Growth of Resorcinol Unidirectional Crystal from Solution at Low Temperature

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ABSTRACT — ***The non linear single crystals of resorcinol were grown by slow evaporation solution growth technique. The grown crystal was confirmed by powder X- ray diffraction. The functional group of grown crystal was found by FTIR studies and UV –VIS transmittance studies were performed to know the thermal and optical behavior respectively. In this matter the entire solution can be converted into crystal so that 100% solute crystal efficiency can be achieved. The structure measurement that performed to check the structure, quality and homogeneity of the grown crystal. Resorcinol is an important agent for dyes and chemical industries. In a wide range of application including rubber production.***

Keywords — Growth from solution, Non linear optical material, XRD, FTIR, UV, Viscosity, Surface Tension, Dielectric constant, organic materials.

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1. INTRODUCTION

Now days nonlinear optical (NLO) materials are widely used in many different areas of Solid State Physics. In the present organic materials are getting attention because of they have good nonlinear optical (NLO) properties over organic counterparts. Crystal grown used many method to grow single crystal mainly, Solution growth, Melt growth, Vapour Growth. Each method is having there own limitations for example conversion, SEST growth yields small size single crystal with different crystallographic faces [1]. Nonlinear optical single crystals are used in area of Fiber Optics Communication, Optical Conversion, Optical data storage etc. In the past two decades organic NLO single crystals are gaining attention due to large NLO coefficient and the possibility of structural engineering for taiel made NLO applications due to availability of reactive bond in organic crystals comparative to inorganic counterparts [2], because of the corrosive action of the chemical used in dye industry, carefully consideration must be given to the construction material employed. The material which can be used for a given purpose should be specified if possible, but frequently experience, or chemical scene, so to speak must be relied up on [3]. The determination of crystal system in a solid in carried out after X-ray and neutron diffraction studies. This comes strictly in the area of materials. Few more distinctive features between planes and direction can be noted. For example planes and there negatives are identical, while this is opposite in case of direction [4]. In the case of inorganic materials, significant efforts have been made to grow large size crystals due to their potential applications in non linear optical applications. Here a more practical S. R. method is used. All the reported crystal growth methods at elevated temperature leads to thermal-induced grown in defects in addition to the complicated instruments, difficulty in large size crystal and multi-step process involved, it offer a solution growth method at room temperature involving less sophisticated equipment to grow in directional single crystal with cylindrical morphology, 100% solute crystal conversion efficiency and ease in scaling up of crystal diameter. The solution growth method is the basic method to be tried because it is very simple and there are no problems with thermal decomposing [5].

2. EXPERIMENTAL STUDIES

A suitable seed crystal was selected from the harvest crystals. Two method of crystallization were attempted to grow bulk crystals. In the first method, a saturated solution was prepared and the seed crystal was hung in side the solution that was optimally closed for controlled evaporation. The size of the crystal depends on the amount of the material available in the

solution, which in turn is decided by the solubility of the material in that solvent. A good quality crystal was obtained in a period of four weeks.

Fig. 1 Grown Crystal for Resorcinol

From fig. (1), it is clear that the portions of the crystal are of good quality which is not perfectly transparent and has no defects [6]. The crystal with specific orientation can be grown from solution by Sankarnarayanan – Ramasamy (SR) method. By this method the entire solute can be converted in to crystal so that 100% solute crystal efficiency can be achieved [7]. Analytical grade chemical such as resorcinol organic material consist of 110.2 molecular weight and molecular formula of this resorcinol $C_6H_4O_2$, which are used as a received without further purification. It is purely soluble in water at a particular phase from 5 degree Celsius to ten degree Celsius temperature and kept over ten to fifteen days to avoid the dust particles for preparing seed crystal by using S. R. method. We have separated transition and from without mixture using resorcinol fresh materials. In this paper optical and spectral properties are reported along its thermal and morphological studies.

3 RESULT AND DISCUSSION

3.1 X-ray diffraction study:

In the arrange structure the organic material resorcinol molecule no effect of modulation in the structure the modulated order is prefect and the molecules remain partially disorder. The structure of the low temperature phase could not be determined owing the poor quality of the crystal affected by reconstructive phase transition. In this case structure belongs to orthorhombic crystal.

The X – ray diffraction pattern is shown in figure sharp peak is observed in the diffractogram of resorcinol crystal. The XRD exhibits regular pattern, which is a characteristics of the amorphous compound also exhibited nearly similar. XRD pattern of resorcinol doped with sodium chloride crystal.

