

Optical, Structural and Elemental Analysis of New Nonlinear Organic Single Crystal of Urea L-Asparagine

R. N. Jayaprakash^a, P. Sundaramoorthi^{b*}, T. Dhanabal^c

^a) Department of Physics, Adhiyamaan College of Engineering, Hosur-635109, Tamilnadu, India.

^b) Department of Physics, Thiruvalluvar Government Arts College, Rasipuram-637401, Tamilnadu, India.

^c) Department of Chemistry, Muthayammal College of Engineering, Rasipuram, Namakkal-637408, Tamil Nadu, India.

Abstract

Single crystal of urea L-asparagine was synthesized and crystallized by slow evaporation solution growth method at room temperature. The bright, transparent and colourless crystal was obtained with average dimension of $11 \times 0.7 \times 0.3 \text{ cm}^3$. The elemental analysis of the compound confirms the stoichiometric ratio of the compound. The sharp and well defined Bragg peaks obtained in the powder X-ray diffraction pattern confirm the crystalline nature of the compound. The optical property of the compound was ascertained through UV-visible spectral analysis. The various characteristics absorption bands in the compound were assigned through fourier transform infra-red (FTIR) spectroscopy. The single crystal unit cell parameters of the compound show that the grown crystal belongs to orthorhombic system with space group P. The nonlinear optical property study indicates that the compound has SHG efficiency 0.5 times greater than that of standard potassium dihydrogen phosphate (KDP).

Keywords: Optical; Elemental analysis; Structural properties;

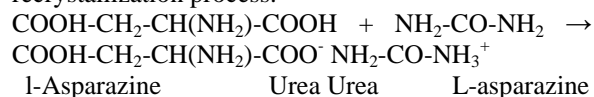
I. Introduction

The nonlinear optical crystals have been given much importance, because of their potential applications such as telecommunication, optical information process, frequency conversion and optical disk data storage [1-2]. Amino acids are interesting materials for NLO applications as they contain a proton donor carboxyl acid (COO^-) group and a proton acceptor amino (NH^{+2}) group in them. Most recent works have demonstrated that organic crystals can have very large nonlinear susceptibilities compared with that of inorganic crystals, but their use is impeded by poor mechanical properties and the inability to produce large crystals [3]. Organic crystals show prominent properties due to their fast and nonlinear response. Over a broad frequency range, they have not only inherent synthetic flexibility and large optical damage threshold, but also have some inherent drawbacks, such as volatility, low thermal stability and weak mechanical strength [4]. The naturally occurring amino acid l-asparagine plays a role in the metabolic control of some cell functions in nerve and brain tissues and is also used by many plants as a nitrogen reserve source [5]. Recently, the growth and characterization of the single crystals of the NLO materials, viz., l-asparaginium picrate [6] and l-asparagine monohydrate [7] have been reported. Based on the above facts, we have reported the synthesis, spectral

and nonlinear optical characterization of urea l-asparazine crystal.

II. Material synthesis and crystal growth

Urea L-asparagine single crystal was synthesized by mixing analytical grade urea and L-asparagine in the stoichiometric ratio 1:1 in triple distilled water. The solution was stirred continuously and to make the solution homogenous, it was slightly heated and then left undisturbed for the precipitation of urea L-asparagine. Slow evaporation method was employed for the growth process. Saturated solution of urea L-asparagine was prepared at 35°C using triple distilled water and stirred thoroughly for 3 h. Then solution was filtered and transferred into 100 ml clean beaker and it was covered with perforated sheet. The solution beaker was housed in constant temperature bath at 35°C for solvent evaporation. Care was taken to minimize the temperature gradient and mechanical shock. Well defined morphology with good transparency single crystals were harvested from the mother solution. The synthetic reaction of Urea L-asparazine crystal is shown on scheme 1. The Good quality crystals were extracted in order to study various characterization studies. The purity of the synthesized salts was enhanced by repeated recrystallization process.



Scheme 1. Synthetic reaction of Urea L-asparazine crystal



Photograph of as grown single crystals of urea L-asparagine

2.2. Characterization techniques

The unit cell parameters are determined by using the single crystal X-ray diffraction data obtained with a four-circle Nonius CAD4/diffractometer (MoK α , $\lambda = 0.710738 \text{ \AA}$).

III. Result and discussion

3.1 Elemental analysis

The elemental analysis shows that the compound Urea l-asparagine contains C: 32.24 % (32.30 %), H: 6.69 % (6.64 %) and N: 17.62% (17.57 %). The results indicate that both experimental and theoretical values (given in brackets). The difference between experimental and

calculated percentages of carbon, nitrogen and hydrogen are very close to each other and are within the experimental errors. Thus, confirms the formation of the compound in the stoichiometric proportion.

3.2. Powder X-ray diffraction pattern method

The powder X-ray diffraction pattern of the synthesized compound is shown in Figure 1. The sharp and well defined Bragg peaks observed in the powder X-ray diffraction pattern confirm its crystalline nature of the compound. The powder X-ray diffraction pattern was scanned from 0° to 80° at a scan rate 1°/min.

