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Growth and characterization of barium doped cadmium tartrate crystal by solution gel method

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ABSTRACT

The doping effect of Barium ion in Cadmium Chloride solution on the size and transparency of Cadmium Tartrate crystal are presented in this paper. Doped crystals were synthesized by controlled diffusion of Cadmium Chloride into Gel with Tartaric acid at room temperature. The Barium ions enhance the size of transparency of the doped crystals. The crystal structure of the compound was confirmed by X-ray diffraction and Field Emission Scanning Electron microscopes (FESEM), Energy Dispersive Analysis X-Ray diffraction (EDAX) etc.

Keywords: Crystal growth, Gel method, XRD, FESEM, EDAX etc.

INTRODUCTION

Single crystals are the backbone of the modern technology of logical revolution [1, 2, 3]. The impact of single crystal, is clearly visible in industries like semiconductors, optics etc. This type of crystal inventions of LASER, and the field of the nonlinear optics and the practical implementations was possible with the applications of nonlinear optical crystal. Now a day great attention has been devoted on the growth and characterization of doped Tartrate crystal with the aim of identifying new materials for practical purposes [4, 5]. The effect of doping on various purpose of crystal are of great interest from solid state science as well as technological point of view. The crystals of Cadmium Tartrate grown in silica gel medium in doped with Barium, Strontium, Lithium, Calcium have already been reported [6]. The growth of Barium doped Cadmium Tartrate crystals yet had not been reported. In the present work we have attempted to grow pure and Barium doped Cadmium Tartrate crystal by Gel Technique. This Growth experiment yield crystal in the Gel using solution Gel Technique [7].

MATERIALS AND MEHTODS

Crystal Growth

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. A single diffusion method (Henish 1973) was employed to grow pure and Barium doped Cadmium Tartrate crystal in the gel method [8]. The AR grade (Loba) chemicals were used for the present work. The crystallization apparatus employed was borosilicate glass tubes (25mm diameter and 200mm length). Gel was prepared by mixing Sodium Meta Silicate solution of appropriate specific gravity and one molar solution of Tartaric Acid so that the

desired pH of the mixture could be obtained. The specific gravity and pH were varied between 1.02 gm/cc and 1.05 gm/cc and 4 to 5 respectively. After mixing the solution was allowed to set for about 48 hours. Over the set gel, one molar 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 (Ba^{++} 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 change. The test tubes were kept undisturbed at room temperature. To grow doped crystal, an aqueous solution of Barium 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 [9]./The best quality crystals were grown for 4.2 pH as shown in fig.1

Fig 1

Chemical reaction

The following reaction is expected to take place in the formation of Barium cadmium tartrate crystal,

$$2(C_4H_6O_6) + {}_xBaCl_2 + {}_{(1-x)}CdCl_2 \rightarrow Ba_xCd_{(1-x)}\,(C_4H_4O_6)_{2\,x}\,H_2O + 4HCl.$$

RESULTS AND DISCUSSION

The various optimum conditions for the growing crystal were found and are given in table no.1

Table 1 Optimum conditions for growth of BCT Crystal

Condition	Barium Cadmium Tartrate
Density of sodium meta silicate solution	1.04 g/cc
Concentration of tartaric acid	1M
Volume of tartaric acid	7ml
Volume of sodium meta silicate solution	24ml
pH of the Gel	4.2
Concentration of $BaCl_2$	0.2M
Concentration of $CdCl_2$	1.0M
Temperature	Room Temperature

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.1shows photographs of Barium Cadmium Tartrate crystals inside the test tube and Fig.2 shows the different morphologies of Barium Cadmium Tartrate crystals grown under different conditions of growth [10]. The crystals grown are whitish, milky white and transparent, semitransparent and rectangular in shape well defined crystals of Barium 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

Table 2 Effect of concentration of reactants and habits, quality and size of thecrystal

Concentrations of reactants in gel	Concentrations of reactants above gel	Habits	Quality	Size(mm)
$C_4H_6O_6$ 1M(8ml,pH 4.2)	$Bacl_2$,$CdCl_2$ 0.5&1M,20ml	Prismatic	Opaque	2x2x2
$C_4H_6O_6$ 1M(8ml ,pH 4.2)	$BaCl_2CdCl_2$ 0.5&1 M(25ml)	Prismatic	Good transparent	2x1x2

Table 3 Chemical composition of BaCdTr crystals % weight

Composition	Cd^{2+}	Ba^{2+}	$C_4H_6O_6$	water
Experimental	15.10	12.02	60.90	12.50
calculated	15.46	12.06	60.91	11.30

3.1. Powder X - Ray Diffraction

Table 4 Powder diffraction data of BCT crystal

λ =1.54056Å.

