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Nucleation and growth of barium tartrate crystals in silica gel

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ABSTRACT

Barium tartrate crystals were grown by single diffusion gel technique in a sodium metasilicate gel media at ambient temperature. The effect of various parameters like pH of the gel, gel density, Concentration of reactants, gel aging on the growth of these crystals was studied. The optimum conditions required for the growth of these crystals are worked out. Opaque, whitish, star shaped crystals were obtained. An attempt has been made to explain growth mechanism.

Keyword: Gel technique: Barium tartrate: Star crystals.

INTRODUCTION

A Variety of crystals are required for the purpose of research and application can be grown in silica gels. 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 materials for practical and industrial purposes [1-5]. Single crystals are the backbone of the modern technological revolution. Compounds of tartaric acid find several practical applications in science and technology because of their interesting physical properties such as dielectric, ferroelectric, piezoelectric and non-linear optical properties [6-9]. The art of growing crystals 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 has been reported by many investigators using gel method [10-12]. The purpose of the present paper is to report growth and influence of

various parameters on the growth mechanism of crystals of Barium tartrate in silica gel at ambient temperature.

MATERIALS AND METHODS

The silica gel method was employed in the present work for the growth of Barium 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 Barium chloride, solution was prepared by dissolving these compounds in an appropriate amount of distilled water to give the required morality. 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 Barium 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 $BaC_4H_4O_6$.
The following reactions took place

$$C_4H_6O_6 + BaCl_2 \rightarrow BaC_4H_4O_6.\ x\ H_2O + 2HCl$$

RESULTS AND DISCUSSION

The Various optimum conditions for growing crystals were found and are given in the table 1. Different parameters such as gel density ,concentration of reactants, pH of gel, impurities in the solvent, gel setting time, gel aging time, etc have considerable effect on the growth rate.

Effect of gel density:-It was observed that the transparency of the gel decreases as the gel densities increases .Gels with higher densities set more rapidly than the gels with lower densities. It may be noted that star shaped whitish crystals of Barium tartrate were obtained with sodium metasilicate of density 1.04 gm/cm^3.

Effect of concentration of reactants:-As the concentration of tartaric acid is increased more volume of sodium metasilicate was required to adjust the pH value around 4.2. Increased in concentration of tartaric acid provides more tartrate ions combine with barium ions. With series of experiments the optimum conditions were obtained good quality crystals were grown at 1M concentration of tartaric acid.

Effect of concentration of supernatant:-Barium chloride is used as supernatant with different concentration from 0.2M to1.2M is added over the set gel. It was observed that at 0.2M of concentration of supernatant very few nucleations were observed with very small size of the crystals and crystals were not well defined. Table 2 gives an Effect of concentration of supernatant.

Effect of pH of gel:-It was observed that as the pH increased the transparency of the gel decreased. In the present work good crystals of Barium tartrate are obtained at pH 4.2.The crystals growing at higher pH values were not well defined. This was due to contamination of the crystals with silica gel. It was observed that as the pH of gel increased the number of crystals decreased.

Effect of gel aging time:-Gels were allowed to age for different period before adding the feed solutions. It was observed that as aging time of gel increased, the number of crystals decreased. This is because gel aging reduces the cell size and consequently the rate of diffusion of ions into the gel [13]. Table 3 gives Effect of gel aging time.

Table 1: Optimum conditions for growth of Barium tartrate

Conditions	Barium tartrate
Density of sodium meta silicate solution	1.04 gm/cm^3
Concentration of tartaric acid	1M
Volume of tartaric acid	7ml
Volume of sodium meta silicate solution	17ml
pH of the gel	4.2
Concentration of $BaCl_2$	1M
Temperature	Room temperature

The systematic growth experiments were performed by adding $BaCl_2$ as feed solution of strength varying from 0.2M to 1.2 M over the set gel of pH range 4 to 5 and gel density range 1.02 to 1.05 gm/cm^3. This type of growth has been reported for barium and lead tartrate [14], didymium tartrate [15] .

Figure 1Shows optical photo graph of growth of Barium tartrate single crystals in silica gel at different pH. Figure2 illustrates different morphologies of star shaped whitish crystals of Barium tartrate**.**Figure3 shows Enlarged photographs of some slelected morphology of grown crystals.

Table 2 Effect of concentration of supernatant
pH = 4.2
Aging period = 9 days

Test tube No.	Sodium metasili-cate (1.04 gm/cm^3) ml	Tartaric acid (1M) ml.	Conc. Of Supernatant M.	No. Of Nuclei	Observation
1	17	7	0.2	2	Very few nucleation's', crystal size is very small, crystals are not well defined.
2	17	7	0.4	5	Nucleation density increases, crystals are not well defined, crystal size increased slightly.
3	17	7	0.6	10	Well shining isolated crystals of various star shape and size are observed.
4	17	7	0.8	15	Well shining isolated crystals; whitish star shaped, faces were well developed with large size crystals.
5	17	7	1	25	Good crystals are observed. opaque whitish star shaped single crystals are obtained.
6	17	7	1.2	30	Numbers of crystals are large and are not isolated.

