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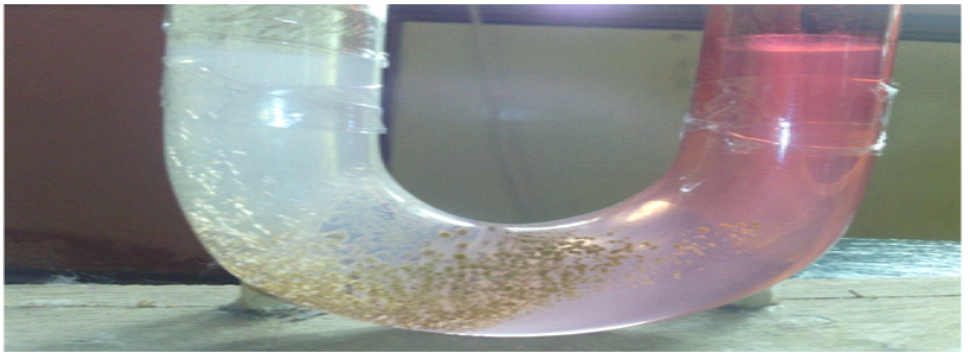

STUDY OF THE GROWTH KINETICS OF COBALT OXALATE CRYSTAL IN AGAR AGAR GEL

S. U. Patil[1] and R. T. Chaudhari[2]
[1]Physics Research Laboratory, Shri V. S. Naik, Arts,
Commerce and Science College, Raver, India.

ABSTRACT

Transition metal oxalate crystals were grown in agar-agar gel The cobalt oxalate crystals were grown by single and double diffusion techniques. The effect on the growth was studied by changing different parameters; like concentration of gel, concentration of reactants, aging period, reversing of reactants, neutral gel and also by using different sizes of the test tube. The nucleation was controlled by using these parameters and optimum conditions were obtained. Such grown crystals were found in different shape and color. The Surface morphology was studied by optical microscopy.

KEYWORDS: Crystalgrowth, Agaragar gel, Optical Microscopy,

INTRODUCTION :

Due to the simplicity and many other benefits, the crystal growth using gel technique has become very popular [1] and is used by many researchers to grow the crystal of good quality and morphology. This method is useful to grow the oxalates because they are insoluble in water and decompose before melting point. Many researchers have grown these crystals by using this technique in silica gel[2, 3, and 4] and gelatin gel, however very few researchers used the agar agar gel. The agar agar gel is not pH dependent and again makes the method simple. Khan et al in 1976 reported the growth of transition metal cobalt oxalate in silica gel [5].YuniarPoncoPrananto et al also found fascinating results while synthesis of low solubility transition metal cobalt oxalate using silica gel [6].

The cobalt oxalate was indirectly useful in the development of supercapacitors [7]and for other uses [8,9].The growth of transition metal cobalt oxalate crystal in agar agargel is not yet found reported. In this paper, the growth study of the cobalt oxalate crystal in agar-agar gel is being reported.

2. Materials

In the present work, the borosilicate glass tubes were used as crystallization apparatus. Test-tube of different diameters of 10, 15, 20 and 25mm and 250 mmlength were used for single diffusion. The U tube of 25mm diameter and 200mm length was used for double diffusion. The chemicals and materials, used were of AR grade (S.D. fine Co. Ltd.).

3. Experimental

For single diffusion,the test tubes were filled by the first reactant (cobalt chloride) of desired volume and molarity. Hot agar agar gel was poured in the test tubes and was kept for setting. The second reactant (oxalic acid) of desired volume and molarity was gently poured along the walls of test tube on the set gel and allowed to diffuse into the gel medium. The open end of tube was closed with cotton plugs and kept undisturbed at room temperature. The ions of supernatant solution reacted with ions of first reactant through capillaries formed in gel medium.

For double diffusion, the gel of desired volume and percentage was poured in U tube and kept for setting. First reactant of desired volume and molarity was poured slowly in one limb and simultaneously second reactant of desired volume and molarity was also poured slowly in another limb of U tube on the set gel. Next day the nucleation were observed near the gel solution interface. After 30 days in single diffusion and 45 days in double diffusion, the crystals were harvested by washing them carefully with double distilled water. As grown crystals were collected and observed under optical microscope.

4.Growth Study

4.1 Effect of gel concentration

Effect of gel concentration on the growth of cobalt oxalate was observed by preparing various concentrationsof 0.25,0.50,0.75,1.0 and 1.50%, while other growth parameters like first reactant (0.75M),second reactant (1.0M),aging period (2 days) and volume first reactant (2 ml),second reactant (25 ml)were kept constant. After setting the gel,the second reactant was poured over the gel. Next day, the precipitationwas observed near the interface of gel. After two days, the observed precipitation was fond to be disappearing and nucleation growth was observed in test tube.Due to the higher concentrationfrom 0.25 to 0.5 %of gel,the size of growing crystals was found to be increasing.

