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Single Crystal of Pure Zinc Tartrate and Zinc-Cadmium Mixed Tartrate Grown By Gel Technique and Its Characterization

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ABSTRACT: The growth of pure zinc tartrate and zinc - cadmium mixed tartrate single crystals are successfully carried out in silica gel by single diffusion method. Gel method is used to grow this tartrate crystal because the gel medium provides effective alternative method to grow these crystals near ambient temperature. The optimum conditions were established by varying various parameters such as pH of gel solution, gel concentration, gel setting time, concentration of reactants etc. Crystal having different morphology were obtained, whitish semitransparent, star shaped, needle shaped crystal of zinc tartrate and zinc-cadmium mixed tartrate crystals were obtained. The crystal structure of the compound was confirmed by X-ray powder diffraction, FTIR, Energy Dispersive Analysis by X-ray (EDAX), and TGA/DTA.

KEYWORDS: Gel growth, Zinc, Cadmium Tartrate, XRD, EDAX, FTIR and TGA/DTA.

I. INTRODUCTION

The gel growth technique is one of the simplest techniques for the growth of crystals under ambient condition, which are sparingly soluble in water and decompose at low temperature [1]. However, attempts have been made to grow crystals, which are soluble in water, by the gel growth technique [2]. Since, gel growth technique has several advantages over few disadvantages; it has attracted the attention of several researchers. In the literature, many tartrate compounds are reported which have been grown by the gel technique; for example, lead tartrate [3], cadmium tartrate [4]-[5], iron tartrate [6], cobalt tartrate [7]-[9], mixed iron-manganese tartrate [10] and ternary iron-manganese-cobalt tartrate [11] as well as ternary iron-manganese-nickel tartrate [12]. Lead tartrate finds application as an additive in gasoline to increase the performance of the engine [13], where as cadmium tartrate is well known for its piezoelectric application [14]. Cadmium Tartrate crystals, $\text{CdC}_4\text{H}_4\text{O}_6 \cdot 5\text{H}_2\text{O}$, is structural with other eletroseramic divalent metal ions. Some divalent metal ions tartrates are interesting piezoelectric and ferroelectric compound exhibiting non-linear optical and spectral characteristics and hence are used in transducers and many linear and non-linear mechanical device Cadmium Tartratecrystals are significant. Cadmium Tartrate crystal is insoluable in water (0.004 gm/100 ml H_2O) and it decompose before melting and does not vaporize or sublime on hearing. The gel method is very attractive and simple technique to grow such type of materials at room temperature. In the present course of investigation it has been decided to grown pure zinc tartrate and zinc – cadmium mixed tartrate crystals by single diffusiongel technique. The grown crystals were characterized using EDAX, powder XRD, FTIR and TGA/DTA.

II. EXPERIMENTAL PROCEDURES

2.1 Crystal growth

The test tube diffusion method was employed to grow pure zinc tartrate and zinc – cadmium mixed tartrate crystals in a gel medium. 1 M sodium meta silicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$)was titrated with 1 M tartaric acid till the mixture attained the

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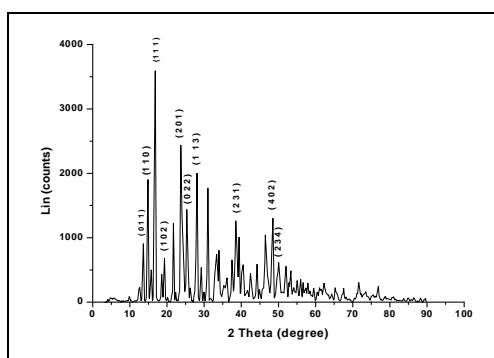
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III. RESULT AND DISCUSSION

