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Growth of Star Shaped Copper Iodate Crystals in Gel and Effect of Various Parameters on Crystal Growth.

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ABSTRACT

The present manuscript reports several aspects regarding the growth procedure of copper iodate $Cu(IO_3)_2$ H_2O crystal, optimum growth conditions and the kinetics i.e. influence of different growth parameters to obtain optimization conditions for the growth of these crystals. It is also predicts the results obtained from the different techniques used for the characterization of gel grown crystals of copper iodate.gel technique ,Aging effect density,Nucleation,growth parameters at ambient temperature are discussed.

Keywords: Copper iodate, Crystal growth, Single diffusion method,Gel technique,starshaped crystal.

I. INTRODUCTION

Growth of copper iodate crystals by gel method is a promising technique for growing crystals of many substances. Now a day, most of the solid state investigations are made by using well developed crystals. An effective efficient process is one, which produces adequately perfect crystals for their use at minimum cost. The aim of the present work is to put the gel method with standard performance and potentiality so that more perfect larger crystals should obtain at ambient temperature.

Copper iodate crystals cannot be crystallized by high temperature methods, as the material starts decomposing before melting. Therefore conventional high temperature methods for its growth are not applicable. Gel method is only the alternative technique to grow the crystal of appreciable size and good quality as reported in the present work at ambient temperature, moreover, this method is simple and inexpensive. Hence the crystals of copper iodate were grown by gel method.

The present manuscript reports several aspects regarding the growth procedure of copper iodate $Cu(IO_3)_2$ H_2O crystal, optimum growth conditions and the kinetics i.e. influence of different growth parameters to obtain optimization conditions for the growth of these crystals. This article also predicts the results obtained from the different techniques used for the characterization of gel grown crystals of copper iodate.

II. EXPERIMENTAL PROCEDURE

Preparation of gel:

Initially, different concentration of solutions of sodium Meta silicate taken for e.g. 10gm, 21gm, and 21.5gm 22gm in distilled water to get 250cc solution. The solution is constantly stirred and then filtered by Dr Watts's filter paper. It is then kept in to an airtight bottle free from dust and contamination. Density of

the solution was measured using Specific gravity bottle. A solution of different molarities prepared by adding proper amount of chemicals to the double distilled water. The chemicals used are copper nitrate, copper chloride, potassium iodate, acetic acid and sodium meta silicate.

When the solution of sodium Meta silicate is mixed with any of mineral or organic acid, gel formation takes place due to the polymerization in the resultant solution. In the present work, various concentrations of acetic acid, copper chloride, potassium iodate used with sodium Meta silicate tried for optimum condition to obtain good quality crystals of copper iodate.

Single diffusion method:

This method used to obtain good quality crystal of copper iodate in gel medium. In actual procedure, 5cc of 2N acetic acid was taken in a small beaker, to which sodium meta silicate solution of density 1.04 gm/cc was added drop by drop with constant stirring by using magnetic stirrer, till pH of the solution reaches a value 4.4 .A digital pocket sized pH meter of HANNA instrument is used for this purpose. A 5cc of copper chloride or copper nitrate solution was added with constant stirring in mixture of acetic acid and sodium Meta silicate solution. Continuous stirring process avoids excessive ion concentration which otherwise causes premature local gelling and makes the final medium inhomogeneous and turbid .The pH of the mixture was maintained at 4.4.

This mixture was then transferred to the test tube, a mouth of test tube closed using cotton plug used to avoid contamination of the exposed surface with atmospheric impurities and to keep the gel at atmospheric conditions. The setting time was 10-13days. The completely set gel was left for aging for 96 hours to 120 hours. Potassium iodate was used as supernatant having different molarities like 0.1M, 0.4M, 0.5M. 1M. were added over the copper chloride set gel. As the concentrations of supernatant increases, the numbers of nucleation centers were also found to be increased. Alternation method of supernatant and reactant also used to obtain good quality crystal of copper iodate.

