

Gel Growth, Powder XRD and FTIR Study of Magnesium Levo-Tartrate Crystals

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ABSTRACT: There are many applications of different tartrate compounds. In the present investigation Magnesium Tartrate crystals were grown by single diffusion reaction silica gel technique. Crystals having shiny white colour and spherulitic morphology were grown within silica gel column. More numbers of tiny crystals were grown at gel-liquid interface. The powdered samples of grown crystals were characterized by powder XRD and FTIR spectroscopic techniques. The results are discussed.

KEYWORDS: Gel Growth, Crystal growth, Magnesium tartrate, XRD, FTIR.

I. INTRODUCTION

At the beginning of seventies, scientific bases of gel growth techniques for low solubility crystals have been established. Afterwards, such methods of crystal growth were applied to soluble materials with a low molecular weight [1,2,3]. Growth and Characterization of some tartrate crystals were reported by Henisch [1], Henisch et al [4], Patel and Rao [5]. Growth of some crystals of tartrate compounds like calcium tartrate [4,6], 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.

Tartaric acids are available in the three different chiral forms, viz., levo-tartaric acid or L-(+)-tartaric acid, dextro-tartaric acid or D-(-)-tartaric acid and meso-tartaric acid. All the three types can be used to grow tartrate crystals but generally levo-tartaric acid and dextro-tartaric acid are reported to grow the different crystals [14,15]. Some mixed tartrate crystals like calcium-barium tartrate [16], calcium-strontium tartrate [7,17], iron-manganese tartrate [18] etc. were also reported. In addition to mixed tartrate crystals, ternary crystals like cadmium-zirconium-sodium [19], iron-manganese-cobalt [20] and iron-manganese-nickel [21] were also reported. Many of the tartrate compounds including pure and mixed tartrate deserve special attention due to its medical, pharmaceutical and industrial applications, e.g., 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 anemia in animals, ferrous tartrate as a catalyst in manufacture of champagne, tanning action of ferrous tartrate to tan skin, the use of manganese tartrate crystals in chemical temperature indicators, strontium tartrate used in ammunition units, zinc tartrate with other compounds form a bright coating and used as protecting powder for metals etc. [18,22-27]. Because of wide applications of such compounds, many more authors also studied the growth and characterizations of different compounds [28-32]. The purpose of publish the paper is, according to us, to report for first time the growth and characterization of magnesium levo-tartrate crystals in silica gel at the ambient temperature.

II. MATERIALS AND METHOD

GROWTH OF CRYSTAL

The gel growth method has attracted the attention of many researchers as it has been proven the successful method to grow the crystals of such compounds having sparing solubility in water or decompose before melting. There are some

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advantages of gel technique, i.e., gel method is simple, cheap, highly economical, at every stage crystals can be practically observed, crystal can be harvested without damaging the crystal faces etc.

In the present study, the first time attempt has been made to grow the magnesium levo-tartrate crystals by gel growth method. Single diffusion reaction technique has employed for crystal growth process. The borosilicate glass test tubes were used as crystallization apparatus having length 20 cm and internal diameter 2.5 cm, which were placed vertically on wooden stand. Silica gel was used as growth media. The gel prepared from mixing aqueous solution of sodium metasilicate having 1.05 specific gravity and 1M solution of levo-tartaric acid. Initially, stock solution has prepared, i.e., Sodium metasilicate was added in distilled water in proper ratio and stirred so it becomes milky. This was left to settle down physical dense impurities for a week. After this it was filtered by Whatman filter papers twice. Filtered solution was centrifuged up to 10,000 RPM for 40 minutes, so all extra impurities could settled down. Then again it was filtered before using. By using this stock solution, required solution of 1.05 specific gravity was prepared. Simultaneously, levo-tartaric acid solution of 1M was prepared by mixing distilled water, stirred and filtered. 1M levo-tartaric acid and 1.05 specific gravity sodium metasilicate solution were mixed so that the pH of the mixture set to 5.00. This mixture was transferred into different test tubes and keep at rest to convert in the form of gel. After gelation took place, 15ml aqueous solution of 2M magnesium nitrate was gently poured without disturb the gel surface.

III. EXPERIMENTAL TECHNIQUES

Powder X-ray diffraction analysis

The crystalline phase of the title compound is identified at room temperature by using Powder X-ray diffraction technique. Powder X-ray diffraction technique has been used as a non-destructive fingerprinting technique in the laboratory and industry for several decades. Every solid crystalline compound gives its own unique X-ray diffraction pattern consisting of a set of Bragg peaks. The diffraction pattern for a compound can be considered analogous to a fingerprint or barcode with the peak positions determined by the unit cell symmetry and lattice parameters. When the XRD data recorded, it can be used these fingerprints to identify not only what phases are present in the sample but also index the pattern to obtain information about the unit cell size and shape in addition to know the cell parameters of the crystal [33,34]. For the present study, X-ray diffraction pattern was recorded by Philips X'pert X-ray diffractometer having CuK α radiation with wave length 1.540598 Å.

