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Characterization of Sodium Chloride Doped with Resorcinol

Rita A.Gharde*, Divakar T. Chunarkar[1] and Santosh A. Mani[2]

[1,2]Department of Physics, University of Mumbai, Santacruz (E), Mumbai 400 098, INDIA.
[3] K.J.Somaiya College of Engineering, Vidyavihar (E) Mumbai 400 077, INDIA.
E-mail: gharde.rita@gmail.com

Abstract

Sodium chloride also known as salt, common salt and table salt. It is an ionic compound with the formula NaCl. The single crystal of pure resorcinol and sodium chloride doped were grown by low temperature solution growth method using distilled water as a solvent in the crystal growth. The lattice constant of doped crystal were found. In this crystal sodium chloride is used in very less quantity as compared to the resorcinol 10:2 ratio. The samples were characterized by X- Ray Diffraction (XRD), Fourier transform Infrared (FTIR) Spectroscopy, Ultraviolet – Visible (UV) Spectroscopy, dielectric study. Crystalline and non linear optical studies carried out on the growth sample justifies that the crystal, quantity is as good as the quantity of crystal grown by S.R.Method. In the present study ”uniaxially solution – crystalline method” unidirectional growth at room temperature and also 100% solute crystal conversion efficiency have been achieved for the first time from the solution growth method.

Keywords: Crystal growth, Solubility, XRD, FTIR, UV etc.

1. Introduction

Materials with non linear properties have wide application in modern optical and optoelectronics devices. Crystal possess high efficiency of frequency conversion, moderately high damage threshold wide range of optical transparency[1].The standard name of resorcinol is benzene,1-3 diol, but it is more commonly referred to as resorcinol. It is obtained by fuming resins, hydrocarbon secretion of plants and with

potassium hydroxide. The hazards nature of resorcinol extends to the environment, it is consider dangerous to the environment. It is primilarly dangerous to aquatic organism, as it is dissolve easily and may infiltrate their system with relative ease [2]. Resorcinol is organic compounds having 110.2 molecular weight and C_6H_4 $(OH)_2$ molecular formula. It is white and lightly transparent as a larger crystal. In this doping, inorganic and organic crystal nonlinear optical materials have mechanical, thermal and spectral properties are studies [1]. The choice of the solvent is of great importance. It proved to be easier to grow large perfect single crystals if the solute has a solubility exceeding about 3 – 5 mole percent. Low viscosity is also desirable to facilitate transport of the solute by diffusion and stirring. In this case both sample are doped easily with the volality of the solvent can be controlled over a wide range, this also increase its versatility [3].

In recent years, several studies dealing with inorganic, organic and semi organic molecules and materials for non linear optics are being reported. In this paper, the spectroscopic and thermal studies of the inorganic non linear optical materials resorcinol doped with sodium chloride seeded single crystal are presented by using S.R. method [4].

2. Experimental

The experimental setup is shown in Figure (1), basically same as that describe by S.R. method. It was employed in house by a controllable cooling assembly transparent growth vessel made out by borosilicate glass. An outer glass shield tube protect and the holed the inner growth ampoule. It is well known that the evaporation rate of water in to the atmosphere as a function of temperature, humidity and air velocity. The evaporation process in the atmosphere is the diffusion of the water molecules, coming out of surface through the air layer covering the water surface. Seed crystals were prepared by making a saturated solution of resorcinol at 31^oC. The solution was filtered in to beaker and kept over 2 – 3 days at room temperature. In this method the following main points have to be considered; seed mounting, seed selection and crucible selection, heater temperature, bath temperature and feeding of the solution. figure (1) shows the growth of resorcinol doped with the sodium chloride single crystal. The main part is the seed mounting which is in the bottom of the growth vessel. The top of the growth vessel is divided in to inner container arrangement with 50 mm and 40 mm diameter and other container arrangement with 8 mm diameter and 50 mm length. The entire bottom set up was made to rest in a water bath.

The temperature of top and bottom part can be changed separately by using different heater attachment. Every crystal growth technique has its own advantage and limitations. The experimental conditions were closely monitored and found that a seed crystal start growing after 10 days. In a time span of 40 – 45 days a good quality single crystal of doped sample has been harvested from the glass tube. The grown crystal was separated by cutting the wall of the ampoule carefully. Disc shaped specimens required for various measurement cut by the crystal cutter perpendicular to the crystal boule. Care was taken to minimize mechanical stress damage, which affects the crystalline perfection and also influence other physical properties of the specimen.