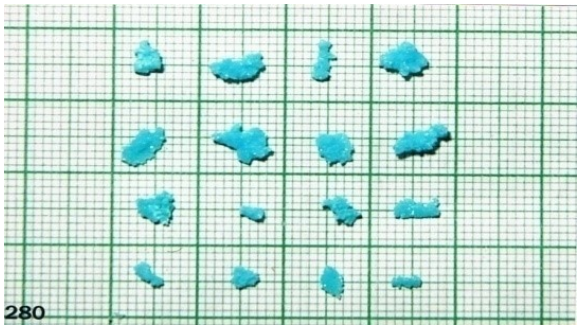

Figure 1. Shinning Star shaped crystals of copper iodate grown using copper chloride gel4FVNM

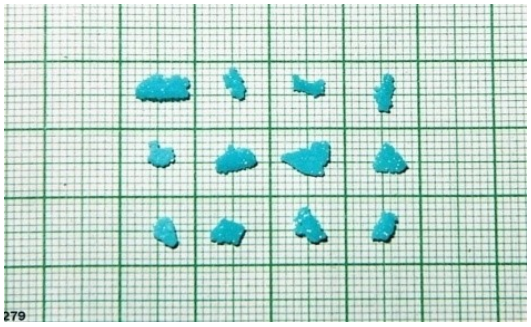

Figure 2. Shinning crystals of copper iodate grown using copper nitrate gel

III. RESULTS AND DISCUSSION

The optimum growth conditions for the growth of copper iodate crystals are represented in Table 1. Copper nitrate and copper chloride solutions are used to compared thickness transparency and quality of the crystal. The thickness of crystals grown by using copper nitrate as reactant is quiet effective in comparison when copper chloride is used as reactant. At a same time, crystals grown with reactant copper chloride are more transparent when same grown with copper nitrate. Crystals of copper iodate grown with help of copper chloride are shown in figure 2.1 and 2.4. A shape of copper iodate crystals in gel containing copper nitrate turns from spherical to star shaped are crowded shown in figure 2.2 and 2.3. The size of

copper iodate crystal in nitrate gel is small but are also transparent as shown in figure 2.

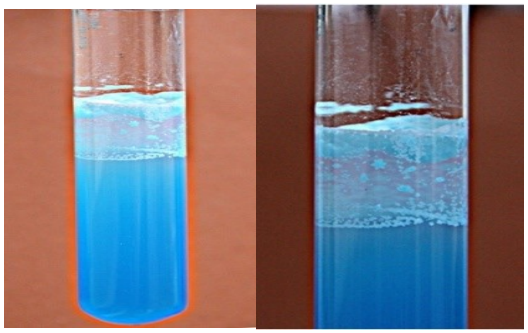

Figure 2.1 Figure 2.2

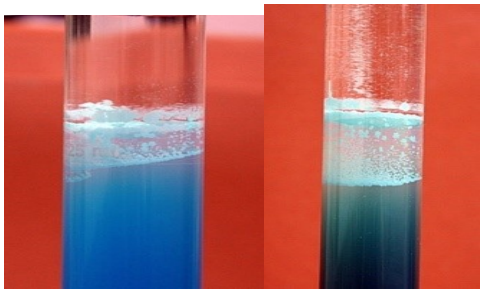

Figure 2.3 Figure 2.4

Table 1. Optimum condition for growth of $Cu(IO_3)_2$ crystals.

