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Archives of Physics Research, 2015, 6 (1):26-30
(http://scholarsresearchlibrary.com/archive.html)

Archives of Physics Research
Scholars Research Library
ISSN : 0976-0970
CODEN (USA): APRRC7

Synthesization and structural studies of Barium Tartrate crystals by silica gel technique

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ABSTRACT

In the present research work, single crystals of Barium tartrate crystals were grown by a simple gel technique using diffusion method. The optimum growth conditions were optimized by varying various parameters such as pH of the gel solution, gel concentration, gel setting time, concentration of the reactance. The crystalline nature of the grown crystal was confirmed using powder X-ray diffraction techniques (XRD). Surface morphology and compassion of materials were studied in scanning electron microscopy (SEM), and dispersive analysis of X-ray (EDAX).

Key words: Crystals growth technique, XRD, SEM, EDAX.

INTRODUCTION

Many investigators were found that the semiconductor nanoparticles exhibit structural, electronic, optical, luminescence and photoconducting properties very different from their bulk properties. It is also very attractive because of their possible application in solar cell, photo detector, laser, LED, high-density magnetic information storage and many others insemiconductor industries [1-7].

Non-linear optical (NLO) materials have great impact on information, optical computing and optical communication technology and could be used to double or triple the frequency of laser light for high speed processing of data [8]. N.Vijayan et al [9] reported that non-linear optics has wide applications in the field of telecommunication and optical information storage devices. Which exhibits optical and electronic properties has been reported and grow by G. Madhurambal and P. Anbu Srinivasan [10]. The new type of hybrid NLO materials have been explored from organic-inorganic complexes with stronger ionic bond such as L-arginine phosphate monohydrate (LAP) and others [11-12] have been grown as a single crystal. The S.H.G. efficiency of gel grown BGHC was found to be five times that of potassium dehydrogenate phosphate (KDP) [13]. Using gel techniques grew many tartrates, oxalates and iodated by researchers. Calcium tungstate is the most widely used phosphor in industrial radiology and medical diagnosis [14-16], was grown by S.K.Arora and B.Chudasama [17] using flux method. The DL-Methionine [$C_5H_{11}NO_2S$] is one of the essential amino acids in humans. It has two crystalline forms, viz., α- and β-methionine. Ramachahdran and Natarajan have grown β-DL-methionine in silica gel [18].

MATERIALS AND METHODS

Gel was prepared by mixing the solutions10 ml solution of Na_2SiO_3 (1M) was take in beaker and in that solution acetic acid was added. Then this mixture was stirred by the magnetic stirrer. In this stirring process, the $BaCl_2$ solution of 1M was added drop by drop. This process is continued till the solution becomes milky and 4 pH of the

solution obtained. This solution was transferred in glass tube of diameter 2.5cm and 15cm in length. The mouth of tube was covered by cotton plug. The tube was kept for the setting. After setting the gel, it was left for aging. After two days the supernatant of 1M concentration was poured over the set gel by using pipette. These tubes were kept undisturbed by covering the cotton plug on the mouth of tubes. After two day pouring the supernatant, next day the small nucleation growth was observed at below the interface of gel. This nucleation growth was increased in size. Good quality spherulites were grown in 45 days. In single diffusion, after a few days spherulitic, and good quality crystals growth of Barium tartarte crystals. The grown Barium tartrate crystals were observed in small size. The reaction taking place is as follows;

$2CH_3COOH + Na_2SiO_3 \rightarrow 2CH_3COONa\downarrow + SiO + H_2O$

$2CH_3COONa + BaCl_2 \rightarrow (CH_3COO)_4 Ba \downarrow + 2NaCl$

$(CH_3COO)_4 Ba + C_4H_6O_6 \rightarrow C_4H_4BaO_6\downarrow + 2CH_3COOH$

The optimum conditions for growing crystals are given in table 1.

