

Growth and Spectral Analysis of Pure and Potassium Doped L-Prolinium Trichloro Acetic Acid Crystal

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Abstract: *In current study growth and analysis of pure and potassium doped L-prolinium trichloro acetic acid single crystals have been performed by alike characterization technique. The single crystal has been harvested by slow solvent evaporation technique. The functional groups of grown crystals have been identified by Fourier transform infrared technique. The constituent elements of pure and doped crystal have been confirmed by employing the EDS analysis. The transmittance window of grown crystal has been evaluated within 200-1100 nm by means of UV-Visible spectral analysis. The tolerance to thermal exposure was confirmed by using the TG/DTA technique.*

Keywords: Crystal growth, Spectral analysis, Optical studies, Thermal studies

I. INTRODUCTION

Currently there is an increasing interest in developing compounds with large non-linear optical susceptibilities. Some organic compounds are the target to develop such materials due to their highly polarizable electronic clouds and the strong impact of changes of the molecular structure on their NLO properties. Amino acids is one of the promising building block material which have been widely studied and there are a number of research papers reporting NLO compounds synthesized by a combination of an amino acid with an inorganic counterpart [1-5]. Among the amino acids, all except glycine, are characterized by chiral carbons, a proton donating carboxyl (-COOH) group and a proton-accepting amino (-NH₂) group. L-proline is vital amino acid amongst the family because it is the only one in which the amine group is part of a pyrrolidine ring, thus making it rigid and directional in biological systems [8]. L-proline has been exploited for the formation of salts with different organic and inorganic acids [9]. L-prolinium picrate exhibits second harmonic generation (SHG) efficiency 52 times higher than KDP [10]. L-prolinium trichloro acetic acid (LPTCA) is significant organic material that has gained attention in the field of crystal growth due to its remarkable properties such as high optical quality, high thermal stability, and tunable nonlinear optical properties. Recently, doping of LPTCA with various organic and inorganic materials has been studied to enhance its properties for different applications. The present study focuses on growth and spectral analysis of pure and K doped LPTCA crystals. The aim of this study is to investigate the effect of doping on the crystal morphology and spectral properties of LPTCA crystals. The spectral analysis of the LPTCA crystals was performed using UV-VIS spectroscopy. The results of this study will enable better understanding of crystal growth and effect of doping on optical and thermal properties of LPTCA crystals. The results will be evaluated for exploring the potential applications of K-doped LPTCA crystals in optics, sensors and nonlinear optical devices.

II. EXPERIMENTAL DETAILS

L-prolinium trichloroacetic crystal growth was achieved by dissolving the high purity grade L-proline and acetic acid in double distilled water in equimolar proportion. The solution was stirred for 4-5 hours and filtrate was kept for slow

evaporation to obtain the pure LPTCA crystal [1]. potassium (K) doping was achieved in saturated solution of LPTCA material by adding 0.1 mol of potassium. The K-LPTCA crystal were obtained by slow evaporation within period of two weeks. Generally, the presence of the impurities is found to have a strong influence on the growth rate [4] and to gain purity pure and K-LPTCA crystal were repeatedly recrystallized Fig. 1a and Fig. 1b shows the pics of harvested pure and K⁺ doped L-PTCA crystals respectively.



Fig1.(a) L-PTCA



Fig1(b). K⁺ doped L-PTCA

III. CHARACTERIZATION

The FT-IR spectra of L-PTCA crystals were recorded in the range 400–4000 cm⁻¹ by BRUKER method to study the functional groups in sample. The metal element detected by EDS. Linear optical properties of the crystals were studied using a UV–vis–NIR spectrometer in the region 200–1100 nm. The thermal stability was identified by thermogravimetric (TG) and differential thermal analyses (DTA). Thermogravimetric and differential thermal analysis of L-PTCA crystals were carried out between temperature range 25 °C and 900 °C in nitrogen atmosphere at heating rate of 10 °C/min.

