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Synthesis and characterization of L-Cystine Sodium Nitrate

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ABSTRACT

In the present work, L-Cystine Sodium nitrate has been grown by a slow evaporation method. The Powder X-ray diffraction analysis was confirmed the crystalline nature. The FT-IR spectral analysis confirmed the various functional groups present in the grown crystal. The good optical property of a crystal was determined by UV-Vis transmittance spectrum. The TGA –DSC analysis confirmed that the crystal was stable up to 231.21^{o}C. NLO studies of the grown L-Cystine Sodium nitrate crystals was determined using Kurtz Powder technique; which is 0.65 times that of KDP.

1. Introduction

Non-linear optical (NLO) materials showing wide range of applications in the field of telecommunication, optical information, storage devices, etc. These materials have large non-linearity, high resistance, too large induced damage, low angular sensitivity and good mechanical hardness[1-5].Amino acid nonlinear optical materials are often formed by weak Vander Waals and hydrogen bonds and hence posses high degree of delocalization. The basic structure of organic NLO materials is based on the π bond system. Due to the overlapping of π orbital, the delocalization of electronic charge distribution leads to a high mobility of electrons [6-7]. Amino acids are bifunctional organic molecules that contain both a proton donor carboxylic (COO-) and proton acceptor amino (NH_2) group. This dipolar nature of amino acids shows peculiar physical and chemical properties. L-Cystine is sulfur containing protenogenic amino acids framed by oxidation of two Cystine molecules which are covalently linked to make a disulfide bond. In the L-Cystine molecule, the functional groups such as NH2 and COOH have a strong tendency to coordinate with inorganic cations and metals [8-11]. Sodium Nitrate was a potential inorganic material having wide range of applications such as electro optic modulator, harmonic generators and parametric generators [12-14].

2. Experimental Procedure

2.1. Synthesis

Slow evaporation of L-Cystine and hydrochloric acid are taken in equimolar ratio in aqueous solution. The solution was stirred continuously using magnetic stirrer till a clear solution was obtained. To the clear solution accurately weighed sodium nitrate was added slowly and stirred well using a magnetic stirrer to get the homogenous mixture. The clear solution was filtered off using whatmann filter paper and the solution was kept undisturbed at room temperature. The growth of the crystal is noted periodically. The growth period was about 25 days. The harvested crystals were observed to be transparent, colourless and non-hygroscopic. The grown crystal was shown in Fig.1.

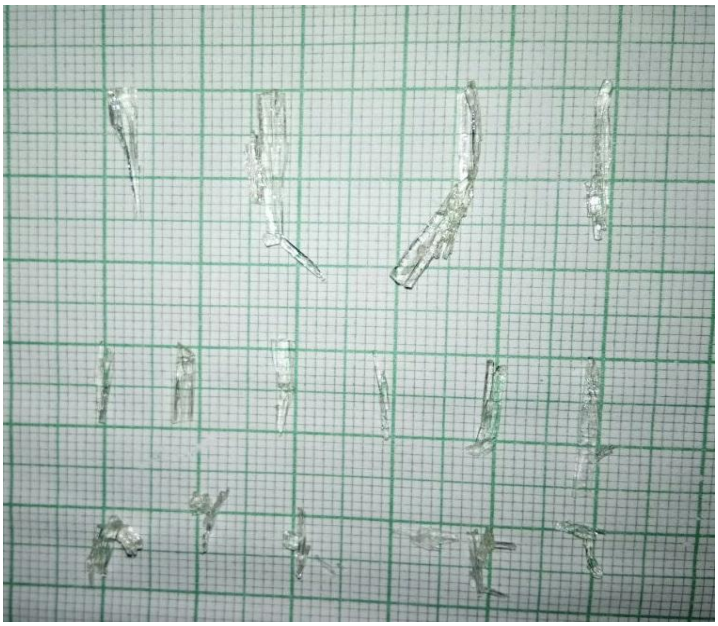

Fig.1. Morphology of L-Cystine Sodium Nitrate.

