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Growth and Characterization of NLO Single Crystal: BIS L-Alanine Potassium Chloride (BIS-LAKCL)

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Abstract: Nowadays the nonlinear optical crystals have greater attention in the field of optoelectronics. Single crystal of Bis L-Alanine Potassium Chloride was synthesized by solution growth method and good optical quality crystal was grown by slow evaporation solution growth technique (SESGT). X-ray diffraction analyses were reveals that the crystal system is triclinic and it crystalline nature good based on the very sharp single peak. The functional groups of the grown crystals were identified and confirmed by the Fourier Transform Infrared Spectrum (FTIR) analysis. From the UV-Vis spectroscopy analysis shows that the grown crystal has good optical transmission in the range of 238 nm to 1100 nm. Thermal and Mechanical property of Bis LAKCl have been studied by using the conventional method and Vicker's microhardness test respectively. The relative SHG efficiency of the Bis LAKCl crystal was found by Kurtz-Perry Powder technique.
Keywords: Single crystal XRD, UV-Vis spectrum, FTIR, Vicker's Microhardness.

1. Introduction

In this technological world nonlinear optical material (NLO) plays a vital role in the field of photonics and optoelectronics. Semi organic crystals with good NLO property make them attractive for the applications of optical communication, optical processing, frequency conversion and optical processing. Recently greater attention devoted by the researchers on the growth of amino acid mixed organic crystals due to their greater merits in the view optical applications compare to other materials [1]. In NLO applications the amino acid crystals has greater importance due their chiral symmetry and crystallize in noncentrosymmetric space groups. Amino acid crystals have very good structure for the NLO applications such as proton donating and proton accepting group. Many amino acid crystals were reported with good NLO property i.e., L-alanine cadmium chloride [2], L-valine cadmium chloride [3]. This manuscript defines the crystal structure of Bis L-alalnine Potassium Chloride (Bis LAKCl) and the crystalline nature is studied by the powder X-ray diffraction. Functional groups have been identified by the FTIR studies. The optical absorption is studied by the UV-Vis spectrophotometer, mechanical strength was evaluated by the Vicker's microhardness tests.

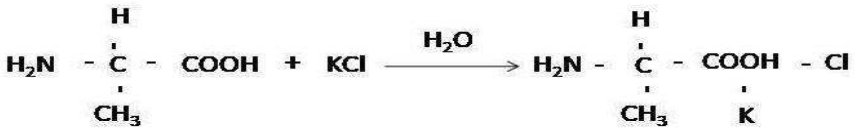
2. Experimental

2.1. Material Synthesis and Crystal Growth

The materials L-alanine and potassium chloride were taken in the ratio of 2:1. Based on this ratio the Bis L-alanine Potassium Chloride (Bis LAKCl) supersaturated solution was prepared with the pH value of 5.8 [4]. Solution was filtered using the Whatman filter paper and transferred to Petri dish. Recrystallization process was carried out for 5 times to reduce the impurities in the compound (Bis LAKCl). Final solution was poured in the Petri dish covered with small holes plastic sheet, tied on top with rubber band to control the evaporation.

The good optical quality transparent crystal has been grown by the slow evaporation solution growth technique (SESGT) with the dimensions 19 x 8 x 3 mm^3.