IV. RESULTS AND DISCUSSION

4.1 FT-IR

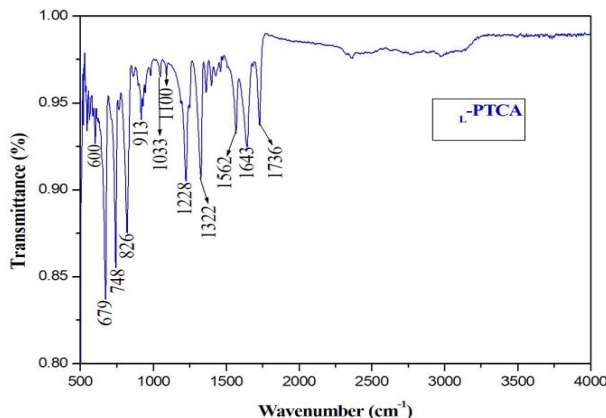


Fig.2a Pure LPTCA

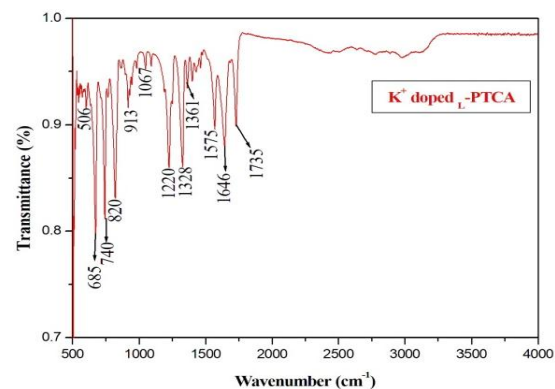


Fig. 2b K⁺ doped LPTCA

In this study, pure L-prolinium trichloro acetic acid and potassium doped LPTCA were characterized using FTIR spectroscopy. The FTIR spectra were recorded using a BRUKER Alpha-ATR spectrophotometer. In the FTIR spectra the main peaks observed were at 1736 cm⁻¹ (C=O stretching of carboxylic acid), 1562 cm⁻¹ (N-H) bending of amine) and 1322 cm⁻¹ (C-C stretching of proline). In the potassium doped sample, the additional peak observed was at 1575 cm⁻¹, due to the presence of K⁺ ion. In addition, the shift in frequency of the peaks was observed in the potassium doped sample with respect to spectrum of LPTCA crystal. The C=O stretching peak shifted to 1735 cm⁻¹, indicating the formation of a hydrogen bond between the oxygen of the carboxylic acid group and the potassium ion. The N-H bending peak also shifted from 1562 cm⁻¹ to 1565 cm⁻¹ due to the interaction with the K⁺ ion.

EDS:

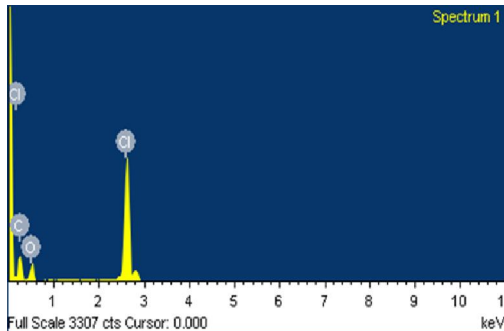


Fig.3a pure LPTCA

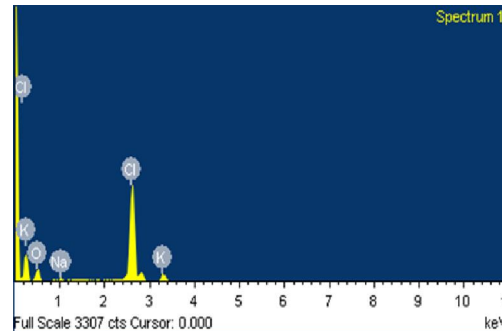


Fig.3b. K doped LPTCA

EDS (Energy Dispersive Spectroscopy) is an analytical technique that is used for the elemental analysis of a sample. In this study, pure and K-doped LPTCA crystal were characterized using EDS analysis. The sample was loaded onto the sample holder and placed under the electron microscope for analysis. In the EDS spectra of LPTCA crystal, the main elements observed were carbon, oxygen, and chlorine. The presence of these elements is consistent with the chemical composition of LPTCA. The EDS analysis revealed that there is no contamination in pure LPTCA crystal. In the K-LPTCA sample the additional peak observed was due to presence of potassium ions. The presence of K^+ ion was confirmed by the energy peak evident in the spectrum. The results of the EDS analysis showed that the sample was doped with potassium ions.

4.2 UV-Visible-NIR Spectral Analysis

UV Vis NIR spectroscopy is a widely used analytical technique employed for linear optical analysis of materials. It is used to determine important information such as electronic transitions, molecular structure, and concentration of light – absorbing species in a sample. In this study, pure and K-LPTCA crystal were characterized by UV VIS NIR spectrometer. Within 200-1100 nm range. In the spectral analysis of pure and K-LPTCA crystal sample, the cut-off wavelength was observed at 298 nm wavelength. The transmittance of pure and K-LPTCA crystal is found to be 61% and 70% respectively. The transmittance spectrum reveals that transmittance of LPTCA is increased due to K^+ doped LPTCA crystal as compared to that of pure LPTCA crystal

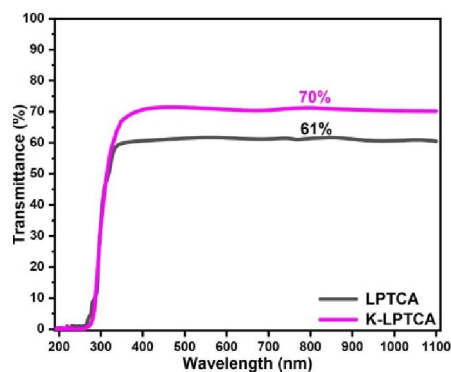


Fig. 4 Transmittance of pure and K doped LPTCA

4.3 TG/DTA

TG/DTA is used to measure the thermal stability of a sample. It evaluates the weight loss and heat flow of a sample within given range of temperature. In this study, pure and K doped LPTCA crystal were characterized using TG/DTA analysis. The TG/DTA analysis was carried out on a Perkin Elmer thermo gravimetric analyser. The TG and DTA analysis was simultaneously performed within 25 to 900 °C at a heating rate of 10 °C/minute. A flow of nitrogen gas

helped prevent oxidation of the samples. The TG curve obtained for the pure LPTCA crystal showed a prominent weight loss starting at 107 °C, which evidences absence of moisture in crystal. There was a significant change in DTA curve at around the same temperature as the weight loss, indicating the endothermic process. The K-LPTCA crystal showed weight loss at 109°C compare to pure LPTCA. The observed curve can be attributed to thermal degradation of the crystal.

