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ISSN: 2278-1862

*Journal of Applicable Chemistry*

2021, 10 (2): 220-226

(International Peer Reviewed Journal)

# Growth of Copper Iodate Crystal Using Gel Technique and Its Characterization

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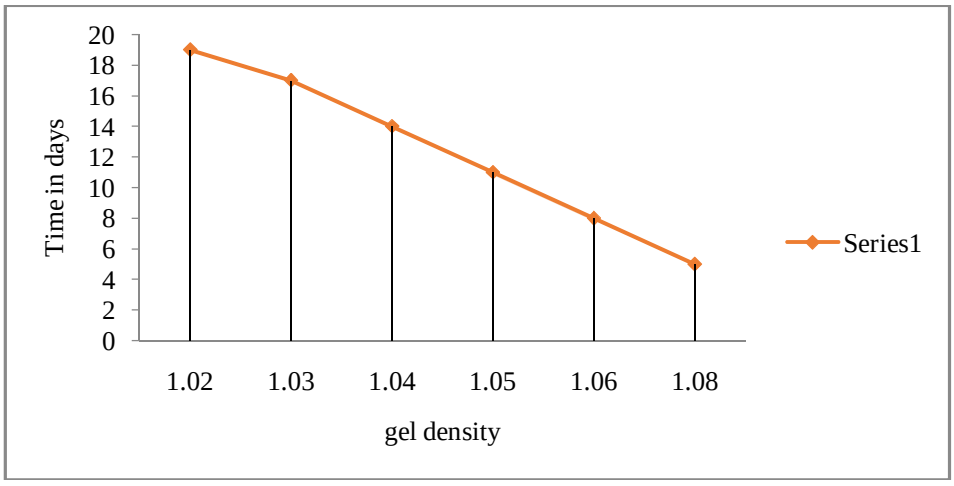
**Accepted on 12th February, 2021**

## ABSTRACT

*Crystals of copper iodate are grown using chlorides and nitrites by applying alternate diffusion technique in gel. Different parameters of gel are investigated, structural and chemical compositions are determined, Chemical contents are determined by chemical compositional Analysis.*

### Graphical Abstract




Plot of Effect of gel density on setting time.

**Keywords:** Gel technique, Iodate, Star shape, Chemical Composition.

## INTRODUCTION

In present work, a heavily conducting material copper is taken under consideration with iodate. The crystal of copper iodate are having large variety of application. A copper and iodine which are applicable in medicine, industry. The grown crystals of cooper iodated are star shaped shining and quite attractive and are of multi shaped shining crystal. The band gap of these crystal exhibits variety

of applications in semiconductor and in electronics. A characterization of growing crystal was performed using structural analysis [**1-3**].

## MATERIALS AND METHODS

A copper iodate crystal are grown using chemical reaction method. This method involves combination of two different solutions of soluble salts by diffusion through a gel with subsequent nucleation and the crystal growth occure, which continues due to the gradual precipitation of insoluble product [**4-5**]. Initially, different concentration of solutions of sodium Meta silicate taken for e.g. 10 gm, 21 gm, and 21.5 gm 22 gm 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 [**6**]. 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.

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 4.2. A digital pocket sized pH meter of HANNA instrument is used for this purpose [**7-8**]. 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 4.2, Number of experiments was carried out to secure appropriate range of pH values which in turn gives good gel allowing to grow good quality crystals.

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 2.5 cm and height 25 cm 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 4 days. i.e. 96 to120 h. 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

