Effect of L-Isolucine on the growth of Sulphamic acid Single Crystals

Fredselin R.S vithel[1]and R. Manimekalai[1*]

[1]Research scholar, Department of Physics, A.V.V.M Sripushpam College, Poondi, Tamil Nadu, India.

[1*]PG and ResearchDepartment of Physics, A.V.V.M Sripushpam College, Poondi, Tamil Nadu, India.

*Corresponding Author: maniabi64@gmail.com

Abstract

The crystals of sulphamic acid (SA) and L-isolucine added sulphamic acid (LISA) were grown by slow evaporation method at room temperature. The XRD analysis revealed that both SA and LISA crystals exhibited orthorhombic crystal structure. The thermal stability of the grown crystals were found by thermo gravimetric and differential thermal analysis (TGA/DTA), and exhibited that the grown crystals were thermally stable up to 261^{o}C for pure SA and 257^{o}C for GSA crystals. The antibacterial activity of the grown was analyzed. The third order nonlinear refractive index, absorption coefficient and optical susceptibility were calculated by the Z-scan technique.

Keywords: Crystal growth, sulphamic acid, Single XRD, TG-DTA, antibacterial activity, Z-scan.

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1. Introduction

Presently huge awareness has been devoted to the growth and characterization of pure and doped bulk single crystals to identify new materials for numerousapplications. It has played an important role in latest technology due to their supreme physical and chemical properties. Therefore a huge research activities have been focussed on the growth and characterization of pure and additive single crystals [1-4].To strengthen the properties of crystals generally dopants/additives have been added [5]. The inorganic crystals are familiar in the area of photonics, owing to high speed information processing, high optical data disk storage, frequency conversion and optical communication [6-11]. Amid overall inorganic crystals, sulphamic acid has got great observation because of its high thermal and chemical stability, better transmittance and good optical and nonlinear properties [12-15].

It is well known that sulphamic acid (H_2NSO_3H, SA) and its derivatives get distinctive observation in NLO property due to its two planar rings which arise from the strong donor-acceptor intermolecular interaction and delocalized π-electron configuration and having excellent blue light transmittance property [16-17]. Amid overall inorganic acids, sulphamic acid is somewhat invariable and crystallizes in non-centrosymmetric structure and hence gives good NLO property. Sulphamic acid is an odorless non-volatile solid. It is also non-hygroscopic and non-corrosive. Further it serves as a good green catalyst in organic synthesis, with outstanding piezoelectric and NLO properties. Sulphamic acid is an inorganic material having orthorhombic structure along with Pbca space group. It forms Zwitterions when dissolved in water and acquires well known catalytic features which prevail to remote from other acidic catalysts [7, 18-23]. Due to these advantages, the Japanese industrial committee has qualified SA as the standard substance for titrimetric analysis [24].

Owing to high thermal stability, elevated catalytic behavior and superior optical properties many research activities have been completed based on sulphamic acid with various dopants and additives. Previous studies revealed that SA was doped with various rare earth elements like lanthanum, dysprosium, terbium, neodymium, cesium, gadolinium, Yttrium and cerium and also with metallic elements copper, manganese, and nickel. Moreover sulphamic acid has been doped with compounds like LiCl,NaCl and KCl,$ZnSo_4$& $MnSo_4$, $FeSo_4$, Al_2Cl_3 and thiourea [6-8, 13-14, 16-17, 25-33].Here,in the present work the amino acid and L-isolucine added with sulphamic acid and the crystals were grown by slow

evaporation method and its structural, thermal and optical properties were investigated.

2. Experimental Methods

The crystals of pure sulphamic acid and L-isolucine added sulphamic acid were grown by slow evaporation technique using the AR grade sulphamic acid and L-isolucine powders. To grow L-isolucine added sulphamic acid crystal the AR grade sulphamic acid and L-isolucine in the molar ratio 3:1 were taken and dissolved in deionized water. The dissolved mixture was stirred well using magnetic stirrer for four hours at room temperature until well liquefied. The saturated solution was filtered using a filter paper to remove any non-miscible and unstirred chemical. The filtered transparent solution was shifted to crystal growth vessels, enclosed by aluminum foil sheet with few punched holes and stored in a dust free atmosphere. The solution was permitted to crystallization at room temperature. Good transparent and colorless crystals were collected after 20 days. Fig. 1shows the photograph of the grown sulphamic acid (named as SA) and L-isolucine added sulphamic acid (named as LISA) crystals.

