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SYNTHESIS GROWTH AND CHARACTERIZATION OF A NEW NLO MATERIAL: L-THREONINE LEAD CHLORIDE

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Abstract

A new NLO material Lead doped L-Threonine was grown from aqueous solution by a slow evaporation technique at room temperature. Crystals were characterized by X-ray diffraction analysis whose results they crystallize on orthorhombic system. The UV-Visible spectroscopic study reveals that crystal has good optical transparency and lower cut-off wavelength was found to be 330 nm. The functional group of grown crystal was found by FTIR analysis. The nonlinear optical property was confirmed by Kurtz Perry powder technique. The detailed structural analysis of the compound under progress.

Key words: Lead doped L-Threonine, XRD, UV-Visible Spectroscopy, FTIR and NLO.

1. Introduction

Materials with second order of non - linear optics have greatly attracted due to their possible applications in new technologies of optoelectronics (Madurambal *et al*., 2013; Sangeetha *et al*., 2012). Organic materials have been of particular interest, since non-linear optical responses on these materials are of microscopic origin, thus offering an opportunity to utilize theoretical models along with synthesis flexibility to design & produce new materials (Mojumdar *et al*., 2011; Jiang *et al*., 1999). Most of the organic crystals have inadequate transparency, poor optical quality and low laser damage threshold (Ravindran *et al.*, 2011). Moreover growths of large sized single crystals have excellent mechanical and thermal properties but they possess relatively modest nonlinearity. Due to the above reasons, a lot of research has been carried out on semi organic materials which have combined properties of both organic and inorganic materials (Franke *et al*., 1961). Amino acids are bi-functional organic molecules that contain both a carboxylic group as well as an amino group. In solid state, amino acid contains protonated amino group and de-protonated carboxylic group. This dipolar exhibits peculiar physical and chemical properties. Therefore the efforts have been made to grow amino acid mixed crystals in order to make them suitable for device applications. It has been shown that some complexes of amino acids with organic or inorganic acids exhibit NLO properties (Vijayaraj *et al*., 2015). This interest has been fuelled by the possibility of using them in the technological devices. It is known that the crystals grown by amino acids mixed with heavy metals exhibits good NLO properties along with second harmonic generation SHG (Nedunchezhian *et al*., 2015; Vijayaraj *et al.*, 2016). Therefore, it is

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useful to synthesize and characterize the crystals of L-Threonine complexes with lead chloride. Semi organic Lead doped L-Threonine was grown by slow evaporation method. The grown crystals were subjected to various characterizations like powder X-ray diffraction, UV, FTIR and second harmonic generation studies.

2. Experimental

Synthesis

Single crystals of Lead doped L-Threonine was grown by preparing L-Threonine and lead chloride taken in 1:1 equimolar ratio in deionised water at room temperature and stirred well to yield a homogeneous mixture of solution. The solution was filtered to remove insoluble impurities using Whatman filter paper of pore size 10 micrometers. Then, the solution of L-Threonine lead chloride was taken in a beaker with a perforated lid in order to control the evaporation rate and kept at room temperature for crystallization. Finally a well defined single crystal was obtained after 15 days by slow evaporation method. The photograph of the grown crystal of Lead doped L-Threonine is shown in Fig.1.

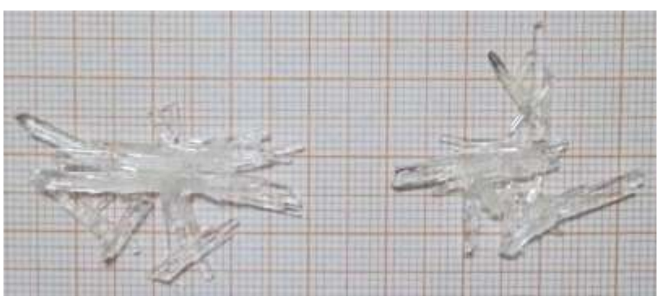

Fig – 1: Lead Chloride doped L-Threonine Crystals

Characterization Methods

The optical absorption spectra of the grown crystals were recorded using Perkin Elmer Lambda 35 model UV-Visible spectrometer in the wavelength region 190 – 1100 nm. The FTIR spectrum of Lead doped L-Threonine was recorded by PERKIN ELMER RX9 spectrometer using KBr pellet technique in the wavelength range 4000 – 400 cm^{-1}. Powder X-ray diffraction study of the grown crystal was carried out by using Bruker's X-ray diffractometer

