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Synthesis and thermal, optical, dielectric and mechanical properties of L-asparagine sodium nitrate single crystal

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ABSTRACT

Single crystal of L-asparagine sodium nitrate (LASN) was grown from aqueous solution by room temperature solution growth technique. The grown crystal was characterized by single crystal X-ray diffractometry (XRD), FTIR and optical absorption spectrum. The micro hardness test of the sample revealed that the crystal belongs to the soft category of materials. The dielectric response of the crystal with varying frequencies was studied and reported. The thermal stability of the compounds has been determined by TG-DTA curves. The SHG relative efficiency of LASN crystal was found to be inferior than that of KDP.

1. Introduction

The search of new nonlinear optical (NLO) materials has been of great interest in the recent years because of their significant impact on laser technology, optical communication and optical data storage technology etc [1]. In the recent past, amino acid family crystals are gaining importance because of their higher second order NLO coefficients [2]. Considerable efforts have been made to combine amino acids with interesting organic and inorganic materials to produce outstanding materials like niobates and borates [3]. Such type of materials have already been reported by many researchers [4]. Recently, L-asparagine L-tartaric acid (LALT), L-asparagine thiourea (LATM), L-asparagine nitrate (LAsN) and L-asparagine cadmium chloride monohydrate proves to be good NLO application materials [5–7]. Recently, investigation of Mn^{2+} doped L-asparagine monohydrate single crystal has been found to improve the crystallinity, optical transparency and mechanical strength that is useful for optoelectronic application as reported by Shakir et al. [8]. The present work involves synthesis of, a new semi organic L-asparagine sodium nitrate monohydrate (LASN) material has been synthesized and bulk single crystal has been grown by slow evaporation technique for the first time. The grown single crystal has been subjected to different characterization analysis in order to know is suitability for device fabrication [9].

2. Material synthesis

The title compound was synthesized form of L-asparagine monohydrate and sodium nitrate taken in the ratio 1:1. The calculated amounts of the reactions were thoroughly dissolved in double distilled water. Then, it was mixed with continuous stirring for about 5 h using a magnetic stirrer. The solution was filtered well to remove suspended impurities and allowed to crystalline by the slow evaporation technique at room temperature. After 3 weeks well defined single crystals of LASN were synthesized. The pH value of L-asparagine sodium nitrate is found to be 6.0. The photograph of as-grown crystal is shown in Fig. 1.

3. Results and discussion

3.1. Single crystal XRD

A Bruker Kappa APEXII single-crystal X-ray diffractometer with Mo $K\alpha$ ($\lambda = 0.71073$ Å) radiation was used to estimate the cell parameter of LASN crystal. The single crystal X-ray diffraction study was carried out to confirm the cell parameters of LASN crystal. The crystal belongs to orthorhombic structure with $P2_12_12_1$ space group. The estimated cell parameter is $a = 5.57$ Å, $b = 9.80$ Å, $c = 11.78$ Å and Volume $V = 643$ Å^3.

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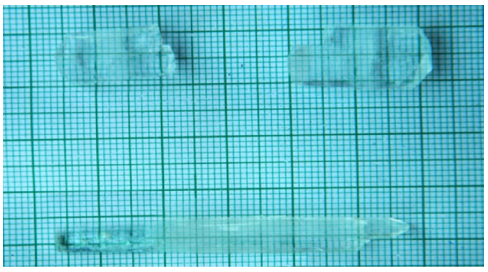

Fig. 1. Photograph of grown LASN single crystal.

3.2. Fourier transform infrared spectral analysis

The room temperature Fourier transform infrared (FTIR) spectrum of LASN was recorded in the region 400–4000 cm^{-1} in order to analyze the synthesized compound qualitatively for the presence of functional groups in the molecule at a resolution of 1 cm^{-1} using a Perkin-Elmer FTIR spectrometer, equipped with a TGS detector, a KBr beam splitter. Spectrum of LASN is shown in Fig. 2. The absorption peaks from 2500 to 3500 cm^{-1} include overlap of peaks due to O—H stretch, N—H stretch and C—H stretch vibrations. These OH, NH and CH stretching vibrations are not clearly resolved in the FTIR spectrum. The presence of hydrogen bonding is confirmed by the broadening of absorption peak. The stretching, vibration of NH stretching in NH^{3+} groups give rise to band at 3444 cm^{-1}. In the crystal, there exist extensive hydrogen bonding interactions whose vibrational frequencies strongly depend on the length of these bonds. The weak hydrogen bonds are characterized by strong, broad, and multicomponent absorption extending into the regions 3300–2500 cm^{-1}, while the strong ones give broad and very strong absorption below 2500 cm^{-1}. The C—C—COO vibration occurs at 1145 cm^{-1} [10]. The band observed at 910 cm^{-1} is attributed to CH_2 rocking vibration in FTIR spectrum [9]. The C—C and C—N stretching appears at 806 cm^{-1} and 892 cm^{-1}, respectively, in the FTIR spectrum is identified. The presence of C═O is evident from the strong peak at 1715 cm^{-1}. The peak at 834 cm^{-1} corresponds to nitrate group which confirms the presence of nitrate in the grown crystal [11]. The observed vibrational frequencies confirmed the presence of functional groups present in the grown crystal.