As the concentration was increased from 1.0 to …., 1.50% the number of nucleationwere increased as shown in fig.1 and fig.2. However the sizes of the crystals were very small. But when the concentrationof gel was decreased from 1.0% to 0.5% it was found that the number of nucleationwere decreased and widely separated cobalt oxalates were observed in the test tubes.Due to these experiments it was found that when thepercentage of gel was 0.5 % the large sized separated crystals were grown. The same result wasobserved for both straight and U tube of same size 20 mm x250 mm.

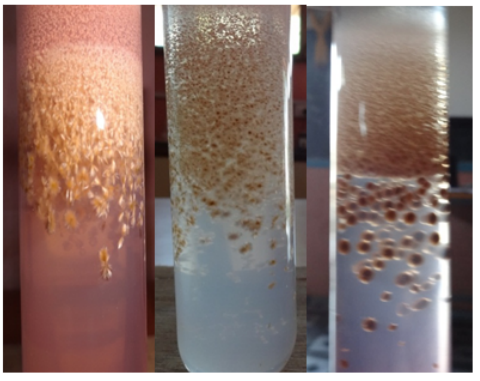

Fig 1: Growth of cobalt oxalate crystal in different concentrations

Fig 2 Growth of cobalt oxalate crystal in double diffusion

4.2 Effect of concentration of reactants.

To observe the effect of concentration of reactants on the growth of cobalt oxalate cryatals, the both reactants were prepared in the concentrations of 0.25M.....,1.0M, while the other growth parameters were kept constant, such as gel concentration (0.5%),aging period (2 days) and volume of first reactant (2 ml) and volume of second reactant (25 ml).

It was found that the number of nucleation was decreased by decreasing the concentrationsof both reactants such as 0.75M and 1M respectively. The same result was observed for both diffusion methods of same size test tubes (20cm x2.5cm).

Meanwhile it was observed that if both reactants were of same concentration (more or less) the rate of diffusion was same and nucleations were found to be 2 cm below interface. In this 2 cm region of nucleation again it was found that when the concentration was of reactants was less but in a equal proportion the crystals were widely separated.

For different concentrations it was observed that, the diffusion rate was faster for higher concentration of second reactant to that of first reactant.

The best result was obtained when the percentage of gel was at 0.50% and the molarity of first reactant was 0.75M and concentration of second reactant was decreased up to 1M,the nucleation growth was controlled and large size cobal toxalate crystals was obtained.

The experimental arrangement to observe the effect of concentration on growth of crystal is shown in fig3.

Also due to different concentrations of reactants the color of the grown crystals changed from pink, dark maroon to dark brown. The same effect was reported by S. J. Joshiet.al. [10].The grown

crystal in different colors are shown in fig.4 and 5.

Fig 3: Experimental arrangement to examine the effect of concentrations on growth of cobalt oxalate crystals

Fig 4: Growth of Pink color cobalt oxalate crystal

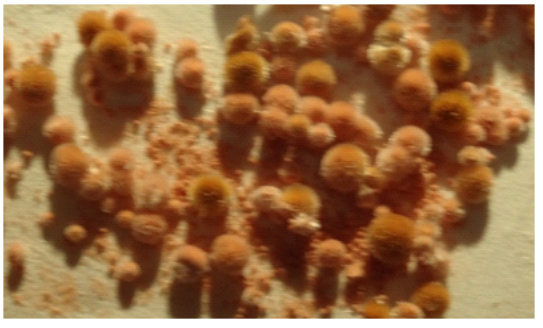

Fig 5: cobalt oxalate crystal in maroon brownish color

4.3. Effect of amount of solutions.

When the amount of first reactant was changed from 1 ml to 5 ml, it was found that due to increasing the amount of solution, the number of nucleation were increases, while the size of grown crystals was found to be decreases. For the growth of cobalt oxalate crystal, the best result was obtained when the amount of solution of first reactant was 2ml.

Similarly amount of second reactant was change from 10 ml to 30ml; it was found that when amount was 10ml, the growths of nucleation were observed at near the interface. While for the 15 ml amount of solution, the growth was observed at 2 cm from the interface.For 20 ml amount of solution, the nucleation was found in the middle of gel column. This result may be due to the gravitational force acting on the second reactant.

