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Archives of Physics Research, 2014, 5 (5):31-36
(http://scholarsresearchlibrary.com/archive.html)

Archives of Physics Research
Scholars Research Library
ISSN : 0976-0970
CODEN (USA): APRRC7

Optical, structural and elemental analysis of calcium tartrate crystals grown by gel method

[1]S. S. Sonawane and [*]R. R. Ahire

[1]Department of Physics, J. J. T. University, Jhunjhunu (Rajastan)
[*]Department of Physics, S. G. Patil College, Sakri, Dist: Dhule (M.S.)

ABSTRACT

In present study, single crystals of calcium tartrate (CaTr) were grown by a simple gel technique, using single diffusion method. The optimum growth conditions were optimized by varying various process parameters such as gel concentration, pH of the gel, gel aging, concentration of reactants etc. The test tubes were used as crystallization vessels, while silica gel as growth medium. Gel was prepared by mixing the solution of tartaric acid and sodium meta silicate. After setting the gel, it was left for aging. After two or three day's duration, the supernatant calcium chloride of different molarities was poured over the set gel. After 48 to 60 hours of pouring the supernatant, the small nucleation growth was observed below the interface of gel. In the present work few prismatic, semitransparent crystals, semi transparent and some whitish, crystals of calcium tartrate were grown. These crystals were characterized by using EDAX, SEM and UV spectroscopy.

Keywords: Gel technique, Calcium tartrate crystal, UV spectroscopy, SEM and EDAX,

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INTRODUCTION

The term 'crystal' has precise meaning within material science and solid state physics. It refers to solid objects that exhibit well defined geometric shapes. Crystals having different shapes are found in nature. The shape of these crystals is dependent on the types of molecular bonds between the atoms as well as on the conditions under which they formed. Growth of crystals by gel method is promising techniques for growing single crystals of substances which are sparingly soluble in water and decompose before their melting point and thermodynamics consideration reveal that since the growth proceeds at near ambient temperature, the grown crystal would contain relatively lesser concentration of non-equilibrium defects. The growing crystals are held in the gel in a strain free manner, thus limiting effects due to impact of the bottom or the sides of the container. In addition in this method almost complete suppression of large-scale movements like convection is achieved which otherwise can be harmful to the crystals perfection. In gel method, the rate of diffusion of reactants can be controlled, since the gels are network of cavities of several thousand of angstroms, in diameter, communicating through slightly smaller orifices. The gel growth technique appeared quite attractive for growing crystals, on account of its unique advantages in terms of crystals produced and the simplicity of the process. Moreover the method is inexpensive and within the scope of the small laboratory.[1-5].

MATERIALS AND METHODS

Good quality crystals were grown in gels in a variety of ways; the single diffusion technique was used in the present work for of the calcium tartrate crystals growth. The growth process involves the diffusion of calcium chloride solution in to a gel in which tartaric acid is impregnated before. The silica gel was used as a growth media. The chemicals used for growth tartrate were $C_4H_6O_6$, $Cacl_2$, and Na_2SiO_3all chemicals were of AR grade. The crystallization apparatus consist of borosilicate glass test tubes of length 25 cm and diameter 2.5 cm placed

vertically on wooden stands. Tartaric acid, calcium chloride solutions were prepared by dissolving these compounds 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. Tartaric acid solution of particular strength was taken in a 100ml beaker and sodium metasilicate solution of a suitable gravity was added drop wise using a tephlon cock burette, constantly stirring the solution in a 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. Here tartaric acid acted as a lower reactant. The digital pH meter was used to measure the pH. The solution after noting pH values, being allowed to fall along the side of a test tube without giving chance 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 of the gel by dust particles of the atmosphere. The solution was found to be strongly depends on pH. High pH value gel takes lower time to set than low pH value, depending on the environmental temperature. After ensuring firm gel setting, the saturated mixed solution of calcium 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 ions (Ca++) slowly diffused in to the gel medium where it reacts with inner reactant.[6-9].

