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Growth of heavily conducting metal copper iodide crystals in Gel and its structural analysis with kinetic Parameters

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Abstract: *Copper iodide crystals are grown using simple gel technique at ambient temperature. The various lattice parameters, gel aging time, gel setting time, Effect of pH observed, Kinetic Parameters of Gel growth are Discussed. Different structures habits, quality, size and concentration programing of crystals and its analysis explained. The chemical analysis, effect of reactants of grown crystals are implemented*

Keywords: *Gel method, pH, Aging and Setting Time Monoclinic, FTIR.*

1. INTRODUCTION:

Today crystals have become the base of modern technology in all the respects with this aspect in the mind and ever increasing the demand of copper crystals in the variety of field in science and technology, the work of the growth and the study of some important crystals have been under taken in the laboratory. The advances in the science of the solid state and material science depends upon the availability of good quality crystals [1]. Today with modern technology, with sophisticated instrument many national laboratories and university are growing facilities in laboratories the work of the growth of iodated varieties of crystals, which have the utility in day to day life, still the growth of some crystals by using simple equipment by utilizing fundamental properties of material much of the research work have been carried out and it not less important by considering all aspect and available facilities in laboratories the work of the growth of iodate, iodide and sulphide respectively.

2. Single diffusion method: A gel containing potassium iodide was prepared by using various concentrations of sodium Meta silicate and acetic acid. For this purpose, 5cc, of 2N acetic acid was taken in a beaker, to which sodium Meta silicate solution having different densities was added drop wise with the help of burette. During this procedure the solutions was continuously stirred by means of magnetic stirrer so as to avoid premature local gelling. Then 5cc potassium iodide solution of different molarities were added to this mixture with constant stirring to make the solution homogeneous. The pH of the mixture was maintained at 4.4. The number of attempts were carried out to optimized suitable pH value for growth of good quality crystals.

Table:1 : Optimum conditions for growth of copper iodide crystal:

Sr.No.	Condition Lattice parameter	Copper iodide concentrations
1	Density of sodium Meta silicate	1.04kg/m^3
2	pH of mixture	4.4
3	Amount of 2N acetic acid	5ml
4	Temperature	Room temperature
5	Gel setting time	15 days
6	Gel aging time	5-6 days
7	Concentration of $CuCl_2$	0.4M
8	Concentration of $Cu(NO_3)_2$	1M
9	Period of growth	4 weeks

3. Result and discussion:

Many trials are performed to obtain optimum condition for growing copper iodide crystals. A bigger size only one crystal was grown in gel medium shown in figure 4.8. But, in some cases numbers of crystals of different in sizes are grown inside the gel. It is due to the effect of pH and potassium iodide used as supernatant. Increase in aging of gel reduces number of nucleation centers. Insufficient gel aging often may leads to the fracturing of gel. Higher pH value of gel sets early but crystals obtained are less transparent due to the inclusion of silica gel. When pH less than 4.2, gel takes longer time to set and there is possibility of breaking of gel. Higher concentrations of reactants result on size and quality of crystals near the gel interface. Less concentrations of reactants leads to diminished the formations of size in crystals. Hence, Increase in aging of gel reduces number of nucleation centers and growth rate. Insufficient gel aging often leads to the fracturing of gel at the time of addition of supernatant [2-3].

3.1. Observations:

In present work potassium iodide used as a reactant as shown in figure 1 spherical shaped shinning number of white crystals are observed, in this case when potassium iodide was used as supernatant. The crowded crystals are obtained because of higher concentrations of supernatants. By reducing the concentration of supernatant, quality of the grown crystals can be improved as shown in Figures. In this case copper chloride used as supernatant. It is observed that alternation or exchange of same solutions shows surprising effect on growing in size and quality of copper iodide crystals as shown in Figure 2. The size of grown crystals of 0.5cm X0.8cm X1.2cm. Some time at higher pH equal to 4.5, bigger crystal of size 2cm were obtained. Copper iodide crystals of different size and shape shown in figure 1 and 2, which are multi shaped crystal.

