



Growth and characterization of metal ions doped L-serine NLO single crystals for optoelectronic applications



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ABSTRACT

Single crystals of pure and metal ions (K^+ , Na^+ and Li^+) doped L-serine have been grown by slow evaporation solution growth technique (SEST). Energy dispersive X-ray analysis (EDAX) and inductively coupled plasma optical emission spectroscopy (ICP-OES) prove the incorporation of metal ions into the doped crystals. The lattice parameters have been obtained from single crystal X-ray diffraction (XRD) studies. The presence of functional groups is identified by Fourier transform infrared (FTIR) and FT-RAMAN analyses. The thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) reveal that the thermal stability of lithium doped L-serine crystal is enhanced. The mechanical properties have been studied by vicker's microhardness test. UV-vis-NIR spectroscopy shows that the percentage of transmission is increased in lithium doped L-serine crystals. Potassium and lithium ions doped L-serine crystals have enhanced second harmonic generation (SHG) efficiency indicating that these crystals are potential candidates for non linear optical (NLO) applications.

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1. Introduction

Nowadays NLO materials are having potential applications in the area of telecommunication and optical storage devices [1]. Amino acid crystals are potential candidates for optical second harmonic generation because all amino acids except glycine contain chiral carbon atom and crystallize in non-centrosymmetric space groups. L-Serine is an organic compound under amino acid category. It is one of the naturally occurring proteinogenic amino acids and it exists in zwitter ionic form. The molecule can combine with anionic, cationic and overall neutral constituents. L-Serine crystal belongs to orthorhombic crystal system with space group $P2_12_12_1$.

The presence of small amount of impurities in a crystallisation system can modify the properties, growth kinetics and morphology of the crystal. For NLO applications, a new and better material with modified chemical and physical parameters is needed. Recent reports reveal that the SHG efficiency and mechanical strength of the single crystals are enhanced by substituting the metal ions as minor impurities during crystal growth [2–6]. These reports prompted the present investigation on the effect of metal ions on the properties of L-serine single crystals.

A series of metal organic compounds such as Cu^{2+} and Mg^{2+} doped L-arginine trifluoroacetate crystal, metal ions doped L-lysine monohydrochloride dihydrate single crystal have been reported with improved chemical stability, mechanical and optical properties [7,8]. The aim of the present communication is to study the effect of K^+ , Na^+ and Li^+ ions on the growth, physical and chemical properties of L-serine crystals that may find wide applications in optoelectronic devices.

2. Experimental procedure

2.1. Crystal growth

The calculated amount of L-serine (SRL) was dissolved in deionised water and stirred well to ensure homogeneous solution. The saturated solution was filtered in a beaker and covered with a perforated cover and kept undisturbed for slow evaporation at room temperature. For the growth of the LSPN (L-serine doped with potassium nitrate) and LSSN (L-serine doped with sodium nitrate), LSLN (L-serine doped with lithium nitrate) crystals, 1 M% of potassium nitrate, sodium nitrate and lithium nitrate, were added to the saturated solution of L-serine respectively. The growth condition was optimised and the grown crystals were further purified by successive recrystallization. The pure and the doped crystals of L-serine with good quality and well defined morphology were harvested after a period of 25 days. Fig. 1 shows the photographs of optical quality single crystals of pure and doped L-serine.

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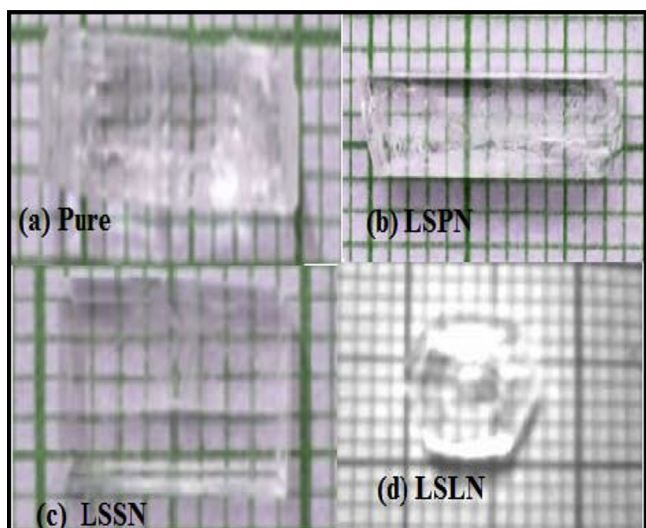


Fig. 1. Photographs of (a) pure L-serine, (b) LSPN, (c) LSSN, and (d) LSLN crystals.