The following reaction is expected to take place in the formation of calcium tartrate crystals.

$C_4H_6O_6 + CaCl_2 \text{-------} \rightarrow C_4H_4O_6Ca + 2HCl$

RESULTS AND DISCUSSION

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

Table 1 Optimum condition for growth of Calcium tartrate crystal

Various process parameter	Optimum conditions
Density of sodium meta silicate	1.04gm/cm^3
Concentration of Tartaric acid	1.0M
Volume of Tartaric acid	5ml
Volume of sodium meta silicate	19ml
pH of gel	4.5
Concentration of Calcium chloride	1.0M
Temperature	Room temperature
Gel setting time	44hr
Gel aging time	30hr
Period of crystal growth	1weeks

Different parameters such as gel setting time, gel aging time concentration of react ants, pH of gel, impurities in the solvent, , etc have considerable effect on growth rate. Figure 1. Show the optical photograph of growth of calcium tartrate inside the test tube. Figure 2(a), (b), (c) illustrates different morphologies of pure calcium tartrate crystals grown under different conditions of growth.

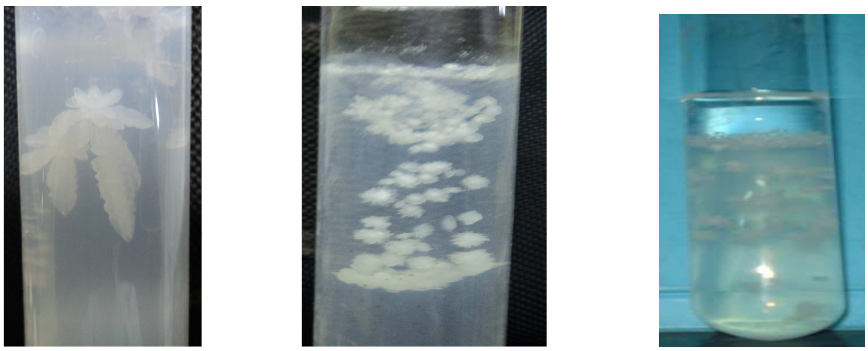


Figure 1. Show the optical photograph of growth of calcium tartrate inside the test tube

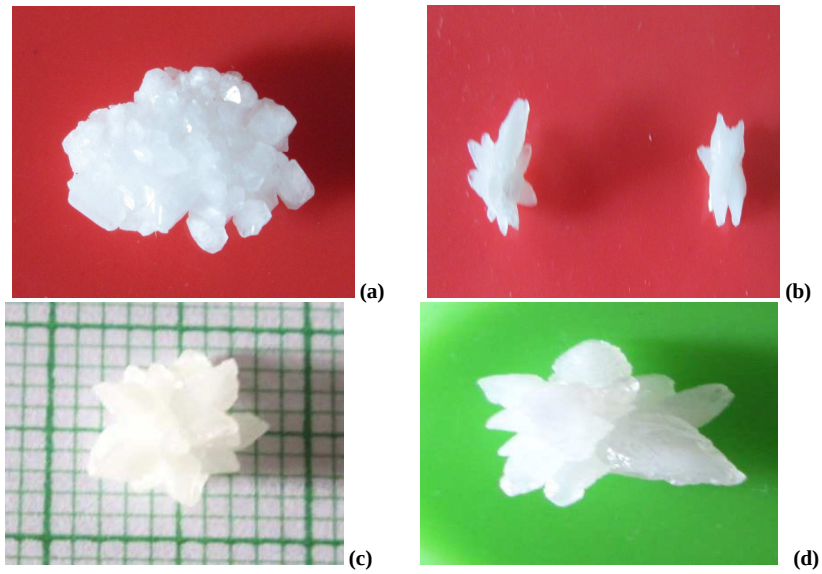


Figure 2.(a,b,c &d) Optical photograph showing growth of calcium tartrate crystals in silica gel at different concentrations of supernatant. Transparent , pale yellowish crystals of calcium tartrate. Transparent and semitransparent, needle shaped calcium tartrate crystals. Magnified needle shaped calcium tartrate crystal. Whitish, semitransparent crystals of calcium tartrate

Characterizations.

