Available online at www.scholarsresearchlibrary.com

Scholars Research Library

Archives of Physics Research, 2014, 5 (1):79-83
(http://scholarsresearchlibrary.com/archive.html)

Archives of Physics Research
Scholars Research Library
ISSN : 0976-0970
CODEN (USA): APRRC7

Growth and characterization of yttrium doped sulphamic acid single crystal

B. Kannan[1], P. R. Seshadri[1*], P. Murugakoothan[2] and K. Ilangovan[3]

[1]PG and Research Department of Physics, A. M. Jain College, Meenambakkam, Chennai
[2]MRDL, PG and Research Department of Physics, Pachaiyappa's College, Chennai
[3]PG and Research Department of Physics, R K M Vivekananda College, Mylapore, Chennai

---

ABSTRACT

Single crystals of Yttrium doped sulphamic acid (Y : SA) were grown from aqueous solutions by slow evaporation technique. Structural characterization of the grown crystals has been carried out by powder and single crystal X-ray diffraction (XRD) methods. (Y : SA) crystals crystallize in tetragonal structure. Second harmonic generation (SHG) for the material of this work was confirmed using Nd : YAG laser. The UV-Visible spectrum showed that the grown crystals have wide optical transparency in the entire visible region. The thermal stability of the crystal has been indentified by thermal studies.

Key words: Growth from solution, X-ray diffraction, FTIR, Optical studies, Powder X-ray diffraction, EDAX

INTRODUCTION

The search for materials for various device applications has led to discovery of many organic, inorganic and semi-organic crystals. The responsibility for the exquisiteness of the crystal is due to their structural simplicity, symmetry and purity. These characteristics endow crystals with unique physical and chemical properties which caused major transformation in the electronics industry. Nonlinear Optical (NLO) materials, have potential applications in optoelectronics, Second Harmonic Generation (SHG), optical storage, optical communication, photonics, electro-optic modulation, optical parametric amplifiers, optical image processing, etc [1-4]. Suphamic acid and its derivatives have wide industrial applications. It has been noted that, when doped the rare earth elements are capable of improving and inducing some important characteristics to the pure sample. Hence in the present work, systematic studies on the growth and characterization of Yttrium doped sulphamic acid (Y : SA) is reported. The crystalline perfection, structural, presence of funtional groups, optical, thermal, behaviours and the presence of the dopant in (Y : SA) were revealed by X-ray diffraction, FTIR, UV-vis-NIR, TG/DTA, EDAX, studies respectively.

MATERIALS AND METHODS

Preparation of the solution to grow the desired crystal is an important stage in solution growth. the saturated aqueous solution of (Y : SA) has been prepared using the pure and dry salts of SA and yttrium in the ratio 1 : 0.1. The saturated solution is filtered using the filter paper. The filtered solution is transferred into the growth beaker and the growth vessel is sealed using a perforated filter paper in order to slow down the evaporation of the solvent. The growth vessel should be carefully kept in a pollution and disturbance free atmosphere [5]. The seed crystals were obtained in a period of 10 days. The Fig.1(a) and 1(b) show the photographs of pure SA and (Y : SA) crystals respectively.

Fig.1(a) Pure Sulphamic acid crystals

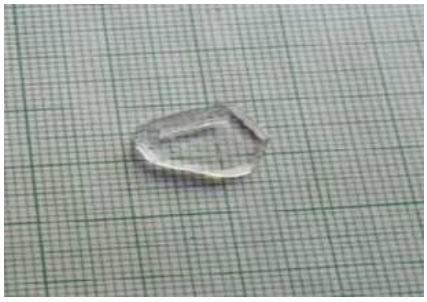

Fig. 1(b) Y : SA single crystal

RESULTS AND DISCUSSION

3.1. EDAX

EDAX is an analytical technique used for the elemental analysis in the given sample [6]. The presence of various elements in Y : SA single crystal has been identified from the EDAX spectrum as shown in Fig.2. The weight percentages of Nitrogen, Oxygen, Sulphur and Yttrium are tabulated and given in table 1. This confirms the presence of the dopant yttrium inside the crystal lattice of the sulphamic acid single crystal.

