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Investigations on Growth and Electrical, Thermal, Mechanical and non-Linear Properties of Vanillin

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Abstract: Single Crystals of organic material vanillin were grown by solution growth technique. XRD analysis reveals that the grown crystal is found to be monoclinic structure with space group $P2_1$. The dielectric studies were carried to establish the dielectric nature of the grown material. From the dielectric studies the solid state parameters were calculated. The thermal and mechanical stabilities were also analyzed. Finally the Second Harmonic Generation (SHG) efficiency was measured to confirm the NLO behavior of the material.
Keywords: Solution growth technique, XRD analysis, dielectric studies, solid state parameters, SHG efficiency.

1. Introduction and Experimental:

Due to the potential applications of nonlinear optical materials in the areas of optoelectronics, tele communication, single processing etc.there has been considerable interest in the field of nonlinear chemical flexibility [1,2]. Vanillin with molecular formula $C_8H_8O_3$ is also one of the organic materials which are capable of exhibitin nonlinear optical behaviour with more SHG efficiency.In the present work, vanillin was first grown using solution growth technique.The grown crystal was then subjected to various characterization studies.

Single crystal of vanillin were grown using isothermal evaporation.The solubility of vanillin in the mixed solvent methanol-chlorofom (4:1 ration) was found to be enhanced. When the mixed solvent was used, the evaporation rate was more than sufficient in order to achieve required supersaturation which yields good quality crystals. Fig.1 shows the as-grown single crystals of vanillin with dimensions of 7.86 x 6.59 x 2.21 mm^3.

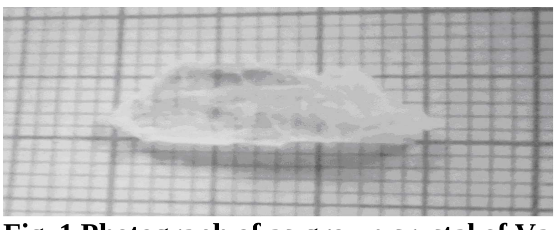

Fig. 1 Photograph of as-grown crystal of Vanillin

1. Results and discussion:

Single crystal XRD study was analysedfor the grown crystal using EnrafNonius CAD 4 singlecrystal diffractometer coupled with computer program.The calculated unit cell parameters are found to bea=14.07Å, b=7.87Å and c=15.01Å; α=γ=90º, β=115.14º andvolume V=1498 (Å) ³. The space group is recognized as $P2_1$ (non-centrosymmetric) which fulfills the condition for the material to exhibit NLO behaviour.Single crystals of Vanillin of thickness 1 mm were subjected to dielectric studies at room temperature for various frequencies ranging from 50 Hz to 5 MHz.It is observed that the value of dielectric constant and dielectric loss are high in the lower frequency region. The larger value of dielectric constant at lower frequencies is due to the contribution from all the four polarization viz. electronic, ionic, dipolar and space – charge polarizations. The low value of dielectric constant and dielectric loss at higher frequencies suggest that the grown crystal contains minimum density of defects with high optical quality [3].

The ac conductivity of vanillin was evaluated using the relation-

$$\sigma_{ac} = 2\pi f \varepsilon_o \varepsilon_r \varepsilon''$$

where ε_o is vacuum dielectric constant, ε_r is dielectric constant, f is the frequency of applied ac and ε'' is the dielectric loss. Fig. 2shows the variation of ac conductivity with frequency. The acconductivity is almost zero up to 20 MHz and starts increasing from 20 MHZ onwards. This is due to the fact that the relaxation time of charge carriers is not matching with the time period of the applied ac up to 20 MHz and so the conductivity of the material is almost zero.

