crystal

Single crystal x-ray diffraction

The good quality grown of GSN and GSNLP crystals were subjected to single crystal X-ray diffraction analysis using the Bruker instrument the cell parameter and space group were found. The given Table 1 shows the cell parameter and space group.

Table 1 Powder XRD pattern for GSNLP crystal

Sample	Lattice Parameters			α, β, γ	Structure
	a (Å)	b(Å)	c (Å)		
GSNCrystal	14.30	5.24	9.10	$\alpha=90^{\circ}$, $\beta=109^{\circ}, \gamma=90^{\circ}$	Monoclinic
GSNLP Crystal	10.91	4.44	5.22	$\alpha=90^{\circ}$, $\beta=109^{\circ}, \gamma=90^{\circ}$	Monoclinic

VIBRATIONAL SPECTRAL ANALYSIS

Fourier Transform Infrared Spectroscopy

The functional group present in grown crystal is determined using FTIR spectroscopy. The observed bands along with their vibrational assignments have been tabulated. The range of GSN and GSNLP crystal is found to be 4000 – 500cm⁻¹. The presence study of GSNLP crystal, FTIR spectrum also shows the presence of functional group¹¹. The recorded pattern of GSN and GSNLP is shown below.

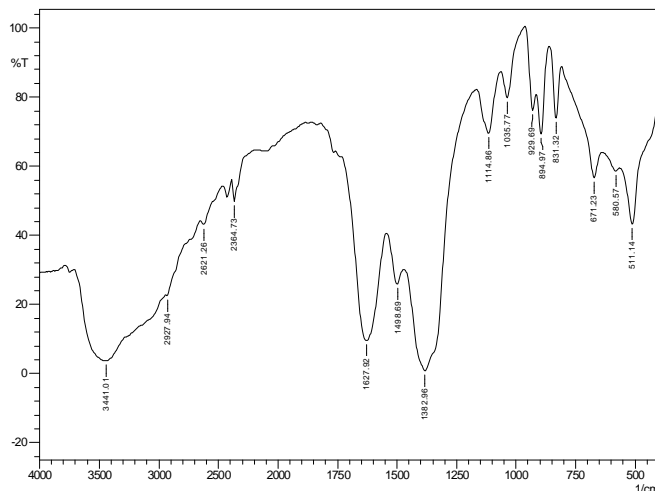


Figure 3: FTIR spectrum for GSN crystal

Table 2 Frequency and space group

Frequency	Assignments
511	Co ^{o-} rocking
671	NO ₃ plane bending
1035	CH ₂ rocking
1114	NH ₃ ⁺ rocking
1627	C=O symmetric stretching
2927	CH ₂ symmetric stretching
3441	NH ₃ ⁺ asymmetric stretching

The FTIR spectrum of GSNLP is as shown in figure 4. The stretching frequency around 3572 cm⁻¹ clearly indicates O-H stretching of monomeric alcohol, and the presence of hydrogen bonding. In addition, stretching frequency at 2233 cm⁻¹ shows the presence of C=H stretching. The stretching frequency at 1519 indicates NH stretching. The stretching vibration 1463 cm⁻¹ show presence of C-H

bend. The presence amine group NH stretching frequency around 1352 cm^{-1} . The stretching vibration C-N stretching indicate that around 1126 cm^{-1} . The N-H bending for L proline is observed at the lower frequency region of 3230 cm^{-1} . Satiskumar et al., reported the presence of functional group of L proline such as OH, C=O and NH bending. The present study of GSNLP crystal, FTIR spectrum also shows the presence of functional group¹². The peaks at 833 cm^{-1} are assigned to stretching modes of CH_2 stretching.

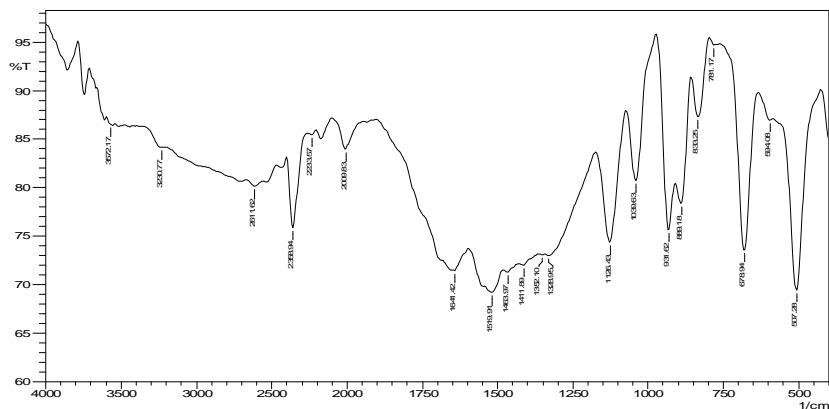


Figure 4: FTIR for GSNLP crystal

TABLE 3: Vibrational band assignments of GSNLP crystal

S.No	Wave number cm^{-1}	Assignments
1	3572	O-H stretching
2	2233	C=H stretching
3	1519	NH stretching
4	1463	C-H bend
5	1352	NH stretching
6	1126	C-N stretching
7	931	O-H bend
8	833	CH_2 stretching

