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Synthesis, growth and characterization of L-valine cadmium chloride monohydrate—A novel semiorganic nonlinear optical crystal

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ABSTRACT

Single crystals of L-valine cadmium chloride monohydrate (LVCC), a novel semiorganic nonlinear optical material were synthesized and grown from aqueous solution by slow evaporation method at room temperature. The powder X-ray diffraction pattern and FT IR spectrum analysis confirmed the formation of the new crystal. LVCC has good optical transmission in the entire visible region, which is an essential requirement for a nonlinear crystal. The thermal studies reveal that the material has good thermal stability. The LVCC crystal was characterized by energy dispersive X-ray analysis. The optical second harmonic generation conversion efficiency of LVCC was determined using Kurtz powder technique.

1. Introduction

Nonlinear optics (NLO) is an innovative area of research and development which will play a key role in the field of optoelectronics and photonics [1]. The apparent development of semiorganic materials, where the organic ligand is ionically bonded with inorganic host refined the search of new materials with high optical nonlinearities which is an important area due to their optical applications such as optical communication, optical computing, optical information processing, optical disk data storage, laser fusion reaction, laser remote sensing, color display, medical diagnostics, etc. [2]. In recent years, complexes of amino acids have been surveyed as attractive materials for NLO applications [3,4]. Amino acids are interesting materials for nonlinear optical applications, they contain a proton donating carboxyl group (COO^-) and a proton accepting amino (NH_3^+) in them except for glycine [5]. Complexes of amino acid with inorganic salts are promising materials for optical SHG, as they have a tendency to combine the advantage of the organic amino acid with that of the inorganic salt. Hence L-arginine, L-histidine, L-threonium, L-alanine and L-valine have been subjugated for the formation of salts with different inorganic acids. As a result very good semiorganic materials such as L-arginine phosphate monohydrate (LAP) [6], L-histidine hydrochloride [7], L-analine cadmium chloride [8], L-valine hydrochloride [5] are some of examples which proved very suitable materials for NLO applications.

In the present study, L-valine is combined with cadmium chloride monohydrate (LVCC) for the first time grown a new semiorganic nonlinear optical material by slow evaporation technique at room temperature. The grown crystals were subjected to various characterization studies such as X-ray diffraction, FT IR, UV–vis, thermal EDAX and second harmonic generation (SHG) efficiency studies.

2. Material synthesis and crystal growth

All the starting materials were pure. The synthesis and growth process were carried out in aqueous solution. The LVCC has been synthesized by taking L-valine and cadmium chloride monohydrate in 1:1 equimolar ratio. The calculated amounts of L-valine and cadmium chloride were dissolved in double distilled water using a magnetic stirrer. An adduct LVCC was formed according to the reaction:

$$CdCl_2{\cdot}H_2O + NH_2CH_3CH_2CH_2CHCOOH \rightarrow Cd(NH_2CH_3CH_2CH_2CHCOOH)Cl_2{\cdot}H_2O$$

The solution was kept in undisturbed condition and allowed to crystallize by slow evaporation technique at room temperature. Care was taken to minimize the temperature fluctuations and mechanical disturbance. After 25–30 days colorless, transparent crystals of LVCC were harvested. The purity of the synthesized salt was improved by successive recrystallization process. The photograph of as-grown crystal is shown in Fig. 1.

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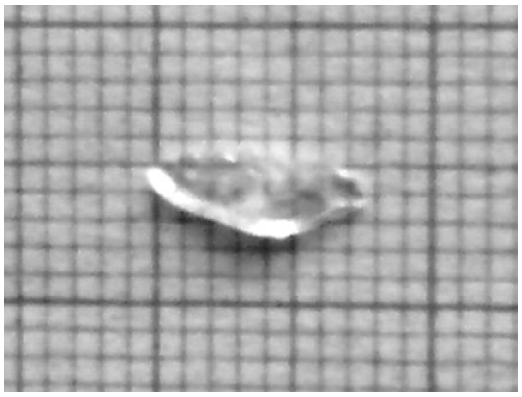

Fig. 1. As-grown LVCC crystals.