3.1 X-ray Diffraction

Lattice Parameters of Pure Zinc Tartrate Crystal

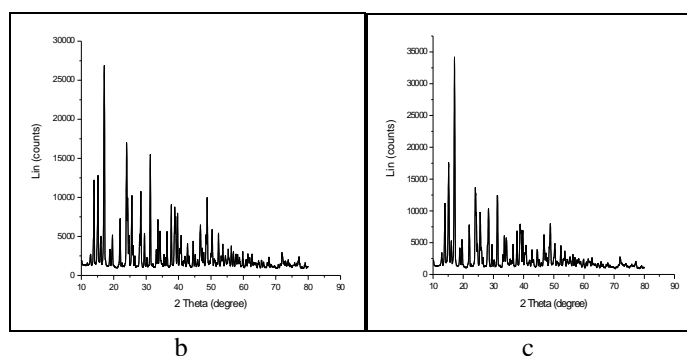
To study the crystal structure of the zinc tartrate Crystal by powder X-ray diffraction method. To obtain unit cell dimensions by single crystal XRD study of pure zinc tartrate Crystal. The lattice parameters a, b, & c found out by 7.945, 8.968, & 11.256 respectively ($a \neq b \neq c$). The interaxial angle $\alpha = \beta = \gamma = 90^\circ$ and volume is 802.0 \AA^3 . The XRD spectrum reveals that the zinc tartrate is polycrystalline having orthorhombic structure with space group $P2_12_12_1$.



.Figure (3.1): (a) Powder XRD spectrum for Sample- I

Powder XRD study for Zn-Cd mixed Tartrate Crystals

Powder X-ray diffraction was recorded with $\text{CuK}\alpha$ radiation recorded 2θ within the range of $10\text{-}80^\circ$. The powder XRD was carried out on Bruker Axs at Sophisticated Analytical Instrument Facility (STIC), Cochin and PANalytical X'pert Powder XRD System, Chennai. The present study of the powder XRD spectrum are shown in figure (3.1) (b) to (e) for the Sample-II to Sample-V Zn-Cd mixed tartrate crystals. The XRD spectrum illustrates that the growing crystals are polycrystalline. The XRD pattern was used to identify the material and to determine the lattice parameters of the grown crystal. Due to random distribution of crystallites a polycrystalline solid is isotropic.



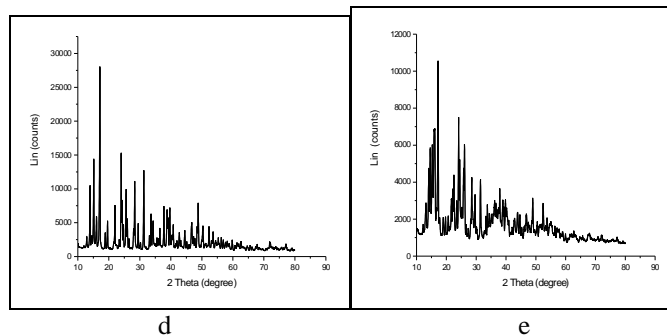


Figure (3.1): (b) Powder XRD spectrum for Sample- II to V

3.2 EDAX Analysis

EDAX stands for Energy Dispersive Analysis by X-ray and it is a technique used for identifying the elemental composition of the samples. When a beam of electron strikes a specimen, a fraction of the incident electrons excites the atom of the specimen, which then emits X-ray when they return to their ground state. EDAX is a technique, which makes it possible to identifying elements and knows their concentration by analyzing the energies of X-ray photons emitted as a result of bombardment by an electron beam [15]. The atomic and weight % of Sample-II to Sample-V is present in Table (3.2).The EDAX spectra are shown in figure (3.2) (a) to (d).