2.2. Characterization

The presence of metal ions in the crystal lattice of the grown crystal was determined by EDAX and ICP analyses. The single crystal XRD studies were carried out by employing Enraf Nonius CAD 4 diffractometer. The FTIR spectrum of the sample was recorded between 4000 cm^{-1} and 400 cm^{-1} using BRUKER spectrometer by KBr Pellet technique. The FT-Raman spectrum of the grown crystal was recorded using BRUKER:RFS 27 FT-Raman spectrometer. Thermal analyses were carried out by employing NETZSCHSTA 409 in the temperature range from $50\text{ }^{\circ}\text{C}$ to $750\text{ }^{\circ}\text{C}$ at the heating rate of $10\text{ }^{\circ}\text{C}/\text{min}$ in nitrogen atmosphere. The Vicker's microhardness test was carried out to know about the mechanical strength of the crystals. The optical transmittance spectra were recorded using Lambda-35 UV-vis-NIR spectrometer. The SHG measurements were performed on the powdered crystals using Kurtz Perry powder technique.

3. Results and discussion

3.1. EDAX and ICP analyses

Small crystal of LSPN was subjected to EDAX analysis. The powdered samples of LSSN and LSLN were subjected to ICP analysis, as the presence of sodium and lithium ions cannot be detected by EDAX analysis due to their low atomic numbers. The results are displayed in Table 1.

The presence of K^+ , Na^+ and Li^+ metal ions were traced. The low percentage of incorporation of dopants may be due to their larger ionic radii. Even though the amount of doping is less, it plays an important role in the enhancement of NLO property in the doped crystals.

Table 1
EDAX and ICP of doped L-serine crystals.

Material	EDAX analysis	ICP analysis
LSPN	0.07 Wt% of K	–
LSSN	–	0.749 mg/L of Na
LSLN	–	0.481 mg/L of Li

Table 2
Single crystal XRD of pure and doped L-serine crystals.

Cell parameters	L-Serine (pure) [9]	LSPN	LSSN	LSLN
a (A.U)	8.571	8.595	8.52	8.553
b (A.U)	9.325	9.342	9.25	9.306
c (A.U)	5.615	5.621	5.565	5.594
$\alpha = \beta = \gamma$	90°	90°	90°	90°
Cell volume (A.U ³)	448.7	451.3	438	445.3

3.2. Single crystal XRD analysis

The single crystals of doped L-serine were subjected to single crystal XRD. The lattice parameters are compared with the reported values of the pure L-serine crystals [9] and are listed in Table 2. It is observed that the pure and the doped L-serine crystals crystallize in orthorhombic structure and belong to $\text{P2}_1\text{2}_1\text{2}_1$ space group.

3.3. FTIR and FT-Raman studies

Spectroscopic studies were employed to elucidate the presence of functional groups. IR and Raman vibrational spectra of pure and doped L-serine crystals are shown in Figs. 2 and 3, while the wave numbers and the assignments of some characteristic vibrations are listed in Table 3. The FTIR and Raman spectra of pure L-serine agree well with the literature values [10,11].

The absorption band at 3453 cm^{-1} is assigned to OH stretching vibrations (caused by absorption of water by KBr). The absorption band at 3095 cm^{-1} is caused by NH_3^+ symmetric stretching vibrations. The peaks at 2997 cm^{-1} , 2959 cm^{-1} and 2904 cm^{-1} in Raman spectrum are due to CH_2 and CH groups. Peaks in the region $2039\text{--}2728\text{ cm}^{-1}$ in FTIR are caused by sum tones. These tones are absent in Raman spectra which confirm the assignments [12]. COO^- anti symmetric stretching vibrations are observed in the region between 1599 cm^{-1} and 1627 cm^{-1} . The peaks at 1467 cm^{-1} and 1413 cm^{-1} are attributed to CH_2 bending and COO^- stretching vibrations respectively. The peaks around 1380 cm^{-1} in doped crystals which are assigned to stretching vibration of nitro group are absent in pure L-serine crystal. It confirms the inclusion of nitro

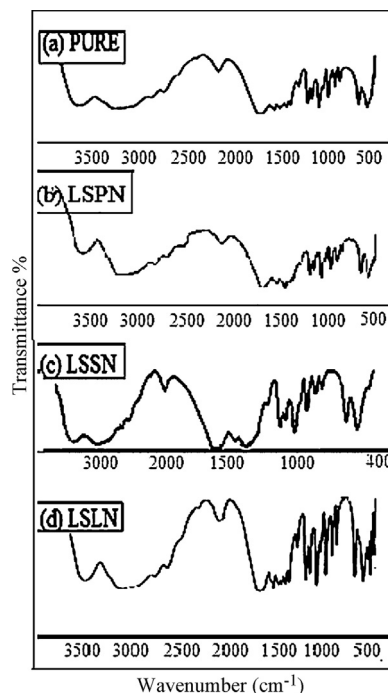


Fig. 2. FTIR spectra of (a) pure L-serine, (b) LSPN, (c) LSSN, and (d) LSLN crystals.

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