Journal of Crystal Growth 340 (2012) 118–122

Contents lists available at SciVerse ScienceDirect

Journal of Crystal Growth

journal homepage: www.elsevier.com/locate/jcrysgro

Growth and characterization of large single crystals of L-serine methyl ester hydrochloride

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ARTICLE INFO

Article history:
Received 28 July 2011
Received in revised form
2 December 2011
Accepted 3 December 2011
Communicated by M. Fleck
Available online 9 December 2011

Keywords:
A1. Characterization
A1. Recrystallization
A1. Solubility
A1. X-ray diffraction
A2. Growth from solutions
A2. Single crystal growth

ABSTRACT

Single crystal of L-serine methyl ester hydrochloride (LSMEHCl), was grown using slow evaporation solution growth technique. The grown crystal was confirmed by single crystal X-ray diffraction and the presence of functional groups were identified by FT-IR analysis. From the Optical transmission spectra of the grown crystal the optical energy band gap is found to be 5 eV and the refractive indices (n_x, n_y and n_z) were found using Brewster's angle method. The melting point of the material obtained using melting point apparatus is 168 °C. The thermal stability of the crystal was investigated. The second harmonic generation was tested with different particle sizes of powdered LSMEHCl by Kurtz–Perry powder method using Nd:YAG laser, which established the existence of phase matching.

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1. Introduction

Nonlinear optical (NLO) materials capable of generating the second harmonic frequency, play an important role in the field of optoelectronics and photonics [1,2]. Organic materials are attractive due to their nonlinearities, ultra fast response and high laser damage threshold [3,4]. Many natural amino acids individually exhibit the nonlinear optical properties [5] because they have a donor NH_2 and acceptor COOH and also intermolecular charge transfer is possible.

L-serine is an organic compound under an amino acid category. It is one of the naturally occurring protenogenic amino acids. L-serine exists in a zwitterionic form; the molecule can combine with anionic, cationic and overall neutral constituents. Although the crystal structure of L-serine methyl ester hydrochloride (LSMEHCl) was reported [6], no detailed analysis of characteristic properties, is available in the literature. Hence in the present investigation we report the bulk crystal growth and characterization of the title compound. The SHG behavior of the material is observed and reported for the first time. The present study includes the growth of L-serine methyl ester hydrochloride [$C_4H_{10}NO_3^+ \cdot Cl^-$] by slow evaporation solution growth technique at room temperature using methanol as solvent. The molecular structure of the LSMEHCl crystal is shown in Fig. 1. The esterification of the carboxyl group of amino acids plays an important role in the synthesis of peptides, especially due to the increased solubility in organic solvents [7].

2. Experimental procedure

2.1. Solubility measurement

The low temperature solution growth technique is widely used for the growth of organic, inorganic and semiorganic materials. The estimation of solubility of the proposed material in chosen solvent is an important step in crystal growth from solution. Hence the solubility studies for the title compound were performed at different temperatures from 30 °C to 50 °C. The commercially available L-serine methyl ester hydrochloride (Sigma Aldrich 98%) was purified by repeated recrystallization process and the recrystallized materials were used for solubility study and growth. The temperature of the solution was maintained above the chosen constant temperature and continuously stirred using a motorized magnetic stirrer to ensure homogeneous temperature and concentration throughout the entire volume of the solution. The solubility of the title compound linearly increases with increase of temperature as shown in Fig. 2.

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doi:10.1016/j.jcrysgro.2011.12.008

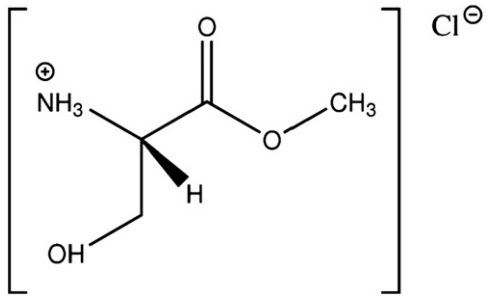

Fig. 1. Molecular structure of LSMEHCl.

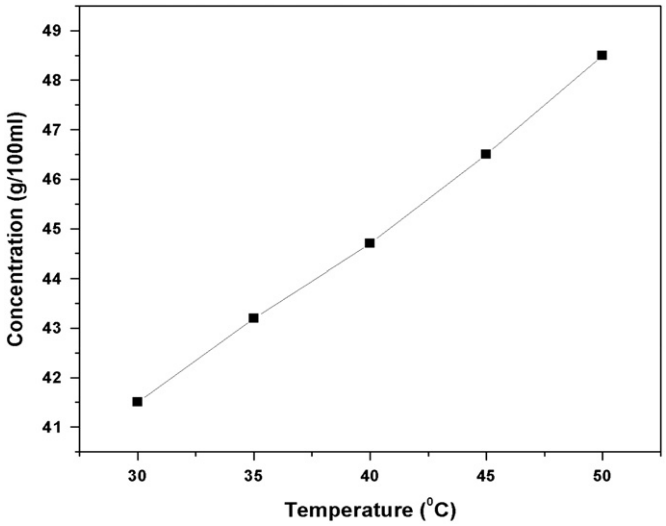

Fig. 2. Solubility of LSMEHCl.