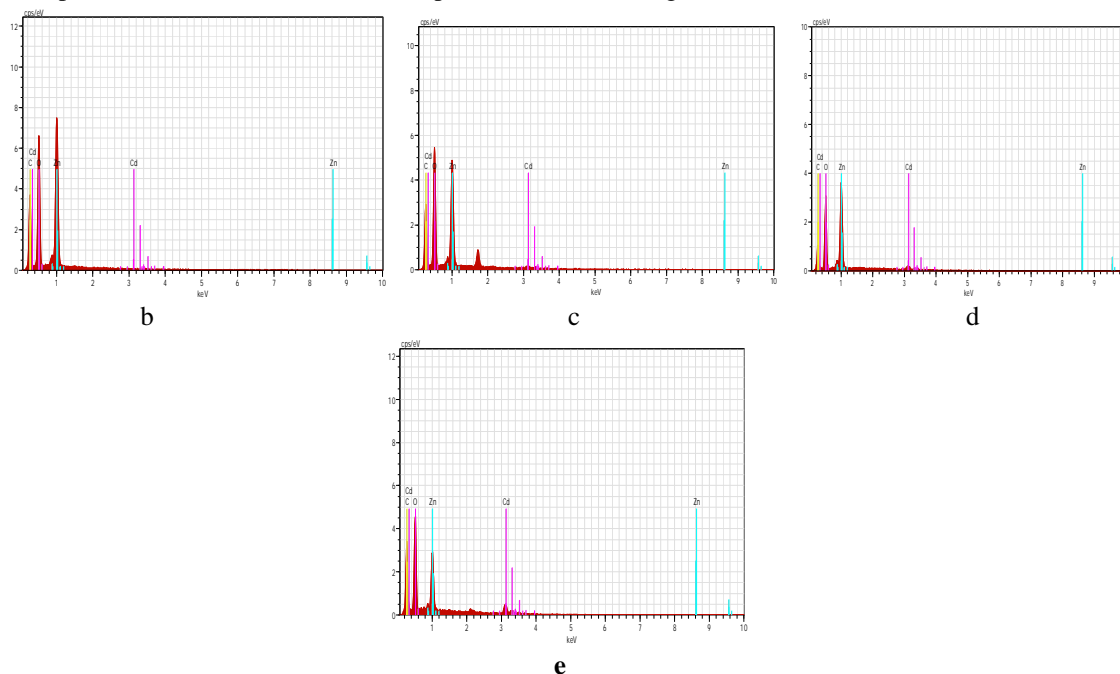


Figure (3.2) The EDAX spectra for Sample- II to V

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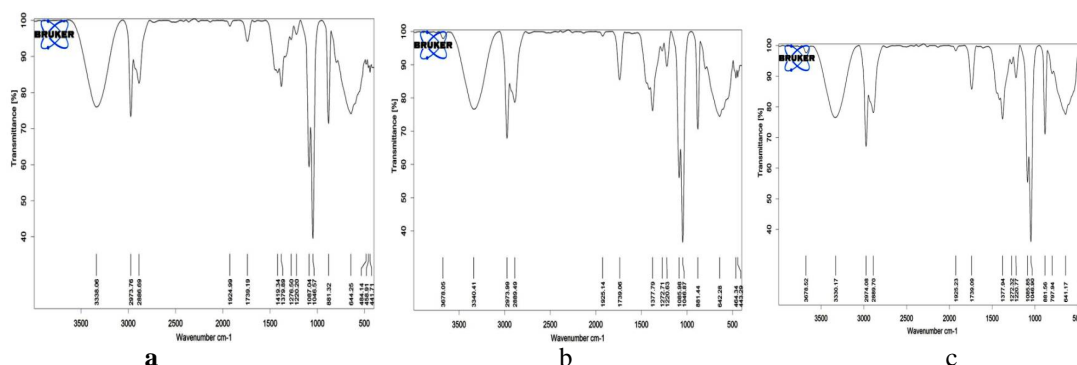
Table (3.2) EDAX result for Sample-II to Sample-V

Crystals	Elements	Atomic %	Weight%
Sample –II	Zn	3.61	2.98
	Cd	0.01	0.08
Sample – III	Zn	1.16	1.29
	Cd	0.06	0.26
Sample- IV	Zn	0.65	0.93
	Cd	0.32	0.70
Sample – V	Zn	0.61	0.81
	Cd	0.45	0.86

From the EDAX analysis we found that when the volume % of cadmium chloride increases, the atomic and weight (%) of cadmium in all grown crystals increasing and the zinc atomic and weight (%) is decreasing.

3.3 FTIR Study

The FTIR spectrum for a particular chemical compound is a unique characteristic of that compound alone. Reflecting as it does the geometry, bond strength and atomic masses of the substance. Therefore, an important use of FT-IR is the identification of unknown chemical. The FTIR spectrum recorded for the grown crystals of pure zinc tartrate and zinc-cadmium mixed tartrate with observed band is shown in the fig 3.3. The spectrum is taken at Department of Nanoscience and Technology, Coimbatore, using the liquid samples in ATR mode in the range from 500 to 4000 cm^{-1} on Brucker- Tensor27 IR spectrometer. It can be observed from the spectrum that the band at 3678.05 cm^{-1} to 2973.99 cm^{-1} are attributed to asymmetric and symmetric O-H stretching of O-H bond, which generally indicating the presence of water of crystallization and the peaks at 2889.49 cm^{-1} are assigned to C-H stretching vibrations. The absorption band at 1595 cm^{-1} is due to C=O stretching vibrations. The C-O stretching vibrations give rise to absorption with 1480 cm^{-1} to 1236 cm^{-1} . The peak 1377 cm^{-1} is due to -O-CH stretching mode. These peaks may be assigned respectively to -C-O asymmetric and symmetric stretching of C-O. The peaks with in 1147 cm^{-1} to 1010 cm^{-1} are due to out plane O-H deformation and C-O stretching. The absorption situated below 921 cm^{-1} is due to metal-oxygen vibrations [16]. It is confirmed that in the present work water of crystallization and metal oxygen bonding is present. In the present study FTIR spectrum Sample-I to Sample-V is shown in the figure (3.3)