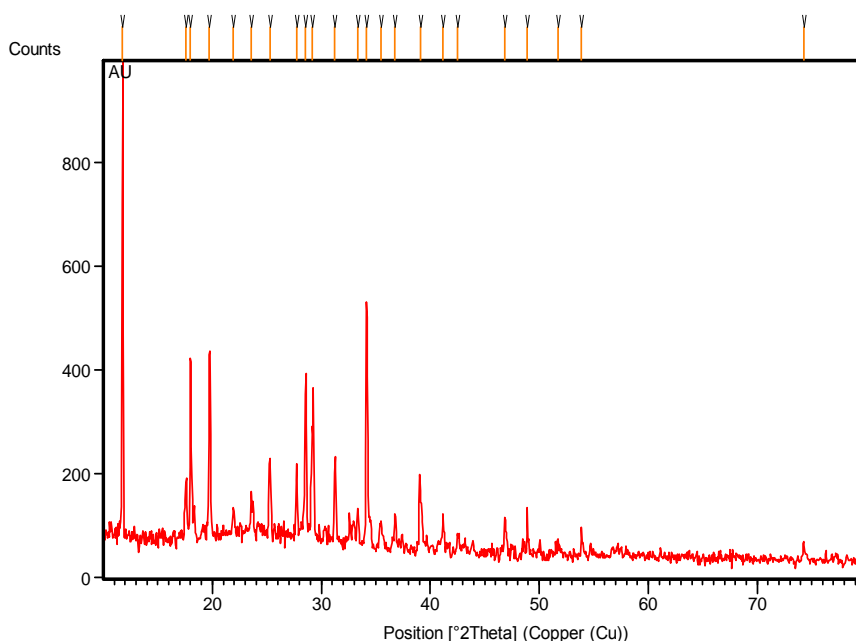


Figure 1: Powder X-ray diffraction pattern of Urea –L asparagines crystal

3.3 Single crystal XRD analysis

The unit cell parameters are $a = 5.56 \text{ \AA}$, $b = 9.77 \text{ \AA}$, $c = 11.77 \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 90^\circ$, $\gamma = 90^\circ$ and volume = $640(2) \text{ \AA}^3$. From the data, the grown crystal belongs to orthorhombic system with space group is P.

3.4 UV -transmittance spectral study

The UV-visible transmittance spectrum for the compound is shown in Figure 3. The spectrum of the compound was recorded in the region between 200-800 nm. The compound shows a lower cut-off wavelength at 230 nm. The transmittance occurs between 200 and 800 nm is approximately 95%. The peak observed at 230nm is due to the $n-\pi^*$ transition. The absence of absorption

in the entire visible region indicates that the compound suitable for optoelectronic application. The crystal has sufficient transmission in the UV and visible region. The higher intensity of absorption band presented in the UV region is due to the conjugated systems presented in the grown material. Vivek et al. [7] reported that the l-asparaginium l-tartrate single crystal has 72%. In this case, urea-L asparagine single crystal reveals better transmittance than l-asparaginium l-tartrate single crystal. We confirmed that urea doped l-asparagine single crystal is suitable material for NLO applications.

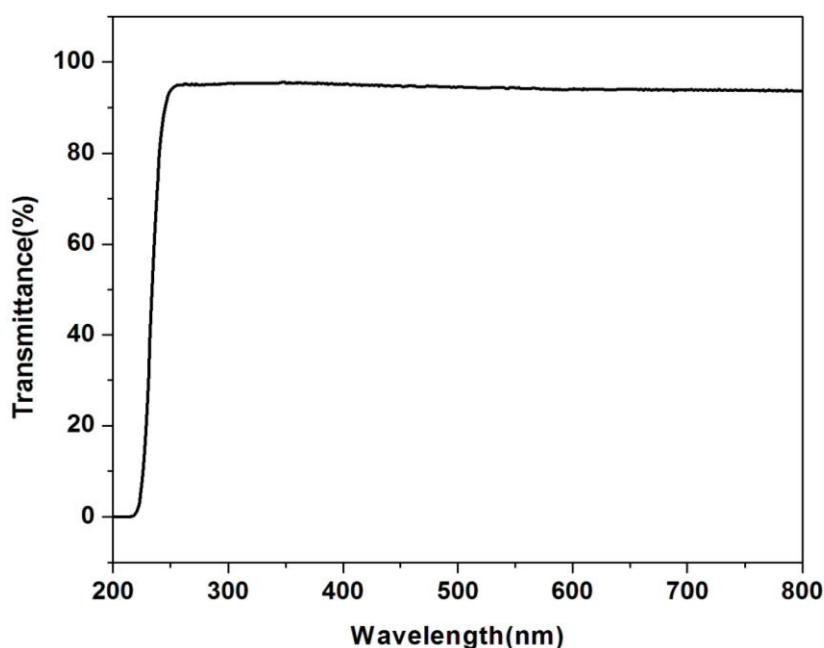


Figure 2. UV- transmittance spectrum of Urea –L asparagines crystal

3.5 FTIR analysis

The FTIR spectrum of the compound is shown in Figure 3. The absorption at 580 cm^{-1} is due to the COO- wagging vibration. The C=C stretching vibration is observed at 760 cm^{-1} . The peak at 805 cm^{-1} is assigned to CH_2 rocking vibration. The C-H in plane bending vibration is observed at 1108 and 1176 cm^{-1} . The absorption observed at 1251 cm^{-1} is characteristics of CH_2 rocking vibration. The CO