Figure: 1 Growth of crystal in Gel of copper chloride

Figure :2 Growth of hexagonal top faced crystal

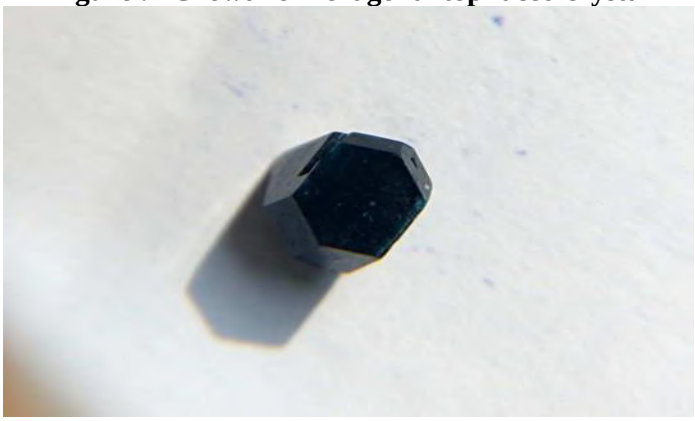


Figure : 3 Growth of hexagonal top side faced crystal

Figure :4 Growth of single big crystal

3.2. Effect of various parameters on crystal growth:

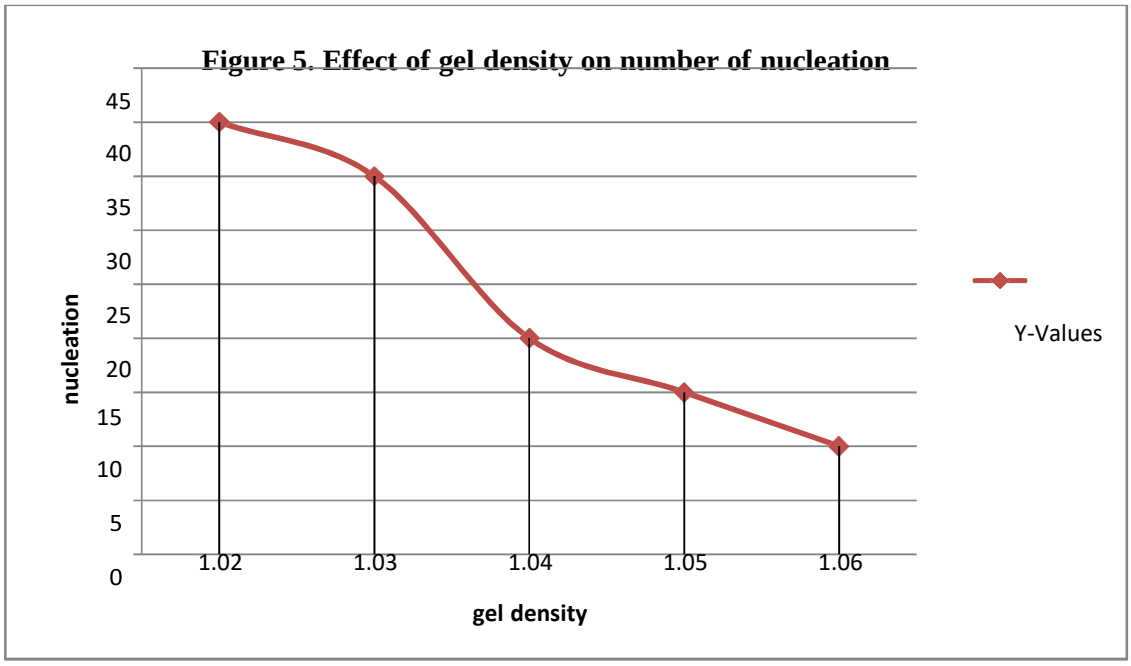
In gel growth technique, nucleation control can be achieved to some extent by changing a variety of gel parameters viz. concentration of feed solution, gel density, gel aging, concentration programming etc. Number of reviews showed that nucleation can be controlled using intermediate neutral gels. Nucleation control by concentration programming has been described by Henish. Influences of gel aging and gel densities on crystal growth rate have been studied by several workers. Effect of different gel parameters are discussed in the following sections according to the results obtained [4-6].

