Available online at www.pelagiaresearchlibrary.com

Pelagia Research Library

Advances in Applied Science Research, 2014, 5(6):224-228

ISSN: 0976-8610
CODEN (USA): AASRFC

Growth and characterization of strontium tartrate crystals in silica gel

D. K. Sawant[1*], H. M. Patil[1] and K. D. Girase[2]

[1]Department of Physics, JES's Arts, Science and Commerce College, Nandurbar (MS), India
[2]Department of Physics, SVS's Arts, and Science College, Dondaicha (MS), India

---

ABSTRACT

Single Crystals of strontium tartrate were grown by a gel technique using single diffusion method. The effect of various parameters like pH of gel, gel density, gel aging, and concentrations of reactants etc. on the growth of these crystals was studied. Whitish, pale yellow, transparent, semitransparent, diamond shaped, some are needle shaped crystals of strontium tartrate were obtained. *Maximum size 4 mm × 5mm, thickness 3mm crystals were obtained. The grown crystals were characterized by X-ray powder diffractometry (XRD), Infrared spectroscopy (FT-IR). The results of these observations are described and discussed.*

Keywords: Gel technique, strontium tartrate, FT-IR, SEM.

INTRODUCTION

Crystal growth is a heterogeneous chemical process in which conversion from one phase to another phase of a compound is involved. In the field of crystal growth gel technique has become more popular and has been used by several investigators [1-6]. Most of the tartrate compounds are insoluble in water and decompose before melting. Hence single crystals of such type of compounds cannot be grown by either slow evaporation or melt technique .In this situation gel method is the appropriate one for their growth. The growth of single crystals of Calcium tartrate was reported (7), single crystals of Strontium tartrate was reported (8). Thermal studies on tartrate crystals grow by gel method were reported by many investigators (9-11).Thermal behavior of Strontium tartrate was reported (8) . Tartrate crystals are of considerable interest, particularly for basic studies of some of their interesting physical properties. Some crystals of this family are ferroelectric [12-14], some others are piezoelectric [15] and quite a few of them have been used for controlling laser emission [16].

In the present paper we reported the growth of single crystals of strontium tartrate grown in silica gel. These crystals are identified and characterized by Infrared spectroscopy (FT-IR) and Scanning Electron Microscope (SEM)

MATERIALS AND METHODS

Good crystals can be grown in gels in a variety of ways with all variant with two basic and extremely simple methods as single diffusion and double diffusion. The single diffusion method was employed in the present work for the growth of strontium tartrate crystals the crystallization apparatus used essentially consists of simple glass test tubes of length 25 cm and diameter 2.5 cm.

Double distilled water was used for dilution, wherever required, throughout the study. Tartaric acid and Strontium Chloride solution was prepared by dissolving this compound in an appropriate amount of distilled water to give the required molarities. Gels of required specific gravity were prepared by adding to the solution of Sodium metasilicate, a calculated amount of redistilled water and a stock solution was kept ready for doing further experiments.

Sodium metasilicate solution of a suitable specific gravity was taken in a 50 ml beaker and tartaric acid solution of particular strength was added drop wise using a tephlon cock burette, constantly stirring the solution in the beaker by magnetic stirrer. Stirring is done to avoid the excessive local ion concentration, which may otherwise cause premature local gelling and make the final medium inhomogeneous and turbid. The Systronic digital pH meter model No.335 was used to measure the pH the solution. After noting the pH value, this solution was gently poured into the test tube, being allowed to fall along the side of the test tube without giving chances for the formation of the bubbles. Test tubes were then closed with rubber corks or cotton to prevent evaporation and contamination of the exposed surface by dust particles of atmosphere.

