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Synthesis and Characterisation of Undoped and Methyl Orange (Dye) doped L-Alanine Acetate Single Crystal

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Abstract : The potential application of amino acid crystals, used in NLO applications leads to intensive investigation about the material. In the present work, L-alanine acetate and methyl orange (dye) doped L-alanine acetate crystal was grown by slow evaporation method. The samples were synthesized and purified by repeated re-crystallization process and the solubility of the sample was found at different temperatures. The cell parameters were calculated using Powder X-ray diffraction analysis and the functional groups were identified using the Fourier Transforms Infra-Red Spectroscopic (FTIR) studies. From UV-Vis-NIR spectral study, it is noted that the transmission in the lower cut-off wave length, reflecting the good transmittance property of the crystal in the entire visible region. The optical emission bands were estimated by the Photoluminescence Studies. The optical band gap was also calculated for the grown crystal. NLO property of the crystal is confirmed by Kurtz-Perry powder technique. The mechanical properties such as hardness value, yield strength and stiffness constant were estimated by the Vicker's Microhardness measurement. EDS analysis shows the elements present in the grown samples. From TG/DTA analysis, the decomposition point for methyl orange doped L-alanine acetate crystal is found to be at 290° C which shows more thermal stability compared to that of undoped L-alanine acetate crystal. Keywords: L-Alanine complex; dopant; NLO; XRD;Hardness; Dye; and Spectroscopy.

1. Introduction

Nonlinear optical compounds have been extensively studied in recent years for their potential application in optoelectronics, signal conversion, optical data storage, frequency conversion, light modulation, optical memory storage, optical second harmonic generation, optical switching and Photonics^{1, 2}. Semi-organic nonlinear optical materials possess several attractive properties such as high damage threshold, wide transparency range, less deliquescence and high optical nonlinearity, which make them suitable for frequency doubling³. In view of this, there has been considerable interest in the synthesis of semi organic materials having good transmission in UV as well as visible region, high mechanical and thermal stability ⁴. Semi-organic nonlinear materials gain importance over inorganic materials because of their large polarizability and wide transmission window ^{5, 6}.

Amino acid gives various advantages particularly like crystallized in noncentrosymmetric space group and its crystals have subjected to extensive investigation by several researchers for their excellent characteristics such as optical, electrical and thermal properties⁷. The importance of amino acid in NLO applications are due to its molecule chirality, absence of strongly conjugated bonds and zwitterionic nature. Amino acids contain carboxylic (COOH) group and the proton acceptor amino (NH₂) group, known as zwitterions which create hydrogen bonds⁸. Due to this bipolar nature, amino acids have physical properties which make them ideal candidates for applications⁹.

L-alanine is the simplest semi-organic amino acid having SHG 1.18% efficiency equivalent to one-third of KDP crystal. In general, amino acid single crystals possess wide transparency in UV-Visible region¹⁰. When L-alanine is mixed with acetic acid combined to form L-alanine acetate gives various properties which is reported in the literature elsewhere ^{11,12,13}. Doping NLO crystals with various dopants can alter physical and chemical properties such as optical, thermal, mechanical etc., and the NLO crystals are doped used for various applications in optical switching, fibre optic communication etc., ^{14,15,16,17,18}. Organic dyes when doped in KDP crystals which improve the growth rate, enhance the optical and dielectric properties^{19,20}. Methyl orange is an organic dye used as a coloring agent in several applications, such as textile, paint, ink, plastic, cosmetic industries and photocatalytic degradation properties^{21,22}. In the present work undoped and methyl orange (dye) doped

L-alanine acetate crystal was grown by slow evaporation method and the results of growth, solubility, structural, optical, mechanical and thermal properties are discussed.

2. Experimental and Characterization Techniques

2.1 Synthesis

Analar Reagent (AR) grade of L-alanine, acetic acid and methyl orange were purchased commercially and the undoped L-alanine acetate salt was synthesized by taking L-alanine and acetic acid in the equimolar ratio and dissolved in deionized water.

The dissolved saturated solution was heated at 50°C forthe synthesis of L-alanine acetate salt. 5wt% of methyl orange was added into the aqueous solutions of L-alanine acetate salt. Solubility measurement was carried out using a constant temperature bath by gravimetric method^{23,24,25}.

2.2 Characterizations

The FTIR spectra were recorded for powdered samples of undoped and methyl orange doped L-alanine acetate using Perkin- Elmer FTIR spectrometer by KBr pellet technique in the range of 4000 cm⁻¹ - 400 cm⁻¹ were taken to analyse the functional group. The grown crystals were cut and polished and the specimen of 1.5 mm thickness was subjected to the transmission measurement using a spectrophotometer (LAMBDA 35) in the spectral region of 190–1100 nm. Vickers microhardness measurements were carried out on the grown crystals using a Vickers microhardness diamond indenter. The indentations were made using a Vickers pyramidal indenter for various loads from 25 to 100 g. Vickers microhardness number (H_v) for the sample crystals is calculated using the following relation

