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Growth and characterization of calcium doped cadmium tartrate crystal by silica gel method

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ABSTRACT

In the present investigation, Calcium Cadmium Tartrate crystals were grown in silica gel at ambient temperature. The doping effect of Calcium ion in Cadmium Chloride solution on the size and transparency of Cadmium Tartrate crystal are presented in this paper. The crystals have been synthesized by controlled diffusion of Cadmium Chloride into Gel with Tartaric acid at room temperature. The Calcium ions enhance the size of transparency of the doped crystals. 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 Calcium-Cadmium Tartrate were obtained. The crystal structure of the compound was confirmed by powder X-ray powder diffraction, scanning electron microscope (SEM), and Energy Dispersive Analysis by X-ray (EDAX).

Keywords: crystal growth, Gel method, XRD, SEM, EDAX

INTRODUCTION

The subject of crystal growth has held a high level of useful information, both of scientifically and technologically [1]. Hence an understanding of how crystals are grown is an important aspect of the science material [2]. The better quality crystal in industries and modern technology of logical revolution. The impact of single crystals is clearly visible in industries like semiconductors, optics etc. and the field of the nonlinear optics and the practical implementations were possible with the applications of nonlinear optical crystal. Growth of the crystal of Calcium doped Cadmium Tartrate (CaCdTr) [3]. Now a day great attention has been devoted the growth and characterization of doped Tartrate crystal with the aim of identifying new materials for practical purposes.[4]. The doping effects on various crystals are of great interest from solid state science as well as technological point of view. The crystal of Cadmium Tartrate grown in silica gel medium in doped with Barium, Strontium, Lithium, Calcium have already been reported [5]. In the present work we have attempted to grow pure and Calcium doped Cadmium Tartrate crystals.

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MATERIALS AND MEHTODS

Crystal Growth

In the present investigation the Calcium Cadmium Tartrate crystal were grown by single diffusion gel method. Most of the Tartrate compounds are insoluble in water and decompose before melting. Hence, such type of compounds cannot be grown by either slow evaporation or melt technique. But can be grown by solution gel method [6]. A single diffusion method (Henish 1973) was employed to grow pure and Calcium doped Cadmium Tartrate crystal in the gel method. The AR grade (Loba) chemicals were used for the present work. The crystallization apparatus employed was borosilicate glass tubes (25mm diameter and 200mm length). Gels were prepared by mixing Sodium Meta Silicate solution of appropriate specific gravity and 1M solution of Tartaric acid so that the desired pH of the mixture should be obtained. The specific gravity and pH were varied between 1.02 gm/cc and 1.05 gm/cc and 4 to 5 pH respectively. After mixing the solution was allowed to set for about 48 hours. Over the set Gel, 1M Cadmium Chloride solution was gently poured with the help of a pipette, so as to allow the solution to fall steadily along the walls of the tube without disturbing the gel surface. The supernatant ions (Ca⁺⁺ and Cd⁺⁺) slowly diffuse into the gel medium where it reacts with inner reactant. The open end of the test tube was closed with cotton to avoid dust from the entering into the glass tube. The solution was faint milky and transparent, initially, but with lapse of time its color slightly changes. The test tubes were kept undisturbed at room temperature. To grow doped crystals, an aqueous solution of Calcium Chloride of varying concentration 0.2- 1.0 M was mixed with the top solution. After one month the crystal was taken out from the test tube and cleaned for the further characterization. The best quality crystals were grown for 4.2 pH, as shown in fig (b) [7].