group stretching vibration is found at 1410 cm^{-1} . The peak at 1673 cm^{-1} is due to NH_2 group deformation mode. The combination band and overtone of NH_2 group are observed at 2035 cm^{-1} . The absorption at 2895 cm^{-1} is assigned to CH_2 asymmetric stretching vibration. The NH_2 stretching vibration is found at 3057 cm^{-1} . The peak at 3462 cm^{-1} is characteristics of OH stretching vibration.

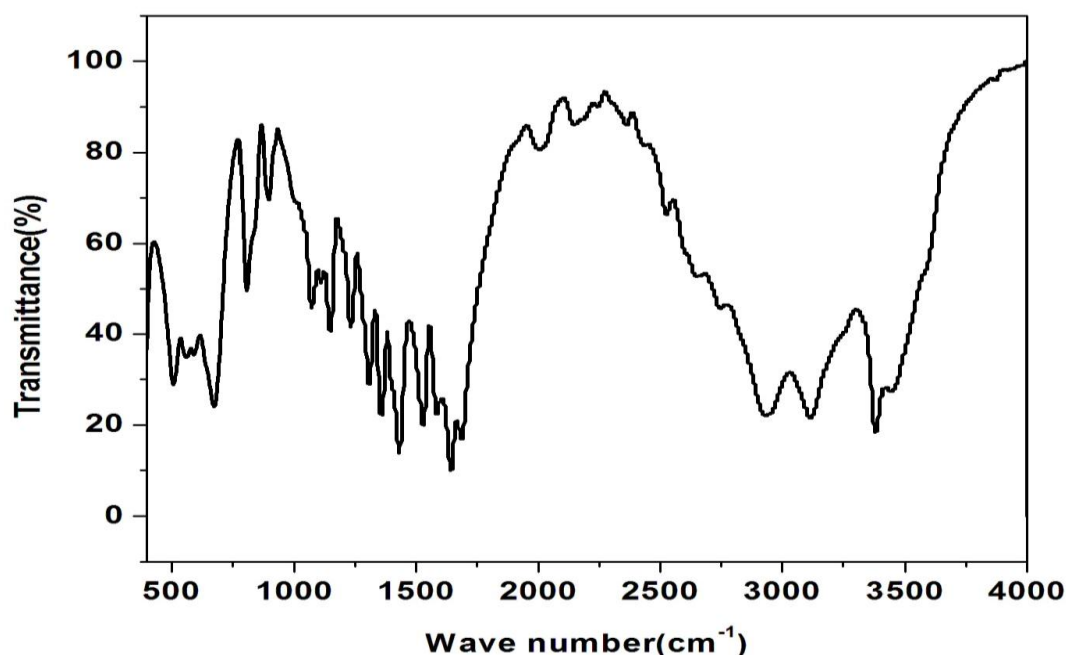


Figure 3. FTIR spectrum of Urea –L asparagine crystal

3.6 NLO studies:

The nonlinear optical property of the grown crystal was tested using Kurtz-Perry powder technique by passing a Q-switched, mode locked Nd:YAG laser of 1064 nm and pulse width of 8 ns (spot radius of 1 mm) on the powder sample of TMPP. The input laser beam was passed through an IR reflector and then directed on the microcrystalline powdered sample. Photodiode detector and oscilloscope assembly detected the green light emitted by the sample. The second harmonic generation efficiency of the crystal was evaluated by taking the microcrystalline powder of KDP as the reference material. For a laser input pulse of 6.9 mJ, the SHG signal (532 nm) of 3.9 mV and 8.0 mV were obtained for KDP and samples respectively. Hence, it is observed that the SHG efficiency of compound has 0.5 times greater higher than that of KDP.

IV. Conclusions

Single crystal of urea L-asparagine was synthesized by slow evaporation solution growth technique at room temperature. The stoichiometric ratio of the compound was studied by using elemental analysis. The sharp and well defined Bragg peaks obtained in the powder X-ray diffraction pattern confirm its crystallinity. The UV-visible spectral analysis was used to study the optical property of the compound. The various characteristics absorption bands present in the compound were assigned through FTIR spectroscopic technique. The single crystal unit cell parameters of the compound indicate that the crystal

belongs to orthorhombic system with space group P. The synthesized compound exhibits SHG efficiency 0.5 times greater than that of standard KDP.

V. Acknowledgement

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