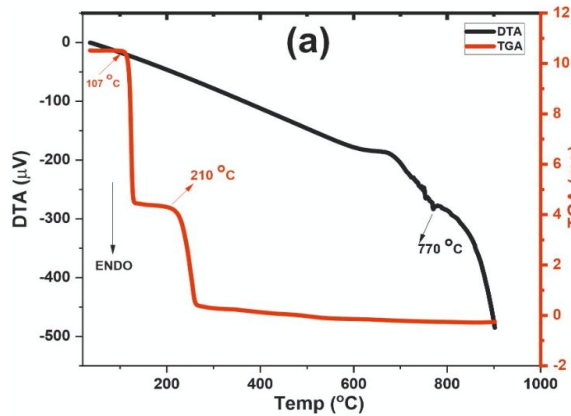


Fig.5a Pure LPTCA

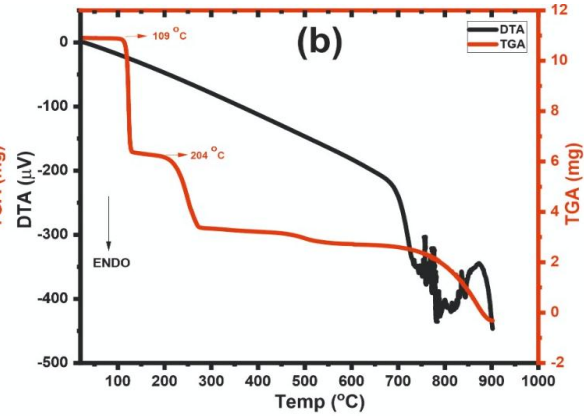


Fig.5b K doped LPTCA

V. CONCLUSION

In the present work single crystal of pure and K-LPTCA were grown by slow evaporation solution growth method. The functional groups of grown crystals were qualitatively identified by FTIR spectral analysis. K metal has been detected by EDS analysis. UV–Visible–NIR spectra revealed that transmittance of LPTCA crystal has been enhanced by 9% due to incorporation of K expressing the improved optical quality of K-LPTCA crystal. Thermal stability of the grown sample was studied by TG/DTA analysis. TG analysis revealed that LPTCA and K-LPTCA crystal is thermally stable up to 107 °C and 109 °C respectively. Conclusion derived in the present work evidently show that K⁺ doped LPTCA crystal is potential candidate as compared to LPTCA single crystals for device applications

VI. ACKNOWLEDGMENT

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REFERENCES

- [1]. Investigation on growth, structural, optical, thermal, dielectric and mechanical properties of organic L-prolinium trichloroacetate single crystals: K. Boopathi et al., Materials Research Bulletin 47 (2012) 2299–2305
- [2]. Structural, optical, mechanical and dielectric studies of pure and doped L-Prolinium Trichloroacetate single crystals: N. Renuka et al. / Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 137 (2015) 601–606.
- [3]. In-depth behavioral study of l-Prolinium Trichloroacetate single crystal: An efficient candidate for NLO applications: Kanika Thakral et al/ Arabian Journal of Chemistry (2019) 12, 4887–4896.
- [4]. The study of impurities effect on the growth and nucleation kinetics of potassium dihydrogen phosphate: J. Podder, Journal of Crystal Growth, Volumes 237–239, Part 1, April 2002, Pages 70-75
- [5]. Molecular structure, vibrational, factor group, optical and second order polarizability analysis of the l-prolinium trichloroacetate: A computational approach: Mohd. Shkir et al. / Optik 136 (2017) 327–335
- [6]. Synthesis, growth and characterization of L-prolinium trichloroacetate single crystal for nonlinear optical applications: D. Kalaiselvi, R. Jayavel, OPTOELECTRONICS AND ADVANCED MATERIALS – RAPID COMMUNICATIONS Vol. 4, No. 9, September 2010, p. 1400 – 1403

- [7]. Solid State Parameters, Structure Elucidation, High Resolution X-Ray Diffraction (HRXRD), Phase Matching, Thermal and Impedance analysis on L-Proline trichloroacetate (L-PTCA) NLO Single Crystals: P Kalaiselvi et al, Spectrochim Acta A Mol Biomol Spectrosc . 2014 Nov 11;132:726-32
- [8]. Synthesis, Characterization and biological activity of pure and Metal ions doped L-Proline Amino acid: V.Renuga, International Journal of Scientific and Research Publications, Volume 4, Issue 7, July 2014 ISSN 2250-3153
- [9]. L-Proline picrate and 2-methylpyridinium picrate: K.Anitha et al/Acta Cryst. (2006). C62, 567-570
- [10]. Synthesis, Crystal Growth, Structural, Optical, Thermal and Mechanical Properties of Semiorganic Nonlinear Optical Material: L-Cystine Dihydrochloride: Journal of Minerals and Materials Characterization and Engineering, Vol.9 No.5