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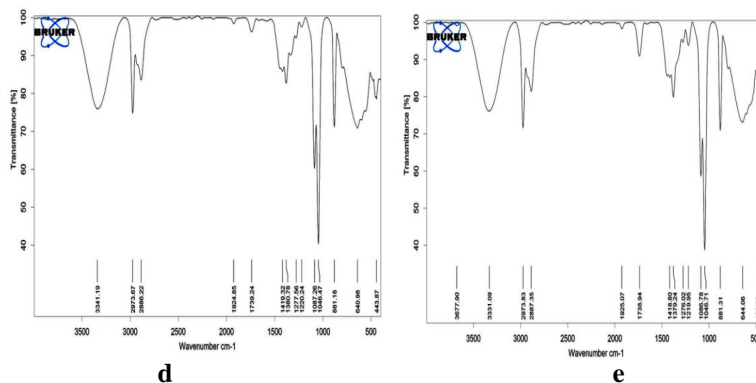


Figure (3.3): FTIR spectrum for Sample- I to V

Table (3.3): FTIR spectral data for samples (I-V)

Assignments	Wave number (cm ⁻¹) Sample (I)	Wave number (cm ⁻¹) Sample (II)	Wave number (cm ⁻¹) Sample (III)	Wave number (cm ⁻¹) Sample (IV)	Wave number (cm ⁻¹) Sample (V)
Free O-H Stretching	3338.06, 2973.76	3678.05, 3340.41, 2973.99	3678.52, 3330.47, 2974.08	3341.19, 2973.67	3677.90, 3331.09, 2973.83
C-H Stretching (asymmetrical)	2886.69	2889.49	2889.70	2886.22	2887.35
Bonded O-H Stretching	2633.88	2633.51	2677.52	2667.83	2689.80
C=O Stretching	1419.99, 1379.89	1377.79	1377.94	1419.32, 1380.78	1418.80, 1379.24
C-O Stretching	1276.50, 1220.20	1272.71, 1220.63	1272.32, 1220.77	1277.56, 1220.24	1276.02, 1219.95
Out plane O-H deformation and C-O stretching	1087.04, 1046.57	1085.98, 1046.87	1085.85, 1046.90	1087.26, 1046.47	1086.78, 1046.71
Meal- Oxygen bonding	Below 921	Below 921	Below 921	Below 921	Below 921

3.4 Thermal Studies

The TGA and DTA for the present samples were carried out from the 40 °C to 730 °C at a heating rate of 10 °C/min in an atmosphere of air. Powdered samples were used for this study. The analyses were carried out at Sophisticated Analytical Instrument Facility (STIC) Kochi. In the present study pure zinc tartrate and Zn-Cd mixed tartrate crystals have been grown and characterized for TGA and DTA analysis.