3.3. Effect of gel density:

The gels of different densities were obtained by mixing sodium Meta silicate solution of specific gravity 1.02 to 1.08 with 2N acetic acid, keeping pH value constant. It was observed that transparency of the gel changes with increase of gel density. Gels with higher densities required less gel setting time compared to the gels with lower densities. It may be noted that well defined and transparent crystals were obtained with sodium meta silicate of density 1.04 gm/cc. on the other hand, gels with densities below 1.04 gm/cc required longer time to set and still gels were not stable . Density of 1.02 gm/cc was the lower practical limit.[7-8] The effect of gel densities on quality of crystal is listed in a table 1. Figure 5 shows the variation of gelation time with gel density .it is observed that the gelation time decreases with increase in gel density. Table 2 shows the effect of density on number of nuclei formed. It has been graphically shown in fig.5. A greater gel density implies smaller pore size and poor reaction among the pores and thus decreasing the nucleation density. Researchers showed that diffusion coefficient becomes distinctly smaller as gel densities increased.

Table 2: Effect of gel density on number of nucleation. pH =4.4 feed solution 1M copper chloride and 0.4 M potassium iodide

Test tube No.	Acetic acid (cc)	Potassium iodide incorporated In gel 0.4 M, cc	Density of gel	Number of nuclei(crystal) formed	Observation
1	5	5	1.02	34	Small but large crystals
2	5	5	1.03	30	Number of crystals decreases
3	5	5	1.04	17	Tetragonal but large number of crystals
4	5	5	1.05	13	Tetragonal big sized crystal
5	5	5	1.06	7	Few dark multi shaped crystals
6	5	5	1.07	6	Low nucleation not well defined crystals



3.4. Effect of pH of gel:

The pH of the gel was varied by changing the composition of acetic acid and sodium Meta silicate. Table 4.4 shows the effect of different pH value on gel setting time and the quality of crystals obtained the optimum value of gel pH to get ideal gel is found to be 4.4.At pH value less than 4.4, the time for gelation increases and the resultant gel was unstable, but for pH value greater than 4.4, the gelation occurred very soon and the resultant gel wasnot transparent. Fig 4.14 shows the graph of pH against setting time in hours. In the present work pH value of 4.4 is the optimum condition to grow good quality crystals[9-10].

Table 4.: Effect of pH on gel (Aging period=5 days, feed solution 0.4M potassiumiodide)

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Test tube No.	Acetic acid (cc)	Copper chloride incorporated In gel 0.4 M, cc	Sodium meta silicate 1.04 gm/cc	pH of mixture	Gel setting time (hours)	Observation
1	5	5	15	2.5		Gel is not set
2	5	5	16	3	365	Gel is not set
3	5	5	16.5	3.5	335	Whisker crystals
4	5	5	17	4.0	300	Small growth of whisker crystals
5	5	5	18	4.2	240	Longer whisker crystals

