

# Synthesis, Growth and Characterization of Undoped L-Lysine with Zinc Acetate Single Crystal for NLO Application

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**Abstract-** Semiorganic nonlinear optical material L-Lysine with zinc acetate was synthesized at room temperature in single crystal was grown by slow evaporation technique. The grown crystal was confirmed by Powder X-ray diffraction analysis and the major peaks were indexed. The single x-ray diffraction analysis reveals that the crystal belongs to monoclinic system. FTIR vibrational spectral study was used to identify the functional groups present in the grown crystal. The optical transparency of the grown crystal material was investigated by UV-Visible Spectrum. The Vickers Microhardness studies reveal the mechanical strength of the grown crystal. The L-Lysine zinc acetate was studied to be thermal stability of the grown crystal TGA and DSC. The SHG (Second harmonic generation) of the crystal was tested by Kurtz-Perry Powder method.

**KeyWords:** L-Lysine, Zinc acetate, FTIR, UV-Visible, Vickers hardness, Single Crystal XRD, Powder Crystal XRD, TG-DSC, NLO Studies.

## I. INTRODUCTION

Nonlinear optical studies with organic, inorganic molecules are called nonlinear optics. The NLO crystals with high conversion efficiency for second harmonic generation (SHG) and transparent of ultraviolet ranges for various devices in the field of opto electronic application [1-3]. In the field of nonlinear optical crystal growth, amino acids play a vital role. Amino acids based organic crystals are interesting materials for NLO applications due to the fact that all the amino acids have molecular chirality, wide transparency ranges in the visible and uv spectral region and zwitterionic nature of the molecule[4]. Complexes of amino acids with inorganic salts are promising materials for optical second harmonic generation (SHG) as they tend to combine the advantages of organic amino acids and inorganic salt. Organic materials are often formed by weak van der Waals and hydrogen bonds and hence possess a high degree delocalization [5]. The semiorganic NLO material has a significance on laser technology for optical communication and device fabrication. The amino acid is organic material for NLO applications to be present of (COOH) group in the materials for ferroelectric properties [6-8]. The L-Lysine possesses hydrogen atom which offer change in transfer to form semiorganic crystal for Nonlinear application. The literature was analyzed for anisotropic studies of L-alanine, L-arginine, glycine based on amino acid group of materials to be synthesized [11-13]. The L-Lysine with Zinc Acetate was

single crystal by slow evaporation technique. The grown crystal study reports detailed evaluation of optical and structural, mechanical, SHG application and Thermal studies for single crystal [14-16].

## II. EXPERIMENTAL

### A. SYNTHESIS AND CRYSTAL GROWTH

Single crystal of were grown from L-Lysine with Zinc acetate was synthesized by dissolving in high purity AR grade in room temperature by 1:3 molar ratio in deionized water. The solution was stirred by magnetic stirrer in the room temperature that was maintained at 45°C to avoid decomposition.



Fig. 1. The photograph of grown crystal for L-lysine Zinc acetate

In single crystals of L-Lysine Zinc acetate at 34°C were grown by slow evaporation method. The grown crystal size of 16x7x3 mm<sup>3</sup> was harvested in 25 days a single crystal with good transparency in the L-Lysine Zinc acetate as shown in figure1.

