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Growth of Copper Iodate Crystal in Gel and its Characterisations

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ABSTRACT

Copper iodate crystals are grown using simple gel technique at ambient temperature. The various lattice parameters of grown crystals are obtained , gel aging time ,gel setting time ,Effect of pH observed, Different characterisation like X-ray Diffraction h,k,l values are determined, FTIR spectroscopic Analysis and Thermal behaviour of grown crystal like Thermogravimetric Analysis TGA are Discussed .Effect of Doping, Molar concentrations, PH of reactants and Alternate Diffusion methods in gel for size of crystal studied

Keywords : Gel method, Monoclinic, XRD, FTIR, TGA.

I. INTRODUCTION

Crystal growth is simple chemical process in which its reaction can be slowed down using Gel Technique. It is also used for phase change in chemical reaction of compound .it has been used by many researchers. Gel method is simply applicable at ambient temperature and pollution free.[1]. The effect of various parameters like gel aging, gel setting time, suppression nucleation centres, power of Hydrogen, solubility for crystal can be implemented in this technology.

In presented paper copper iodate crystals grown which are insoluble in water and they decompose before the melting point. The work on copper iodate crystal is reported only in silica hydro-gel. Hence the purpose of the present paper is to report the growth and influence of various parameters on the growth mechanism of star-shaped crystals of copper iodate in gel at ambient temperature.

Actually, copper is considered as the most promising interconnecting material in integrated circuits due to its low resistivity and high migration performance of cation and Anion and its deposition in silica gel [2-3]. Iodate used by researchers in electrochemical experiments at around pH 4 , A fundamental understanding of iodate-based Cu can only be attained by highlighting how the reduction characteristics of (IO_3)_on Cu depend on pH, (IO_3) concentration and rotation rate. The electrochemical literature does not appear to contain any systematic studies on IO3 reduction on Cu. More variation of pH can be implemented in Gel technique by incorporating and Doping of supernatant. But once the air Bubble get fixed, they grow larger and lenticular in shape during the period of gel setting

and it is impossible to knock them away from growth medium. since such bubbles have a degree of long-range order, they always tend to hinder the growth of larger crystals [4].

Copper (Cu) is environment friendly and easily available material that possesses a unique combination of low electrical resistivity and high conductivity (394 W m–1 K–1), excellent malleability, reasonably good corrosion resistance at ambient temperature [5-6]. Owing to these excellent combination of properties Cu and iodate are also engineering and Medically used materials for conduction of electricity and Thyroid problem in patients having iodine Deficiency. The crystals of copper iodate exhibit NLO properties and piezoelectric properties. The nonlinear and devices find large applications in optical communication, image processing and wave guide coupling crystals [7-8]. The purpose of present paper is to report the growth and influence of various parameters such as gel reactants, growth of mechanism of copper iodate crystals in gel with effect of ionic and molar concentration, its optimum conditions and properties.

II. METHODS AND MATERIAL

2. The growth of copper iodate crystal was carried out in silica gel. All chemicals such as acetic acid, copper chloride, potassium iodate and sodium metasilicate were of AR grade. In the present work, silica gel was preferentially used for the growth of crystals by single diffusion technique. 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 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, Number of experiments were carried out to secure appropriate range of pH values which in turn gives good gel allowing to grow good quality crystals of copper iodate.

It was observed that the mixture of solution with pH value less than 4.2, gelation takes quiet large time of the order of several days. However, in the pH range 4.2 to 4.5, there was appropriate waiting in gelation time. The gel setting time required for the gel solutions of pH greater than 4.5 was short. Borosil glass test tubes of diameter 2.5cm and height 25cm were used as crystallizing vessels. 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. Initially the mixture appeared in test tube was bluish, However with lapse of time its color changed towards dark blue when gel was completely set. The setting time was 10-13days. The completely set gel was left for aging for 4days. i.e. 96 hours to120 hours. It is also observed that the aging of gel reduces the diameter of the capillaries in gel so that speed of the reaction is automatically controlled. 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. For this, numbers of test tubes were set up for the observation. Alternation method of supernatant and reactant also used to obtain good quality crystal of copper iodate.

The chemical reaction inside the gel can be expressed as

$XCl_2 + 2YIO_3 \rightarrow X(IO_3)_2 + 2YCl$

Where X=Cu and Y=K

III. RESULTS AND DISCUSSION

2.1 Observations

Figures 1shows 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 CuCl2 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 2. Star Shaped one beautiful (2mm) shinning crystal is observed. In the test tube of Figure 3. Circular Shaped Crowded Crystals and few small star shaped 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.