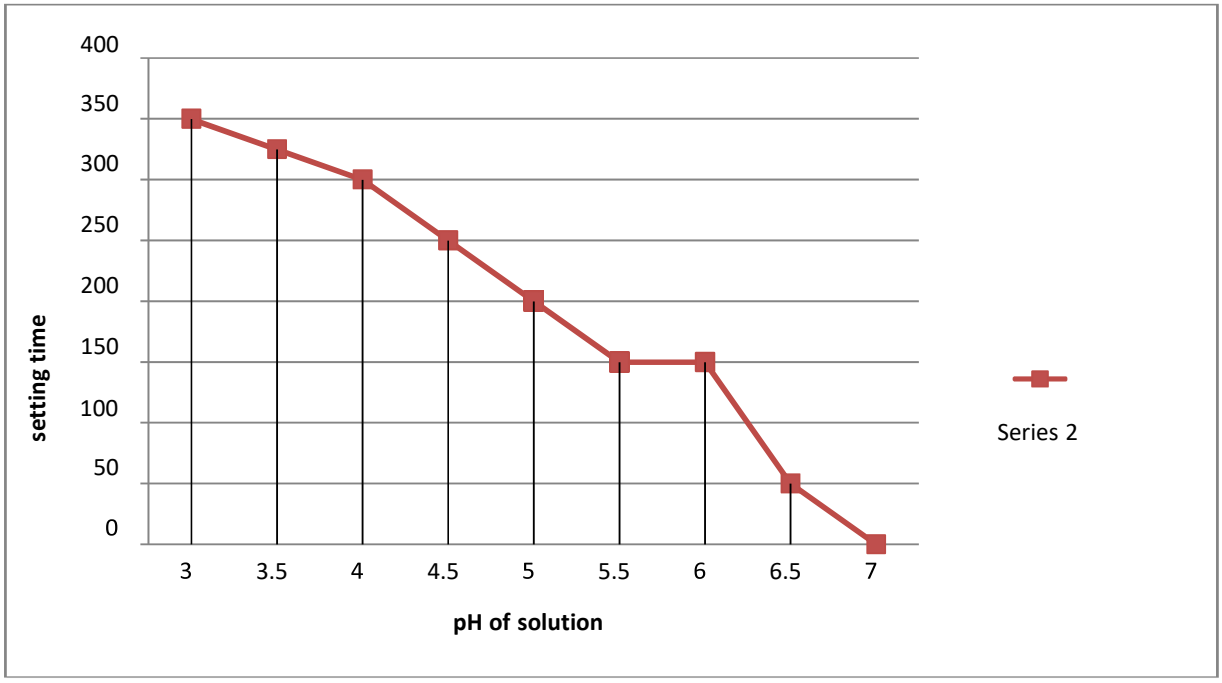

Figure 6. Plot of effect of pH on gel

3.5 : Effect of gel aging:

Gel aging plays an effective role on the growth of crystals as reported by Henish et al. to investigate the effect of aging on gels, gels of same pH and density were allowed to age for various periods before adding the feed solution. It was found that the nucleation densityof potassium iodide gel decreases as the aging increases [11].Aging of gel reduces number of nucleation centers and growth rate, The reason may be the formation additional cross linkages between siloxane chains with increasing gel age, resulting in a gradually diminishing cell size, this in turn reduces nucleation centers, since many nuclei find themselves in cells of very small size, where further growth is not possible

Table 4.: Effect of gel aging time pH: 4.4, feed solution 0.4 M copper chloride

Test tube no.	Acetic acid (cc)	Copper chloride incorporated In gel 0.4 M, cc	Sodium meta silicate 1.04 gm/cc	Aging Time (hours)	Number of crystals	Observation
1	5	5	19.5	24	39	High nucleationwhisker and dendritic growth
2	5	5	19.5	48	37	Opaque dendriticgrowth ,high nucleation
3	5	5	19.5	72	34	Low nucleation Whisker growth
4	5	5	19.5	96	32	Few cubical tetragonal crystals

