JES Journal of Engineering Sciences

Vol 11, Issue 4 ,April/2020
ISSN NO:0377-9254

COMPARATIVE STUDY OF THE SURFACE MORPHOLOGY OF STRONTIUM DOPED BARIUM TARTARATE AND CADMIUM TARTARATE CRYSTALS BY SILICA GEL METHOD

S. K. Bachhav [1] K.B.Patil [2]

1' Department of physics, Arts, Commerce and Science College Varangaon India. (M.S)
2' Department of physics, Arts, Commerce and Science College Varangaon India. (M.S)

Abstract

Silica gel method is used for growth of strontium doped Barium tartarate and cadmium tartarate crystals by silica gel method . The XRD pattern shows that barium tartarate, Cadmium tartarate crystals are polycrystalline in nature and having orthorhombic, tetragonal structure.. Surface morphology and compassion of materials were studied in scanning electron microscopy and dispersive analysis of x- ray.SEM shows triangular, pentagonal, rod , and plat like shape and broad at the border like structure of the grown crystals The incorporation of strontium in the Barium Tartarate and cadmium tartarate crystals well confirmed by EDAX. and affected significantly by doping.

Keywords : Silica gel technique, XRD, SEM, EDAX.

Crystals habit of various crystals grown under different conditions and also different technique Commercially, the tartrate compound can be used in various applications like as antimony in veterinary drugs [1], ferroelectric applications of sodium-potassium tartrate [2], potassium - chromium tartrate in medicine [3] and where described by Buckley [1], Hartman [2], kern [3], chernor [4], Burton [5]. Number of face with respect to their properties such as dielectric, magnetic, ferroelectric, piezoelectric, optical and other pertinent characteristics [7-12].tors such as degree of saturation, type of solvent [6] pH of the gel media [7] presence of impurities [8] the in growth temperature also presumably affect significantly the morphology of the crystal [9].

1. Experimental work

In the present work, the crystals of barium tartrate and cadmium tartarate with strontium as a diffusible were grown in silica gel by chemical reaction, single diffusion method. Barium chloride , cadmium tartarate and strontium chloride together were taken as the inner reagent and Tartaric acid was used as the outer reagent. To grow well defined crystals of strontium doped Barium tartrate and cadmium tartarate, several experiments were performed by varying the growth parameters like density of gel, gel age, pH of gel and morality of inner and outer reactants, in order to establish the optimum conditions for the growth. The chemicals used in this work are sodium metasilicate $Na_2SiO_3.5H_2O$, acetic acid CH_3COOH, barium tartrate ($BaCl_2$), cadmium chloride ($CdCl_2$) strontium tartarate ($SrCl_2$) (all are AR grade) and tartaric acid ($C_5H_6O_6$), GR grade. Silica gel was prepared by adding the sodium meta silicate solution of specific gravity 1.05 gm/cc drop by drop with constant stirring by using magnetic stirrer into the 5 cc (1M) acetic acid till the pH value 4.2 was set for the mixture. To the above sodium meta silicate solution of pH 4.2, the inner reagent which was the mixture of 15cc and 10cc aqueous solution of 0.05M $BaCl_2$ and 0.05M $CdCl_2$ 0.1M $SrCl_2$ was added with constant stirring.

This mixture was then transferred to the test tube of length 15 cm and 2.5 cm diameter. To keep the solution free from dust and impurities. Care was taken to cover the test tube. The gel was usually set within 5 days. It was left for two more days for ageing and then the outer reagent; the aqueous solution of 0.1M $C_5H_6O_6$ was added on to the top of the gel. The outer reagent was added slowly along the sides of test tube using a pipette and not directly on to the gel medium. Due to the diffusion of the outer reagent into the gel medium and its reaction with the inner reagents, crystals started growing. Nucleation was observed within 24 hours after addition of the outer reagent spherical shape, translucent, prismatic, shining,

crystal aggregates, spherulitic crystals were observed. All experiments leading to the growth of crystals were carried out at room temperature. The reaction between barium chloride and cadmium tartarate, strontium chloride and Tartaric acid in gel medium resulted in the growth of Sr doped barium chloride, cadmium tartarate crystals. Same procedure was adopted to grow 0.05M and 0.1M Sr doped barium tartrate cadmium tartarate crystals.