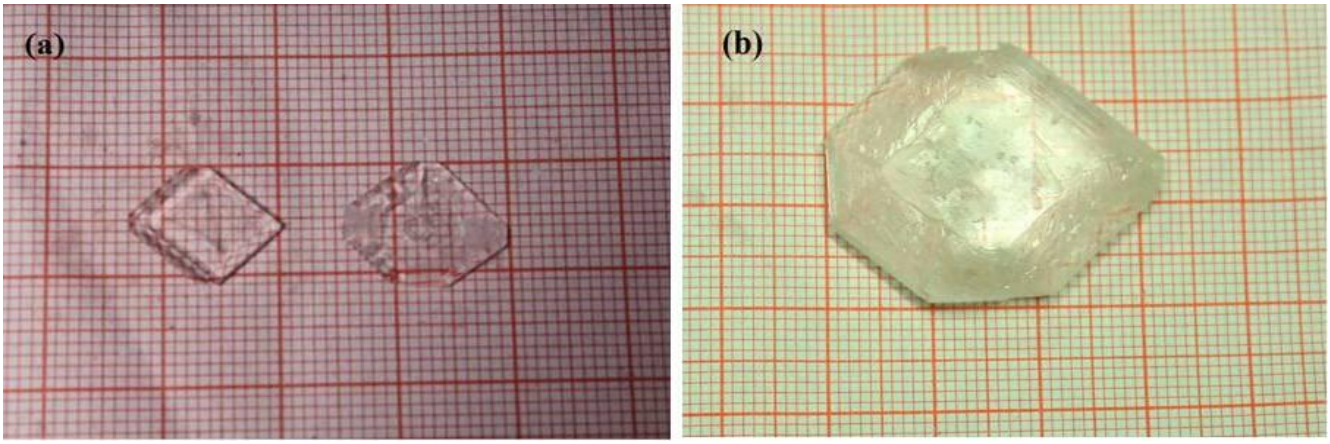

Figure1. Photographs of the grown crystals (a) SA (b) LISA

3. Results and discussions

3.1. Single crystal X-ray diffraction

The single crytal X-ray diffraction analysis was studied using BRUKER AXS KAPPA APEX 11 CCD Diffractometer. L-isolucine added sulphamic acid single crystals possessed orthogonal geometry. The cell parameter values were a=8.21A^{o} , b=8.09A^{o}, c=9.28A^{o} with angles α=β=γ=90^{o} and cell volume were 603 A^{3}. The a, b, c values varied as compared to that of pure sulphamic acid. The addition of L-isolucine did not change the crystal structure. The cell axes and the cell volume were changed slightly compared to pure SA.

3.2. TG-DTA Analysis

Thermogravimetry (TG) and Differential Thermal Analysis (DTA) of SA and LISA crystals were done at a heating rate of 10^{o}C/min from 27 ^{o}C to 800^{o}Canalysis in the N_2 atmosphere. Fig. 2 shows the TG-DTA curves of SA and LISA crystals. It was found that both crystals follows the same trend up to approximately 261 ^{o}C and no weight loss has been observed up to this temperature. A sudden drop was appeared at 261 ^{o}C and lowers down up to 448^{o}C which denotes the weight loss due to the evaporation of all solvent material and water molecules. Similar peak drop was observed in previous reports related to the decomposition temperature of the crystal [7]. From DTA curves it understood that the endothermic peaks occurred at approximately 212^{o}C and 261^{o}C in SA crystal and slightly lowered temperature LISA crystal probably at 205^{o}C and 257^{o}C respectively were related to loss of water molecules and solvent elements. Similarly the sharp endothermic peak at 448^{o}C for SA crystal and 447^{o}C for LISA crystal confirmed the decomposition temperature of the crystals and was consistent with TGA results. Hence the thermal stability of these crystals were good and useful for high temperature applications below 250^{o}C.