The gel in the pH range 4 to 5 was usually found to set in 5 to 8 days, depending on the environmental temperature, After ensuring firm gel setting, the saturated solution of Strontium Chloride (supernatant) of particular strength was poured over the set-gel with the help of a pipette, the solution being allowed to fall along the wall of the test tube to prevents the gel surface from cracking. The supernatant solution slowly diffused in to the gel medium, where it reacts with the inner reactant, giving rise to the slow precipitation of $SrC_4H_4O_6$.
The following reaction took place

● $C_4H_6O_6 + SrCl_2 \rightarrow SrC_4H_4O_6 . x\ H_2O + 2HCl$

RESULTS AND DISCUSSION

The various optimum conditions for the growing crystals were found and are reported given in table 1.

Table 1: Optimum conditions for growth of Strontium tartrate.

Conditions	Strontium tartrate
Density of sodium meta silicate solution	1.04 gm/cm^3
Concentration of tartaric acid	1.25M
Volume of tartaric acid	7ml
Volume of sodium meta silicate solution	17ml
pH of the gel	4.2
Concentration of $Srcl_2$	1M
Gel setting time	60hrs
Gel aging time	48hrs
Period of crystal growth	1week
Temperature	Room temperature

Figure 1. Whisker growths of Strontium tartrate crystals in test tube

Different parameters such as concentration of reactants, pH of gel, gel setting time, gel aging time, etc have considerable effect on growth rate. The grown crystals are Whitish semitransparent, pale yellow, rhombohedra shaped, needle shaped crystals were obtained. Some of them were transparent diamond shaped, due to fast growth rate twined crystals are obtained; faces are well developed and polished. Higher concentration of supernatant increased the nucleation density. Perfect crystals were obtained for pH values, between 4 and 5. It was found that strontium tartrate does not nucleate at pH values less than 3. Most of the crystals were larger in size and few crystals close to the gel solution interface were smaller in size. The crystals growing deep into the gel were larger in size

Density of nucleation was reduced and size of the crystals growing deep into the gel was increased by concentration programming. Aging period reduced the nucleation. Figure1 shows whisker growth of strontium tartrate crystals inside test tube. Figure 2.(a) shows Yellowish,Octahedral, some are needle shaped crystals of $SrC_4H_4O_6$.

Figure 2.(b) shows Whitish, semitransparent, Diamond shaped crystals of $SrC_4H_4O_6$.

Fig.2 (a) Yellowish,Octahedral, some are needle shaped crystals of $SrC_4H_4O_6$

Fig.2 (b) Whitish, semitransparent, Diamond shaped crystals of $SrC_4H_4O_6$

Characterization

Strontium tartrate crystals were characterized by FTIR.and SEM

3.1 Fourier Transform infrared (FT-IR) Spectral analysis.

FT-IR is used for structural analysis. In the present study IR spectrum of strontium tartrate sample was recorded using Shimadzu FTIR-8400 at University Institute of Chemical Technology, North Maharashtra University Jalgaon. Figure 3.shows FT-IR spectrum of strontium tartrate. The IR spectra of these grown crystals was recorded in the wave number range 400cm^{-1}- 4000cm^{-1}.The band at 3531.78$cm^{=1}$, 3381.33$cm^{=1}$, 3158.54$cm^{=1}$ are due to O-H stretching mode and water of crystallization. The band at 2380.95cm^{-1} is assigned to C-H stretching vibrations. Strong asymmetrical band at 1591.33 cm^{-1} is attributed due to C=O stretching in Carboxylate ion, and the peaks at 1373.38cm^{-1}, 1274.03cm^{-1}, 1127.43cm^{-1} are due to O-H in plane bending. The bands at 1078.24cm^{-1}, 1000.12cm^{-1} are due to C-O stretching mode. The absorption bands are found at 928.76cm^{-1}, 841.96cm^{-1} are due to the metal oxygen bonding. The absorption between 689.57cm^{-1}, 640.39$cm^{-1}$and 477.40cm^{-1} are due to presence of water of crystallization.

It is confirmed that Water of crystallization and metal oxygen bonding is presented in the reported.