2.2. Measurements

Bruker FTIR Spectrometer under the range of 400- 4000cm^{-1} was used to record the FTIR spectrum for the identification of functional groups. Lamda 25 model UV-Vis-NIR spectrometer in the range 190-800nm is used to record the UV-Vis spectrum. Using SDT Q600V20.9 TGA analyzer the thermo gravimetric and differential thermal analysis were carried out in the temperature range between 0 °C and 1000° C at a heating rate of 10 °C /min under nitrogen atmosphere. The Kurtz Perry Powder method with a Nd:YAG laser source of wavelength =1.064nm is used to determine the SHG efficiency.

3. Results and Discussion

3.1 .FT-IR Studies

Fourier Transform Infrared Spectroscopy was recorded in the range of 400-4000cm-1 using Bruker FT-IR Spectrometer and the recorded spectrum is shown in Fig.2. Vibrational spectroscopy provides an important tool to understand chemical bonding [15]. The strong absorption peaks at 2883.73cm^{-1} is due to C-H stretching. The O-H stretching vibration is attributed at 2631.60cm^{-1}. The functional peak observed at 1718cm^{-1} is due to –C=O vibration. The frequencies observed at 1592& 1575cm^{-1} are attributed to N-H bending vibrations. The N=O stretching vibration is attributed with 1393cm^{-1} -1353cm^{-1}. The C-C-N bending vibration is observed at 1156.11cm-^{1}. The C-S sulfides vibration is attributed at 579.64cm-^{1}. The assignments of the observed vibrational frequencies are listed in Table.1.

Table.1. FT-IR spectrum of L-Cystine Sodium Nitrate crystal

Wavenumbers(cm-1)	Assignments
2883.73	C-H stretching
2631.60	O-H stretching
1718.48	C=O stretching
1592.41 & 1575.38	N-H bending
1393.95 & 1353.91	N=O
1156.11	C-C-N bending
579.64	C-S sulfides