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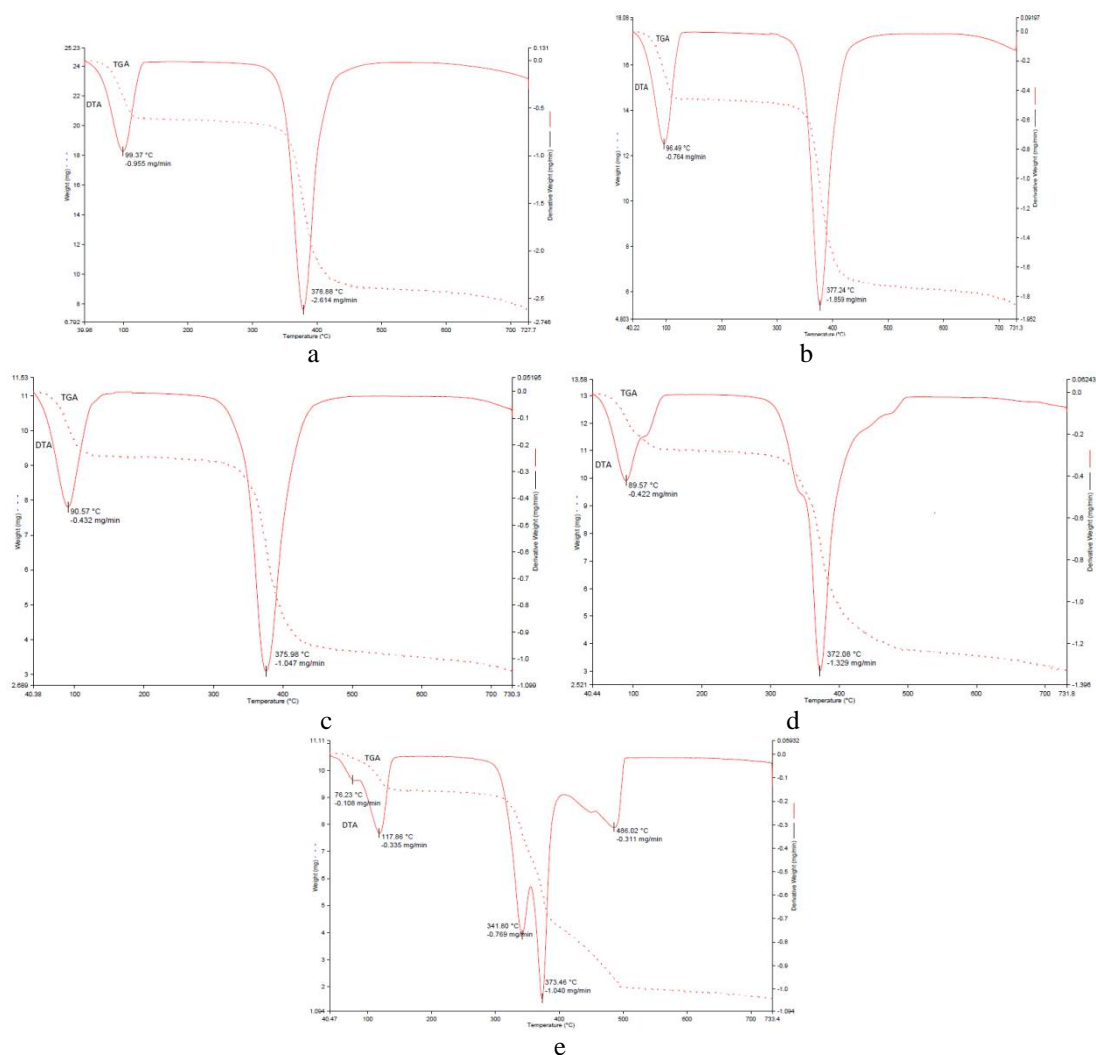


Figure (3.4): (a) TGA/DTA curves for Sample (I-V)

For the crystals grown at gel-liquid interface of samples (I-V), the first stage of dehydration is completed up to 260 °C and 130 °C, respectively, which results into formation of anhydrous zinc-cadmium mixed tartrate. The first endothermic peak in DTA at 99.37 °C the compound stable up to 59°C and then starts losing crystalline water up to 158°C, during this temperature approximately 14.5% weight loss was takes place. The residue material remains stable in the temperature range 158 °C to 310°C. The second endothermic peak is observed at 378.88 °C. The major stage of decomposition process occurs between 310°C to 451°C, during this temperature approximately 43.05% weight loss was takes place. It may due to the decomposition C₄H₄O₅. Then the remaining residue material may be zinc oxide [17]. The sharpness of this endothermic peak shows the good degree of crystallinity and purity of the sample.

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Table (4.3) Thermal stability of Sample- I to Sample-V

Samples	Thermally stable upto($^{\circ}\text{C}$)
Sample- I	59
Sample- II	49
Sample- III	55
Sample- IV	58
Sample- V	61

IV. CONCLUSIONS

The pure zinc tartrate and zinc-cadmium tartrate crystals were grown in gel by using single diffusion as supernatant solution. The XRD spectrum reveals that the sample is poly crystalline and orthorhombic structure with space group $P2_12_12_1$. The FTIR spectrum revealed the presence of water molecules, O-H band, C-O, C-H and carbonyl C=O functional groups with metal-oxygen vibrations. The C-OH in plane bending and out of plane bending is identified. The presence of metal zinc and cadmium identified was confirmed by chemical analysis. The presence of water molecules was confirmed by TGA and DTA analysis. EDAX confirmed the presence of Zn and Cd.

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