Fig. 2 XRD for Resorcinol Crystal

There are lightly change in resorcinol and its doped crystal in both samples as shown in fig. (2).The powder X-ray diffraction pattern was recorded for resorcinol using a rich seifert diffractometer over the angle range 4.0 to 60 degree at a scan rate 5.0 d/m .The strong peak of resorcinol at 9,14 and 15 with high intensity 3665 as shown in figure (2). The lattice parameter are a = 4.3530, b = 3.414 and c = 3.3814 $\alpha = \beta = \gamma = 90$ degree.

3.2 FTIR Study:

The FTIR spectra of the crystal were recorded in the resorcinol crystal in the frequency range of 450 to 4000 cm^{-1}. Using JASCO spectrometer FTIR model 410 at a resolution of 90 and with a scanning speed of 2 mm/s. the recorded FTIR spectra were compared with the slandered spectra of the functional groups. The strong but board peak at a 3256.40 cm^{-1} is due to the presence of C – H stretching in the hydroxyl group. The very strong peak observed that 1490.38 cm^{-1} indicates the presence of C=H groups as shown in fig. (3).

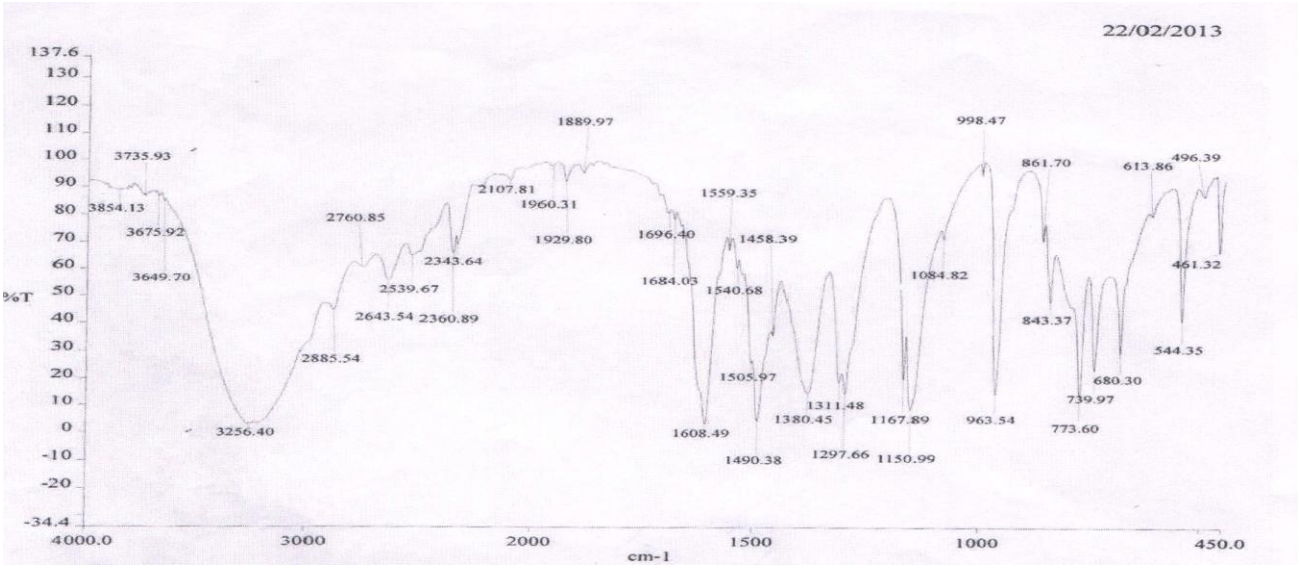

Fig.3 FTIR for Resorcinol Crystal

R1.pk

R1.sp 1776 4000.00 450.00 3.16 100.00 4.00 %T 16 1.00

REF 4000 92.09 2000 98.56 600

3854.13	88.56	3735.93	86.54	3675.92	86.54	3649.70	84.49	3256.40	3.16
2885.54	44.63	2760.85	60.67	2643.54	55.96	2539.67	65.19	2360.89	65.89
2343.64	69.39	2107.81	92.71	1960.31	98.07	1929.80	92.22	1889.97	95.39
1696.40	81.09	1684.03	78.59	1608.49	3.30	1559.35	67.35	1540.68	60.69
1505.97	25.73	1490.38	4.40	1458.39	35.76	1380.45	14.85	1311.48	18.76
1297.66	14.54	1167.89	20.17	1150.99	8.73	1084.82	71.94	998.47	94.87
963.54	14.36	861.70	70.84	843.37	47.95	773.60	13.61	739.97	23.53
680.30	29.55	613.86	80.33	544.35	41.22	496.39	87.29	461.32	67.04

Table. Data for FTIR for Resorcinol Crystal

3.3 UV Study:

The UV – VIS transmission spectrum of the crystal was recorded in the wavelength range of 190-1100 nm and in shown in fig. 4. It is seen that the UV transparency cut of occur in 862.0 nm and there is perfect transparency as compared to the doped crystal and remarkable no absorption in the entire region of the spectra it is an important requirement for non linear optical material for possible application. The sample recorded at room temperature in the

frequency range of materials. In this material constraint growth in the S. R. method experiment does not affects the crystalline perfection.