4.4. Effect of size of test tubes.

The various test tubes of different diameters and heights were used to observe the effect. The size of test tubes were 15X2.0, 15X 2.5, 20X 2.5 and 25X3.0cm.In the 15X2.0 cm size of test tubes, the morphology of crystal was increased, but the size of grown crystal was decreased. While in the 25X3.0cm size of test tube it was found thenucleation was widely separatedwith larger in size of cobalt oxalatecrystals. On the basis of morphology and size of crystal, the best result was obtained for the 20X2.5 cm size of test tubes as shown in fig 6.

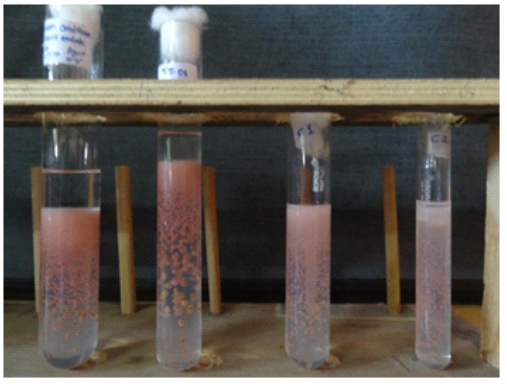

Fig 6: Experimental arrangement for the growth of cobalt oxalate crystals using test tubes

4.5. Effect of gel aging period.

When the gel aging period was short, the gel was not set in the test tubes and the growth was not found in test tubes. However it was also observed that if period was more than 3 days the nucleation was observed near the interface. The best result was observed for the aging of gel was 2 - 3 days for single diffusion and double diffusion respectively for the growth of good quality crystals.

5.0. Growth of cobalt oxalate crystal using neutral gel.

The neutral gel plays the important role to control the nucleation and increases the size of grown crystal[11].The experiment for the growth of the cobalt oxalate crystals using neutral gel was performed in two ways;

1. One layer of neutral gel.
2. Four layer of neutral gel.

For the first method, test tubes of size 25 cm X 2.0 cm were used. In these test tubes the cobalt chloride solution of concentration (0.75 M) was mixed with 0.5% agar-agar gel. After setting the gel it was kept for aging for another 2 days. On the set gel again 0.5% hot agar agar gel solution was gently poured and kept for setting and aging. After two days aging period anoxalic acid of concentration (1M) was poured and kept undisturbed. Similarly other test tube was filled with neutral gel of concentration 1.0% and 1.5 % respectively. The formation of layers is shown in fig 7.

Fig 7: Different neutral gel layers for the growth of cobalt oxalate crystals

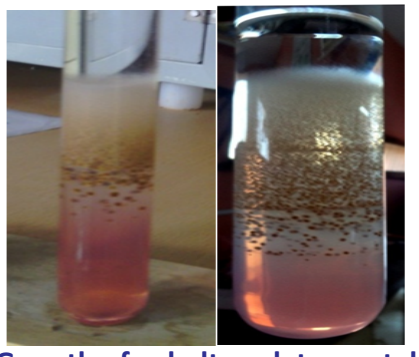

Fig 8: Growth of cobalt oxalate crystal in neutral gel

It was found that the large size cobalt oxalate crystals were grown in the 0.5% of neutral gel region in both of these methods.

The density of nucleation remained same at the interface.Thenumbers of nucleation weredecreased due to decreasing the concentration of neutral gel. It was also found that the size of grown crystals was increased due to decreasing the gel concentration.The surprising result was obtained in these experiments that the nucleationgrowth was not seen below the neutral gel region as shown in fig 7 and fig 8.

Table 1 shows the optimum conditions for the growth of cobalt oxalate crystals.

Table 1: Optimum conditions for the growth of cobalt oxalate crystal.

Condition	Single Diffusion	Double Diffusion
Percentage of gel	0.5%	0.5%
Molarity of cobalt chloride	0.75M	0.75M
Molarity of Oxalic Acid	1M	1M
Volume of cobalt chloride	2 ml	5 ml
Volume of Oxalic Acid	20 ml	20 ml
Gel setting period	24 Hrs	24 Hrs
Gel aging period	2 days	3 days
Period of growth	45 days	60 days
Temperature	Room Temp	Room Temp
Quality	Large size Spheruletic pink colour	Small size Spheruleticbrownish colour
Size	5 to 6 cubic mm	3 to 4 cubic mm

6.0 CONCLUSION

The cobalt oxalate crystals can be grown in agar agar gel by single diffusion method and double diffusion method. Various growth parameters affected the growth of cobalt oxalate crystal. Neutral gel controlled the nucleation but could not increase the size of crystals. Morphology was found to be same in single diffusion, double diffusion and neutral gel method. The crystals are in color of pink, red brown having in spherulitic shapes.

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