FTIR Spectroscopic analysis

Infrared spectroscopic study deals with infrared region of the electromagnetic spectrum. Basically, IR Spectroscopic method covers a range of techniques, mostly based on absorption spectroscopy. FTIR is useful technique to identify organic and/or inorganic substances. It can also be useful to quantitate some components of an unknown mixture. This technique is equally applicable to the analysis of solids, liquids and gases [21]. FTIR spectroscopic analysis of some crystals comprising of pure and mixed tartrate compounds were reported by earlier researchers [8,9,31-36]. The FTIR spectrum of powdered sample of grown magnesium tartrate crystals was recorded in the range of 400 cm⁻¹ to 4000 cm⁻¹ using a Perkin Elmer Fourier transform infrared spectrometer Model: Spectrum GX.

IV. RESULTS AND DISCUSSION

The magnesium tartrate crystals were grown by single diffusion reaction technique in silica gel medium. Various growth conditions were maintained to study the best growing environment for the growth of good quality crystals as per the method described in the section I. Out of them, Table 1 shows the optimum conditions selected for the growth of good quality crystals.

Parameters	Values
Specific Gravity of sodium metasilicate solution	1.05
pH of the silica gel	5.00
Concentration of levo-tartaric acid	1 M

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Concentration of magnesium nitrate	2 M
Volume of magnesium nitrate	15 ml
Temperature	Room temperature
Colour and nature of grown crystals	Shiny white
Morphology	Spherulitic
Diameter of largest crystal	4 mm
Approximate numbers of crystal in one Test tube	28 to 30
Gelation time	4 days
Period of crystal growth	90 days

Table 1: Selected Optimum growth conditions to grow the crystals.

The grown crystals were gently pulled out from test tubes and cleaned by double distilled water. Photograph in figure 1(a) shows the growing crystals within the test tube during the growth period, while figure 1(b) shows the photograph of grown crystals. The grown crystals of shiny white colour having spherulitic morphology.



Figure 1(a): Growing crystals within the test tube Figure 1(b): Grown crystals of shiny white colour having spherulitic morphology

The powdered sample of grown crystals was studied by powder X-ray diffractometer and the results were analyzed. 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 Win-PLOTR package [37] is used to determine the observed diffraction peak positions of the crystal. Analytical indexing of the powder pattern and determination of the space group are performed using EXPO 2009 [38-39], which is designed to launch the most common indexing programs.

The X-ray diffraction pattern of the powdered sample is depicted in the Figure 2. It shows the well-defined diffraction peaks with indexed peaks, which indicates that the grown material has good crystalline nature.

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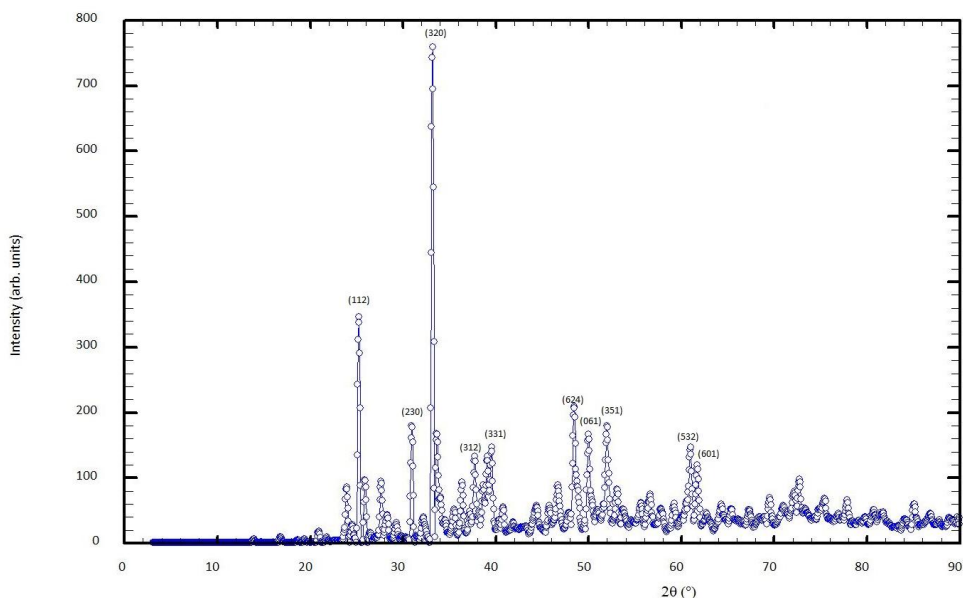


Figure 2: powder XRD pattern of magnesium tartrate crystal using Winploter Software

The lattice parameters are listed in Table 2. The obtained values of lattice parameters, viz., a,b,c and α,β,γ as well as crystal structure, lattice type along with space group. From the powder XRD pattern, it is confirmed that grown crystal has orthorhombic structure.

