Synthesis, Growth and Characterization of a novel Organic crystal Glycine Potassium Iodide (GPI)

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ABSTRACT

Glycine Potassium Iodide (GPI) is a new organic crystal has been synthesized and single crystals have been grown from its aqueous solution by Slow Evaporation Solution Technique(SEST). The solubility of the material was measured at various temperatures in de-ionized water. Good quality of crystal of size 32x18x15 mm³ has been harvested over a period of 48days. The grown crystals were characterized by Single crystal X-ray diffraction studies shows that GPI belongs to hexagonal system and identify the crystalline nature. The grown crystals have been subjected to powder X-ray diffraction to identify the intense peaks on various planes. The UV –VIS- NIR Spectrum of the grown GPI crystals shows less optical absorption and good transmittance in the entire visible region enabling its use in optical applications.

Key Words: SEST, solubility, Single crystal X-ray diffraction, Powder X-ray diffraction and UV - VIS - NIR Spectroscopy.
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INTRODUCTION

Organic materials attract much interest to chemists, material scientists and optical physics because of their superior performance with respect to NLO properties such as the large NLO efficient, ultrafast nonlinear response time, and high optical damage threshold [1-5].

Amino acids and their complexes belong to a family of organic materials that have been considered for photonic applications [6]. Out of 20 amino acids glycine is the simplest of all. Glycine family crystals have been subjected to extensive research by several researches for their efficient NLO properties [7-10].

In the present work, a systematic investigation has been carried out on Glycine Potassium Iodide (GPI) single crystal by slow evaporation solution growth technique. The grown crystal were

characterized by single crystal X-ray diffraction analysis, Powder X-ray diffraction analysis and UV-Vis-Spectroscopy analysis are reported in this paper.

MATERIALS AND METHODS

Experimental

All reagents used in the synthesis were of analytical grade. The crystal determination was performed of a Bruker APEX II CCD area detector diffractometer equipped with graphite-monochromatized MoKα radiation (λ = 0.7103 Å). Powder X-ray diffraction analysis was carried out with help of an instrument SEIFERT,JSO – DEBYEFLEX 2002, Germany(λ = 1.540598Å Cu, reflection scan mode, scintillation counter detector). UV - VIS – NIR transmission spectrum of crystal was obtained at room temperature on a Spectrometer with a wide wavelength range of 200-2500nm. Crystal used in this test was polished before hand and the thickness of the crystal was 1mm**.**

Synthesis and Crystal growth

The starting materials for synthesis were of AR grade. Glycine and Potassium Iodide were taken in 1:1 stoichiometric ratio. The amount of Glycine salt was calculated and dissolved in deionized water for one and half hours to get complete dissolution. Then the appropriate amount of Potassium Iodide was added to the solution. The resultant solution was continuously stirred for 4 to 5 hours. The chemical reaction is as follows:

$$CH_2NH_2COOH + KI\text{----------}\rightarrow C_2H_5NO_2.KI$$
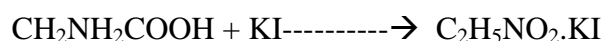
The complete dissolved solution was filtered using micro filter paper and taken in a Petri dish. It was optimally closed using a perforated polythene paper and kept in undisturbed conditions. The solution was allowed to evaporate at room temperature. After a growth period of 48 days, a well developed and optically transparent GPI single crystal of dimension 32x18x15 mm^3 was harvested and the photograph of as grown single crystal of GPI was presented in fig.1.

