Engineering and Scientific International Journal (ESIJ)
Volume 10, Issue 3, July – September 2023
ISSN 2394-7187(Online)
ISSN 2394 - 7179 (Print)

FTIR, SEM, EDAX Study of Manganese Tartrate Crystals

P. A. Savale
Assistant Professor, Department of Physics, SES's Arts and Science College, Bhalod Dist. Jalgaon (M.S.) India
**Corresponding Author: pa_savale@yahoo.co.in*

Abstract — In the present investigation, single crystals of manganese tartrate crystals were grown by using silica gel as a growth medium. These single crystals were grown by simple gel technique using diffusion method. The optimum growth conditions were optimized by varying various parameters viz., pH of the gel solution, gel concentration, gel setting time, concentration of the reactance, growth period and temperature for these crystals. The shiny white brownish coloured and spherulitic morphology crystals grown within silica gel column were obtained. Crystals having different morphologies were obtained with a maximum size of 5.5mm × 5.2mm × 3.5mm. The grown crystals are characterized by FTIR, SEM and EDAX. The functional groups present in the crystals were identified by using Fourier Transform Infrared Spectroscopy (FTIR) analysis which shows that the presence of O=H, C=O, C-O, C-H and metal-oxygen bonds. The scanning electron microscope (SEM) study reveals that the morphology of the crystal having orthorhombic structures. The analysis of EDAX has shown the presence of manganese and oxygen.

Keywords *— Gel Technique; Manganese Tartrate; FTIR; SEM; EDAX.*

1. Introduction

Compounds of different tartrates draw attention of many researchers because of their various applications in science and technology as well as in the field of pharmaceutical and even in the medical sciences, in addition to industrial purposes. For example, various ferrous tartrate crystals are used to avoid anaemia in animals [1] and use of manganese tartrate crystals in chemical temperature indicators [2]. Growth and Characterization of some tartrate crystals were reported by Henisch and Henisch et al [3], Patel and Rao [4]. Growth of some crystals of tartrate compounds like calcium tartrate [5, 6], strontium tartrate [7], barium tartrate [8, 9], ammonium tartrate [10], zinc tartrate [11], sodium tartrate [12], cadmium tartrate [13] and iron tartrate [14] were reported by earlier researchers.

The compounds of Tartaric acid find numerous applications in semiconductor and optics industries with the invention of lasers. The field of non-linear optics touched the new heights and practical implementations were possible with the various applications of non-linear optical crystals [15]. Many of the tartrate compounds like pure and mixed tartrate attracts extraordinary attention due to its applications in medical, pharmaceutical and industrial such as, injections of Na-Cr tartrate increase the susceptibility of the transplanted sarcoma to the effect of X-rays, calciphylatic responses of various ferrous tartrate compounds to prevent anaemia in animals, ferrous tartrate as a catalyst in manufacture of champagne, tanning action of ferrous tartrate to tan skin, strontium tartrate used in ammunition units, zinc tartrate with other compounds form a bright coating and used as protecting powder for metals[16-20].

In the present investigation, single crystals of manganese tartrate were grown by a simple gel technique using diffusion method. The optimum growth conditions were established by varying various parameters such as pH, concentration gel solution, setting time of the gel solution and concentration of the reactance. The optimum growth conditions for these crystals were determined. These crystals were characterized by using FTIR, SEM and EDAX.

2. Experimental Analysis

Manganese Tartrate ($C_4H_4MnO_6$) crystals were grown by single diffusion method in silica gel medium at room temperature. The Sodium Meta Silicate (Na_2SiO_3) solution and acetic acid (CH_3COOH) was prepared by dissolving 22gm (Na_2SiO_3) into the 250ml distilled water and 15ml (CH_3COOH) dissolving into 250ml distilled water respectively. Then (Na_2SiO_3) was added into 6ml (CH_3COOH) drop by drop by maintaining the pH 4.2 with continues till the solution becomes milky. After that 15ml solution of Manganese Chloride ($MnCl_2$) with 1M added into the gel solution. This mixture was then transferred to the test tube of 15 cm × 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 10ml tartaric acid ($C_4H_6O_6$) with 1M was allow to fall steadily along the wall of the tube above the set gel, next day the small nucleation growth was observed at below the interface of gel. This nucleation growth was increased in size. The chemical reaction inside the gel can be expressed as follows:

$$2CH_3COOH + Na_2SiO_3 \rightarrow 2CH_3COONa\downarrow + SiO + H_2O$$
$$2CH_3COONa + MnCl_2 \rightarrow (CH_3COO)_4 Mn \downarrow + 4NaCl$$