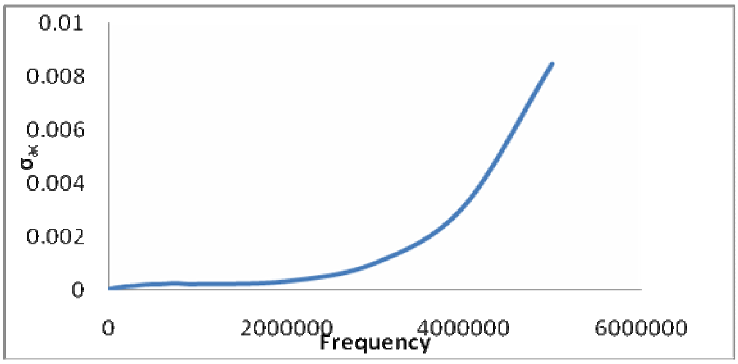

Fig. 2 Plot of ac conductivity Vsfrequency

The TGA and DTA results are shown in Figs. 3 and 4 respectively. The weight loss starts at 160^{o}C and is maximum at 265.3^{o}C. The maximum weight loss is confirmed by the endothermic peak of DTA curve at about 265.3^{o}C. It is understood that the material vanillin is thermally stable up to 160^{o}C. In the DTA curve, and exothermic peak is observed corresponding to 87.2^{o}C which is due to evaporation of trapped solvent molecules.

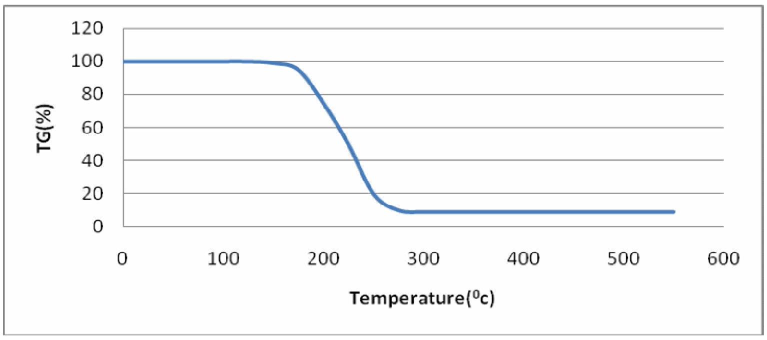

Fig. 3 TGA traces of Vanillin

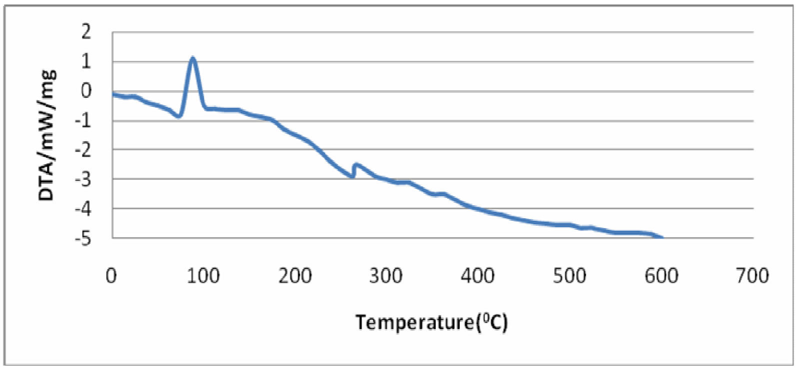

Fig. 4 DTA traces of Vanillin

The Vickers hardness number was calculated using the formula [4,5]

$$H_v = \frac{1.8544\, P}{d^2} \text{ kg/mm}^2$$

where P is the applied load and d is the average diagonal length of the impression. It is observed that the microhardness increases with load. Beyond a load of 50g, significant cracking occurs around the indentation mark. This may be due to the release of internal stress generated locally by indentation. Hence, the material can withstand the stress developed due to the maximum load of 50g.

The solid state parameters Plasma Energy (E_P), Fermi Energy (E_F) and Polarizability (α) were calculated using the following relation[6].The polarizability (α) of the grown crystal can be obtained using the following relation

$$\alpha = \left(\frac{(\hbar\omega p)^2 S_O}{(\hbar\omega p)^2 S_O + 3E_P{}^2}\right)\frac{M}{\rho} \times 0.396 \times 10^{-24}$$

where, $$S_0 = 1 - \left(\frac{E_P}{4E_F}\right) + \left(\frac{E_P}{4E_F}\right)^2$$

It is understood that the polarizability(4.806×10^{-23} cm^3)of vanillin crystal is found to be more than that ($2.14\times 10^{-23}cm^3$) of KDP crystal. Since the polarizability is responsible for NLO behavior, it is concluded the vanillin crystal is found to possess enhanced SHG efficiency due to higher value of polarizability.The nonlinear optical property and the efficiency were tested using Kurtz and Perry powder technique. It is concluded that the grown crystal vanillin is a promising material showing higher SHG efficiency for NLO applications.

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