ISSN: 2278 – 1323
International Journal of Advanced Research in Computer Engineering & Technology (IJARCET)
Volume 1, Issue 6, August 2012

UNIDIRECTIONAL SEEDED SINGLE CRYSTAL GROWTH FROM SOLUTION OF SULPHAMIC ACID AND ITS CHARACTERIZATION

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ABSTRACT

Unidirectional seeded single crystal growth by SR Method. The growth crystal has a cylindrical morphology with good optical quality by this method. The entire solute can be converted in crystal so that, 100 % solute crystal efficiency can be achieved. The huge sulphamic acid crystal grown from a seed crystal in a super saturated aqueous solution. In the instrument a vertically designed "L" bend was used to avoid the spurious nucleation. Growth from solution is often the only alternative if the substance decomposes below its melting point or undergoes a phase change between its melting point and temperature at which crystal is to be used. The growth rate at which perfect crystal can be grown from solution are appreciably smaller than in process of growth from melt. The liner rates of growth from solution are commonly 10mm per day compare to rates of 10mm per minute from the melt. In SR method orientation seed was fixed at the bottom of the glass and ampoule. And crystal of diameter 40 mm & length 60mm successfully grown by SR method.

Key word :– Growth from solution, XRD, FTIR, Single Crystal Growth, Inorganic compound

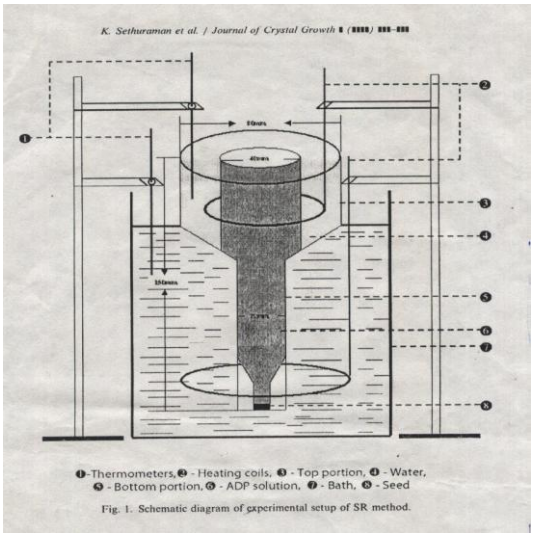
1. INTRODUCTION

Now day nonlinear optical materials are widely used in many different areas of solid state physics. In the recent past organic non linear optics(NLO) material are gaining attention because of their good nonlinear properties over inorganic counter parts. The organic nonlinear optical (NLO) crystal plays an important role in second harmonic generation (SHG), frequency mixing, Electro optic modulation and optical parametric oscillation. Crystallization from the solution is an important process and is a two step process nucleation and crystal growth. The driving force for crystallization is the degree of super saturation which has

been commonly expressed as the difference in concentration between the super saturated and saturated solution. Sulphamic acid is a classical inorganic compound. It is an important and industrial chemical with an annual manufacturer of several kilotons. It is moderately soluble in water. It is stable at room temperature and normal atmosphere. The unidirectional seeded single crystal is a promising NLO material. A novel growth and S.R. Method introduce for growth of single crystal from aqueous solution. This technique already applied to grow some organic and inorganic crystal.Sankarnarayan method is better than conventionally growth crystal. The goal is to be combining the potential high polarizability of organic molecules such as aromatic cycle, with thermal, mechanical and chemical stability of mineral compound. This method is important and extremely flexible for the development of new material applicable over a very board range of frequency. The advantage is of semi organic is that, the crystal can grow from aqueous solution and they form three dimensional crystals which can be easily cut and polished. Good quality and large size nonlinear optical crystal are need for laser application. For practical application, we need good optical transparency and also crystal should with stand high optical power and should have chemical stability. The grown crystal was characterized by X – Ray powder diffraction analysis, FTIR study etc.

2. EXPERIMENTAL SETUP

The schematic diagram of S.R. method apparatus is shown in fig.1. It consists of growth ampoule made out of glass with seed mounting pad. The diameter of the growth portion and top portion of the ampoule was 20mm & 80mm respectively. A ring heater kept at the top of the growth ampoule & bottom of the growth ampoule was connected to the temperature. The temperature around growth ampoule kept constant with the help of temperature controller till the process is completed. The grown crystal fig.2.



❶-Thermometers, ❷ - Heating coils, ❸ - Top portion, ❹ - Water, ❺ - Bottom portion, ❻ - ADP solution, ❼ - Bath, ❽ - Seed

Fig. 1. Schematic diagram of experimental setup of SR method.

Fig. 1 Schematic diagram of S.R. Method Apparatus.

Fig. 2 Single crystal of S.A.

The ring heater was moved downwards using a translation mechanism designed "L" bend, the growth condition of this method depend on the heating point.

The solubility experiment was carried out in constant temperature bath for the temperature 30° C, 35° C, 45°C & 55° C.

The growth experiment was performed using demonized water and mixed solvent with the help of solubility curve by the slow evaporation technique. The temperature of solution was increase slowly. The color of the solution becomes slightly yellowish after a few days at temperature above 60° C so that temperature of solution was kept always below 60° C.

