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Growth, Structural, Optical and Mechanical Properties of Oxalic Acid Single Crystals

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***Abstract:*-- Single crystals of oxalic acid were grown by slow evaporation method. The grown crystals have been subjected to single and powder X-ray diffraction, FTIR, UV-Visible and micro hardness studies. It is found that oxalic acid single crystals crystallizes in monoclinic crystal system and the powder X-ray diffraction analysis was done to verify the lattice parameters and crystalline nature of the crystals. The vibrational frequencies of various functional groups present in the sample have been derived from FTIR analysis. The percentage of transmittance in visible region of the crystal was recorded using the UV-Visible Spectrophotometer. The mechanical strength of the crystal was found out using Vickers micro hardness test.**

***Keywords:*-- Organic crystals, X-ray diffraction, FTIR analysis, Mechanical properties.**

1. INTRODUCTION

In recent years, organic materials are attracting a great deal of attention for possible use in optical devices because of their large optical nonlinearity, low cut-off wavelengths, short response time and high laser damage thresholds. Organic single crystals are aromatic hydrocarbon compounds which contain benzene ring structure composed of carbon and hydrogen atoms. They can be used for fast neutron scintillations in combination with photomultiplier tubes, silicon devices. A Carboxylic acid is an organic compound that contains a carboxyl group. Carboxyl group (COOH) is a functional group consisting of a carbonyl group(C=O) with a hydroxyl group (O-H) attached to the same carbon atom. Oxalic acid is an organic compound with the formula $C_2H_2O_4$. It is a colourless crystalline solid that forms a colourless solution in water. Oxalic acid is a hydrogen-bonded material. It is the only possible compound in which two carboxyl groups are joined directly and for this reason oxalic acid is one of the strongest acids in organic compounds. A. N. Winchell et al, reported the anhydrous oxalic acid is dimorphous [1]. The alpha-phase (I) is orthorhombic, space group Pcab and crystals are dipyramidal in shape forms. The beta-phase (II) is monoclinic with space group P21/c, and tends to have a prismatic habit. Oxalic acid dihydrate is monoclinic with space group P21/n [2, 3]. Race C. Moulton et al, grown the Single crystals of oxalic acid dihydrate by using slow evaporation method. The dihydrate is monoclinic,. a=6.119 Å, b=3.640 Å, c= 12.051 Å and space-group P21/n [4]. Structure of oxalic acid dihydrate and modification of deuterated oxalic acid dihydrate was studied using XRD analysis [5-8]. The structure of N-Methyl urea-Oxalic acid (2:1) reported by S. Harkema et al [9]. Y. Wang et al, reported the temperature-dependence studies of alpha-oxalic acid dihydrate at five different temperature [10]. A number of novel amino acids mixed with nonlinear organic crystals have L-alaninium oxalate [11], L-prolinium tartarate [12], and L-alaninium succinate [13], undoped and Mn doped glycinium oxalate and oxalic acid doped with tryptophan crystals were studied [14-16]. In the present investigation, an attempt has been made to grow an optical quality oxalic acid dihydrate single crystal by low temperature solutions growth technique. In addition, FT-IR, XRD and optical studies and mechanical hardness have been performed in details.

2. EXPERIMENTAL PROCEDURE

Oxalic acid sample was mixed in double distilled water and stirred well using magnetic stirrer to get homogeneous solution about 3 hours. The solution was filtered using whatman filter paper and kept for slow evaporation growth process. After 30 few days transparent and colourless crystals were harvested. Grown crystals of oxalic acids are depicted in figure (1).

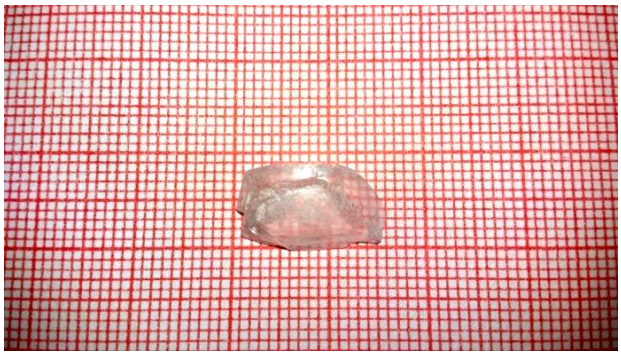

Fig.1. Grown crystal of Oxalic acid

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3. RESULT AND DISCUSSION

3.1. XRD Studies

A small portion of the single crystals of oxalic acid dihydrate was crushed and subjected to powder X-ray diffraction analysis. The narrow, sharp and high intensity peaks reveal that the grown crystals were of high degree of crystallinity. The lattice parameter values of oxalic acid was a = 6.114 Å, b = 3.587 Å, c = 12.011 Å and V=253.1 $Å^3$ Fig (2) shows the XRD pattern of the powder sample [13-16].

