Optical, Thermal and Electrical Characterization of Urea Sulphamic Acid Single Crystals

E. Chinnasamy¹, A.Venkatesan²,³, M.E. Rajasaravanan³, S.Senthil¹,a

¹ – Department of Physics, Govt Arts College for Men, Nandanam, Chennai, India
² – Department of Physics, Aringnar Anna Arts College, Villuppurm, India
³ – Department of Physics, Government Arts College, Salem, India
a – ssatoms@yahoo.co.in

Keywords: single crystal XRD, FT-IR, UV-Vis spectral studies and PL studies, TG/DTA, dielectric studies.

ABSTRACT. Urea Sulphamic acid single crystal was grown by slow evaporation techniques at room temperature. The cell parameters of the grown crystal were determined by single crystal X-ray diffraction analysis. The presence of functional groups in the crystal lattice has been qualitatively determined by FT-IR analyses. Optical characterization were analysed by UV-Vis and photo Luminescence (PL) spectral studies and the band gap energies of the USA single crystals have been calculated. Thermo gravimetric and differential thermo gravimetric analysis (TG/DTA) indicates the thermal stability of the grown crystal. The dielectric properties of the grown crystal have been studied.

Introduction. The search of materials for various device applications has led to discovery of many organic, inorganic and semi organic crystals. The responsibility for the exquisiteness of the crystal is due to their structural simplicity, symmetry and purity. These characteristics endow crystals with unique physical and chemical properties which caused major transformation in the electronics industry [1-2]. Nonlinear Optical (NLO) materials have potential applications in optoelectronics, Second Harmonic Generation (SHG), optical storage, optical communication, photonics, electro optic modulation, optical parametric amplifiers, optical image processing, etc [3]. In recent years more emphasis is given to inorganic materials due to their much matured NLO applications than organic materials and owing to their good transparency, chemical stability, and mechanical properties [4]. Also research into the growth of large single crystals from aqueous solution is currently serving as the important avenue to general progress in understanding many fundamental concepts of crystallization [5]. Hence, in the present work, a systematic study on the growth and characterization of Urea Sulphamic acid (USA) is reported. The grown single crystals have been subjected to single crystal XRD, FTIR, UV-visible and Photoluminescence spectroscopy, TG/DTA and dielectric studies respectively.

Experimental Procedure. In the present study, USA crystals were grown by slow evaporation solution growth technique. Urea and Sulphamic acid were taken in equimolar ratio and dissolved in Millipore water. The solution was stirred up to saturation state. The solution was filtered and covered with dust free polyethylene sheet then placed at room temperature [6]. After a period of 15 to 20 days, good quality and highly transparent Urea Sulphamic acid (USA) seed crystals has been grown and the good quality seed crystals are allowed to grow and the crystals of size 13x5x4 mm³ was harvested. The photograph of the as grown single crystal is shown in Fig.1

© 2017 The Authors. Published by Magnolithe GmbH. This is an open access article under the CC BY-NC-ND license
http://creativecommons.org/licenses/by-nc-nd/4.0/
Results and Discussion

**Single Crystal X-Ray Diffraction.** Single crystal X-ray diffraction analysis of USA crystal was carried using an Enraf Nonius CAD4 single crystal X-ray diffractometer with an incident CuKα radiation, and the calculated lattice parameter values are a= 8.076 Å, b= 8.098 Å, c= 9.218 Å, α=β=γ=90° and V = 602.8 (Å³). The lattice parameter values show that the grown USA crystal belongs to orthorhombic structure, which is confirmed with the reported values.

**FT-IR Spectral Analysis.** The FTIR spectroscopy study is effectively used to identify the functional group present in the material. The FTIR spectrum for USA is recorded using BRUKKER IFS 66V spectrometer by KBr pellet technique in the range 400-4000 cm⁻¹ and is show in Fig. 2. The FTIR spectrum of Urea Sulphamic Acid seems to be complex because of various functional groups present in the crystal. The functional group assignments for USA is summarized in Table 1.

