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Study of Growth, EDAX, Optical properties and Surface Morphology of Zinc Tartrate Crystals

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Crystals of Zinc Tartrate were grown by a simple gel technique using single diffusion method. The optimum growth conditions were established by varying various parameters such as pH of gel solution, gel concentration, gel setting time, concentration of reactants etc. Crystals having different morphologies and habits were obtained. The crystals were characterized using Scanning Electron Microscope (SEM), UV, and Energy dispersive X-ray spectroscopy (EDAX). A result of the UV-Visible spectroscopy confirms nonlinear optical property of the crystals.

Keywords: Gel growth, Zinc tartrate crystals, SEM, UV, EDAX.

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1. INTRODUCTION

In recent years, crystal growth in gel has attracted many investigators [1-2]. The technique for growth of single crystals has been developed over the years to meet the need for basic research and applications. Good single crystals are essential for variety of scientific and commercial purposes [3-6]. The gel medium prevents turbulence and being chemically inert, it provides a three dimensional crucible which permits the reagents to diffuse at a desirable controlled rate. A series of pure and mixed crystals have been grown by several researchers with the aim of identifying new material for practical and industrial purposes [7-11]. Single crystals are the backbone of the modern technological revolution. Compounds of tartaric acid find several particle applications in science and technology because of their interesting physical properties such as dielectric, ferroelectric, piezoelectric and non linear optical properties [12-15].

An NLO material is a compound in which a nonlinear polarization is invoked on application of intense electric field. This electric field results from the external application of an intense laser-source. The nonlinear material is different from the linear material in several aspects. A nonlinear material is one, whose electrons are bound by very short springs. If the light passing through the material is intense enough, its electric field can pull the electrons so far that they reach the end of their springs. The restoring force is no longer proportional to the displacement and then it becomes non linear. The electrons are jerked back roughly rather than pulled back smoothly and they oscillate at frequencies other than the driving frequency of light wave. These electrons radiate at new frequencies, generating the new wavelength of light. The exact values of the new wavelength are determined by conservation of energy. The energy of the new photon generated by the nonlinear interaction must be equal of the photons used.

The art of growing crystal in gel is not new for researchers because of its simplicity, inexpensiveness and crystals can be grown at ambient temperature. But the challenges and opportunities in understanding the growth features and morphology of grown crystal remain there. Crystals of great interest from both solid state sciences as well as technological point of view have been reported by many investigators using gel method [16]. The purpose of the present paper is to report growth and influence of various parameters on the growth mechanism of crystals of zinc tartrate in silica gel at ambient temperature. The result of studies of the optical properties, giving confirmation about nonlinear behavior of the material is also reported.

2. MATERALS AND METHODS

Zinc tartrate shows poor solubility in water, hence it was thought worthwhile to grow such a kind of material by chemical reaction at controlled rate using gel method. The crystallization apparatus for growth of zinc tartrate crystals consist of borosilicate glass tube of length of 25 cm and diameter 2.5 cm placed vertically on plastic stand. Silica gel was prepared by acidifying pure sodium meta silicate of specific gravity 1.04 gm/cm^3, with tartaric acid of a concentration in accordance with the requirement of a particular pH value.

The tartaric acid solution was added slowly to sodium meta silicate solution with continuous stirring to avoid any local ion concentration, which would otherwise cause premature local gelling and make the final solution inhomogeneous. Here tartaric acid acted as a lower reactant. A fixed amount of gel solution with the desired value of pH was then transferred to several test tubes. The test tubes were sealed with cotton plug to prevent fast evaporation and contamination of exposed surface of the gel. The solution was then allowed to set.

Gel setting time was found to be strongly dependent on pH. High pH value gel takes lower time to set than low pH value. After confirming the gel setting, aqueous solution of zinc chloride of required concentration was then poured slowly along the sides of the tube to avoid

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breaking of the gel. Zinc chloride solution acted as upper reactant whose ions move through the narrow pores of the silica gel leads to reaction between these ions and the ions present in the gel as lower reactant.

