

Ag-Mn Mixed Tartrate Crystals: A Growth and Spectrometric Studies

Mansi Tailor¹, Mayuri Dave², Vimal Joshi^{3*}

Research Student, Research Laboratory, Department of Physics, Shri R.K. Parikh Arts and Science College, Petlad,
Dist: Anand, Gujarat, India¹.

Research Student, Research Laboratory, Department of Physics, Shri R.K. Parikh Arts and Science College, Petlad-
Dist: Anand, Gujarat, India².

Principal, Department of Physics, Shri R.K. Parikh Arts and Science College, Petlad, Dist: Anand, Gujarat, India³.

ABSTRACT: The different tartrate compounds draw attention of many researchers because of its several applications in science and technology as well as in the field of medical and pharmaceutical sciences. In the present investigation, authors present their research on the crystal growth of silver-manganese mixed tartrate crystals using the method of single diffusion gel growth technique in silica gel made from aqueous solution of sodium metasilicate and tartaric acid. During the growth of silver-manganese mixed tartrate crystals, two types of morphology of grown crystals have observed. One is flower shape crystals having light pink colour and another is spherulitic crystals having light brown colour. The X-ray diffraction and FT-IR spectroscopic studies were carried out on the powdered sample of grown crystals. The results are discussed.

KEYWORDS: gel growth, crystal growth, silver-manganese mixed tartrate crystal, powder XRD, FT-IR

I. INTRODUCTION

The gel growth technique is one of the oldest techniques to grow the crystals at ambient temperatures, which are sparingly soluble in water or decompose before melting. Study of growth, characterization and applications of several crystals have been carried out by many authors [1-4]. Many types of pure and mixed tartrate crystals have been grown in hydro silica gel. Growth and characterization of different crystals of pure and mixed tartrate compounds were elaborately reported by earlier researchers [5-15].

Interest in the morphology of crystals started in 17th century when Kepler [16] observed dendritic snow crystals in 1611 and Steno [17] was attracted by variety of polyhedral forms of rock crystals[18-19]. Different types of tartrate crystals can be grown in hydro silica gel with the help of single diffusion technique[2]. Mainly tartrate crystals have attracted attention due to their interesting physical properties and application like dielectric and ferroelectric[20-23]. Growth and characterization of pure and mixed crystals were reported earlier by many researchers[24-27].

In the present investigation, growth of Silver-Manganese mixed tartrate crystals and their characterized by FT-IR spectroscopy and powder X-ray diffraction techniques were reported. Unit cell parameters and crystal structure of grown crystals were calculated by powder x-ray diffraction technique with the help of powder X software. The functional group present in the grown crystals were reported by FT-IR spectroscopy.

II. EXPERIMENTAL TECHNIQUES

Crystal growth

In the present study single diffusion gel growth technique was employed to grow Silver-Manganese mixed tartrate crystals. The glass test tubes were used as crystalline apparatus. Dimension of glass test tubes were 25mm internal diameter and 150mm length. Hydro silica gel was used as a gel media to grow Silver-Manganese mixed tartrate crystals. The hydro silica gel was prepared by aqueous solution of sodium meta silicate having 1.05 specific gravity was acidified by 1M tartaric acid in such a manner that pH 5.0 could be set for gel mixture. This gel mixture was

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transformed in to different glass test tubes and allowed to set into gel form. The gel set within 2 to 3 days. After setting gel, 10ml of supernatant solution was gently poured over a set gel without break the gel. The supernatant solution was prepared by mixing different proportion of 1.5M AgNO_3 and 1.5M $\text{Mn}(\text{NO}_3)_2$.

The growth of crystals has started after three to four weeks. Before crystal growth start, it was observed that some precipitates formed near Gel-Liquid Interface (GLI). The formation of precipitates were denser near the GLI and density of precipitates become decreases as move away from GLI. This precipitates were formed in the band shape. The growth of crystals were observed in the layered form. Two types of crystal morphology were observed in the test tubes. One was pinkish colour floret shape and another was spherulitic brown colour. The morphology of both the crystals were different. In the present study, authors present the characterization of the pink crystals grown by the supernatant solution containing mixture of 2 ml AgNO_3 and 8 ml $\text{Mn}(\text{NO}_3)_2$. Fig. 1 shows the precipitates and grown crystals within the test tube. Whereas, the grown crystals depicted in the fig.2.