3.3. Thermal studies

The thermal behavior of LASN was studied by the thermo gravimetric (TG) and differential thermal analyses (DTA). The TG/DTA for LASN has been recorded using SDTQ 600V8.3 Build101 instrument. An alumina crucible was used for heating the sample. The thermo gravimetric analysis (TGA) and differential scanning calorimetric (DSC) analysis of LASN crystal was carried out on the sample weight of 1.164 mg between room temperature to 1000 °C at a heating rate of 20 °C/min in nitrogen atmosphere and the resulting thermogram

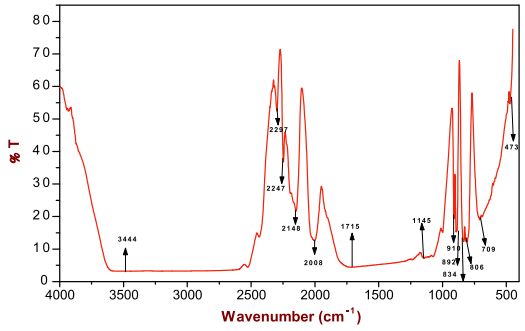

Fig. 2. FTIR spectrum of LASN.

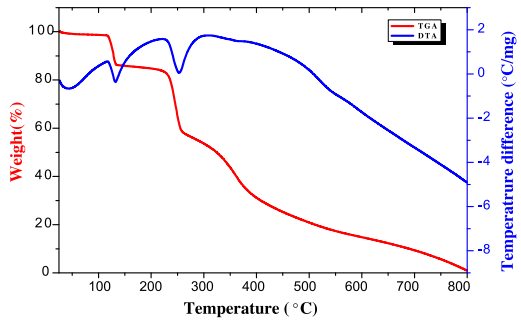

Fig. 3. TGA and DTA curves of LASN.

is shown in Fig. 3. From the TG curve it is obvious, that the material is stable and there is no phase transition up to 122 °C. The loss of weight in the region between 122 °C and 131 °C may be due to the elimination of NO species when the crystal starts decomposing. Further heating induces decomposition of the residue and weight loss occurs in very small steps due to the release of volatile substances in the compound, probably ammonia. The much more gas of CO and CH4 are liberated with higher temperature around 300 °C.

The DTA curve of the crystal reveals that no endothermic or exothermic peak is observed below 118 °C suggesting its structure is stable in the temperature range. Also, there is no phase transition in the respective region. This ensures the suitability of the materials for possible applications in laser. The exothermic peak at 118 °C indicated the decomposition of the compound. The decomposition processes continued up to 400 °C which is signed by the exothermic peak accompanied by the loss of weight of about 75%. The final residue weight left was about 25% after heating to 500 °C. Syed Suresh Babu et al. [9] studied the DSC curves of L-asparagine monohydrate (LAM) and Sr^{2+} ion doped LAM. In the present study, the DSC curve of LASN crystal shows the peak slightly higher in value and shift accordingly compared to LAM crystal. There is a rise in temperature in the decomposition temperature of LASN crystal which is attributed to the increased lattice energy caused by the addition of metal sodium ion.

3.4. Optical measurement

The UV spectrum was recorded using Lamda 35 spectrophotometer with a LASN single crystal of 2.5 mm thickness in the range of 200–900 nm. The transmission range and transparency cut off are very important for the crystals used in the laser and communication field. To determine the transmission range and hence to know the suitability of the crystals for optical applications, the UV–vis spectra were recorded in the range of 200–900 nm (Fig. 4). UV–vis spectrum gives information about the structure of the molecule

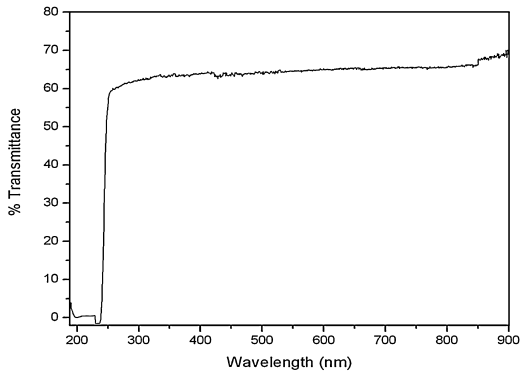

Fig. 4. UV–vis spectrum of LASN.