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Growth of Urea Salicylic Acid (USA) Crystal

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Abstract: A new organic NLO crystal Urea salicylic acid (USA) was grown by slow evaporation method using the mixed solvent of ethanol and water in equal ratio. The crystal structure parameters were observed by single and powder x-ray diffraction (XRD) studies. The various functional groups present in the grown crystal were confirmed by Fourier transform infrared (FTIR) spectroscopy studies. Thermo gravimetric analysis (TG) and differential thermal analysis (DTA) were carried out to find the thermal stability of the USA crystal. The optical absorption study explained the good transmission window of the title crystal and its suitability for optical applications. The second harmonic generation (SHG) efficiency of USA was confirmed by Kurtz Perry technique.
Keywords: Slow evaporation method; X ray diffraction; Fourier transform infrared spectrum; Thermal analyses; Nonlinear optical crystal.

1.1. Introduction

Nonlinear optics (NLO) is at the forefront of current research because of its importance in providing the key functions of frequency shifting optical modulation, optical switching and optical memory for the emerging techniques in areas, such as telecommunications, signal processing and optical interconnections [1-4]. Organic crystals have been extensively studied due to their nonlinear optical coefficients being larger than those of inorganic crystals. Urea is an organic compound with the chemical formula $(NH_2)_2CO$. The molecule has two amide (—NH_2) groups joined by a carbonyl (C=O) functional group. It has shown interesting properties for nonlinear optical applications [5]. The growth of urea salicylic acid (USA) compound is formed from the strongly organic compound of urea and weak carboxylic acid. In the present work, the single crystal of Urea salicylic acid (USA) was grown by slow evaporation method at room temperature. The grown USA crystal was subjected to different characterization studies, such as single and powder X ray diffraction, Fourier transform infrared spectroscopy (FTIR), thermo gravimetric and differential thermal analyses (TG-DTA), ultraviolet - visible spectroscopy (UV-vis) and second harmonic generation (SHG) to confirm its structural, spectral, thermal, linear and nonlinear optical properties.

1.2. Experimental method

The urea salicylic acid (USA) salt was synthesized using high-purity urea and salicylic acid in equal molar ratio by slow evaporation method. The calculated amount of salts were dissolved in the mixed solvent of water and

ethanol with equal molar ratio at room temperature. Transparent and colourless crystals were harvested in a period of 15 days. The photograph of the grown crystals is shown in Fig. 1.

2. Result and Discussion

2.1. Single crystal X ray diffraction study

The grown USA crystal was subjected to single crystal X-ray diffraction study using an Enraf CAD-4 diffractometer with Mo Kα radiation. The obtained results confirmed that the title crystal belongs to monoclinic structure. The cell parameters of the title compound are a = 4.92 Å, b = 11.16 Å, c = 11.56 Å, α = 90°, β = 90.83°, γ = 90° and volume, V = 634.58 $Å^3$.

2. 2. Powder X-ray diffraction analysis

The powder X-ray diffraction study of USA crystalline sample was carried out, using Siemens D500 X-ray diffractometer with Cu Kα radiation. The X-ray diffraction pattern for the grown sample is shown in Fig. 1. The well-defined Bragg's peak at specific 2θ angles have shown high crystallinity of USA. The evaluation of lattice parameters from powder XRD pattern and peak indexing were carried out using the software Proszki Version 2.4.

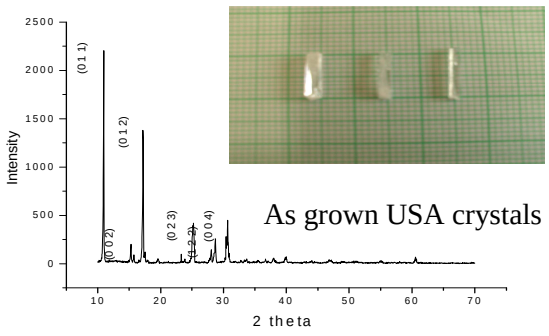

Fig.1. Powder X-ray diffractogram of USA Crystalline sample

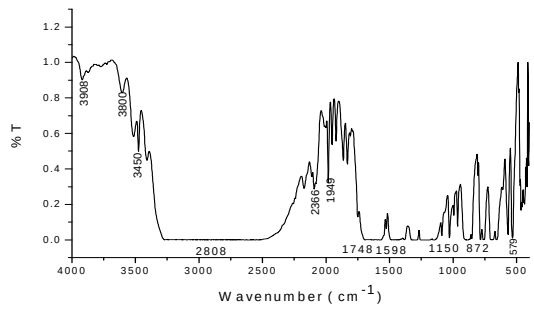

Fig.2. FTIR spectrum of USA crystalline sample

2.3. FTIR analysis

The FTIR spectrum of USA crystalline sample was recorded in the range of 4000 – 400 cm^{-1} using a Perkin – Elmer grating infrared spectrometer by KBr pellet method. The FT-IR spectrum analysis of grown USA crystal is shown in Fig.2. The presence of N-H stretching is shown at the peaks of 3606 cm^{-1} and 3410 cm^{-1}. The broad envelope in the high energy region between 3410 and 2500 cm^{-1} is due to NH_2 hydrogen bond symmetric and asymmetric vibrations [6]. The peak at 1750 cm^{-1} is assigned to C = O stretching which confirms the presence of carboxylic group in the grown crystal.

