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Advances in Applied Science Research, 2014, 5(6):66-71

ISSN: 0976-8610
CODEN (USA): AASRFC

Crystal growth and characterization of strontium doped barium tartrate crystals by silica gel method

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ABSTRACT

The single crystals of Strontium doped Barium tartrate crystals were grown by Single diffusion technique, in silica gel at room temperature. Effect of Strontium doping on the Barium tartarate has been studied. The XRD pattern shows that Strontium barium tartarate crystals are polycrystalline, having orthorhombic structure. SEM pictures reveal that crystals are grown by layer deposition. The elemental analysis was done by EDAX. The chemical analysis is done by FTIR to denote the functional group of grown crystal. The thermal stability was studied by the TGA, DTG and DSC.

Keywords: Single diffusion, XRD, SEM, FTIR, THERMAL ANALSYSIS.

INTRODUCTION

Now a day's crystal growth is the rapid growing field in research because of huge demand of crystalS for several applications and recently researchers focused on the tartrate. 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 so on. Many people studied various tartrate compounds likes calcium-strontium mixed levo tartrate [4], zinc tartrate [5] & cadmium tartrate [6] with respect to their properties such as dielectric, magnetic, ferroelectric, piezoelectric, optical and other pertinent characteristics [7-12].

Many researchers grow the series of pure and mixed crystals to find out the new material for various purposes [13-17]. There are various techniques for growing crystals like melt growth, vapour phase, solution growth and etc. The gel technique has attracted more attention towards it because of its simplicity and cost effectiveness. The crystals can be grown at room temperature.

Barium Tartrate (BaTr) is a quite interesting compound and hence some attempts have been made to grow Strontium doped Barium tartarate crystals. Here Strontium doped barium tartaric crystals are grown by gel technique in silica gel medium and the as grown crystals analyzed under the various characterizations.

MATERIALS AND METHODS

The Strontium doped barium tartarate crystals were grown by single diffusion method in silica gel medium at room temperature. The sodium Meta silicate solution and acetic acid was prepared by dissolving 22 gm sodium Meta silicate in to the 250ml distilled water and 15 ml acetic acid dissolving in to 250 ml distilled water respectively. Then sodium Meta silicate added in to 6 ml acetic acid drop by drop by maintaining the pH 4.2 with continues starring. After that 15 ml solution of $BaCl_2$ with 0.1M and 10 ml solution of $SrCl_2$ with 0.05M added in to the gel solution.

This mixture was then transferred to the test tube of 15 × 2.5 cm dimension. The open end of the tube was closed with cotton, to prevent evaporation and contamination of the exposed surface and stored the tubes at room temperature.

After setting the gel in 4-5 days, the 10 ml tartaric acid with 1M allow to fall steadily along the wall of the tube above the set gel, to prevent the gelled surface from cracking. Crystals were visible within about a week and well shaped crystals grow approximately within one month. Figure 1 shows the Strontium doped barium tartarate crystals. The reaction between Barium Chloride, Strontium Chloride and Tartaric acid in gel medium resulted in the growth of Sr doped Barium Chloride crystals.

$CH_3COOH + Na_2SiO_3 \rightarrow 2CH_3COONa\downarrow + SiO + H_2O$
$2CH_3COONa + BaCl_2 + SrCl2 \rightarrow (CH_3COO)_4\,BaSr\downarrow + 4NaCl$
$(CH_3COO)_4\,BaSr + C_4H_6O_6 \rightarrow C_4H_4BaSrO_6\downarrow + 4CH_3COOH$