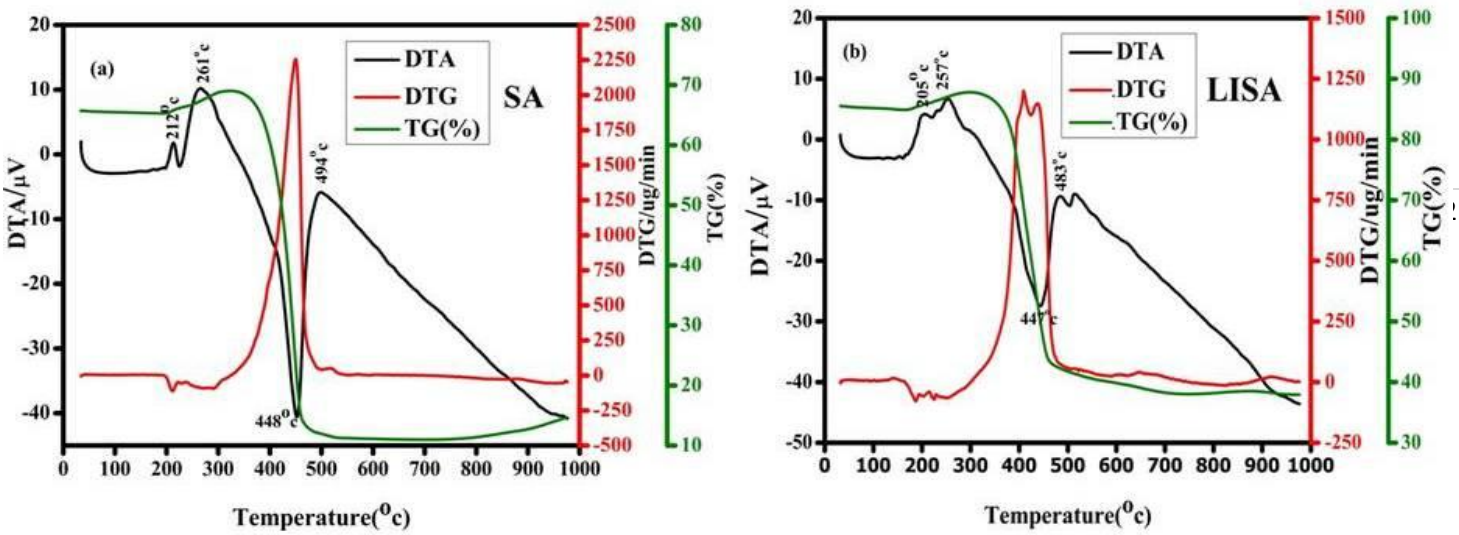

Fig. 2 TGA-DTA curves of (a) SA and (b) LISA crystal

3.3 Antibacterial activity

The antibacterial activity of SA and LISA crystal was studied against gram positive bacteria(Bacillus, staphylococcus aureus) and gram negative bacteria(Escherichia coli,klbsiela,serattia) using Disk Diffusion Kirby – Bauer method. Mueller- The zone of inhibition of E.coli have 22mm of half lifes for known standard antibiotic, whereas in LISA it was increased. Similarly in klebsiella, bacillus, staphylococcus aures and serattia, the absorbed zone of inhibition was increased while comparing with standard antibiotic zone of inhibition. Table .1 shows the antibacterial activity of SA and LISA crystal. From this we decided that the grown crystals are more effective than standard antibiotic. Zone of inhibition higher for gram negative bacteria Escherichia coli.

Table 1. Antibacterial effect of LISA crystal.

