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Synthesis, crystal growth and structural characterization of lithium fumarate semi-organic single crystals

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ABSTRACT

Crystalline substance of Lithium fumarate was synthesized. Single crystals are grown using slow evaporation solution growth method. The unit cell parameters of the grown crystals are determined by single crystal X-ray diffraction. The UV-vis-NIR transmittance spectrum was recorded in the range 200-1100 nm to determine the cut off wavelength. Thermogravimetric/ Differential thermal analysis are carried out on the grown crystals to study its thermal behavior. The mechanical stability of the crystal was analyzed by Vickers micro hardness studies.

KEY WORDS: Crystal Structure, Crystal Growth, X-ray techniques, Thermal Properties, Optic Materials.

1. INTRODUCTION

Materials with large optical nonlinear properties are of great interest for applications such as frequency conversion, telecommunication, optical computing, optical information processing and high optical disk data storage. Investigation of semi-organics is led by the need for materials which combine large nonlinear optical characteristics with resistance to physical and chemical attack. The advantages of semi- organic materials are that they can be grown from aqueous solution and form large three-dimensional crystals. In the search for such semiorganics, Fumaric acid draws more attention due to its tendency to form cyclic motifs linked by intermolecular hydrogen bonds. This paper reports the synthesis, crystal growth and characterization studies of lithium fumarate (LF) single crystal.

2. EXPERIMENTAL

2.1. Synthesis, Growth and Characterization: Crystalline substance of LF was synthesized using analar grade lithium hydroxide and fumaric acid in a stoichiometric ratio of 1:1. The calculated amount of the lithium hydroxide was dissolved in the deionized water. Fumaric acid was slowly added to the reaction. The resultant mixture was stirred well for six hours. The prepared solution was filtered. The filtered solution was kept undisturbed in an environment conducive for single crystal growth. Photograph of as grown non hygroscopic LF crystal is shown in figure. 1.

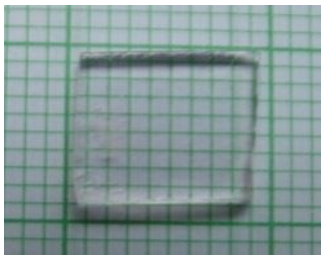

Figure.1. As grown LF Crystals

3. RESULTS

The grown crystals are subjected to single crystal X-ray diffraction studies in which the measurements was carried out at 293K using Bruker AXS Kappa Apex II CCD diffractometer. LF crystal belongs to the monoclinic system with the lattice parameters a= 8.5440(6) Å, b= 8.3608(5) Å, c = 7.8289(6) Å, α = 90˚, β=113.4(2) ˚ and γ= 90˚.

The UV-vis-NIR spectrum is studied in the wavelength range 200-1100 nm. Studies were carried out without any antireflection coatings on 1.5 mm thick LF sample. The lower cut off wavelength is 290 nm which is shown in figure.2. The grown crystal has wide transmission in the entire range and the optical band gap was found to be 4.24 eV.

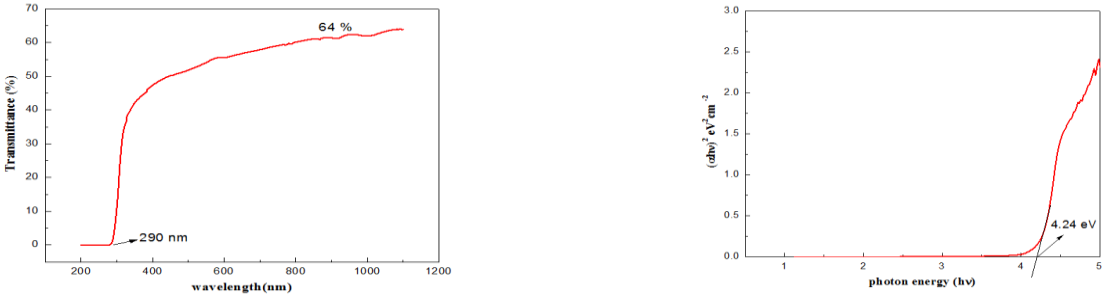

Figure.2.Transmittance and optical bandgap spectrum of LF crystals

The thermal behavior of LF was studied using thermogravimetric (TG) and Differential thermal analyses (DTA) in the temperature range 35-450˚C. An endothermic peak is observed at 271˚C in DTA curve which is accompanied by the weight loss in TG curve. This is assigned as the melting point of LF. The second endotherm is observed at 316˚C. Micro hardness measurement was carried out to determine the mechanical stability of LF. Load ranging from 1g to 50g was applied on LF and observed that H_v increases with the load which is reverse indentation size effect.

4. CONCLUSION

Single crystals of lithium fumarate is successfully grown using slow evaporation method. LF belongs to monoclinic system. The cut off wavelength is 290nm. LF is thermally stable up to 271˚C. Reverse indentation size effect is observed in LF.

REFERENCES

Ledoux I, New advances in molecular engineering for quadratic nonlinear optics, Synthetic Metals, 54 (1-3), 1993, 123 – 127.

Yuan DR, Xu D, Zhang N, Liu MG, Jiang MH, Organic Nonlinear-Optical Crystal MHBA For Compact Blue-Violet Laser, Chinese Physics Letters, 13 (11), 1996, 841 – 843.