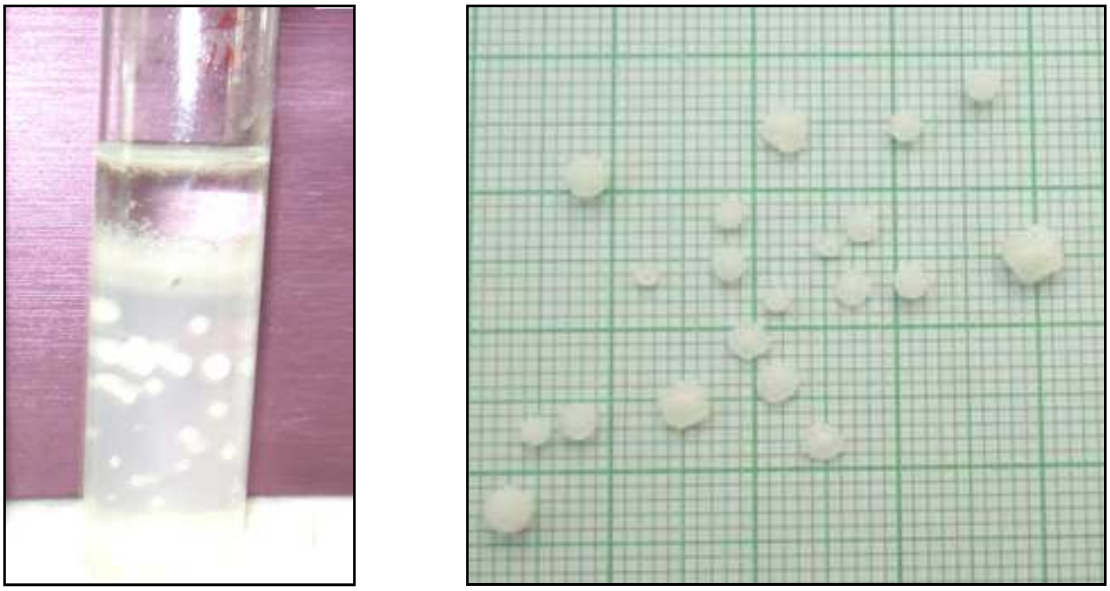

Fig.1 0.05M Sr doped of Barium tartarate crystals

As grown crystals were characterized for structural, morphological, elemental and thermal properties. The structural characterisation of sample was carried out by X – Ray diffractometer (Bruker - CuKα radiation) within the 2θ range of 20° - 80°. Surface morphological study was carried out by using Scanning Electron Microscope (Zeiss EVO 50), operating with an accelerating voltage 10 KV. The elemental analysis of the sample was carried out by Energy dispersive X-ray Analyzer (EDAX) attached with SEM. The FT-IR spectrum of the powdered crystalline sample in KBr medium was recorded within 450 to 4000 cm^{-1} by using "Perkin Elmer model 783". The thermal decomposition behaviour of grown crystals was studied by thermo gravimetric analysis (TGA) and differential scanning calorimeter analysis (DSC) within the range of 27 to 600 °C at 10 ºC/min heating rate in the nitrogen atmosphere.

RESULTS AND DISCUSSION

3.1. XRD Analysis

The XRD pattern of 0.05M Strontium doped barium tartarate crystal grown by single diffusion technique is shown in figure 2. The XRD pattern reveals that sample is polycrystalline in nature having orthorhombic phase. The 2Θ peaks observed at 26.00°, 27.60°, 31.10°, 33.70°, 35.20°, 36.10°, 38.60°, 40.70°, 44.50°, 51.90° and 53.30° which corresponds to the (230), (102), (171), (212), (232), (103), (112), (262), (233), (211) and (114) plane of reflection respectively. These results are well in agreement with JCPDS data card no (01-1278 and 26-0192). The grain size was found to be 47.83 nm by using Debye–Sherrer's formula:

$$D = \frac{0.94\,\lambda}{\beta \cos\theta}$$

Where, λ is the X-ray wavelength, θ is the Bragg angle and β is full width at half maxima. The lattice parameters a, b, c and volume was found to 7.59, 23.78, 7.53 and 1360.17Å respectively.

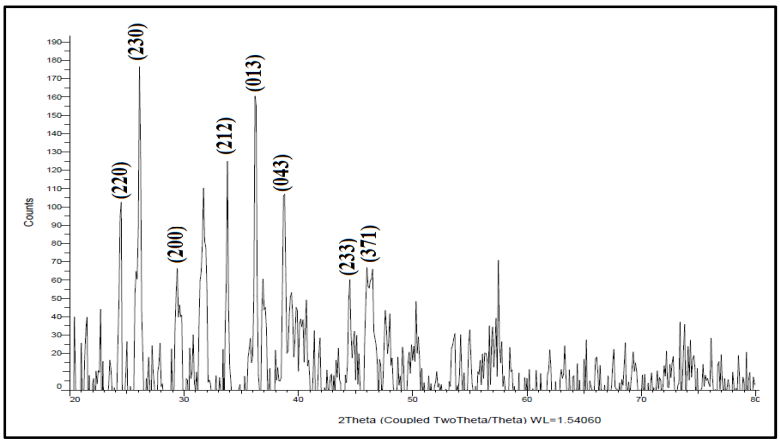

Fig. 2 XRD spectrum of Sr-doped Barium tartrate crystals

3.2. Scanning electron microscopy (SEM)

The morphology of Strontium doped barium tartarate was studied by Scanning electron microscopy (SEM). Figure 3 illustrates the SEM images of same sample.