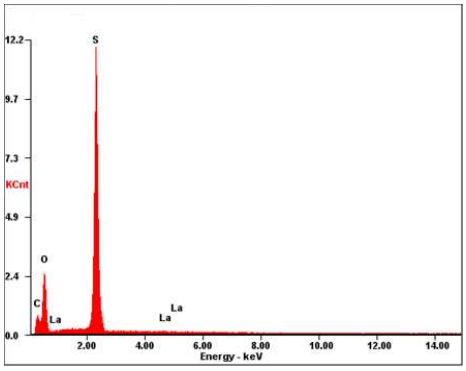

Fig. 2. EDAX spectrum of Y : SA

Table 1: Elemental composition of Y : SA crystal

Element	Wt.%	At.%
N	11.27	17.51
O	38.52	51.33
S	46.61	30.99
Y	3.60	00.53

3.2. X-ray diffraction

Single crystal X-ray diffraction

Single-crystal X-ray diffraction is most commonly used for precise determination of a unit cell, including cell dimensions and positions of atoms within the lattice. ENRAF NONIUS FR 590 single crystal X-ray diffractometer has been used to measure the cell parameters of both pure SA and Y : SA single crystals. The observed lattice parameters are given in table 2. While analyzing the data given in the table it can be seen that in doped crystal 'a" and 'b' are equal with different 'c' value indicating that it crystallizes in tetragonal structure whereas the pure SA crystal is orthorhombic, this clearly indicates that the dopant inside the crystal lattice have some effect on the structural parameters.

Table 2. Lattice parameters of pure SA and Y : SA single crystals

Parameters	Pure SA	Y : SA
a Å	8.100	8.080
b Å	8.049	8.080
c Å	9.220	9.550
V $Å^3$	604.8	597

Powder X – ray diffraction

The powder X – ray diffraction patterns of pure and doped SA are shown in Fig. 3(a) and 3(b). The crystallinity of both pure and doped crystals is quite clear from diffractograms because of the occurance of sharp peaks at specific Bragg's angles. The 'd' spacing and (h k l) values for prominent peaks in the spectrum were identified. The extra peaks indexed (413), (206) and (226) obtained in Fig.3(b) in comparison with the Fig.3(a) may be due to the incorporation of the yttrium into the pure SA crystal lattice, from the diffractograms it is clear that the entry of the dopants in the modified composition of SA crystals lead to a change in the intensity of peaks when compared to the peaks of pure SA, which shows that the doping has brought about a change in the internal structure of crystals due to change in bond lengths [7]

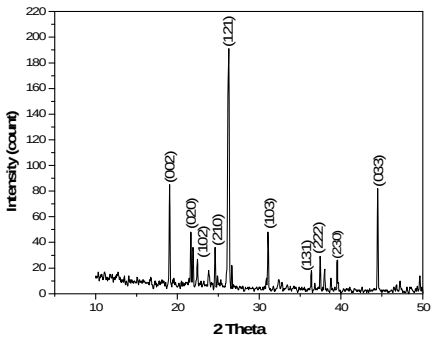

Fig.3(a). Powder X – ray diffractogram of Pure SA

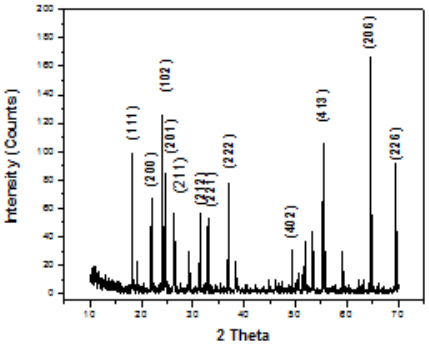

Fig.3(b). Powder X –ray diffractogram of Y : SA

3.3. Fourier Transform Infrared Spectroscopy

The FTIR spectra of Y : SA crystal was recorded with the help of BRUKKER IFS 66v spectrometer by KBr pellet technique in the range 500-4000 cm^{-1} and is shown in Fig 4. The FTIR spectroscopy study is effectively used to identify the functional groups present in the material. The functional group assignments for Y : SA is summarized in Table 3.

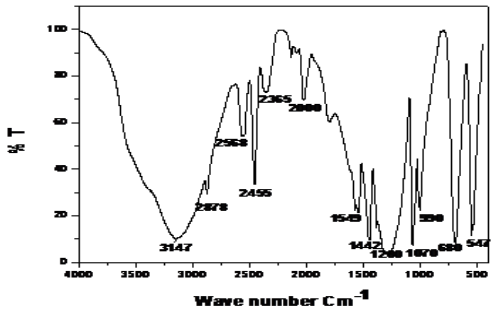

Fig. 4 FTIR spectrum of Y : SA

Table 3 : Vibrational band assignment for Y : SA single crystal

Wavenumber (cm^{-1})	Assignment
3147	Degen. NH_3^+ stretching
2878	Sym. NH_3^+ stretching
1549	Degen. NH_3^+ deformation
1442	Sym. NH_3^+ deformation
1200	Degen. SO_3^- stretching
1078	Degen. SO_3^- deformation
689	NH_2 and N-H Wagging
547	Degen. SO_3 deformation

3.4. UV-vis-NIR Spectroscopy

The absorbtion spectra of Y : SA crystal is measured in the wavelength range 200-800 nm using Philips PV8700 UV-visible scanning spectrometer. The recorded absorption spectrum is shown in Fig 5. It is observed that the, crystals have good transmission in the entire visible and IR region. The lower UV cut of wavelength is at 250 nm.

