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Growth and characterization of L-alanine admixtured urea single crystal

R.N. Jayaprakash [a], P. Sundaramoorthi [b,*]

[a] Department of Physics, Adhiyamaan College of Engineering, Hosur 635109, Tamil Nadu, India
[b] Department of Physics, Thiruvalluvar Government Arts College, Rasipuram 637401, Tamil Nadu, India

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ABSTRACT

Single crystals of urea with L-alanine were grown by low temperature solution growth method from aqueous solution. The cell parameters and crystalline nature of the grown crystals were determined from single and powder X-ray diffraction studies. The qualitative analyses of the presence of functional groups in the grown crystals were estimated from FTIR analysis. The UV–visible spectrum reveals the optical property of the grown crystal. The second harmonic generating (SHG) properties are demonstrated using Kurtz–Perry powder technique. Photoluminescence shows that the grown crystal has a blue fluorescence emission. Photoconductivity study reveals the negative photoconductivity nature of the crystal. The low value of dielectric loss with high frequency for these samples suggests that the samples possess enhanced optical quality with lesser defects and this parameter is of vital importance.

1. Introduction

Wideband optical communicating systems are gradually displacing electronic methods due to their speed, bandwidth and reliability and in recent years, demand for such optical systems has been increasing in high speed integrated electro optic devices. The extended field of nonlinear optical (NLO) applications has stimulated search for the highly nonlinear optical materials [1–3]. Organic materials have been of particular interest because of their high order of optical nonlinearity, large electro-optic co-efficient with low frequency dispersion, large nonlinear response over a broad frequency range and large structural diversity [4–6]. Urea is the organic nonlinear optical material used in the processing of generation and mixing of frequencies in a spectrum including the UV [7–11] because of its unique properties such as transparency and large birefringence. Lack of large size optically single crystals prevents the utilization in the NLO applications; moreover growth process is difficult to control. In order to overcome these drawbacks, a variety of dopants like L-alanine, L-valine, L-histidine, etc., have been incorporated for investigation, thereby giving quality bulk single crystals with better properties than pure. Amino acid family crystals are playing an important role in the field of nonlinear optical organic molecular crystal. Among them L-alanine is simplest natural amino acid with methyl group as a side chain and it has the zwitterionic form ($^{+}NH_3{}^{-}C_2H_4{}^{-}COO^{-}$) both in crystal and in aqueous solution over a wide range of pH, which favors crystal hardness for device application [7]. L-Alanine is an efficient organic NLO compound under the amino acid category. The second-harmonic generation (SHG) efficiency of L-alanine is about one-third of KDP. In the present study, we report the effect of L-alanine on urea. Crystal structure and the presence of functional groups of grown crystals were analyzed by powder X-ray diffraction and Fourier transforms Infrared respectively. Their linear and nonlinear optical study has also been carried out. Herein, we report the results of our all studies in this communication.

2. Experiment

Amino acids urea and L-alanine were mixed in the stoichiometric ratio 1:1 in triple distilled water and the temperature of the solution was maintained at about 50 °C with continuous stirring. L-Alanine admixture urea was obtained by the evaporation of the solvent. Then the purity of the synthesized salts was enhanced by successive recrystallization process. For growth process, saturated solution was prepared by using triple distilled water and stirred thoroughly for 2 h. Then solution was filtered and transferred into 100 ml beaker having perforated holes kept in the constant temperature bath set at 40 °C for solvent evaporation. Well defined morphology with good transparency single crystals (Fig. 1) were harvested from the mother solution.

* Corresponding author. Tel.: +91 4344 261 111; fax: +91 04344 260573.
E-mail address: prakashnandhi@yahoo.co.in (P. Sundaramoorthi).

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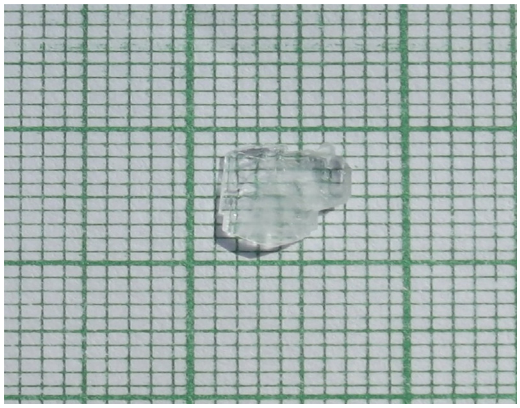

Fig. 1. As grown single crystal of urea L-alanine.

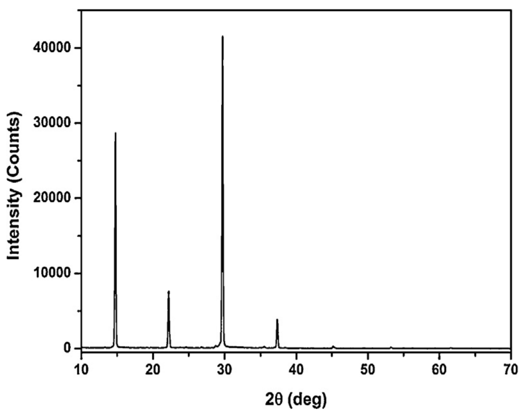

Fig. 2. Powder X-ray diffraction pattern.