Fig.1 – Photograph of as - grown crystal of GPI

RESULTS AND DISCUSSION

Characteristic studies
1. Solubility Studies
The solubility of GPI in double deionised distilled water was determined in the temperature range 30 – 55°C in steps of 5°C and the results were plotted on the solubility diagram(fig. 2). From the graph, it is observed that the solubility of GPI sample in water increases as the temperature increases and thence the title compound has positive temperature coefficient. The results indicate that there is a positive slope and solubility of GPI in water is sufficient for the growth of good quality single crystals of GPI in reasonable size.
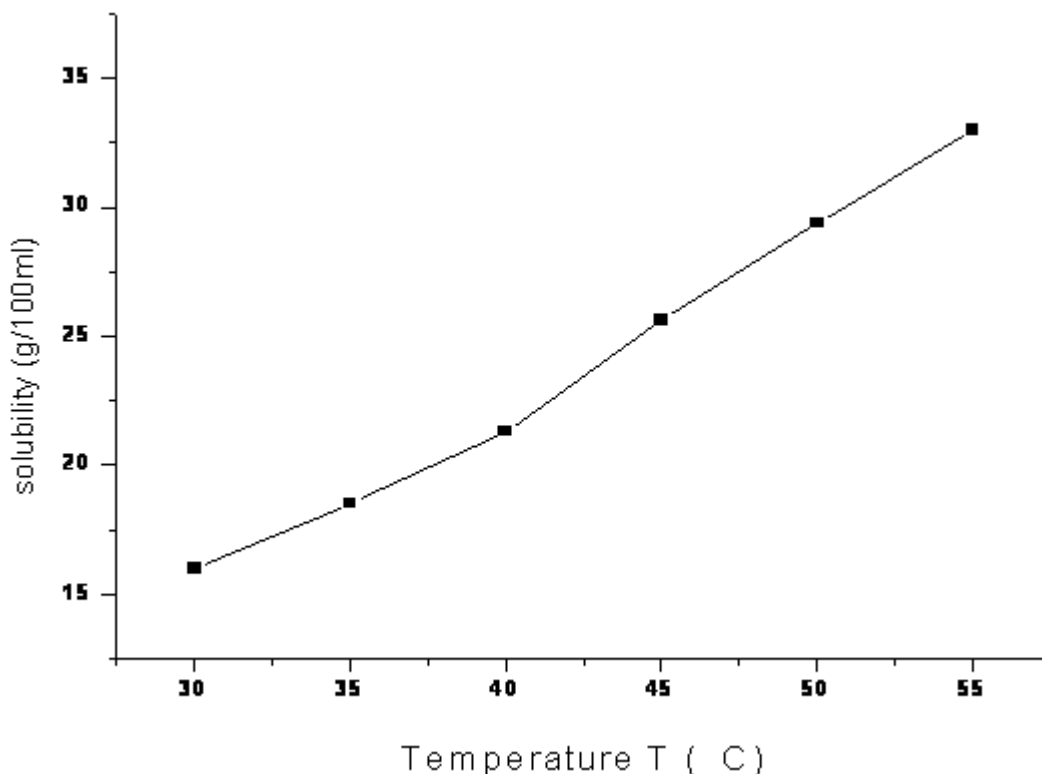

Fig. 2   Solubility curve of GPI

**Table 1 : Unit cell information--Glycine Potassium Iodide (GPI)**

Parameter	Value
Cell axes (Å)	
a	6.9876(67)
b	6.9876(67)
c	5.4499(52)
Cell angles (deg)	
α	90.000(0)
β	90.000(0)
ν	120.000(0)
Cell Volume (Å^3)	230.45(4)
Crystal System	Hexagonal
Space group	P65
Density (g.cm^{-3})	1.033
Z	1
Abs. Coeff.(mm^{-1})	0.115

2. Single crystal X-ray diffraction

Single crystal X-ray diffraction study was carried out on the as grown GPI single crystal. The present study shows that GPI crystallizes in a trigonal system. Its space group is P65.The unit cell parameters and crystallographic data are listed in Table 1.