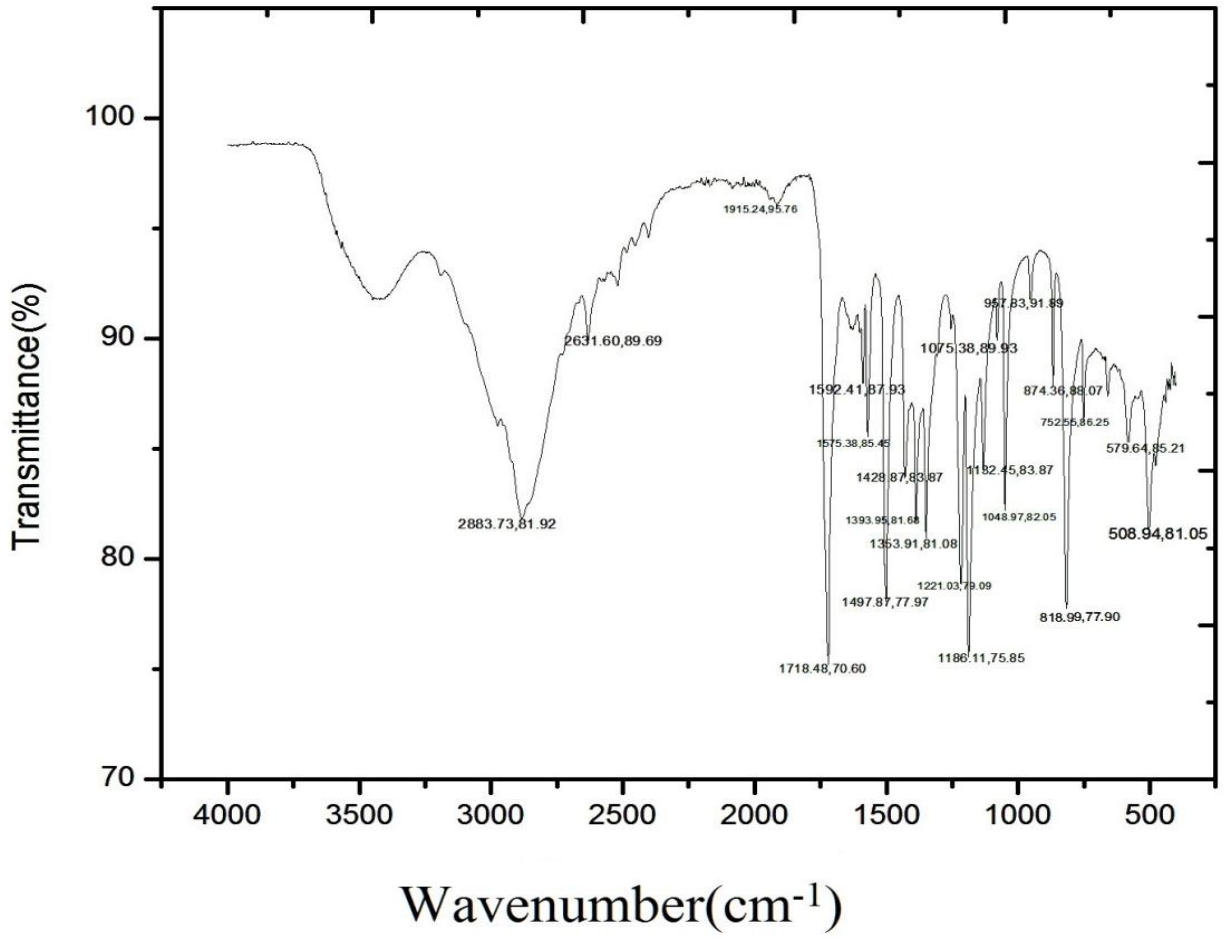

Fig. 2.FT-IR spectrum of L-Cystine Sodium Nitrate

3.2. XRD Analysis

L- Cystine sodium nitrate crystals were crystals were powdered and X-ray powder diffraction data were collected at room temperature. An automatic high resolution X-ray diffractometer is used for XRD analysis. The sample was grinded into fine powder and made to expose in a beam of X-ray. High intense beam of X-rays with a scanning speed 0.001°per min of wavelength λ=1.5406A° is used to X-ray analysis. The observed diffraction peaks were recorded and indexed using Computer program over a range of 20-80°. POWDERX refinement software is used to calculate the lattice parameter by accepting two-theta value as input and was found to be matched with the reported data and is shown in Fig.3. The collected XRD data are given in Table.2.

Table.2. Powder XRD data of L-Cystine Sodium Nitrate

2-Theta	hkl values
21.120	106
22.897	011
28.010	10 13
29.448	101
31.356	020
34.900	117
43.552	121

