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COMPARATIVE STUDIES ON GEL GROWN OXALATE CRYSTALS

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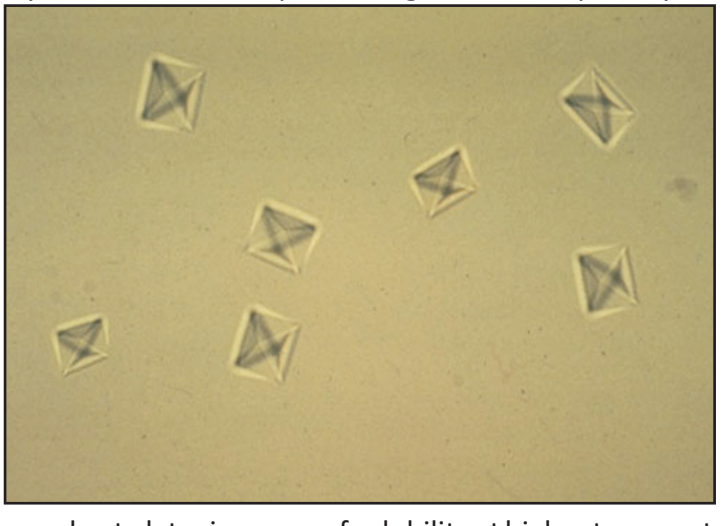
ABSTRACT

An attempt is made in this study to find suitable method of growth of oxalates. Different methods of growth are described. It is further concluded that the best option for the growth of oxalate crystal is gel medium. A summative study of barium oxalate, strontium oxalate and cadmium oxalate is described in support of methods for the growth of oxalates.

KEYWORDS : Crystal growth, gel method, barium oxalate, strontium oxalate, cadmium oxalate.

INTRODUCTION:-

The growths of crystals of various substances have gained considerable concern of several investigators for their practical importance, often for scientific investigations but principally for technological and industrial applications. Hardly any solid state physics investigation is made now a day without an attempt of using well developed crystals. The availability of good single crystals of required properties and characteristics may lead to important applications.



Out of the various techniques for the growth of crystals from melt, vapour or solution, those that require elevated temperature have some inherent difficulties as,

- Crystalline imperfections are more opt to occur due to the lattice disruption by pronounced thermal vibrations.
- The chances of lattice contamination by impurities are profusely increased due to later increase of solubility at higher temperature.
- Point defects and lattice strains are frequently introduced into the growing matrix during the range of coding cycle.

Of all the methods of crystallization at room temperature, the gel method is believed to hold substantial promise of future development due to the following advantages as, the crystals can be observed practically in all stages of their growth, gel medium considerably prevents convection current and turbulence, by remaining chemically inert and harmless, the gel framework acts like a

three dimensional crucible in which the crystal nuclei are delicately held in the position of their formation and growth, thereby preventing damage, if any, due to impact with either bottom or the walls of the container, the gel is soft and yields suitable environment for the growth of crystals, thermodynamic considerations reveal that since the growth process at near ambient temperature, the grown crystals would obtain relatively lesser equilibrium concentrations of defects, since the gel reduces, in effect, that the speed of chemical reaction, crystal could be made to grow much larger than those formed by a similar reaction in water or in molten stage by double decomposition process, all the nuclei are spatially separated thereby minimizing mutual interactions, one can control diffusion rates and nucleation probability and thus design one's own crystallization equipment for obtaining different size and morphology of different crystals and the growth procedure is highly economical; it yields a good crop of crystals with the simple and almost inexpensive equipment. The method can be exploited even in smaller laboratories which do not possess sophisticated equipment to grow crystals.

Many oxalate salts are insoluble in water and decompose before melting; therefore solution method is more suitable technique than the slow solvent evaporation and melts techniques [1] for growing oxalate crystals. In solution method, water solution and gel medium were specially used to get good crystals.

In gel method silica hydro gel and agar gel were commonly used for the growth of oxalate crystals. Barium oxalate [2-3], Barium copper oxalate [4], Barium cadmium oxalate[5], Cadmium oxalate [6-10] were grown in silica hydro gel and Barium oxalate [11-12], Cadmium oxalate [13-14] were grown in agar gel.

In the present work, growth of gel grown barium oxalate [12], strontium oxalate [15] and cadmium oxalate [16] crystals reported earlier in addition to the growth of barium oxalate using additives as reported for the growth of strontium oxalate [15] and cadmium oxalate [16] is reported and comparative summary of the optimum conditions established for the growth of such crystals is presented.

EXPERIMENTAL

The growth of barium oxalate, strontium oxalate and cadmium oxalate crystals were carried out in agar-agar gel and reported [Dalal and Saraf 2009). A single glass tube of length 20 cm and diameter 2.5 cm was used as a crystallizing vessel.