The following reaction was expected to take place inside the gel

$$C_4O_6H_6 + Zncl_2 \rightarrow ZnC_4H_4O_6 + 2HCl$$

Crystals of zinc tartrate are whitish, semi-transparent, diamond shaped. Crystals of size 3mm x 4mm and thickness about 2 to 3 mm are obtained.

Different parameters such as concentration of reactant, pH of gel, impurities in the solvent, gel setting time, gel aging time, etc. have considerable effect on growth rate. Near the interface of gel, dendritic growth is observed due to fast growth rate. However as the reactants percolates through the gel, the controlled reaction occurs below the gel interface, at the depth of 3 to 4 cm. Hence good quality, semitransparent, well developed crystals are observed. This results due to decrease in concentration of reactants, below gel interface. Table 1 gives the optimum conditions for the growth of zinc tartrate crystals in silica gel.

Table 1 – Optimum condition for growth of zinc tartrate crystals

No	Process parameter	Optimum condition
1.	Density of sodium meta silicate	1.04 gm/cm^3
2.	Concentration of Tartaric acid	0.9 M
3.	Volume of Tartaric acid	5 ml
4.	Volume of sodium meta silicate	17 ml
5.	pH of gel	4.4
6.	Concentration of Zinc chloride	0.5 M
7.	Temperature	Room temperature

In present work, Fig. 1 shows ZnTr crystals in test tubes and Fig. 2 shows photographs of semi-transparent crystals of zinc tartrate growing under different conditions. Fig. 3a illustrates different habits of pure zinc tartrate crystals grown under different conditions. Fig. 3b shows crystal in magnified form. Few whitish, some pale yellowish, semi-transparent crystals, were observed.

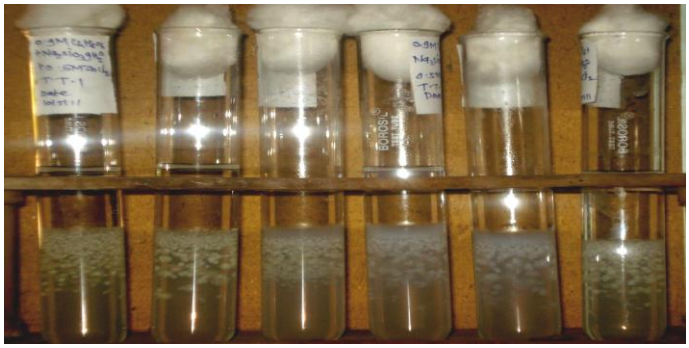
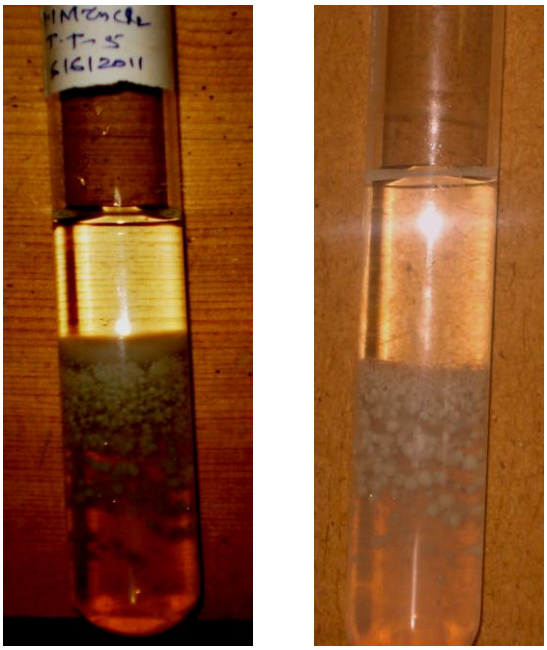
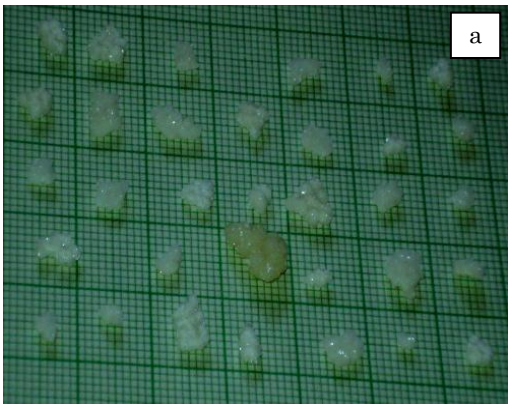