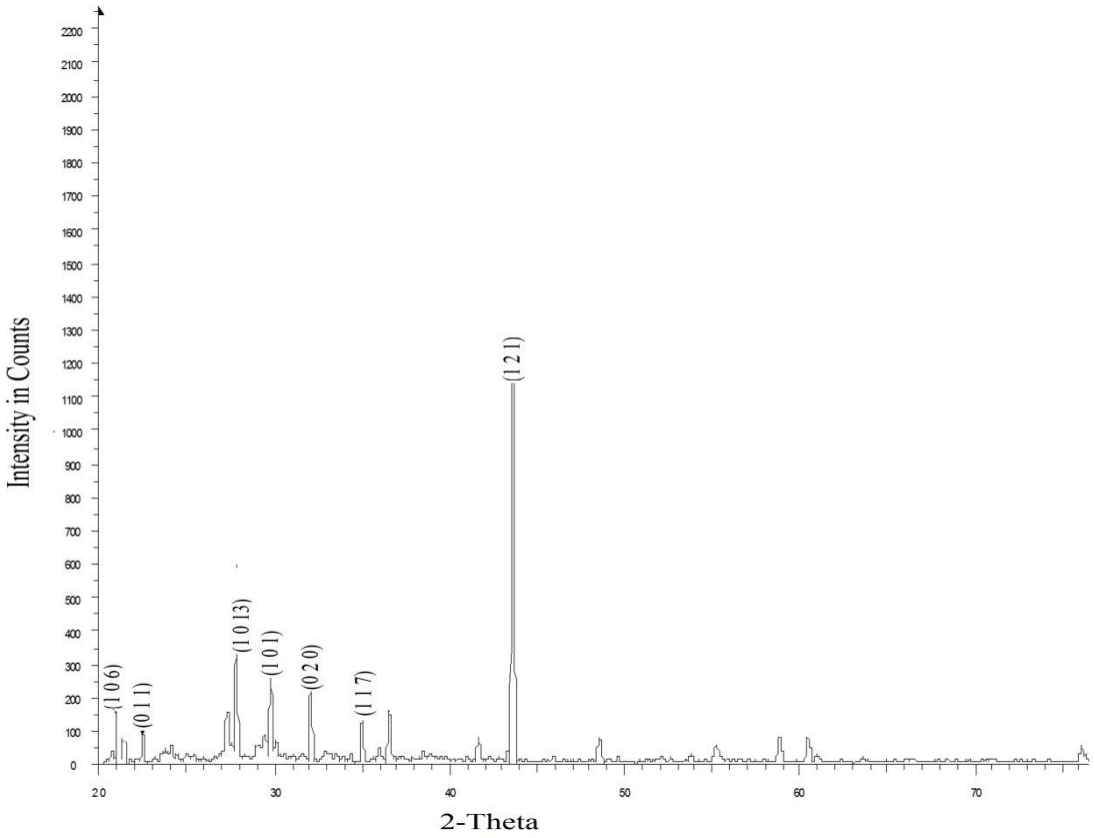

Fig.3. X-ray diffraction pattern of L-Cystine Sodium Nitrate

3.3. UV Spectral Analysis

The UV –Visible-NIR transmittance spectrum was recorded using Lambda 25 model UV-Vis-NIR spectrometer in the range 190-800nm covering the entire UV and visible region Fig.3 shows the recorded optical transmittance spectrum of L-Cystine Sodium nitrate crystal. The lower cut off wavelength is observed at 202.40nm. A complete transparency in visible region was interesting which was required for NLO applications.

