



The Gel Growth of Pure and Mixed Strontium and Manganese Tartrate Crystals

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Abstract:

Several tartrate compounds deserve special attention due to their applications; for example; manganese tartrate crystals being temperature sensitive find applications to sense temperature changes; strontium tartrate find application in the tracer compound in ammunition units. The attempt is made to grow pure and mixed Strontium and Manganese crystals by single diffusion gel growth technique using silica hydrogel. The coloration and morphology of the grown crystals changed upon changing the composition of the crystals. The FT-IR spectroscopic study confirmed the presence of C=O, C-O, O-H and metal-oxygen bonds. However, the absorptions due to metal-oxygen bond vibration changed systematically on varying the composition of manganese in the crystals. The powder XRD study indicated a flip in the structure of mixed crystals from strontium tartrate type to manganese tartrate type on increasing the content of manganese in the mixed crystals. The results are discussed.

Keywords: Crystal Growth, FTIR technique, Sol-gel preparation, X-ray technique

1. Introduction

Manganese tartrate crystals being temperature sensitive can be used to sense and measure the temperature. The change in coloration of these crystals occurs at 410⁰ C from pink to black. The coloration change is almost instantaneous and occurring within 1 to 2 seconds. Gvozdev and Erunov described explored this method.

There are some other applications of tartrate compounds; for example, ferroelectric applications of sodium – potassium tartrate and calcium tartrate, ferroelectric - ferroelastic applications of sodium ammonium tartrate , dielectric applications of lithium thiallate tartrate and use of strontium tartrate in the tracer compound in ammunition units . Looking at various applications of different tartrate compounds the present authors have grown strontium and manganese mixed tartrate crystals by the gel growth technique and characterized them by powder XRD and FT-IR studies.

2. Experimental

2.1 Crystal Growth

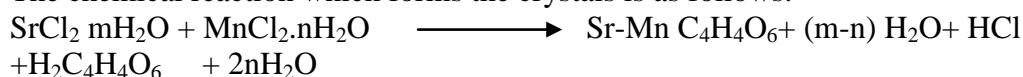
The crystallization apparatus employed for the present investigation were consisted of glass test tubes of 25mm diameter and 140mm length. The AR grade chemicals were used to grow the crystals. One of the reagents, 1 M levo tartaric acid, was mixed with sodium metasilicate solution to prepare the gel in such a way that the pH of mixture was maintained 4.5. The specific gravity of the gel was chosen 1.04. After setting the gel, the supernatant solutions containing various amounts of SrCl₂ and MnCl₂ having concentrations of 1M were gently poured without disturbing

the gel surfaces, in respective test tubes. The growth of crystals was completed within the period of one month.

The following supernatant solutions added on the set gel:

- (1) 10 ml 1M SrCl₂ (Solution-1),
- (2) 8 ml 1M SrCl₂ + 2 ml 1M MnCl₂ (Solution-2),
- (3) 6 ml 1M SrCl₂ + 4 ml 1M MnCl₂ (Solution-3),
- (4) 4 ml 1M SrCl₂ + 6 ml 1M MnCl₂ (Solution-4),
- (5) 2 ml 1M SrCl₂ + 8 ml 1M MnCl₂ (Solution-5),
- (6) 10 ml 1M MnCl₂ (Solution-6)

The chemical reaction which forms the crystals is as follows:



2.2 Characterization techniques

The FTIR spectra of mixed Sr-Mn levo tartrate crystals were recorded in the range from 400 cm⁻¹ to 4000 cm⁻¹ using SHIMADZU, FTIR 8400 spectrometer. The powder samples in KBr medium were used. The powder X-ray diffraction analysis was carried out on PHILIPS X'PERT MPD System using Cu K α-radiation and data were analyzed by REFEDT.BAS software.

3. Results and Discussion

Many workers have reported the growth of Mn levo tartrate crystals, mixed Fe-Mn levo tartrate, ternary Fe-Mn-Co levo tartrate as well as strontium tartrate and mixed Ca-Sr levo tartrate. In present study mixed Mn-Sr levo tartrate crystals were grown and the growth observation suggested that as the concentration of MnCl₂ increased in the supernatant solution, the coloration changed gradually from light yellow to pinkish brown. It was also noted that from the supernatant solution (4) onwards the white spongy precipitates were observed. These precipitates have been explained elsewhere in detail. It was also found that the small crystals were grown at the gel-liquid interface, which was due to direct reaction between two reactants. Various growth morphologies were observed such as prismatic, star shaped, rectangular shape, etc. Figure 1 shows the growing crystals in gel for Sr_{0.8}Mn_{0.2}C₄H₄O₆ 3H₂O supernatant solution.