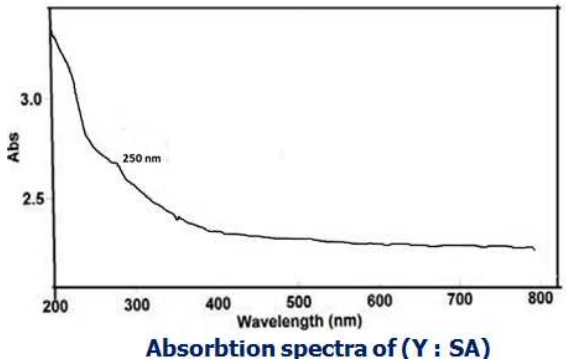

Fig.5. UV-vis-NIR specturm of Y : SA single crystal

3.5. Thermal studies

Thermal analysis for Y : SA was performed using NETZSCH-STA 409 instrument in nitrogen atmosphere, alumina was taken as reference material [8]. The resulting TG-DTA trace is shown in Figure 6. There is no weight loss between 100 and 150^{o}C. This indicates that there is no inclusion of water in the crystal lattice, which was used as the solvent for crystallization. From the TG curve it is observed that the Y : SA exhibit three stage decomposition. The first and second minor decomposition takes place at 160^{o}C and 260^{o}C with weight losses of 2.45% and 5.10% the third major weight loss takes place at 370^{0}C with the weight loss of of 88.83 % of the initial mass.

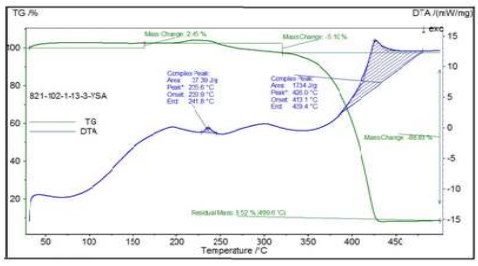

Fig 6. TG - DTA thermograms of Y : SA

These stages of decompositions are well supported by the endothermic peaks at the respective temperatures in the DTA.

3. 6. Nonlinear optical test

The SHG efficiency of Y : SA is measured by using the Kurtz-Perry powder technique. A fundamental wave with a pulse width of 8 ns, repetition frequency of 10 Hz, a beam diameter of 1 mm, energy of the laser pulse around 300 mJ and a wave length of 1064 nm radiated from Nd:YAG laser source was focused on the samples by a lens with focal length of 120 mm [9]. The grown single crystal of Y : SA was powdered with a uniform particle size and densely filled into the quartz cell. A sample of potassium dihydrogen phosphate (KDP), also powdered to the identical size as the experimental sample was used as a reference material in the SHG measurement. The transmitted fundamental wave was absorbed by a $CuSO_4$ solution and the second harmonic signal was detected by a

photomultiplier tube and displayed on a storage oscilloscope. The generation of the second harmonics was confirmed by the green radiation for the doped crystal, it was observed that its NLO efficiency is 0.8 times the standard KDP, but for the pure SA there is no green emmision observed, hence it can be seen that the dopant induces some NLO property to the grown sample.

CONCLUSION

Good quality single crystals of pure SA and Y : SA have been grown from slow evaporation technique. The crystallinity of the grown sample has been confirmed by X ray diffraction analysis. Various functional groups present in the grown crystal have been identified by FTIR spectroscopy. The optical transparency has been revealed by UV-vis-NIR study and thermal stability has been confirmed by thermal analysis. Finally it is observed that the dopant induces the NLO property to the grown crystal.

REFERENCES

[1] R. Ramesh Babu, R. Ramesh, R. Gopalakrishnan, K. Ramamurthi, G. Bhagavannarayana, *Spectro Chemica Acta Part* A **76** (**2010**) 470 – 475
[2] F.A. Kanda, A.J. King, *J. Am. Chem. Soc*. **73** (**1951**) 2315.
[3] R.L. Sass, *Acta Cryst*. **13** (**1960**) 320–324.
[4] D. Balasubramanian, R. Sharkar, V. Siva Sankar, P. Murugakoothan, P. Arulmozhichelvan and R. Jayavel (**2008**). *Materials Chemistry and Physics*. 107(1): **57**.
[5] A. Sundara Raj, P. Muthusubramanian, *J. Mol. Struc. Theochem*. **89** (**1982**) 291–296.
[6] S.J. Hickling, R.G. Woolley, *Chem. Phys. Lett*. **166** (**1990**) 43–48.
[7] R. Valluvan, K. Selvaraju, S. Kumararaman, *Mater. Chem. Phys*. **97** (**2006**) 81–84.
[8] T.Arumanayagam, S.Ananth, P.Murugakoothan *Spectrochim.Acta Part* A **97** (**2012**) 741 -745.
[9] P.Vivek, P.Murugakoothan *Advanced Materials Research* **584** (**2012**) 51 - 55.