

SINGLE CRYSTAL OF CADMIUM TARTRATE GROWN BY GEL TECHNIQUE AND ITS CHARACTERIZATION

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Abstract: The growth of Cadmium tartrate single crystals was successfully carried out by using gel growth technique at near ambient temperature using silica gel. 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 Cadmium Tartrate were obtained. The cell parameter values were found using single crystal X-ray diffraction analysis. The optical absorption study was done by UV-Visible spectral analysis. TGA/DTA studies explain the thermal properties of the crystal.

Keywords- XRD, TGA/DTA & UV-Visible spectral analysis.

Introduction

Crystals are the back bone of today's technological development. Crystals are the unacknowledged pillars of modern technology. The world crystal production was estimated to be more than 20,000 ton per year in the year 1999 (D. Elwell, H. J. Scheel at 1975). Out of that the largest share of about 60% was from semiconductor materials, for instance, silicon, gallium arsenide, indium phosphate, gallium phosphate, cadmium telluride and its alloys. Optical crystals and acousto-optic crystals had about equal shares of 10%, whereas laser and nonlinear-optic crystals and crystals for jewelry and watch industry had shares of a few % only. However, over the last decade the demands on synthetic biomaterials have increased significantly with the progress in fields of medicine, material science and engineering, biochemistry, pharmaceuticals and nanotechnology. The growth of crystals occurs either in nature or artificially in a laboratory. The Mother Nature grows a variety of crystals in the crust of Earth, which are mainly the natural mineral crystals including diamond, precious stones and rocks. Mostly these crystals were grown from the molten state by freezing. It is also possible to form crystals directly from a gas without passing through the liquid state; example is the hoarfrost, i.e., ice crystals which are grown from water vapour in the air. Also, a few mineral substances which are found mainly around volcanoes, where sulphur and

ammonium chloride crystals are formed from the gases emitted during eruption. There are other examples of crystals grown in nature, today, the growth of crystals does not remain the phenomena only occurring in nature, but it has become a well advanced as well as widely used laboratory technique.

A crystal is a solid material whose constituent atoms, molecules, or ions are arranged in an orderly repeating pattern extending in all three spatial dimensions. A systematic and scientific study of crystals including process of crystallization, internal structure, external morphology, properties and classification of crystals is known as "*Crystallography*". The study of the formation of crystals is covered under the subhead "*Crystal Growth*". The process of crystal formation is known as *crystallization*. It is interesting to note that the crystal growth can be regarded as an ancient subject, owing to the fact that the crystallization of salt and sugar was known to the ancient Indian and Chinese civilizations. The subject of crystal growth was treated as a part of crystallography and never had an independent identity until the last century. It is important to note that both structural crystallography and the science of crystal growth emerged from curiosity about the large variety in crystal forms existing in nature. The fundamental aspects of crystal growth had been derived from early crystallization experiments in the 18th and the 19th century (H.J. Scheel et al 1993). Theoretical understanding started with the development of thermodynamics in the late 19th century and with the development of nucleation and crystal growth theories and later on the increasing understanding of the role of transport phenomena in the 20th century. There always has been a requirement of good quality crystals for various applications (H.J. Scheel et al 2003). The demand of the modern day science and technology has tempted scientists to synthesize and grow several new varieties of crystals. The requirement for better, cheaper, and larger single crystals has driven extensive research and development in crystal growth. This has brought the field of crystal growth into the lime light. As a result the congeries of crystals is ever expanding day by day (P.S. Raghavan & P. Ramasamy 2000).

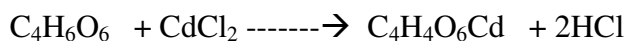
Material and Methods

Experimental

Good quality crystals were grown in gels in a variety of ways; the single diffusion technique was used in the present work for of the cadmium tartrate crystals growth. The growth process involves the diffusion of cadmium chloride solution in to a gel in which tartaric acid is impregnated before. The hydro silica gel was used as a growth media. The chemicals used for growth tartrate were $C_4H_6O_6$, Na_2SiO_3 and $CdCl_2$ all chemicals were of AR grade. The

