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Effect of zinc acetate addition on crystal growth, structural, optical, thermal properties of glycine single crystals

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KEYWORDS

Crystal growth; X-ray diffraction; Optical studies; Thermal analysis; Spectral studies; Nonlinear optical crystals **Abstract** In the present study, γ -glycine has been crystallized by using zinc acetate dihydrate as an additive for the first time by slow solvent evaporation method. The second harmonic conversion efficiency of γ -glycine crystal was determined using Kurtz and Perry powder technique and was found to be 3.66 times greater than that of standard inorganic material potassium dihydrogen phosphate (KDP). The analytical grade chemicals of glycine and zinc acetate dihydrate were taken in six different molar ratios: 1:0.2, 1:0.4, 1:0.6, 1:0.7, 1:0.8, and 1:0.9 respectively to find out the γ -polymorph of glycine. The lower molar concentration of zinc acetate yield only α -polymorph where as the higher molar concentration of zinc acetate inhibits the γ -polymorph of glycine which was confirmed by single crystal XRD and powder XRD studies. Inductively coupled plasma optical emission spectrometry (ICP-OES) was carried out to quantify the concentration of zinc element in the grown glycine single crystals. The concentration of zinc element in the presence of grown γ glycine single crystal is found to be 0.73 ppm. UV-Visible-NIR transmittance spectra were recorded for the samples to analyse the transparency in visible and near infrared region (NIR). The optical band gap E_g was estimated for γ -glycine single crystal using UV–Visible–NIR study. Functional groups present in the samples were identified by FTIR spectroscopic analysis. Differential scanning calorimetry technique was employed to determine the phase transition, thermal stability and melting point of the grown crystal.

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1. Introduction

Nonlinear optical (NLO) crystals with high second harmonic generation efficiencies and transparent in the visible and ultraviolet ranges are required for various devices in the field of optoelectronics and photonics (Calark, 1988). Amino acid crystals have been grown and studied by several researchers for their excellent nonlinear optical properties in the last two decades (Vijayan et al., 2009). The area of active research for

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many decades is understanding and controlling solution crystallization and polymorphism (He et al., 2006). In the recent past, growth and characterization of γ -glycine crystals have been reported by several researchers and it was observed that γ -glycine single crystals have been grown using additives such as sodium nitrate (Narayan Bhat and Dharmaprakash, 2002), potassium chloride (Sekar and Parimaladevi, 2009), potassium fluoride (Dillip et al., 2011), lithium nitrate (Esthaku Peter and Ramasamy, 2010), lithium bromide (Balakrishnan et al., 2008), lithium acetate (Dhanraj and Rajesh, 2009), potassium bromide (Zulifigar Ali Ahamed et al., 2013), ammonium nitrate (Narayana Moolya et al., 2005), ammonium sulphate (Anbuchudar Azhagan and Ganesan, 2013), ammonium acetate (Anbuchudar Azhagan and Ganesan, 2012), ammonium carbonate (Anbuchudar Azhagan and Ganesan, 2013), magnesium chloride (Dillip et al., 2012), phosphoric acid (Parimaladevi and Sekar, 2010), and strontium chloride (Anbuchezhiyan et al., 2010). In this paper we report the crystal growth and characterization studies of γ -glycine crystals grown from aqueous solution of glycine in the presence of zinc acetate for the first time. The second harmonic generation efficiency of γ -glycine grown from zinc acetate additive is 3.66 times that of inorganic material potassium dihydrogen phosphate (KDP), where as the second harmonic generation efficiency of γ -glycine grown from lithium bromide additive is 3 times that of potassium dihydrogen phosphate (Balakrishnan et al., 2008). The highlight of the present work is the second harmonic efficiency of the grown crystal is slightly higher than the work reported earlier (Balakrishnan et al., 2008).

2. Experimental

2.1. Chemicals used

Glycine ($C_2H_5No_2$ aminoaceticacid) SD-fine AR (99.5%) M.wt: 75.07 g/mol, zinc acetate dihydrate ($C_4H_6O_4Zn\cdot 2H_2O$) SD-fine AR (99.5%) M.wt: 219.50 g/mol and double distilled water were used for the crystal growth experiment.