Characterization: a) FTIR Analysis

The functional group was identified by FTIR using JASCO model 610 FTIR spectrometer in the range 6000 to 2000 cm-1 as shown in fig.3. The presence of band between 4000-1 to 3800 cm-1 are mainly due to N-H stretching. The band observe at 2400 cm-1 arises symmetric vibration of NH3+ group and the band at 4000 cm-1 is an a symmetric stretching. The strong band 5600 cm-1 correspond to SO3 stretching vibration. This peak was also observed at FTIR spectrum. It always shows that, reflectance range of active band 3000 to 500 cm-1. The band observed with the wave number of 1400 cm-1 in reflectance is goes unsymmetrical stretching vibration. From the FTIR analysis of the compound of sulphamic acid was identified. The band observed 1200 to 1300 cm-1 & strong band also found 1350 & 1450 cm-1 as shown in fig.4.

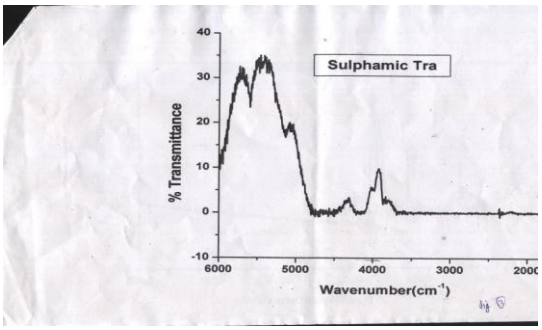


Fig . 3 FTIR spectrum of S. A.

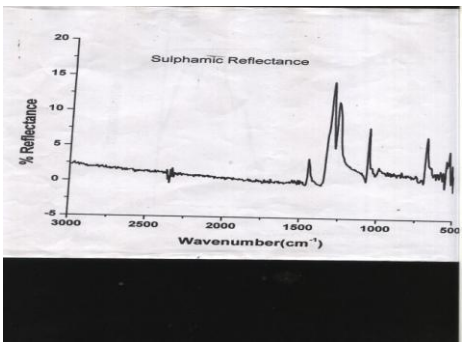


Fig. 4 FTIR spectrum of sulphamic acid

b) X-Ray powder Diffraction Analysis

The peak observed from fig.5, X – Ray diffraction spectrum were analyzed & indexed using software package and the data obtained by the powder XRD analysis are in accordance with the single seeded crystal XRD data ,shown in the data spectrum table. The single crystal XRD reveals that the sulphamic acid crystal belongs to orthorhombic structure.

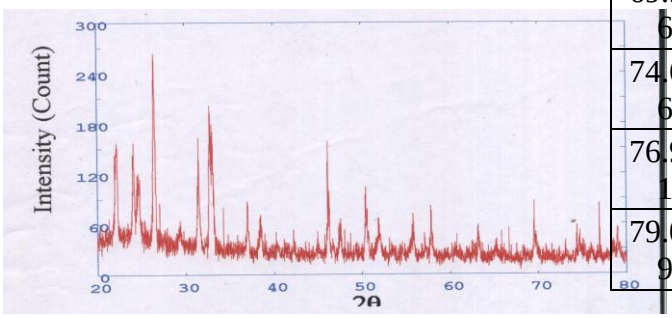


Fig. 5 Powder X-Ray Diffraction pattern of sulphamic acid

2Theta	d (A)	Height	Area	FWHM
22.194	4.00208	105.8	2008.6	0.3800
24.110	3.68824	104.3	1341.5	0.2600
24.633	3.61118	74.5	1234.9	0.3600
26.491	3.36189	209.0	2851.0	0.2800
29.379	3.03769	23.7	426.7	0.3600
31.457	2.84159	100.8	1308.1	0.2600
32.760	2.73146	155.5	1659.4	0.2200
36.997	2.42785	54.8	685.0	0.2600
38.45	2.339	44.9	655.4	0.3000
0	38			
46.148	1.96545	132.5	1017.0	0.2400
47.52	1.911	32.7	586.9	0.3800
50.416	1.80863	64.4	899.7	0.2800
51.915	1.75985	34.1	544.7	0.3200
55.803	1.64609	31.8	492.2	0.3600
57.845	1.59275	45.3	569.2	0.2600
63.146	1.47120	26.3	439.2	0.3400
69.536	1.35080	56.7	566.0	0.2200
74.666	1.27019	27.2	271.5	0.2000
76.941	1.23820	25.7	282.0	0.2200
79.019	1.21077	34.2	341.7	0.2000

XRD spectrum Table

RESULT AT A GLANCE:

Sulphamic acid seeded single crystal was successfully grown by slow evaporation solution grown technique. The solubility of sulphamic acid in demonized water at saturated solution the FTIR and X–Ray diffraction studies confirmed the vibration frequency of sulphamic acid. The crystal also found to be NLO material having good quality in transparent medium.

ACKNOWLEDGEMENT

I am extremely grateful to Dr. A.D. Yadav Head of the Department of

Physics, University of Mumbai, I am Indebted to Dr. Rewatkar (Lecturer in Physics) Dr. Ambedkar M. Dikshabhumi Nagpur, Swati & Nandu Meshram for experimental setup, as well as for data information. I am also thankful to Mr.Chetan Sir, Dept. of Physics, University of Mumbai, Mumbai.

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