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Optimizing growth conditions for copper tartrate crystals in GEL medium

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Abstract

In this study, copper tartrate crystals were synthesized using the gel growth method. Since the compound decomposes before reaching its melting point, conventional high-temperature growth techniques are unsuitable. Additionally, due to its limited solubility in water, the gel method serves as an effective alternative for obtaining high-quality crystals at ambient conditions. This approach is both cost-effective and straightforward. The crystals of copper tartrate were grown in a sodium metasilicate gel medium using the single diffusion technique at room temperature. Various factors influencing crystal formation, including gel pH, gel concentration, setting time, and reactant concentrations, were systematically examined. The impact of these parameters on crystal growth was analysed to optimize conditions for producing well-formed copper tartrate crystals Cu(C4H4O6).

Keywords: Gel method, single diffusion technique, copper tartrate crystal growth, gel density optimization, pH influence on gel, aging effects in gel, reactant concentration impact

1. Introduction

Copper tartrate crystals can exist in two distinct forms:

- Anhydrous copper tartrate ($CuC_4H_4O_6$)
- Copper tartrate dihydrate ($CuC_4H_4O_6 \cdot 2H_2O$)

These crystals are typically synthesized in laboratory conditions for research purposes. Their structural, spectroscopic, magnetic, and thermal properties have been extensively studied.

The molecular and crystal structures of copper tartrate have been examined in various studies. Notable contributions include research by Prout *et al.*, who analysed the crystal structure of copper (II) meso-tartrate and d-tartrate trihydrates. Jian, Zhao, and Wang explored novel two-dimensional coordination polymers of tartrate copper (II), while Jethva, Dabhi, and Joshi conducted in-depth studies on gel-grown copper levo- and dextro-tartrate crystals. Additionally, Ariponnammal and Velvizhi investigated the structural, spectroscopic, and magnetic characteristics of these compounds.

The crystal structure of the dihydrate form, which shares isostructural characteristics with its cobalt counterpart, has been analyzed using X-ray single-crystal diffraction. It crystallizes in the P space group, with lattice parameters a = 490.60 pm, b = 667.31 pm, c = 1088.85 pm, and angles $\alpha = 98.855^\circ$, $\beta = 91.119^\circ$, and $\gamma = 92.841^\circ$. The three-dimensional network is formed by trans-configured $CuC_4H_4O_6 \cdot H_2O$ octahedra interconnected via shared tartrate ligands and hydrogen bonding along different crystallographic planes.

In contrast, the anhydrous form, $CuC_4H_4O_6$, crystallizes in the P2 space group (Z = 2) with unit cell dimensions of a = 548.9 pm, b = 512.4 pm, c = 941.8 pm, and $\beta = 90.5^\circ$. The structure exhibits a slight monoclinic distortion. Infrared (IR) and Raman spectroscopic analyses have been performed, and group theoretical studies discuss factors such as hydrogen bond strength, lattice distortion, and vibrational modes based on the mutual exclusion principle.

2. Methods and Material

The growth of copper tartrate crystals in a gel medium was achieved using the single diffusion method. The experimental setup consisted of borosilicate glass test tubes (25 cm in height and 2.5 cm in diameter), along with a magnetic stirrer, pH meter, burettes, pipettes, beakers, and a specific gravity bottle for precise measurements. The gel was prepared using acetic acid and

sodium metasilicate at varying concentrations. For this, 5 mL of 2N acetic acid was taken in a beaker, and sodium metasilicate solution of different densities was slowly added drop by drop using a burette. To prevent localized gelation, the solution was continuously stirred with a magnetic stirrer. Once the solution became uniform, 5 mL of copper chloride solution (at different molarities) was introduced with constant stirring to maintain homogeneity. The pH was set at 4.4, and further experiments were conducted to determine the optimal pH conditions for producing high-quality crystals. The prepared solution was carefully transferred into test tubes and covered with a cotton plug to prevent contamination from atmospheric impurities while allowing controlled gelation. The gel took approximately 12 days to set completely, after which it was aged for 3 days. Aging is a critical step, as it regulates nucleation by reducing the capillary diameters within the gel matrix, leading to better crystal growth. A tartaric acid was used as the supernatant, which was carefully poured over the set gel to facilitate the slow diffusion of reactants and controlled crystal formation. Additional experiments were performed by altering the positions of the reactants to observe their effects on crystal growth.
The chemical reaction inside the gel can be expressed as