Figure (1): Doped crystal of resorcinol with sodium chloride.

3. Results and Discussion

3.1 Single crystal X-ray diffraction analysis

In order to determine the lattice parameter of the grown single crystal of resorcinol doped with Sodium Chloride, the sample was subjected to crystal X-ray diffractometer. From this measurement we found that, the grown single crystal belongs to orthorhombic structure.

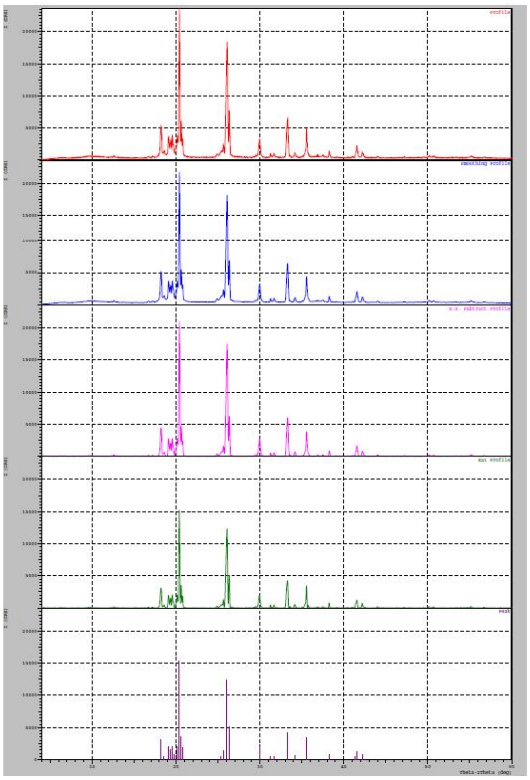

Figure (2): XRD of doped crystal.

It is nearly same crystal structure of fresh resorcinol. It is electrolyzed product of sodium chloride solution, examined for the resorcinol hydroquinone and catchpoll

were identified by in chlorination process system having noncentrosymetric nature with space group.

Table (1): XRD data of doped crystal.

*** Basic Data Process ***

Group : Standard
Data : IR

Strongest 3 peaks

no.	peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	13	35.4893	2.52744	100	0.15910	7989	65692
2	4	19.1721	4.62563	49	0.12650	3942	28027
3	9	29.7090	3.00470	42	0.20070	3369	38109

Peak Data List

peak no.	2Theta (deg)	d (A)	I/I1	FWHM (deg)	Intensity (Counts)	Integrated Int (Counts)
1	18.1000	4.89713	4	0.16740	328	3656
2	18.2400	4.85985	4	0.17840	325	2593
3	18.9600	4.67689	4	0.06900	317	1617
4	19.1721	4.62563	49	0.12650	3942	28027
5	19.8400	4.47139	5	0.15780	437	4109
6	20.1150	4.41088	10	0.24820	762	9298
7	20.4428	4.34088	10	0.21020	788	8756
8	25.7690	3.45447	3	0.39150	269	8581
9	29.7090	3.00470	42	0.20070	3369	38109
10	29.9600	2.98009	4	0.11060	298	2399
11	31.6915	2.82111	16	0.12640	1273	9879
12	35.2200	2.54614	10	0.19820	826	9691
13	35.4893	2.52744	100	0.15910	7989	65692

The determined cell dimension of lattice parameters are a = 2.52744, b = 4.62563, c = 3.00473 and $\alpha = \beta = \gamma = 90^{o}$.

The maximum growth rate using S.R. method was 2 mm per day. To raise the growth rate of the crystal, the temperature of solution was increased slowly but maintaining the room temperature to 70 degree temperature. The powder XRD pattern of doped crystal as shown in figure (2) . The calculated (hlk) planes satisfy the general reflection condition. XRD of this doping sample is nearly same as compared to fresh resorcinol crystal, but more variation of colour between them. In this XRD, peak numbers 13, 9, 4 are strong peak with intensity of 7989, 3942, 3369.The general reflection condition of space group observed from the structure determination of the crystal. The grown crystal were cut and polished by using indigenously fabricated crystal cutting medium. The single sharp diffraction curve indicates that, the crystal lattice perfection is extremely good. The specimen is nearly perfect single crystal without any internal structure grain boundaries and dislocation or very low density of the dislocation which could not be high resolution. The behaviour of resorcinol single crystal structure and resorcinol doped single crystal nearly same.

