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FTIR Spectroscopic and XRD analysis of gel-grown, cadmium levo-tartrate crystals

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Abstract
In the present investigation, pure cadmium levo-tartrate crystals were grown by single diffusion gel growth technique in silica hydro gel medium. Dendrite type pale yellow crystals were obtained. The powder XRD suggested the monoclinic nature of the grown crystals. The FTIR spectroscopy revealed the presence of water molecules, O-H, C-H, C-O and C=O functional groups.

Keywords: Cadmium levo-tartrate crystals, gel growth, FTIR, powder XRD.

1. Introduction
The metal tartrate compounds find various applications in different fields, for example, ferroelectric, dielectric, optical and thermal properties of calcium tartrate [1], piezoelectric application of cadmium tartrate [2] and the addition of lead tartrate in gasoline to prevent knocking in motors [3]. The gel growth technique is elaborately described by Henisch [4]. This technique is found to be suitable to grow tartrate compound crystals and many authors have reported the growth of metal tartrate crystals [5-7], mixed metal tartrate crystals [8-12] and ternary metal tartrate crystals [13, 14] in gel medium. In the present investigation the author has attempted to grow the pure cadmium levo-tartrate crystals and characterize them by Powder XRD and FTIR spectroscopy.

2. Experimental
The single diffusion gel growth technique was employed for the growth of pure cadmium levo-tartrate crystals. The silica hydro gel was used as a growth medium. To prepare the gel, a solution of sodium meta silicate of 1.05 specific gravity was acidified by 1 M solution of levo-tartaric acid in such a manner that the pH of the mixture was set at 4.5. The gel solution was poured in to glass test tubes of 15 cm length and 2.5 cm diameter and allowed to set in the gel form. The supernatant solution, 1 M cadmium nitrate tetrahydrate, was poured on the set gel carefully without damaging the gel. All the chemicals were AR grade and obtained from Ranbaxy chemicals.
The following reaction is expected to occur.

$$Cd(NO_3)_2{\cdot}4H_2O + H_2C_4H_4O_6 + nH_2O \rightarrow CdC_4H_4O_6{\cdot}\ nH_2O + 2HNO_3 + 4H_2O \quad (1)$$

The amount of HNO_3 produced is very less in comparison to the nutrients being supplied to the growing crystals and hence no major limitation is imposed [7-12].
The growth was completed within twenty days. The crystals were 1 to 2 cm long, dendrite type and pale yellow in color. Photograph of the grown crystals is shown in the figure 1.

Fig 1: Growth of dendritic crystals of pure cadmium levo-tartrate

The grown crystals were characterized by different techniques. The Powder XRD patterns were recorded on Philips X'pert MPD by using Cu K_α radiation and the data were analyzed by software powder-x. The FTIR spectra were recorded on Perkin Elmer Spectrum GX spectrophotometer in the range from 400-4000 cm^{-1} in KBr medium.

3. Result and Discussion

Mechanism of dendrite crystal growth was studied by Fujiwara and Nakajima [15]. Dendrite type growth morphology has been observed by several authors in the gel grown pure crystals, such as lead tartrate [16], cadmium tartrate [17], ammonium tartrate [18] and lanthanum tartrate [19] as well as in the mixed crystals, such as lead-cadmium mixed levo-tartrate [10], lead-iron mixed levo-tartrate [11] and lead-cobale mixed levo-tartrate [12]. In the present study to grow pure cadmium levo-tartrate crystals, the supernatant ions of Cd^{+2} slowly diffused into the gel medium where they react with inner tartrate anions. The crystal growth was started about two days after pouring the supernatant solution at the gel liquid interface in the form of a thin layer of very small crystalline particles. The dendritic nature is found may be due to instability occurring in the diffusion reaction process as the rapid growth of crystals takes place in one direction.

A. 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 cadmium levo-tartrate with observed band is shown in the fig 2. The spectrum is scanned in the region 400 to 4000 cm^{-1} using Perkin Elmer Spectrum GX spectrophotometer.

