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Growth and characterization of pure and L-alanine doped urea cobalt chloride NLO materials

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ABSTRACT

A Semi organic crystal of L-Alanine doped Urea Cobalt chloride a nonlinear optical material are synthesised and Grown by the slow evaporation technique at room temperature. The lattice dimension was carried out from powder X- ray diffraction method. The dielectric constant of the crystal was premeditated as a function of frequency. The L-Alanine doped urea Cobalt chloride crystal has good optical transmission in the entire UV-visible region. The different functional bond frequencies associated with the crystal from FT-IR analysis. The nonlinear optical properties of the crystal have been confirmed by Kurtz- powder second harmonic generation method.

Key words: Powder X-Ray diffraction, UV –Visible, FT-IR, SHG efficiency.

INTRODUCTION

Semi organic nonlinear optical materials wide range of application in the field of telecommunication, optical information storage device. These materials have large nonlinearity, high resistance, too large induced damage, low angular sensitivity, good mechanical hardness. Amino acids are strong applicant for optical second harmonic generation because they contain chiral carbon atom and crystallize the non Centro symmetric space groups [1-5]. In the present investigation of L-Alanine doped Urea Cobalt chloride (AUCC) single crystals were grown by slow evaporation technique. The grown crystals were characterized by FTIR, Powder XRD studies, Dielectric Studies, UV –Visible, FT-IR analysis, SEM analysis and SHG studies. The results obtained from various studies of L-Alanine doped Urea Cobalt chloride (AUCC) crystals are reported and discussed.

MATERIALS AND METHODS

2.1. Crystal growth

The material of the L Alanine doped urea cobalt chloride (UCC) was synthesized by slow evaporation technique. UCC prepared from Urea and Cobalt chloride in 1:3 stoichiometric Ratio according to the following chemical reaction. To avoid the decomposition, maintained room temperature this solution was prepared from deionised water. The solution was stirred with magnetic stirrer and heated at 50°C, formed UCC. The one mole of solution of L- Alanine was the doped solutions of the UCC were growing by slow evaporation method [6-7]. The Single crystal sizes of 15.12.12 mm3 with good transparency were harvested in 30 days as shown in Fig 2.

 $CoCl_2 + 3 [CO (NH_2)_2] \longrightarrow [CO [CO (NH_2)_2]_3] Cl_2]$



Figure 1.Urea Cobalt Chloride



Figure 2.L-Alanine doped Urea Cobalt Chloride

CHARACTERIZATION

The grown crystal of L-Alanine doped Urea Cobalt Chloride were subjected at various characterization via Powder X-ray Diffraction, Dielectric studies, UV-Visible, FT-IR analysis, SEM analysis, SHG test.

3.1. Powder X-Ray Diffraction

The fine powder of crystal was recorded pattern is shown in Fig. The powder sample was scanned in 1° for a time interval of 10 seconds over a 20 range of 10° to 70°. The sharp and well Bragg's peaks at specified 20 angle shown crystalline nature and purity of the crystal. New peaks in the XRD pattern of the grown crystal confirm incorporated in cobalt chloride. The lattice parameter are obtained are a=6.0870Å, b=4.0210Å, c=9.0880Å. The structure is confirmed to be orthorhombic with space group is P2₁2₁2₁



Figure: 3.Powder XRD analysis for L-Alanine doped Urea Cobalt Chloride single crystal

3.2. Dielectric studies

Single crystals of L-Alanine doped Urea Cobalt Chloride of thickness 1 mm were subjected to dielectric studies at room temperature for various frequencies ranging from 100 Hz to 5 MHz using HIOKI 3532-50 LCR HITESTER. The dielectric constant is evaluated using the relation $\dot{\varepsilon}=Cd/\dot{\varepsilon}0A$. Where d is the thickness and A is the area of the cross section of the grown crystal. The variation of dielectric loss as a function of frequency at room temperature is shown in figure-4. From the graph, the dielectric loss is seen to decrease with increase in frequency. The very low value of dielectric constant at higher frequencies is important for the fabrication of materials towards ferroelectric, photonic and electro optic devices [8-10]



Figure: 4 Dielectric loss Vs log of frequency curve for L-Alanine doped Urea Cobalt Chloride single crystal

3.3. UV Analysis

The UV-Vis transmittance spectrum (Lambda 35) of L-Alanine doped Urea Cobalt Chloride single crystal was recorded in the range 190-1100nm and shown in figure 5.3.a. The crystal shows a good transmittance in the visible region. The UV cut off wavelength was around 212nm. The spectrum further indicates that the crystal has wide optical window from 200-600nm combined with the (85%) very good transparency window makes the material suitable for optoelectronics applications.