3.2 FTIR analysis

The functional groups were identified by FTIR spectroscopy studies using Bruker 66V FTIR spectrometer. In the range on X-axis 450 cm^{-1} to 4000 cm^{-1} as shown in figure (3). The strong band at 3256.99 cm^{-1} corresponds OH stretching vibration. The presence of band between 1500- 2000 cm^{-1} is mainly due to O – H stretching. The band observed at 1490.34cm^{-1} is assigned to the symmetric stretching of mode. The good qualities of inorganic crystal were needed for device fabrication. The single doped crystal responsible for the salinity of ocean of the extracellular. The OH stretching frequency decrease from 3256.99 to 2885.6cm-1.This shows that sodium has coordinate through oxygen atom fluid of many multicellular organisms as the various ingredient edible salt and resorcinol single crystal. It is commonly used as a condiment and food preservative. This crystal some time used as a cheap and safe desiccant because it is appear to have hygroscopic properties. It is generally used in dyes and azo dyes manufacturing industries.

The FTIR analysis of pure and doped crystal shows difference of 3000cm^{-1}.The pure resorcinol gives 3256.40cm^{-1} and doped crystal gives 3256.99$cm^{-1}$of band formation as shown in figure (3).The graph are very sharpness and clear. From this graph band observed shows very little difference from 450-3256.99 cm^{-1}. From this band gap we have to observe that, this types of crystal used in dyes and azo dyes and plant as an operation. It gives the pure material i.e.99.9 % maximum. It is used in UV absorbent, adhesive, resins explosive. The weak medium band can arises to functional group bending of aromatic OH group. It is used in the fluorescence tracer for many applications. The fluorescence of this molecule is very intense and excitation occurs in nanometer.

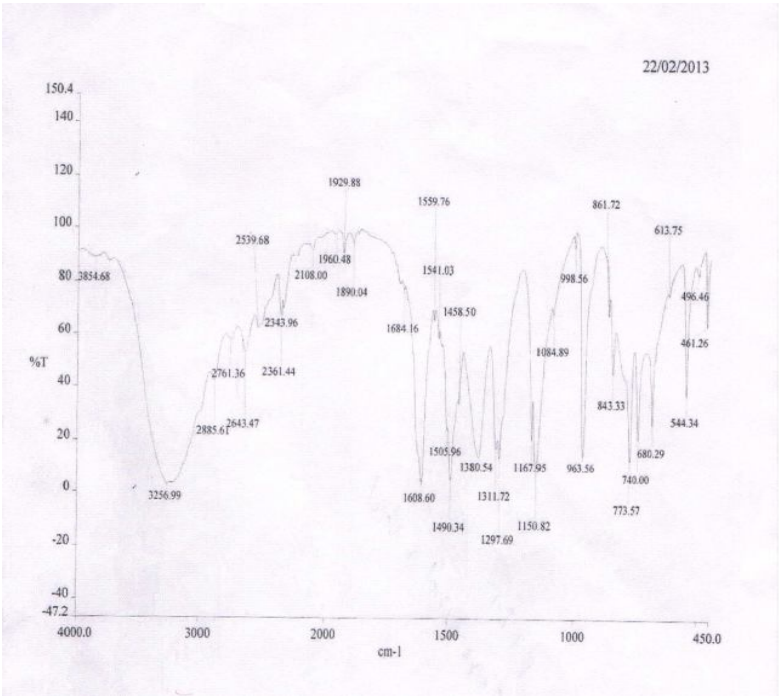

Figure (3): FTIR of doped crystal.

R2.pk

R2.sp 1776 4000.00 450.00 3.16 100.00 4.00 %T 16 1.00

REF 4000 91.46 2000 98.44 600

3854.68	89.01	3256.99	3.16	2885.61	42.14	2761.36	58.07	2643.47	53.38
2539.68	62.68	2361.44	67.50	2343.96	70.26	2108.00	91.92	1960.48	97.79
1929.88	91.16	1890.04	94.91	1684.16	77.28	1608.60	3.53	1559.76	65.59
1541.03	58.96	1505.96	24.46	1490.34	4.92	1458.50	33.87	1380.54	14.02
1311.72	17.48	1297.69	13.48	1167.95	20.81	1150.82	10.03	1084.89	68.55
998.56	93.64	963.56	14.56	861.72	68.18	843.33	45.43	773.57	12.76
740.00	21.09	680.29	26.76	613.75	76.23	544.34	37.80	496.46	84.05
461.26	64.34								

Table (2): FTIR data doped crystal.

