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Research Article

COMPARATIVE ANALYSIS ON OPTICAL, THERMAL AND STRUCTURAL CHARACTERIZATION OF BARIUM TARTRATE CRYSTAL GROWN IN THE PRESENCE AND ABSENSE OF MAGNETIC FIELD

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ABSTRACT

There are many reports presenting the growth and influence of various parameters such as gel density, concentration of reactants etc on the growth mechanism of barium tartrate crystal. Characterization on single crystals of barium mixed strontium tartrate crystals and barium mixed calcium tartrate tetrahydrate crystals are reported. To the best of our knowledge there are no literature available on barium tartrate crystals grown under the influence of magnetic field. In this paper we report the comparative analysis of the optical, thermal and structural properties of barium tartrate crystals grown in the presence and absence of magnetic field. There is small variations in the refractive index, bandgap energy, PXRD pattern and cell parameters.

INTRODUCTION

The gel method for growing single crystals offers the advantage of a room temperature growth technique and is particularly suited to the preparation of materials which dissociate, decompose or react with the container at elevated temperature[1]. There are reports on the growth of crystals under different growth conditions such as variations of concentrations of inner and outer reactants, pH of gel and application of magnetic field during the growth period[2,3]. Single crystals are the backbone of the modern technological revolution. Tartrate materials are used mostly, for their dielectric, ferroelectric, piezoelectric or nonlinear optical properties[4]. Characterization on single crystals of manganese doped barium tartrate crystals[5] and barium mixed calcium tartrate tetrahydrate crystals[6] are reported. Barium substitution introduces anisotropy in the spectroscopic splitting factor and change in C(3)–H bond length. The report on Mn doping on barium tartrate identifies few extra peaks with lower intensities. The intensity of the prominent peak is decreased[7]. Growth under the influence of magnetic field has scarcely been studied by employing the gel technique and the field is in an early stage of development with many opportunities to create new species. The influence of magnetic field on some tartrate crystals are reported[8,9]. A magnetic field may be considered as affecting nucleation and crystal growth rate, polymorphism and colloidal stability. It is now being applied to study the crystal growth of proteins and other compounds. In research on many poorly soluble inorganic salts, a marked affect was only found for diamagnetic salts of weak acids (Carbonates and Phosphates); a magnetic field (0.27 T) increased their nucleation and growth rates. No effect on paramagnetic salt was recorded. An increase in average growth rate about 41% and the average crystal size of 38% higher were observed due to the influence of magnetic field on crystallization from solutions was reported. In this paper, we are reporting the optical, structural and thermal analysis of barium tartrate crystal grown in the presence and absence of magnetic field. Barium tartrate crystal formed are aggregates of complex shapes[10].There is no literature available on the growth of barium tartrate crystals under the influence of magnetic field to the best of our knowledge.

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Experimental Work

Single diffusion method was employed to grow these crystals. The chemical reaction taking place in the gel medium was as follows.

$$C_4H_6O_6 + BaCl_2 \rightarrow BaC_4H_4O_6 + 2HCl$$

Borosilicate glass test tubes of length 25 cm and diameter 2.5 cm were placed vertically on thermocole stand. Sodium meta silicate solution of 1.04 gm/cm^3 was prepared. 1M tartaric acid of 9.5 ml quantity was added to 10 ml of sodium meta silicate solution taken in the test tube. The pH of the gel was maintained at 4.2. 1M barium chloride solution of 10 ml was added over the set gel without any gel breakage. The barium ions diffused through the narrow pores of the gel to react with tartrate ions, which are present in the gel as inner reactant. More number of crystals of small size were formed near the interface. Though the number of crystals formed was found less, when we go deeper from the interface, the size of the crystals were found to be large. For growing the crystals under the influence of magnetic field the set gel was subjected to the magnetic field of 182 Gauss, 5 hours before adding the outer reactant . The barium tartrate crystals growing in the absence of magnetic field (BaTr)and in the presence of magnetic field(BaTrmf) are shown in Figure 1.