3.2. Scanning Electron Microscope (SEM)

The study of the surface of the crystal gives valuable information about its internal structure. In the present work powdered sample of strontium tartrate crystals was examined by using SEM technique at the National Chemical Laboratory, Pune. Figure 3.2(a) illustrates SEM photographs of crystals of strontium tartrate crystal. An enlarged SEM image is shown in figure 3.2 (b). It shows plate like crystal morphology. These crystals are grown by layer deposition. Thick and thin layers are seen in figure. The individual plates of samples are flat and the plates with the sharp edges were observed. On some plates further plate like growth was observed. Boundaries of the plate like structure are not very sharp. The presence of small grain structures, along with the plate like microstructures;

interlocked with each other is observed. On higher magnification plate like structure is clearly seen. The SEM images of strontium crystals are reported [17].

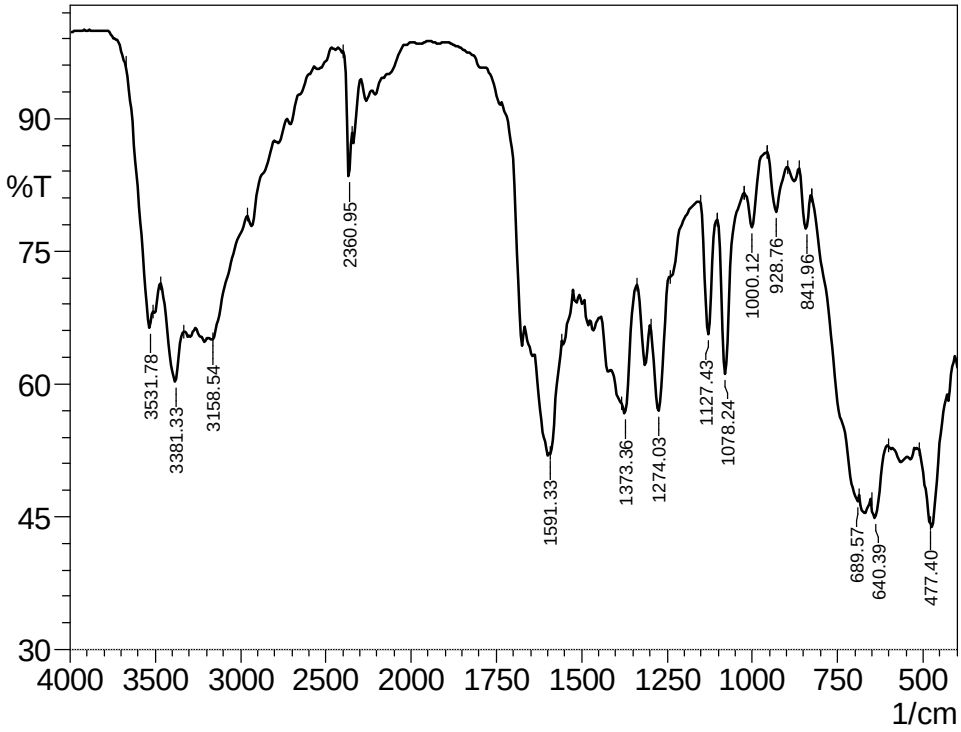
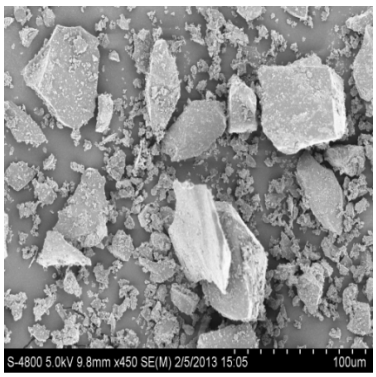