The chemical reaction of synthesis process in given below-



(Bis L-alanine) + Potassium Chloride ⟶ Bis L-alanine potassium chloride

Fig 3.1 Chemical reaction

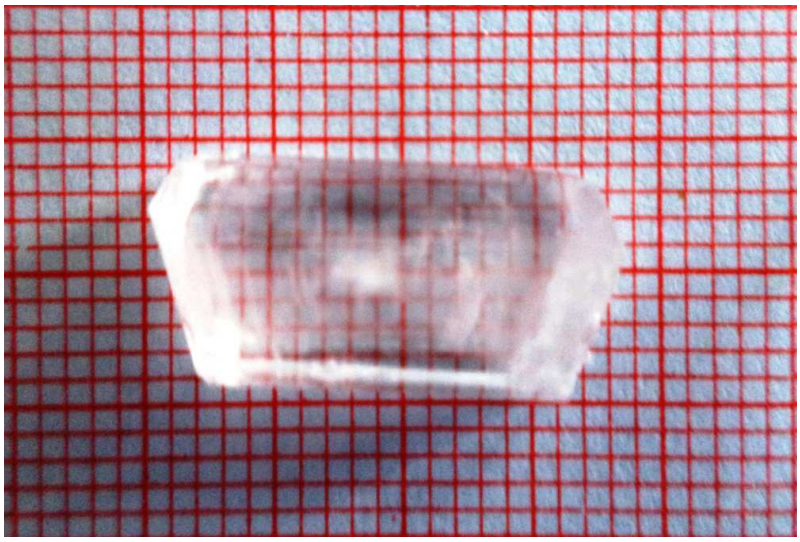


Fig 3.2.Grown Bis LAKCl single crystal

2.2. Characterization Studies

The crystal was characterized by the single crystal and powder XRD, FTIR, UV-Visible Spectroscopy, Microhardness studies, Thermal studies and Kurtz-Perry powder SHG technique. The single crystal X-ray diffraction analysis was carried out using the ENRAF NONIUS CAD4 diffractometer to determine the unit cell parameters of the grown crystal. Powder X-ray diffraction analysis was carried out using a D8 ADVANCE BRUKER with CuKa (λ=1.5418 Å). Using the PERKIN ELMER RX1 Fourier Transform Infrared spectrometer the spectrum was recorded using KBr pellet technique in the frequency range of 400-4000 cm^{-1}. The UV Vis absorption spectrum was recorded using carry 5000 scan UV-Vis-NIR spectrometer [Varian] for Bis LAKCl crystal.

2.3. Results and Discussion

2.4. X-RAY Diffraction Analyses

2.4.1. Single Crystal X-RAY Diffraction Analysis

Single crystal XRD reveals the unit cell parameters of the grown good transparent nonlinear optical single crystal. The cell parameters of the grown crystal show that the crystal belongs to triclinic. The parameters were given in the table.4.1. The cell parameters almost similar with reported literature about L-KCl [2].

Table 4.1. The unit cell parameters of the Bis LAKCl crystal

BIS L-Alanine Potassium Chloride	Crystal Data
Lattice Parameters	a = 10. 6453Å b = 16.0435Å c = 5.3489 Å $\alpha = 96.6299^o$, $\beta = 98.6734^o$, $\gamma = 95.7711^o$.
Cell Volume (V)	891.8159 $Å^3$

2.4.2. Powder XRD Analysis

The powder XRD of Bis L-alanine potassium chloride is shown in Fig.4.1. Finely crushed powder of BIS LAKCL crystal was subjected to powder X-ray diffraction analysis using a D8 ADVANCE BRUKER with CuKa (λ=1.5418 Å) radiation. The sample was scanned over the range of 10 to 80 degree at a scan rate of 0.02°/minute. The X-ray diffraction pattern of BIS LAKCL was recorded and indexed.

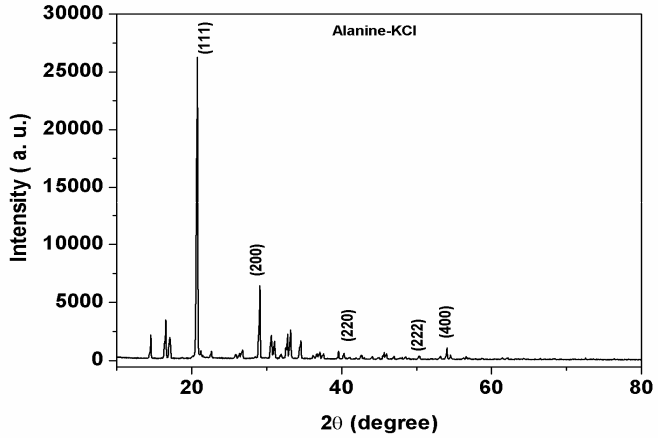


Fig.4.1.Powder XRD pattern of the Bis LAKCl

2.5. FTIR Analysis

Using the PERKIN ELMER RX1 Fourier Transform Infrared spectrometer functional group of the Bis LAKCl crystal was identified. The spectrum was recorded using KBr pellet technique in the frequency range of 400-4000 cm^{-1} [4] and is shown in fig 4.2. The confirmed functional were listed in the table.4.2.