Figure 1 BaTr and BaTrmf Crystals Growing in Gel

RESULTS AND DISCUSSION

FTIR Analysis

The FTIR spectrum was recorded in the range of 4000 cm^{-1}-400 cm^{-1} using spectrum RXI FTIR Spectrometer. The spectrum of BaTr and BaTrmf are shown in the Figure 2 and Figure 3 respectively. The functional groups were identified. There are 5 additional peaks in BaTrmf at 3874 cm^{-1}, 3686 cm^{-1} 3874 cm^{-1}, 2147 cm^{-1}, 1344 cm^{-1}, which may be due to the orientations of atoms in the direction of applied magnetic field. The assignment of vibrational frequencies of BaTr and BaTrmf are given in Table 1. The absorption peak at 2832 cm^{-1} of BaTr and 2870 cm^{-1} of BaTrmf are assigned to O–H water stretch. The peak at 709.06 cm^{-1} of BaTr and 710.01 cm^{-1} of BaTrmf may be due to metal oxygen bonding. The carbonyl group and CH stretching are also identified. The assigned vibration peaks are in good agreement with the reported values[11].

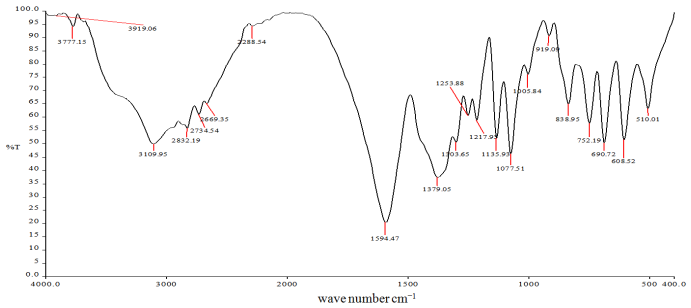

Figure 2 FTIR Spectrum of BaTr

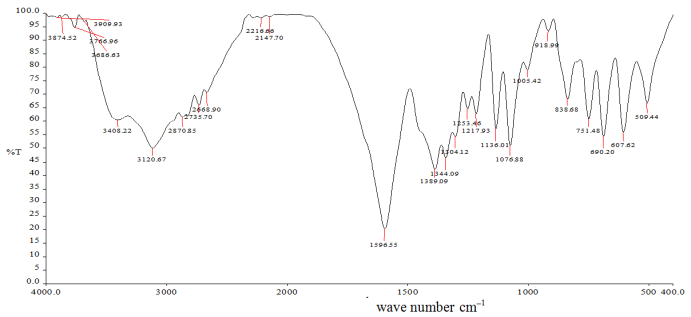

Figure 3 FTIR Spectrum of BaTrmf

Table 1 Vibration Frequencies and Assignments of BaTr and BaTrmf

Reported[11] in cm^{-1}	BaTr in cm^{-1}	BaTrmf in cm^{-1}	Assignments
3108.75	3109.15	3120.67	–OH stretching
2850	2832.19	2870.85	C–H stretch
1601.29	1594.47	1596.55	C=O stretch
1390.2	1379.05	1389.09	λ(C=O) + δ(O–C=O)
1138.23	1135.93	1136.01	δ(C–H) + π(C–H)
1079.97	1077.51	1076.88	Out of plane OH deformation
1005	1005.84	1005.42	C–Hvibration
837	838.95	838.68	C–C
691.96	690.72	690.20	CO_2 deformation
511.72	510.01	509.44	Ba–O

UV Spectral Analysis

The UV spectrum in the spectral range of 190 – 1100 nm was recorded using Lambda 35 Perkin-Elmer spectrophotometer. The UV vis NIR transmittance spectrum of BaTr and BaTrmf are shown in Figure 4 and Figure 5. The percentage of transmittance of the crystals grown in the absence of magnetic field is greater than BaTrmf. The crystals show optical transmittance in the entire visible region. The band gap energy is calculated as 6.4 eV for BaTr and 6.48 eV for BaTrmf. The refractive index of BaTr is 1.85. The refractive index of BaTrmf is 1.84.