Fig.1(a) single diffusion method



Fig.1(b) Calcium Cadmium Tartrate crystal

Chemical reaction

The following reaction is expected to take place in the formation of Barium Cadmium Tartrate crystal,

 $2(C_4H_6O_6) + {}_{X}CaCl_2 + {}_{(1-x)}CdCl_2 \rightarrow Ca_xCd_{(1-x)}(C_4H_4O_6)_2 {}_{x}H_2O + 4HCl.$

RESULTS AND DISCUSSION

The various optimum conditions for the growing crystal were found and are given in Table 1

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Condition	Calcium Cadmium Tartrate		
Density of sodium meta silicate solution	1.04 g/cc		
Concentration of tartaric acid	1M		
Volume of tartaric acid	8ml		
Volume of sodium meta silicate solution	28ml		
pH of the Gel	4.5		
Concentration of CaCl ₂	0.2M		
Concentration of CdCl ₂	1.0M		
Temperature	Room Temperature		

Table1 Optimum conditions for growth of Calcium Cadmium Tartrate

Different parameters such as concentration of reactants, pH of gel, impurities in the solvent, gel setting time, gel aging time etc have considerable effect on growth rate. Fig. 2 shows optical photographs of Calcium Cadmium Tartrate crystals inside the test tube and Fig. 3 illustrates the different morphologies of Calcium Cadmium Tartrate crystals grown under different conditions of growth. The crystals grown were whitish, milky white and transparent, semitransparent and rectangular in shape well defined crystals of Calcium Cadmium Tartrate crystals were obtained. Some of them were transparent small diamond shape due to fast growth rate twin crystal are obtained faces are well developed and polished Fig.2 [8].



Fig.	2
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Table 2 Effect of concentration of reactants and characteristics of the crystal

Concentrations of reactants in gel	Concentrations of reactants above gel	Habits	Quality	Size(mm)
C ₄ H ₆ O ₆ 1M(8ml,pH 4.2)	$Cacl_2, CdCl_2$ (20ml)	Prismatic	Opaque	2x2x2
$C_4H_6O_61M$ (8ml, pH 4.2)	CaCl ₂ CdCl ₂ 0.5 M(25ml)	Prismatic	Good transparent	2x2x2

Table 3 Chemical composition of CCT crystals % weight

Composition	Cd ²⁺	Ca ²⁺	$C_4H_6O_6$	water
Experimental	15.10	15.02	60.90	10.50
calculated	15.46	15.06	60.91	10.30

Powder X-Ray Diffraction



Fig. 3 Powder XRD of Calcium Cadmium Tartrate

Table 4 Powder diffraction data of Calcium doped Cadmium tartrate crystal $\lambda = 1.54056$ Å.

From present work				From JCP.	DS file		
20	Observed d-value	Intensity I/Io	h k l values	20	Standard d-value	Intensity I/Io	h k l values
17.445	5.07956	12296	100	17.099	5.07950	505	100
32.531	2.75019	3743	103	32.733	2.75010	371	103
34.382	2.60622	13287	200	34.595	2.60628	49	200

In the above table, the observed d-values and *h k l* plane are compared with standard data of 2002 JCPDS v. 2.3, 26-0282.

X-Ray diffraction

X ray diffraction study of Calcium Cadmium Tartrate crystals were carried out using BRUKER AXSD8 advance model X ray diffraction with CuK α 1 (λ =1.54056 Å) radiation in the 20 range 10⁰ - 80⁰. The scanning speed of the specimen was 2⁰/min. In the present study of X-Ray powder pattern of Calcium doped Cadmium Tartrate crystals grown in gel medium were obtained and used to identify the grown material. The XRD pattern of CaCdTr is shown in fig. 3. The spectrum match with the data reported in JCPDS files No26-0282. From this diffractogram intensity and *hkl* values were computed. The observation table give the index XRD data for the grown crystals value and *hkl* plane were calculated the unit cell parameter satisfy the condition for hexagonal system that is a = b \neq c and α =90 β =90 γ =120⁰. From X-ray diffraction study it may be concluded that the grown crystal of CaCdTr crystal have hexagonal system. The observed and calculated d values are given in Table 4.

Parameter	Calcium doped cadmium Tartrate crystal
system	Hexagonal
а	5.983
b	5.983
с	9.654
α	90^{0}
β	90^{0}
γ	120^{0}
v	56.53

Table 5 Unit Cell parameter

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The diffracting index observed (d) values are in good agreement with calculated values. It is very interesting to note that CaCdTr crystals are hexagonal with non Centro symmetric space group where as Cadmium Tartrate crystals in Centro symmetric monoclinic space group [9,10].

