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GROWTH AND CHARACTERIZATION OF CALCIUM TARTRATE CRYSTAL BY GEL METHOD

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ABSTRACT

The crystals of Calcium Tartrate were grown by gel method. The crystals were grown using silica gel as the growth medium. The structural and optical properties of the grown crystals were characterized by FTIR and UV- Visible spectroscopy. FTIR spectra provide the information about functional groups present. The structure of the crystal was analyzed by Single crystal XRD. Single crystal XRD provide the cell parameters and the structure of the grown crystal. The thermal behavior of the crystal was observed from TG-DTA studies.

KEYWORDS

Gel growth, FTIR analysis, Thermal Analysis, XRD studies, UV-Visible studies.

1. INTRODUCTION

Crystal growth involves a variety of research fields ranging from surface physics, crystallography and material science to condensed matter. Though it has been studied more than 100 years, crystal growth still plays an important role in both theoretical and experiment research fields, as well as in applications [1]. Scientifically and technologically crystal growth and characterization have became an interested

research area in the past decades. All basic solid materials are made up of single crystals and they are backbone of the modern technology. The influence of single crystal is noticed in the semiconductors, optics and acoustics, in various medical applications and in jewelers industries [2].Compounds of tartaric acid find several practical applications in science and technology because of their interesting physical properties such as dielectric, ferroelectric, piezoelectric and non-linear optical properties [3]. The gel method is found to be more promising than the high temperature crystal growth methods [4]. Most of the tartrate compounds are insoluble in water and decompose before melting. The gel method can be conveniently used for mass production of crystals. Crystals with dimensions of several mm can be grown in a period of 3 to 4 weeks. The crystals grown by this technique have high degree of perfection. The method is extremely simple and expensive.

2. EXPERIMENTAL

The calcium tartrate single crystals were grown by gel technique using single diffusion method at room temperature. Prepare 10ml of 1M sodium metasilicate. After that 1M of tartaric acid is mixed with 10ml of prepared sodium metasilicate solution. Tartaric acid was mixed with sodium metasilicate by continuous stirring. Stirring was done to avoid premature local gelling. The gelling solution was transferred to test tubes. Test tubes were then closed with rubber corks or cotton to prevent evaporation and contamination of the exposed surface by dust particles of the atmosphere. The gel was set within 24 hours and it leave for another 24 hours for firm gel. After two days the supernatant 5ml of 1M calcium chloride was poured over the set gel by using pipette and kept undisturbed by covering with the cotton plug or rubber cork. Crystals were found within 2 days. The crystals were

harvested after 3 weeks and stored in a clean container. The grown crystals are shown in fig.1.

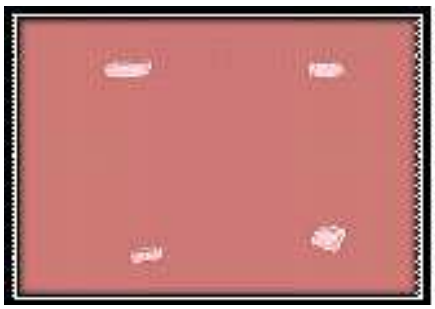

Fig. 1. Photograph of the grown crystals.

2.1. DETERMINATION OF DENSITY

The density of the single crystal was determined by the floatation method. In this method we used three well miscible liquids. They were Bromoform, Carbon tetrachloride and Xylene. Bromoform of density 2.89gm/cc was used as the denser liquid; xylene of density 0.89gm/cc and carbon tetrachloride of density 1.59gm/cc were used as the less density liquids. The low density liquid was taken in a test tube. The transparent small size single crystal was put in the test tube. By the movement of the single crystal upward or downward we concluded that the crystal density is lower or higher than the test solution density. From that we added suitable density liquid, until the crystal remained stationary in the middle of the solution. This was kept undisturbed for a significant length of time to take the crystal as liquid is equal to the density of the crystal. Then the density of the solution was determined by using specific gravity bottle of capacity 5ml. The density of the crystal was calculated using the relation,

$$\rho = \frac{W_3 - W_1}{W_2 - W_1}$$

Where,

W_1 - weight of the empty specific gravity bottle

W_2 - weight of the gravity bottle with pure water

W_3 - weight of the gravity bottle with solution

ρ - Density of the solution

Table. 1. Density values for the parents and complex

Sl. No.	Substance	Density (gm/cc)
1.	Calcium chloride	2.15
2.	Tartaric acid	1.74
3.	Calcium Tartrate	1.87