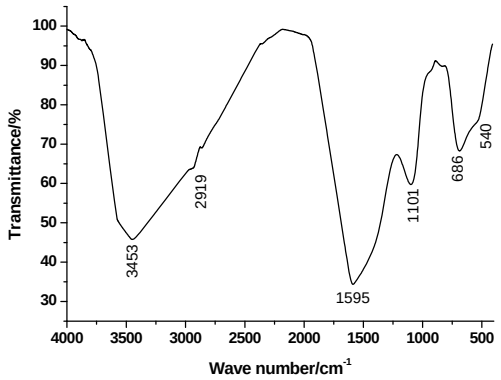

Fig 2: FTIR spectrum of pure cadmium levo-tartrate crystals

It can be observed from the spectrum that the band at 3453 cm^{-1} is due to O-H stretching and water of crystallization. The band observed at 2919 cm^{-1} is due to asymmetrical C-H stretching vibrations. The absorption band at 1595 cm^{-1} is due to C=O stretching vibrations. The absorption band at 1101 cm^{-1} is due to C-O stretching vibrations. The absorptions bands found between 500 and 700 cm^{-1} are due to the metal-oxygen bonding vibrations.

B. Powder XRD Study

The powder XRD pattern of the grown crystals of pure cadmium levo-tartrate is shown the fig 3. The unit cell parameters were computed by using computer software Powder-X. Dabhi [20] has reported the monoclinic unit cell parameters of pure cadmium levo-tartrate crystals as: a = 6.1286 Å, b = 12.3554 Å, c = 7.6101 Å and $\beta = 116^0\ 14'$, while the reported unit cell parameters of monoclinic cadmium tartrate pentahydrate are: a= 6.129 Å, b=12.312 Å, c= 7.627 Å and $\beta = 116^0\ 14'$ with space group $P2_1$ [21]. Altogether, the structural phase transition was observed in cadmium tartrate tetrahydrate crystal at different temperatures by taking powder XRD at 26 ^oC, 60 ^oC and 70 ^oC from space group $P2_12_12_1$ to $P2_1$ and finally to P_{nmn} corresponding to paraelectric phase, ferrolelectric phase and anhydrous phase of cadmium tartrate [22]. In the present study, the monoclinic unit cell parameters of pure cadmium levo-tartrate are obtained as a= 5.9940 Å, b=10.92 Å, c= 7.70 Å and $\beta = 116.5^o$. The deviation from the reported values of unit cell parameters of cadmium levo-tartrate may be due to the different set up of experimental parameters such as, specific gravity of the gel, pH of the gel, concentration of the levo-tartaric acid and supernatant solution, i.e., whether the supernatant solution is the solution of cadmium nitrate, cadmium chloride, cadmium sulphate etc.

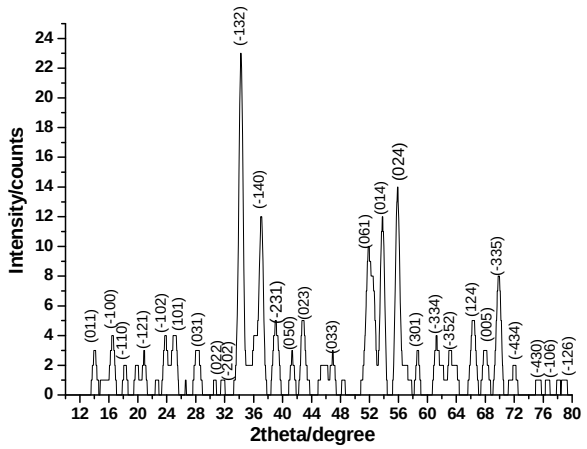

Fig 3: Powder XRD patternof pure cadmium levo-tartrate crystals

4. Conclusions

The pure cadmium levo-tartrate crystals were grown in silica gel by using the cadmium nitrate tetrahydrate as supernatant solution. The FTIR spectrum of the grown crystals indicated the presence of O-H, C-H, C-O and C=O functional groups with metal-oxygen vibrations. The powder XRD suggested the monoclinic crystal structure. The deviation from the reported values of unit cell parameters of cadmium levo-tartrate may be due to the different set up of experimental parameters.

5. Acknowledgments

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