2.2. Crystal growth

In series the analytical reagent chemicals of glycine and zinc acetate dihydrate were mixed in different molar ratios such as 1:0.2,1: 0.4, 1:0.6, 1:0.7, 1:0.8, and 1:0.9, respectively in 100 ml double distilled water to identify and confirm the presence of γ -polymorph in the aqueous solution. The prepared solutions were placed in a separate 100 ml beaker and the mixtures were stirred well continuously up to 6 h to get the homogenous saturated solutions. The saturated solutions were filtered with whatman 41 filter paper. The filtered saturated solutions were closed with a perforated cover and kept in a dust free environment and allowed to crystallize by slow evaporation at ambient temperature. After a nucleation period of 5-7 days of solvent evaporation, the solution becomes supersaturated and tiny crystals were found in the beakers containing molar ratios such as 1:0.9, 1:0.8, 1:0.7, and 1:0.6, respectively. The nucleation period τ for the grown crystals is presented in Table 1. From the growth experiment, it is clear that higher molar concentration of zinc acetate dihydrate (0.6-0.9) induces quick nucleation sites than that of lower molar concentration of zinc acetate (0.2, 0.4).

Table 1Nucleation table.	
Molar ratio of Glycine and zinc acetate	Nucleation period τ in days
1:0.2	14
1:0.4	11
1:0.6	7
1:0.7	6
1:0.8	5
1:0.9	5

2.3. Crystal morphology

The morphology of the grown crystal changes from α to γ -polymorph when the concentration of zinc acetate exceeds 0.4 M. The α - and γ -polymorphs of glycine were confirmed by Single crystal XRD, Powder XRD and FTIR studies. The grown crystals were harvested after a period of 18 days. The harvested seed crystal of α -glycine and its bulk size are shown in Figs. 1 and 2. The morphology of the crystal resulted from lower molar concentrations of zinc acetate is shown in Figs. 3 and 4. Higher molar concentrations of zinc acetate lead to γ -polymorphs which are presented in Figs. 5–8.

3. Results and discussion

3.1. Single crystal XRD and ICP-OES studies

Single crystal X-ray diffraction data were recorded using EN-RAF NONIUS CAD4-MV31 X-ray diffractometer with MoK_{α} radiation ($\lambda = 0.71073$ Å) to reveal the crystal structure and unit cell dimensions of the grown crystal. The unit cell dimensions were obtained for the grown single crystals from single crystal XRD analysis and are presented in Table 2. This study confirms the α - and γ -polymorphs of glycine. The cell values closely agreed with the reported values (Sekar and Parimaladevi, 2009). To determine the concentration of zinc element present in the grown crystals, the inductively coupled plasma optical emission spectrometry (ICP-OES) analysis using Perkin Elmer Optima 5300 DV ICP-OES spectrometry was carried out. The presence of zinc element in the grown



Figure 1 Harvested seed crystal of α -Glycine.



Figure 2 Bulk size of α -Glycine crystal.



Figure 3 α -Glycine crystal (1:0.2).



Figure 4 α -Glycine crystal (1:0.4).

crystals is listed in Table 3. Emission spectrometry is based on the principle that atoms or ions in an excited state tend, to revert back to the ground state and in doing so emit characteristic wavelength and the intensity of that light is proportional to the concentration of that particular element in the sample solution. This technique is used for qualitative and quantitative determination of zinc present in the γ -glycine. For this



Figure 5 γ -Glycine crystal (1:0.6).



Figure 6 γ -Glycine crystal (1:0.7).



Figure 7 γ -Glycine crystal (1:0.8).

analysis, the grown crystals were crushed into pieces and finely grounded in an agate mortar. These powdered samples were transferred into a 25 ml volumetric flask with the help of a funnel and were diluted to volume with deionized water. This



Figure 8 γ-Glycine crystal (1:0.9).

diluted sample was analysed by ICP-OES system. The amount of zinc element present in the γ -glycine sample prepared from 0.9 M concentration of zinc acetate was found to be 0.73 ppm and its characteristic wavelength is 213.89 nm. It is determined that the concentration of zinc is so low as to form a complex and hence it can be concluded that the zinc can be incorporated in the void space within the crystal lattice of γ -glycine (Dillip et al., 2012; Anbuchezhiyan et al., 2010).