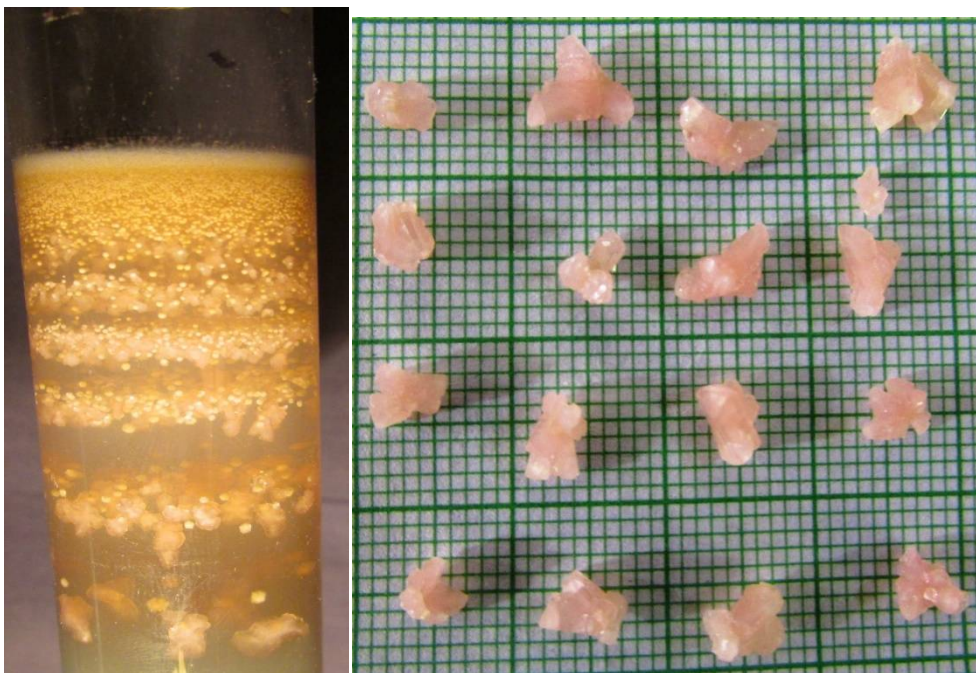


Fig.1 Precipitates and grown crystals within the test tube Fig 2 Grown crystals

Characterization Techniques

FTIR spectroscopic analysis

Fourier Transform Infrared Spectroscopy is a non-destructive technique which is used to obtain an infrared spectrum of absorption and emission. This technique is applicable to the analysis of solids, liquid and gases. FTIR is tool for identifying the functional group present in given sample.

In the present study, the FT-IR spectrum of powdered sample of grown Silver-Manganese mixed tartrate crystals were recorded in the range of 400cm^{-1} to 4000cm^{-1} using Perkin Elmer Fourier Transform Infrared Spectrometer Model: Spectrum GX.

Powder XRD analysis

Crystalline nature and unit cell parameters can be studied by powder X-ray diffraction technique.

In the present study, powder X-ray diffraction pattern of grown Silver-Manganese mixed tartrate crystals was recorded using Philips Holland, Model: X'pert MPDX-ray diffractometer with CuK_α radiation.

III.RESULT AND DISCUSSION

Fourier Transform Infrared Spectroscopic (FTIR) Analysis

The FTIR spectrum of grown pink crystals of Silver-Manganese tartrate was recorded in the wave number range of 4000cm⁻¹ to 400cm⁻¹ is shown in fig.3.

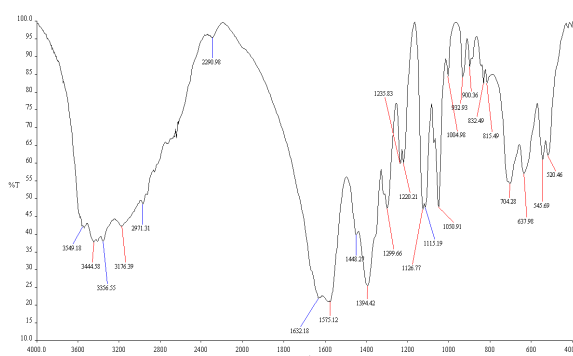


Fig.3 absorption spectrum of powdered sample of grown crystals

The absorption peaks at 3549.18cm⁻¹, 3444.58cm⁻¹,3176.39cm⁻¹ and 2971.31cm⁻¹ are due to stretching of O-H bond, which normally indicates the presence of water crystallization. The strong absorption peak at 2290.98cm⁻¹ and 1394.42cm⁻¹ are due to combination of C-H stretching like δ and π. The absorption peaks at 1632.18cm⁻¹ and 1575cm⁻¹ are due to the carbonyl C=O group. The strong peak at 1448.27cm⁻¹ is assigned to C=O symmetric and δ(O-C=O) mode. The absorption peaks at 1299.66cm⁻¹, 1235.83cm⁻¹ and 1220.21cm⁻¹ are due to O-H plane bending. The C-H stretching vibrations give rise to the absorption at 1126.77cm⁻¹, 1115.19cm⁻¹ and 1050.91cm⁻¹. The absorption peak at 832.49cm⁻¹ and 815.49cm⁻¹ are due to C-C stretching. The absorption peaks at 704.28cm⁻¹ and 637.98cm⁻¹ are due to water crystallization in Silver-Manganese tartrate crystals.The absorption peaks at 545.69cm⁻¹ and 520.46cm⁻¹ are due to the metal–oxygen stretching vibrations in Silver-Manganese tartrate crystals. The observed vibrational frequencies and their assignments are listed in table 1.