OPTICAL CHARACTERIZATION

UV-VIS-NIR spectral studies of GSN crystal

The grown GSN crystal has a wide transparency in the near UV region and entire visible region. The absorption spectrum of GSN crystal is recorded at room temperature. The UV cut off wavelength has been found to be 235 nm. Good optical transparency in the entire visible region and near UV cut off wavelength of GSN crystal suggests its suitability for NLO devices.

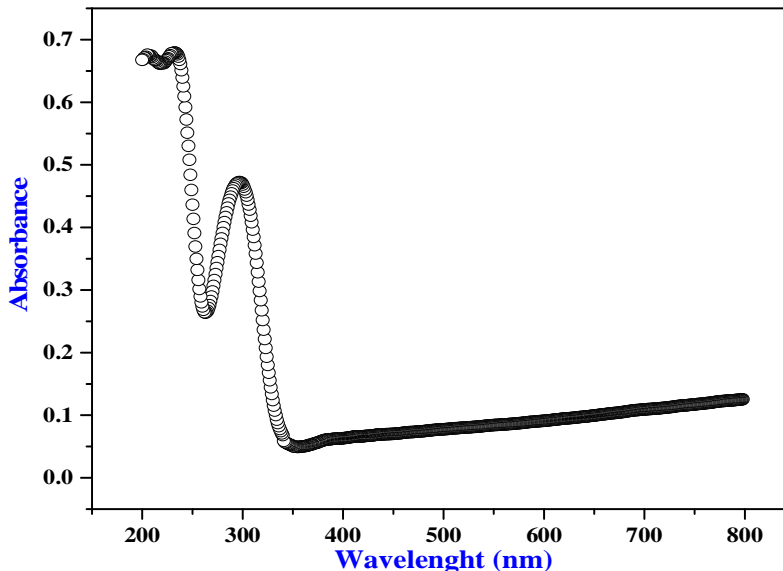


Figure 5:UV-Vis -NIR Spectrum of GSN Crystals

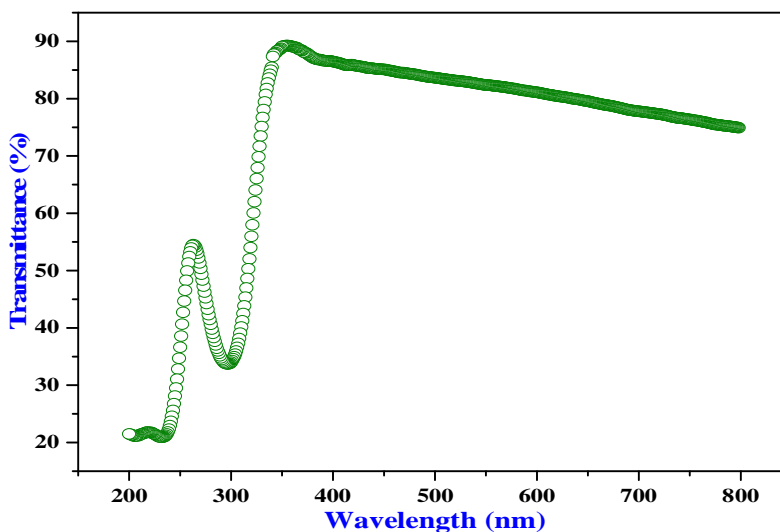


Figure 6:UV-Vis -NIR Spectrum of GSN Crystals

UV-VIS-NIR spectral studies for GSNLP crystal

The grown crystal of GSNLP was subjected to UV- absorption spectra. The peak was absorbed in the range of 0 - 1500 nm and shown in Figure 7.