**UV-Visible Absorption Spectrum.** The absorption spectra of USA crystal is measured in the wavelength range 200-800 nm using Philips PV8700 UV-visible scanning spectrometer. The recorded absorption spectrum is show in Fig.3. It is observed that the, crystal have good transparency window in the entire visible and IR region. The lower cut off wavelength is observed at 254 nm [7]. The optical band gap is obtained by plotting the graph between hν and (αhν)² and is shown in Fig.4. From the Tauc’s plot, the optical energy band gap is determined as 4.74eV.
Table 1. Fundamental Vibrational Assignments of USA crystal.

<table>
<thead>
<tr>
<th>Wave number (cm(^{-1}))</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>3096</td>
<td>Degeneracy. NH(_3^+) stretching</td>
</tr>
<tr>
<td>2877</td>
<td>Symmetric. NH(_3^+) stretching</td>
</tr>
<tr>
<td>1535</td>
<td>Degeneracy. NH(_3^+) deformation</td>
</tr>
<tr>
<td>1440</td>
<td>Symmetric. NH(_3^+) deformation</td>
</tr>
<tr>
<td>1239</td>
<td>Degeneracy. SO(_3^-) stretching</td>
</tr>
<tr>
<td>1061</td>
<td>Symmetric. SO(_3^-) deformation</td>
</tr>
<tr>
<td>683</td>
<td>N-S Stretching</td>
</tr>
<tr>
<td>529</td>
<td>Degeneracy. SO(_3^-) deformation</td>
</tr>
</tbody>
</table>

Fig. 3. UV absorption spectrum of USA crystal.

Fig. 4. Optical Band gap of USA crystal.

**Photoluminescence Analysis.** Aromatic compounds or the molecules with multiple conjugated double bonds are expected to give high degree of resonance stability and can be expected strong fluorescence. The emission spectrum has been recorded at room temperature by exiting the molecules with wavelength of 362 nm. The emission spectrum is shown in Fig.5. Strong emission from green to red is observed with three peaks at 503, 609 and 749 nm for this excitation. The property of having strong emission in this range may lead to potential application of this material in optoelectronic device [8].
Thermal Analysis. The thermal stability and physiochemical changes of USA crystal were analyzed by recording the TG–DTA spectrum as shown in Fig. 6. It reveals that USA is thermally stable up to 191.0°C and after this the sample undergoes appreciable weight loss. The change in weight loss confirms the decomposing nature of USA sample. The DTA spectrum confirms the melting point of the sample through a sharp exothermic peak at 191.0°C. Moreover, the endothermic peak at 409.4°C reveals the volatile nature of the sample. After that no sharp peak was observed, which confirms that the material is thermally stable up to 409.4°C.

Dielectric Studies. Variations in dielectric constant and dielectric loss as a function of room temperature and frequency are shown in Fig. 7a and 7b. In Fig. 7a, it is seen that the value of dielectric constant is found to increase with temperature and it becomes independent of frequency at higher frequency region. The decrease in dielectric constant of USA crystal at low frequencies may be attributed to the contribution of the electronic, ionic, orientation and space charge polarizations which depend on the frequencies [9]. The low value of dielectric loss at high frequencies suggests that the sample possess enhanced optical quality with lesser defects and this parameter is of vital importance for NLO applications [10].
Summary. Good optical quality single crystals of USA have been grown from slow evaporation technique. The crystallinity of the grown sample has been confirmed by X-ray diffraction analysis. Various functional groups present in the grown crystal have been identified by FTIR analysis. The optical transparency has been revealed by UV-visible study and thermal stability has been confirmed by thermal analysis. The low dielectric constant and dielectric loss of USA at higher frequencies show that the material is a more suitable candidate of nonlinear optical application.

References


Cite the paper