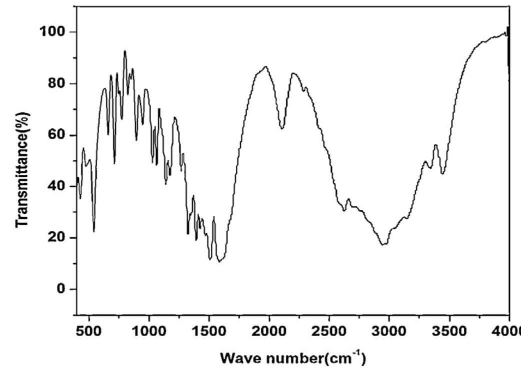

Fig. 3. FTIR spectrum of the grown crystal.

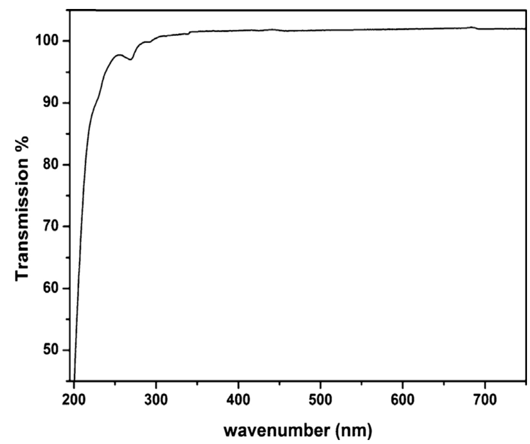

Fig. 4. UV–vis–NIR transmission spectrum of the grown crystal.

3. Results and discussion

3.1. Single and powder X-ray diffraction study

Single crystal X-ray diffraction data recorded using MACH 3 Nonius CAD-4 X-ray diffractometer with Mo Kα radiation ($\lambda = 0.7107$ Å) for the grown crystals. It is confirmed from this study that the title compound crystallizes in orthorhombic P system and have the lattice parameter values $a = 5.26$ Å, $b = 9.57$ Å, $c = 12.03$ Å and $\alpha = 90^\circ$, $\beta = 90^\circ$, $\gamma = 90^\circ$ and the volume is 605 Å^3. Powder X-ray diffraction analysis has been carried out using Philips Powder X-ray diffractometer using CuKα radiation to identify the lattice parameters. The sharp and well defined Bragg's peaks confirmed the crystallinity of the grown crystals. Powder X-ray diffraction pattern of the grown crystals was shown in Fig. 2.

3.2. FTIR spectral analysis

The infrared spectra were taken using Nicolet Instrument Corporation using KBr pellet technique to analyze the presence of functional groups in the crystal sample qualitatively. The FTIR spectrum of grown crystal has been shown in Fig. 3.

3.3. UV–vis–NIR spectral analysis

To analyze optical window width of the title compound, UV–vis–NIR spectrum was measured in the range from 190 to 1100 nm in solution mode and is shown in Fig. 4. It was found that the crystal was transparent from 250 to 1100 nm. As observed in the spectrum of Fig. 4 absence of absorption in the entire visible region, it is an essential condition for optoelectronics applications [12]. As a result it can be used as a potential material for SHG in the visible region.

3.4. Second harmonic generation study

The second harmonic generation behavior of the powdered material was tested using the Kurtz and Perry method [13]. A high-intensity Nd:YAG laser ($\lambda = 1064$ nm) with a pulse duration of 10 ns was passed through the powdered sample. The SHG behavior was confirmed from the output of the laser beam having the bright green emission ($\lambda = 532$ nm). The second harmonic signal of 14 mV for L-alanine admixture urea was obtained for an input energy of 2.9 mJ/pulse. The SHG value of KDP crystal gives a signal of 6.9 mV/pulse for the same input energy. Thus, it is observed that the SHG efficiency of the L-alanine admixture urea is 2.02 times higher, than that of the standard KDP crystal.

3.5. Photoluminescence (PL) analysis

The photoluminescence property of the L-alanine admixture urea was studied using FP-6500 Spectrofluro meter-67 at room temperature. The emission spectrum was recorded in the range 200–550 nm. Fig. 5 shows photoluminescence spectrum of L-alanine admixture urea single crystal. The emission spectrum shows broad peak centered at 440 nm L-alanine admixture urea. This intensity peak at 440 nm is due to the protonation of amino group to the carboxyl group. After 440 nm the intensity of photoluminescence spectrum is becomes minimum. The maximum intensity peak at 440 nm is ascribed to n–π* transition of carbonyl group. The lowering of photoluminescence intensity at higher wavelength region may be accredited to a comparatively low