The powder XRD study was carried out to study the change in the structural behavior of the grown crystals. Figure (2) shows the powder XRD pattern of crystals grown for the supernatant solution Sr_{0.8}Mn_{0.2}C₄H₄O₆ 3H₂O. The planes were also assigned for different reflections. The powder XRD patterns for all pure and mixed Sr-Mn levo tartrate crystals were analyzed and the unit cell parameters were determined, which are given in Table (1). It can be noticed from the table that the pure Sr tartrate type crystal structure is attained only up to the certain composition of the crystals (Sr_{0.6}Mn_{0.4}C₄H₄O₆ 3H₂O) then there is a sudden flip to the Mn levo tartrate type crystal structure. This may be due to larger ion radius of Sr (1.32 Åpm) compared to Mn (0.89 Å) changes the bond and ultimately the lattice attains the MnC₄H₄O₆ 2H₂O crystal structure after the flip.

Figure (3) is the FTIR spectrum of Sr_{0.8}Mn_{0.2}C₄H₄O₆ 3H₂O mixed levo tartrate crystals. The assignments of the FTIR spectrum are given in the table (2). Similarly the FTIR spectra were taken for all other pure and mixed Mn-Sr levo tartrate crystals. It has been found that the effect of increases the Mn concentration in mixed tartrate crystals does not invite systematic changes in absorption due to the metal-oxygen vibrations. The FTIR spectra indicate the presence of water of crystallization C = O, C – O, O – H, and metal oxygen bond. However, the values of water of hydration for each crystal is found from the thermal analysis and reported elsewhere.

4. Conclusions

1. The values of lattice parameters of $\text{Sr}_{0.8}\text{Mn}_{0.2}\text{C}_4\text{H}_4\text{O}_6 \cdot 3\text{H}_2\text{O}$, $\text{Sr}_{0.6}\text{Mn}_{0.4}\text{C}_4\text{H}_4\text{O}_6 \cdot 3\text{H}_2\text{O}$ are corresponding to the strontium levo tartrate lattice parameters, but $\text{Sr}_{0.4}\text{Mn}_{0.6}\text{C}_4\text{H}_4\text{O}_6 \cdot 2\text{H}_2\text{O}$ and $\text{Sr}_{0.2}\text{Mn}_{0.8}\text{C}_4\text{H}_4\text{O}_6 \cdot 2\text{H}_2\text{O}$ corresponds to the value of lattice parameters of manganese levo tartrate crystal structure. A sudden flip in the crystal structure is observed on increasing the volume of MnCl_2 in the supernatant solutions.
2. The FTIR spectra indicate the present of water of crystallization $\text{C} = \text{O}$, $\text{C} - \text{O}$, $\text{O} - \text{H}$, and metal oxygen bond.