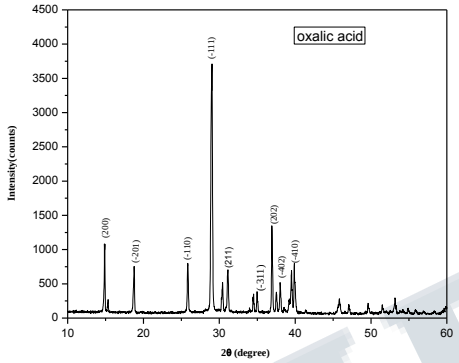

Fig.2. Powder XRD pattern of oxalic acid

3.2. FTIR Analysis

FTIR studies are important in the investigation of molecular structure of crystals. This study involves the stretching, bending, twisting and vibrational modes of atoms in a molecule and hence to identify the functional groups of samples. From the figure (3) it is observed that the CH_2 Asymmetric stretching vibrations of were observed at 3427 cm^{-1} and 2000 cm^{-1} [16]. The C=C stretching for vibration of were observed at 1689 and 1500 cm^{-1}. The peak observed at 1346 cm^{-1} was assigned to OH in plane bending. The C-O stretching vibration was observed at 1246 cm^{-1}. OH out of bending mode was observed at 767 cm^{-1} and 710 cm^{-1}.

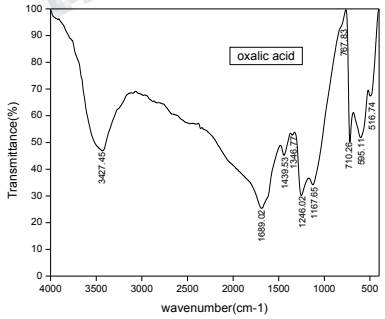

Fig.3. FTIR spectrum of oxalic acid

3.3. UV-Visible spectral studies

The optical transmission spectrum of oxalic acid powder sample was recorded by UV/VIS spectrophotometer in the range 190nm – 1100nm.The optical transmission spectrum of oxalic acid powder sample is shown in Figure (4, 5). Which shows that oxalic acid is optically transparent in the entire visible region and low cut-off wavelength at 245 nm. Between 430 and 1100 nm, there is no absorption of wavelength which is clearly indicates that grown crystals can be used as window material in optical instruments. The Forbidden energy band gap of the sample is 5.11 eV.

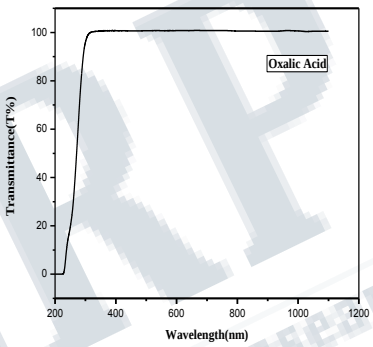

Fig.4. UV-Visible spectrum of oxalic acid

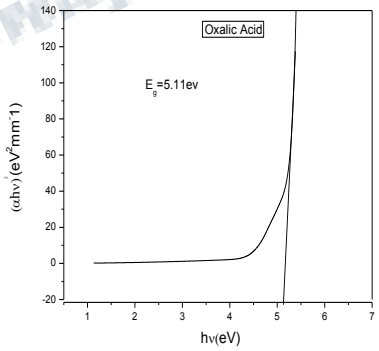

Fig.5. UV-visible spectrum of Tauc's plot

3.4. Microhardness studies

Hardness is one of the important mechanical properties to determine the plastic nature and strength of a material. The hardness number was calculated using the relation H_v = 1.8544(P/d^2) kg/mm^2. Where P is applied load (gm) and d is the diagonal length (μm) of the indentation. The plot between hardness number and load is shown in fig 6.It is observed that the hardness number increases with increase in load and it reveals that the oxalic acid crystal exhibits reverse

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indentation effect. The plot of log (P) against log (d) is almost a straight line [18].

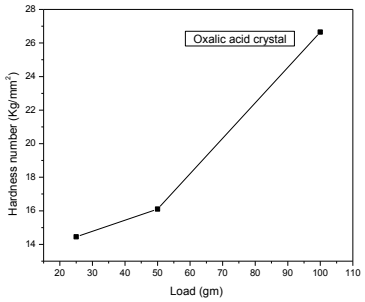

Fig.6. Plot of hardness vs load

4. CONCLUSION

Organic oxalic acid crystals were grown by using slow evaporation solution growth technique. From powder crystal XRD studies, the crystal structure is monoclinic, a = 6.114 Å, b = 3.587 Å, c = 12.011 Å and V = 253.1 (Å)^3. The various functional groups and the modes of vibrations were identified by FTIR spectroscopy. The UV-Visible transmittance spectrum gives a cutoff wavelength is 245 nm and energy gap value is 5.11eV. The hardness studies the oxalic acid crystal exhibits reverse indentation effect.

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