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Growth, structural, optical, electrical and mechanical studies on urea phthalic acid single crystals

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ABSTRACT

Optically good quality organic crystals of urea phthalic acid (UPA) were grown successfully by slow evaporation solution growth technique at room temperature. The crystalline nature of the crystal was assessed by powder XRD analysis. The presence of various functional groups has been identified by using FT-IR spectral analysis. UV–vis spectra show good transparency in the entire visible region. Dielectric constant and dielectric loss were measured for the grown crystals. Photoconductivity studies were also taken for the grown crystals. Mechanical studies of the crystals were studied using Vickers microhardness tester.

1. Introduction

Some new materials with unique optical properties are essential for using photons instead of electrons in the transmission of information. These demands of NLO materials attract the researchers toward growth of crystals that produce second harmonic generation. Among NLO materials organic NLO materials are generally believed to be more versatile than their inorganic counter parts due to their more favorable nonlinear response. Because of its capable electro optical properties it can be utilized in optical device fabrication which can replace the electronic switching circuits [1–6]. Urea is an organic NLO material which attracts many researchers due to its high nonlinear optical properties. Its mechanical and optical properties were comparable to those of inorganic ADP and KDP crystals [7–9]. Its crystallization is problematic due to its polar electrical characteristics which enhance the interaction between growth surfaces and molecules of solvent and solute. With some better efforts improved results have been achieved by solution and vapor growth technique. Generally organic acids form stable hydrogen bonded system with urea. Some of the reported promising urea based organic NLO crystals such as urea succinic acid, urea L-malic acid, urea cinnamic acid and urea DL-tartaric acid [10–13].

In the present work urea and phthalic acid single crystals were grown by slow evaporation solution growth technique at room temperature. The grown crystals were characterized by various characterization techniques such as UV, FT-IR, powder XRD, dielectric, photoconductivity and microhardness studies and the obtained results were discussed.

2. Experimental

2.1. Crystal growth

In the present study UPA crystals were grown by slow evaporation solution growth technique. Commercially available (AR grade E-Merck) urea and phthalic acid were taken in the stiochiometric ratio of 1:1 in methanol and stirred separately for about half an hour using a magnetic stirrer. After that these solutions were mixed together and stirred well for about 1 h and the saturated solution was filtered using Whatman filter paper. The filtered solution was transferred into crystallizing vessel. To control the evaporation of the solvent beaker was covered with thin plastic sheet. After 20 days good quality crystals were harvested from the solution. The as grown crystal is depicted in Fig. 1.

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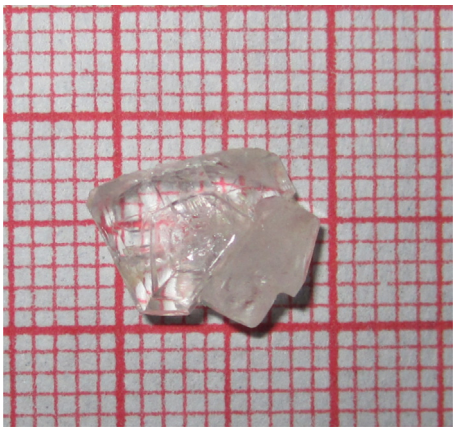

Fig. 1. Photograph of as grown crystals of UPA crystals.

3. Results and discussion

3.1. Powder XRD analysis

The grown crystals were finely crushed into microcrystalline powder using mortar pestle and then subjected to analysis. Powder X-ray diffraction of the grown crystal has been recorded using RICH SIEFERT X-ray powder diffractometer equipped with CuKa ($\lambda = 1.5405$ Å) radiation. The recorded spectrum is shown in Fig. 2. The samples were scanned over the 2θ range of 5–80° at a scan rate of 2°/min. The sharp and well defined Bragg's peaks at specific 2θ authenticate the crystalline nature and purity of the crystal. The highest peak intensity was observed at 70,855 counts/s. While compared to previous powder XRD results the title compound shows very high crystalline nature. This confirms the purity of the sample [10,14,15].