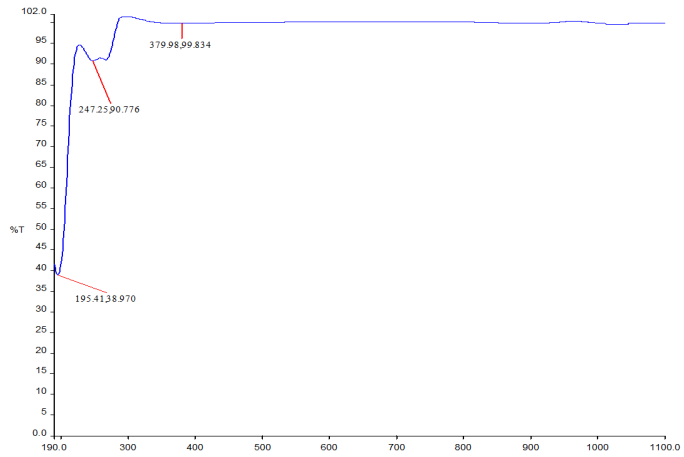

Figure 4 UV Spectrum of BaTr

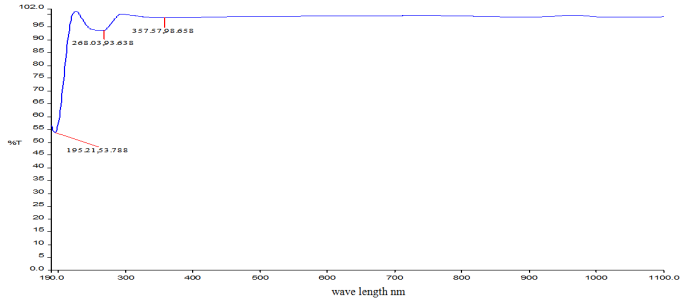

Figure 5 UV Spectrum of BaTrmf

Thermal Analysis

Thermogravimetry analysis

The thermal characterization of gel grown crystals of barium tartrate was done by measuring the change in their physico chemical properties as a function of increasing temperature with time. The experimental data shows that the material is stable upto 225° C beyond which it exhibits marked tendency to decompose.TGA was carried out in the nitrogen atmosphere at 20 ml / min using Perkin-Elmer make at the rate of 30 deg / min between the temperature range 35-900° C. The TGA of BaTr and BaTrmf are shown in the Figure 6 and Figure 7 respectively. From the TGA analysis it is inferred that the mode and the number of decomposition stages of BaTr and BaTrmf are different. The decomposition of pure and doped barium tartrate crystal is discussed in literature[12-16]. The samples are thermally stable upto 223° C. The percentage values of weight losses were calculated for different stages of thermograms and were found to be matching with observed ones. The decomposition details of BaTr and BaTrmf are given in Table 2 and Table 3 respectively. From the TGA curve it is inferred that decomposition starts at slightly higher temperature for BaTrmf. The thermal decomposition of BaTr has three stages. The first stage is decomposition of 0.5 H_2O. In the second stage the anhydrous BaTr decomposes into barium oxalate. The product of decomposition of third stage is barium oxide. The thermal decomposition of BaTrmf has two stages. The first stage is decomposition of 0.5 H_2O. In the second stage the anhydrous BaTrmf decomposes into barium oxide and barium carbonate. Barium carbonate is used as a pyro-colorant to produce green colour in fire creekers as well as in signaling one stage[17]