2.2. Crystal growth

The single crystals of L-serine methyl ester hydrochloride were successfully grown from slow evaporation solution growth technique at room temperature using methanol as solvent. The saturated solution of L-serine methyl ester hydrochloride was obtained by dissolving the purified charge material into the methanol solvent with continuous stirring of the solution using a magnetic stirrer at room temperature (35 °C) with the help of solubility data. On reaching saturation, the equilibrium concentration of the solute was gravimetrically determined. The beaker containing the solution was optimally closed for controlled evaporation and housed in constant temperature bath (± 0.01 °C). Transparent single crystals of size 27 mm × 5 mm × 3 mm were obtained from mother solution after 35 days. The grown crystal is shown in Fig. 3.

3. Characterization studies

The single crystal X-ray diffraction analysis of LSMEHCl crystal was carried out using ENRAF NONIUS CAD4 X-ray diffractometer and its lattice parameters were determined. Fourier transform infrared spectrum was recorded by the KBr pellet technique using a BRUKER 66V FT-IR spectrometer to confirm the functional groups present in the title compound in the range of 400–4000 cm^{-1}. UV–vis spectrum was recorded in the region of 200–800 nm using VARIAN CARY 5E spectrometer. The thermal behavior was studied by thermogravimetric analysis using a NETZSCH STA 409C/CD thermal analyzer in the nitrogen atmosphere. The melting point of the material was measured by melting point apparatus (VEEGO MODEL: VMP-PM). The second harmonic generation (SHG) of the crystal was tested by the Kurtz–Perry powder method.

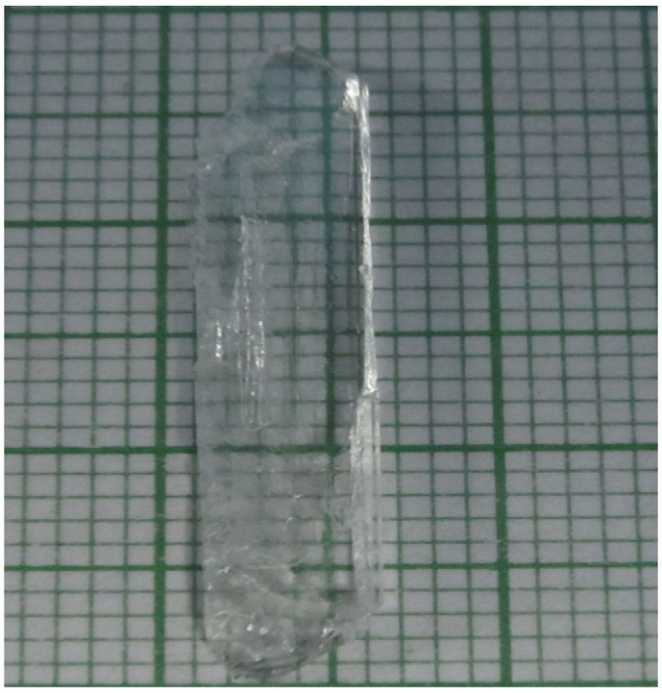

Fig. 3. As grown single crystal of LSMEHCl.

4. Results and discussion

4.1. Single crystal X-ray diffraction analysis

The single crystal XRD study enumerates that the LSMEHCl belongs to the monoclinic crystal system with space group $P2_1$ and the lattice parameters are $a=5.217(6)$ Å, $b=6.437(7)$ Å, $c=11.740(14)$ Å, $\alpha=\gamma=90^\circ$, $\beta=90.496(17)^\circ$ and $V=394$ Å^3. The obtained single crystal XRD data are in good agreement with the reported literature values [6].

4.2. FT-IR spectral analysis

The FT-IR spectrum of LSMEHCl was recorded between 4000 and 400 cm^{-1} by the KBr pellet technique. The resulting spectrum is shown in Fig. 4. The intense broad band in the high energy region includes N–H stretching vibration of NH_3^+ at 3367 cm^{-1}. It also includes O–H stretching vibration of alcoholic group. The CH_2 stretching vibration occurs at 2956 cm^{-1}. The fine structures at 2662, 2552, 2491, 2420 cm^{-1} are due to hydrogen bonding. The C=O stretching occurs at 1750 cm^{-1}. The N–H deformation occurs at 1597 and 1508 cm^{-1}. The CH_2 bending vibrations are at 1474 and 1382 cm^{-1}. The –COO– vibrations occur at 1298 and 1254 cm^{-1}. The groups of peaks lying between 1180 and 950 cm^{-1} are due to C–O vibration and OH deformation. The NH_3^+ torsional oscillation occurs at 582 cm^{-1}. The peak at 1943 cm^{-1} is attributed to combination band of the peaks at 1037 and 900 cm^{-1}. There are also series of combination bands between 2200 and 2000 cm^{-1}. From the FT-IR analysis it is confirmed that L-serine is in the form of an ester with the amino group protonated.

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