Figure 3.1- FT-IR Spectrum of Strontium tartrate crystal



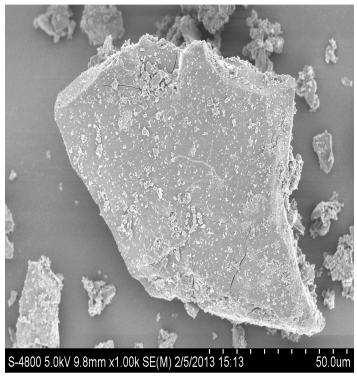


Figure 3.2. (a) **Figure 3.2. (b)**
Figure3.2 (a) SEM image of Strontium tartrate crystal
Figure 3.2(b) Magnified SEM image of Strontium tartrate crystal

CONCLUSION

From the experiments on the growth of strontium tartrate crystals in the system $Srcl_2$ -Na2SiO3-C4H6O6, the following conclusions may be drawn:

1. The growth of strontium tartrate crystals is accomplished by allowing diffusion of strontium chloride through silica gel impregnated with tartaric acid in a single-gel-single tube system.
2. The crystals exhibit Diamond, Octahedral morphology even under varied conditions of growth. Maximum size of the grown crystals under optimum conditions was 3mm in thickness.
3. The crystals are identified and characterized by different methods.
4. The absorption property of a gel plays an important role during the crystallization of strontium tartrate crystals.
5. SEM photographs shows plate like crystal morphology of the Strontium tartrate..

Acknowledgement
The authors are grateful to Head, Department of Physics, Pratap College, Amalner, for providing laboratory facilities. Our special thanks to authorities of NCL, Pune for help in SEM and the Director University Institute of Chemical Technology, North Maharashtra University Jalgaonfor providing FT-IR facilities. The authors also thanks to Principal, J.E.S's Arts and Science College Nandurbar for encouragement and necessary support

REFERENCES

[1] Trivedi,S.G and Joshi, M.S., (**1970**), *Kryst. Und.Tech*.15, 1131.
[2] Ittyachen, M.A. and Kurien, K.V., (**1979***)*, *J. Cryst. Growth*,47, 743.
[3] Joshi, M.S., Mohan Rao, P., and Antoni, A.V., (**1981**), *Bull. Mater. Sci*., 2,127.
[4] Armington, A.F. and O' Cornnar, J.J. (**1968***)* *J. Cryst.Growth*, ¾, 367.
[5] Srinivasan N and Natarajan S, *Indian J. Phys*., 70A, (**1996**), 563.
[6] Arora, S.K. (**1981**), *Prog. Crystal Growth Characterization*, 4, 345.
[7] Henisch H.K, Dennis J and Hanoka J.I **1965** *J. Electro Chem,Soc*. **112** 627**(1965).**
[8] Rahimkutty M.H,Rajendra Babu ,K.Shreedharan *Bull. Mater. Sic.* 24 249-252**(2001).**
[9] Henisch H.K. Crystal growth in gels University park, PA: The Pennsylvania university Press**(1973).**
[10] Kotru P.N,Gupta N.K,Raina K.K and Koul M.L, *Bull.Mater.Sci*. **8** 547**(1986)**.
[11] Oswal,S.K.,Ph.D.thesis,N.M.U.Jalgaon**(1995).**
[12] M. M. Abdel-Kader, FI-Kabbany, S. Taha, M. Abosehly, K. K. Tahoon, and A. EI-Sharkay, *J. Phys. Chem. Sol*, Volume 52,Page 655, **1991**.
[13] H. B. Gon, *J. Cryst. Growth*, Volume 102,Page 501,**1990**
[14] C. C. Desai and A. H. Patel, *J. Mat. Sci. Lett*, Volume 6, Page **1066**, 1987.
[15] V. S. Yadava and V. M. Padmanabhan, *Acta. Cryst, B* Volume 29,Page 493, **1973**.
[16] L. V. Pipree and M. M. Kobklova, *Radio Eng. Electron Phys*, (USA),Volume 12,Page 33, **1984**.
[17] Sachin J. Nandrea, Sharda J. Shitoleb and Rajendra R. *Ahirec Advances in Applied Science Research*, **2013**,4(5):223-231