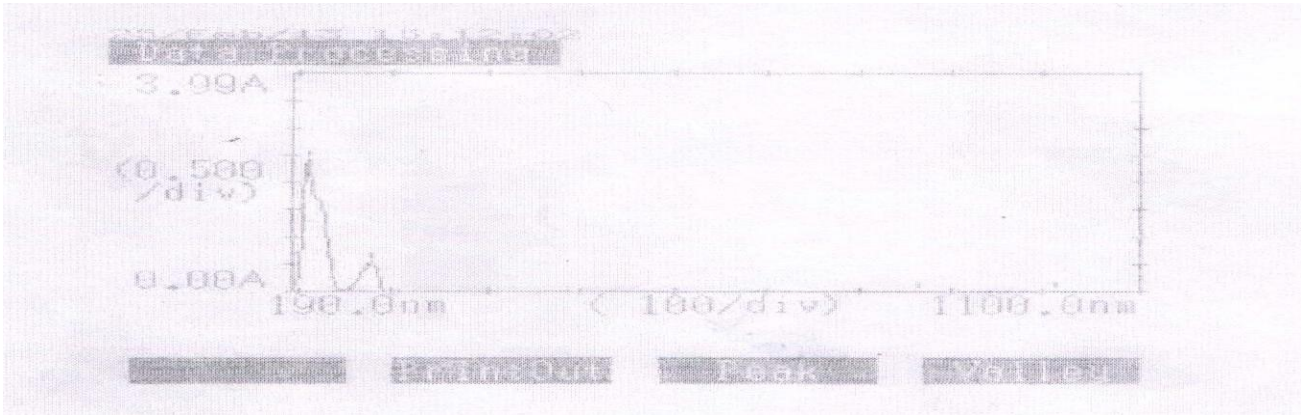

Fig. 4. UV for Resorcinol Crystal

3.4 Dielectric constant:

It uses a parallel plate capacitor carrying affixed charge and not connected to the battery to provide a uniform external electric field in to which we place a dielectric slab. The over all effect of aliment and induction is to separate the centre of positive charge of the entire slab slightly from centre of negative charge. The slab as a whole, although remaining electrically neutral, becomes polarized. The net effect is a pile-up of positive charge on the right face of the slab and of negative charge on left face. Within the slab no excess charge appear in any given volume element. In this resorcinol crystal the dielectric constant found that 3.22 this is more quantity as compared to the doped sample.

3.5 Surface tension:

It is the force in dynes acting at aright angles to the surface of the liquid along one centimeter length of the surface. It is generally represented by the symbol gamma and is expressed in dynes cm-1. The existence of strong intermolecular force of attraction in liquid gives rise to another important property. According to kinetic theory, molecular kinetic energy is proportional to absolute temperature. The rise in temperature in the liquid is therefore accompanied by increase in energy of its molecule. In this case, the intermolecular force decreases with increase in the energy of molecules, the intermolecular force of attraction decrease with rise in temperature. Hence the surface tension of these materials decreases with rise in temperature. At critical temperature, since the surface of separation between the liquid and its vapor disappear, the surface tension fall to zero. In case of resorcinol the surface tension is 20.23 dynes/cm.

3.6 Viscosity:

The viscosity of nearly developed slow and fast curing of resorcinol adhesive increased very rapidly. The flow is the characteristics properties of viscosity. The flow of liquid molecules can be analyzed in term of molecular laminar layer arranged one over another. In this sample we used Ostwald's viscometer method which is based on Poiseuille' law. This connects the flow of liquid through capillary tube with the coefficient of viscosity of the liquid. The viscosity of this resorcinol crystal is very low 56.012 P. at 29 ^{O}C.

4. CONCLUSION

The transparent organic crystal of resorcinol were grown by S R method at room temperature. The characteristics by single crystal x-ray diffraction get orthorhombic structure. The FTIR studies were carried out shows the functional group. This material is fully hazards type therefore avoids skin and eye contact.

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