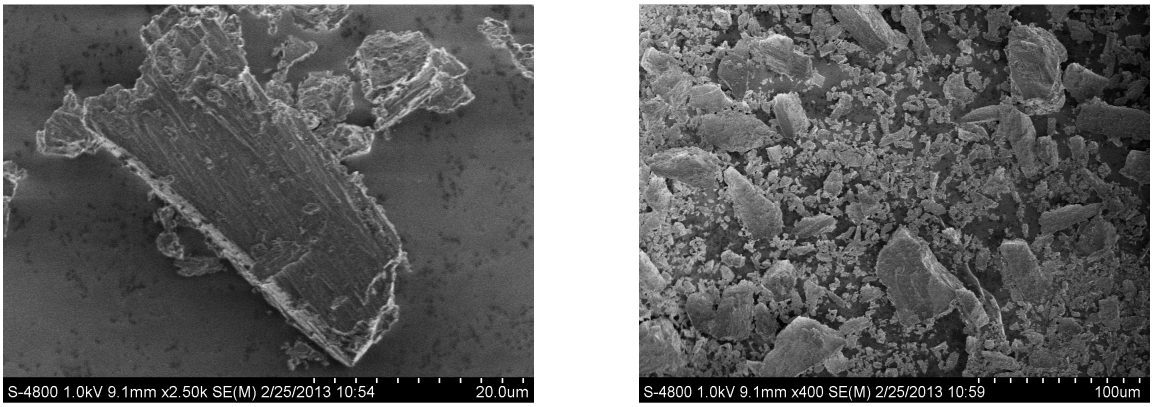

Fig. 3a-b. SEM picture of 0.05M Sr doped Barium tartarate crystals

The SEM photographs shows crystals are grown by layer deposition having triangular, pentagonal, rod and plate like crystals morphology. The individual plates of samples are flat and the plates with the broad edges were observed. It was found that the morphology of the crystals was affected significantly by the doping.

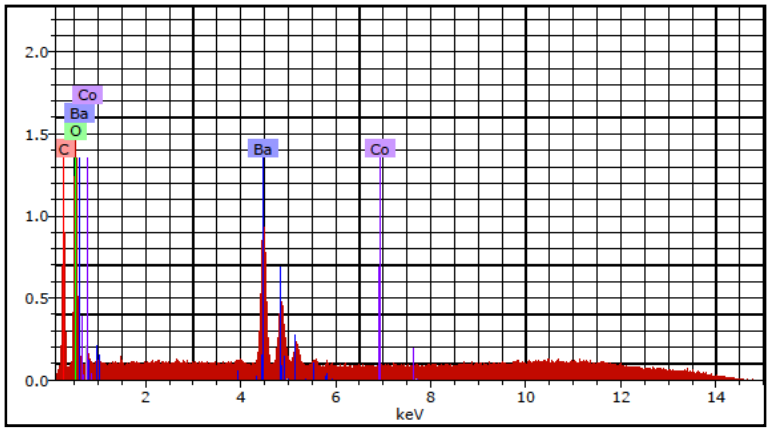

Fig. 4 EDAX spectrum of 0.05M Sr doped Barium tartarate

3.3. EDAX Analysis

The elemental analysis of grown crystal has been done by EDAX in binding energy region within 0 to 15 KeV. Figure 4 shows the EDAX pattern of Strontium doped Barium tartarate crystals. The spectrum shows the detection of expected elements. The atomic percentage of present element C, O, Co and Ba was found to be 36.07, 55.55, 1.08, 7.29 percent respectively.

3.4.FT-IR

The functional group of Strontium doped barium tartarate crystal involved in vibration frequency have been identified using FTIR spectroscopy. The FTIR spectrum of gel grown single crystal is shown in figure 5. The spectrum shows the peaks within the range of 450 to 4000 cm^{-1}.