3. CHARACTERIZATION

The calcium tartrate crystal was grown by single diffusion gel method at room temperature. The grown crystals were subjected to single crystal X- ray diffraction to obtain the unit cell parameters and structure of grown crystals, using Nonius Mach3 four circle diffractometers. The FTIR spectrum was recorded by using a SHIMADZU

Spectrophotometer by KBr pellet technique from 4000cm^{-1} – 400cm^{-1}. The energy gap was measured using UV-Visible spectrophotometer. The thermal behavior of the crystal was analyzed by TG-DTA analysis from 30^0 C to 900^0C at the rate of 10^0 C/min.

4. RESULTS AND DISCUSSIONS

4.1. SINGLE CRYSTAL XRD STUDIES

Using X-ray diffraction method, the preliminary cell values a, b, c, α, □ and □ for the single crystals grown was found out. The cell parameter values of the complexes are given in Table 2.

Table 2. XRD data

Cell values	$CaCl_2$	Tartaric acid	Complex
a	6.24(Å)	7.72 (Å)	9.185 (Å)
b	6.43(Å)	6.00 (Å)	9.635 (Å)
c	4.20(Å)	6.20 (Å)	10.560 (Å)
α	90^0	90^0	90^0
β	90^0	100^0	90^0
γ	90^0	90^0	90^0
V	25.41$Å^3$	286.38 $Å^3$	934.6 $Å^3$

4.2. FTIR ANALYSIS

FTIR spectrum of calcium tartrate is shown in fig.2.

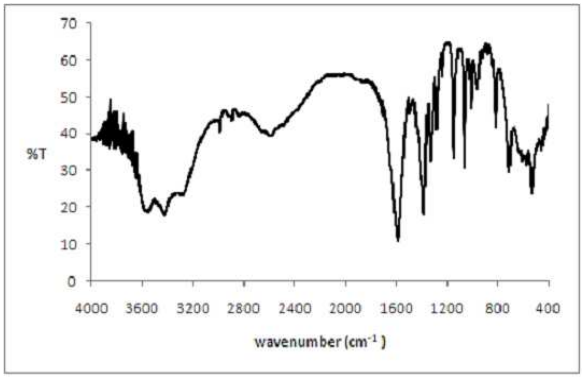

Fig.2. FTIR for complex crystal

The two strong peaks at 3571.32 cm^{-1} and 3423.76 cm^{-1} are due to O - H stretching mode and water. The peak at 2987.84 cm^{-1} is due to the C- H stretching vibration. The band occurs at 1587.47cm^{-1} is due to the C=O stretching of carbonyl group. The strong peak at 1385.90 cm^{-1} is assigned to C=O symmetric stretching. The peak at 1147.68cm^{-1} is attributed to C–H vibrational modes. The peak observed at 1282.71 cm^{-1} is assigned to OH plane bending [5]. The peaks of various intensities at 1061.85 cm^{-1} and 1011.70 cm^{-1} are due to out of plane O–H deformation and C–O stretching. The absorption between 963.48 cm^{-1} and 534.30 cm^{-1} is due to metal oxygen bonding (Ca - O) [6].

4.3. THERMAL STUDIES

The TG-DTA for the grown crystal is shown in fig.3.

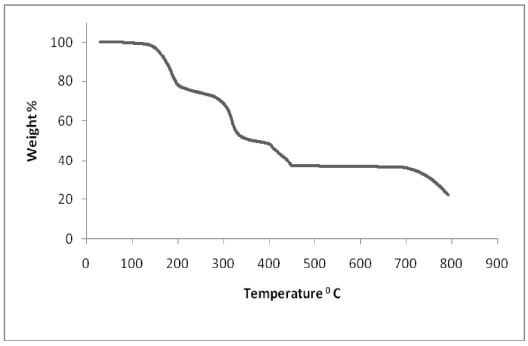

Fig.3. TGA for complex

The thermal decomposition of the crystals occurs in four stages between of 154 - 725^{0}C. Decomposition of the sample at 154^0 C and terminates at 791^{0}C. There are four stages of decompositions. At first stage the crystal loss water and become anhydrous in the temperature range of 154 to 210^{0}C. Then it decompose into calcium oxalate at the temperature range of 293 - 335^0 C and further it decompose into calcium carbonate at 404^0 C. Then it turns into calcium oxide at 725^0 C and the sample remains as calcium oxide for the remaining temperature. The weight loss percentages of the crystal were tabulated below [6].

