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Synthesis, growth and characterization studies on non linear optical crystal-Trisglycine Zinc Chloride

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ABSTRACT

A new semi organic non-linear optical single crystal of Trisglycine Zinc Chloride (TGZC) has been grown by slow evaporation solution growth technique. The crystal system and lattice parameters crystals were determined from the single crystal X-ray diffraction analysis. Fourier transform infrared (FTIR) studies confirm the various functional groups present in the grown crystal. The transmittance and absorbance of electromagnetic radiation is studied through UV- Visible spectrum. The mechanical property of grown crystals has been analyzed by Vicker's microhardness method. The second harmonic generation test has been confirmed by the Kurtz powder technique.

Key words: Crystal growth, nonlinear optical material, solution growth, X-ray diffraction.

INTRODUCTION

Nonlinear optical materials (NLO) exhibiting second harmonic generation have been great demand over the last few decades due to technological importance in the fields of optical communication, signal processing and instrumentation[1-3]. Most of the organic NLO crystals usually have poor mechanical and thermal properties and are susceptible for damage during processing even though they have large NLO efficiency. Also it is difficult to grow larger size optical quality crystals of these materials for device applications. Purely inorganic NLO materials have excellent mechanical and thermal properties but possess relatively modest optical nonlinearity because of the lack of extended π-electron delocalization[4,5]. Hence it may be useful to propare semi-organic crystals which combine the positive aspects of organic and inorganic materials resulting in useful NLO properties. In semi-organic materials, the organic ligand is ionically bonded with inorganics. These crystals have higher mechanical strength, chemical stability, large nonlinearity, high resistance to laser induced damage, low angular sensitivity and good mechanical hardness[6,7].

Amino acids are interesting materials, as they contain proton donor carboxyl acid(-COOH) group and proton accepter amino (-NH_2) group which provide the ground state charge asymmetry of the molecule required for second order nonlinearity[8,9]. Literature reveals that amino acid impurities have improved the material properties[10]. Glycine is the simplest amino acid. It is reported that glycine addition has enhanced the nonlinear optical property of Zinc–Tris–thiourea sulphate[11]. Glycine hydrofluride[12], Glycine barium dichloride[13], Glycine thiourea[14] and Glycine sodium chloride[15] are some of the examples which proved applications in the field of Nonlinear optics.

In this paper, we report the growth of Trisglycine Zinc chloride (TGZC) single crystals by slow evaporation solution growth technique and to study its characterization.

MATERIALS AND METHODS

Synthesis

The starting material was synthesized by taking Trisglycine and Zinc Chloride in 3:1 ratio. The required amount of starting materials for the synthesis of Trisglycine Zinc Chloride (TGZC) salt was calculated and dissolved in deionized water. Zinc Chloride was then added to the solution slowly by stirring. The prepared solution was allowed to dry at room temperature and the salt were obtained by slow evaporation technique. The purity of the synthesized salt was further improved by successive recrystallization process.

2.1.Crystal growth

The saturated solution of TGZC was prepared at room temperature from the recrystallized salt. The solution was then filtered twice to remove the suspended impurities and allowed to crystalline by slow evaporation technique at room temperature. A good optical transparent crystal harvested in a growth period of four weeks is shown in Fig.1.

Characterization

The grown crystals of TGZC were confirmed by single crystals X-ray diffraction analysis using ENRAF NONIUS CAD4 diffractometer. The functional groups were identified by Fourier transform infrared studies using Perkin Elmer spectrum RXI FTIR spectrometer in the range of 400 – 4000 cm^{-1}. The optical properties of the crystals were examined by using Lambda 35 UV – Vis spectrometer. The thermal behavior of the grown crystal was tested by SDTQ600 V8.3 thermal analyzer. The microhardness measurement of TGZC crystal were carried out using a Leitz Weitzler Vicker's microhardness tester. To confirm the nonlinear property, Kurtz powder SHG test was performed in the TGZC crystals.