From present work				From JCPDS file			
2θ	Observed d-value	Intensity	h k l values	2θ	Standard d-value	Intensity	h k l values
18.648	4.75441	1081	111	18.659	4.75438	68	101
28.483	3.13116	363	300	28.772	3.13106	26	200
30.439	2.93426	1120	212	30.441	2.93430	756	211
32.415	2.75976	1419	203	32.589	2.75970	911	112
37.325	2.40723	928	222	37.837	2.40728	311	103
44.551	2.03215	708	410	44.608	2.03220	17	402
45.155	2.90632	718	105	45.831	2.90645	4	431
46.153	1.96524	104	304	46.073	1.96521	3	204

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.

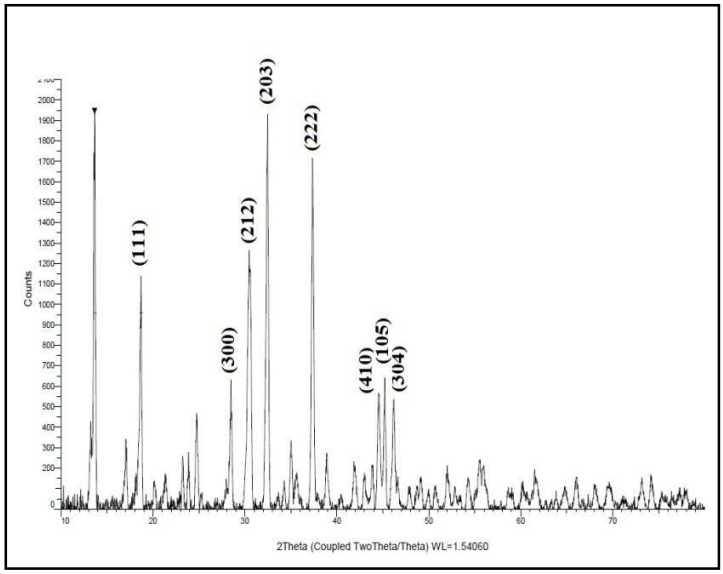

Fig. 3 Powder X-Ray diffraction of BCT Crystal

X-Ray diffraction

X ray diffraction technique is used to investigate the inner arrangement of atoms or molecule in the crystalline material. The grown BaCdTr crystals/were subjected to powder diffraction pattern of the grown crystal was carried out using BRUKER AXSD8-Advance model Germany. X ray diffraction with CuKα1 radiation of wave length (λ=1.54056 Å) operating at a voltage of 40KV and a current 20 mA. The scanning rate was maintained at $2°/min$ over a 2θ range of 20 - $80^{0.}$ In the present study of XRD powder pattern of Barium doped Cadmium Tartrate crystal is shown in fig. 3. The sharp peaks with maximum intensity characterize the XRD pattern, indicating the formation of well defined crystallites. The spectrum match with the data reported in JCPDS file No26-0282 [11]. From this diffraction pattern intensity and (h k l) values were computed. The observation table give the index XRD data for the grown crystals value and (h k l) plane were calculated the unit cell parameter satisfy the condition for hexagonal system that is a= b≠ c and α=β=90^0γ = 120°. The observed and calculated d values are given in Table no. 1The diffracting index observed, the (d) values are in good agreement with calculated values. It is very interesting to note that BaCdTr crystals are hexagonal with non Centro symmetric space group where as Cadmium Tartrate crystals in Centro symmetric monoclinic space group [12].

Unit Cell parameter

Parameter	BCT crystal
system	Hexagonal
a	10.74
b	10.74
c	10.2
α	90^0
β	90^0
γ	120°
v	74.46

Percentage of crystallinity is very good and it is 88.6% and crystal size is 5.2nm.and FWHM is 17.114. Analysis composition determines by chemical analysis 49.9% Ba. and 50.1% 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 [13]. In The present work elemental analysis of gel grown Barium Cadmium Tartrate crystal was carried out using BRUKER-X-Flash detector at the NMU Jalgaon. Fig. 2 represents that EDAX spectrum of Barium Cadmium Tartrate crystal. When a beam of electron strikes a specimen, a fraction of the incident electron excites the atom of the specimen, which then emits X-ray when they return to their ground state. 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 [14]. EDAX carried out standard less at 15.0KV energy showed the following result for the given sample of Barium Cadmium Tartrate crystals. The pulse rate 2.97 Kcps. and peak ranging from 0-5 Ev. It is clearly indicates that the presence of Barium Cadmium Tartrate sample characteristic peak of the sample appear in between the 0-5 Kev. The relative concentration of the Barium Cadmium Tartrate is observed 48% and 56% [15].