$$CuCl_2 . 2H_2O + C_4H_6O_6 \rightarrow Cu(C_4H_4O_6) + 2HCl + 2H_2O$$

3. Results and Discussion

Observations indicated that nucleation commenced approximately 7 to 8 days after the introduction of the reactants. Crystal nuclei were primarily formed at the interface between the gel and the supernatant solution, with ery few observed within the gel framework itself. A notable trend was identified in which the density of nuclei was inversely related to the distance from the gel interface, meaning that fewer nuclei formed as the distance from the interface increased. The nucleation process was found to be influenced by several parameters, including pH, gel density, aging duration, and reactant concentrations. Under conditions of a stable concentration gradient, the crystal growth rate reached equilibrium, facilitating the development of well-formed copper tartrate crystals. During the experimental period, an unexpected drop in ambient temperature was recorded. This temperature reduction led to a decrease in the solubility of the supernatant solution, impacting the overall crystallization process. Additionally, in test tubes containing a high concentration of copper chloride, the by-product hydrogen chloride (HCl) formed at a controlled rate above the gel interface. The precipitated HCl exhibited a white, opaque appearance. Interestingly, the growth of HCl whiskers followed an upward trajectory within the supernatant solution rather than extending downward into the gel, a phenomenon not previously observed in similar experiments. This unique behaviour suggests that external environmental factors, including temperature variations and reactant concentrations, played a important role in dictating the directionality and morphology of crystal growth within the gel medium. Further investigation is required to understand the underlying mechanisms governing this unexpected crystallization pattern.

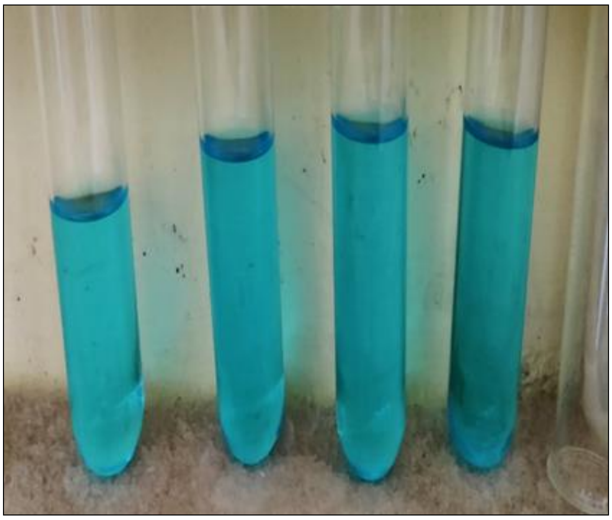

Fig 1: Blue solution in test tubes

This occurrence was primarily linked to a sudden drop in room temperature due to the cold season. The unusual behaviour observed for different concentrations of 0.4M, 0.5M, 1M, and 1.5M of $CuCl_2.2H_2O$ is illustrated in Figure 1.
The *fiber-like* byproducts were carefully eliminated, and fresh tartaric acid solution was introduced over the gel in each test tube. A substantial number of microcrystals formed near the gel interface, prismatic crystals of notable size appeared slightly farther from the interface. The quantity of crystals decreased as the distance from the gel interface increased, likely due to a reduction in the diffusion rate of the supernatant. Another contributing factor could be gel aging, as crystals forming in this region develop within an older gel. As the gel matures, the number of nucleation sites and the overall growth rate of crystals decline. Insufficient gel aging may lead to fractures during the introduction of the supernatant, which can affect the crystallization process. A high concentration of reactants leads to the formation of a dense layer composed of numerous microcrystals that accumulate at the interface. This phenomenon is likely due to a steep diffusion gradient near the gel interface. Conversely, when the reactant concentration is lower, crystal growth occurs at a slower rate, resulting in the development of well-defined prismatic crystals due to the gradual diffusion and limited reactant availability.

Fig 2: Growth of copper tartrate crystal

As shown in Fig-2, the optimized copper tartrate crystals formed near the gel interface within the test tube. Under higher magnification, these crystals exhibit a distinct prismatic shape. They appear opaque at the centre but become more transparent toward the edges**.** Large-sized crystals were successfully grown.