Table 1.optimum condition for growth of Barium tartarte crystals

Condition	Single diffusion
Density of sodium meta silicate	1.05 gm/cm^3
Amount of 1M acetic acid	5 ml
pH of the gel	4.0
Temperature	Room Tempwrature
Concentration of Barium Chlorid	14 ml
Concentration of 1M Tartaric acid	5 ml
Gel setting period	3 days
Growth of period	45 days
Size of Crystals	3.5mmX2.5mmX2.5mm

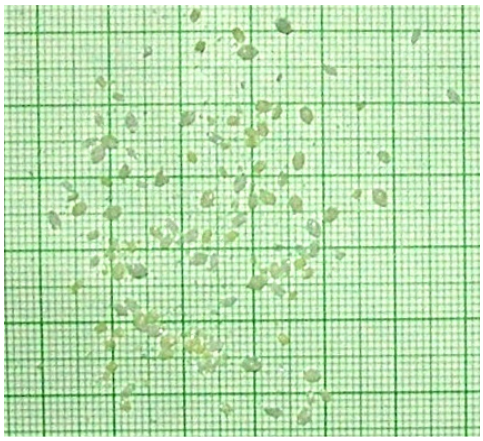

Figure 1: White coloured opaque crystals of Barium tartrate crystals

Figure 2: Crystals growth in the test tube

RESULTS AND DISCUSSION

The grown BaTr crystals were subjected to the single crystals X-ray diffraction. Studies to confirm orthorhombic nature and the values of the lattice parameters obtained from single crystal XRD studies and identify the diffraction planes, powder X-ray diffraction pattern of the powdered sample was obtained using a powder X-ray diffractometer (Bruler, D-Advance) having wavelength of λ=1.54060 Å. The sample was scanned over the range of 20^0 - 80^0 for 2θ values. Surface micro structure was investigated by Scanning electron microscopy [SEM) by using EVO 50. The quantitative compositional analysis of the Barium Tartrate crystal was carried out by EDAX. (Energy dispersive X-ray Analyzer) technique attached with SEM.

3.1 X- Ray Diffraction Analysis

The crystal structure of the sample compound was studied by powder X-ray diffraction method by the instrument Miniflex-Rigaku model Japan with CuKα radiation of wavelength λ=1.54056Å. The recorded diffraction pattern of the BaTr crystals is shown in the figure 3 and XRD data of barium tartrate crystal shown in table 2.

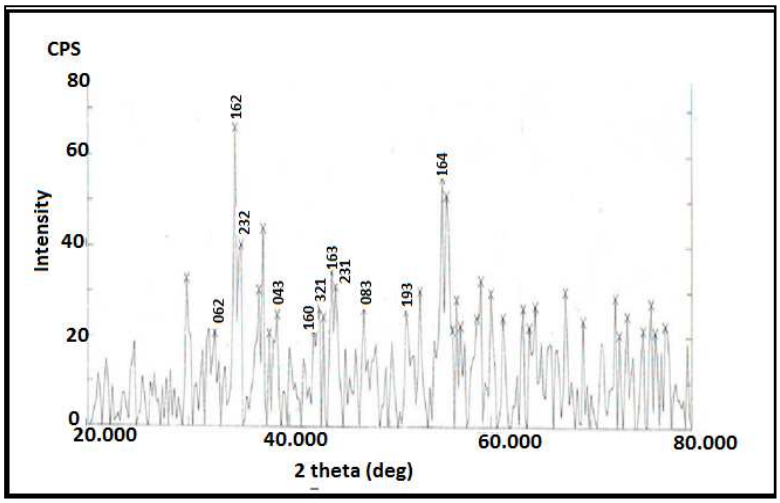

Figure 3: XRD pattern of barium tartrate Crystal (pH 4.2)

Table 2: XRD data of barium tartrate crystal λ =1.54056Å

observed data values			standard data			
2θ	*d-value*	*Intensity*	*2θ*	*d-value*	*Intensity*	*h k l*
32.600	2.7444	21	32.765	2.7310	16	0 6 2
34.600	2.5902	66	34.868	2.5710	25	1 6 2
35.200	2.5474	40	35.393	2.5340	12	2 3 2
38.800	2.3189	25	38.887	2.3140	6	0 4 3
42.400	2.1300	21	42.339	2.1330	2	3 6 0
43.00	2.0832	26	43.210	2.0919	2	3 1 2
43.400	2.0832	24	43.384	2.0840	12	2 9 2
44.200	2.0473	34	44.323	2.0420	2	1 6 3
44.600	2.0299	31	44.692	2.0260	2	2 3 3
47.400	1.9163	26	47.331	1.9190	8	0 8 3
51.600	1.7698	26	51.562	1.7710	1	1 9 3
55.200	1.6626	55	55.330	1.6590	2	1 6 8

Determination of Grain size from XRD spectra

The grain size is determined by measuring the width of the line with highest intensity peak and calculated by using the formula:

Grain size $D = 0.9 \lambda / \beta \cos\theta$

Where, β is full width of half maxima in radian and D is grain size of the crystal.