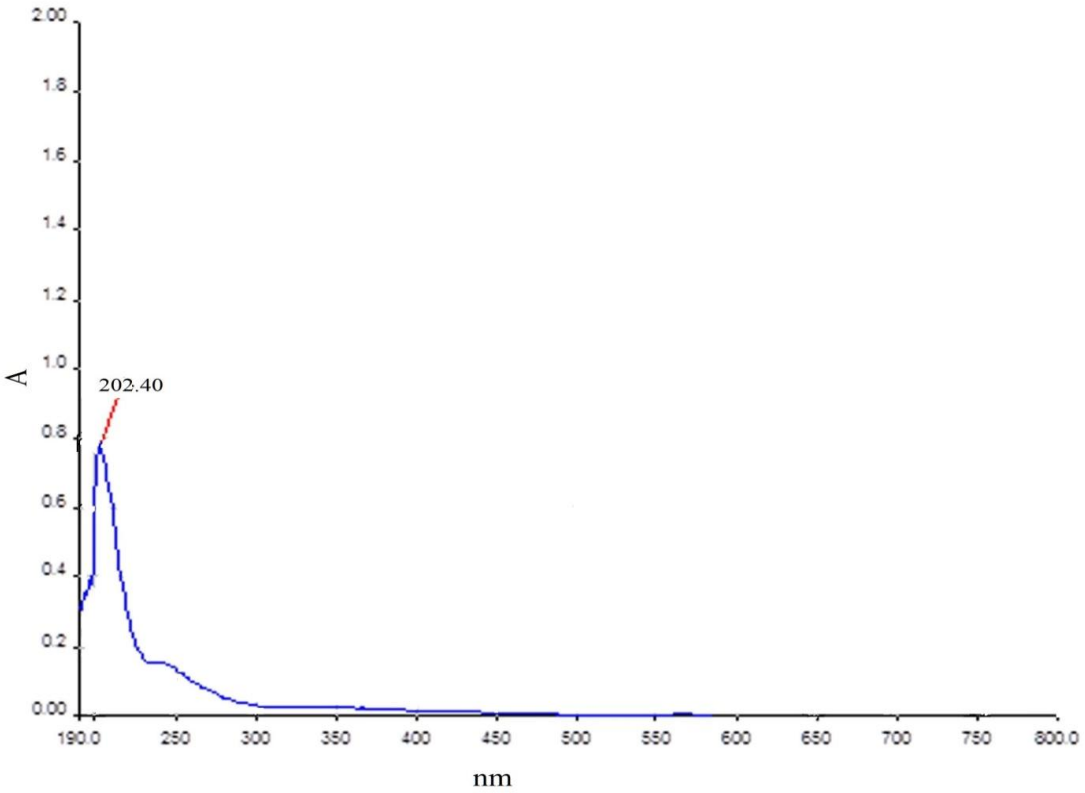

Fig. 4.UV-Visible spectrum of L-Cystine Sodium Nitrate

3.4. Thermal Analysis

Thermogravimetric and differential thermal analysis was carried out using SDT Q600V20.9 TGA Analyzer. The TG/DSC traces of L-Cystine Sodium nitrate crystallization sample are shown in Figs.5 &6 - respectively. For heating the sample and the analyses were carried out in an atmosphere of nitrogen at a heating rate of 10°C/min in the temperature range of 0°c to 1000°c. The initial mass of the material subjected to the analyses was 7.8520mg. The decomposition of L-Cystine Sodium Nitrate starts at 231.21°c and ends at 911.11°c. The DSC spectrum shows an exothermic peak at 304.42°c. The material starts to melt at 223.52C.