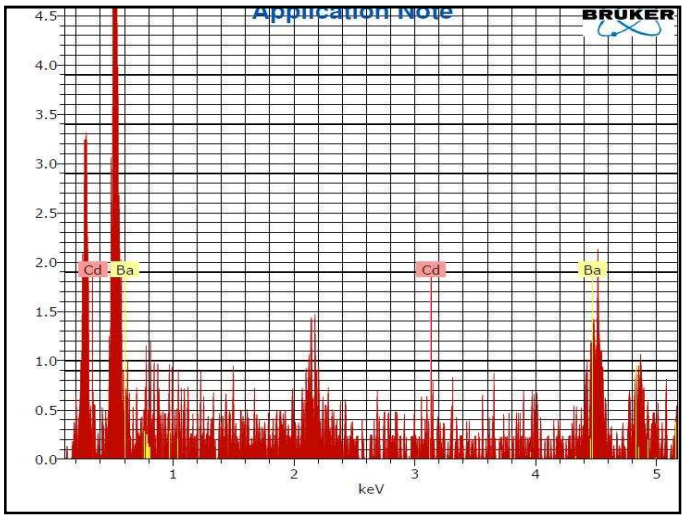

Fig. 4 EDAX spectrum of barium cadmium tartrate

Table-shows the value of elemental content of the crystal as measured by EDAX technique (At%) and the theoretical calculation from molecular formula (Wt%).

Table 5 Value of elemental content of barium cadmium tartrate

Element	AN	series	Unn.c Wt%	Norm.c Wt%	Atom.c Wt%	Sigma Wt%
Cd	48	L-series	0.47	2.52	3.06	0.20
Ba	56	L-series	18.35	97.48	96.94	1.49
Total			18.82	100.00	100.00	

Scanning Electron Microscope (SEM) of BCT Crystal

In the present work powder sample of Barium Cadmium Tartrate crystal was examined by using SEM technique. The studies of the surface exposure to air of doped $BaCdcl_2$ were examined by FESEM [16]. The crystal gives valuable information about its internal structure such as smooth and continuous surface. Fig. 5 (a) & (b) shows illustrate FESEM photograph of crystal of Barium Cadmium Tartrate crystal. An enlarged FESEM image is shown in fig. 5 (b). This technique combines the resolution and analytical power with ease of operation. Image can be formed form a very wide range of material [17]. These materials can be examined with low energy secondary electron with high energy back scattered electron or with other emission such as light, heat and sound. The high depth of field of the FESEM image makes it. Especially suitable for the study of the fracture and complex microstructure such as found in composite material [18]. Fig. 5 (a) & (b) shows flower like crystal morphology. It shows all boundaries are clear. These crystals are grown by point to point deposition. The corner and edge of the crystal serves as the initiation point for the growth layers [19, 20].

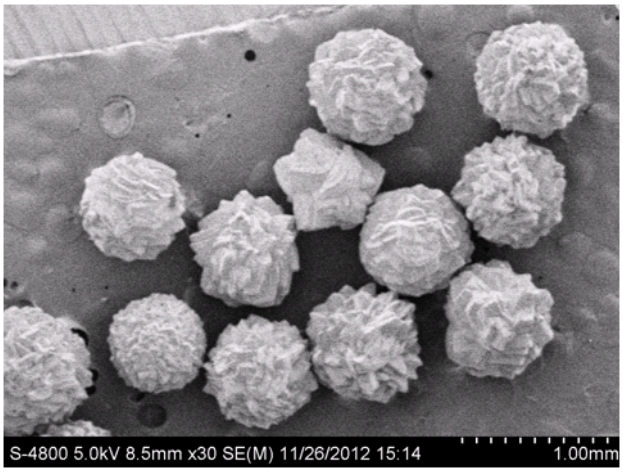

Fig. 5(a) FESEM Image of BCT crystal Fig. 5(b) Enlarged Images of BCT

CONCLUSION

1)Gel method is found suitable for growing barium cadmium tartrate crystal.
2)The growth of single crystal of barium cadmium tartrate crystal was accomplished using single test tube diffusion method. Optimum condition for growth ware worked out.
3)Different habits of barium cadmium tartrate crystal can be obtained by changing parameters like gel density, gel ageing, pH of gel, concentration of reactants etc.
4)EDAX studies revels that grown crystal are BCT indeed. Water of Crystallization present in grown crystal and presence of barium and Cadmiumis confirmed in the crystal.
5)The FESEM Images shows morphology of BCT crystal studies suggested 2-D layer deposition growth.

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