Percentage of crystallinity is very good and it is 91.6% and crystal size is 9.2nm and FWHM is 14.079. Analysis composition determines by chemical analysis 59.9% Ca. and 40.1% Cd. and space group Pm-3m (221).

Energy Dispersive Analysis by X-ray (EDAX)

Energy Dispersive analysis by X-ray (EDAX) is used for the quantitative analysis and is also called as elemental analysis. In the present work elemental analysis of gel grown Calcium Cadmium Tartrate crystal was carried out at the NMU in chemical sciences Jalgaon. Fig 4 shows EDAX spectrum of Calcium Cadmium Tartrate crystal. When a beam of electron strikes a specimen, a fraction of the incident electron excite the atoms of the specimen, which then emit X-ray, when they returned to their ground state [11, 12]. The energy of these X-ray is strictly related to the atomic number of the element excited and therefore their detection forms the basis of elemental analysis in the electron microscope. EDAX carried out standard less at 15.0KV energy showed the following result for the given sample of Calcium Cadmium Tartrate crystals. The pulse rate 1.55 Kcps. and peak ranging from 0-5 eV [13]. It is clearly indicates that the presence of Calcium Cadmium Tartrate sample characteristic peak of the sample appear in between the 0-5 Kev. The relative concentration of the Calcium Cadmium Tartrate is observed 48% and 56%.



Fig.4 EDAX spectrum of Calcium Cadmium Tartrate

Table 6 shows the value of elemental content of the crystal as measured by EDAX technique (At%) and the theoretical calculation from molecular formula (Wt%).[14,15].

Element	AN	series	Unn.c Wt%	Norm.c Wt%	Atom.c Wt%	Sigma Wt%
Ca	20	K-series	10.99	98.60	99.60	0.76
Cd	28	L-series	0.16	1.40	0.50	0.16
Total			11.15	100.00	100.00	

Scanning Electron Microscope (SEM) of Calcium Cadmium Tartrate crystals

In the present work powder sample of Calcium Cadmium Tartrate crystal was examined by using SEM technique [16]. The study of the surface of crystal gives valuable information about its internal structure. Figs. 6 a & b shows SEM photograph of crystal of Calcium Cadmium Tartrate crystals. This technique combines the resolution and analytical power with ease of operation. Image can be formed form a very wide range of material from metal to ceramic, semiconductor to polymer [17]. These materials can be examined with low energy secondary electron with

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high energy back scattered electron or with other emission such as light, heat and sound. The high depth of field of the SEM image makes it. Especially suitable for the study of the fracture and complex microstructure such as found in composite. Figure 6 (a) and 6 (b) shows plate and rock like crystal morphology. It is very clear shows all boundaries. These crystals are grown by layer by layer deposition [18, 19].



Fig. 6 (a) and (b) SEM Image of Calcium Cadmium Tartrate

CONCLUSION

1)Gel method is found suitable for growing Calcium Cadmium Tartrate crystal.

2) The growth of single crystal of Calcium Cadmium Tartrate crystal was accomplished using single test tube diffusion method. Optimum condition for growth ware worked out.

3)Different habits of calcium cadmium tartrate crystal can be obtained by changing parameters like gel density, gel ageing, pH of gel, concentration of reactants etc.

4)Calcium Cadmium Tartrate crystals were grown and powder XRD confirms the Unit cell parameter value with those available in the literature matches its value J.C.P.D.S. data. The XRD Shows that grown crystal is Calcium Cadmium Tartrate crystal belong to hexagonal system.

5)EDAX studies revels that grown crystal are CCT indeed. Water of Crystallization present in grown crystal and presence of Calcium and Cadmium is confirmed in the crystals.

6)The SEM Images shows morphology of CCT crystal studies suggested 2-D layer deposition growth.

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