**Figure 1.** Growing stage of spherical to star shaped crystal of copper iodate in copper nitrate gel.

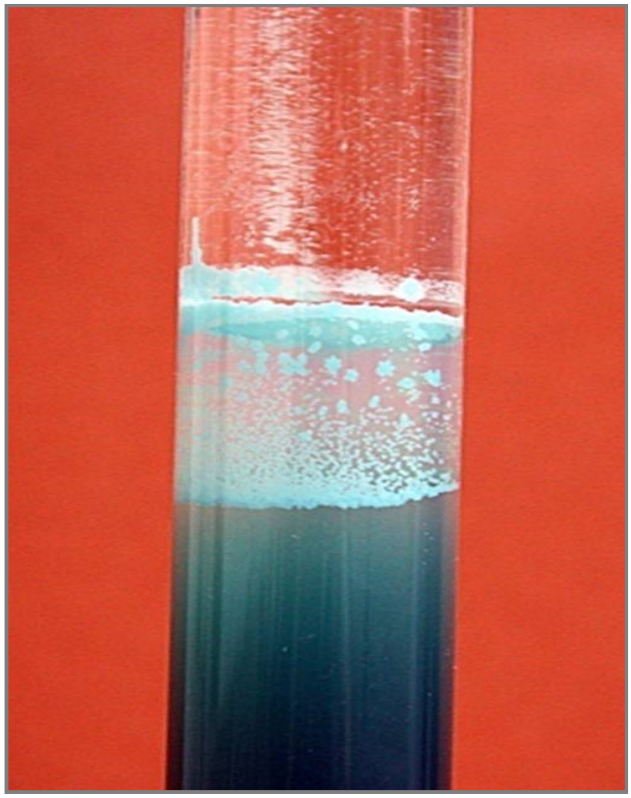


**Figure 2.**Transparent star shaped grown crystal of copper iodate in copper chloride gel

Formation of nuclei depends on number of parameters such as pH of solution maintained, concentrations of reactants, aging of gel and density of gel. It was observed that initial nucleation takes place on the surface of the gel and supernatant solution and rarely inside the gel. Number of nuclei is inversely proportional to the distance from the gel interface. Nucleation in gel takes place after 9 to 11 days. Generally this time varies few hours to few days depending upon the ambient temperature and circumstances [9].

## RESULTS AND DISCUSSION

The optimum growth conditions for the growth of copper iodate crystals are represented in table 1. Different parameters such as gel density, gel setting time, gel aging time, concentration of reactant, pH of gel, period of growth etc have the considerable effect on the growth rate. 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 1. A shape of copper iodate crystals in gel containing copper nitrate turns from spherical to star shaped are crowded shown in figure 2. The size of copper iodate crystal in nitrate gel is small but are also transparent as shown in figure 1.

**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 | 5 mL |
| 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 |

**Effect of gel density:** According to Henish, the proper range of specific gravity of growing good quality crystal is 1.03 to 1.06, by keeping pH constant. 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 as gel density decreases, transparency of the gel increases. As a rule very dense gels produce poor crystals [10]. On the other hand, gels of insufficient density take long time to form and are mechanically unstable. It is observed that the nucleation density decreases as the gel density increases. Table 1 shows the effect of gel densities on the quality of copper iodate crystals with its setting time. Figure 3 shows the variation of time of gelation with gel density. It also indicates effect of gel density on number of nucleation centers. In present work sodium met silicate solution specific gravity 1.04 gm/cc and acetic acid (2N) with 4:1 ratio is an ideal combination for gel formation in the present case of copper iodate. The effect of gel density on nucleation density of copper chloride as shown in figure 3.

**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 star shaped required crystal.

**Chemical Analysis:** Today in a modern industrialized society, most manufacturing industries depends on both qualitative and quantitative chemical analysis to insure that the raw material used most certain specialization, they also check the quality of the product. For this purpose analytical methods must be employed which are quick and which can be readily adopted for research and

application orientated work. The determination of element of the crystal or of the foreign substance obtained using chemical analysis [11-12]. Chemical analysis is further divided into two types Qualitative analysis and Quantitative analysis. Qualitative analysis includes semi micro test for deflection of cation and anions present in the substance by different sophisticated technique such as EDAX, Spectrophotometer, micro analyzer etc.

**Table 2.**Effect of gel density on setting time: pH=4.4 , Feed solution 0.4 M $KIO_3$

| Test tube no. | Acetic acid 2N (mL) | Copper chloride incorporated In gel 1M | Density of gel (gm $mL^{-1}$) | Gel setting time (days) | Observations |
|---|---|---|---|---|---|
| 1 | 5 | 5 | 1.02 | 17 | Gel is not stable |
| 2 | 5 | 5 | 1.03 | 16 | Long time required to set gel |
| 3 | 5 | 5 | 1.04 | 14 | Few spherical crowded crystals |
| 4 | 5 | 5 | 1.05 | 12 | Number of crystals decreases spherical and star shaped crystals exist |
| 5 | 5 | 5 | 1.06 | 10 | Less transparent |
| 6 | 5 | 5 | 1.07 | 07 | Not well defined crystals |