	SA	**LISA**	**Standard antibiotic AMIKACIN**
Bacteria name	**Zone of inhibition[mm]**	**Zone of inhibition[mm]**	**Zone of inhibition[mm]**
E.COLI	28	36	22
KLEBSIELLA	30	34	19
BACILLUS	29	34	20
STAPHYLOCOCCUS AUREUS	26	34	22
SERATTIA	33	31	19

3.4 Z-scan measurements:

The third-order NLO properties of LISA crystal was investigated by the Z-scan technique. Z-scan is a very acceptable technology to examine the third order nonlinear refractive index (n_2) and nonlinear absorption coefficient (β). The graphs observed for the open aperture and closed aperture method of LISAand SA crystal is shown in Fig. 3(a), 3(b) and 4(a), 4(b). Here, an open aperture Z-scan method was carried out to calculate the nonlinear absorption coefficient (β),from the assembled data, we examined the maximum absorption at focus which obtained in a shape of the valley that showed the reverse saturable absorption (RSA) along with positive absorption coefficient (β) showed two-photon absorption (TPA), occurred in the grown crystal of LISA. This is a very important characteristic for obtaining any optical limiting applications. The calculated values of third order nonlinear refractive index (n_2) were 2.72 x 10^{-9} cm^2/w. The nonlinear absorption coefficient (β) is 2.98 x 10^{-4} cm/w. The positive value of nonlinear refraction revealed the self-focusing nature. The third order susceptibility ($\chi^{(3)}$) of LISA crystal was 3.47 x 10^{-6}esu.