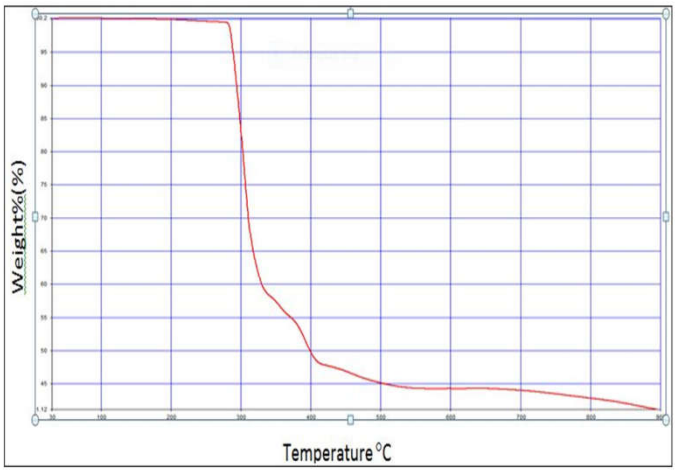

Figure 6 TGA of BaTr

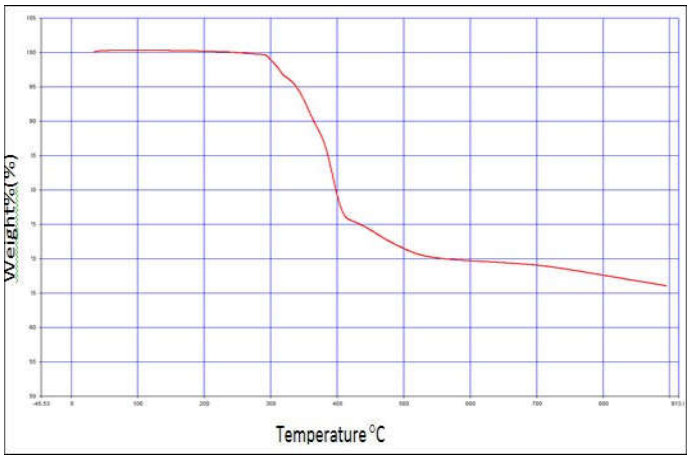

Figure 7 TGA of BaTrmf

Table 2 Thermal decomposition Reactions of BaTr

TGA Stage	Temperature range	Mass loss % Observed	Calculated	Reactions
I	243° C – 281° C	3	3.05	$BaC_4H_4O_6.5H_2O \rightarrow BaC_4H_4O_6$
II	281° C – 305° C	20	20.38	$BaC_4H_4O_6 \rightarrow \begin{matrix}COO\\COO\end{matrix} \diagdown\diagup Ba$
III	300° C – 900° C	35	32	$\begin{matrix}COO\\COO\end{matrix} \diagdown\diagup Ba \rightarrow BaO$

Table 3 Thermal decomposition Reactions of BaTrmf

TGA Stage	Temperature range	Mass loss % Observed	Calculated	Reactions
I	294.76° C – 415.79° C	25	23.5	$BaC_4H_4O_6.O.5H_2O \rightarrow BaC_4H_4O_6$
II	415.79° C – 900° C	9	9.76	$BaC_4H_4O_6 \rightarrow 0.5BaCO_3+0.5BaO+CO$

Differential thermal Analysis

The DTA curve of BaTr and BaTrmf are shown in Figure 8 and Figure 9 respectively. The DTA curves show parallel peaks corresponding to weight losses in TG curve. A sharp endothermic peak is observed at 300º C for BaTr. A sharp endothermic peak is observed around 400º C for BaTrmf. The DTA of BaTr shows a small plateau at 400° C. The DTA of BaTrmf has a small sharp endothemic peat at 300° C .The well defined sharp peak at 400° C attribute to major weight loss.

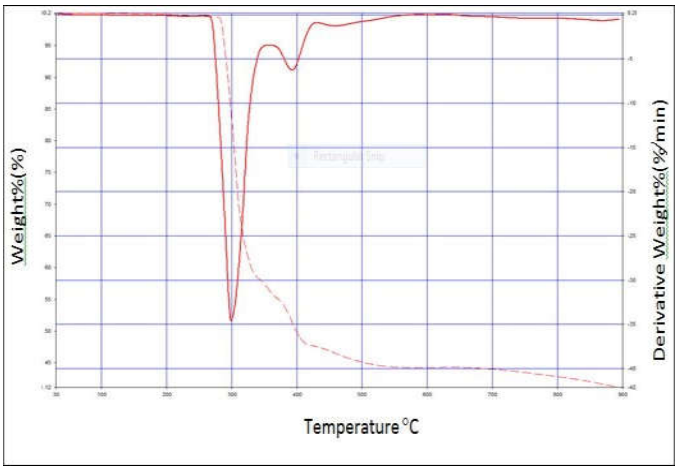

Figure 8 DTA of BaTr

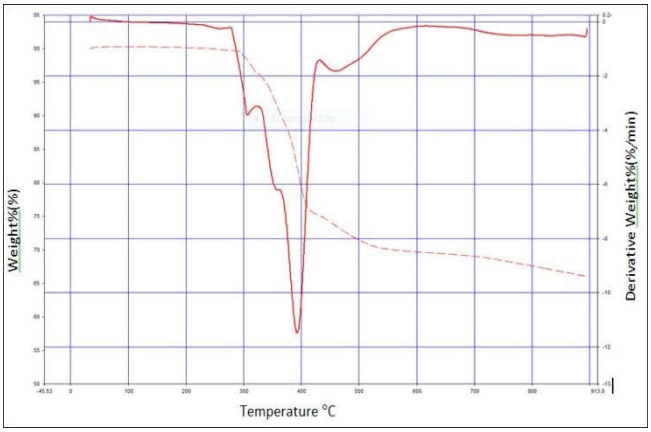

Figure 9 DTA of BaTrmf

Differential scanning calorimetry

DSC is carried out in the nitrogen atmosphere at 20 ml / min using Perkin-Elmer make at the rate of 30 deg/min between the temperature range 35° C – 400° C. The DSC curves of BaTr and BaTrmf are shown in Figure 10 and Figure 11 respectively. The thermogram of BaTr shows an endothermic peak at 330° C with heat flow 22.75 mw. The thermogram of BaTrmf shows an endothermic peak at 330°C with heat flow 24.1 mw.