$H_v = 1.8544 P/d^2 kg/mm^2$

Where P is the applied load in kg and d is the diagonal length of indentation impression in millimetre and 1.8544 is a constant of a geometrical factor for the diamond pyramid²⁶. Yield strength of the material can be found out using the relation, yield strength (σ_y) = (H_v/3) and the stiffness constant (C₁₁) for different loads was calculated the formula C₁₁ = H_v^{7/4} where H_v is the microhardness of the material^{27, 28}. The single crystals were powdered and were irradiated by an incident radiation (1064 nm) of pulse width 8 ns from a Q-switched quanta RAY GCR Nd:YAG laser. KDP was used for calibrating the SHG intensity. The output power of the crystal was measured using a power meter and the NLO property of the crystal was confirmed from the estimation of strong green radiation of the crystal. The compositions of the samples are analysed by EDAX detector (model-JEOL Model JED – 2300).Thermogravimetric (TG) analysis is carried out to find the degradation temperature, absorbed moisture content of materials, the level of inorganic and organic components in materials and decomposition/melting points of materials. Also a differential Thermogravimetric curve (DTG) or derivative weight loss curve can be used to tell the point at which weight loss is most apparent.

3. Result and Discussions

3.1 Solubility and Crystal Growth

From Solubility measurements, the solubility increases which depends on the temperature showing positive temperature coefficient of solubility. By gravimetric method, it is noted that the solubility of L-alanine acetate gets increased when methyl orange is added as a dopant which is due to the fact that the solvent is able to accommodate a marginally increased amount of solute for the saturation at the same temperature²⁹.



Fig. 1: Solubility curves for Undoped and Methyl orange(dye) doped L-alanine acetate samples



Fig. 2:Grown Crystal for Undoped and Methyl orange (dye) doped L-alanine acetate samples

3. 2 Structural Studies

The grown samples of undoped and methyl orange doped L-alanine acetate crystal structure was analysed by single crystal X-ray diffraction studies using anENRAF NONIUS CAD4 diffractometer with MoK_a radiation (λ =0.71073 Å). The structure was solved by direct methods and refined by full-matrix least square methods using SHELXL programs ³⁰. The undoped L-alanine acetate crystals of cell parameter values are found which are close agreement to that of reported literature ³¹. The single crystal XRD data for methyl orange(dye) doped L-alanine acetate are given in the table 1.It is the necessary provision for SHG.

Sample	Cell Parameters	Volumem ³	Space Group	Crystal
	(Å)			System
Methyl	a = 5.423	463.77	$P2_{1}2_{1}2_{1}$	Orthorhombic
Orange (dye)	b = 6.418		Non-	
doped	c = 13.325		Centrosymmetric	
L-alanine	$\alpha = 90^{\circ}$			
acetate crystal	$\beta = 90^{\circ}$			
(5Wt%)	$\gamma = 90^{\circ}$			

Table 1: Lattice constants for methyl orange (dye) doped L-alanine acetate crystals

3.3 FTIR Studies

The Vibrational spectroscopic study was carried out with a view of obtaining an insight into the structural aspects of crystals. From the absorption bands/peaks of the spectra, the functional groups of the samples were identified. In the spectra, the very broad peak around 3280 cm⁻¹ is due to the stretching vibration of NH₃⁺. The peak in the region between 3100-2700 cm-1 is due to CH stretching mode, which results in the bonding of CH₃to O or N. Several bands from overtone and combination bands are weak in the region 2050-1650 cm⁻¹. The peaks at around 1600 cm⁻¹ is due to symmetric stretching vibration of C=O³². The in-plane OH bending vibration peaks are found in the region 1420-1400 cm⁻¹. The peaks in the region 1400-1310 cm⁻¹ results from COO⁻ group in carboxylic acids. The CH₃ bending modes are found in the bands are 1455 and 1412 cm⁻¹³³. The strong peaks in the region around1150 cm⁻¹ are assigned to C-N stretching vibrations³⁴. The peaks in the region of 700-750 cm⁻¹ are NH-Wagging³⁵. The peaks around 530 cm⁻¹ region are 0-H bending vibrations ³⁶.From the results it is noted that the spectrum of the doped sample is almost similar to undoped except that some shifts in the peak positions and variations in transmittance. Hence the crystals are expected to interact among same groups and ions. Yet significant difference could not be observed for doped crystals, except for broadening and shifting of absorption peaks/band in the FTIR spectra. This may be due to the inclusion of dopants in small quantity into the interstitials of the host samples.



Fig. 3: FTIR spectrum of a) Undoped and b) Methyl Orange (dye) doped L-alanine acetate crystals

3. 4 UV-Vis-NIR Spectral Studies

It is observed from the spectrum that the lower cut off wavelength of undoped and methyl orange doped L-alanine acetate crystalsare 255nm and 242 nm respectively. The optical energy band gap was calculated using the formula $E_g = 1240/\lambda$, Where λ is the wavelength of the light in nm and it is found to be 4.8625eV and 5.1239 eV for undoped and methyl orange doped L-alanine acetate crystal. As there is no significant absorption in the range 300–1100 nm, this is an advantage of using amino acids, where the absence of strongly conjugated bonds leads to wider transparency range in the visible and UV spectral regions. This transmittance is essential for the material to be used in Infrared Diode, IR Sensor Modules, Photovoltaics, LASER and SHG device applications ^{37,38,39}.