Parametric Analysis of Crystal Growth Behaviour

The growth rate of crystals is significantly influenced by several key parameters. Factors such as the size of the gel cell, the density and age of the gel, and the pH level play a crucial role in determining the rate of crystal formation. These parameters affect not only the number of nuclei formed (nucleation density) but also influence the growth dynamics, external shape (habit), and overall quality of the resulting crystals. Additionally, the concentration of the chemical reactants and the method used to vary this concentration (concentration programming) greatly affect the size, morphology, and structure of the crystals. The influence of each of these factors on crystal development is discussed in detail in the subsequent sections, based on the experimental results

Nucleation and Growth

Nucleation was observed after 7 to 8 days, mainly at the gel-supernatant interface. The number of nuclei decreased with increasing distance from the interface. Various parameters were studied to optimize crystal growth:

Table 1: Effect of pH on Nucleation and Growth

Test Tube No.	Acetic Acid (2N) (mL)	$Cucl_22H_2O$ (1M) (ML)	Gel pH	Gel Setting Time (Days)	Observations
1	5	5	3	No setting	Gel remained loose even after 1 month.
2	5	5	3.5	15	Few small crystals near the gel interface.
3	5	5	4	13	More microcrystals, transparent and well-isolated.
4	5	5	4.4	12	Prismatic, well-grown crystals.
5	5	5	5	7	Thick layer of microcrystals near the interface.

Although the initial pH of the gel does not necessarily reflect the pH after gelation, it significantly influences the gel structure, as well as the nucleation and growth of crystals. In the current study, experiments were conducted by keeping the amount of reactant the supernatant tartaric acid constant over the set gel. The pH of the gel was altered by adjusting the proportions of acetic acid, sodium metasilicate, and the second reactant, $CuCl_2$ $2H_2O$. The impact of pH variation on the crystal growth rate was systematically investigated.

Table 1 displays the relationship between different pH levels, the gel setting time, and the resulting crystal quality. It was found that a pH of 4.4 offers optimal conditions for forming a stable and transparent gel, which supports the growth of high-quality crystals. At pH levels below 4.4, the gel takes longer to set and tends to be mechanically unstable. In contrast, when the pH exceeds 4.4, gelation occurs too quickly, resulting in gels that lack transparency.

Despite the fact that the pH may shift after gelation, the initial pH remains a critical factor in determining the overall characteristics of the gel and the effectiveness of crystal growth. Based on the findings of this study, a pH value of 4.4 is ideal for achieving the best crystal quality.

Effect of Gel Density on Nucleation Density (Ph-4.4, Tartaric acid as a supernatant)

Henisch reported that the ideal specific gravity range for obtaining high-quality crystals is between 1.03 and 1.06. In the present study, gels with varying densities were prepared by mixing sodium metasilicate (specific gravity ranging from 1.03 to 1.06) with 2N acetic acid, while maintaining a constant pH. It was observed that as gel density decreased, the transparency of the gel improved. Generally, gels with higher density tend to produce crystals of lower quality, while those with lower density take longer to solidify and exhibit reduced mechanical strength. Additionally, an inverse relationship between gel density and nucleation density was identified higher gel density led to fewer nucleation sites. Table -2 presents the data on the number of nuclei formed at different gel densities, while Figure 3 illustrates the trend between gel density and nucleation density. In this experiment, the most suitable composition for gel preparation was found to be a 4:1 ratio of sodium metasilicate solution (specific gravity 1.04 g/cm³) to 2N acetic acid**.**

Table 2: Gel density affects crystal formation

Test Tube No.	Acetic Acid (2N) (mL)	$CuCl_2\ 2H_2O$ (1M) (ML)	Gel Density (g/cm³)	Number of Nuclei Formed	Observations
1	5	5	1.02	50	Large number of micro crystals near the gel interface.
2	5	5	1.03	42	Thick layer of attached micro crystals at the interface.
3	5	5	1.04	30	Some well-isolated crystals observed.
4	5	5	1.05	18	Crystals at the gel interface were very close together.
5	5	5	1.06	12	Grains of irregularly shaped crystals observed.
6	5	5	1.07	7	Few small opaque crystals of indefinite shape.