Absorption PeakPositions (cm ⁻¹)	Assignments
3549.18,3444.58,3176.39,2971.31	O-H stretching (water)
2290.98	Combination C-H stretching
1632.18,1575.12	C=O stretching
1448.27	C=O symmetric and δ(O-C=O)
1394.42	Combination C-H stretching
1299.66,1235.83,1220.21	O-H plane bending
1126.77,1115.19,1050.91	C-H stretching
832.49, 815.49	C-C Stretching
704.28, 637.98	Water crystallization in silver-manganese tartrate
545.69, 520.46	Metal–oxygen stretching

Table 1 Peak assignments of powdered sample of grown crystals

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Powder X-ray Diffraction Analysis

The recorded powder XRD pattern for the powdered sample of grown pink colour Silver–Manganese mixed tartrate crystals is shown in fig. 4. The experimental 2θ range is 3° – 90° with a step size of 0.05° and a counting time of 60s per step. The program of graphic tool for powder diffraction named as powder X [28]. The powder X software is also used to determine the observed diffraction peak positions of the crystal and analytical indexing of the powder pattern. The grown crystals found to be crystallized in nature having orthorhombic structure.

The unit cell parameters of are found to be,

$$a = 12.9898 \text{ \AA}, b = 14.1298 \text{ \AA}, c = 12.1211 \text{ \AA} \text{ and } \alpha = \beta = \gamma = 90^\circ$$

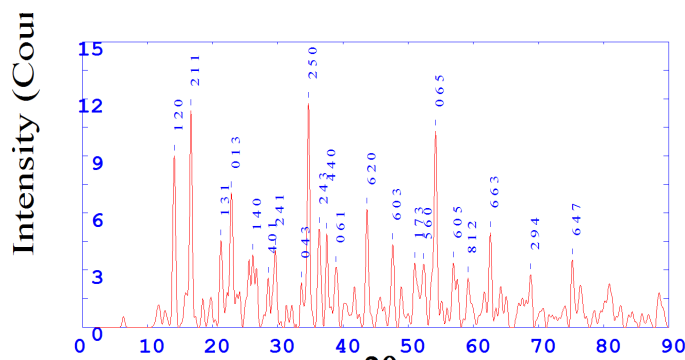


Fig.4 Powder x-ray diffractogram grown crystals

The calculated and observed values of 2θ and d corresponding to $h k l$ parameters are summarized in Table 2.

Hkl	2θ		D		Intensity (Exp.)
	(Exp.)	(Calc.)	(Exp.)	(Cal.)	
1 2 0	14.122	14.259	6.26627	6.20634	9.00
2 1 1	16.678	16.695	5.31147	5.30588	11.38
1 3 1	21.283	21.347	4.17135	4.15904	4.55
0 1 3	22.875	22.874	3.88457	3.88467	7.07
1 4 0	16.177	26.121	3.40154	3.40866	3.79
4 0 1	28.534	28.431	3.12573	3.13682	2.58
2 4 1	29.625	29.694	3.01299	3.00622	4.03
0 4 3	33.623	33.675	2.66331	2.65937	2.34
2 5 0	34.713	34.587	2.58215	2.59130	11.77
2 4 3	36.350	26.480	2.46955	2.46106	5.18
4 4 0	37.528	37.593	2.39469	2.39071	4.91
0 6 1	38.955	38.928	2.31016	2.31174	3.15
6 2 0	43.691	43.694	2.07010	2.06996	6.21
6 0 3	47.637	47.615	1.90745	1.90828	4.34
1 7 3	51.012	51.022	1.78887	1.78853	3.36
5 6 0	52.386	52.397	1.74515	1.74481	3.32
0 6 5	54.211	54.262	1.69062	1.68916	10.31
6 0 5	56.941	56.984	1.61588	1.61477	3.38
8 1 2	59.204	59.227	1.55939	1.55884	2.59
6 6 3	62.605	62.604	1.48261	1.48262	4.97
2 9 4	68.804	68.828	1.36338	1.36295	2.76
6 4 7	75.193	75.172	1.26258	1.26289	3.54

Table 2 Powder diffraction data of pink colour Silver –Manganese mixed tartrate crystals.

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IV. CONCLUSION

The pink colour crystals of Silver –Manganese mixed tartrate crystals are successfully grown by single diffusion technique in hydro silica gel. The grown crystals are of pinkish colour having floret type morphology. With the help of powder X-ray diffraction analysis, it is found that powdered sample is crystalline in nature and confirms the unit cell parameters and d-values. The FTIR spectrum shows presence of various functional group like O-H, C-C, C=O and metal oxygen bond.

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