Growth and Characterization of Sulphamic Acid and L-Methionine Added Sulphamic Acid Single Crystals

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ABSTRACT

Sulphamic acid (SA) and L-methionine added sulphamic acid crystals (LMSA) were grown by slow evaporation technique at room temperature. The Single crystal XRD studies revealed that the grown crystals possessed orthorhombic crystal structure. The presence of various functional modes was confirmed by RAMAN analysis. The EDAX analysis confirmed the presence of L-methionine in the grown LMSA crystal. The surface morphology of crystals was studied. The larger size crystals revealed the excellent crystallinity. The stability of the crystals was ascertained from thermo gravimetric and differential thermal analysis. The third order nonlinear refractive index, nonlinear absorption coefficient and third order optical susceptibility were calculated by the Z-scan technique.

Keywords: L-methionine, Sulphamic acid, Slow evaporation, Transparent crystal.

INTRODUCTION

The creation of passive and active photonic devices primarily makes use of nonlinear optical materials [1]. Optics and electronics are two fields where the utilization of single crystals is evident. Sulphamic acid (SA) is one of the most generally used inorganic compounds in industry [3]. In multistage flash evaporation (MSF) desalination plants, SA identifies good applications for heat exchangers, demisters, cleaning, cooling water systems, etc. [2]. Both orthorhombic structure and zwitterionic form of sulfuric acid are present in SA. At low temperatures, large single crystals of pure SA can be formed. Due to these outstanding qualities, SA has been recognised as a standard material for titrimetric analysis by the British analytical techniques committee, IUPAC, and Japanese Industrial Standard (JIS) [4-6].SA has large applications as anti-corrosive agent [7, 8]. Both pure and doped SA crystals have been reported so far by various methods such as slow cooling method, unidirectional solution growth method, aqueous solution growth method etc. These studies also suggested the reported crystals for optoelectronics and various device applications. In addition to that studies have shown that dopping methionine plays an important role in the improvement of crystallization and the growth behaviour of crystals [9-21].

Sulphamic acid has found wide application as a catalyst, especially in polymerization process and used as a catalyst for many organic reactions. It is used as a fire retardant of fire proofing agent, as a cross-linking agent for polymers, in the dyeing of fabrics, as a cleaning agent for air, as herbicide, in pulp bleaching, for scale removal and cleaning metal, in pigment printing, and in producing cigarettes of reduced toxicity [22]. Dopants play a crucial role in improving the physical properties of single crystals [23]. Several researchers have investigated the incorporation of L-methionine with both organic and inorganic materials. Studies have demonstrated that single crystals, such as ammonium dihydrogen phosphate and potassium dihydrogen phosphate doped with L-methionine, exhibit improved nonlinear optical (NLO) properties and enhanced sensor applications compared to the pure one [24-26]. To best of our knowledge L-Methionine added Sulphamic acid crystal was not grown by any other researcher. In this present work pure sulphamic acid crystal and L-methionine added sulphamic acid crystals were grown by slow evaporation technique at room temperature and the structural and optical properties were discussed.

EXPERIMENTAL METHODS

Table 1 Experimental details of SA and LMSA crystal growth

Parameter	**SA Crystal**	**LMSA Crystal**
Solvent	Double-distilled water	Double-distilled water
Growth method	Slow evaporation technique	Slow evaporation technique
Growth temperature	Ambient temperature (28–32°C)	Ambient temperature (28–32°C)
Stirring duration	3–4 hours	3–4 hours
Crystallization period	20 days	20 days
Number of growth batches	Multiple batches	Multiple batches
Reproducibility of growth	Consistent crystal growth observed	Consistent incorporation of L-methionine and crystal quality observed