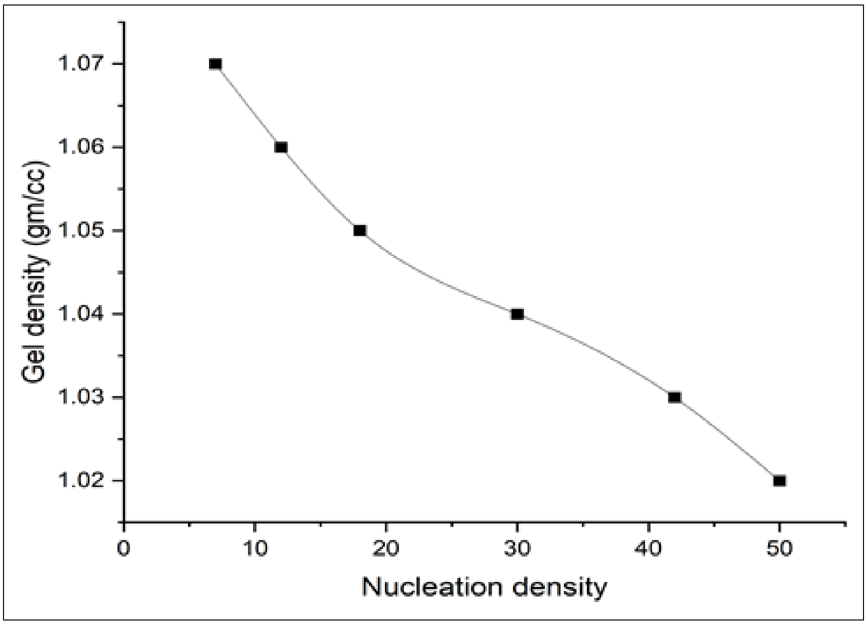

Fig 3: Correlation between Gel density and Nucleation density

Effect of Gel Aging on Nucleation

The influence of gel aging was examined by allowing gels of identical pH and density to age for different time intervals. After aging, a supernatant solution of fixed molarity was added to the gel as the feed solution. It was observed that as the gel aging time increased, the number of Copper tartrate crystals formed decreased. This decline is attributed to a reduction in pore size and diffusion rate, as well as a decrease in nucleation density. Prolonged aging also leads to more significant water loss from the gel. Water evaporation affects the gel in two stages: prior to setting and after the framework is formed. Before setting, the evaporation increases gel density, potentially enhancing the framework, but it can also introduce discontinuities due to shrinkage. Table 3.

Table 3: Gel aging time influences crystal growth.

Test Tube No.	Acetic Acid (2N) (mL)	$CuCl_2\ 2H_2O$ (1M)ML	Sodium meta silicate 1.04(gm/cc)	Gel Aging Time (Hours)	Number of Crystals	Observations
1	5	5	20	24	50	Few microcrystals formed
2	5	5	20	48	44	Increased nucleation density.
3	5	5	20	72	32	Reduced nucleation, crystals near interface.
4	5	5	20	96	22	Needle shaped prismatic crystals.
5	5	5	20	120	18	Well-grown prismatic transparent crystals.

Table-3 presents data on how aging time affects both the quantity and quality of the crystals, and Figure-4 illustrates the relationship between aging time (in hours) and the number of crystals formed. Based on the findings, a gel aging time of 120 hours was identified as optimal. At this duration, the gel maintains structural integrity without becoming overly dry, brittle, or fragile. Additionally, this aging period effectively reduces the number of nucleation centres, promoting the growth of high-quality crystals