3. Powder X-ray diffraction

The purified samples of the grown crystals have been crushed to a uniform fine powder and subjected to a powder X-ray diffraction using a powder X-ray diffractometer. The K_α radiations (λ = 1.540598A) from a copper target were used. The specimen was scanned in the reflection mode in the 2θ range 10 – 70°. Fig. 3 represents the indexed powder diffractogram for the grown crystal of GPI. It was found that the intense peak at 2 θ =14.72° The sharp peaks found in spectra shows good crystallinity of the grown crystals. The well defined Bragg's peaks at specific 2θ angles show the high crystallinity.
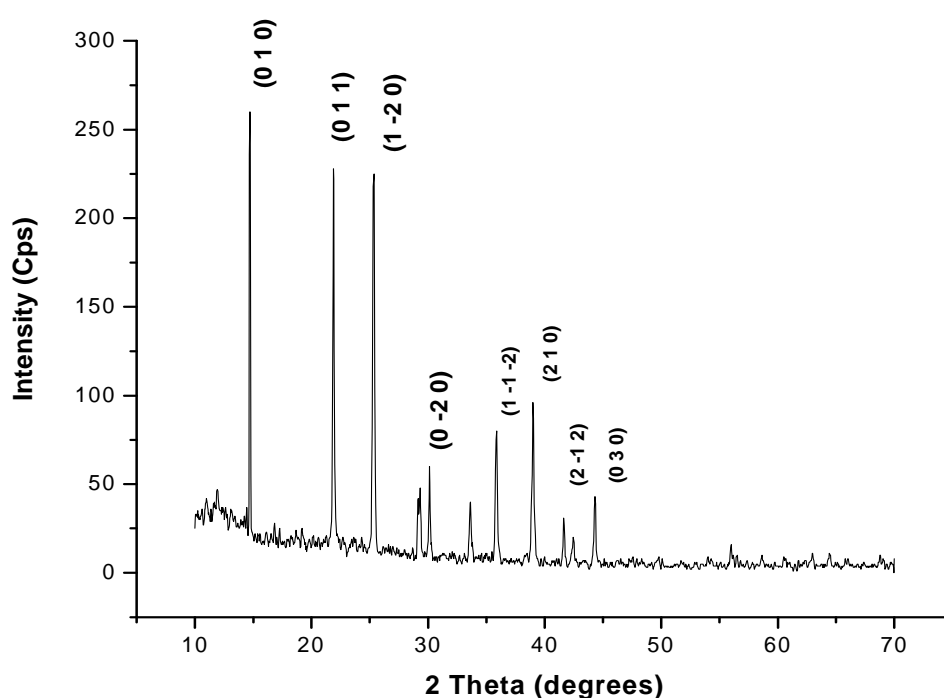


Fig.3   Powder XRD pattern of GPI

4. Optical absorbance of GPI

The optical absorption spectrum of GPI single crystal was recorded. The spectrum was recorded in the range of 200 – 2500 nm is shown in the fig 4 . The lower cut-off wavelength(λ) of the GPI crystal was found to be at 341 nm. Using the relation E_g = 1240/λ, the band gap energy was found to be 3.64 eV. The optical absorption spectrum shows that absorption was very less in the entire visible region and part of IR region.