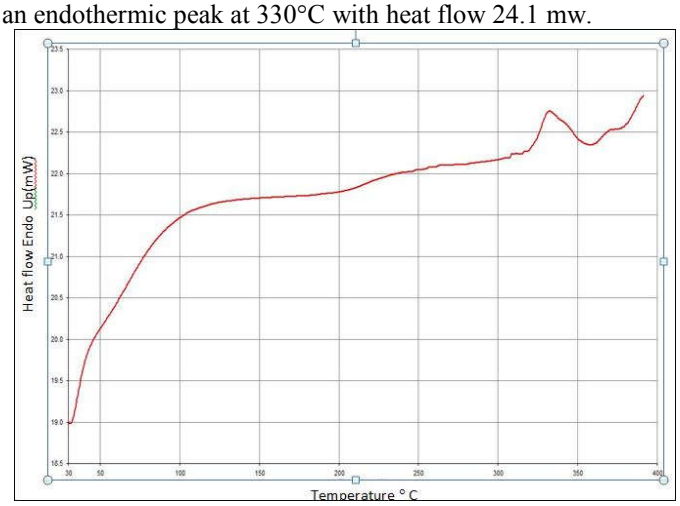

Figure 10 DSC of BaTr

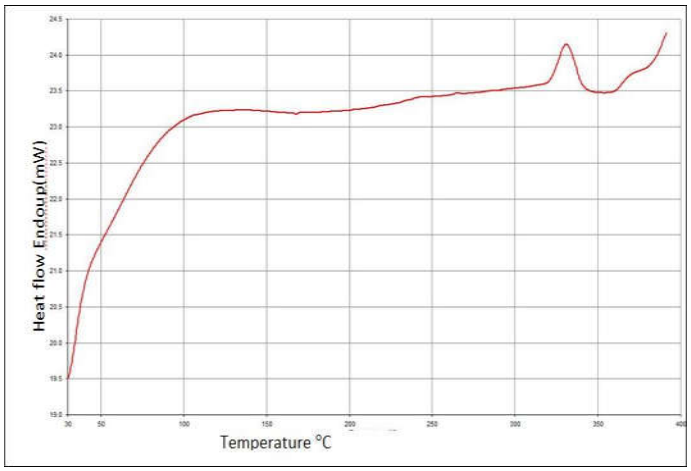

Figure 11 DSC of BaTrmf

Structural Analysis

Single crystal XRD Analysis

Single crystal XRD data of the grown crystals were collected using Bruker Kappa Apex III diffractometer with MoK_α radiation (λ = 0.71073 Å). From the single crystal XRD study, the system of grown crystals were found to be orthorhombic with space group $P2_1\ 2_1\ 2_1$. The crystallographic data of BaTr and BaTrmf are given in the Table 4.

Table 4 Comparative Crystallographic Data of BaTr and BaTrmf

Parameters	BaTr	BaTrmf	Reported [18]
a Å	8.22	8.26	8.182
b Å	8.43	8.47	8.40
c Å	9.06	9.09	9.028
Crystal system	orthorhombic	orthorhombic	orthorhombic
α, β, γ	90°	90°	90°
Space group	$P2_1\ 2_1\ 2_1$	$P2_1\ 2_1\ 2_1$	$P2_1\ 2_1\ 2_1$
Volume (Å^3)	628	636	621

Powder XRD Analysis

The powder XRD studies were carried out using Rigaku Ultima 3 diffractometer with CuK_α radiation (λ=1.54056 Å). The samples were scanned over the required range of 2θ values (10° – 80°). The powder XRD patterns of BaTr and BaTrmf are shown in Figure 12 and Figure 13 respectively. The patterns confirm that the crystals are essentially in single phase. The XRD data was analyzed using Expo 2014 and chekcell software [19,20]. The 2θ values, d values and the indices of major peaks observed in the spectra of the samples BaTr and BaTrmf are given in Table 5 and Table 6 respectively. The indices of the major peaks for BaTr in decreasing order of intensity are (362), (416), (222), (320), (210), (132),(002), (211), (013), (101). For the crystal BaTrmf the major peaks in decreasing order of intensity are (210), (322), (101), (013), (214),(011),(230),(311),(002),(200) and (201). The most important feature of this data is the elevation of the (210) planes from the eighth position in BaTr to the first position in BaTrmf. There is change in the order of (013) and (101) planes of BaTr to (101) and (013) of BaTrmf. This signifies that the application of 182 Gauss magnetic field during the growth of

barium tartrate crystals, resulted in a modification of the lattice planes. This modification would have resulted from the orientation of a considerable number of unit cells against thermal disordering, in a direction corresponding to the least value of magnetization energy [21]. The intensity of powder pattern of BaTr is greater than BaTrmf. The cell parameters calculated from the powder XRD data are in good agreement with single crystal XRD data. The grain size is calculated as 2.37 nm for BaTr and 1.278 nm for BaTrmf.