Fig. 1 – Crystals in test tubes



(a) (b)

Fig. 2 – ZnTr crystals in the test tube under variation of upper reactant concentration (a) 1.2 M, (b) 0.9 M



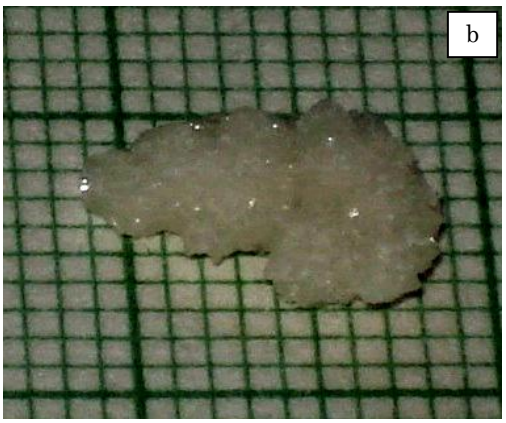


Fig. 3 – Various habits of ZnTr crystals (a) ZnTr crystal in magnified form (b)

3. RESULTS AND DISCUSSION

3.1 Scanning Electron Microscope (SEM) of Zinc Tartrate Crystals

This technique combines the resolution and analytical power with much ease of operation. Images can be formed from very wide range of materials, from metals to ceramics and semiconductors to polymers. These materials can be examined with low energy secondary electrons, with high energy 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 fractured surfaces and complex microstructures such as those found in composite materials. The study of the surface of the crystal gives valuable information about internal structure. Fig. 4 illustrates SEM photographs of single crystal of Zinc tartrate crystal. 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. The plates with the sharp edges were observed. On some plates further plate like growth was observed.

3.2 EDAX of Zinc Tartrate Crystal

Elemental Dispersive Analysis by X-rays (EDAX) is used for the quantitative analysis. When a beam of electron strikes a specimen, a fraction of the incident electrons excites the atoms of the specimen, which then emit X-rays when they return to their ground state. The energy of these X-rays is strictly related to the atomic number of the elements excited and therefore their detection forms the basis of elemental analysis in the electron microscope.

Fig. 5 shows E-DAX spectrum of Zinc tartrate. Table 2 shows the values of elemental content of the crystals as measured by EDAX technique (**At. %**) and the theoretical calculations from molecular formula (**Wt. %**).

Table 2 – Values of elemental content of Zinc tartrate

No	Elements	Experimental Values	
		Wt. %	At. %
1	C	22.54	48.62
2	O	15.11	24.48
3	Si	2.24	2.06
4	Cl	3.20	2.34
5	Co	0.27	0.12
6	Zn	56.26	22.36
7	I	0.38	0.08

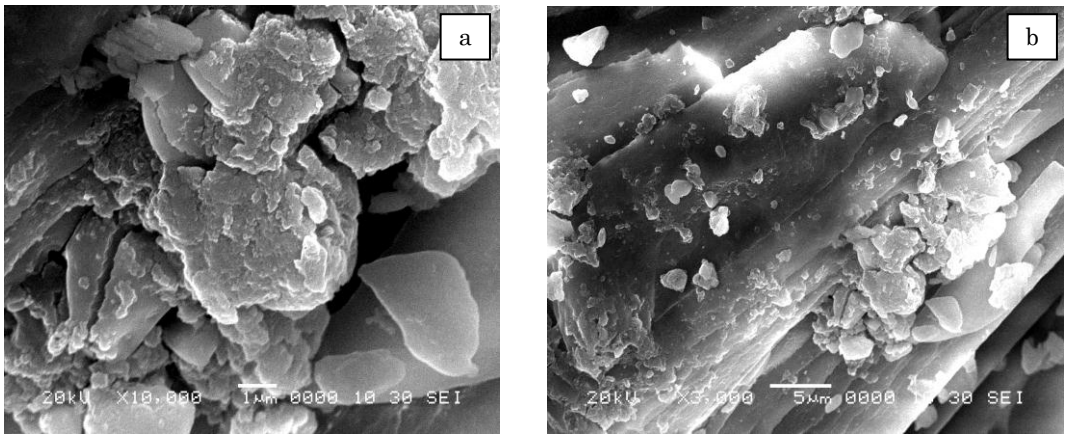


Fig. 4 – SEM image of Zinc tartrate crystal (a), Magnified SEM image (b)