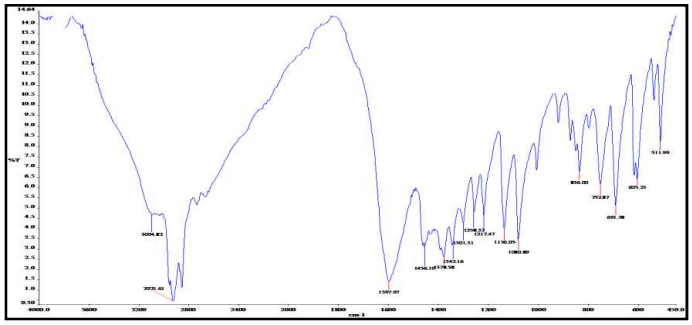

Fig. 5 FTIR Spectrum of Sr: BaC4H4O6 0.05M

The FTIR spectrum recorded for Sr: BaC4H4O6 crystal with observed band is shown in the figure 5. The spectrum is scanned in the region 450 to 4000 cm^{-1} using "Perkin Elmer model 783". The bands around 3379 to 2926 cm^{-1} are attributed to asymmetric and symmetric–OH stretching of water [10-13]. The–OH stretching frequency of Sr: BaC4H4O6 appeared at 2926 cm $^{-1}$.The moderate absorption around 3379to 2926cm^{-1} is probably due to stretching vibration of alkali group. The presence of the –C–O– group is indicated by the occurrence the sharp and intense band at 1598cm^{-1} and 1463cm^{-1} indicate asymmetric –C–H bending. The peaks at 1377 cm^{-1} is due to –O-CH stretching mode. These bands may be assigned respectively to –C–O– asymmetric and symmetric stretching of –C–O–. The bonding mode of water of crystallization overlaps with the new asymmetric –C–O– frequency band. i.e. the region of for the broadness of the absorption around 1598 cm-1. The inplane bonding of –C–H is assigned by the peak at 1463 cm-1. The absorption at 1136 cm-1 are probably due to –O–H bending while –C–OH stretching vibration respectively which represents the co-ordinate –C–OH group. With the help of the assignment made above, 1079 represents C-O stretching shows sharp peaks absorption at 1007 cm^{-1} indicate that C-O bond stretching in alcohol –C-OH group. The absorption siuated below 838 cm^{-1} are due to barium-cobalt-oxygen stretching vibrations table1 shows the assignments of FT-IR spectrum.

Table1: FTIR data of Sr doped Barium tartarate crystals

Sr. No	0.05M	Assignment
1	3379-2926	-OH stretching and water of crystallization.
2	1598	C=O stretching vibrations
3	1463	-C-H indicated asymmetric bending
4	1377-1218	C-O stretching Vibration.
5	1136-1079	C-H stretching vibration.
6	921—513	Metal Oxygen (Ba-Sr-O) stretching vibrations

3.5. Thermo gravimetric analysis TGA

The thermal stability of the grown crystals and the number of water molecules associated with grown crystals determined by conducting thermo gravimetric analysis (TGA).

Figure 6 shows the TGA pattern of grown crystal. The TGA data in the different stages of decomposition are presented in table 2.

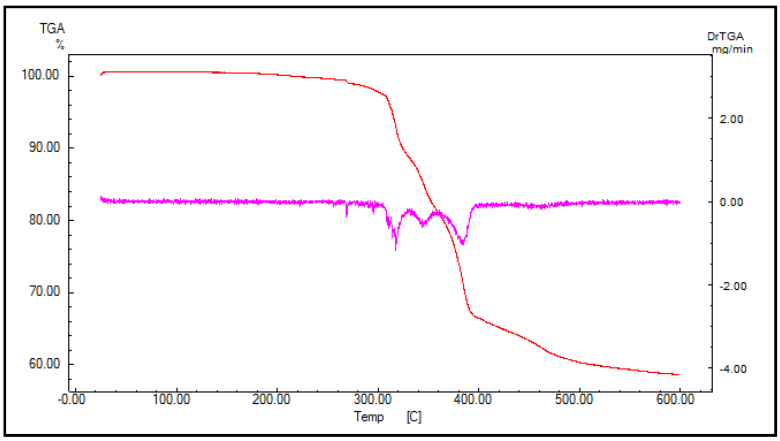

Fig. 6. Spectrum of $SrBaC_4H_4O_6$ 0.05 M Crystals

In stage I, it was observed that in the temperature range of 27.78 – 260 °C in which weight loss of 1.75 %, agrees very well with the calculated weight loss of 2.36 %. It is clear that Strontium barium tartrate crystals are hydrated and the weight loss calculation clearly indicates that Strontium barium tartrate crystals have $0.5H_2O$ water loss

In stage II, In the temp range of 260- 360 °C, the total weight loss of 15.72 % is seen, which is due to the loss of 2CO and $2H_2$ this well arrangement with calculated weight loss of 15.70 %. Then anhydrous Strontium Barium tartarate decomposes into Strontium Barium oxalate. Strontium barium oxalate stable within the temperature range 260-360 °C.