The value of E_g also be determined as follows. Draw a graph between $(\alpha E)^2$ Vs E as shown in figure 5. Draw a tangent of the curve, it meets the X – axis at a point which gives the value of E_g. From the graph, the value of E_g is found to be 3.61 eV.
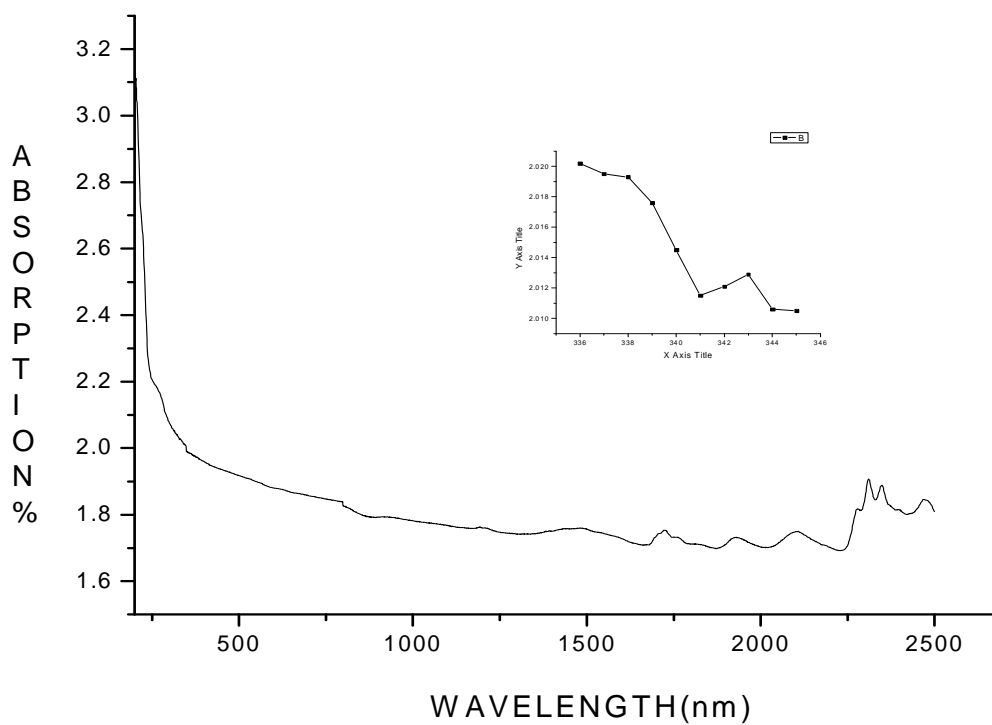
Fig. 4   UV – Vis – NIR spectrum of GPI crystal.
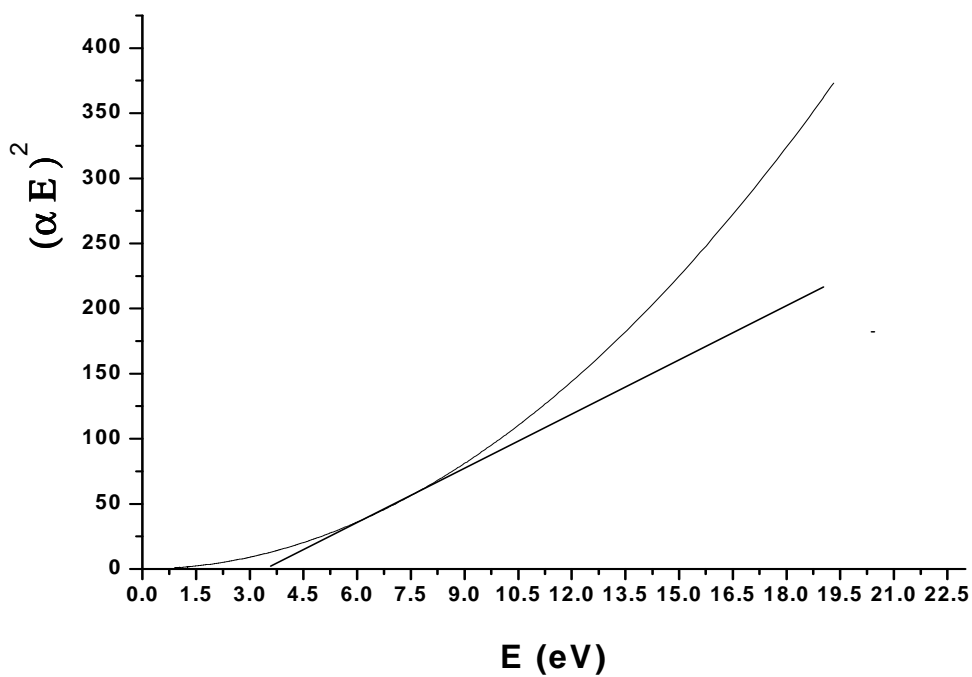
Fig. 5   Eg Calculation of GPI Crystal

CONCLUSION

Optically transparent organic crystals of GPI to the size of 32x18x15 mm^3 have been grown by slow evaporation solution growth technique at room temperature. The solubility curve of the crystals shows that it has positive temperature coefficient. The grown crystal was characterized by single XRD analysis which reveals the crystal structure has hexagonal and to deduce unit cell parameters. The powder XRD analysis reveals that the sharp peaks found in spectra shows good crystalinity of the grown crystals. The optical absorption study reveals high transparency of the crystal with a UV cut off wavelength of 341 nm and the band gap energy of the crystal was found to be 3.64 eV.

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