Lattice Parameters	Values
a	9.1820 Å
b	11.2062 Å
c	7.9529 Å
$\alpha=\beta=\gamma$	90°
Crystal family	Orthorhombic
Lattice Type	P
Space group	P m m m

Table 2: Lattice parameters for magnesium tartrate crystal

The selected values of (hkl) correspond to prominent reflections; experimental and calculated values of 2θ and d; as well as intensity are recorded in Table 3. From Table 3, it is clear that the observed values of 2θ and d, are in consistent with calculated values.

Obs. No.	2θ (Exp.)	2θ (Calc.)	d (Exp.)	d (Calc.)	(hkl)	Relative Intensity
1	25.3197	25.654	3.5176	3.4696	(112)	54.64
2	30.9218	30.835	2.8919	2.8974	(230)	37.24
3	33.0808	33.330	2.7079	2.6860	(320)	100.00
4	37.7049	37.925	2.3858	2.3705	(312)	21.51
5	39.5444	39.691	2.2789	2.2698	(331)	19.79

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6	48.4403	48.548	1.8792	1.8737	(024)	35.09
7	50.0003	50.131	1.8241	1.8182	(061)	22.09
8	51.9853	51.808	1.7591	1.7632	(351)	39.44
9	60.9577	60.791	1.5199	1.5224	(532)	23.08
10	61.7246	61.673	1.5028	1.5027	(601)	23.94

Table 3: Experimental and Calculated crystallographic data for magnesium tartrate crystal

Infrared spectroscopy is one of the powerful tool to identify certain chemical groups and bonds in the sample [40-41]. The FTIR spectrum of the powdered sample of the magnesium tartrate crystal is depicted in the Figure 3. From the various absorption peaks, it is confirmed that the various groups and bonds, viz., O-H stretching, C=O stretching, O-H plane deformation, C-O stretching, O-H deformation out of plane and C-H stretching, Metal–Oxygen bonding are present in the powdered sample of the grown crystals.

Table 4 shows the position of peaks as well as its assignments for the various chemical groups and bond present in the powdered samples of the grown crystal. The results are found in consonance with those reported by earlier researchers [42-43].

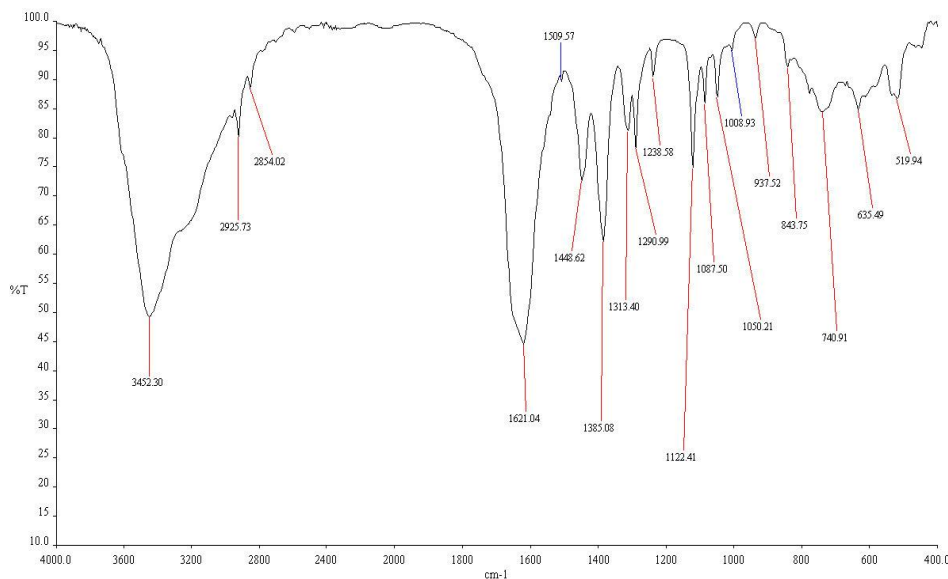


Figure 3: FTIR spectrum of the powdered sample of the magnesium tartrate crystal

Wave numbers in cm^{-1}	Peak assignments
3452, 2925, 2854	O-H stretching
1621, 1509	C=O stretching
1385, 1313	O-H plane deformation
1448, 1290, 1238	C-O stretching
937, 843, 1122, 1087, 1050, 1008	O-H deformation out of plane and C-H stretching
740, 635, 519	Metal–Oxygen bonding

Table 4: FTIR peak assignment for magnesium levo-tartrate crystal.

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V. CONCLUSION

Magnesium levo-tartrate crystals were successfully grown by single diffusion reaction technique with the help of test tube in hydro silica gel. The grown crystals have spherulitic morphology and shiny white colour. With the help of powder XRD study, it is found that the powdered sample is crystalline in nature and confirms the unit cell parameters and d-values having orthorhombic structure. From the FTIR spectroscopic study, the presence of O=H, C=O, C-O, C-H and metal-oxygen bonds were confirmed.

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