3.1. Single Crystal X-ray diffraction analysis

The grown crystals were subjected to single crystal X-ray diffraction analysis to confirm the crystalline and also to estimate the lattice parameters by employing Enraf Nonis CAD4 diffractometer. From the single crystal X-ray diffraction data, it is observed that the TGZC crystal is hexagonal in structure. The lattice parameters were observed to be a=7.024 Å, b=7.013 Å , c=5.479 Å, $\alpha= \beta=90^0$, $\gamma=120^0$ and V=233.5$Å^3$.

3.2. Fourier transform infrared analysis

The FTIR spectrum of TGZC revealed at room temperature in the range of 400 – 4000 cm^{-1} is shown in Fig.2 and all the functional group assignment are summarized in Table.1. The absorption due to Carboxylate group of free Trisglycine is observed at 504.2, 892.8 and 1614 cm^{-1} respectively. In TGZC, these peaks are shifted to 511.8, 883.2 and 1636.8 cm^{-1} respectively. Similarly, the absorption peaks due to NH_2^+ group of free glycine are observed at 1131 and 1505 cm^{-1} respectively. In TGZC, these peaks are shifted to 1116.6 and 1401.8 cm^{-1} respectively from a comparison of the spectra with that of glycine [16].

3.3. Microhardness studies

Microhardness measurements were carried out using Leitz Weitzlert hardness tester fitted with a diamond indentor. The mechanical behavior of the TGZC crystals was analyzed using Vicker's microhardness test at room temperature. The selected surface of the grown crystals were lapped, polished, washed and dried. Hardness measurements were taken for applied load s varying from 25 to 100 gm keeping the indentation constant at 10 sec. for all cases. The Vicker's hardness number (VHN) of grown crystals were calculated using the relation Hv=1.8544 P/d^2 $kg.mm^{-2}$, where Hv is VHN, P- is the applied load in kg, d- is the average diagonal length in mm of the indentation mark. A graph is plotted between hardness number (Hv) and applied load as shown in Fig.3. From the graph, it is found that the hardness value increase with the increase of load. This might be due to the release of internal stress generated locally by indentation[17].

3.4. Second Harmonic Generation Test

The second harmonic generation (SHG) test on the TGZC crystal was performed by Kurtz powder SHG method[18]. The powdered sample of crystal was illuminated using the fundamental beam of 1064 nm from Q-switched Nd:YAG laser. Pulse energy 4 ml/pulse width of 8 ns and repetition rate of 10Hz were used. The second harmonic signal generated in the crystalline sample was confirmed from the emission of green radiation of wavelength 532 nm collected a monochromator after separating the 1064 nm pump beam with an IR–blocking filter. A photomultiplier tube is used as a detector. It is observed that the measured SHG efficiency of TGZC crystal was 0.5 times that of potassium dihydrogen phosphate (KDP).