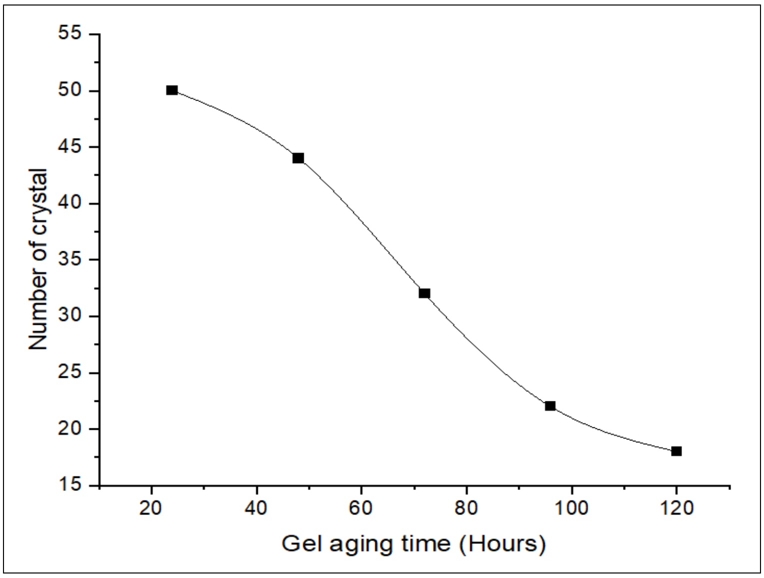

Fig 4: Relation between aging time (in hours) and the number of crystals

Effect of Reactant Concentration

The influence of reactant concentration was examined by maintaining a consistent gel pH and density while varying the molarity of the feed solutions. Tartaric acid and $CuCl_2.2H_2O$ were used as the primary reactants. Solutions of tartaric acid with concentrations ranging from 0.1 M to 0.4M were prepared. The tartaric acid solution was placed above the gel, while the concentration of $CuCl_2.2H_2O$ embedded within the gel was varied.

It was observed that increasing the concentration of $CuCl_2.2H_2O$ in the gel led to a rise in nucleation density. This trend is likely attributed to the greater availability of Cu^{2+} ions within the gel matrix, which promotes nucleation. For optimal crystal growth, the best results were obtained when the $CuCl_2.2H_2O$ concentration in the gel was maintained at 1.0 M, and the tartaric acid solution above the gel was set at 0.4 M.

To further explore the role of reactant positioning, the arrangement was reversed: tartaric acid of fixed molarity was embedded in the gel, and $CuCl_2.2H_2O$ feed solutions with varying concentrations (from 0.5 M to 1.5 M) were placed over the set gel. The results consistently highlighted the importance of appropriate reactant concentrations for achieving quality crystal growth.

Table 4: Effect of Reactant Concentration on Crystal Habit, Quality, and Size of Cu(C4H4O6)

$CuCl_2$ $2H_2O$ in gel(M)	Tartaric Acid in Supernatant (M)	Crystal Habit	Crystal Quality	Crystal Size
0.5	0.1	Irregular, fragmented	Poor	Small
0.8	0.2	Semi-prismatic	Moderate	Medium
1.0	0.4	Well-defined prismatic	Good	Large
1.2	0.4	Dense, clustered	Moderate	Medium
1.5	0.4	Aggregated, distorted	Poor	Small

This table presents a summary of how varying the concentrations of copper chloride $CuCl_2.2H_2O$ and tartaric acid influences the morphological characteristics, structural quality, and dimensions of copper tartrate crystals. Different combinations of reactant concentrations were tested to determine the optimal conditions for high-quality crystal formation. Observations indicate that the concentration of reactants plays a significant role in controlling nucleation rates, crystal habit, and overall growth.

Gradual Concentration Enhancement

After establishing the optimal conditions for reactant concentrations, single diffusion experiments involving gradual concentration enhancement were carried out to study its effect on nucleation control. Tartaric acid was embedded in the acetic acid gel, and feed solutions of $CuCl_2.2H_2O$ were prepared in concentrations ranging from 0.1 M to 0.4 M.The experiment began with a 0.1 M $CuCl_2$ solution gently layered over the gel. After 48 hours, this was replaced with a 0.2 M solution, followed by stepwise increases of 0.1 M every 48 hours, until the concentration reached 0.4 M.

It was observed that at the lower concentrations, nucleation did not occur. As the concentration increased incrementally, a few nuclei appeared. Further enhancement of the concentration resulted in the formation of only a small number of new nucleation sites, which enabled the previously formed crystals to grow without overcrowding. This approach led to moderate improvements in crystal appearance and morphology, with subtle changes in lustre also noted.

While the overall effect of gradual concentration enhancement was limited, it showed some potential in improving crystal quality by promoting more regulated nucleation and growth.

Conclusion

Based on the experimental observations, the following conclusions can be made:

1. Copper tartrate crystals can be successfully synthesized using the gel growth method.
2. The single diffusion technique proves to be effective for controlled crystal growth within a gel medium.
3. The morphology of the crystals can be influenced by adjusting key parameters such as gel density, gel aging duration, pH, and reactant concentrations.
4. An optimal gel density of 1.04 g/cm³ was found to support favorable crystal growth conditions.

5. Aging of the gel plays a significant role in regulating the rate of nucleation, helping achieve better crystal quality.
6. A pH value around 4.4 was identified as ideal for promoting the growth of well-formed copper tartrate crystals.

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