The rate of diffusion seems to be uniform in the complete circular area of the test tube. As the circular ring at the bottom of the transparent region in which crystals are observed quite uniform at the same height i.e., rate of diffusion is constant and uniform as shown in test tube No 3. Again, the region in which crystals are formed is more transparent compared to nitrate solution. The color of copper iodate crystal grown with copper nitrate reactant is blue. While with copper chloride reactant is faint green as shown in Figure 2. Figure 3 and 4 shows optimized star shaped transparent crystals of copper iodate, figure 5 shows crystals of copper iodate on a graph paper with their scaling.

2.2 Effect of concentration of reactants:

The effect of concentration of feed solution can be investigated by preparing the gel of the same pH 4.4. Feed solution of either KIO3, CuCl2 and copper nitrate were tried. Potassium iodate solutions of concentrations 0.1M to 0.4M molarity were prepared. It was observed that as the concentration of the reactant in the gel increases, the nucleation density also increases. This may be due to the more effect of Cu ions in the gel. For the growth of good quality crystal of copper iodate, suitable concentration of reactant incorporated in gel is found to be 0.5M. Number of experiments were performed with interchanging the position of reactants.

It is to mention that the reactant 0.5M of CuCl2 and copper nitrate ,0.4M for KIO3 were taken to grow good quality crystal of copper iodate using copper nitrate and copper chloride. Change in the position of reactants does not affect either the quality of the crystal or the number of nucleation centres. However, the use of KIO3 and CuCl2 yields the better and transparent quality of crystal, in terms of size and shape. Therefore, after getting the optimized condition, all experiment were carried out by incorporating 5cc,1M CuCl2 solution in gel and 15cc,0.4M of KIO3 solution as supernatant was put over the set gel acidified with 2N acetic acid as a feed solution.

2.2a X-ray diffractogram of copper iodate

X-ray diffract gram of gel grown crystal of copper iodate was recorded using Miniflex model, Japan with Cukα radiation of wave length 1. 5408A°. and scanning speed of 10 0/minute. A copper target and nickel filter were used. From the powder diffract gram on data of copper iodate which shows eleven different peaks as shown in figure 5, and corresponding d values and (h k l) values were computed by using computer program POWD [An interactive powder diffraction data interpretation and indexing program] The recorded X-ray diffract gram is as shown in Fig. 5 The study was carried out at Department of Physical sciences, North Maharashtra University Jalgaon, Maharashtra.

These values are computed using computer programmed, POWD is as shown in the table 1. These parameters satisfy the condition for monoclinic system. These calculated values agree with the reported ones. Calculated unit cell lattice parameter of the copper iodate crystal is given in table 2

IV. RESULTS

3.1 Fourier transform infrared spectroscopy

In the present work the FT-IR spectra of copper iodate crystal was recorded using SHIMADZU spectrophotometer at the University Department of Chemical technology, North Maharashtra university Jalgaon (M.S) .The IR spectrum was recorded in the wave number range 500-4000 cm-1 for KBr line. The FT-IR spectrum of the copper iodate crystals is as shown in the figure 6.

The bands at 2910.68 cm-1 are due to O-H stretching. Bands due to vibration involving metal, iodine and oxygen atoms i.e. metal oxygen bonding are found predominantly near 711.55-719.47 cm-1. Fundamental infrared frequencies which observed in all iodate compounds are also found in present FT-IR analysis. The bands at 2312.73 cm-1 is also due to O-H stretching. The existence of acetic group obtained at 1411.94 cm-1 due to co-o stretching. A carbon oxygen band of C=O stretching yield at 1082.10 cm-1 to 914.29 cm-1, This is due to oxygen substituting effect. The fundamental frequencies have been observed are of symmetric stretching at 719.47 cm-1and asymmetric stretching at 700 cm-1. The dominant absorption bonds are found at 800cm-1in all iodate compound (Nakamoto 1970). In present work it match with bands at 719.47cm-1 to 771.55 cm-1. From the spectral analysis, it is clear that in case of copper iodate crystal the O-H stretch bands in the region 2312.73 cm-1 are due to inclusion of water molecules. The other extra bands observed in the spectra are may be due to inclusion of sodium Meta silicate in the grown crystal and water molecules present in copper iodate.

3.2. Thermo Gravimetric Analysis (TGA):-

It was confirmed that the thermal decomposition of copper iodate passes through Cu5 (IO6)2 which is unstable and immediately decomposes to Cu5O It has a one stage course until CuO is obtained an intermediate Orth periodate Cu5(Io6)2 is obtained in this process analogously alkaline earth iodates , however unlike Cu5(Io6)2, immediately after it is obtained begins to decompose to CuO.

