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THE STRUCTURAL AND OPTICAL PROPERTIES OF UREA DOPED LASC NLO SINGLE CRYSTAL

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Abstract - *Growth of single crystals of Urea doped L-Alaninium Sodium Chloride (ULASC) was carried out in its aqueous solution using slow solvent evaporation technique at room temperature. Single crystal XRD proves that ULASC crystals belong to orthorhombic crystal system with space group $P2_12_12_1$. The optical absorption study reveals the optical transparency of the sample over a wide range from 350 to 2000 nm thus having optical transmittance in the entire range covering UV-Vis-. The NLO property of the grown crystals was confirmed by SHG studies. The dielectric studies were carried out with respect to frequency variation and temperature variation.*

***Key Words*: solution growth, NLO, SHG, FTIR, XRD, SEM**

1. INTRODUCTION

The search and design of high efficient nonlinear optical (NLO) crystals for visible and Ultraviolet (UV) regions are extremely important for laser and material processing However, most organic NLO crystals have usually poor mechanical and thermal properties, and it is difficult to grow large optical quality crystals of this class for device applications [1-4].Organic and semiorganic NLO crystals formed with L-alanine have been identified as potential candidates for replacing KDP in nonlinear optical applications [5-7]. Urea doped L-Alaninium Sodium Chloride (ULASC) is promising NLO materials for device fabrication. Keeping this in view, attempts are made to grow and study the structural, Morphological and optical properties of ULASC single crystals.

2. EXPERIMENTAL STUDIES

2.1 Crystal Growth and Morphology

High purity chemicals (Merck 99.9 %) were used to grow crystals by slow evaporation technique. Equimolar amounts of L-alanine and Sodium Chloride were dissolved in double distilled water to prepare the aqueous solution of LASC. The chemical reaction is given below:

$$C_3H_7NO_2 + NaCl + H_2O \rightarrow C_3H_8ONaNO_2.HCl$$

The synthesized salt of LASC was doped with Urea (Merck 99.9%) and ULASC obtained by evaporating the solvent. The solvent was allowed to evaporate and numerous tiny crystals were formed at the bottom of the container due to spontaneous nucleation. The transparent and defect free ones among them were chosen as the seeds for growing bulk crystals. Good optical quality crystals of dimension up to 13 x 10 x 4 mm^3 were harvested after a period of 20 - 30 days. The photograph of as grown crystal of ULASC is shown in Figure 2.1.

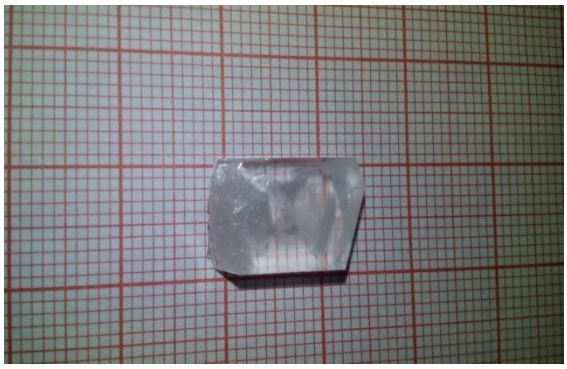

Fig. 2.1 As grown single crystal of ULASC

2.2 Characterization

The single crystal XRD was collected using an automated diffractometer (MESSRS ENRAF NONIUS CAD4-F, The Netherlands). The structure was solved by the direct method and refined by the full matrix least square technique using the SHELXL program. The Fourier transform infrared analysis was carried out between 400 and 4000 cm-1 by recording the spectrum using BRUKER

IFS 66V FT-IR SPECTROMETER. The surface morphology of the ULASC sample was recorded at room temperature using a JEOL/EO-JSM-5610 scanning electron microscope (SEM).

3. RESULT AND DISCUSSION

3.1 single X-ray diffraction studies

The unit cell parameters of ULASC were determined using 25 reflections collected through random search routine with graphite monochromated MoKα (λ = 0.71073 Å) radiation and indexed by the method of short vectors followed by the least squares refinement. Single crystal X-ray diffraction studies confirm that ULASC belongs to orthorhombic structure with space group $P2_12_12_1$. The unit cell parameters are found to be a = 6.045 Å, b = 12.258 Å, c = 5.392 Å and cell volume V = 399.545 $Å^3$. These values are in good agreement with the reported work [8].

3.2 FT-IR Analysis

Figure 3.1 shows the FT-IR spectrum of ULASC. The sample was prepared by mixing it with KBr. The absorption peaks at 3085 cm^{-1} corresponds to C-H asymmetric stretching. The peaks observed at 2937 and 2601cm^{-1} corresponds to OH stretching. The peak observed at 2112cm^{-1} is due to C≡O stretching. The peak observed at 1620 and 1593 cm^{-1} is due to C=O stretching. The peak observed at 1513$cm^{-1}$is due to N-H in plane bending. The peak observed at 1455, 1412, 1361 and 1305$cm^{-1}$is due to C-H bending in plane. The peak observed at 1235cm^{-1} is due to variable asymmetric C-O=C stretching. The peak observed at 1150cm^{-1} is due to asymmetric C-O=C stretching. The absorption peaks at 1112cm^{-1} corresponds to symmetric C-O=C stretching. The absorption peaks at 1014cm^{-1} corresponds to variable C-CHO stretching. The absorption peaks at 918 cm^{-1} corresponds to OH bending (out of plane bending).The absorption peaks at 849, 772 and 648cm^{-1} corresponds to C-H out of plane bending. The absorption peaks at 539 and 486 cm^{-1} corresponds to S-S stretching.