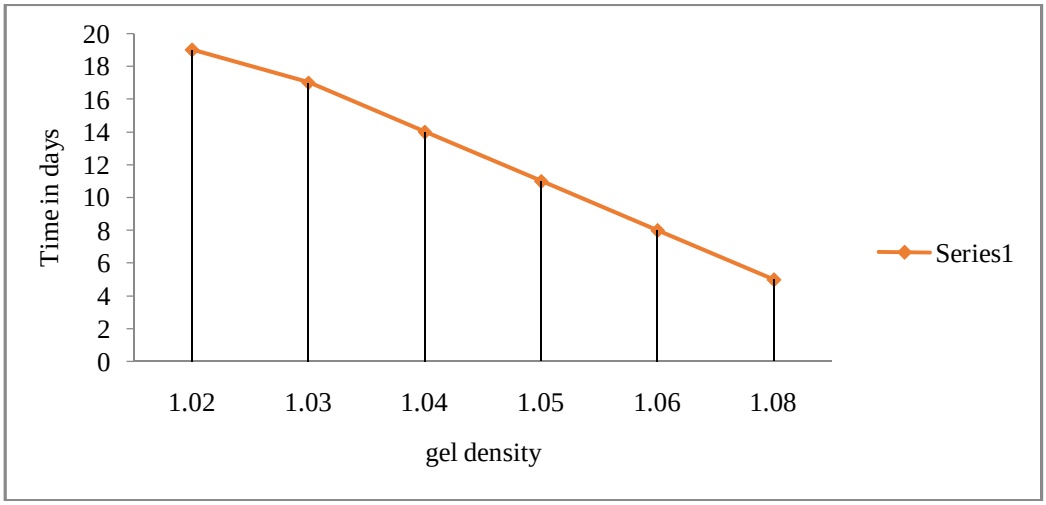



**Figure 3.** Plot of Effect of gel density on setting time.

Quantitative analysis deals with the determination of quantity of each component present. Such type of analysis involves different techniques such as trimetric or volumetric analysis and gravimetric analysis in gravimetric analysis, the substance under study is converted in to an insoluble precipitate, which is collected and weight. In volumetric analysis, the substances to be identified are allowed to react with an appropriate reagent added as a standard solution and the volume is determined. We get chemical analysis in the present work involves estimation and detection of the copper and iodate in the crystal of copper iodate.

**Estimation of copper (Cu):** Copper is estimated gravimetric using dilute sulphuric acid by homogeneous precipitation. 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 dissolve the powder completely. Few mL of dilute $H_2SO_4$ was slowly added with constant stirring until precipitation was obtained. It was allowed standing for same time. Precipitate was then filtered through Dr. Watts's filter paper and washed several times with warm distilled water. In a previously weight precipitate crucible, the filter paper along with the precipitate was ignited first, it was slowly heated to dry the filter paper [13-15].

The precaution should not catch the fire. When the filter paper was completely dried, the precipitate was strongly ignited to get white ash of the residue was cooled. Weight of residue obtained was 0.0351 gm. Weight of sample =0.1 gm, Weight of crucible +sample =13.1831 gm. Weight of empty crucible= 13.148 gm. Weight of the residue = 0.0351 gm.

**Theoretical percentage of copper (Cu):** Theoretically in Cu ($IO_3$) H2O having molecular weight 411.54 gm. The amount of Cu is 63.54 gm Percentage of Cu= 63.54×100/411.54= =15.44% Thus theoretically in 100 gm of Cu $(IO_3)_2$ $H_2O$, there is 15.44% of copper (Cu).

**Practical percentage of copper (Cu):** Practically molecular weight of copper crystal is 146.0786 gm, Amount of Cu is 63.54 gm. Therefore in 0.0351 gm of residue, Amount of Cu= 63.54×0.0351/ 146.0786=0.0152 gm, Thus amount of Cu practically 0.0152 gm, Since 0.1 gm of sample powder, there is 0.0152 gm of Cu. Percentage of Cu=0.0152×100/0.1,Cu=15.2%Thus practically, in 0.1 gm of Cu $(IO_3)_2H_2O$ sample, there is 15.2% of copper.

**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 (14) to get precipitate. Precipitate was then filtered through Dr watts filter paper and washed with distilled water the residue obtained in this procedure was weighted after heating it in oven along with the filter paper. Weight of sample=0.1 gm. Weight of filter paper precipitate=1.2382 gm. Weight of filter paper=1.05 gm, Weight of the residue=0.1182 gm.

**Theoretical percentage of Iodine (I):** Theoretical, in $Cu(IO_3)_2H_2O$ having molecular weight 411.54 gm. Amount of iodine is 252gmTherefore in 0.9116gm of residue AgI Percentage of iodine =252×100/411.54=63.23%Thus theoretical in 100 gm of $Cu(IO_3)_2$ $H_2O$ there is 63.23 % of Iodine.

**Practical percentage of Iodine (I):** Practically, in AgI having molecular weight of 235 gm amount of I is 127 gm. Therefore In 0.9116 gm of AgI residue, Amount of I = 127×0.1182/235= 0.06307 gm=15.0114/235=0.0638 gm, Thus amount of I in AgI is practically 0.0638 gm. Since 0.1 gm of sample powder, there is 0.0638 gm of Iodine Percentage of I = 0.0638×100/0.1I=63.8%Thus practically, in 0.1 gm of Cu $(IO_3)_2$ $H_2O$ sample. There is 63.8% of iodine. The result of chemical analysis that the experimental valves of copper (Cu) and Iodine (I) are in good agreement with the theoretical ones.