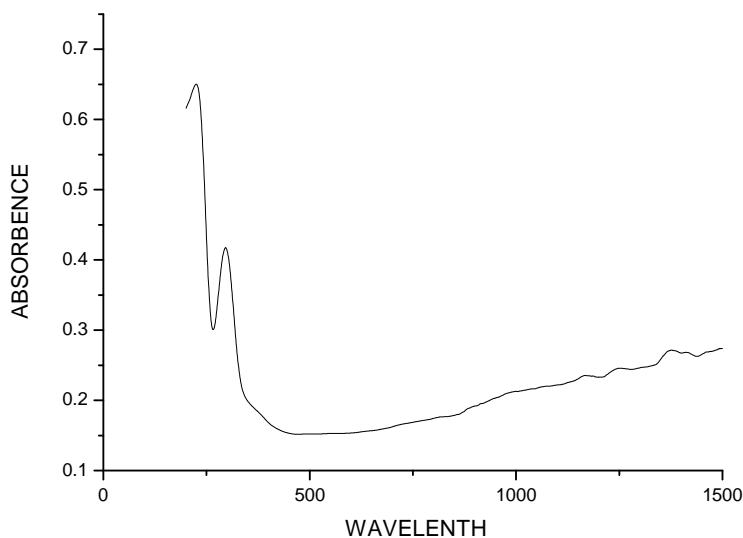


Figure 7:UV-Vis -NIR Spectrum of GSNLP Crystals

There is no absorption in wavelength range 217-1300 nm as in the case of aminoacid and the transmittance is approximately 70% in these wavelength range.The grown crystal has transparent above the 217 nm also it is potential candidate for the optical applications highly.

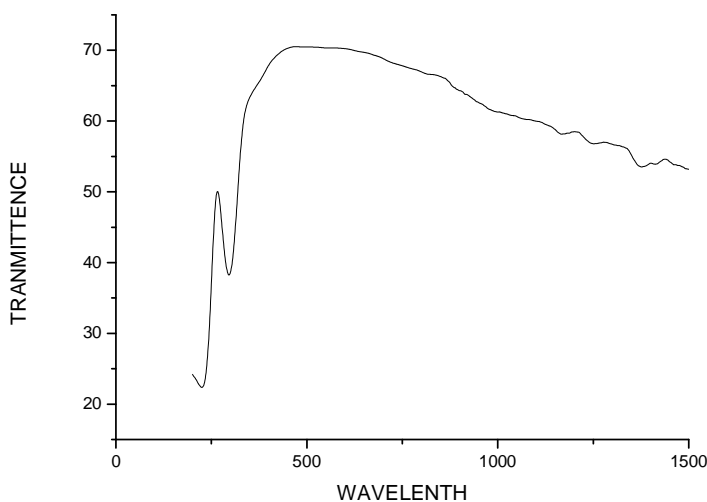


Figure 8:UV-Vis -NIR Spectrum of GSNLP Crystals

Table 4 UV Cut off wavelength

Name of the sample	Cut off wavelength (nm)
GSN Crystal	235
GSNLP Crystal	217

NON-LINEAR OPTICAL CHARACTERIZATION

Second harmonic generation efficiency

The NLO efficiency of the GSN crystal was tested using Kurtz and Perry method. The sample was illuminated using Q- switched Nd-YAG laser operating at the fundamental wavelength of 1064 nm with a repetition rate of 10 Hz was used for this experimental study. The Second harmonic generation signal of 10.48mJ for GSN crystal and 11.5mJ for GSNLP crystal was obtained for an input energy of 0.701 J. But the standard KDP crystal gave an SHG signal of 8.91mJ for the same input energy. It is notices that the SHG efficiency of the grown GSN crystal is 1.17times and GSNLP Crystal is 1.29 times greater than that of KDP.Besky job et al., studied the growth and characterization of glycine sodium nitrate crystal. They stated that non- linear optical efficiency of the crystal is good¹³.

Table 5 SHG efficiency

S.NO	NAMEOF THE SAMPLE	INPUT ENERGY (Joule)	OUTPUT ENERGY (milli joule)	SHG EFFICIENCY
1.	GSN Crystal	0.701	10.48	14.95
2.	GSNLP Crystal	0.701	11.5	16.40
3.	KDP	0.701	8.91	12.71

CONCLUSION

By using slow evaporation method the GSN and GSNLP crystal has been grown successfully at room temperature. XRD peaks confirmed the crystalline nature of the grown crystal and the structure of crystal is monoclinic. FTIR analysis confirms the various functional group present in GSN and GSNLP crystal. The grown crystals were optically transparent above the cut off wavelength and the absorbance of GSNLP is less than GSN crystal such as 217 nm and 235 nm as it lies in UV region and also in visible region. The NLO of grown GSN and GSNLP crystal is 1.17 and 1.29 times greater than the KDP. Hence this crystal can be used in optical communication.

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