Observation:

The habits and morphologies of doped crystals were quite similar to that of pure ones [4]. In the case of Sr doped barium tartrate and cadmium tartarate crystals, similar result were obtained but the size of the crystals increases with the concentration of Sr doped.

2. X-ray powder diffraction analysis (XRD)

X-Ray diffraction technique is used to investigate the inner arrangement of atoms or molecules in a crystalline material . The grown $SrBaC_4H_4O_6$ and $SrCdC_4H_4O_6$ crystal where subjected to powder X-Ray diffraction pattern of the grown crystals was obtained using Diffract meter with copper (K alpha 1) radiation of wavelength 1.54056A^0 operating at a voltage of 40 KV and a current of 20 Ma . The scanning rate was maintained at 1.6^0 / min over a 20 orange of 10-80^0 employing the refraction mode for scanning. The power XRD The sharp peaks with maximum intensity characterize the XRD pattern , including the formation of well defined crystallites

XRD study revealed that Strontium barium tartrate crystal belongs to orthorhombic crystal and Strontium Cadmium tartarte tetragonal system. Calculated hkl values were found to be in good agreement with the JCPED card no. 26-0192. and 89-4045. and 2002 JCPDS v. 2.3, 65-1628.. The crystal structure of Sr doped barium tartrate, Cadmium tartarte is determined to be orthorhombic, tetragonal. Revealing that the incorporation of the dopant has not changed the structure of the parent crystal but Cadmium tartrate dopant has changed the structure of the parent crystal.the slight shift of XRD peaks, variations in intensity and lattice parameters of doped Barium tartarate , Cadmium tartarte crystals indicated that dopend are really doped into the $BaC_4H_4O_6$. $CdC_4H_4O_6$ structure.

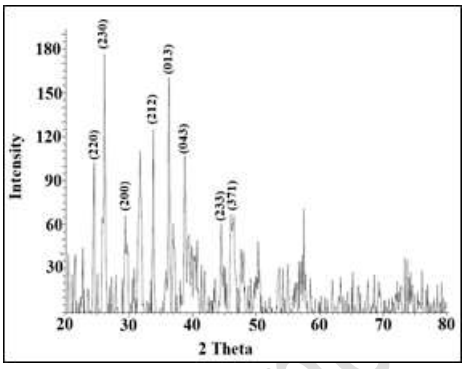

Figure 1. XRD pattern of the crystals of $SrBaC_4H_4O_6$ 0.05M

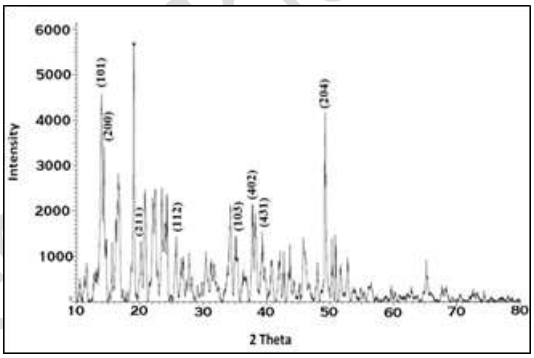

Figure 2. XRD pattern of the crystals of $SrCdC_4H_4O_6$ 0.05M

Table 1. XRD data of 0.05M Sr doped barium tartrate crystals λ =1.54060Å

Samples	wt % obtained from EDAX		at % obtained from EDAX		Ag/S (at %)	Remark
	S	Ag	S	Ag		
B1	30.65	69.35	59.79	40.21	0.673	NS
B2	25.78	74.22	53.88	46.12	0.856	NS
B3	20.70	79.30	46.75	53.25	1.139	NS
B4	16.99	83.01	40.78	59.22	1.452	NS
B5	12.78	87.22	33.02	66.98	2.028	S
B6	11.48	88.52	30.27	69.73	2.303	NS
B7	9.19	90.81	25.39	74.61	2.938	NS
B8	4.60	95.40	13.95	86.05	6.168	NS