The calculated average grain size is 35.42 nm. The analysis of different diffraction peaks indicates the formation of orthorhombic system. By measuring the peak heights above the background in nm and scaling the value up so that the tallest peak has a value of 55 and 66. The preferential orientation is observed from the XRD data is (168) and (162) indicating maximum growth of the crystal in that direction. Orientations of the

crystallites along different h, k and l value were present. The intensity of different peaks could give the relative orientation of a particular h, k and l of plane. The observed d-values and h, k and l plane were compared with standard data of 2002 JCPDS v. 2.3, 26-0192.

3.2 Surface morphology by scanning electron microscopy

In present work Scanning electron Microscopy of powdered sample of gel grown Barium Tartarte was carried out at Chemical Technology, N.M.U., Jalgaon and successive photograph were taken at the magnifaction of 5.00,40.0,50.0 um all the photograph were taken at common width 9.2mm and EHT magnifaction 1.0KV.
The figures 4(a,b,c,d,) illustrate SEM photographs of Barium tartarate crystals. It was found that morphology of the crystals. Triangular, Pentagonal, rod and cubic like shape and broad edges layers are seen in figures. The individual plates of samples are flat and the plates with the broad edges were observed. Small quantities of impurity or excess or deficiency native species can be accommodated in the crystal lattice without break down of the growth surface. However, there is a high probability that the incorporation will be spatially non-uniform. This shows that the mechanism of this growth in related of the mean crystals. Same time further plate like growth in observed.