ISSN 2394-7187(Online)
ISSN 2394 - 7179 (Print)

$(CH_3COO)_4\ Mn + C_4H_6O_6 \rightarrow C_4H_4MnO_6\downarrow + 4CH_3COOH$

Good quality shiny white brownish coloured spherulitic crystals were grown in 40-45 days. The functional groups present in the single crystals were identified using Fourier Transform Infrared spectroscopy (FTIR) analysis. The SEM was done to find the structural morphology of the grown crystals. The presence of manganese and oxygen was confirmed by EDAX analysis.

3. Results and Discussions

Fig.1: Grown ($C_4H_4MnO_6$) crystals

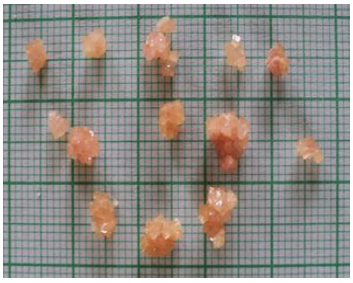

Fig. 2: Shiny white brownish ($C_4H_4MnO_6$) crystals

The Figure 1 shows the crystals of manganese tartrate attached themselves and forming a thick layer at the interface. Fig. 2 shows a few grown crystals of manganese tartrate having different habits with their scaling on a graph paper. The grown crystals are of the 5.5mm × 5.2mm × 3.5mm size. Several optimum circumstances for growing crystals were given in Table 1. For pH 4.2 the spherulitic crystals were observed in the interface of the gel column [21, 22]. Tartaric acid ($C_4H_6O_6$) used as upper reactant. Gel age is the interval time between gel and pouring of the upper reactant.

Table 1. Optimum condition for growth of Manganese Tartrate ($C_4H_4MnO_6$) crystals

Sr. No.	Conditions for growth of Manganese Tartrate ($C_4H_4MnO_6$) crystals	Single diffusion
01	Density of sodium meta silicate (Na_2SiO_3)	1.05 gm/cm^3
02	Amount of 1M acetic acid(CH_3COOH)	6 ml
03	pH of the gel	4.2
04	Temperature	Room Temperature
05	Concentration of Manganese Chloride ($MnCl_2$)	15 ml
06	Concentration of 1M Tartaric acid($C_4H_6O_6$)	10 ml
07	Gel setting period	4-5 days
08	Gel aging period	24 Hrs
09	Growth of period	40- 45 days
10	Quality	Shiny white brownish, Large size, Spherulitic morphology
11	Size of crystals	5.5mm × 5.2mm × 3.5mm

3.1 FTIR Analysis of Manganese Tartrate Crystals

The Fig. 3 shows the FTIR spectrum of the manganese tartrate crystals and Table 2 presents the observed absorption frequencies and their assignments in relation to their characteristic vibrational modes. The FTIR spectrum was recorded using Shimadzu FTIR-8400, Japan (400cm^{-1} to 4000cm^{-1}).

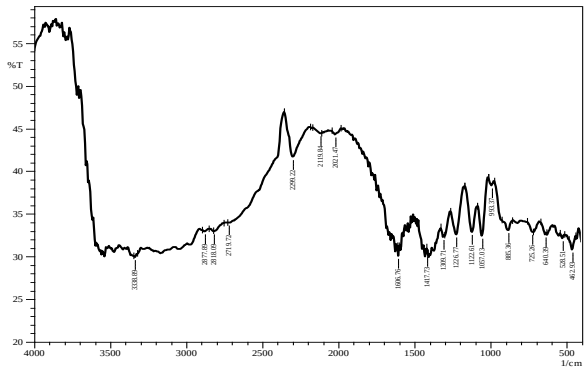

Fig. 3: FTIR spectrum of manganese tartrate crystals (pH 4.2)

The absorption peaks positioned in between 3338.89–2299.22cm^{-1} corresponds to symmetric and asymmetric stretching vibrations of O-H bond due to water of crystallization. The peak at 1606.76cm^{-1} is the band corresponding to carbonyl C=O group. The band at 1309.71cm^{-1} is the band corresponding C-O stretching vibration. The band at 1122.61-885.36cm^{-1} is the bands corresponding to O-H stretching out of plane vibrations and C-H stretching. The band observed at 725.26-462.93cm^{-1} corresponds to metal oxygen bonding [23-25].