3. Results and Discussion

Powder X-ray diffraction

Powder X-ray diffraction study of the grown crystal was carried out by using Bruker's X-ray diffractometer as shown in the Figure 2. XRD analysis has confirmed that the crystal belongs to orthorhombic with space group $P2_12_12_1$. The space group suggests that the grown material is non-centrosymmetric which fulfils the fundamental criterion for the material to exhibit NLO behaviour (Kurtz *et al*., 1968; Dixit *et al*., 2003; Puhal Raj *et al*., 2013).

UV-Vis-NIR spectral analysis

The UV-Visible transmission spectrum is very important for optical material because of its wide transmittance window. The high transparency was confirmed from the recorded spectrum and it was observed that there was no significant absorption in the range 330 – 1100 nm. There is an advantage in the use of amino acids, where the absence of strongly conjugated bands leads to a wide transparency range in the visible and UV spectral regions. The lower cut-off wavelength was found to be around 330nm which combined to good transparency attests to the usefulness of Lead doped L - Threonine material for optoelectronic application (Bright *et al*., 2010).

FTIR Analysis

The FTIR spectra of the sample were recorded in the frequency region of 4000 - 400 cm^{-1} using PERKIN ELMER RX9 FTIR Spectrometer to confirm the vibrational structure of grown crystalline compound. Fig. 4 shows the FTIR Spectrum of Lead doped L-Threonine. The broad absorption of medium intensity peaks at reveals the COO^- symmetric vibration. The peak at 2861.23 cm^{-1} indicates the C-H stretching vibration. The NH_3^+ rocking vibration was 3289.67cm^{-1} and 2921.41cm^{-1} was assigned due to