Tartrate crystals grown were characterized by XRD SEM, UV spectroscopy.

4.1 UV Absorption Spectroscopy

Absorption spectra of calcium tartrate crystals were recorded using a SHIMADZU UV-2450 UV-Vis spectrophotometer over the wavelength range 200nm – 1400nm at Department of Physics; Savitribai Phule Pune University . Figure 3. shows UV absorption spectra of calcium tartrate crystals. From the spectrum, it has been inferred that calcium tartrate crystals have sufficient transmission in the entire visible and IR region. The absorption coefficient is high at lower wavelength and the wide transparency from 200 nm suggesting their suitability for second and third harmonic generations of the 1064nm radiation [10-12].

The band gap energy of the calcium tartrate crystals with the obtained wavelengths are calculated using the following simple conversion equation; Band gap energy (eV) = 1240/wavelength (nm).Band gap energy is presented in the table 2.

Table 2 Band gap energy of calcium tartrate

Crystal	λ (nm)	Band gap Energy (eV)
Calcium tartrate	270	4.59

4.2 Scanning Electron Microscopy (SEM)

This technique combines of the resolution and analytical power with much ease of operation. Images can be formed from a very wide range of materials. From metals to ceramics, semiconductors to polymers. These materials can be examined with low energy secondary electrons, with high energy back scattered electrons or with other emission such as light, heat and sound. The high depth of field of the SEM images makes it especially suitable for the study of the fracture surfaces and complex microstructures such as those found in composite materials.

In the present work powdered sample of calcium tartrate crystals was examined by using SEM technique at the UDCT, NMU, Jalgaon. The study of the surface of the crystal gives valuable information about its internal structure. Figure 5 (a) illustrates SEM photographs of single crystals of calcium tartrate crystal. An enlarged SEM image is shown in Figure 5 (b) Thick and thin layers are seen in figure. It is observed that due growth conditions small voids are created at the grain boundary. Plate like structures are stuck together to flat surface of crystallite. Miss shaped growth features are observed along with small grain size particles. It shows plate like crystal morphology. These crystals are grown by layer deposition. 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.[13].