3.3 UV Analysis

Single crystals are mainly used in optical application. The optical transmission range and the transparency cut off are important. Therefore UV transmission spectroscopy was carried out using a Shimadzu spectrophotometer. The transmission spectrum of doped crystal is shown in Figure (4). Strong absorption peaks corresponding to the fundamental absorption appear at 190.0 nm and the crystal shows transmittance of 95 %, which is shows preparation of the good quality in doping single crystal. Its aqueous solution is dark violet in form and water soluble dye which turn by compound in crystal.

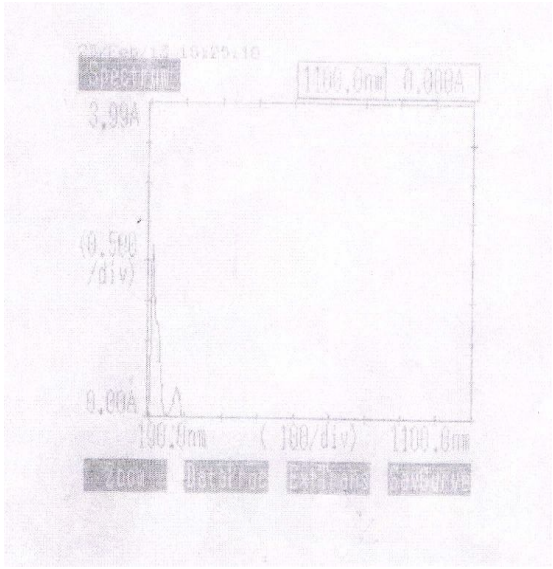

Figure (4): UV of doped crystal.

In this sample tea has a resorcinol doped sodium chloride skeleton structure. The graph plotted between absorption and wavelength is shown in Figure (4), which

illustrate 190.0 nm and 400.0 nm on X – axis and Y – axis 0.0A to 0.5 A. The wavelength of thresholds at which the absorption showing abrupt rise is determined. This can be indicative of the band gap of the material working of this hypothesis and equation utilized the absorbance data as shown in figure (4). It is used in rubber tackier adhesive for wood, veneer and rubber to textile composite medicine. This shows the crystal is good enough and it is an important requirement for non linear optical materials having its application.

3.4 Dielectric Studies

The dielectric constant of the sample essentially involves the accurate measurement of the capacitance of parallel plate condenser with and without the crystal between the plates. The capacitance of capacitor increase when it is filled with an insulating medium. The increases in the capacitance depend on the properties of medium. The typical doping sample dimensions were carried out in the surface are in requirement of the thickness. Dielectric measurements were carried out using simple technique in laboratory by our co – worker and researcher by parallel plate condenser method. The measurements were carried out at room temperature. For pure resorcinol we found 4.0 at a 28^{o} C and in doping sample it is 4.82 at 28^{o} C.

3.5 Viscosity Studies

The viscosity of liquid generally decreases with rise in temperature. The decrease is appreciable, being about 2 % per degree rise of temperature in many cases. This has been explained in term of the "**hole theory"** of the liquid. According to this theory, there are vacancies or "holes" in a liquid. The liquid molecules keep on moving continuously in to these vacancies. As a consequence, the vacancies also keep on moving around as otherwise the liquid will not be able to flow. In this doping crystal when prepared solution the activation energy to move in to a hole. As activation energy becomes increasingly available at increasing temperature, a liquid can flow easily at higher temperature. The coefficient of viscosity thus falls appreciable with rise in temperature. This doped crystal confirmed that brittle in nature, lightly bluish in color and oily. The viscosity of this crystal is very high as compared to the fresh resorcinol crystal i.e. 203 .55gm/ cm.s.

Conclusion

Transparent semi inorganic crystal of resorcinol doped with sodium chloride crystal is brittle and oily materials produced by this non linear optical crystal were successfully grown using submerged seed solution by S. R. method at room temperature. The charecrestics by single crystal XRD 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. Use this material with adequate verification and keep out of reach of children.

Result

The doped crystal of new non linear optical material was successfully grown by S. R. method and characterized by single crystal XRD, UV, FTIR, Dielectric constant and Viscosity studies were carried out. An optical study shows that crystal is lightly bluish and well oils. The fundamental efficiency of the doping crystal was found to be 97 % of purity. Strongest value of the peak is nearly same as compared to previous resorcinol single crystals. S. R. method grown doped sample moderately brittle hardness. Dielectric constant is 4.0 at 28^{O}C i.e. very low as compared to the viscosity of this sample. The viscosity of this sample is 20.355 gm/cm.s and color strongly difference between both sample.

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