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SYNTHESIS GROWTH AND CHARACTERIZATION OF STRONTIUM CHLORIDE DOPED L-VALINE CADMIUM CHLORIDE CRYSTALS GROWN BY SLOW EVAPORATION TECHNIQUE

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ABSTRACT:- A new semi organic non-linear optical material, L-valine cadmium chloride (LVCC crystal) doped with strontium chloride has been grown successfully by slow evaporation technique. The grown crystal was characterized by using powder X- ray analysis and confirmed the identity of the title compound. The presence of functional groups of the grown crystal has been confirmed by Fourier Transform Infrared Spectroscopy (FTIR) analysis. TGA/DTA studies revealed the thermal stability and the mechanical streangth of grown crystals. The optical absorption study was examined by UV-VIS spectrum.

Keywords: Slow evaporation method, XRD,FTIR, UV spectrum, Thermal studies.

INTRODUCTION

Growth of Non Linear Optical (NLO) single crystals with good quality initiates the development of many novel devices in the field of optoelectronics and optical communication such as optical modulator, optical data storage and optical switches [1-4]. Semi Organic NLO materials are generally have a high nonlinear coefficient, high laser damage threshold, high thermal stability and mechanical strength than inorganic crystals [5-6]. In this connection, amino acids are prominent materials for NLO applications, as they contain zwitterions, which create the hydrogen bonds used for the generation of non centro symmetry structures favorable for attractive SHG properties of crystal [7-9]. Except Glycine, generally amino acids having a Proton donating carboxyl group (Coo⁻) and a proton accepting amino (NH₃+) group, having a tendency of combining with that of inorganic salt [10]. As a result good NLO crystals such as L-Valine hydro bromide [11], L-Valine cadmium acetate [12], L-Valine Potassium Chloride [13], L-valine Zinc Sulphate [14-15], L-Valine Nickel Chloride [16], L-Valine Succinate [17], L-Valine Oxalate [18], L-Valine hydro Chloride [19], L-Valine hydro bromide [20], L-Valine Cadmium bromide [21], L-Valine Cadmium Chloride [22] has been grown and its characterization studies have been already reported.

In this present investigation, L-Valine Cadmium Chloride Crystal (LVCC) doped with strontium chloride has been grown from its aqueous solution by slow evaporation method. Attempt has been made to characterize the crystals through the techniques such as powder XRD, FTIR, TGA/DTA and UV-Visible.

Crystal Growth

L-Valine and Cadmium Chloride has been taken in an equimolar ratio of 1:1 and was dissolved in deionized water. 1M of strontium chloride has been added slowly. The solution was stirred continuously using a magnetic stirrer for six hours to yield a homogeneous mixture. The prepared solution was filtered and kept undisturbed at room temperature. Tiny seed crystals with good transparency were obtained due to spontaneous nucleation after a period of fifteen days. The chemical equation governing the reaction is,

 $C_5H_{11}NO_2+CdCl_2$. $H_2O+SrCl_2 \rightarrow Cd[C_5H_{11}NO_2]Cl_2$. $SrCl_2$. H_2O .

The photograph of the grown LVCC crystal doped with strontium chloride is shown in fig.1

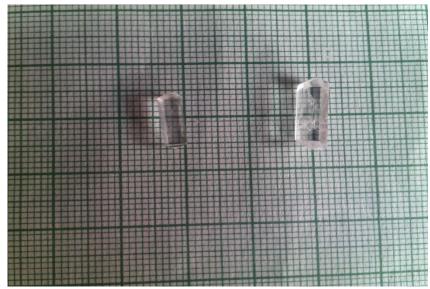


Fig. 1 The Photograph of LVCC Crystal doped with Strontium Chloride

EXPERIMENTAL DETAILS

The LVCC crystal doped with strontium chloride was subjected to powder X-ray diffraction studies. Powder X-ray diffraction pattern was undertaken by X-ray diffraction meter (Model JDX 8030) with CUK α (λ = 1.5418Å). The compositional analysis of the grown LVCC crystal doped with strontium chloride was made quantitatively using energy dispersive X-ray analysis. The presence of functional groups has been confirmed with the help of Perkin Elmer FTIR spectrometer in the range of 400 to 4000 cm⁻¹ using KBr pellet technique. The optical transmission spectrum of LVCC crystal doped with strontium chloride was investigated by λ 35 model Perkin Elmer double beam uv-visible spectrometer in the rage of 190nm to 1100nm. The nonlinear optical property was confirmed by illuminating the sample by using Nd: YAG laser (λ = 1064 nm). **RESULTS AND DISCUSSIONS**

Powder XRD Analysis

The powder XRD pattern of LVCC crystals doped with strontium chloride is shown in fig.2. The sharp and sensitive peaks confirmed the crystalline nature of the grown crystal. The highest intense peaks of the observed powder XRD pattern has been compared with the standard XRD pattern of L-Valine (JCPDS file no.33-1954) and is indexed.