In single diffusion, particularly in conventional method, hot aqueous agar-agar gel solution and a 5ml oxalic acid solution (0.5-1 M) were mixed and kept in test tube for setting. After setting and aging the gel, a 20ml barium chloride (0.5-1M), strontium chloride(0.5-1M), and cadmium acetate solution (0.5-1 M) were added over the set gel separately in the test tube to grow barium oxalate, strontium oxalate and cadmium oxalate crystals respectively. Initially a thin precipitation layer was formed on the surface of the gel. This white precipitation band increases gradually as the diffusion proceeds into the gel. On reversing the reactant, hot aqueous agar-agar gel solution and a 5ml barium chloride (0.5-1M), strontium chloride(0.5-1M), and cadmium acetate solution (0.5-1 M) were mixed separately and kept in test tube for setting. After setting and aging the gel, a 20ml oxalic acid solution (0.5-1 M) was added over the set gel. Nucleation was started readily at the interstitial and inside the test-tube. Heavy nucleation was observed in the test-tube, which was further increased. As in single diffusion techniques, to control nucleation, neutral gel and additive were tried. Neutral gel has reduced the nucleation but did not improve the size of the crystal. To improve the size of the crystal, ammonium chloride solution of different concentrations and quantities were added in the gel. The best results were seen when 8 ml of 4M NH_4Cl solution was added in case of growth of barium oxalate and

strontium oxalate crystal, whereas 8.5 ml of 4M NH4Cl solution was added in case of growth of cadmium oxalate. Transparent prismatic bi-pyramidal platy shaped of average size 6 x 6 x 3 mm crystals were separated after 60 days,

RESULTS AND DISCUSSION

A comparative study of barium oxalate, strontium oxalate and cadmium oxalate in respect of their optimum conditions are described in Table 1.

White precipitation at the interstitial and well inside the tube were observed in conventional method, while spherulite crystals were observed at the interstitial and well inside the gel on reversing the reactants. Spherulitic, transparent and bi-pyramidal growth of crystals at the interstitial and well inside the tube were observed in case of neutral gel. Neutral gel has controlled nucleation up-to some extant but could not improve the size of crystals. NH4Cl was used as an additive for the purpose to suppress the nucleation density and to increase the size of the crystals. Transparent, platy shaped crystals of barium oxalate, strontium oxalate and cadmium oxalate at the interstitial were observed by adding NH4Cl solution along with one reactant during setting of gel. Some good quality and large size crystals of barium oxalate, strontium oxalate and cadmium oxalate are shown in figure 1. Controlled nucleation density and improved quality of crystals might be due to the formation of buffer solute, which may dissolve excess nuclei and provide sufficient nutrient to grow single nuclei.

Table 1 Comparative summary of the optimum conditions established for the growth of barium oxalate, strontium oxalate and cadmium oxalate.

Conditions	Barium oxalate	Strontium oxalate	Cadmium oxalate
% of gel	1.5	1.5	1.5
Concentration of barium chloride, strontium chloride and cadmium acetate	1M	1M	1M
Concentration of oxalic acid	1M	1M	1M
Gel setting period	3 days	3 days	3days
Gel aging	24h	24h	24h
Period of growth	60 days	80 days	60days
Temperature	Room temperature	Room temperature	Room temperature
Quality	Transparent, prismatic and bi-pyramidal	Transparent, prismatic	Transparent, prismatic
Size	6 x 6 x 3 mm	4 x 4 x 3 mm and spherulite of 4 mm diameter	6 x 6 x 3 mm

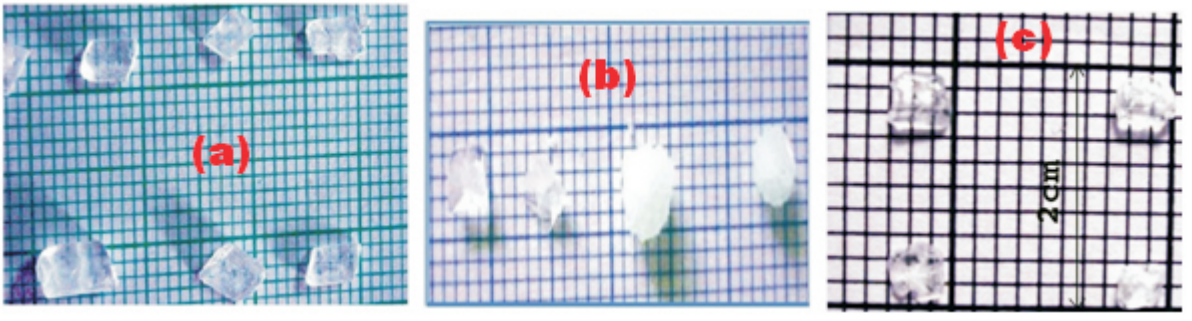


Figure 1: Some good quality crystals of- (a) barium oxalate (b) strontium oxalate (c) cadmium oxalate

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