In stage III, the total weight loss of 11.53% was observed in the temperature range 360-400 °C which corresponds to the loss of CO_2. This weight loss aggress very well with the calculated weight loss 11.51% and decomposes into Strontium barium carbon dioxide.

In stage IV, total weight loss of 7.28% was observer within 400- 600 °C. This loss is attributed to the loss of CO. This is in well agreement with calculated weight loss of 7.33 %. Thus the Strontium barium carbon dioxide finally turns into Strontium barium oxides at 600 °C.

Table2: TGA data of 0.05 Strontium Barium tartarate crystals ($SrBaC_4H_4O_6$)

Stage	Temperature range	Observed % weight loss	Calculated % weight loss	Loss of molecule in stage
I	27.78-260^{0}C	1.75	2.36	-0.5H2O
II	260-360^{0}C	15.72	15.70	-2CO 2H2
III	360-400^{0}C	11.53	11.51	-CO2
IV	400-599^{0}C	7.28	7.33	-CO

3.6. Derivative thermo gravimetric analysis (DTG): DTG

The loss of molecules can be determined by using DTG analysis. The figure 6 shows the spectrum of DTG.

1. In the first stage of decomposition, Major endothermic peak at 255 °C is attributed to loss of $0.5H_2O$. The peak observed in the DTG Curve corresponds to the weight loss 2.36% in the TGA curve.

2. The endothermic peak at 305 °C and 320°C in the second stage of decomposition is attributed to loss of 2CO and H_4. The peak observed in the DTG curve corresponds to the weight loss 17.65% in the TGA curve.

3. The endothermic peak at 365 °C in the third stage of decomposition is attributed to loss of CO_2.The peak observed in the DTG curve corresponds to the weight loss 12.86% in the TG curve.

4. In the fourth stage the endothermic peak 401°C decomposition is attributed to loss of CO. The peak observed in the DTG curve corresponds to the weight loss 8.05% in the TG curve.

Beyond the 600 °C, the reaction proceeds and finally stable residue $Sr:BaC_4H_4O_6$ remains up to the end of analysis.

3.7. DSC Analysis

The DSC analysis of the grown crystals was recorded between 20-400 °C in the nitrogen atmosphere. The figure 7 shows the DSC curve for Sr doped Barium tartarate crystals. The DSC analysis has been done in two stages. From the DSC curve the endothermic peaks are found in both stages. The DSC data is represented in table 3.

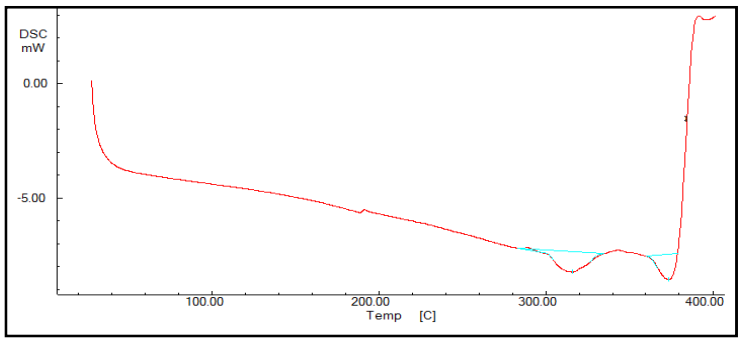

Fig. 7 DSC curve of Sr doped barium tartarate Crystals

Table3: DSC data of Sr doped Barium tartarate Crystals

Stage	Peaks	Temperature °C	On set °C	Endset °C	Heat Mj
I	Endothermic	316.21	301.64	330.50	-119.11
II	Endothermic	373.61	363.43	378.95	-70.82

COLLUSION

The Strontium doped barium tartarate crystals were grown by single diffusion method. The XRD illustrates that the grown crystals are polycrystalline nature and orthorhombic phase. SEM pictures shows crystals are grown by layer deposition. The incorporation of Sr in the crystalline of Barium tartarate is well confirmed by EDAX. The chemical analysis is done by FTIR to denote the functional group of grown crystal. The thermal stability was studied by the TGA, DTG and DSC.

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