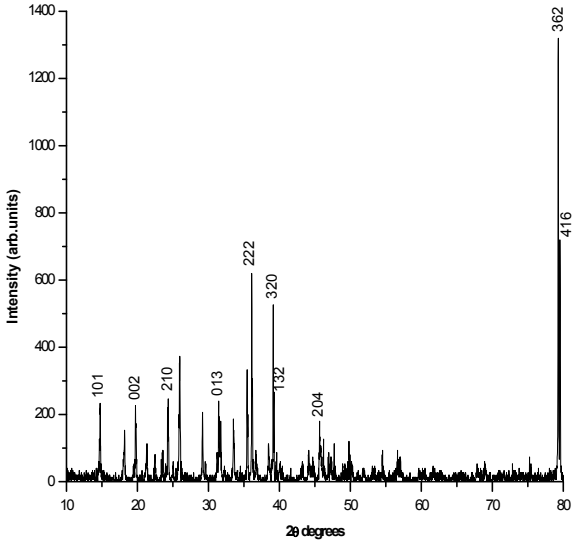

Figure 12 Powder XRD Pattern of BaTr

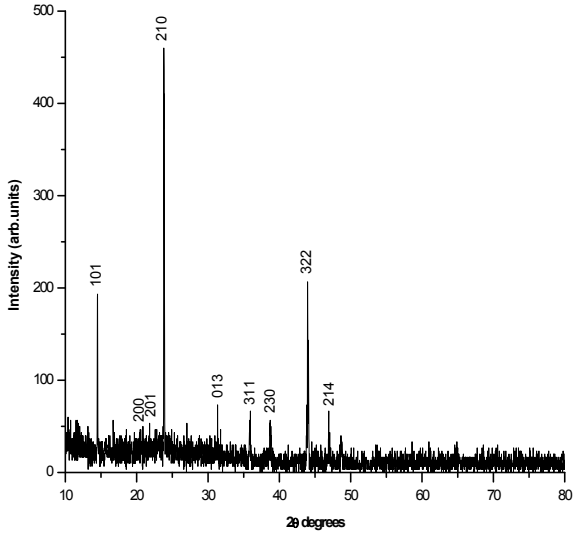

Figure 13 Powder XRD Pattern of BaTrmf

Table 5 hkl indexing of BaTr

2θ in radians	d Å	Relative intensity	indices
45.66	1.9852	13.64	204
19.7219	4.4978	17.17	002
14.7402	6.0048	17.68	101
31.4414	2.8249	18.18	013
24.3378	3.6542	18.69	211
24.3378	3.6542	18.69	002
39.2392	2.2940	20.20	132
25.9569	3.4268	28.28	210
39.1393	2.2997	39.90	320
36.1008	2.4860	46.97	222
79.5210	1.2044	54.55	416
79.2997	1.2072	100	362

Table 6 hkl indexing of BaTrmf

2θ radians	d Å	Relative intensity	indices
22.8218	3.8934	10.14	201
21.8287	4.0682	11.59	200
11.5007	7.6879	12.32	002
35.8599	2.5021	12.32	311
38.7187	2.3237	12.32	230
10.3820	8.5136	13.04	011
46.9301	1.9345	14.49	214
31.3460	2.8513	15.94	013
14.52	6.0953	42	101
43.9623	2.0579	45	322
23.8030	3.7351	100	210

CONCLUSION

BaTr and BaTrmf crystals have been successfully synthesized. The thermal and structural properties of these crystals have been studied. From the thermogram it was found that the percentage of weight loss is maximum for BaTr (58 %) compared with BaTrmf (around 44 %). The decomposition starts at the earlier temperature in BaTr. The changes observed in the powder XRD spectra signify that the application of magnetic field during the growth of barium tartrate crystal's may result in an orientation of the lattice in a direction corresponding to the magnetostatic energy and an increase in the spacing between the Bragg's diffraction planes. This is also well supported by an increase in the volume of BaTrmf by 8 units, observed from single crystal XRD data.

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