Table 2. FTIR spectral analysis of Manganese Tartrate crystal (pH 4.2)

FTIR peaks cm^{-1}	Assignment	Intensity
3338.61, 2719.72, 2299.22	O-H stretching	Strong, sharp
1606.76	C=O stretching	Strong, broad

DOI: **10.30726/esij/v10.i3.2023.103001**

ISSN 2394-7187(Online)
ISSN 2394 - 7179 (Print)

1309.71	C–O stretching	Strong, sharp
1122.61, 885.36	O-H deformation out of plane and C-H stretching	Strong, sharp
725.26,640.39,462.93	Metal Oxygen bonding	Strong, sharp

3.2 SEM Analysis of Manganese Tartrate Crystals

The Fig.4 shows the SEM image of manganese tartrate. The FESEM image was noted using Hitachi S-4800, Japan with X-Flash detector-5030, Bruker, Germany. The scanning electron microscope study reveals that the morphology of the crystal having rectangular shape, triangular shape, looks like cubes of different shapes and sizes of orthorhombic structures [26, 27].

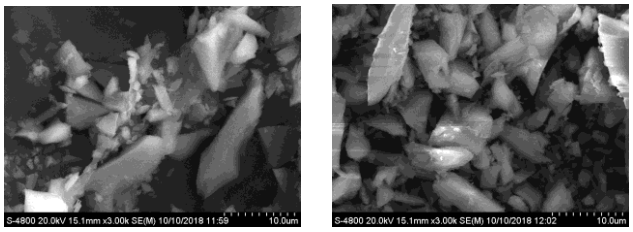

Fig. 4: SEM of manganese tartrate crystals

3.3 EDAX Analysis of Manganese Tartrate Crystals

Fig. 5 shows the EDAX pattern of manganese tartrate crystals. The elemental analysis of gel grown manganese tartrate crystals was carried out by using X-Flash-60 Bruker instrument. Manganese tartrate is one of the most complex systems with many phases and unique chemical composition as it is amplified between silica gel by the distribution of manganese chloride in the gel. The diversity in the stoichiometry of manganese tartrate poses a challenge for the control of size and shape. The energy dispersive x-ray spectrum has shown that the Mn and O are present in the atomic percentage of 8.51 and 55.05 and weight percentage of 26.19 and 49.31 respectively [28, 29]. Table 3 shows the EDAX analysis for manganese tartrate crystal.

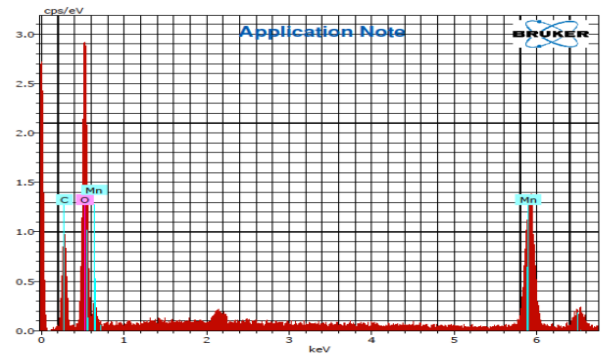

Fig. 5: Shows the EDAX pattern of manganese tartrate crystals

Table 3. EDAX analysis for manganese tartrate crystal.

Spectrum: 9891

El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
C	6	K-series	49.06	24.50	36.44	9.77
O	8	K-series	98.73	49.31	55.05	15.33
Mn	25	K-series	52.43	26.19	8.51	1.61
		Total:	200.23	100.00	100.00	

4. Conclusions

Gel method is found to be suitable for growing the manganese tartrate crystals. Single diffusion method is convenient for the growth of the manganese tartrate crystals. Nucleation control can be achieved by changing a various gel parameters such as pH of gel, density of gel and concentration of feed solutions. The shiny white brownish coloured spherulitic crystals were observed. From the FTIR spectroscopic study, the presence of O=H, C=O, C-O, C-H and metal-oxygen bonds were confirmed. The SEM study reveals that the morphology of the crystal having orthorhombic structures. The analysis of EDAX has shown anhydrous nature of the manganese tartrate crystal with the presence of manganese and oxygen.

Acknowledgements

Authors are grateful to University Institute of Chemical Technology, Kavayitri Bahinabai Chaudhari North Maharashtra University, Jalgaon for providing FTIR, SEM and EDAX facilities.

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DOI: **10.30726/esij/v10.i3.2023.103001**