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ChemTech

International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN : 0974-4290
Vol.6, No.3, pp 1640-1642, May-June 2014

ICMCT-2014 [10th – 12th March 2014]
International Conference on Materials and Characterization Techniques

Growth and Characterization of NLO active Glycine Lithium Sulphatesingle crystals

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Abstract- Extensive studies have been made on the synthesis and crystal growth of nonlinear optical (NLO) materials over the past decade because of their potential applications in the field of telecommunications, optical signal processing, and optical switching. Glycine Lithium Sulphate(GLS), a nonlinear optical material, was grown by temperature reduction method from aqua solution. The grown crystal crystallized in orthorhombic system with $Pna2_1$ space group. Powder X- ray diffraction analysis confirmed the crystalline nature of the grown crystal. FT-IR spectrum was used to identify the presence of functional groups of the compound. TG and DTA studies of grown crystal show three stages of weight loss of the material. Powder Second Harmonic Generation nature of the grown crystal was measured by Kurtz technique.
Key words–GLS, FT-IR, XRD, NLO.

Introduction

Crystals of many amino acids and their complexes are reported to be showing nonlinear optical properties and capable of generating second harmonics because they haveunit cells with no centre of symmetry [1, 2].Glycine is the smallest of the amino acids. It is ambivalent, meaning that it can be inside or outside of the protein molecule. In aqueous solution at or near neutral pH, glycine will exist predominantly as the zwitterion. Complexes of amino acids with inorganic salts are promising materials for optical second harmonic generation (SHG), as they tend to combine the advantages of the organicamino acid with that of the inorganic salt [3]. Glycine molecule can exist in zwitterionic form and hence it is capable of forming compounds with anionic, cationic and neutralchemical compounds. Thus a large variety of glycine coordinated compounds can be formed. The growth of semi-organic crystals has nowadays come into prominence to overcome organic NLO materials such as environmental stability, poor chemical and mechanical stability, red-shift of the cut-off wavelength, low laser damage thresholds and poor phase matching.The semi organic crystals have some advantages such as higher second order optical non-linearities, short transparency cut-off wavelength and stable physico-chemical performance over the traditional inorganic and organic crystals. Since there is a large demand for crystalsbecause of the revolution in electronic industries, it isrequired to synthesize new NLO materials and improvethe properties of the existing materials. In this work, the growth and characterization of glycine lithium sulphate by temperature reduction method from aqua solution were reported.

Experimental Procedure[4-8]

The analytical grade of Glycine and Lithium Sulphate in the stoichiometric ratio was used to grow single crystals. The chemicals were dissolved in deionized water and stirred well for about 5 hours.Saturated solution of GLS was prepared. The beaker containing solution was kept at 30°C in the constant temperature bath. The temperature was lowered at the rate of 0.5°C/day. The first tinycrystal was observed at the bottom of the beaker due to slow evaporation of the solvent. The solution is stirred well for 2 hours for recrystallization.Then the solution were slowly evaporated at a temperature of 25°C over a period of three months., yielded crystals up to several millimeters in diameter (Figure 1).

$$C_2H_5NO_2 + Li_2(SO_4) \longrightarrow Li_2(SO_4)(C_2H_5NO_2)$$

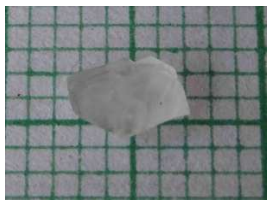

Fig 1. Photograph of as grown GLS single crystal

Results and Discussion:

Powder X- ray Diffraction:

Powder X – ray DiffractionStudies carried out to identifythe structureof the grown crystals. The powder sample of GLS was subjected to powder XRD analysis and the recorded pattern is shown in figure2. This sample was scanned for 2θ values from 10° to 45°. The Lattice parameters were calculated by Least Square fit method and compared with reported studies. The crystal is in orthorhombic system with $Pna2_1$ space group.Powder X ray diffraction analysis confirmed crystalline nature of the grown crystal. The lattice parameters are a = 5.029Å, b =7.635Å, c =16.389Å.