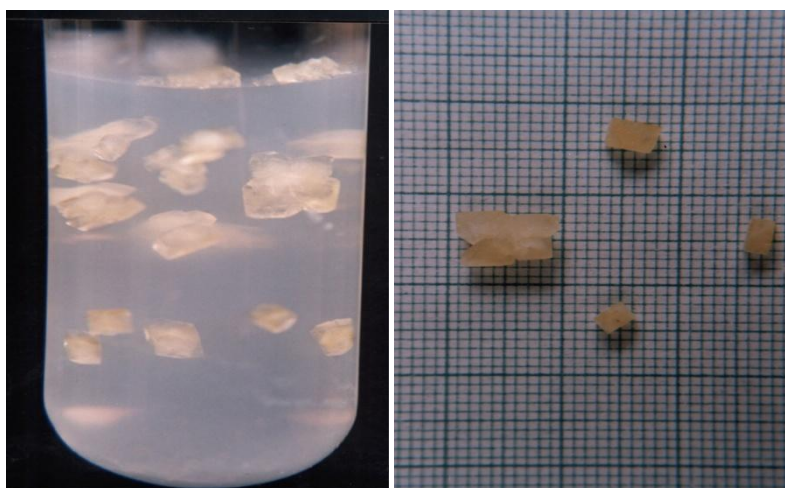


Figure 1

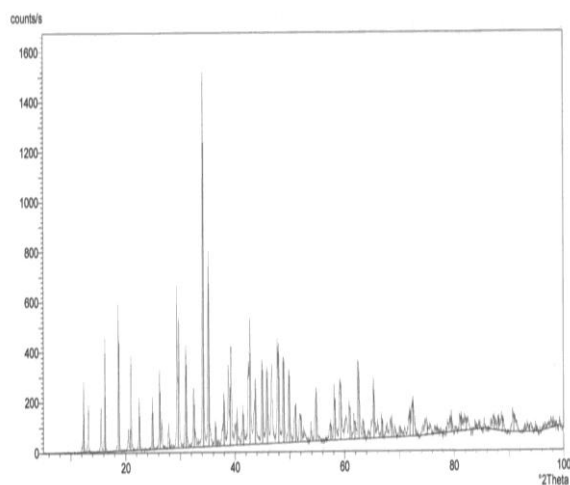


Figure 2

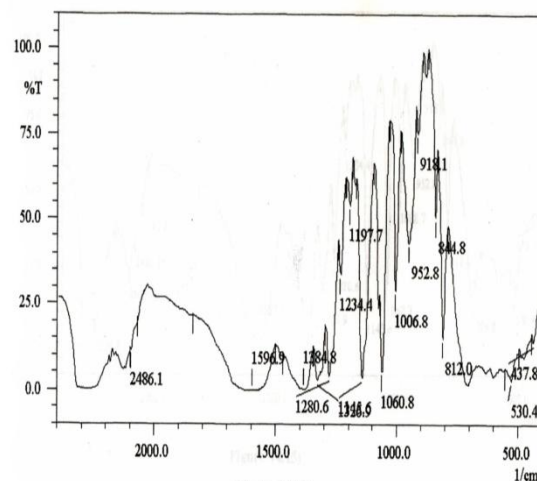


Figure 3

Table 1

Wave number (cm ⁻¹)	Assignments
3450.0	Broad O – H stretching due to water of crystalization
1596.8	C = O stretching
1280.6	C – O stretching
1184.8	C – O stretching
1234.4	C – O stretching
1197.7	C – O stretching
1060.8	O – H deformation out of plane and C – O stretching
1006.8	O – H deformation out of plane and C – O stretching
952.8	O – H deformation out of plane and C – O stretching
918.1	O – H deformation out of plane and C – O stretching
844.8	Metal – oxygen stretching
812.0	Metal – oxygen stretching
530.4	Metal – oxygen stretching
437.8	Metal – oxygen stretching

Table: 2

Sample	a (Å)	b (Å)	c (Å)	Cell volume	Ortho rhombicity	X– ray density gm/cm ³
A1	9.4517	11.0353	9.4115	981.64	7.73	3.92
A2	9.4828	10.9578	9.4503	981.98	7.21	3.83
A3	9.5102	10.9490	9.4325	982.18	7.03	3.74
A4	9.3845	11.6997	5.0426	553.65	10.98	6.04
A5	9.4222	11.6543	5.0855	558.43	10.59	5.74
A6	9.4120	11.6400	5.0706	555.51	10.58	4.85

5. Caption to Figures & Tables

Figure 1 : Photograph of Growing crystals in gel for Sr_{0.8}Mn_{0.2}C₄H₄O₆ · 3H₂O supernatant solution.

Figure 2 : Powder XRD pattern for Sr_{0.8}Mn_{0.2}C₄H₄O₆ · 3H₂O crystals.

Figure 3 : FT-IR spectrum of Sr_{0.8}Mn_{0.2}C₄H₄O₆ · 3H₂O crystals.

Table 1 : Unit cell parameter values for different crystals.

Table 2 : Assignment of various absorptions in FT-IR of Sr_{0.8}Mn_{0.2}C₄H₄O₆ · 3H₂O crystals.

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References

1. Aizu, K. J. Phys. Soc., Pap., 31 (1971) 1521.
2. Gon, H. B. J. Cryst. Growth., 102 (1990) 501.

3. GvozdoV S. P. and A. A.Frunov; Zavedenhil Khim. Teckhnol., 5 (1958) 154.
4. Heiskell, R. H. U. S. Patent (1959) no. 2899291
5. Kansara, S. B. M.Phil. Dissertation, Saurashtra University, June 2003.
6. Sawaguchi, E. and L. E. Cross; Ferroelectrics, 2 (1971) 37.
7. Valasek, Phys. J. Rev., 17 (1921) 475.