Journal of Engineering Sciences
Vol 11, Issue 4 ,April/2020
ISSN NO:0377-9254

Table 2. XRD data of 0.05 M Sr doped Strontium tartrate crystals λ =1.54060Å

Observed Values from XRD			Observed Values from XRD		
Sr No	2θ	d-Values	2θ	d-Values	h k l
1	24.600	3.6159	24.606	3.6149	2 2 0
2	26.000	3.4243	26.009	3.4230	2 3 0
3	29.300	3.0457	29.374	3.0382	2 0 0
4	33.700	2.6574	33.718	2.6560	2 1 2
5	35.900	2.4994	35.936	2.4969	0 1 3
6	38.800	2.3190	38.887	2.3140	0 4 3
7	44.600	2.0299	44.692	2.0260	2 3 3
8	46.300	1.9594	46.308	1.9590	3 7 1

3 . Scanning electron microscopy (SEM)

In the present work powdered sample of $SrBaC_4H_4O_6$ crystals were examined by using LEICA S440 SEM instrument at the North Maharashtra University Chemical technology Laboratory, Jalgaon. Figure 3 illustrate the SEM images of single crystals of 0.05M and 0.1M Strontium doped barium tartrate and cadmium tartarate crystals respectively.

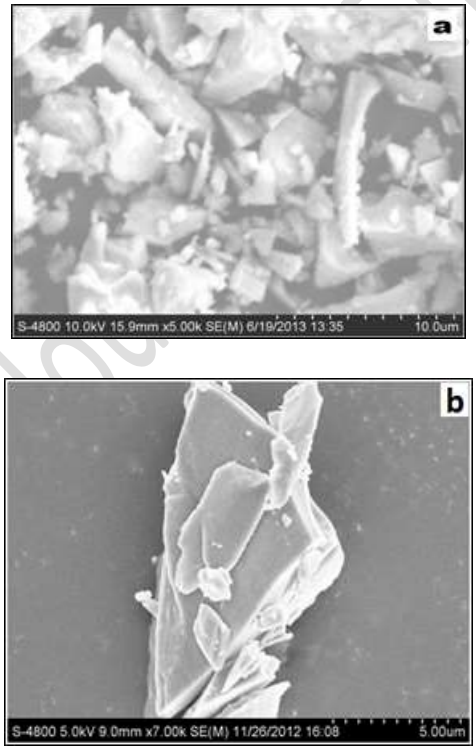

Figure 3. SEM picture of 0.05M Sr-doped SrBaC4H4O6 and $SrCdC_4H_4O_6$ **crystals**

All SEM photo mages shows in fig 3. triangular, pentagonal, rod and plate like crystals morphology and crystals are grown by layer deposition. Broad layers are seen in figured. The individual plates of samples are flat and the plates with the broad edges were observed. On some plates further plate like growth was observed [9]. It was found that morphological changes take place due to doping.

4. Energy dispersive X-ray spectroscopy (EDAX):

The EDAX spectrum is just a plot of how frequently an X-ray is received for each energy level. An EDAX spectrum normally displays peaks corresponding to the energy level for which the most X-rays had been received. Each of these is unique to an atom and therefore corresponds to a single element. Figure 5 shown EDAX spectrum of crystals of barium tartrate and cadmium tartarate doped by Strontium at 0.05M and 0.1M concentration respectively. The peaks show the presence of Ba, C, O, and Cd, Sr, Cd. This is a clear indication of presence of the strontium doped in the crystals.. It was observed that Mass% and atomic % of Ba, Cd and Sr are shown in fig 4 and 5.