**Table 3.** Result chemical analysis

| Element | Theoretical value (%) | Practical value (%) |
|---|---|---|
| Copper | 15.44% | 15.20% |
| Iodine | 63.23% | 63.8% |

## APPLICATION

Non linear optical properties of various iodate crystals is very important .In the recent year this prominent NLO behavior is studied by growing iodate of alkali transition and alkaline earth material. it must be alkaline earth material. Iodide also used to multi cellular process, it supplies oxygen to plant vegetable and fruit trees. It would be interesting to study particular iodide in crystalline form. Copper used to make electrical products and electronics in electric generators and motors, electrical power and lighting fixture. Electrical wiring radio and television sets, computers air conditioning system and other electrical appliances in building construction, equipment and heating, chemical and pharmaceutical manufacturing hence number of characterizations were performed using different techniques.

## CONCLUSION

From the above studies it can be concluded that

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, its pH, gel aging and gel concentration of reactants etc.
6. Chemical compositions of the grown crystal by chemical analysis match with the calculation from molecular formula.

## REFERENCES

[1]. Kumar, Senthikumar, Dhanasekaran, Growth of YCOB single crystals by flux Technique and their characterization, *Cryst. Res. Technol.*, **2008**, 43, 467.
[2]. Zatovsky, Strutynska, Shishkin, Synthesis and characterization of phosphates in the pseudo-ternary melted systems CsO-PO-$M^{II}O$ ($M^{II}$ – alkaline earth), *Cryst. Res. Technol*, **2008**, 43, 362.
[3]. Rathi, Dinakaran and Jerome Das, Characterization of L-aspartate crystals grown in gel medium, *Cryst. Res. Technol.*, **2008**, 43, 729.
[4]. Ghosh, Goswami, Optical and structural properties of lead iodide thin films prepared by vacuum evaporation method, *Crystal. Res. Technol.*, **2008**, 43, 959.
[5]. Fumin Wang, Synthesis, characterization and crystal structure of a novel 3-D copper(II) polymer with bent N-containing ligand, *Cryst. Res. Technol.*, **2008**, 1087.
[6]. ChenWeijun Lin jingzhen, Thermal Behaviour of KAP, RAP and TAP of X- ray Analyzing Crystals, *Journal of synthetic crystal*, **1992**, 04, 12.
[7]. Oueslati, Ben Nasr, Crystal structure and spectroscopic studies of a new organic mono hydrogen mono phosphate dehydrate, *Cryst. Res. Technol.*, **2008**, 43, 108.
[8]. Bauch, Henschel, Schulze, automatic detection and high resolution fine structure analysis of conic X-ray diffraction lines, *Cryst. Res. Technol.*, **2010,** 45, 583.
[9]. Florani, Comparison between fine particles and spin-glass properties in Studies of Magnetic Properties of Fine Particles and Their Relevance to Materials Science, J. L. Dormann, D. Fiorani, Eds., *Elsevier: Lausanne*, **1992**, 135.
[10]. Marinova, Georgiev, Stoilova, Infrared spectroscopic study of $NH_4^+$ and $SO_4^{2-}$ ions included in nickel sulfates and selenates, *Cryst. Res. Technol.*, **2010**, 45, 111.
[11]. Brass, Istomin, L. Nazarova, X-ray diffraction refinement of the crystal structure of anosovite prepared from leucoxene, *Cryst. Res. Technol.*, **2009**, 44(1), 117-122.
[12]. Jianqiang Liu, Mei Zhang, QizhenShi, Synthesis, characterization and crystal structure of a novel 2D Cu (I) complex with flexible ligand, *Cryst. Res. Technol.*, **2009**, 44(1), 123-126.
[13]. Sumesh, Pathak, Srivastav, Analysis of current-voltage-temperature characteristics of In and Cu contacts on n-type MoSe2 single crystals, *Cryst. Res. Technol.*, **2011**, 46, 61-64.
[14]. Cheng-Hsing Hsu, Shu-Fong Yang, Micro structural, optical and electrical properties of $GeO_2$ thin films prepared by sol-gel method, *Cryst. Res. Technol*.**2011**, Vol-46, No. 1, 64-68.
[15]. Karabulut, Yilmaz, Boz, Electrical and optical properties of Co doped TlGaS2 crystals, *Cryst. Res. Technol.*, **2011**, 46 (1), 79-84