crystallization apparatus consist of borosilicate glass test tubes of length 20 cm and diameter 2.5 cm placed vertically on plastics stands. Tartaric acid, cadmium chloride solutions were prepared by dissolving these compounds in an appropriate amount of double distilled water to give the required molarities. Gels of required specific gravity were prepared by adding to the solution of sodium metasilicate, a calculated amount of redistilled water and a stock solution was kept ready for doing further experiments. (A.R. Patel & A.V. Rao 1982) Tartaric acid solution of particular strength was taken in a 100ml beaker and sodium metasilicate solution of a suitable gravity was added drop wise using a teflon cock burette, constantly stirring the solution in a beaker by magnetic stirrer. Stirring is done to avoid the excessive local ion concentration which may otherwise cause premature local gelling and make the final medium inhomogeneous and turbid. Here tartaric acid acted as a lower reactant. The digital pH meter was used to measure the pH. The solution after noting pH values, being allowed to fall along the side of a test tube without giving chance for the formation of the bubbles. Test tubes were then closed with rubber corks or cotton to prevent evaporation and contamination of the exposed surface of the gel by dust particles of the atmosphere. The solution was found to be strongly depends on pH. High pH value gel takes lower time to set than low pH value, depending on the environmental temperature. After ensuring firm gel setting, the saturated mixed solution of cadmium chloride (supernatant) of particular strength was poured over the set gel with the help of a pipette (E. Ramachandran & S. Natarajan 2005). The solution being allowed to fall along the wall of the test tube to prevents the gel surface from cracking. The supernatant ions (Cd^{++}) slowly diffused in to the gel medium where it reacts with inner reactant [6-9].

The following reaction is expected to take place in the formation of cadmium tartrate crystals.



Result and Discussion

The various optimum conditions for the growing crystals were found and are given in table 1. Figure 1 shows different morphology of grown cadmium tartrate crystal such as inside the test tube like Brush shape, Necklace shape crystal & some transparent opaque, diamond shape crystal are observed.

Table 1: Optimum condition for growth of Cadmium tartrate

Condition	Cadmium tartrate
Density of sodium meta silicate solution	1.04gm/cm ³
Concentration of tartaric acid	1M
Volume of tartaric acid	5ml
Volume of sodium meta silicate	16ml
pH of the mixture	4.2
Concentration of CdCl ₂	1M
Temperature	Room temperature
Gel setting time	96 Hours
Gel aging time	130Hours
Period of growth	5 weeks

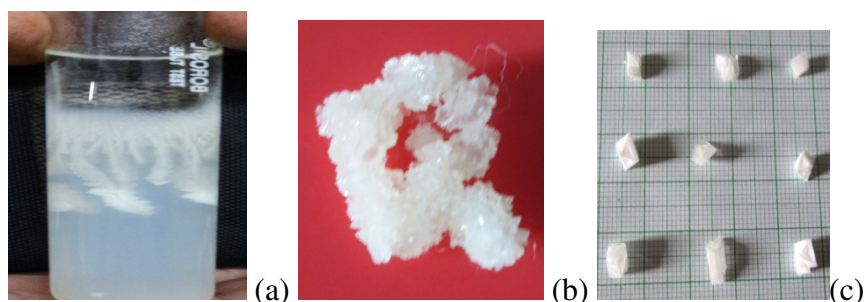


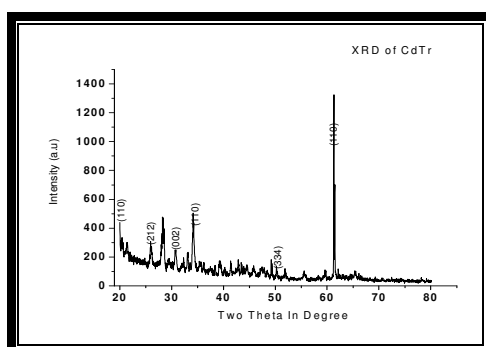
Figure 1. Show the optical photograph of growth of cadmium tartrate inside the test tube. (a) Brush shape (b) Necklace shape crystal (c) illustrates different morphologies of pure cadmium tartrate crystals grown under different conditions of growth.

Characterization

X-ray diffraction of CdTr Crystal - X-ray diffractogram of gel grown cadmium tartrate was recorded using powder rotation photograph method on “Minislex Regaku” X-ray diffractometer at Department of Physical Sciences, North Maharashtra University, Jalgaon. CuK α -radiation (wavelength $\lambda=1.54051 \text{ \AA}$) was used. The sample was rotated in the range (2θ) , 20° - 80° (D.K. Sawant et al 2011). The scanning speed was kept $10^\circ/\text{min}$. The recorded x-ray diffractogram is shown in figure 2. The unit cell parameters is shown in table 1 and system is monoclinic ($a \neq b \neq c$). (S. L. Garud & K.B. Saraf 2005).