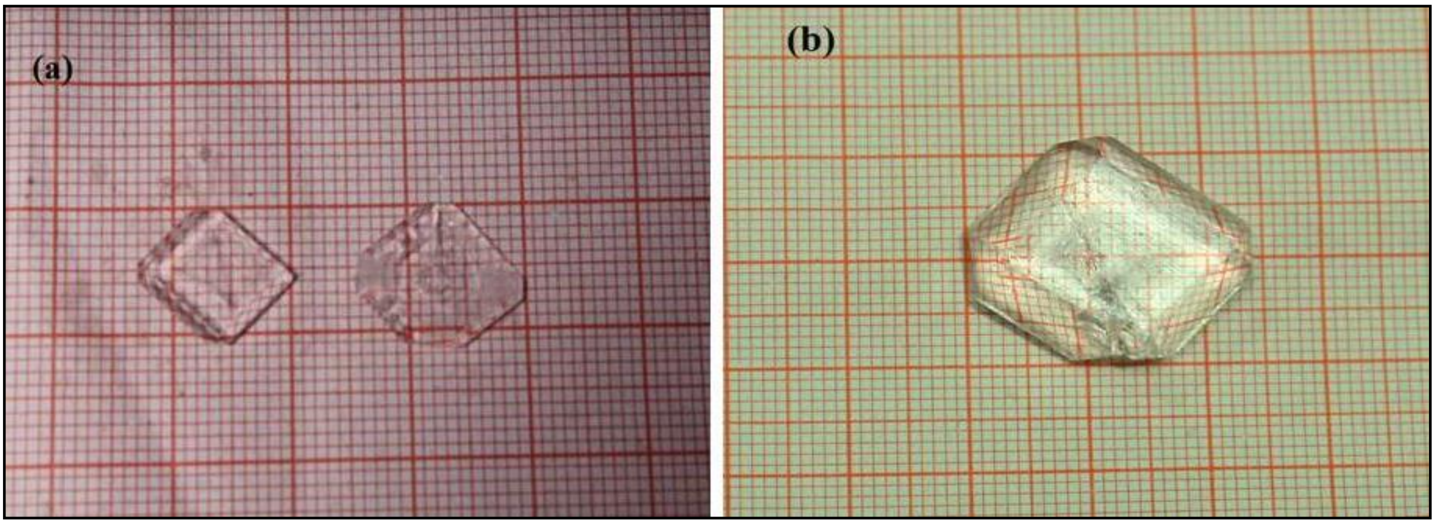

Fig 1 Photographs of the grown a) SA crystal and b) LMSA crystal

Sulphamic acid crystal and sulphamic acid crystal containing L-methionine were produced via slow evaporation method. Sulphamic acid crystal is prepared by disolving sulphamic acid in double-distilled water. For sulphamic acid crystal containing L-methionine, L-methionine powder and high purity sulphamic acid are employed as the initial components in which SA and L-methionine are combined in a 3:1 ratio and dissolved in double-distilled water. For a few hours at room temperature, the dissolved solutions were agitated with a magnetic stirrer to produce a clear solution in separate beakers. The translucent saturated solutions were filtered through filter papers and left to crystallize at ambient temperature in a purified environment. After 20 days, nice transparent crystals that were colorless were gathered and used for characterisation. Fig 1 depicts images of grown crystals. The experimental parameters employed for the growth of SA and LMSA crystals are presented in Table 1. The growth conditions were maintained identical for all batches to ensure reproducibility of the doping effect.

RESULTS AND DISCUSSIONS

Single crystal X-ray diffraction

Single crystal X-ray Diffraction was investigated using BRUKER AXS KAPPA APEX 11 CCD Diffractometer. Table 2 presents the structures and lattice parameters of SA and LMSA single crystals. The measured lattice parameters for SA are a=8.10 Å, b=8.17 Å, c=9.29 Å and $\alpha = \beta = \gamma = 90°$. SA has unit cell volume of 614 Å m^3 and the density is 2.129 mg/m^3. The obtained lattice parameters for LMSA are a=8.12 Å, b=8.16 Å, c=9.26 Å and $\alpha = \beta = \gamma = 90°$. The unit cell volume for LMSA is 615 Å m^3 and the density of cell volume is calculated as 2.129 Mg/m^3. The slight increase in unit cell volume (614 to 615 $Å^3$) suggests that L-methionine is incorporated

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interstitially without altering the orthorhombic structure of sulphamic acid (SA). In its zwitterionic form ($^{+}NH_3$–SO_3^-), SA provides active sites for interaction with L-methionine functional groups (–NH_2, –COOH, –SCH_3). Hydrogen bonding between –NH_2/–COOH of L-methionine and –SO_3^-/–NH_3^+ of SA, along with weak van der Waals interactions from the –SCH_3 group, leads to slight lattice expansion. Similar lattice modifications have been reported in amino acid-doped crystals, such as DL-methionine doped ADP [24]. These additional intermolecular interactions also account for the observed enhancement in thermal stability of LMSA.The single crystal XRD study revealed that both the grown crystals belong to orthorhombic crystal system with a space group of Pbca, which shows the addition of Methionine has not altered the structure of SA.