Condition Lattice parameter	Copper iodate concentrations
Density of sodium Meta silicate	1.04kg/m^3
pH of mixture	4.4
Amount of 2N acetic acid	5ml
Temperature	Room temperature
Gel setting time	13 days
Gel aging time	5 days
Concentration of KIO_3	0.4M
Concentration of $CuCl_2$	1M
Concentration of $Cu(NO_3)_2$	1M
Period of growth	4 weeks

IV. OBSERVATIONS

Figures shows different forms of grown copper iodate crystals inside the test tubes for the different concentrations of $CuCl_2$ solution in the gel. The range of the $CuCl_2$ Solution used was from 0.1 M to 0.5 M .The whisker growth with greater length originating from the interface of the gel was observed in the test tube containing $CuCl_2$ solution of 0.1M. However the dendritic crystal growth was not observed in the test tube containing $CuCl_2$ solution of 0.1 M concentration. As the concentration of $CuCl_2$ solution was increased up to 0.4M, it leads to dendritic growth along with the whisker growth .However there was no growth of shaped crystals. It was observed that in a test tube containing $CuCl_2$ solution of 0.5 M concentration, growth of copper iodate occurs in three phases which are whisker, cubical and star shaped. In present work, potassium iodate used as supernatant, in the test tube of Figure 3.1. Star Shaped one beautiful (2mm) shinning crystal is observed. In the test tube of Figure 3.5. Circular Shaped Crowded Crystals are seen. Large numbers of circular shaped small tiny crystals are seen at the wall of test tube. But at interface large, very crowded crystals are seen. The layer of crystals is very thick. The region of interface and region of crystal has turned transparent instead of blue i.e. the region in which copper nitrate has been completely utilized for crystallization. Figure 3.6. Figure 1.1 and 1.2 shows optimized star shaped transparent crystals of copper iodate on a graph paper with their scaling.

Effect of various parameters on crystal growth of copper iodate :

A. Effect of gel density:

The gels of different densities were obtained by mixing sodium Meta silicate solution of specific gravity 1.03 to 1.06 with 2N acetic acid. It is observed that the nucleation density decreases as the gel density increases. Table 3.2– shows the effect of gel densities on the quality of copper iodate crystals. Fig 3.9 shows the variation of time of gelation with gel density.

Table 3.5 indicates effect of gel density on number of nucleation centers.

pH=4.4 , Feed solution 0.4 M KIO_3

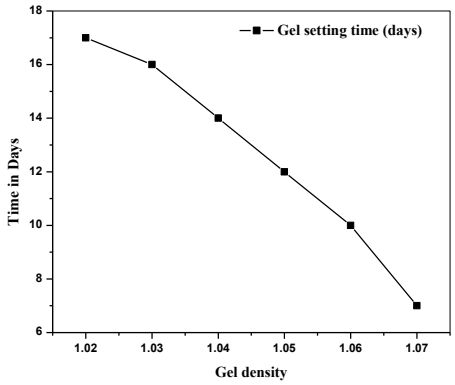

Figure 3.1. Plot of Effect of gel density on setting time

pH =4.4 feed solution 1M $CuCl_2$ and 0.4 M KIO_3:

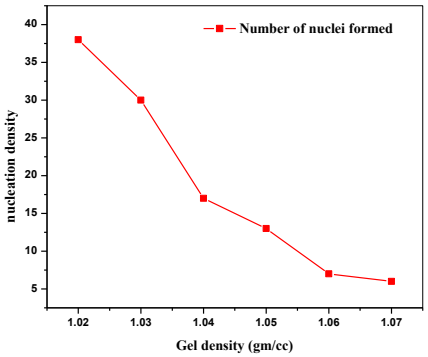

Figure 3.2. Plot of Effect of gel density on nucleation density

B. Effect of pH on gel:

The pH value of gel was varied from 2.5 to 7.0. The effect of the different pH values on gel setting time and the quality of grown crystal is as shown in Table 3.11. The optimum value of gel pH to get ideal gel is found to be 4.4 .At pH values less than 4.4 the time for gelation increased, and the resultant gel was unstable, for pH values greater than 4.4, the gelation occurred very soon and the resultant gel was not transparent. Figure 3.11 shows the graph of pH values against the setting time in hours. In the present work, pH value of 4.4 is the optimum condition for the good quality crystal of copper iodate.