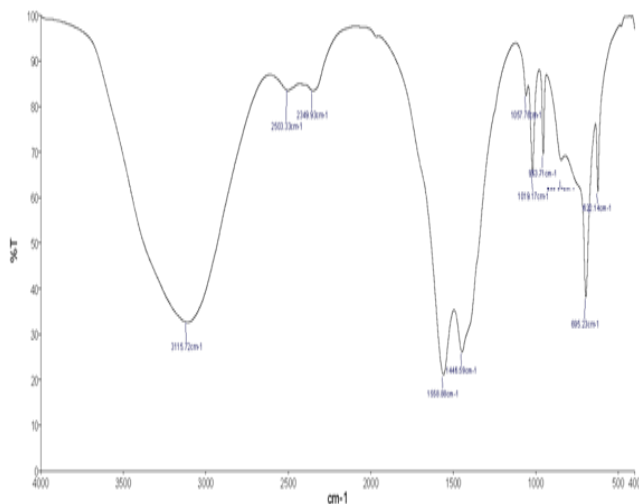
**III.RESULT AND DISCUSSION**

**A. FTIR SPECTRUM ANALYSIS**

The FTIR Spectrum of L-Lysine with Zinc acetate was recorded in the KBr phase in the frequency region 450cm<sup>-1</sup> – 4000cm<sup>-1</sup> using Perkin Elmer spectrometer is shown in fig2. The assignments of various functional groups are given table 1. The stretching frequency at 3115cm<sup>-1</sup> shows the presence of C-H stretching and 2349cm<sup>-1</sup> NH<sub>2</sub> symmetric stretching. In multiple fine structures at the lower energy mode indicate the strong hydrogen bonding of NH<sub>3</sub><sup>+</sup> groups. The strongest band observed at 1558cm<sup>-1</sup> indicates the presence of C=C stretching in the carbonyl group. The presence of strong peak ranges from 1446cm<sup>-1</sup> to 1019cm<sup>-1</sup> for the C-N stretching of carbonyl group. In the lower wave number region, the bands at 953cm<sup>-1</sup> and 695cm<sup>-1</sup> are due to the ring asymmetric, symmetric stretching and plane deformation.

**Table 1: FTIR Spectra for L-Lysine Zinc Acetate**

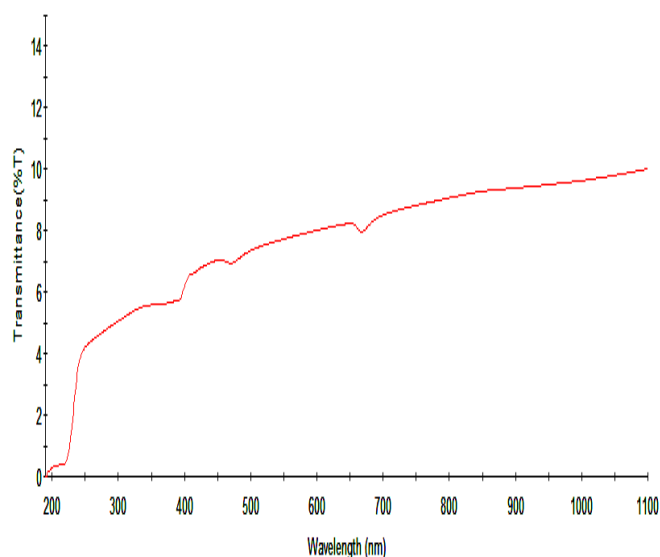
Wave Number (cm <sup>-1</sup> )	Assignments
3115	C-H Stretching
2349	NH <sub>2</sub> Symmetric Stretching
1558	C=C Stretching
1446	CN Stretching
1019	C-N Symmetric Stretching
953	C-H Symmetric Stretching
695	O-H Carbonyl Group



**Figure 2: FTIR Spectrum of L-Lysine Zinc Acetate.**

**B. UV-VISIBLE STUDIES**

The UV-Visible spectral study of L-Lysine with Zinc acetate was carried out in the range of 200-1200nm were recorded transmittance is shown in fig3. The grown crystal exhibits high transmittance above 85% of entire visible region in which the crystal is enabled to be a good material for second harmonic generation from Nd:YAG laser and optoelectronic applications. A sharp peak in absorption is observed around 230nm and this corresponds to the lower cut-off wavelength that is fundamental absorption of the crystal. The optical transparency of L-Lysine with Zinc acetate and hence in the presence of grown crystal.