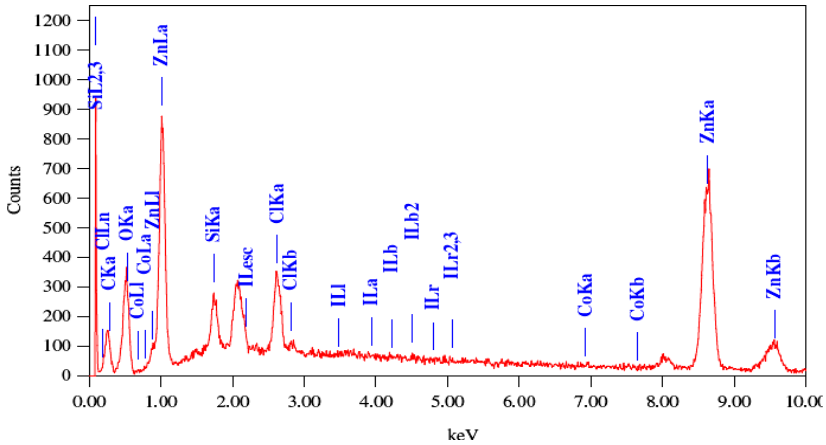


Fig. 5 – Energy dispersive spectrum of Zinc tartrate crystal

3.3 UV- Absorption spectroscopy

Absorption spectra of Zinc tartrate crystals were recorded using a SHIMADZU UV-2450, UV- Visible spectrophotometer over the wavelength 100-600 nm. Fig. 6 shows UV absorption spectra of Zinc tartrate crystals. From the spectrum, it has been inferred that Zinc tartrate crystals have sufficient transmission in the entire visible and IR region. The absorption coefficient is high at lower wavelength and wide transparency from 300 nm. This result suggests zinc tartrate crystals' suitability for second and third harmonic generations for the 1064 nm radiation [17-18].

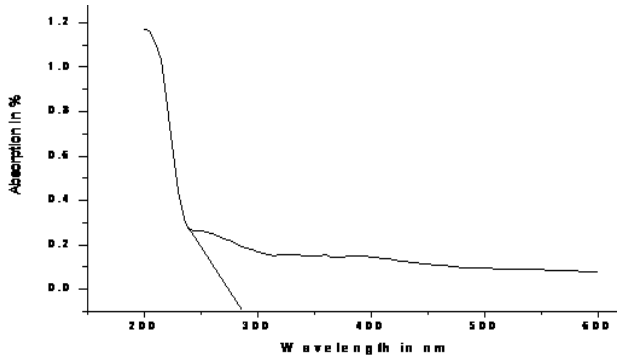


Fig. 6 – Optical absorption spectra of Zinc tartrate

The band gap energy of the zinc tartrate crystals are calculated with the obtained wavelength using the following simple conversion equation:

Band gap energy (eV) = 1240 / wavelength (nm)

The band gap energy of Zinc tartrate crystal is found to be 4.35 eV at the wavelength of 285 nm

4. CONCLUSIONS

i) Gel growth technique is suitable for growing crystals of Zinc tartrate.
ii) Different habits of Zinc tartrate crystals can be obtained by changing parameters like gel density, gel aging, pH of gel, concentration of reactants etc.
iii) By performing UV characterization, it is found that Zinc tartrate crystals have nonlinear optical property.
iv) SEM images show morphology of the Zinc tartrate crystal.
v) EDAX confirms the presence of Zinc in the crystals.

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