3.2. FT-IR studies

The FT-IR spectrum of the grown crystal was recorded using a Jasco-460 plus FT-IR spectrometer by the KBr pellet technique in the wavelength range of 400–4000 cm^{-1}. The recorded spectrum is depicted in Fig. 3. The peak at 3487 and 2669 cm^{-1} is assigned to the symmetric and asymmetric NH_2 stretching vibration. Vibration at 1647 cm^{-1} is due to the NH_2 deformation. The absorption band at 1047 cm^{-1} is assigned to N—C—N stretching vibration. The symmetric and asymmetric stretching of COO— is found to be at 1315 and 1408 cm^{-1} respectively. The C=C and C—O—H stretching vibration is occurred at 1477 and 704 cm^{-1}. CH stretching vibration appears at 1280 and 1315 cm^{-1}.

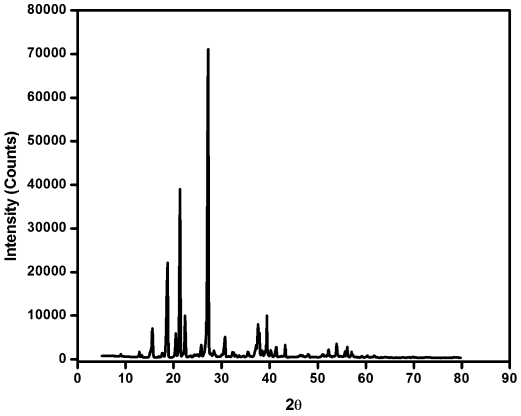

Fig. 2. Powder XRD spetrum of UPA crystal.

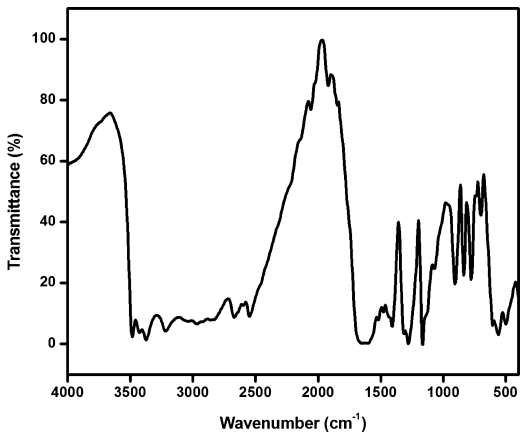

Fig. 3. FT-IR spectrum of UPA crystal.

3.3. Optical transmittance studies

The UV–vis transmittance spectrum of the grown crystals were recorded using a Perkin–Elmer lambda 35 spectrophotometer in the range 200–1100 nm covering the near ultraviolet (200–400), visible (400–800) and then far infrared region (800–1100). The transmittance spectrum is depicted in Fig. 4a. UPA crystal shows maximum transparency of 90% in the entire visible region with lower cut-off wavelength of 310 nm. The optical absorption coefficient (α) was calculated using the recorded transmittance by the given relation

$$\alpha = \frac{2.3036 \log(1/T)}{t}$$

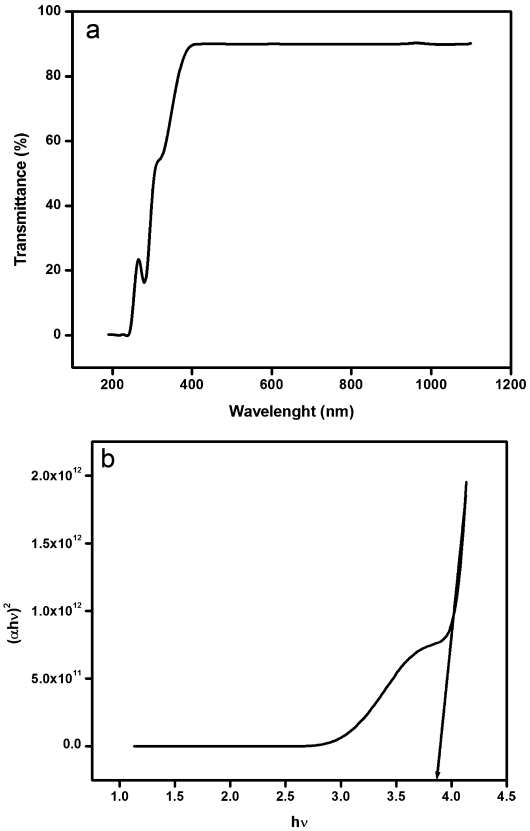

Fig. 4. (a) Optical transmittance spectrum of UPA crystal. (b) Optical band gap of UPA crystal.