**Figure 3: UV-Visible Spectrum of L-Lysine Zinc Acetate.**

**C.VICKERS HARDNESS TEST**

The measurement of hardness determines the resistance deformation. In the use of Microhardness study that the carried out on the grown crystal to across the mechanical property the static indentations were made at room temperature with constant time of 10s for the all indentations. The indentation marks were made on the surfaces by varying the load from 25g to 100g. The Vickers hardness number (H<sub>v</sub>) of the L-Lysine with Zinc acetate crystal was calculated using the relation,  $H_v = 1.8544P/d^2$  Kg/mm<sup>2</sup> Where, P is the applied in Kg, and d is the average diagonal length of the indentation in mm. A graph plotted between H<sub>v</sub> versus applied load (P) is shown in fig4. The H<sub>v</sub> increases as the applied load (P) for the grown crystal of beyond the 100g in the significant crack occurs to which may be due to internal stress generated with indentation. The work hardening coefficient (n) for thee grown crystal was softer material category in the L-Lysine with Zinc acetate single crystal.

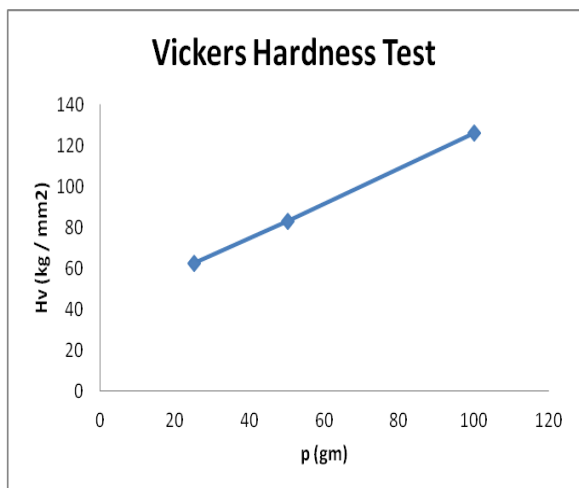


Figure 4: Vickers hardness studies of L-Lysine Zinc Acetate.

**D.X-RAY DIFFRACTION TECHNIQUE**

**1. POWDER CRYSTAL XRD**

The diffraction pattern of the grown crystal was subjected to powder X-Ray diffraction analysis using analytical XPERT PRO powder X-ray diffraction employing CuK $\alpha$  radiation ( $\lambda=1.5405\text{\AA}$ ). The XRD pattern of L-lysine zinc acetate crystal is shown in fig 5. The observed of sharp peaks in spectrum indicated the resemblance of grown crystal to monoclinic structure. The grain size was calculated following Scherrer's formula,

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Where, D is the Crystallite size of the crystal. K is the constant depending on crystalline shape (0.9)  $\lambda$  is the wavelength of X-Ray.  $\beta$  is the full width half maximum and  $\theta$  is the Bragg angle of X-Ray diffraction. The crystalline size was calculated as 72.13nm in the L-Lysine Zinc acetate grown crystal.

**2. SINGLE CRYSTAL XRD**

Single Crystal X-ray diffraction analysis of Pure L-lysine with zinc acetate was recorded using ENRAF NONIUS CAD-4 automatic X-ray diffraction. This analysis reveals that the pure of L-Lysine zinc acetate crystallizes in monoclinic system with space group P<sub>21</sub>. The calculated lattice parameters of the pure L-lysine with zinc acetate crystal are a=7.281 $\text{\AA}$ , b=8.410  $\text{\AA}$ , c=12.898  $\text{\AA}$ ,  $\alpha=\beta=\gamma=90^\circ$  and volume V= 1154.115  $\text{\AA}^3$  Thus, the XRD results confirm that there is no change in crystals structure, but there are small changes in the lattice parameters. The change in the

lattice parameter may due to incorporation of L-lysine with Zinc acetate.