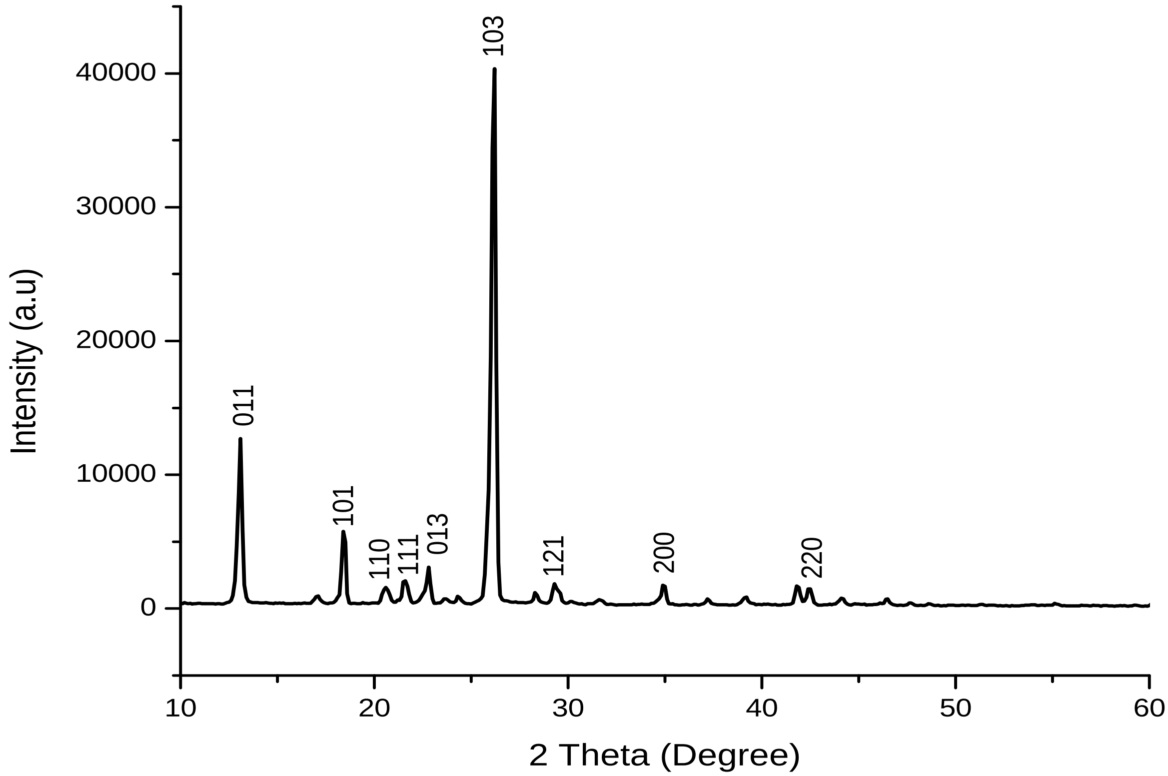

Fig. 2 X-ray diffraction pattern of Lead Chloride doped L-Threonine

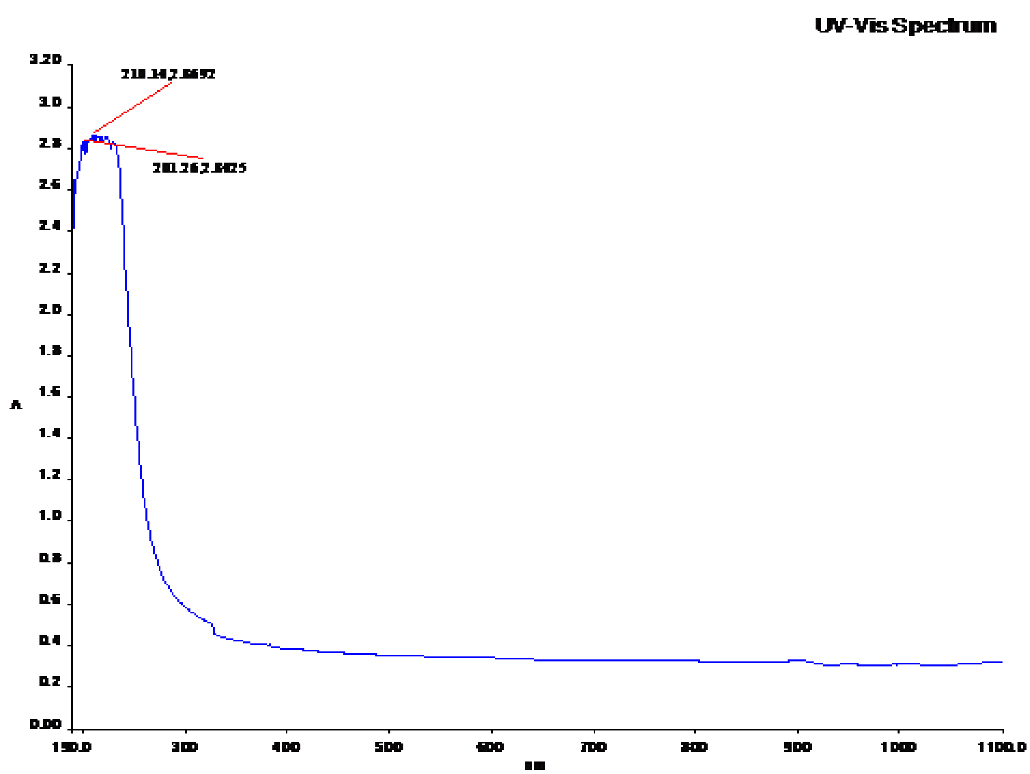

Fig. 3 UV spectrum of Lead doped L-Threonine

Table - 1: Vibrational assignments of Lead doped L-Threonine single crystal

Wave number cm^{-1}	Vibrational assignments
3289.67	NH_3^+ asymmetric stretching
2921.41	NH_3^+ symmetric stretching
2861.23	C-H symmetric stretching
1713.50, 1618.15	NH_3^+ asymmetric deformation
1451.76	COO^- asymmetric stretching
1265.50, 1073.46,821.28	NH_3^+ rocking
716.70, 619.01	C-C-N deformation