3.2. Powder X-ray diffraction analysis

Powder X-ray diffraction studies were carried out using a Bruker D8 advance diffractometer with CuK_{α} ($\lambda = 1.5406$ Å) radiation. The powdered α - and γ -glycine sample from 0.4 to 0.9 M concentration of zinc acetate respectively was scanned over a range 5-70° at the scan rate of 1° min. The reflection peaks corresponding to different crystal hkl planes in the recorded XRD profile were indexed. The indexed powder X-ray diffraction pattern for α - and γ -glycine crystal is shown in Figs. 9 and 10 for comparison. The observed powder Xray diffraction pattern and position of the peaks of α - and γ glycine crystal were found to be in good agreement with the reported pattern (Index to the Powder Diffraction File, 2002). The data obtained from the XRD spectrum such as Angle 2θ , d-value, hkl, peak intensity, intensity (%) and full width half maximum value (FWHM) of every prominent peak in the spectrum of α - and γ -glycine are tabulated in Tables 4 and 5. The sharp and strong peaks in the XRD pattern confirm good crystallinity of the grown crystal. From the XRD data, the lattice parameters of the grown α - and γ -glycine crystal were calculated using UNIT CELL software and are tabulated in Table 6.

3.3. FTIR spectral analysis

The infrared spectroscopy is effectively used to identify the functional groups of the samples. FTIR spectra of α - and γ -glycine sample from 0.4 to 0.9 M concentration of zinc acetate respectively are shown in Figs. 11 and 12 for comparison. In the FTIR spectrum of α -glycine, the peaks observed at 502, 606 and 695 cm⁻¹ are attributed to the carboxylic groups while the peak observed at 1513 cm⁻¹ corresponds to the NH₃⁺ group. The peaks at 1030 and 898 cm⁻¹ are attributed to C–

Table 2 Single crystal XRD cell value results.										
Molar ratio of Glycine and zinc acetate	Olar ratio of Lattice parameters C lycine and nc acetate							Crystal System	Polymorph	JCPDS CARD NO.
	a [Å]	b [Å]	c [Å]	α (degree)	β (degree)	γ (degree)	Cell volume [Å ³]			
1:0.2	5.10	11.93	5.45	90	111.90	90	308	Monoclinic	Alpha	32-1702
1:0.4	5.11	11.97	5.48	90	111.97	90	310	Monoclinic	Alpha	32-1702
1:0.6	7.02	7.02	5.41	90	90	120	233	Hexagonal	Gamma	06-230
1:0.7	7.05	7.05	5.44	90	90	120	235	Hexagonal	Gamma	06-230
1:0.8	7.08	7.08	5.46	90	90	120	237	Hexagonal	Gamma	06-230
1:0.9	7.09	7.09	5.48	90	90	120	238	Hexagonal	Gamma	06-230

Table 3 ICP-OES study results.

Molar ratio of Glycine and zinc acetate	Crystal System confirmed from SXRD cell values	Polymorph	Presence of Zinc element in ppm or milligram per litre	Characteristic wavelength of Zinc element (nm)
1:0.2	Monoclinic	Alpha	0.25	213.81
1:0.4	Monoclinic	Alpha	0.36	213.81
1:0.6	Hexagonal	Gamma	0.45	213.82
1:0.7	Hexagonal	Gamma	0.54	213.84
1:0.8	Hexagonal	Gamma	0.63	213.86
1:0.9	Hexagonal	Gamma	0.73	213.89



Figure 9 Indexed powder XRD profile of α -Glycine.



Figure 10 Indexed powder XRD profile of γ-Glycine.