Fig.1. As grown TGZC crystals

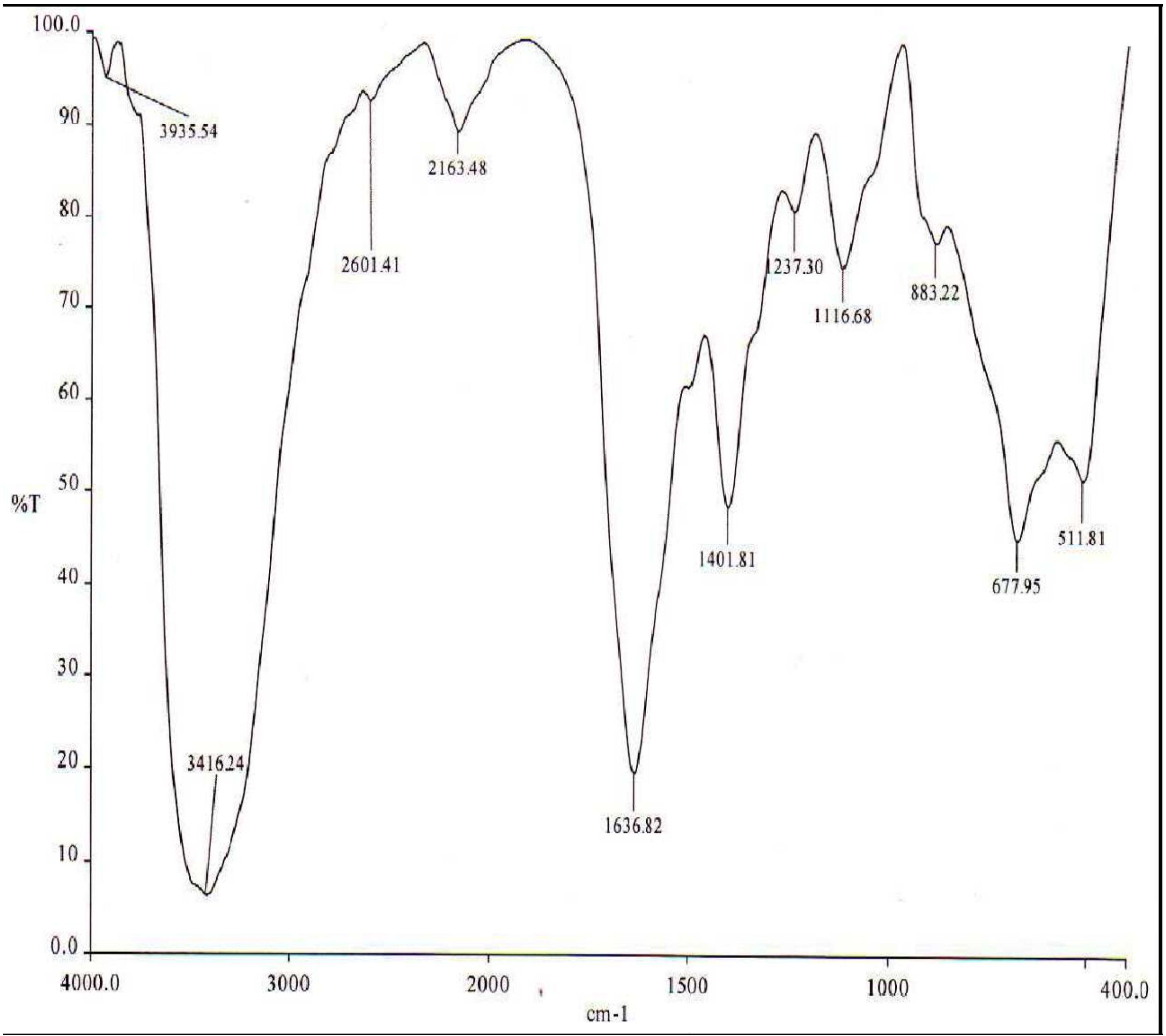

Fig.2. FTIR spectrum of TGZC crystals

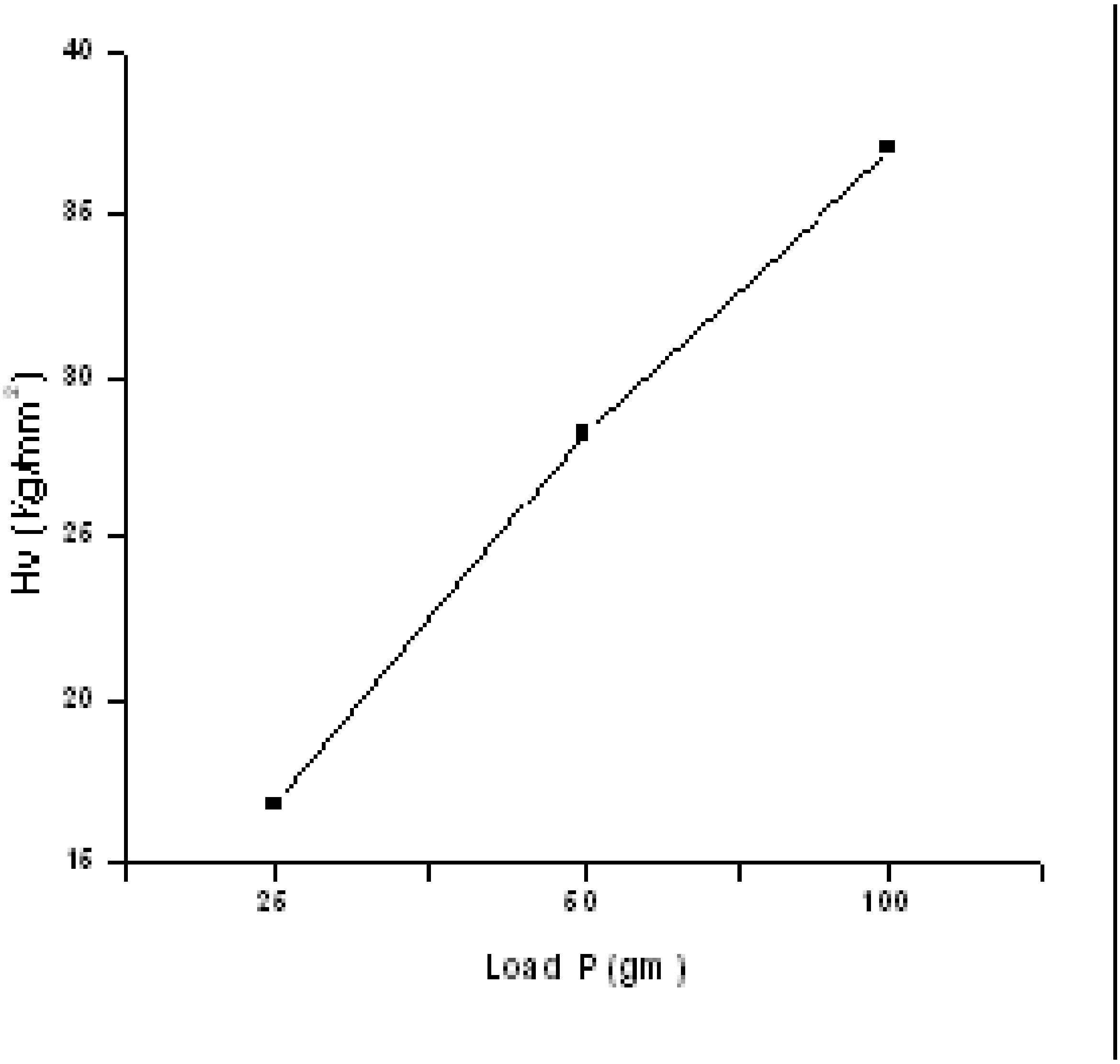

Fig.3. Microhardness Curves of TGZC crystals

Table. 1. FT-IR assignments for TGZC crystals

Wavelength cm^{-1}		Assignments
Glycine[16]	**TGZC**	
504.2	511.81	Corboxylate group
892.8	833.22	Corboxylate group
1131	1116.68	Absorption due to NH_2^+
1384	1237.30	COO group
1505	1401.81	Absorption due to NH_2^+
1614	1636.82	Corboxylate group
2837	2601.41	CH_2 group

CONCLUSION

Single crystal of TGZC, a new semi organic non linear optical material has been grown in solution growth technique. The lattice parameters were found by single crystal X-ray diffraction technique. The FTIR spectrum reveals that the functional groups of the grown crystal. Mechanical property of the grown crystal has been studied by microhardness test and noticed that there is an increase of microhardness number. The NLO behavior of the

TGZC was confirmed by the Kurtz – Perry powder SHG technique. The powder second harmonic generation efficiency measurement shows the grown TGZC crystal having 0.5 times higher nonlinear optical efficiency than potassium dihydrogen phosphate (KDP).

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