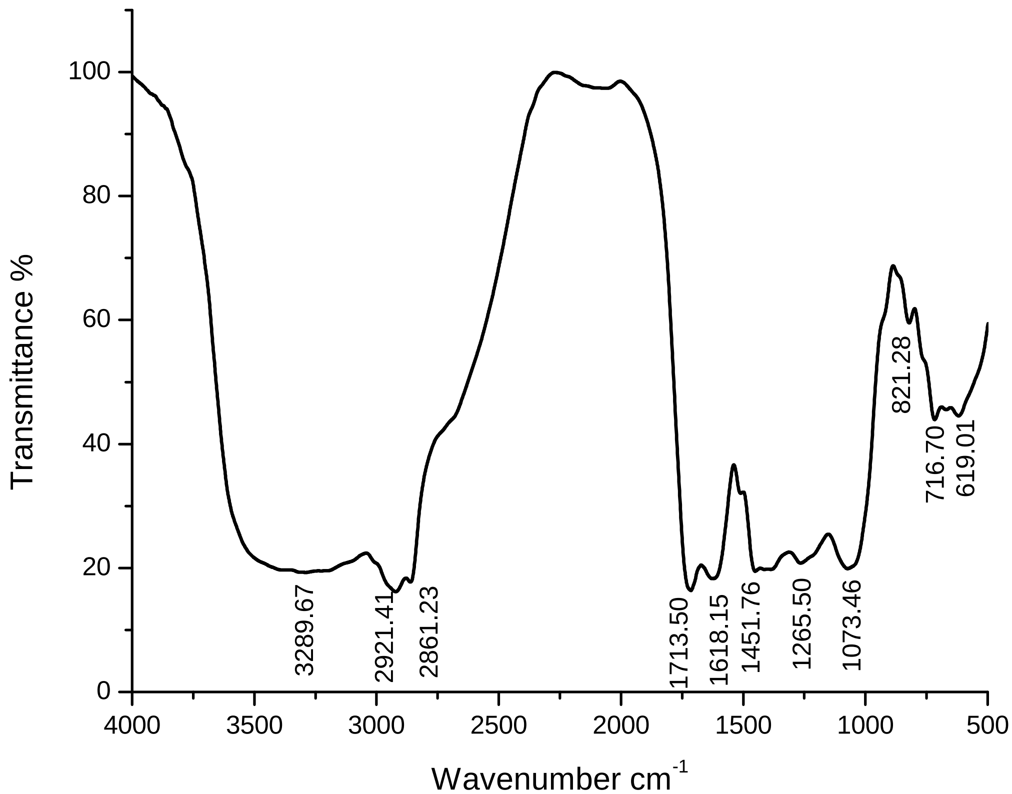

Fig. 4 FTIR spectrum of Lead doped L-Threonine

NH_3^+ asymmetric and symmetric stretching vibration. Due to the stretching vibration involving carbon and nitrogen of amino group exist a peak at 1073.46 cm^{-1}. The asymmetric NH_3^+ deformation occurs at 1618.15 cm^{-1}. The absorption peak at 1713.50 cm^{-1} was due to symmetric NH_3^+ deformation vibration. The peak at 1451.76 cm^{-1} observed at 1265.50 and 821.28 cm^{-1}. The C-C-N deformation was observed at 716.70 cm^{-1} - 619.01 cm^{-1}. Hence, the presence of functional groups was confirmed from the above assignments (Lucia Rose *et al*., 2011; Nagarajan *et al*., 2015).

NLO Test

The SHG behaviour of the powdered material was tested using Kurtz Perry method (Kurtz *et al*., 1968). The sample was ground into very fine powder and tightly packed in a micro capillary tube. Then, it was mounted in the path of Nd:YAG laser beam of 9.6 mj pulse energy obtained by splitting the original laser beam. The output light was passed through monochromator which was detected green light at 532 nm. This confirm the NLO behaviour of the material. The green light intensity registered by a photomultiplier tube and converted into an electrical signal. The same particle size of KDP was used as a reference material. SHG efficiency of Lead doped L-Threonine was greater than that of KDP.

4. Conclusion

Single crystals of Lead doped L-Threonine were grown by slow evaporation technique. Powder XRD confirms the orthorhombic structure of the crystal. FT-IR analysis confirms the Presence of functional groups present in the crystal. The optical transmittance of the crystal was found to be around 330 nm. SHG efficiency shows that the crystal has a higher efficiency than KDP.

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