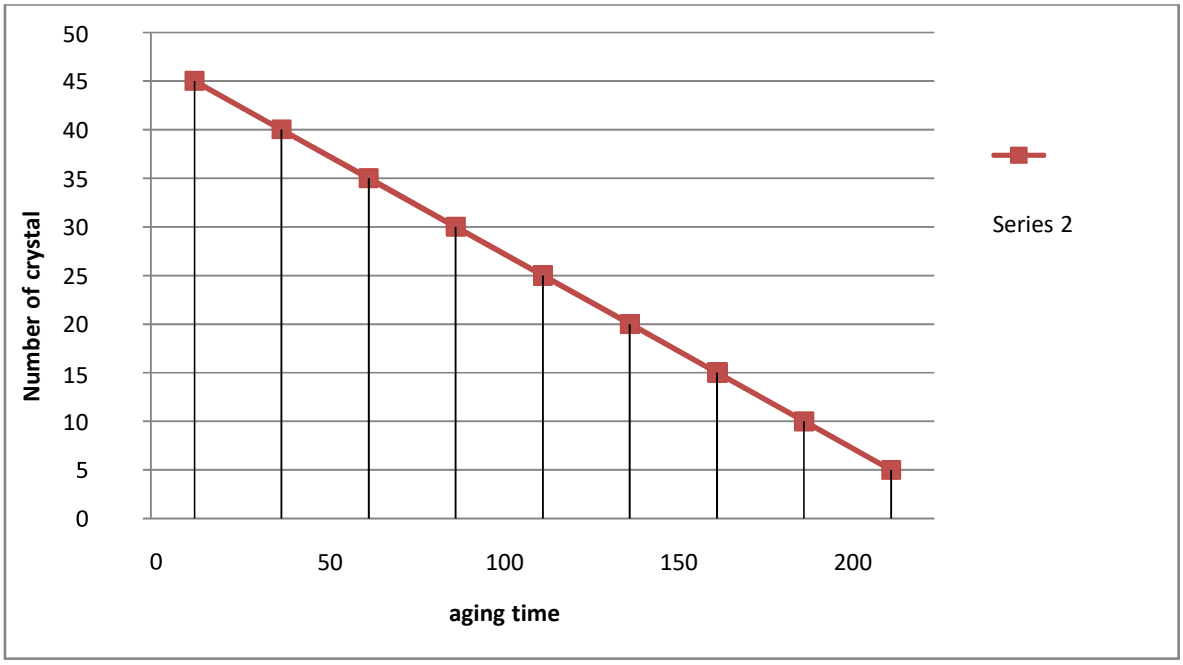

Figure 7.0 Effect of gel on aging time

4. Effect of concentration of reactants:

To investigate the effects of concentration of feed solutions, gel of same pH and density were prepared. feed solution of copper chloride was tried. Copper chloride solutions of 0.1 M to 0.5 M were prepared. Similarly, solutions of copper nitrate having different molarities 0.01 M to 0.1 M and solutions of potassium iodide having different molarities 0.1 M to 0.5M were prepared. By keeping the molarities of reactants incorporated in gel constant say copper chloride and copper nitrate, feed solutions of potassium iodide having different molarities were put over the set gels[12-13].

It was observed that, as the concentration of feed solution increases, the nucleationdensity also increases .this may be due to the enhance availability of cu ions .After repetition of number of experiments, suitable concentrations of reactants, potassium iodide incorporated in gel is found to be 0.4M , and for the feed solution, as copper chloride, it was found to be 1M.

Table.5: The effects of concentrations of reactant on habbit, quality and size of copper iodide crystals

Sr. No	Concentration of reactant above gel	Concentration of reactant in gel	Remark
1	Copper chloride 0.1M to 0.5 M,15ml	Potassium iodide 0.4M,5 ml	Large numberof small crystals are tiny
2	Copper chloride 1M, 15ml	Potassium iodide0.4M,5 ml	Tetragonal big sized crystals are dark in color
3	Copper chloride 1.5 M,15ml	Potassium iodide 0.4 M,5 ml	Tetragonal but small crystals are seen

4.1 Chemical analysis:

Copper is estimated gravimetrically using dilute HCl by homogeneous precipitation

Accurately weighed 0.1 gm of sample in powder form was dissolved in small quantity of double distilled water. Few drops of nitric acid were added while heating to dissolve the powder completely. Few ml of dilute sulphuric acid was slowly added with constant stirring until precipitation was obtained .it was allowed to stand for some time. Precipitate was filtered through filter paper and wash with warm distilled water .in a previously weighed porcelain crucible, the filter paper along with the precipitate was ignited. first it was slowly heated to dry the filter paper[14].