Hydrous copper iodate becomes anhydrous according to reaction

$Cu(IO_3)2\ H_2O = Cu(IO_3)_2$.

Anhydrous copper iodate decomposes at high temperature according to reaction

$5Cu(IO_3)_2 = Cu_5(IO_6)_2 \rightarrow 10_2 \uparrow + 0.5I_2 \uparrow + 110_2 \uparrow + 4.5I_2 \uparrow + 5CuO$

The TGA curve for copper iodate gel grown crystal is as shown in the figure7. by continuous line. The TGA data collected from this curve and the theoretical values as calculated. TGA curve of copper iodate

showed clearly two stage of decomposition up to 600°c.TGA curve did not show an appreciable weight change in the temperature range 28°c to 98°c indicating that the crystals of copper iodate are thermally stable in this range .The first stage of decomposition occurs in the temperature range 98.5°c to 260°c in which weight loss of 6.018% agrees very well with the calculated weight loss of 6.36 %..This clearly indicates that the crystal of copper iodate is hydrated and weight loss calculation clearly indicates its decomposition. The probable loss of molecules in this stage are one O2 and 0.5 I2. Therefore, the grown crystal becomes anhydrous at 2100 c.

There is no further weight loss up to 340.0 showing thermal stability of copper iodate crystals in the temperature range of 2100c to 3400c. The second stage of decomposition occurs in the temperature range 341.350c to 5900c in which observed weight loss of 58.692 %, due to loss of 10 I2 and 4.5 I2 is observed. This is in agreement with the calculated weight loss of 58.0680 %, which is confirmed by the observed residual weight. The observed residual weight of 35.290% up to the end of analysis. This is nearly agreement with the calculated residue weight of 34.0960%, This confirms the presence of copper in the grown crystals.

A. Figures and Tables

Table 1. Lattice parameters

Lattice Parameters	Copper iodate $Cu(IO_3)_2$
System	Monoclinic
a	5.101 A^0
b	6.120 A^0
c	9.226 A^0
α	900
β	107.0 A^0
γ	900
V	249.83 (A0)3

Table 2 : Optimum condition for growth of copper iodate crystals

Condition Lattice parameter	Copper iodate concentrations
Density of sodium Meta silicate	1.04kg/m3
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 KIO3	0.4M
Concentration of CuCl2	1M
Concentration of Cu(NO3)2	1M
Period of growth	4 weeks