Table 4.2. Functional groups of Bis LAKCL single crystals

Wavenumber (cm^{-1})	Assignments
3087.05	C-H asymmetric vibration
1512.27	C=O stretching
1457.16	C-H in plane bending
1360.27	C-H bending
1234.52	C-O-C stretching
1151.82	C-O-C stretching
1110.03	Symmetric C- O-C Stretching
1012.20	C-CHO stretching
919.47	O-H bending
847.64	C-H out of plane bending
772.49	C-H out of plane bending
648.39	C-H bending
539.76	O-C-N deformation

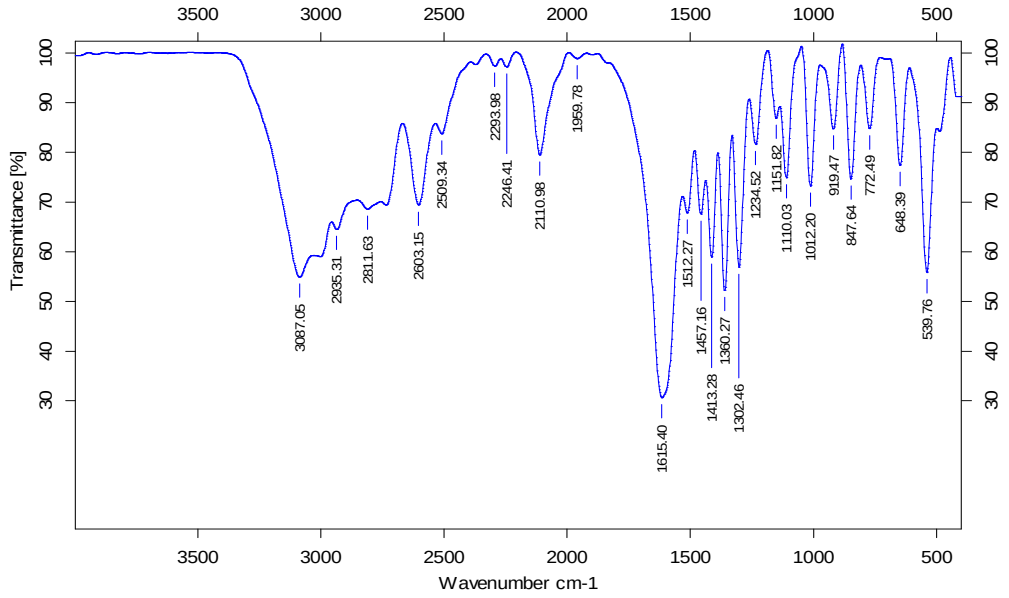


Fig.4.2 FTIR spectrum of Bis LAKCl crystal

2.6. UV-Visible Spectral Analysis

The optical absorption spectrum of Bis LAKCl was observed in the range of 190-1100 nm. The material has good transparency in the visible region and it is the important feature for nonlinear optical crystals. The absorption was drastically reduced from the 201nm to 238nm. After the wavelength of 238 nm the optical absorption is completely nullified and the crystal has highly transparent in the range of 238 nm to 1100 nm. L-alanine potassium chloride (LPC) has the similar cutoff wavelength with minimum variation [4]. The absorption spectrum is shown in fig.4.3.