Table 2 Structure and lattice parameters of pure SA and LMSA single crystals.

Crystal	Structure	A (A°)	B(A°)	C(A°)	Volume (m³)
SA	Orthorhombic (α=β=γ= 90°)	8.10	8.17	9.29	614
LMSA	Orthorhombic (α=β=γ= 90°)	8.12	8.16	9.26	615

Raman analysis

Fig 2 displays the Raman spectra of SA and LMSA crystal. The Raman spectra of SA and LMSA crystals revealed the distinct vibrational bands characteristic of their structural features. The Raman spectra of SA displayed in Fig 2 (a) contains a high-intensity vibration band observed near 1059 cm^{-1} was attributed to the symmetric SO_3 stretching vibration. The bands at 1281 cm^{-1} and 1345 cm^{-1} corresponded to degenerate SO_3 stretching modes. A band at 1017 cm^{-1} was assigned to the degenerate NH_3^+ rocking vibration, while the N–S stretching vibration was observed at 682 cm^{-1} [27]. Additionally, the vibration band at 543 cm^{-1} was ascribed to degenerate SO_3 deformation, and the band at 363 cm^{-1} was due to SO_3 rocking vibrations. The vibration band detected at 256 cm^{-1} was assigned to N–S torsion [28]. All the Raman modes confirmed the orthorhombic structure of the SA crystal as displayed in the Raman spectra. Hence, the structure of the SA crystal was conclusively validated.

The bands seen at the Fig 2(b) corresponds to LMSA spectra. 372 $cm^{-1}$and 242 cm^{-1} peak positions were caused by SO_3^- rocking and N-S torsion vibrations, respectively. The 550 cm^{-1} LMSA vibration bands were attributed to degraded SO_3^- deformation. The band at 689 cm^{-1} of LMSA was caused by C-S astretching mode of vibration [29] . The band, which appeared at 1000 cm^{-1}, showed the arocking avibration of NH_3^+ in L-methionine with sulphamic acid addition. At 1352 cm^{-1} and 1279 cm^{-1} in LMSA, the degraded SO_3^- stretching vibration emerged. The band at 2437 $cm^{-1}$was caused by S-H stretching vibration, and the peak at 1539 $cm^{-1}$was related to NH_3^+deformation. C-H stretching mode was assigned to the band seen at 2873 cm^{-1}[30]. The NH_3^+ stretching vibration linked with the vibration bands noted at 3048 cm^{-1} in LMSA [31-36]. When comparing SA and LMSA, only a negligible peak shift is observed. This proved that the orthorhombic structure was maintained despite the alteration in sub-lattice geometry.