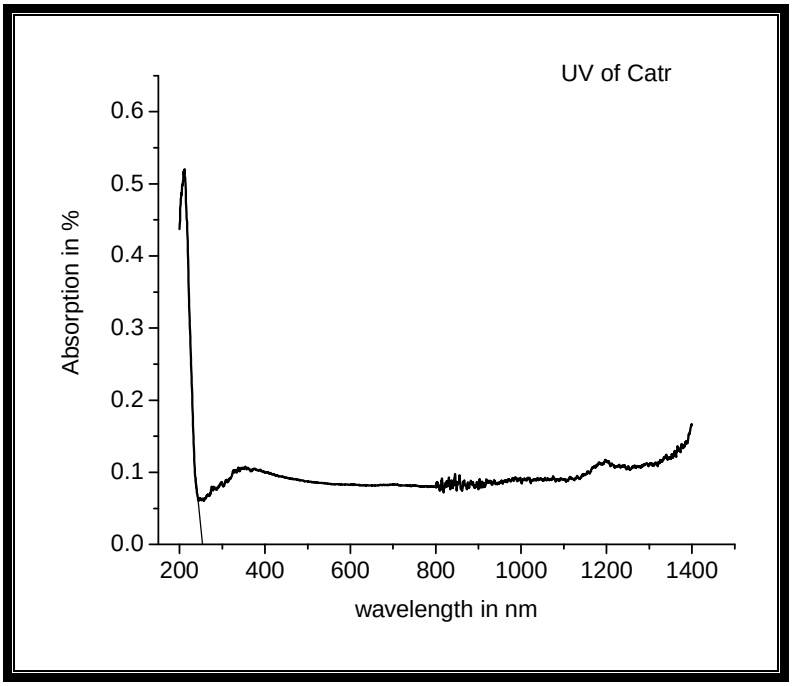
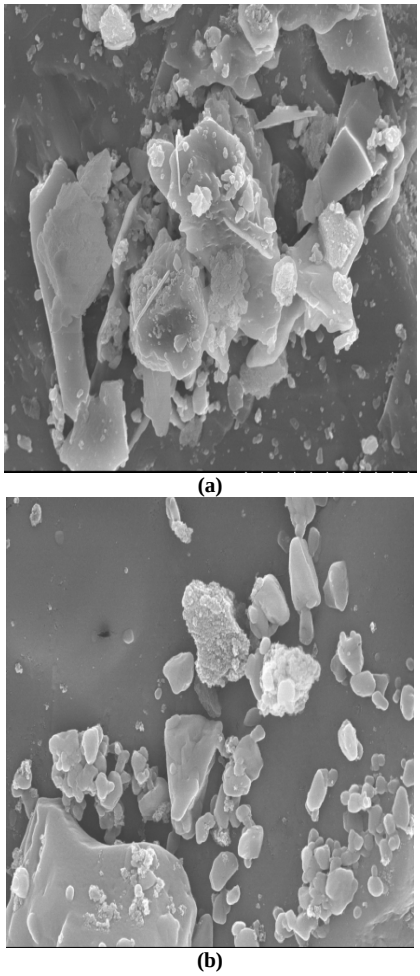


Figure 3 Optical absorption spectra of calcium tartrate



(a)

(b)

Figure 4 (a) SEM image of calcium tartrate crystal. (b) Magnified SEM image of calcium tartrate

4.3 Energy Dispersive Analysis by X-rays (EDAX):

Energy Dispersive analysis by X-rays (EDAX) is used for the quantitative analysis and is also called as elemental analysis. In the present work elemental analysis of gel grown calcium tartrate crystals was carried out at the UDCT, NMU, Jalgaon. Figure 5 shows EDAX spectrum of calcium tartrate.

The peak ranging from 0.7KeV to 3.8KeV clearly indicates the presence of calcium in the sample it also gives the 2.0 counts per second. The relative concentration of the calcium, oxygen and carbon is observed 12.85%, 65.12% and 22.03% respectively.