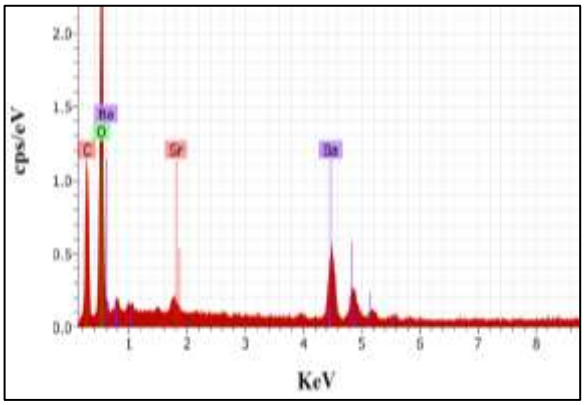

Figure 4. Energy dispersive of 0.05M Sr doped barium tartrate crystal

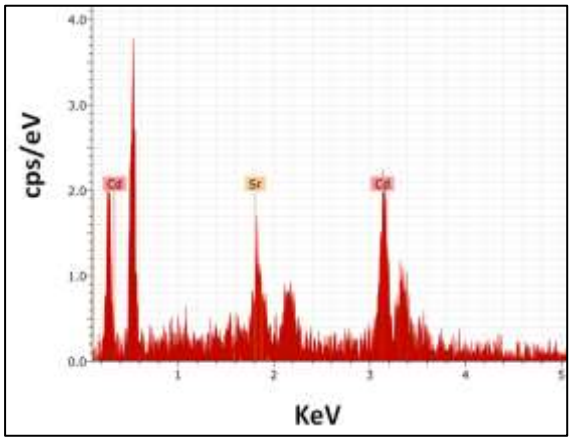

Figure 5. Energy dispersive of 0.05M Sr doped cadmium tartrate crystal

The spectrum of EDAX is shown in figure 5 and 6. The average atomic percentage was found as Sr 3 48 and Ba 40.87 , Sr = 28.87 and Cd = 71.13.

Conclusions:

From the experimental studies, following conclusions are drawn:

1) Signal diffusion method is convenient for the growth of the Strontium doped barium tartrate and Cadmium tartarate crystals.
2) Spherically, translucent, shinning, crystal aggregate, spiky spherulitic, good quality sized crystals were obtained. Size of the doped crystals increases with the concentration of Sr dopant.
3) The powder X-ray diffraction study confirmed that grown crystals are very much crystalline in nature having Strontium barium tartrate is orthorhombic structure incorporation of the dopant has not altered the structure of the parent and Strontium Cadmium tartrate tetragonal structure Strontium Cadmium tartrate incorporation of the dopant has altered the structure of the parent crystal.
4) As a result of Sr doping, the XRD peak value shifts toward lower angle, indicating an increase in the value of lattice constants.
5) The grain size of the Strontium barium tartrate and Strontium Cadmium tartrate crystals increases on Sr doping and subsequent doping shows increasing tendency in grain size.
6) Surface morphology was affected significantly by the doping.
7) EDAX confirms the presence of strontium in the crystals.

References:

1] Jenkins R.X-RAY fluorescence Spectrometry, 2nd Edition, Wiley inters science, (1999).
2] Behrke V.E., Deane K.S., Jenkins R. and Smith D.K., A practical Guide for the preparation of Specimens for X-RAY fluorescence and X-Ray Diffraction analysis.
3] Cullity B. D. Elements of X-ray Diffraction, 2ndEdition.
4] A. B. Patil and K. B. Saraf, Phy. D Thesis- "Growth and Characterization of Doped and undoped Lead Iodide single Crystals", (2003).
5] http://en.Wikipedia.org/ wiki/ Second-harmonic generation.
6] Franken P. Hill, A. Peters C. Weinreich, G. Physical Review Letters, (1961), 7, 118.
7] http://www.rp-photonics.com/frequency_doublling.html.
8] Chatwal G. and Anand S. Instrumental Methods of Chemical Analysis, Himalaya Book Publishing House (1984).
9] Mitra S. Fundamentals of Optical, Spectroscopic and X-ray Mineralogy, Wile Eastern Limited (1989).
10] Chanda M. Atomic Structure and Chemical Band, 3rd Edition, Tata McGraw-Hill publishing company Ltd. New Delhi, (1994)
11] Williams D. H. and Fleming I. Spectroscopic Methods in Organic chemistry 4th edition, Tata McGraw-Hill. Publishing Company Ltd., New Delhi, (1987).
12] Little L. H. Infrared Spectra of Adsorbed Species, Academic Press, London. (1967)