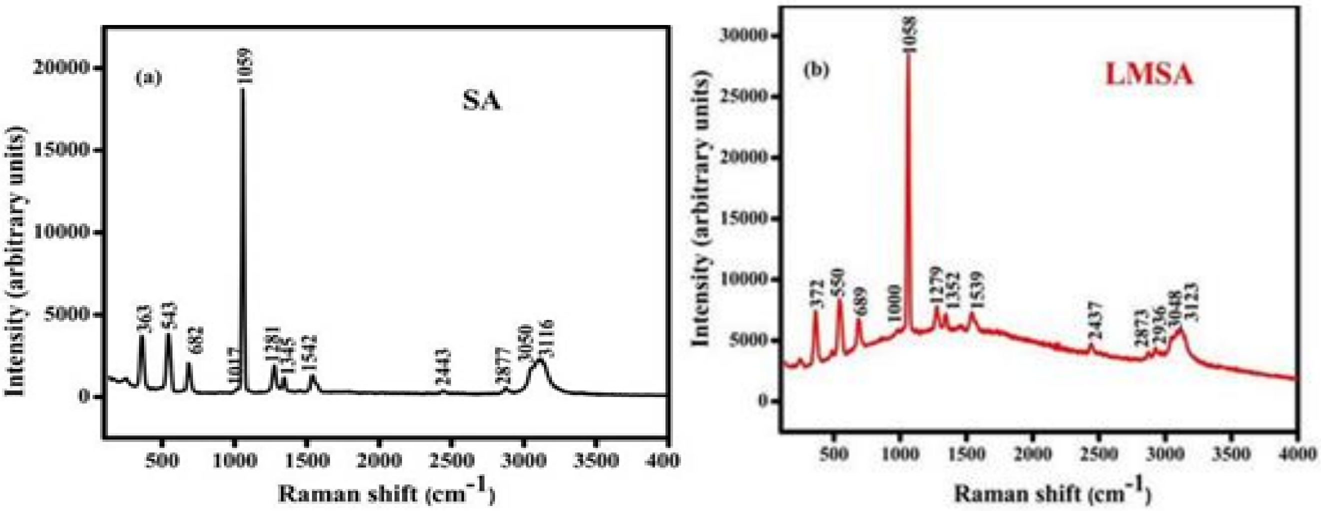

Fig. 2 Raman spectra of a) SA and b) LMSA crystals

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Morphology and compositional analysis

Using scanning electron microscope (SEM), the surface morphology of the SA and LMSA crystals were examined; the image is shown in Fig 3. The SEM scans explored that the particles were not all the same size and that voids and agglomerations were present. The thermal effect (during the synthesis process) causes the crystals overlapping which may be owing to the agglomeration of crystal nucleus. The agglomeration of crystal nuclei may be the reason of the crystals' overlap caused by the temperature effect during the synthesis process. Few spherical shaped smooth microcrystals were grown on the surface. Surface morphology of the crystal is smooth and is crack free. The particle sizes of the SA and LMSA crystals were analyzed using ImageJ. The particle size of the SA crystal was found to be 100 µm, while that of the LMSA crystal was 140 µm. Compared to SA, the LMSA crystal exhibited an increase in particle size.

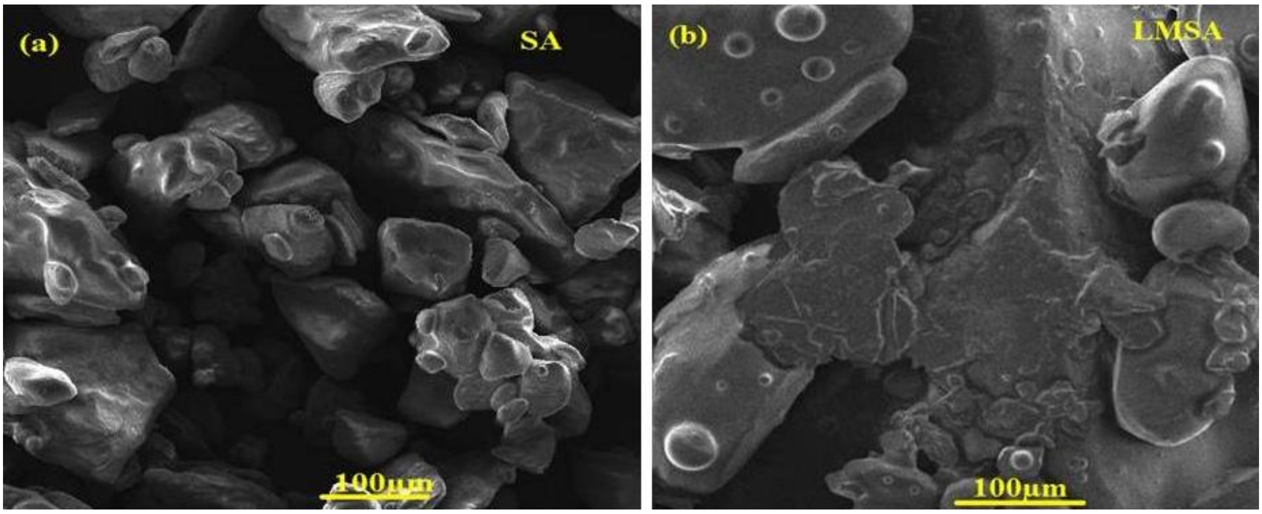

Fig. 3 SEM analysis of a) SA and b) LMSA crystals

The EDAX spectrum of SA and LMSA is shown in Fig. 4. The SA crystal consists of oxygen, nitrogen, and sulfur, while the L-methionine-added sulfamic acid (LMSA) crystal contains oxygen, nitrogen, sulfur, and carbon. The presence of carbon in LMSA confirms the incorporation of L-methionine into the SA crystals.In Table 3a and 3b, the elemental compositions of SA and LMSA are listed.