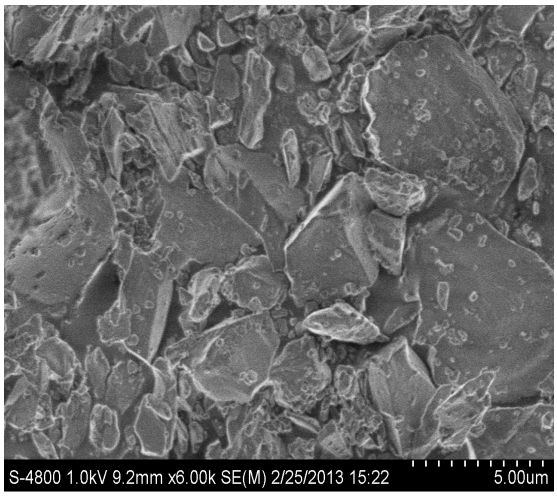

Fig 4.(a) BaC4H4O6

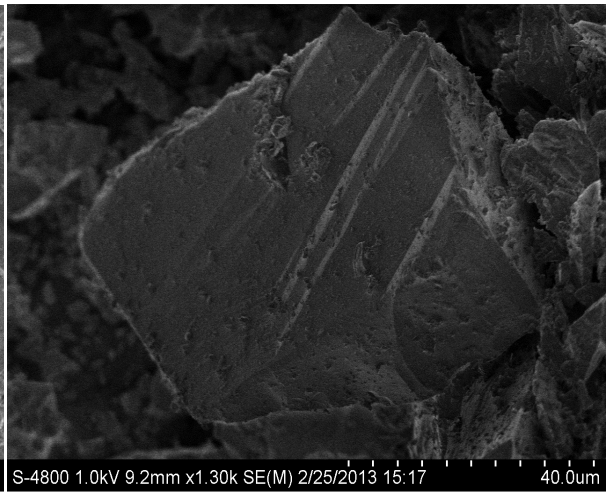

Fig 4.(b) bAc4h4o6

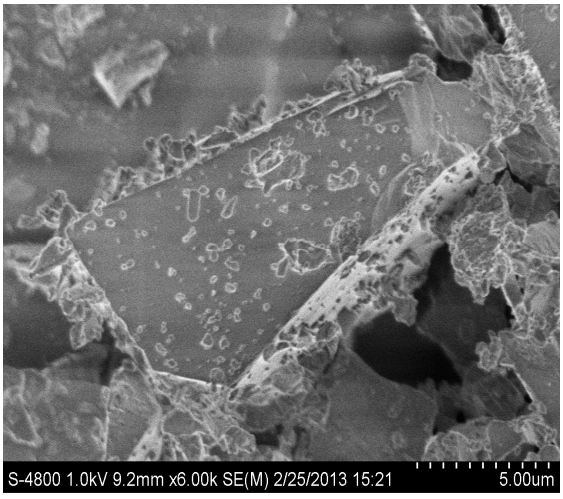

Fig 4.(c) BaC4H4O6

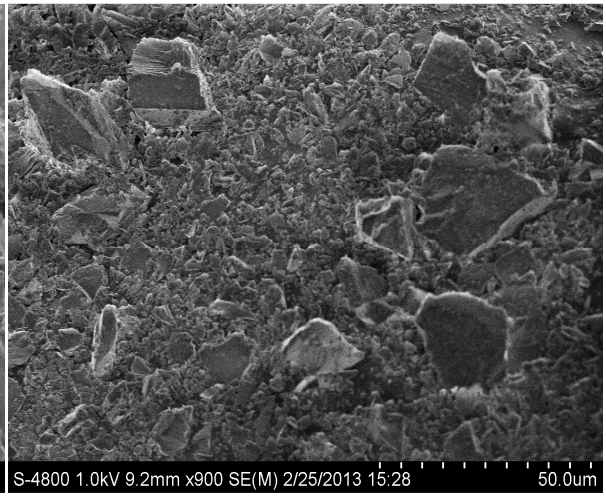

Fig 4.(d) BaC4H4O6

3.3 EDAX

The compositional analysis of barium tartarate crystals is carried out by using EDAX analysis. EDAX pattern of barium tartarate crystals is shown in Fig 5. The EDAX confirms the presences of Barium (Ba), Carbon (C) and Oxygen (O) with their atomic percentage ratio 14.98 : 37 : 47.46 : respectively. Recorded EDAX spectrum revels that there is no evidence of other impurity. It indicates purity of crystals. From the table it is clear that the values (weight% and atomic %) of C, O and Ba in the grown crystal measured by EDAX are very close with the values calculated from the molecular formula.

Table 3. Experimental and calculated composition obtained from (EDAX) various constituent elements present in barium tartrate crystals

Elements	Line	Experimental values		Theoretical values	
		Mass[%]	Atomic[5]	Mass[%]	Atomic[%]
C	K	13.81	37.56	16.27	36.84
O	K	23.24	47.46	21.69	49.12
Ba	L	62.95	14.98	62.04	14.04

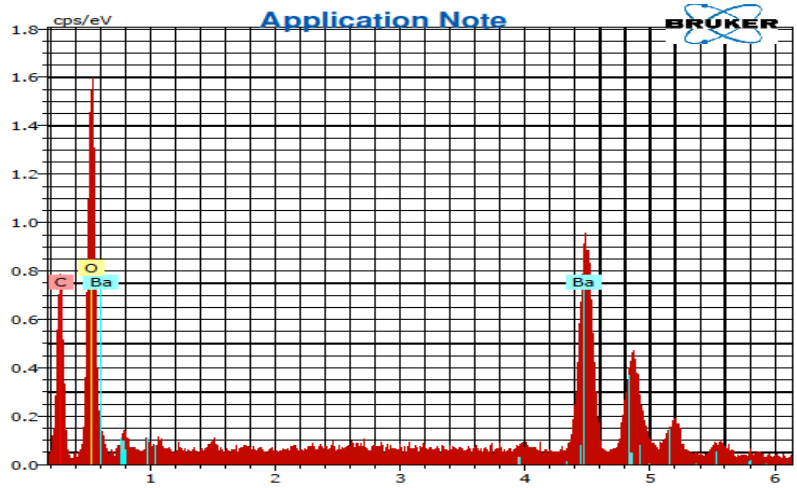

Figure 5. EDAX spectrum recored for barium tartrate crystals

CONCLUSION

Gel method is found suitable for growing Barium tartrate crystals. The growth of single crystals of Barium tartrate was accomplished using the single test tube diffusion method. Different habits of Barium tartarate crystals can be obtained by changing parameters like density, gel aging pH of gel, concentration of reactants, etc. Single crystals of BaTr were grown by controlled diffusion of Ba^{2+} using the silica Gel. BaTr is required to maintain the pH of the gel. The parameters such as temperature affect the growth of BaTr Crystal. Gel growth technique suitable for growing Crystals of BaTr. The grown BaTr crystals were in small size. The structure of BaTr is the semitransparent platy shape and spherulitic. Unit cell parameter values match very well with the reported XRD standard Data values. The BaTr crystals are shining and quite transparent and they are of good quality. SEM photographs shows plate, triangular, pentagonal, and rod like crystal morphology. Chemical compositions of the grown crystal by EDAX show good agreement with the theoretical values and calculated values from molecular formula.

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