Figure 1 : Star shaped crystal of copper iodate

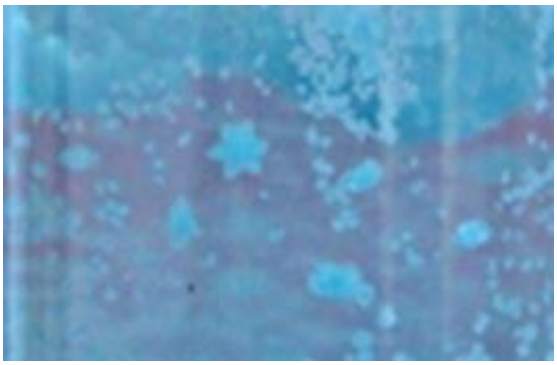


Figure 2 : Growth of copper iodate crystal in Gel

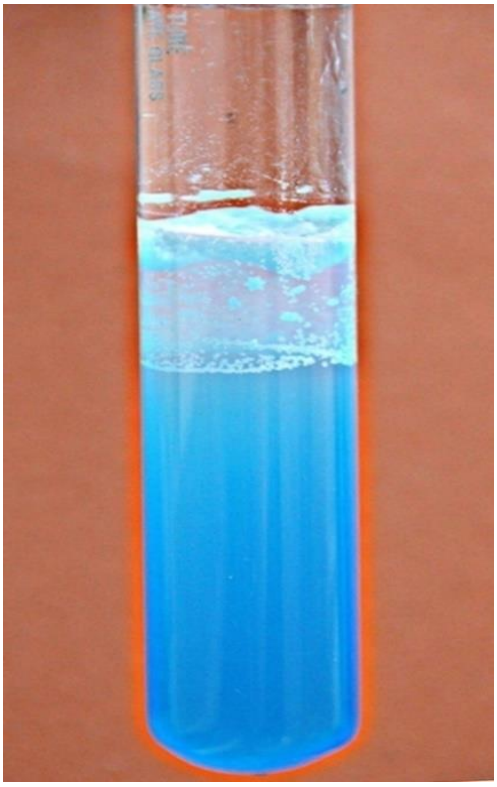


Figure 3 : Growth of spherulite copper iodate crystal in Gel

Figure 4 : Star shaped crystal of copper iodate in nitrate Gel

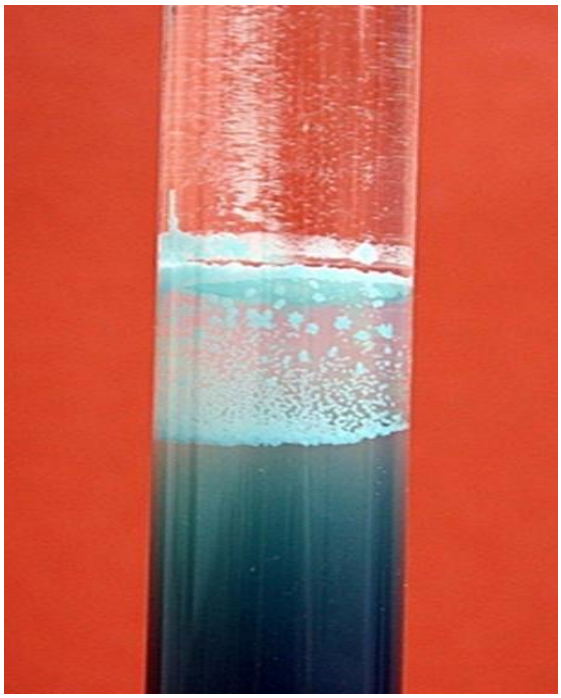


Figure 5. X-ray diffractogram of copper iodate

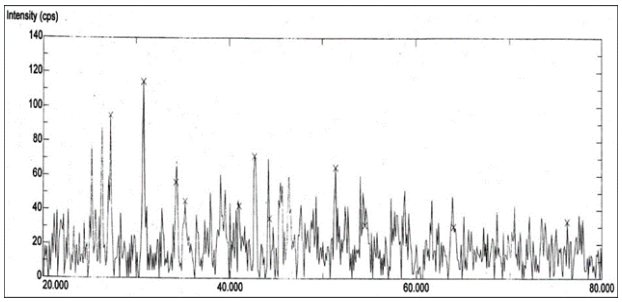


Figure 6. Infrared spectra (IR) of copper iodate

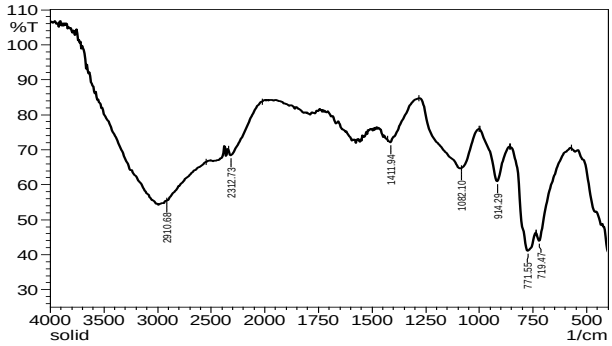
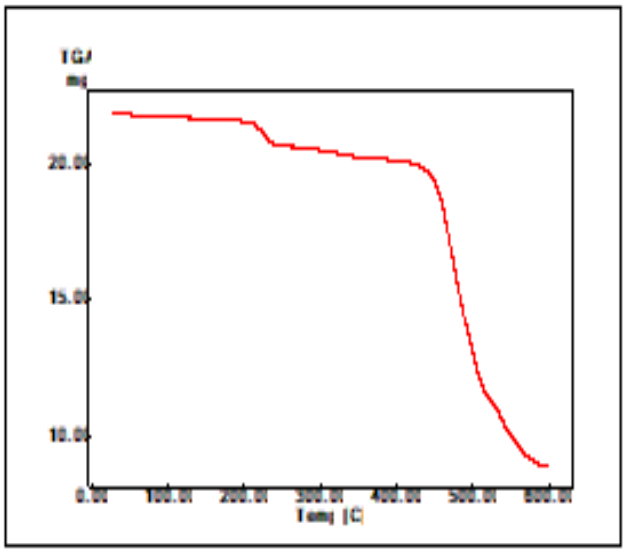


Figure 7 : TGA of copper iodate crystal:



V. CONCLUSION

It is observed that an acidified gel containing copper nitrate leads to growth of blue colour crystal of copper iodate, while gel containing CuCl2 leads to blue shinning and star shaped required crystal. Value of pH lies in between 4.2 to 4.4 to obtain copper iodate crystals at ambient temperature. Monoclinic structure using lattice parameters X ray Diffraction matched with powd data and decomposition of crystal at temperature gives nonexistence of water molecules in crystal it is verified using its Fourier transform infra-red radiations and Thermal behaviour including stretching of bonds.

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