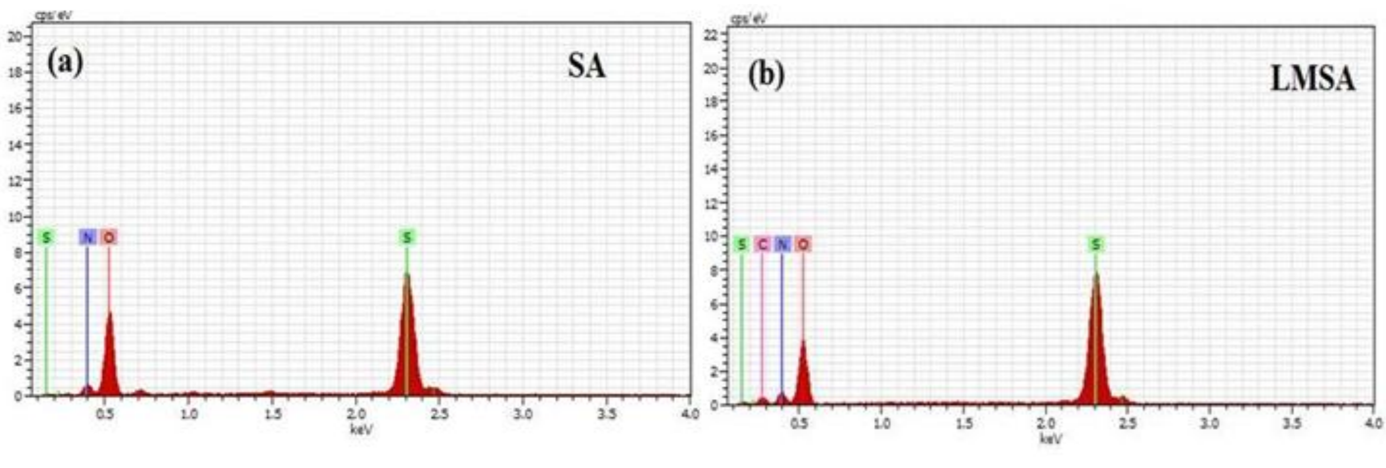

Fig. 4 EDAX analysis of a) SA and b) LMSA crystals

Table 3a Elemental composition of pure SA

Element	Molecular weight (%)	Atomic weight (%)
Oxygen	63.26	69.71
Nitrogen	14.23	17.91
Sulphur	22.51	12.37

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Table 3b Elemental composition of LMSA

Element	Molecular weight (%)	Atomic weight (%)
Oxygen	51.62	53.75
Nitrogen	17.03	20.25
Sulphur	20.14	10.46
Carbon	11.20	15.54

TG/DTA/DTG Analysis

TG/DTA/DTG analysis is a quite useful tool to determine the phase transition of the formed crystal, the water of crystallization, also reliable information's like thermal stability, melting point and decomposition of the grown crystal[37]. Fig 5 displays the results of a TG/DTA/DTG study of SA and L-methionine added in SA. The graph indicates that no significant weight loss was observed for the SA crystal up to 261°C. A sudden and substantial weight reduction occurred between 261°C and 448 °C, as evidenced by a sharp peak at 448 °C, confirming the crystal's decomposition at this temperature, consistent with the findings of Brahmaji et al. [15]. In the DTA curve, endothermic peaks appeared at 212 °C and 261 °C, signifying the complete evaporation of water and solvent molecules. As the temperature increased further, the material experienced weight loss, corresponding to its decomposition, as indicated by the pronounced endothermic peak at 448 °C, which aligns with the TGA curve. For LMSA, no significant weight loss was observed up to 265 °C. The weight loss starts from 265 °C and a rapid weight loss was observed at 453 °C. This manner of weight loss revealed the material's breakdown point at 453 °C. Two exothermic peaks were observed at 209 °C and 265 °C which corresponds to the first and second decomposition stage respectively. The DTA curve shows a strong endothermic peak at 453 °C, which represent the last decomposition state. It is noticed that LMSA crystals are therefore thermally stable up to 265 °C. In light of this, we can state that the crystals that have formed are suitable for applications with working temperatures of up to 265 °C. Compared to SA, the LMSA crystal exhibited minimal variation; however, the temperature shifts toward a higher range. This suggests that the addition of L-methionine to sulphamic acid enhances its thermal stability compared to pure sulphamic acid.