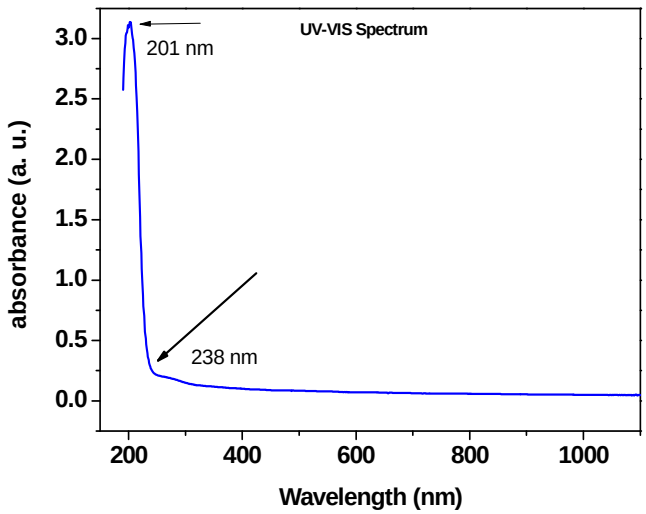


Fig.4.3 UV absorption spectrum of Bis LAKCl

2.7. Thermal Analysis

Melting point of the material was evaluated by the conventional melting point method. FEC meltin point apparatus was used for this analysis.Finely powdered sample was filled in the capillary tube size is 6 cm. the Bis LAKCl was filled in the 3cm level. The capillary tube was kept inside the apparatus in the visible position. Through the lens we can clearly seeing the capillary tube with material. The temperature was increased, when the temperature was reached 140°C the materials colour was changed to brown and finally the material completely melted at 145°C [5].

2.8. Microhardness Test

Bis LAKCl crystal was subjected to Vickers micro hardness test to find the mechanical strength of the grown crystal. The mechanical strength can define the capability of the grown crystal for the suitable industrial applications. The microhardness test was carried out with the load varying from 25 to 100g. Vicker's hardness number of the crystal calculated using the relation [6]

$$H_V=1.8544\ p/d^2$$

The hardness number is decreasing while the load is increasing. From the graph the material has good agreement with Indentation Size Effect (ISE) [7]. The variation of hardness with load is shown in Fig.4.4.

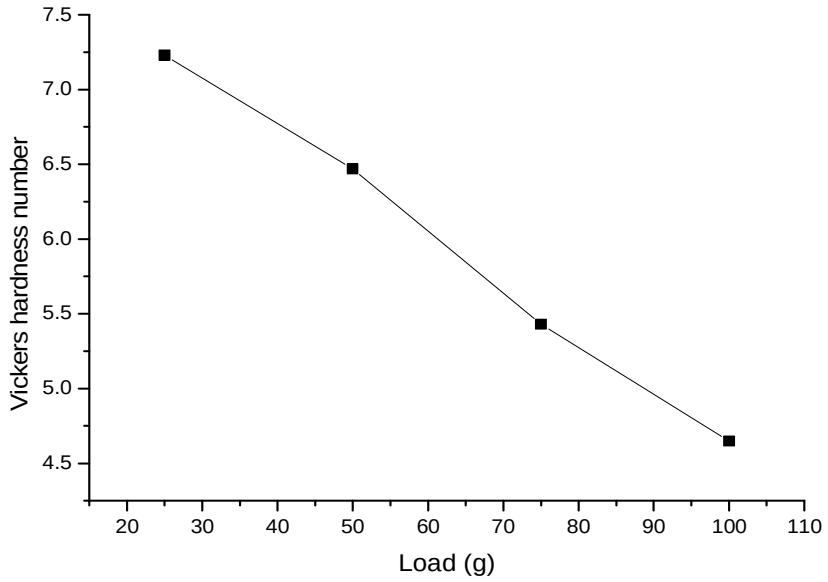


Fig.4.4 Vicker's hardness number with load

2.9. NLO Studies

NLO property of Bis LAKCl crystal was confirmed by Kurtz-Perry powder technique. The good optical quality crystal has been powdered finely with particle size 100- 125 µm and filled in the capillary tube 1.5 mm. The Q-switched Nd:YAG laser emitting a fundamental wavelength of 1064 nm with pulse width 8 ns high intense light falls on the sample [8]. From the output we found that the relative SHG efficiency of Bis LAKCl is 4.2 mv with reference of KDP has 7.0 mv. The SHG property of the grown crystal was confirmed by the green emission of the sample [9].

3. Conclusion

Single crystal of Bis LAKCl was grown slow evaporation solution growth technique (SESGT) at room temperature. The cell parameters are evaluated by single crystal X-ray diffraction analysis. The functional of the grown crystal was confirmed by FTIR spectral analysis. Melting point was determined by conventional method also this material can withstand upto 145°C. Mechanical strength of the grown crystal was estimated by the Vicker's hardness test. From NLO study, it is conclude that the grown crystal has nonlinear optical property.

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