 Table 4
 Powder XRD profile of Alpha glycine single crystal.

h	k	l	Angle 2-theta (°)	d value (Å)	FWHM (degree)	Peak intensity (Counts)	Intensity (%)
0	2	0	14.654	6.040	0.139	80.319	8.032
-1	1	0	18.726	4.735	0.139	742.529	74.253
0	1	1	19.918	4.454	0.139	81.682	8.168
0	2	1	23.633	3.762	0.238	286.201	28.62
0	4	0	29.671	3.008	0.199	1000	100
0	4	4	35.174	2.549	0.159	315.246	31.525
0	4	4	40.597	2.220	0.139	116.389	11.639
1	4	4	42.762	2.113	*****	70.462	7.046

C–N asymmetric and C–C–N symmetric stretching modes of vibration. The absorption peak observed at 1328 cm⁻¹ is attributed to CH₂ twisting mode. The peak observed at 1109 cm⁻¹ corresponds to NH₃⁺ rocking. The strong asymmetric CO₂ stretching vibration occurs at 1609 cm⁻¹. The combination bond occurs at 2121 cm⁻¹. The peaks at 2614 and 3169 cm⁻¹ are due to NH₃⁺ stretching vibration.

The spectrum of γ -glycine displays the peaks at 501, 606 and 683 cm⁻¹ that are attributed to the carboxylic groups

while the peak observed at 1491 cm^{-1} corresponds to the NH₃⁺ group. Frequencies observed at 1040 and 887 cm⁻¹ are attributed to C–C–N asymmetric and C–C–N symmetric stretching vibration. The absorption peaks observed at 927 and 1327 cm⁻¹ are attributed to CH₂ rocking and CH₂ twisting mode. The peak observed at 1124 cm⁻¹ corresponds to NH₃⁺ rocking. The prominent band near 2171 cm⁻¹ may be assigned to combination bond. The other peaks around 2601 and 3105 cm⁻¹ have been attributed to the NH₃⁺ stretching group.

h	k	l	Angle 2-theta (°)	d value (Å)	FWHM (degree)	Peak intensity (Counts)	Intensity (%)
1	0	0	14.581	6.0800	1.660	21.28	2.128
1	0	1	21.827	4.0700	2.233	483.141	48.314
1	1	0	25.301	3.5100	2.046	1000	100
1	1	1	30.192	2.9500	2.617	97.263	9.726
2	0	1	33.644	2.6600	1.692	79.409	7.941
1	0	2	35.956	2.5000	1.574	314.084	31.408
2	1	0	39.084	2.3000	2.048	194.06	19.406
1	1	2	41.866	2.1600	1.666	68.706	6.871
3	0	0	44.471	2.0300	1.649	96.586	9.659
3	0	1	47.812	1.9000	1.642	27.076	2.708
0	0	3	49.726	1.8200	1.307	29.868	2.987
2	1	2	51.870	1.7600	1.923	31.349	3.135
2	2	1	54.833	1.6900	1.681	46.495	4.649
3	0	2	56.388	1.6700	1.567	41.672	4.167

Table 6Unit cell results from powder XRD.

Polymorph	Lattice parameters						
	a [Å]	b [Å]	c [Å]	α (degree)	β (degree)	γ (degree)	Cell volume [Å ³]
α-Glycine	5.62276	12.0726	5.22291	90	113.7109	90	324.6122
γ-Glycine	7.03507	7.03507	5.48962	90	90	120	235.2938



Figure 11 FTIR spectrum of α -Glycine.

The FTIR data comparisons of α - and γ -glycine single crystals are presented in Table 7. The observed functional groups of the grown α - and γ -glycine single crystals are in good agreement with the literature values (Sekar and Parimaladevi, 2009; Dhanraj and Rajesh, 2009; Dillip et al., 2012).

3.4. Differential scanning calorimetry analysis

DSC studies of powdered sample of α - and γ -glycine sample from 0.4 to 0.9 M concentration of zinc acetate respectively

were recorded by employing NETZSCH DSC 200F3 instrument between 30 and 500 °C. The recorded DSC thermograms of α - and γ -glycine sample are shown in Figs. 13 and 14. The α -glycine DSC thermogram shows a peak at 253.9 °C which corresponds to the decomposition of glycine. Whereas in γ -glycine DSC thermogram a minor peak occurs at 187.3 °C, a phase transformation peak from γ to α -polymorph. The sharpness of the exothermic peak shows a good degree of crystallinity. From the DSC curve of γ -glycine, it is observed that an exothermic event begins at 243.5 °C and then a sharp



Figure 12 FTIR spectrum of γ -Glycine.

Table 7 Comparison of FTIR spectrum of α and γ -glycine crystal.