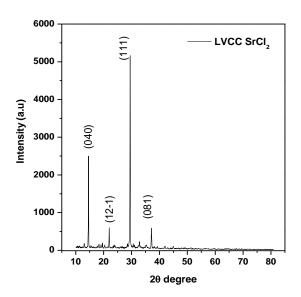


Fig.2 The powder XRD pattern of the grown LVCC crystal doped with strontium chloride FTIR Analysis

The FTIR spectrum of LVCC crystal doped with strontium chloride is presented in fig.3. In the spectrum the narrow envelope found at 3145 cm⁻¹ is due to NH₃⁺ asymmetric stretching vibrations. The medium peak observed at 2978 cm⁻¹ is assigned to NH₂⁺ symmetric stretching vibrations. The peak occurs at 2628 cm⁻¹ is associated with N-H-O valence stretching combination. The narrow peak at 2110 cm⁻¹ may be due to C-O-C stretching vibration. The strong peak at 1588cm⁻¹ is attributed to COO⁻ asymmetric stretching vibration. NH₃⁺ symmetric deformation have been observed at 1508 cm⁻¹. The weak bands at 1425 and 1393 cm⁻¹ are correlated to bending vibration of CH₂ and COO⁻ symmetric stretching vibration respectively. The C-O stretching components are observed at 1351 and 1327 cm⁻¹. C-C stretching mode vibrations appear at 1178 and 1140 cm⁻¹. The band occur at 947cm⁻¹ confirm the rocking vibration of CH₂. The peaks at 896 and 848 cm⁻¹ are owing to the rocking vibration of NH₃⁺. The peaks seen at 824 and 716 cm⁻¹ are attributed to C-H out of plane bending. The absorption frequencies at 775 and 753 cm⁻¹ are due to C=C stretching (Skeletal). The frequencies at 542, 475 and 430 cm⁻¹ are due to the C-Co deformation, C-C deformation and Coo⁻ rocking mode of the compound respectively.

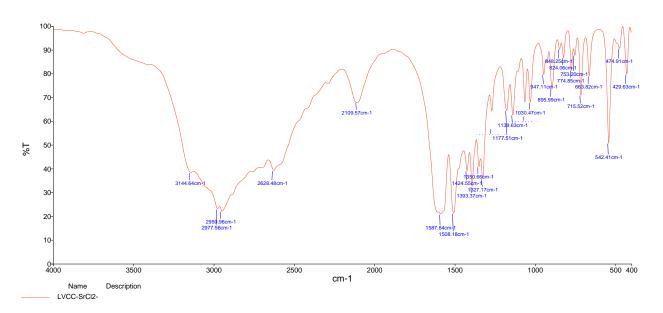


Fig.3 FTIR spectrum of LVCC crystal doped with strontium chloride

Thermal Analysis

The thermogram of LVCC crystal doped with strontium chloride is shown in fig.4. The grown crystal has high amount of thermal stability upto 250°C. The strong endothermic peak observed at 276.9°C is assigned to the melting point of the given compound. The second stage of decomposition was observed at the temperature range of 250°C to 290°C accompanied with a weight loss of 87.5% corresponds to the loss of L-Valine molecules. In the third stage (290°C – 560°C), the remaining 10% loss of the grown LVCC crystal doped with strontium chloride is due to the removal of cadmium and other volatile compounds.

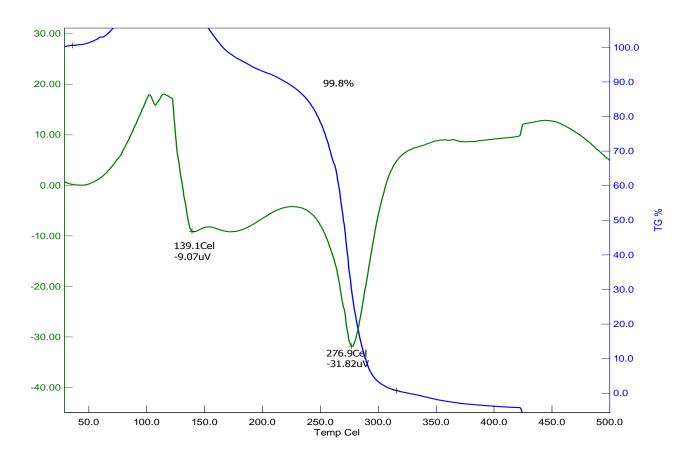


Fig. 4 TGA/DTA spectrum of LVCC Crystal doped with strontium chloride

UV - spectral Analysis

The optical transmission spectrum of LVCC crystal doped with strontium chloride is shown in fig. 5. From the graph it is concluded that the grown crystal have highly transparency over a considerable region of wavelength in uv and visible region. The lower cut off wavelength was found to be 240 nm attest the usefulness of this material for non linear optical applications [23].

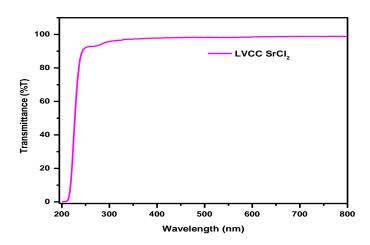


Fig. 5 The optical transmission spectrum of LVCC crystal doped with strontium chloride

CONCLUSION

The title compound L-Valine Cadmium Chloride (LVCC) crystal doped with strontium chloride has been grown by the conventional slow evaporation technique. The sharp peaks from the observed powder XRD pattern of LVCC doped with strontium chloride crystal confirmed the crystalline nature. The functional groups present in the compound has been explained on the basis of FTIR studies. The UV cutoff wave length has been found out as 240 nm. All characterization properties revealed the NLO application of grown crystal.

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