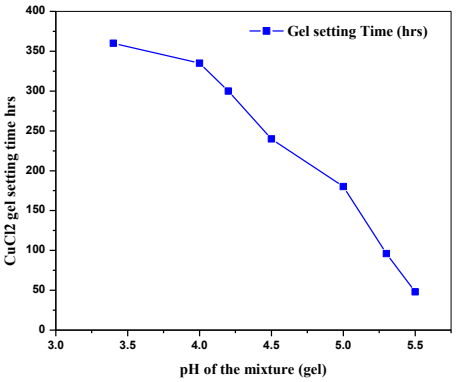

Figure 3.3. Plot of pH against gel setting Time

C. Effect of gel aging:

A feed solution of constant molarity was then added over a set gel. It is found that the number of copper iodate crystals decreases as the aging of gel increases. Aging of gel decreases the diffusion and nucleation density. More aging causes more amount of water evaporation out of the gel. Before the gel is set, the evaporation of water causes an increase in gel density which in turn decreases the diffusivity of reactive ions in the gel, thereby decreasing the number of nucleation sites. After the gel containing copper chloride is set, the evaporation of water causes not only the lack of ionic carriers in the channel of gel frame work, but also discontinuities in the channel due to the shrinkage of gel. Both these effects would adversely affect the diffusion of reactants ions hence the decrease in the number of nucleation sites. Table-3.5 shows the effect of aging time on number and the quality of crystal .Fig3.12 shows graph of aging in hours verses in number of crystal .In the present work aging of 124 hours was found most suitable at ambient temperature.

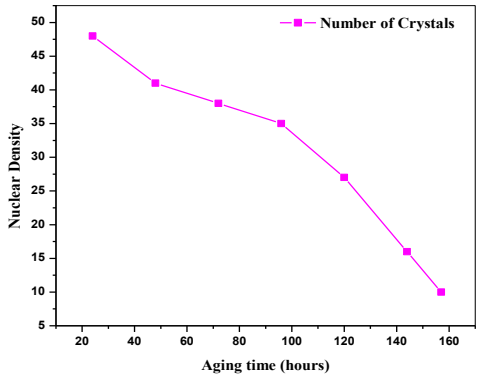

Figure 3.4. A gel aging time on number of nucleation

D. Effect of concentration of reactants:

Feed solution of either KIO_3, $CuCl_2$ and $Cu(No_3)_2$ were tried. Potassium iodate solutions of concentrations 0.1M to 0.4M molarity were prepared. As the concentration of the reactant in the gel increases, the nucleation density also increases. For the growth of good quality crystal of copper iodate, suitable concentration of reactant incorporated in gel is found to be 0.5M . It is to mention that the reactant [0.5M of $CuCl_2$ and $Cu(No_3)_2$,0.4M for KIO_3] were taken to grow good quality crystal of copper iodate using copper nitrate and copper chloride .. However, the use of KIO_3 and $CuCl_2$ yields the better and transparent quality of crystal, in terms of size and shape. All experiment were carried out by incorporating 5cc,1M $CuCl_2$ solution in gel and 15cc,0.4M of KIO_3 solution as supernatant was put over the set gel acidified with 2N acetic acid as a feed solution .Table 3.6 summarizes effect of concentration of reactant on quality,habit, and size of crystal.

E. Colour:

It is observed that an acidified gel containing copper nitrate leads to growth of blue colour crystal of copper iodate,while gel containing $CuCl_2$ leads to blue shinning and starshaped required crystal.

V. CONCLUSION:

1. Star shaped copper iodate crystal can be grown by simple gel technique.
2. The effect of pH, concentration of reactants, gel aging and setting, gel density and room temperature is important to grown crystals.
3. It is observed that the colour of copper nitrate is dark blue as compare to copper chloride, copper chloride crystals are shining and transparent but both are star shaped.
4. Single diffusion gel growth technique is suitable to grow copper iodate crystals.
5. Different habits of copper iodate crystals can be obtained by changing parameter like gel density,itspH,gel aging and gel concentration of reactants etc.

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