Fig. 4:Optical Transmittance spectra for a)Undoped and b) Methyl orange(dye) doped L-alanine acetate crystal

3. 5 Photoluminescence Studies

The emission spectra of the grown samples recorded in the range of 250 -700 nm and they are presented in figure 5. Here the excitation wavelength used was 240 nm.



Fig. 5:PL Emission spectra for a)Undopedand b)Methyl Orange doped L-alanine acetate crystal

The emission peaks / bands are observed for the undoped L-alanine acetate crystal are at 350, 445 nm,490 nm and 685 nm. The PL spectrum of methyl orange doped L-alanine acetate crystal consists of only two emission bands one at 411 nm and a strong red band at 625 nm. The results indicate that the grown crystals

have the brightemission in the visible region which indicates that the samples are the promising materials for the use of sensitizers to other visible luminescent organic/inorganic entities²³.

3.6 Vicker's Microhardness Test

Mechanical Property of the samples plays an important role of the device fabrication. Resistance and the lattice offers to local deformation were measured 40 . The structure and molecular composition in crystals greatly influence the mechanical properties such as hardness, yield strength and stiffness constant. Plots between the hardness number and the l-alanine acetate applied load are presented in the figure6(a). When methyl orange doped L-alanine acetate crystal shows that the hardness values are increased compared to the undoped one. The addition of dopants into L-alanine acetate crystal most possiblywhich increases the strength of bonding with the anchor material and hence increases the hardness value. The variations of hardness value, yield strength and stiffness constant with the applied load are presented in the figures 6 (b) and 6 (c). From results, it is observed that yield strength and stiffness constant of samples increase with increase of the applied load.



Fig. 6: Plots of a) Hardness Value, b) Yield Strength and c) Stiffness constant versus applied load for Undoped and Methyl orange (dye) doped L-alanine acetate crystal

3. 7 Measurement of SHG efficiency

The analysis of second-order nonlinearity like second harmonic generation (SHG) of the grown crystals was achieved by Kurtz powder method. From the observed value, the SHG efficiency of the undoped and methyl orange doped L-alanine acetate crystals are 1.13 and 1.25 times respectively than that of standard KDP crystal ⁴¹.So the dopant increases the efficiency of methyl orange(dye) doped L-alanine acetate crystal.

3.8 Energy Dispersive X-Ray Analysis (EDS)

The recorded EDS spectra of undoped L-alanine acetateand methyl orange doped L-alanine acetate crystals are shown in the figures7 (a) and (b). The obtained results show that the grown crystals have the elements such as oxygen, carbon, nitrogen, sodium and sulphur.



Fig. 7: EDS spectrum of a)Undopedb) Methyl Orange (dye)doped L-alanine acetate crystal

3.9 Thermogravimetric Analysis

Here, TG/DTG thermal curves for undoped and methyl orange doped L-alanine acetate crystals were recorded in the nitrogen atmosphere at a heating rate of 10 °C / min for a temperature range of 35°C to 600°C 42 . TG/DTA curves as shown in the figures 8 (a) and (b). From the results, it is noted that the samples have thermal stability around 200°C. Since there is no weight loss upto 150 °C, the samples have no water of crystallization.



b)Methyl Orange (dye) dopedL-alanine acetate crystal

The endothermic peaks in the figures indicate the decomposition points of the samples. The values of decomposition point are noted to be 276°C and 290°C for undoped and methyl orange doped L-alanine acetatecrystals respectively. It is to be mentioned here that there is a heavy weight loss at these temperatures and hence these temperatures are referred as decomposition points and not melting points. The sharp peak indicates to good crystallinity of the crystal. The results of microhardness studies indicate that the mechanical strength increases when the sample is doped with methyl orange and hence the value of decomposition point increases when the sample is doped with methyl orange⁴³. From the results shows that the crystal is high thermal stability for various Laser applications.

4. Conclusion

Undoped and methyl orange (dye) doped L-alanine acetate crystal was successfully grown by slow solvent evaporation method. Solubility measurement shows that the solubility increases with temperature thereby showing positive temperature coefficient of solubility. A structural study reveals that the prepared crystals are orthorhombic in nature. Photoluminescence studies shows the emission peaks at 350, 445, 490 and 685 nm for undoped L-alanine acetate and 411nm, 625 nm for methyl orange doped L-alanine acetate which indicates the suitability of these materials as sensitizers. The studies on the mechanical properties of the prepared crystals show the increase in the yield strength and stiffness constant with increase in the applied load. SHG measurement shows that the SHG values of the grown crystals are 1. 13 and 1.25 times greater than that of KDP crystal. The presences of O, C, N, Na and S are noted from EDS analysis and the decomposition point is noted from TG/DTA analysis.

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