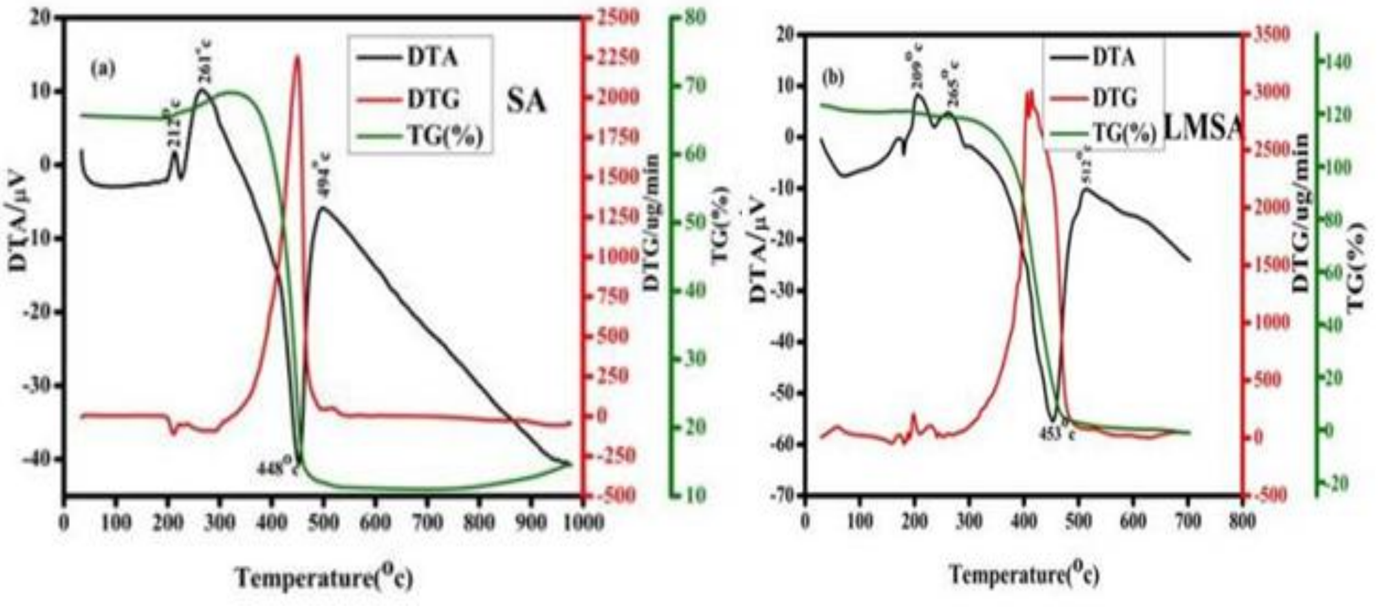

Fig. 5 TG-DTA curves of a)SA and b)LMSA crystal

Z-scan measurements

The third-order nonlinear optical characteristics of a 1 mm thick LMSA crystal were estimated using the Z-scan technique. A semiconductor continuous wave laser with a wavelength of 532 nm and a power of 100 mW was used to perform the Z-scan experiment. A convex lens with a focal length of 103 mm was employed to focus the crystals. Z-scan is a very good technology for analyzing the nonlinear absorption coefficient (β) and third order