2.4. TG-D TA analyses

The TG-DTA thermogram of the grown USA crystal was recorded between 30º C and 1000º C at a heating rate of 10 K/min. in the nitrogen atmosphere using the instrument NETSZCH STA 409 C/CD. The spectral curves are shown in Fig. 3. The TG thermogram reveal that the loss of weight from the temperature 158.3 ^{0}C confirms the decomposing nature of USA sample. The DTA spectrum shows a sharp endothermic peak at 158.3 ˚C which is attributed to the decomposition of the material as shown in TG spectrum. Thus from the thermal studies, it is understood that the title compound is stable in the working temperature and retain its texture upto 158.3 ºC.

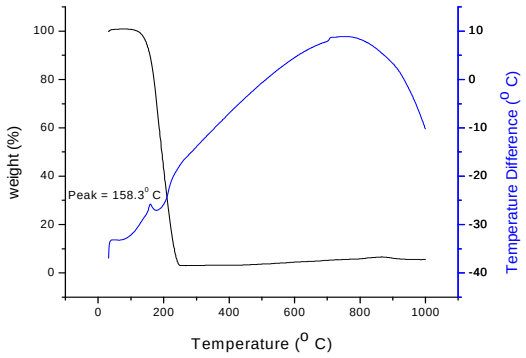

Fig.3. TG-DTA curves of USA crystalline sample

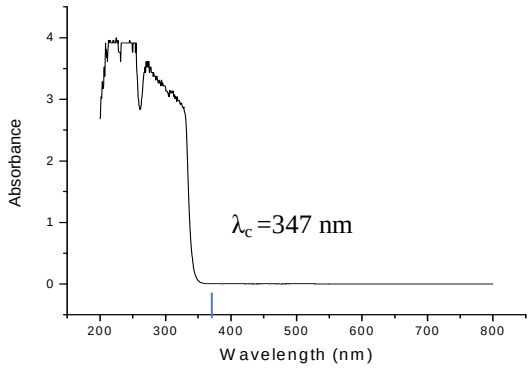

Fig.4. UV-vis-NIR spectrum of USA Crystalline sample

2.5. UV-vis studies

The UV-vis optical absorption spectrum of USA crystal was recorded in the range 200-800 nm using Varian Cary SE UV-vis-NIR spectrometer. The absorption spectrum of title crystal is shown in Fig.4. From the UV absorption spectrum, it is observed that the crystal is transparent in the range 340 to 800 nm without any absorption peak, which is an essential requirement for NLO crystals. It is evident that USA crystal has UV cut off wavelength (λ_c) at 347 nm. The calculated band gap value of the grown USA crystal is 3.58 eV. This indicates that USA is a higher band-gap energy material with large transmittance in the visible region.

2.6. Second harmonic generation (SHG) study

A quantitative measurement of the second harmonic generation (SHG) efficiency of USA crystal was determined by the modified version of powder technique developed by Kurtz and Perry [7]. In this technique, the sample was packed as a polycrystalline powder into a cell sandwiched between two glass slides and exposed to a Q-switched ND:YAG laser emitting 1064 nm, 10 ns laser pulses with spot radius of 1 mm to assess the SHG intensity. The powder SHG efficiency of USA is 0.55 times that of the KDP crystal.

2.7. Conclusion

The optically good quality single crystal of USA has been successfully grown by slow evaporation technique from the mixed solvent of ethanol and water with equal molar ratio at room temperature. The structural, spectral, thermal, linear and nonlinear studies of the grown USA crystal indicate that the crystal is suitable for photonic applications.

References

1. Chemla, D.S. and Zyss, J., Eds., Nonlinear Optical Properties of Organic Molecules and Crystals, Academic Press, New York, 1987. 23-27.
2. Nalwa, H.S. and Miyata, S., Nonlinear Optics of Organic Molecules and Polymers CRC Press, Boca Raton, 1997. 129.
3. Prasad, P.N. and Williams. D., Introduction to Nonlinear Optical Effects in Organic Molecules and Polymers, Wiley, New York, 1991.
4. Thomas Joseph Prakash J., Felicita Vimala J. and Lawrence M., Growth and Characterization of Organic NLO Material, J. Comp. Appl., 2010, 8,18-21.
5. Selvam A., Pandi S., Selvakumar S. and Srinivasan S., Growth and characterization of urea citric acid (UCA) single crystal by slow evaporation process, J. Appl. Sci. Res., 2012, 4, 2474- 2478.
6. Bellamy L., The Infrared Spectra of Complex Molecules, Wiley, New York, 1958, 161.
7. Kurtz S.K. and Perry T.T., A powder technique for the evaluation of nonlinear optical materials, J. Appl. Phys., 1968, 3798.

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