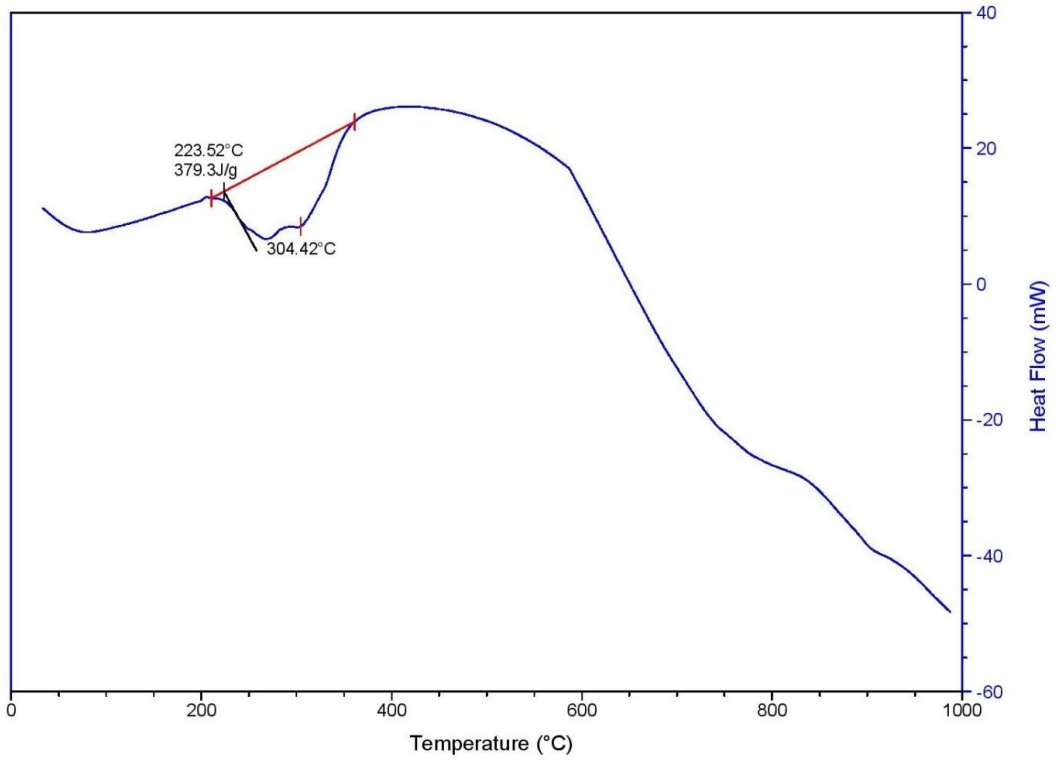

Fig. 5 TGA spectrum of L-Cystine Sodium Nitrate

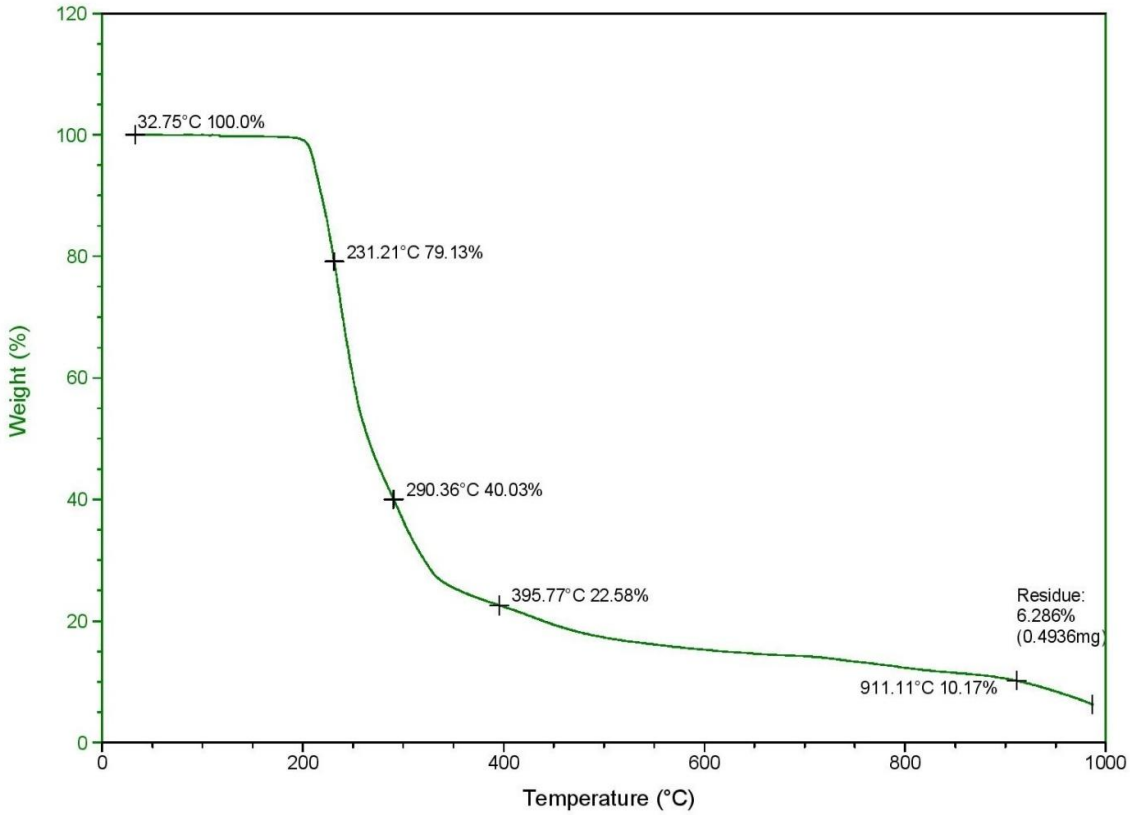

Fig. 6 DSC spectrum of L-Cystine Sodium Nitrate

3.5. NLO Studies

Taking KDP as a reference material the second harmonic generation efficiency of the grown crystal is determined by using Kurtz Perry Powder method. Q-switched laser beam of wavelength λ=1.064nm is made to illuminate the microcrystalline powder packed in the glass tube of pulse width 10ns. The photomultiplier detects the emitted radiation from the crystal and the same was recorded. Emission of green light (λ=532nm) from the sample confirms that the crystal has NLO potential. It was observed that the efficiency as 13mV and the conversion efficiency of the grown crystal are 0.65 times that of KDP crystal.

4. Conclusion

Single crystals of L-Cystine sodium Nitrate was grown by slow evaporation method. Powder XRD studies confirms that the grown crystal. FT-IR spectrum confirms the presence of C-H,C=O, N-H,N=O,C-S and C-O groups were identified. The UV-Vis –NIR spectrum shows that it has a good optical transmittance with lower cut off wavelength 202.40nm.The grown crystals thermal stability is relatively high on analyzing the TGA. By performing the NLO test using Kurtz Powder technique as Nd:YAG laser source the SHG efficiency of the grown sample is studied and thus confirmed that the grown crystal may be used for NLO device fabrications.

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