Sl.No	Temperature range ^{o}C	Weight loss %
1.	154-210	19.74
2.	293-335	23.98
3.	404-454	15.5
4.	725	Remain stable as CaO

The DTA for the crystal is shown in fig.4.

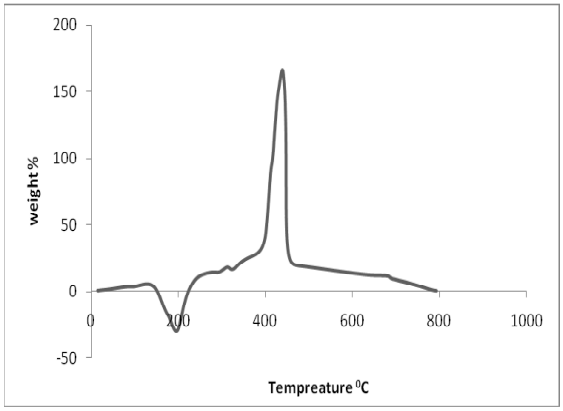

Fig.4. DTA for crystal

From the DTA figure it is observed that there is one exothermic peak at 435^{0}C. The exothermic peak corresponds to calcium carbonate compound.

4.4. UV- VISIBLE STUDIES

The UV spectrum was recorded in the spectral range of 200 - 900nm. The UV-Visible transmittance spectrum is shown in fig.5.

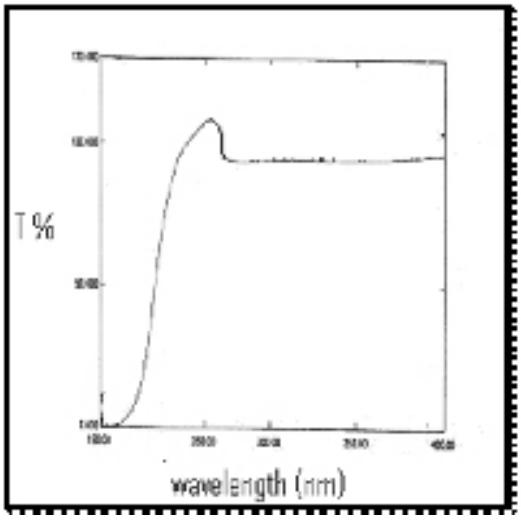

Fig.5. UV-Vis Transmittance spectra

For optical applications, the material considered must be transparent in the wavelength region. The grown crystals were 96% transparent in the UV region. The value of energy gap was calculated from UV-Vis absorption spectra. The absorption spectrum is shown in fig.6.

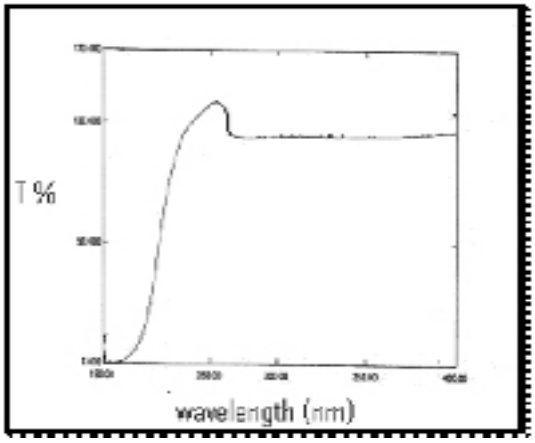

Fig.6. UV-Vis absorption spectra

The maximum absorption was observed at 331nm.The energy gap of the material was calculated using the formula,

$$E = \frac{hc}{\lambda}$$

E- Energy gap

h- Planck's constant

c- Velocity of light

λ- Wavelength

Therefore the energy gap of the crystal is 3.15eV.

4.5. CONCLUSIONS

Calcium tartrate crystals were grown in silica gel by single diffusion method. From the density measurement the density values are in between the parent values. Single crystal XRD provide the cell parameters and the values are different from that of the parents. The grown crystal belongs to orthorhombic structure. From the FTIR studies, vibrational assignments of bands in the infrared spectra are performed for calcium tartrate crystal. The FTIR spectrum of the grown crystals reveal the presence of O–H, C–O and C=O bonds. From TG- DTA the crystal was stable up to 154^0C and the crystals were decomposed into calcium oxide through different stages. From UV -Visible studies, we conclude that grown crystal have transparency of 96%.

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