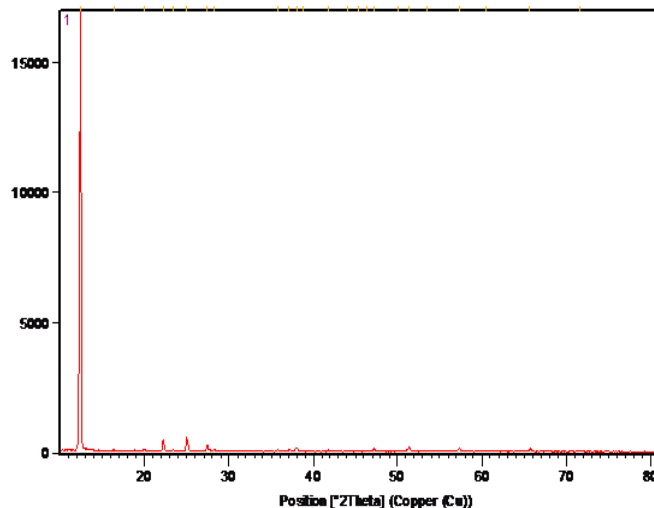


Figure 5: Powder X-Ray Diffraction Pattern of L-Lysine Zinc Acetate.

**E.THERMAL ANALYSIS**

**1.TG-DSC STUDIES**

The thermal stability changes of L-Lysine with Zinc acetate single crystal were studied by thermo gravimetric analysis and differential scanning calorimetric analysis. The curve of L-Lysine with Zinc acetate is shown in fig.6. TG/DSC. The thermal decomposition starts at 50°C to 900°C the acetate complexes with inorganic leads the decomposition of the Zinc acetate and metal oxides. Due to the addition of L-lysine with Zinc acetate the thermal stability is increased by nearly 5 °C. The thermal decomposition of L-Lysine with Zinc acetate is exothermic peak at 150°C in the DSC analysis which shows the melting point of the complex on the 350°C and the allythiourea metal halides to be thermal stability of the L-Lysine with Zinc acetate good quality in single crystal.

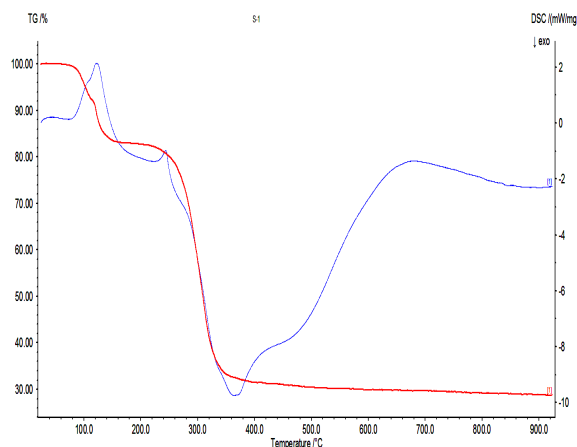


Figure 6: Thermal analysis TG-DSC of L-Lysine Zinc Acetate.

### F. NONLINEAR OPTICAL STUDY

The SHG efficiency measurements have been carried out using Kurtz and Perry technique. A Q-switched Nd:YAG laser beam of wavelength 1064 nm with input beam energy of 1.5mJ to 3mJ and pulse width 6 ns with a repetition rate of 10 Hz was used. The grown crystals of L-lysine with Zinc acetate crystal was powdered with uniform particle size and tightly packed in a microcapillary of uniform bore and exposed to the laser radiation. The bright green light ( $\lambda = 532$  nm) emission has been observed which indicates the SHG behaviour of the grown crystals. The relative SHG efficiency of pure L-Lysine with Zinc acetate (850mJ) is nearly 1.4 times that of KDP respectively.