Figure: 5 UV spectra for L-Alanine doped Urea Cobalt Chloride single crystal

3.4. FT IR Analysis:

FT-IR spectra of L-Alanine doped Urea Cobalt Chloride single crystal were recorded using PERKINELMER RX1in the frequency region 400-4000 cm-1 by KBr pellet technique. FT-IR spectra of growing single crystal were shown in Fig-5.4. The broad bond lying in the range of 3089.50 and 2935.32 cm⁻¹ is due to the N-H stretching vibration of the NH₂ group of thiourea. The Amino group of absorption bands was noted at 846.56 cm⁻¹. The peak at 2293.76 and 2247.60 and 1232.66 cm⁻¹ is due to CH₃ stretching. The peak at 2600.13 and 2509.15 cm⁻¹ is due to NH₃⁺ stretching vibration. The sharp absorption peak at 2110.71 cm-1 is due to a combination of NH₃⁺ symmetric stretching and torsion oscillation. The C=O stretching vibrations occur at 1620.15 cm⁻¹.



Sample Name: SB-1 Figure: 6. FT-IR Analysis for L-Alanine doped Urea Cobalt Chloride single crystal

The frequencies pertaining to1509.68 cm⁻¹ show the presence of the carboxyl group .The peaks at 1456.67 cm⁻¹ are due to NH₂ bending vibration. The peak at 1411.50 cm⁻¹ is due to symmetric stretching of C-COO⁻. N-C-N vibration is a peak at 1096 cm⁻¹. The peaks at 1301.32 cm⁻¹ corresponds to C-N symmetric stretching vibration. The C-O symmetric stretching is observed at 1011.03 cm⁻¹. The peaks at 1152.43, 1109.06 cm⁻¹ corresponds to rocking deformation of NH₃⁺ vibration. The C-C-N symmetric stretching vibration is confirmed by the presence of peak at 918.16 cm⁻¹. Due to C-CH₃ bending, a strong absorption peak was formed 846.56, 771.23 cm⁻¹. The peak at 647.66 is due to COO- plane deformation. The peak at 538.05 cm⁻¹ represents torsion oscillation of NH₃⁺. So the crystal is devoid of covalent bonded water to cobalt.

3.5. SEM Analysis

The surface morphology and nature of the grown crystal analysis was studied by scanning electron microscope (SEM) and the image is shown in Fig-5.6. It shows that the grown crystal is a rectangle like shape.



Figure: 7. SEM microgram for L-Alanine doped Urea Cobalt Chloride single crystal

3.6. SHG Efficiency

The grown crystals were subjected to the NLO study to measure the efficiency with respect to pure KDP. The SHG property of a grown crystal was tested by the Kurtz and Perry powder method. The fundamental beam of wavelength 1064nm from a Q-switched Nd:YAG laser with a pulse energy3mJ/pulse, pulse width 8ns, and repetition rate 10 Hz was used. The second harmonic efficiency of the crystals was established by the emission of green radiation from the crystals. The measured output for pure KDP and L-Alanine doped Urea Cobalt Chloride were 24 mV respectively. SHG efficiency of L-Alanine doped Urea Cobalt Chloride 2.1 times than that of pure KDP crystals.

CONCLUSION

Good optical quantity of NLO transparent crystals of pure, L-Alanine doped Urea cobalt chloride are successfully grown by slow evaporation technique. Structural Characterization was carried out by Powder X-ray diffraction and lattice parameters have been evaluated. Powder XRD studies revealed that orthorhombic system. The dielectric loss with the frequency of the L-Alanine doped Urea Cobalt chloride single crystals proves that these materials possess an enhanced optical quality with lesser defects. UV –Visible spectrum was shown in good optical transmittance in the entire visible region. The FT-IR analysis shows the bands belonging to spectrum of pure and L-Alanine doped Urea cobalt chloride. The SHG efficiency of L-Alanine doped Urea cobalt chloride was found to be 2.1 times greater Urea respectively.

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