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nonlinear refractive index (n_2). Fig 6 and 7 depict the graph obtained using the SA and LMSA crystals open aperture and closed aperture methods, respectively. Closed aperture Z-scan curve of both ZA and LMSA indicate the valley followed by a peak, which reveals that the positive nonlinearity with a self-focusing effect [38]. Since the laser used is continuous wave the nonlinearity observed in the sample is purely thermal [39]. Here, the nonlinear absorption coefficient (β) was determined using an open aperture Z-scan technique, which occurs in the SA and LMSA and is represented by a valley with a positive absorption coefficient (β). For the purpose of getting any optical limiting applications, this property is crucial. The third order nonlinear refractive index (n_2) values were determined as 4.72 x 10^{-9} cm^2/w and 2.42 x 10^{-9} cm^2/w, nonlinear absorption coefficient (β)= 2.79 x 10^{-4} cm/w and 2.72 x 10^{-4} cm/w, the positive value of nonlinear refraction revealed the self-focusing nature. Real and imaginary value of third order susceptibility of SA and LMSA are 5.48 x 10^{-6} cm^2/w, 1.09 x 10^{-6} cm/w and 2.98 x 10^{-6} cm^2/w, 1.22 x 10^{-6} cm/w respectively. The third order susceptibility ($\chi^{(3)}$) of SA and LMSA are 5.58 x 10^{-6}esu and 2.82 x 10^{-6}esu respectively. It is important to note that the third-order nonlinear optical susceptibility ($\chi^{(3)}$) of LMSA is lower than that of pure SA. This reduction suggests that the incorporation of L-methionine does not enhance the third-order nonlinear response of sulphamic acid under the present growth conditions. The observed decrease in ($\chi^{(3)}$) is significantly larger than the typical experimental uncertainty associated with CW Z-scan measurements, indicating that the reduction is likely intrinsic rather than arising from experimental error alone. Several factors may contribute to this behavior. First, the incorporation of L-methionine molecules into the SA lattice may dilute the density of highly NLO-active sulphamic acid units, thereby reducing the effective nonlinear polarization. Second, the addition of methionine may introduce localized lattice defects or microstrain, as evidenced by the slight lattice expansion observed in single-crystal XRD analysis, which can increase scattering losses and reduce nonlinear efficiency. Third, intermolecular interactions between the functional groups of methionine ($-NH_2$,$-COOH$,$-SCH_3$) and the zwitterionic SA framework may alter charge-transfer pathways and weaken the electronic delocalization responsible for nonlinear optical response. Such modifications in charge distribution can influence the third-order susceptibility significantly.

Despite the reduction in ($\chi^{(3)}$), the LMSA crystal still exhibits appreciable nonlinear optical behavior comparable to several reported amino acid-based semi-organic NLO crystals. For example, L-methionine barium bromide (LMBB) crystals reported by Shalini et al. [26] showed effective optical limiting behavior with third-order nonlinear optical response in the same order of magnitude. Similarly, L-methioninium hydrogen maleate crystals reported by Natarajan et al. [16] demonstrated useful NLO characteristics arising from intermolecular hydrogen bonding and charge-transfer interactions. The ($\chi^{(3)}$)value obtained for LMSA remains sufficiently high for potential applications in optical limiting, optical switching, and photonic device fabrication [40]. Furthermore, LMSA offers the additional advantage of improved thermal stability compared to pure SA, which may be beneficial for practical device operation under elevated temperature conditions.