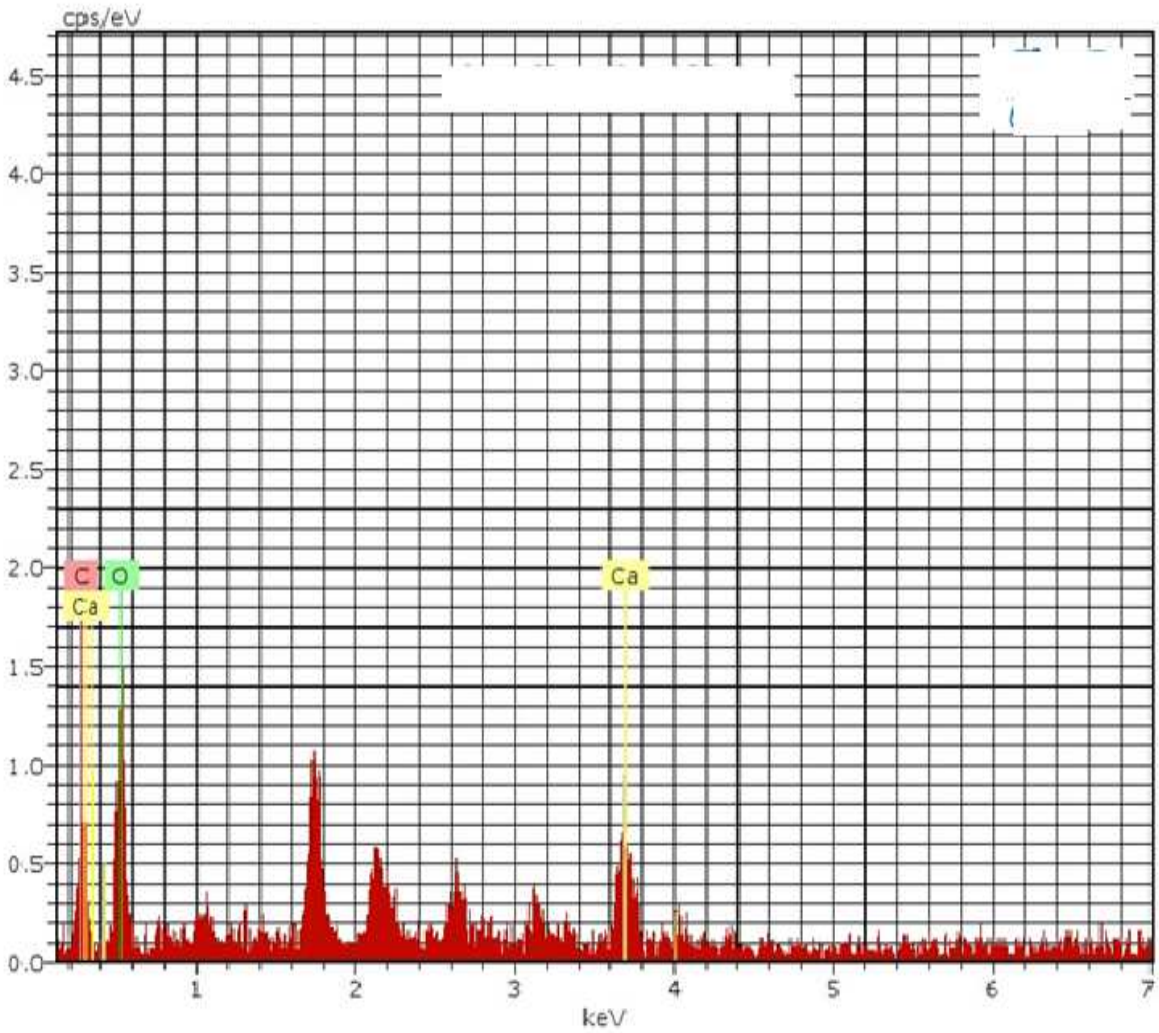


Figure 5 EDAX Spectrum of calcium tartrate

CONCLUSION

From the experiments on the growth of Calcium tartrate crystals in the system, the following conclusions may be drawn.

1)Gel method is found suitable for growing calcium tartrate crystals.

2)The growth of calcium tartrate crystals was accomplished by allowing diffusion of calcium chloride solution through silica gel impregnated with tartaric acid in single diffusion gel tube system, with all growth conditions; calcium tartrate crystals assume Transparent, pale yellowish crystals of calcium tartrate. Transparent and semitransparent, needle shaped crystals..

3)The crystals obtained in silica gel with average size of 6x4x3mm by single diffusion method.

4)Different habits of calcium tartrate crystals can be obtained by changing parameters like gel density, gel aging, pH of gel, concentration of reactants etc.

5)The UV spectroscopy, SEM and EDAX studies suggested different characteristics features and morphology of grown crystals.

Acknowledgement
The authors are grateful to Dr.P.P.Patil, School of Physical Science, N.M.U., Jalgaon for providing XRD facilities. Department of UDCT, N.M.U., Jalgaon for providing. SEM facilities. We are thankful to Dr Sanjay Dhole, Savitribai Phule Pune University for providing the facility of UV spectroscopy and also indebeted to the Dr. D.L. Torwane, Principal, S.G.Patil Arts, Comm. and Science College, Sakri for encouragement.

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