Table 1: Calculated unit cell parameters

Lattice Parameters	Cadmium tartrate
System	Monoclinic
a	6.129A ⁰
b	12.314 A ⁰
c	7.627 A ⁰
$\alpha\neq\beta\neq\gamma$	90 ⁰

**Figure 2:** XRD of CdTr Crystal

TGA of CdTr Crystal - The thermograms were obtained with the help of Diamond TGA/DTA thermal analyzer available at Shivaji University Kolhapur Recrystallized alumina sample holders were used and the heating rate was 10⁰C/min. The TGA/DTA thermograms were recorded in the temperature range from 30⁰C to 100⁰C. The experiment was carried out under a flow rate of 60ml/min in nitrogen atmosphere. Microcrystalline (powdered) samples of calcium tartrate were taken for TGA/DTA, studies and the weight of the sample was 3.0150mg. The sample was hold for 1.0min at 30⁰C to evaporate water due to moisture and then heated from 30⁰C to 1000⁰C at the rate of 10⁰C/min. Figure 3 shows the TGA of cadmium tartrate crystal. Table 2 summarizes the TGA curve (G. Sivakumar et al 1999 & S.J.Nandre et al 2013).

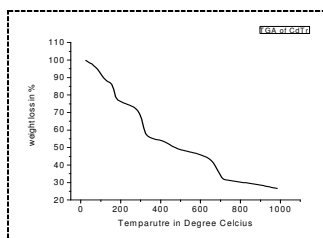
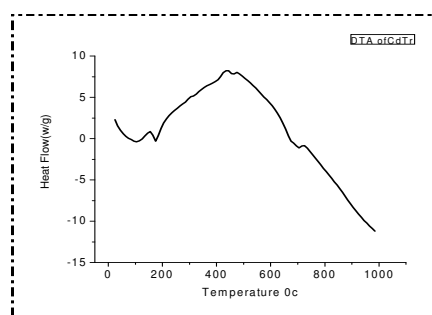
**Figure 3.** TGA of CdTr Crystal

Table 2: Results of decomposition process of Cadmium tartrate crystals

Sr.No	Decomposition Stage	Temperature in $^{\circ}\text{C}$	Observed weight loss in %
1	I	30-150	8.388
2	II	150-170	5.274
3	III	170-190	8.431
4	IV	190-270	5.966
5	V	270-380	17.60
6	VI	380-610	9.658
7	VII	610-720	12.89
8	VIII	720-1000	26.54

DTA of CdTr crystal- The DTA curve for cadmium tartrate gel grown crystals is as shown in the figure 4 and the DTA data collected from this curve is tabulated in the table.(s.J.Nandre et al 2013).

**Figure 4:** DTA of CdTr**Table 3:** Results of decomposition process of Calcium tartrate crystals

Peak Temperature in $^{\circ}\text{C}$	Nature of Reaction
102.55 $^{\circ}\text{C}$	Endothermic
168.0 $^{\circ}\text{C}$	Exothermic
175.64 $^{\circ}\text{C}$	Endothermic
440.0 $^{\circ}\text{C}$	Exothermic
459.90 $^{\circ}\text{C}$	Endothermic
703.55 $^{\circ}\text{C}$	Endothermic

UV Absorption Spectroscopy of CdTr Crystal- Absorption spectra of cadmium tartrate crystals were recorded using a SHIMADZU UV-2450 UV-Vis spectrophotometer over the wavelength range 200nm–900nm at Department of Physics; Savitribai Phule

University Pune. Figure 5 shows UV absorption spectra of cadmium tartrate crystals. Band gap energy (eV) = 1240/wavelength (nm). Band gap energy is presented in the table 4. (S.J.Nandre et al 2012).

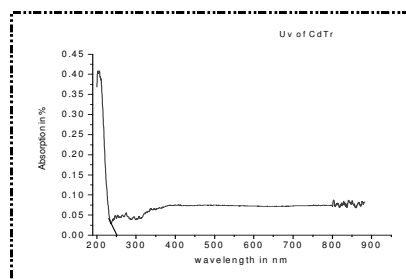


Figure 5: UV absorption spectra of CdTr

Band gap energy (eV) = 1240/wavelength (nm).

Crystal	λ (nm)	Band gap Energy (eV)
Cadmium tartrate	255	4.86

Acknowledgement

The authors are grateful to Dr. P.P. Patil, School of Physical Science of NMU Jalgaon, for providing XRD facilities. Authors, thankful to Dr Sanjay Dhole Savitribai Phule University Pune for providing the facility of UV spectroscopy. Also thankful to Dr. N.G. Deshpande, Department of Physics, Shivaji University Kolhapur and Dr. S.J. Nandre, Uttamrao Patil Arts and Science college Dahivel for fruitful scientific discussion. Special thanks to Dr. D.L. Torwane Principal, S.G. Patil Art's, Com. and Science College, Sakri for continuous encouragement.

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