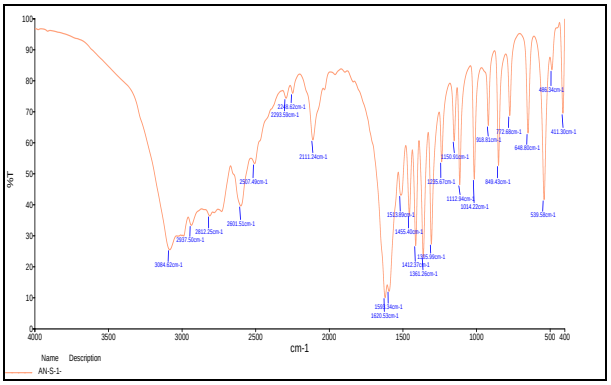

Fig. 3.1 FT-IR spectrum of ULASC

3.3 UV-Vis Spectroscopy

The UV-Vis-NIR spectrum gives information about the structure of molecule because the absorption of UV and visible light involves the promotion of the electrons in the σ and π orbitals from the ground state to higher energy states. The UV-Vis optical absorption spectra of the samples were recorded in the range of 200-1100 nm. The samples were cut into thin slabs of thickness 1.5-2 mm and then the spectra were recorded at room temperature. From the UV absorption spectrum (Fig.3.2), it is evident that ULASC crystal has UV cut-off around 260 nm and it can be seen from the absorbance curve that LPAN is absorption from 300 to 1100 nm, which is sufficiently low for SHG laser radiation at 1064 nm or other applications in the blue region [9].

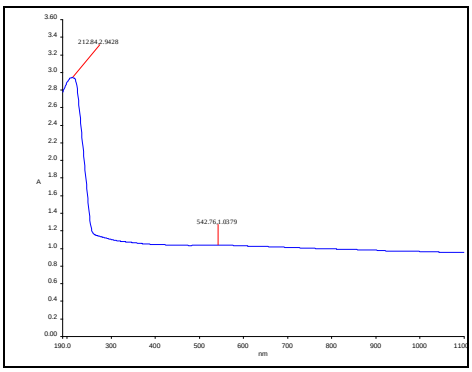

Fig.3.2 Optical absorption spectrum of ULASC

3.4 Non Linear Optical Test

Second harmonic generation test was done on the ULASC sample using Kurtz and Perry technique. The source used was Q-switched, mode-locked Nd^{3+}:YAG laser

emitting 1.06 μm fundamental radiation. The input laser beam was passed through IR reflector and then directed on the microcrystalline powdered sample packed in a capillary tube of diameter 0.154 mm. For the SHG efficiency measurements, microcrystalline material of KDP was used for comparison. When a laser input of 6.2 mJ was passed through ULASC, second harmonic signal of 173 mV is produced and the experiment confirms a second harmonic efficiency of nearly 1.4 times that of KDP (124 mV). Thus SHG efficiency of ULASC sample is comparable with other promising amino acid based NLO crystals.

3.5 Scanning Electron Microscopy analysis

The surface morphology of the ULASC sample was recorded at room temperature using a JEOL/EO-JSM-5610 scanning electron microscope (SEM). Figure 3.9 presents the SEM micrograph taken on the as grown crystal with urea does not show much void/gap, therefore, confirm that sample is of high density. It indicates the appearance of smooth surface. It is observed that few micro particles are also seen on the natural crystal faces, which shows that it can add more molecules to grow in to a large crystal.

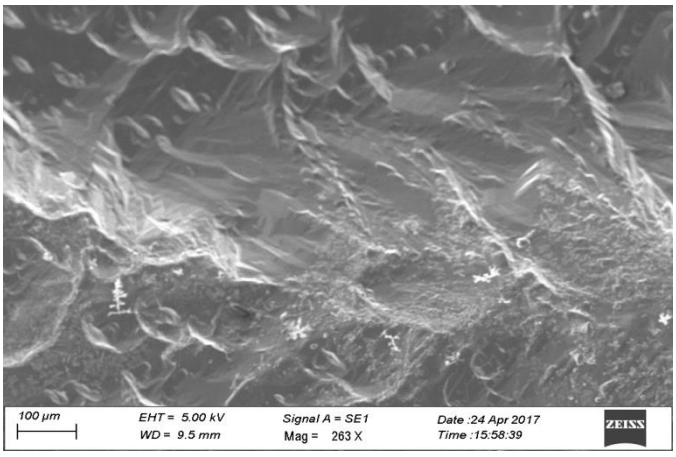

Fig.3.3 SEM micrograph of ULASC

4. CONCLUSIONS

Single crystals of ULASC are conveniently grown by slow solvent evaporation technique. It is estimated from the X-ray diffraction studies that ULASC crystals are orthorhombic structure with space group $P2_12_12_1$. The UV-Vis-NIR spectrum reveals the wider transmission window of ULASC and a low cut-off wavelength of 260 nm. From the SEM analysis shows that the crystal may grow in larger size for device fabrication.

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