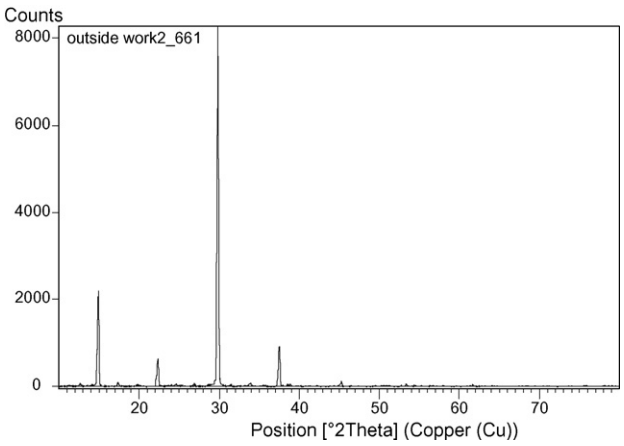

Fig. 2. Powder XRD patterns of LVCC crystals.

3. Results and discussion

3.1. Powder X-ray diffraction

Powder X-ray diffraction data were collected for the grown single crystals. The pattern was recorded using a JEOL JDX services instrument with Cu Kα (λ = 1.5406 Å) radiation. The powdered sample was scanned in the range 10–90 °C at a scan rate of 2°/min. The powder X-ray diffraction pattern of LVCC is shown in Fig. 2. The prominent well defined Bragg's peak at specific 2θ angle reveals the high crystallinity of LVCC crystals. The *d*-spacing and their relative intensities of the diffraction peaks tabulated in Table 1.

Table 1
X-ray powder diffraction data of LVCC crystals.

2θ	FWHM	*d*-Spacing [Å]
12.6673	0.3264	6.98252
14.9671	0.1224	5.91438
17.3929	0.3264	5.09459
19.7788	0.6528	4.48509
22.3602	0.2856	3.97279
29.7921	0.1224	2.99650
29.8918	0.1224	2.98674
31.4202	0.3264	2.84485

3.2. FT IR spectrum studies

The infrared spectral analysis is effectively used to understand the chemical bonding and it provides information about molecular structure of the synthesized compound. The FT IR spectra were using Thermo Nicolect V-200 FT IR spectrometer by KBr pellet method in the range 4000–400 cm^{-1} as shown in Fig. 3. The peak around 3000, 2628 cm^{-1} is due to N–H stretching vibration. The peak around 1507 cm^{-1} is NH_3^+ symmetric deformation [9]. The peak at 1472 cm^{-1} is due to CH_3 asymmetric stretching. The peak obtained at 1395 for COO^- symmetric stretching [5]. CH_3 rocking curve obtained at 1269 cm^{-1}. The peak obtained at 1032 cm^{-1} is due to the C–N stretching vibration CH_2 rocking curve obtained at 889 cm^{-1}. The obtained frequency values are shown in Table 2.

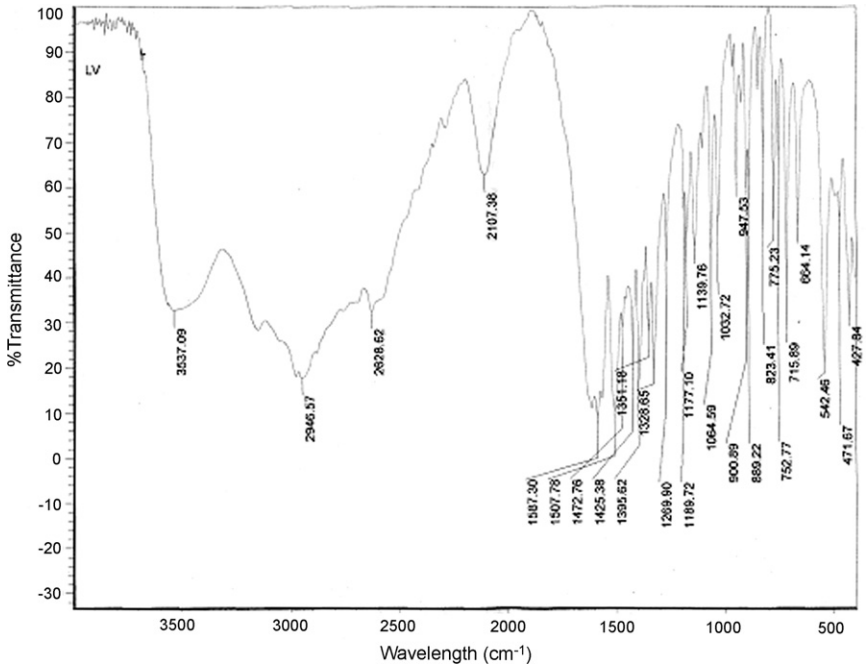

Fig. 3. FT IR spectrum of LVCC crystals.