Table 3: Effect of gel aging time
pH = 4.2
Concentration of Supernatant = $BaCl_2$ 1M
Density of gel = 1.04 gm / cm^3

Test Tube No	Sodium Metasi-licate (ml)	Tartaric acid 1M (ml)	Aging time (Hours)	Number of crystals obtained (appr)	Observations
1	17	7	24	Many	Immediate nucleation's, no dendrites are developed at the interface.
2	17	7	48	35	Nucleation observed after 2 hours, (Induction period) crystals are below the interface. Nucleation is observed in controlled manner.
3	17	7	72	25	Induction period increased up to 6 hours, star crystals were observed below the interface, good quality crystals, faces are developed.
4	17	7	96	15	Well-developed crystals, crystals near interface are of different morphology than those well inside the gel, crystals are opaque, whitish, star shaped are observed.
5	17	7	108	5	Nucleation density is very limited, improved crystal quality, large size crystals were grown, and crystal faces were well developed.
6	17	7	120	0	No nucleation observed after a long time.

Fig1. (a) pH=4.2 **Fig1. (b) pH=4.3**

Fig 1. (c) pH= 4.5

Figure 1 Optical photo graph showing growth of Barium tartrate crystals in silica gel at different conditions pH (a) pH=4.2 (b) pH=4.3 (c) pH=4.5

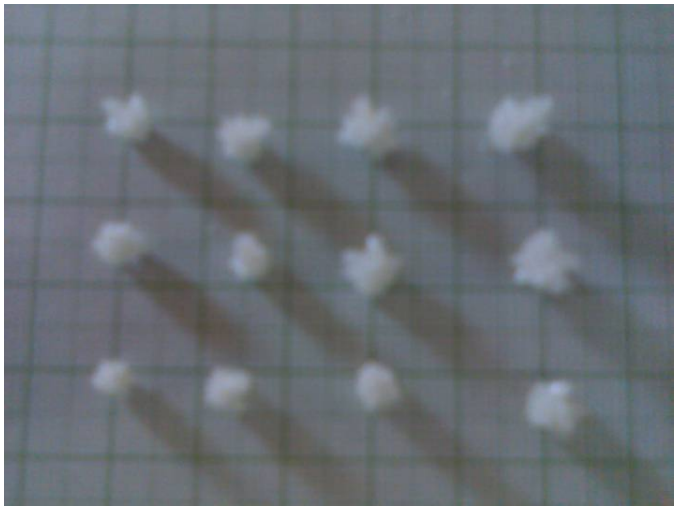
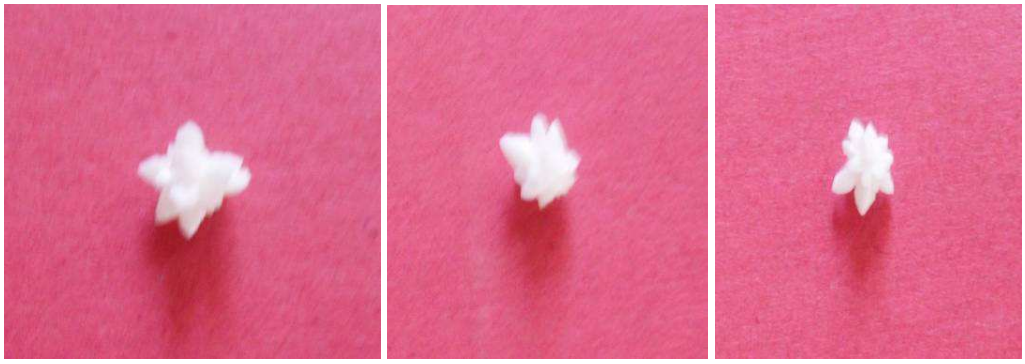
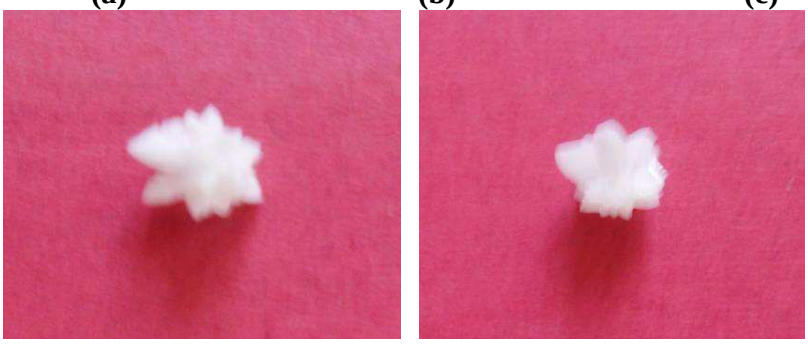


Figure 2 Optical photo graph of enlarge star shaped whitish crystals of Barium tartrate



(a) **(b)** **(c)**



(d) **(e)**

Figure 3 Enlarged View of some selected morphology of grown Barium tartrate crystals.

CONCLUSION

- Whitish star shaped crystals of Barium tartrate were grown at 1.04 gm/cm^3 densities and pH of 4.2
- It was found that Barium tartrate does not nucleate below 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.
- The average length of the grown crystals was found to be 0.5-1.5 cm.
- 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 density and improves the quality of the grown crystals.
- Higher concentration of supernatant increased the nucleation density.

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