4.2 Estimation of iodine: -

Initially accurately weight 0.1 gm of sample in powder form was dissolved in small quantity of double distilled water few drops of nitric acid were added while heating to dissolved the powder completely. Few ml of dilute silver nitrate ($AgNO_3$) was then added to get precipitate. Precipitate was then filtered through Dr watts filter paper and washed several times with warm distilled water the residue obtained in this procedure was weighted after heating it in oven along with the filter paper. The result of chemical analysis that the experimental valves of copper (Cu) and Iodine (I) are in good agreement with the theoretical ones. The result of chemical analysis as shown in table 6.

Table 6: Result chemical analysis

Element	Theoretical	Practical value (%)
Copper	20.14%	20.11%
Iodine	79.86%	79.82%

5. CONCLUSION:

- The crystal of copper iodide can be grown by using gel technique Single diffusion gelgrowth technique is suitable copper iodide crystals.
- Different habits of copper iodide crystals can be obtained by changing their parameters.
- Chemical composition of the grown crystal by chemical analysis and EDAX match withthe theoretical calculation from molecular formula.
- Unit cell parameter value and d values match very well with the reported ones
- The structure of copper iodide is tetragonal confirmed by x-ray diffraction.

REFERENCES:

1. Joshi MS and Trivedi SG 1981 Growth of single crystals of barium iodate and strontium iodate in silica gels. Cryst. Res. Technol. 16(1) 19-26
2. Garud SL, Mahajan NK and Saraf KB 2009 Study of gel grown mixed crystals of BaxCa (1x)(IO3)4. Bull. Mater. Sci. 32(2) 187-92

3. Garud SL and Saraf KB 2008 Growth and study of mixed crystals of Ca Cd iodate. Bull. Mater. Sci. 31(4) 639-43
4. Morosin B, Bergman JG and Crane GR 1973 Crystal structure, linear and nonlinear optical properties of Ca (IO3)2.6H2O. Acta. Crysta. B29 1067-72
5. K.P.Joshi ,K.B.Saraf Acrylitic Starlite Crystals Grown using Gel Method and its Characterization, AJCER,VOL4(1)28-32pp Jan,2011
6. K.P.Joshi , K.B.Saraf , Growth of Multi-shaped Crystals of Copper iodide in Gel and its Characterization AJCR ,Vol4(2)84-87p.p,April,2011
7. K.P.Joshi,Semiconducting copper sulphide grown Crystal in Gel and its characterisation.IORD,Vol2,2348-0831p.p.Feb,2015
8. K.P.Joshi ,Optical Structural electronical and Band gap determination of newly grown semiconducting Copper sulphide crystal using Gel method,IJAREEIE,Vol4(2)2320-3765p.p.Feb,2015
9. K.P.Joshi, Growth of copper Sulphide and iodide Crystal in Gel and its Characterisation,JORD,Vol7(9)166-179p.p.Dec,2017
10. K.P.Joshi, B.S.Nandekar, Barun .K.Mehata Growth of star shaped crystal of copper iodate in Gel and Effect of various Parameters on Crystal Growth ,Volume 4, Issue 4, April 2018International Journal of Scientific Research in Science and Technology (IJSRST)2395-6011(Print)2395-602X (Online) 5
11. K.P.Joshi, Growth of copper Sulphide and iodide Crystal in Gas-Gel and its Characterization IJSRST international Journal of Scientific Research in science and Technology.(IJSRST), Volume 4, Issue 4, 449-454 April 2018,Online, 2395-602X,
12. K.P.Joshi, Synthesis of Semiconducting Copper Sulphide crystal grown in Gas- gel and its characterization, IJSRST international Journal of Scientific Research in science and Technology.(IJSRST), Volume 4, Issue 4, 40-4s April 2018,Online,2395-602X,5.327
13. De Kock and AJR 1980 Handbook on Semiconductors vol3, edKellar SP and Moss TS (Amsterdam: North Holland) p 247
14. Nyrit J 1982 Industrial Crystallization (Amsterdam: Elsevier) p 203 [7] Nassau K, Shiever JW and Prescot BE 1973 Transition metal iodates. I. Preparation and characterization of the 3d iodates. J. Solid State Chem. 7 186-204.