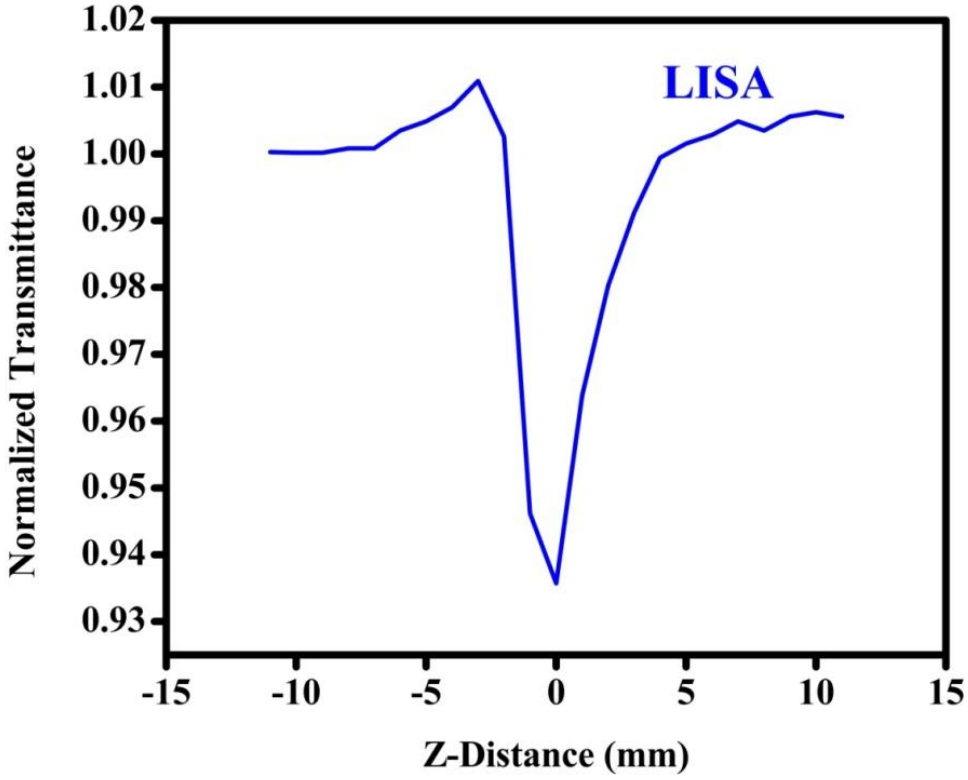

Fig.3a. Z-scan open aperture of LISA crystal

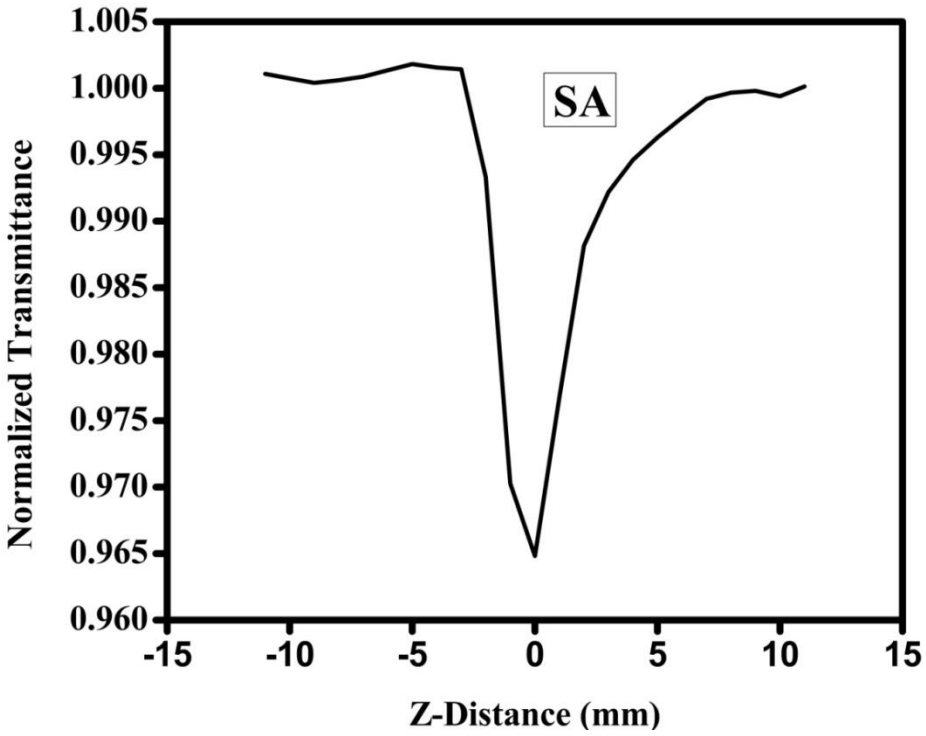

Fig.3b. Z-scan open aperture of SA crystal

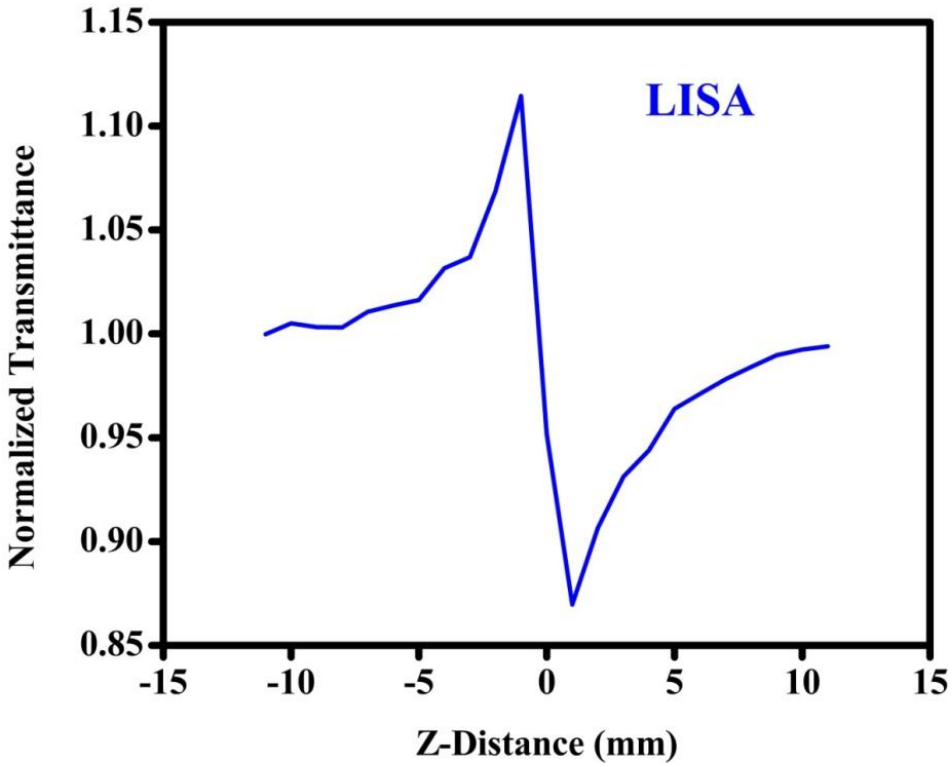

Fig.4a Z-scan closed aperture of LISA crystal

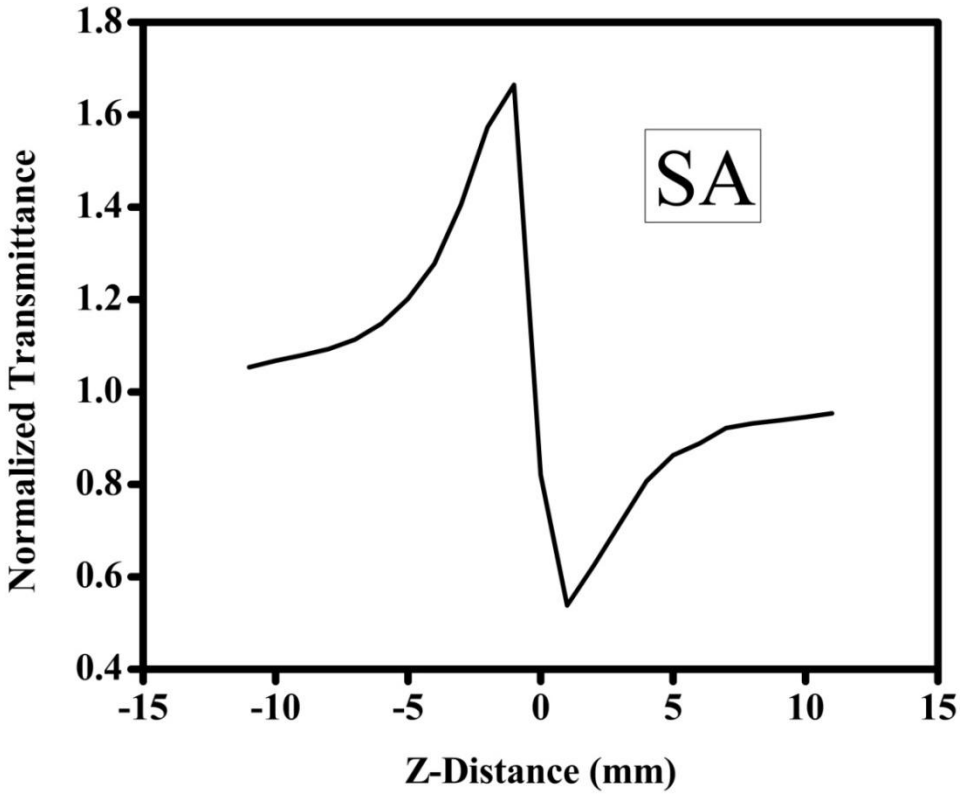

Fig. 4b Z-scan closed aperture of SA crystal

4. Conclusion

Pure sulphamic acid and L-isolucine added single crystals were synthesized at room temperature using slow evaporation technique. X-ray analysis affirmed the presence of the characteristic pattern related to orthorhombic crystal system. The thermal analysis exhibit the material is useful for high temperature applications below 250 ^{o}C.The strong intense blue emission finds application in optoelectronic devices.The antibacterial activity showed that upon addition of isolucine in pure SA the antibacterial activity was enhanced, which will be helpful for killing pathogenic bacteria's. The third order nonlinear refractive index, absorption coefficient and optical susceptibility were calculated by the Z-scan technique and it revealed that the SA and amino acids added SA crystal possessed self focussing and two-photon absorption process.

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