### IV. CONCLUSION

The Pure L-Lysine With Zinc acetate single crystal was grown by solution growth method at room temperature. The single crystal analysis of lattice parameter and Space group is to be calculated. The powder X-Ray Diffraction was indexed of monoclinic system. The FTIR was confirmed to be analysis of functional group of grown crystal. UV-visible to be analysis of the transmittance is found to be maximum in the visible near infrared region in Pure BTZA crystal at the lower cutoff wavelength region in 230nm. The Vicker's microhardness was calculated in order to understand the mechanical stability of the grown crystals. Hardness measurement also shows that harder than pure L-Lysine with zinc acetate crystals. The TGA/DSC analysis of allythiourea metal halides thermal stability in the pure L-Lysine with Zinc acetate crystal. The thermal decomposition of L-Lysine zinc acetate is exothermic peak at 150°C in the DSC analysis that shows the melting point of the complex compared to other than metal based complex 350°C and the metal halides to be thermal stability of the pure good quality in single crystal. The SHG was observed using a Q-switched Nd:YAG laser and its efficiency were found to be better than KDP. In Pure L-Lysine zinc acetate single crystal, a grown crystal belongs to the potential for ferroelectric material applications in future.

### REFERENCES

- [1] R.N. Shaikh, Mohd.Anis, M.D. Shirsat and S.S. Hussaini IOSR Journal of Applied Physics (IOSR-JAP) e-ISSN: 2278-4861. Volume 6, Issue 1 Ver. I (Jan. 2014), PP 42-46
- [2] R. Sumalatha, C. Babu, G. Srinivasan, G. Suresh, International Journal of Engineering Science Invention ISSN (Online): 2319 – 6734, ISSN (Print): 2319 – 6726
- [3] V Vasudevan, R Ramesh Babu, A Reicher Nelcy, G Bhagavannarayana and K Ramamurthi Bull. Mater. Sci., Vol. 34, No. 3, June 2011, pp. 469–475. Indian Academy of Sciences.
- [4] Suresh Sagadevan1, Priya Murugasen International Journal of Recent advances in Physics (IJRAP) Vol.3, No.1, February 2014.
- [5] K. Rajesh. A. Mani, and P. Praveen Kumar International Journal of Engineering Science Invention ISSN (Online): 2319 – 6734, ISSN (Print): 2319 – 6726

- [6] A.Venkatesan, S. Arulmani, S. Senthil, M. E. Rajasaravanan IJEDR Volume 5, Issue 1 ISSN: 2321-9939, (2017)
- [7] J. Thomas Joseph Prakash M. Lawrence International Journal of Computer Applications (0975 – 8887) Volume 8– No.3, October 2010
- [8] T. Uma Devi, N. Lawrence, R. Ramesh Babu, S. Selvanayagam, Helen Stoeckli-Evans, and K. Ramamurthi Crystal Growth & Design, Vol. 9, No. 3, 2009
- [9] R. Tamilselvi, K. Arunadevi, D. Sivavishnu, P. Ramadoss International Journal of Scientific Reports Tamilselvi R et al. Int J Sci Rep. 2017 Apr;3(4):100-105
- [10] N. Kanagathara and G. Anbalagan Hindawi Publishing Corporation International Journal of Optics Volume 2012, Article ID 826763, doi:10.1155/2012/826763
- [11] V.Kathiravan, M.Rajalingam, M.Narmathaa, P.Sasikala IOSR Journal of Applied Physics (IOSR-JAP) e-ISSN: 2278-4861. Volume 9, Issue 6 (Nov. - Dec. 2017), PP 09-16.
- [12] Koteeswari Pandurangan and Sagadevan Suresh Hindawi Publishing Corporation Journal of Materials Volume 2014, Article ID 362678
- [13] S. Chennakrishnan, S.M. Ravikumar, D. Sivavishnu, M. Packiyaraj, S.Varalakshmi Mechanics, Materials Science & Engineering, March 2017 – ISSN 2412-5954
- [14] K.Manimekalai, R.Rajasekaran International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056 Volume: 04 Issue: 07 July -2017
- [15] J. Martin Sam Gnanaraj, M. Iniya Pratheepa, M. Lawrence, Optics & Laser Technology Volume 107, November 2018, Pages 478-483
- [16] A.Sathya, P.Yasotha, P.Sagunthala International Journal of Chemical and Physical Sciences, ISSN:2319-6602 IJCPS Vol. 4 Special Issue-6 APST–2015.