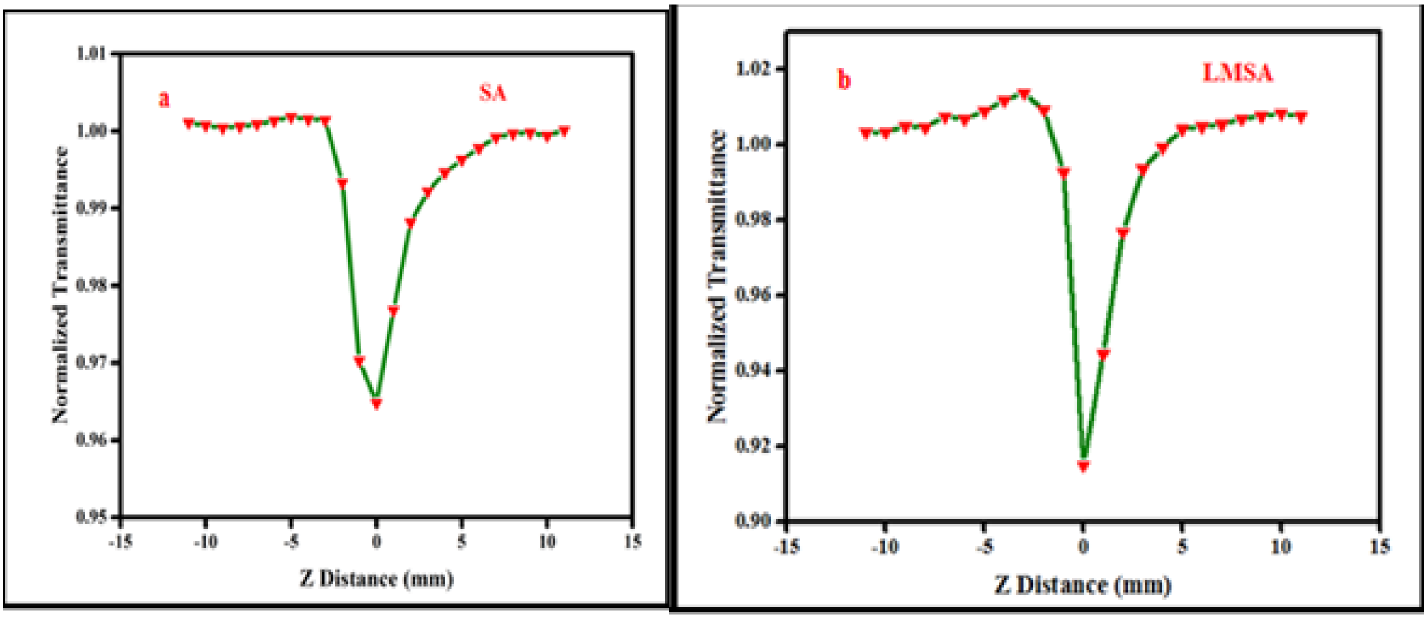

Fig. 6 Z-scan open aperture of a) SA and b) LMSA crystal

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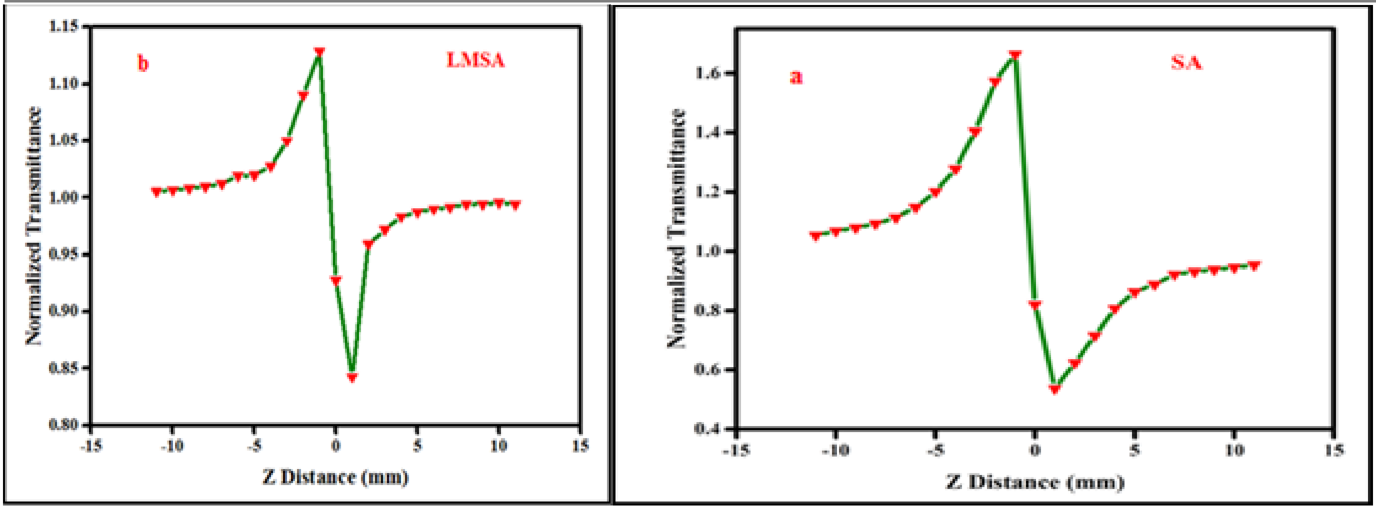

Fig. 7 Z-scan closed aperture of a) SA and b) LMSA crystal

CONCLUSION

The sulphamic acid (SA) and L-methionine added sulphamic acid (LMSA) crystals were successfully grown by the slow evaporation technique at room temperature. Single crystal XRD analysis confirmed that both crystals belong to the orthorhombic crystal system. Raman and EDAX analyses verified the incorporation of L-methionine into the SA lattice without altering the crystal structure. TG-DTA-DTG studies revealed that the LMSA crystal exhibits enhanced thermal stability up to 265°C compared to pure SA, making it suitable for optoelectronic devices operating at elevated temperatures. Z-scan measurements confirmed positive nonlinear refractive index and nonlinear absorption coefficients, indicating self-focusing and optical limiting behavior. The obtained n_2 and β values suggest that the grown crystals are promising candidates for third-order nonlinear optical applications such as optical limiters, optical switches, optical data processing devices, and frequency conversion applications. The improved thermal stability and nonlinear optical response of LMSA demonstrate its potential advantage over pure SA for practical photonic and optoelectronic applications.

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