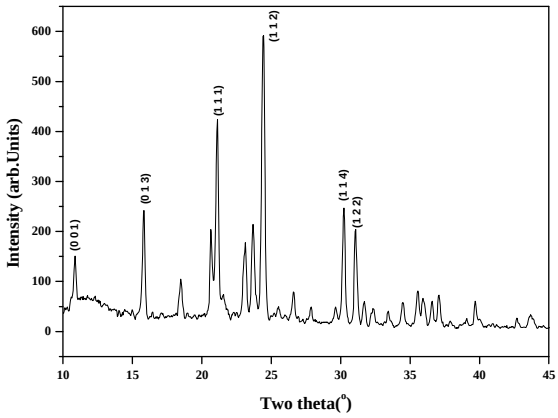

Fig 2. Powder XRD pattern

FT-IR Spectrum

FT-IR studies of GLS crystal were carried out in wave number range from 4000 to 450 cm^{-1}. FT-IR spectrum of GLS crystal is shown in Figure3. The O-H asymmetric stretching produces the characteristic peak at 3202 cm^{-1}. Presence of band in the region 2700–3000cm^{-1} is the characteristic region for the identification of C–H stretching vibrations. The scaled vibrations are around 2969 cm^{-1}. It is inferred from the spectra that the peaks at 1645 $cm^{-1}$and 1165 cm^{-1} are due to the C=O stretching of carboxylic group.The C-N band is observed at 1310 cm^{-1}. The C-N stretching and C-C stretching modes are observed at 995 cm^{-1} and 649 cm^{-1} respectively.

TGA/DTA

Single crystals of Glycine Lithium Sulphate (GLS) crystalwas subjected to thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) simultaneously using STA 409C instrument, in the nitrogen

atmosphere at a heating rate of 10 K/min. Figure 4 shows the resulting TGA and DTA traces of the crystal. The decomposition of the material starts at 250 ºC. The material is found to be thermally stable up to 250 ºC. GLS crystals show three stages of weight losses of 40.52%, 3.288 and 2.714%.The DTA trace of GLS shows that, there is a sharp endotherm matching with the decomposition of GLS.

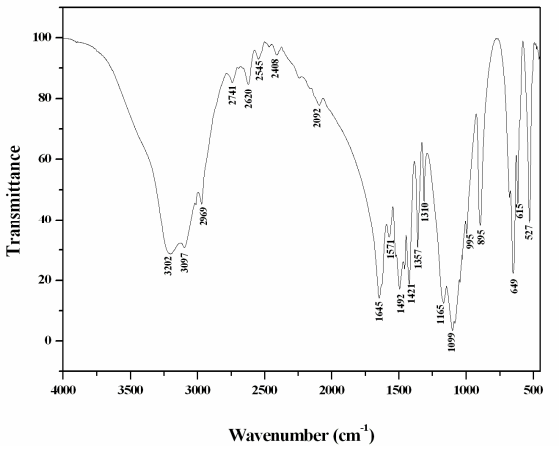

Fig 3. FT- IR spectrum of GLS

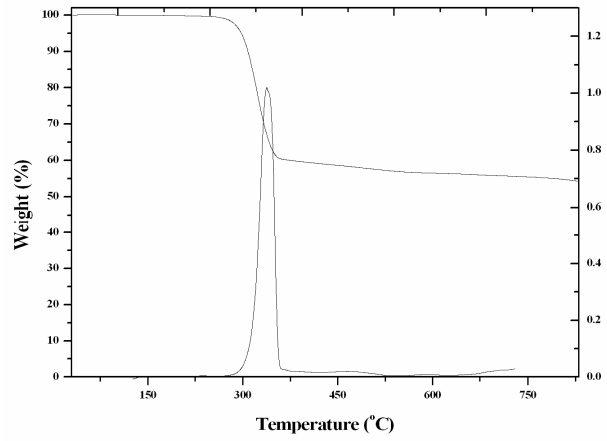

Fig 4. TG-DTA thermogram of GLS crystal

NLO Studies

The crystal was grounded into powder and packed between two transparent slides. The samples are tested using Q switched Nd-YAG Laser of wavelength 1064 nm,Quanta ray series supplied by Spectra Physics USA and coherent Molectronpowermeter USA with input power 0.68J. The output SHG was detected by the emission of green flash from the GLS crystals which confirms the material is non linear active nature.

Conclusion

Glycine Lithium Sulphate was synthesized and grown by temperature reduction method from aqua solution. The grown crystal is orthorhombic structure with non centrosymmetry and its space group $Pna2_1$, confirmed by Powder XRDanalysis.FT-IR isused to identify the functional groups of the GLS. TG and DTA studies of GLS show three stages of weight loss of the material. Non Linear property of GLS is studied by Kurtz Perry Technique,which is comparable with the SHG of KDP.

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