-		
α-Glycine	γ-Glycine	Tentative assignments
502	501	COO ⁻ rocking
606	606	COO ⁻ wagging
695	683	COO ⁻ bending
898	887	CCN Symmetric stretching vibration
_	927	CH ₂ rocking
1030	1040	CCN Asymmetric stretching vibration
1109	1124	NH ⁺ ₃ rocking
1328	1327	CH ₂ twisting
1409	1392	COO ⁻ symmetric stretching
1513	1491	NH_3^+
1609	1584	Strong asymmetric CO ₂ stretching
2121	2171	Combinational bond
2614	2601	NH ⁺ ₃ stretching vibration
3169	3105	NH_3^+ stretching vibration

peak appears at 256.9 °C which corresponds to decomposition point of the sample. The phase transition temperatures are reported by several researchers such as 172, 189, and 182 °C (Narayan Bhat and Dharmaprakash, 2002; Balakrishnan et al., 2008; Dhanraj and Rajesh, 2009). It is concluded from their studies that the transition temperature between α and γ -polymorphs can range between 165 and 201 °C (Perlovich et al., 2001). Thus, in the present work, the phase transition temperature appears at 187.3 °C.

3.5. Optical transmittance studies

Good optical transmittance and lower cut-off wavelength are essential properties for NLO crystals. The UV transmission spectra of α -glycine and γ -glycine grown in the presence of 0.4 and 0.9 M concentrations of zinc acetate respectively are shown in Figs. 15 and 16 for comparison. The lower



Figure 13 DSC thermogram of α -Glycine.



Figure 14 DSC thermogram of γ -Glycine.



Figure 15 UV spectrum of α -Glycine.

cut-off wavelength of γ -glycine was at 212 nm and it was determined by chromophores such as the aminoacid and carboxyl group, which show no absorption and are transparent in the UV region. The bandgap of γ -glycine is calculated using the formula $E_g = 1240/\lambda$ (nm). It is found to be 5.84 eV. The percentage of transmittance of γ -glycine is 94%. The observed spectrum and band gap value are in good agreement with the literature values (Narayan Bhat and Dharmaprakash, 2002; Sekar and Parimaladevi, 2009; Dillip et al., 2011; Esthaku Peter and Ramasamy, 2010; Balakrishnan et al., 2008; Dhanraj and Rajesh, 2009).

3.6. SHG efficiency measurement

The second harmonic generation efficiency of the grown γ glycine crystal was checked using the technique developed by Kurtz and Perry technique (Kurtz and Perry, 1968). The crystal was ground into powder and densely packed in a micro capillary tube. A Q-switched Nd: YAG laser beam of wavelength 1064 nm was made to fall normally on the sample cell. The emission of green light confirmed the SHG of γ -glycine crystal. Thus, the efficiency of γ -glycine crystal is 3.66 times higher than that of potassium dihydrogen phosphate (KDP). The SHG % Transmittance



300 400 500 600 700 800 900 1000 1100 1200 Wavelength (nm)

Figure 16 UV spectrum of γ -Glycine.

signal of γ -glycine crystal is 165 mV, while the standard inorganic material potassium dihydrogen phosphate (KDP) gave a signal of 45 mV for the same input energy.

0 ↓ 200

4. Conclusion

Optically good quality transparent α - and γ -glycine single crystals have been grown by the slow evaporation method at ambient temperature. The cell parameters have been determined by Single crystal XRD and powder XRD analysis and they agree well with the reported values. From the ICP analysis, it is found that the amount of zinc element is 0.73 ppm in the trace level of γ -glycine crystals prepared from 0.9 M concentration of zinc acetate. The FTIR spectra reveal the various functional groups present in the grown crystals. The UV-Visible–NIR transmission spectrum of γ -glycine shows a wide transparency window between 358 and 1098 nm with an energy gap of 5.84 eV, making it a potential candidate for NLO applications. DSC thermogram of γ -glycine shows a phase transition from γ - to α -glycine at around 187.3 °C. The SHG efficiency of the grown γ -glycine crystals is 3.66 times that of potassium dihydrogen phosphate (KDP) sample, which indicates the suitability of γ -glycine crystals for applications in nonlinear optical devices and optoelectronic devices.

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