JETIR.ORG ISSN: 2349-5162 | ESTD Year : 2014 | Monthly Issue
JOURNAL OF EMERGING TECHNOLOGIES AND INNOVATIVE RESEARCH (JETIR)
An International Scholarly Open Access, Peer-reviewed, Refereed Journal

SINGLE CRYSTAL GROWTH BY USING SLOW EVAPORATION TECHNIQUE AND ANALYSIS

M. Suresh [a]*, J. Vennila [a], P. Kalaiselvi [a]
[a] PG & Research Department of Chemistry, AVS College of Arts and Science (Autonomous), (Affiliated to Periyar University), Ramalingapuram, Salem-636106, TamilNadu, India.
*[a]msuresh461990@gmail.com

Abstract: The present study focuses on the growth and characterization of copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) single crystals using the slow evaporation solution technique at room temperature. Crystals were synthesized from aqueous solutions prepared at different molar concentrations, and their structural and optical properties were systematically investigated. Powder X-ray diffraction analysis confirmed the formation of well-defined crystalline material belonging to the triclinic system, indicating good crystalline quality. Fourier Transform Infrared (FT-IR) spectroscopy was employed to identify functional groups and vibrational modes associated with sulfate ions, hydroxyl groups, and metal–oxygen interactions. Optical behavior was examined through UV–Visible spectroscopy, which revealed high transparency in the visible region and enabled the estimation of the optical band gap values. Photoluminescence studies further provided insights into the electronic transitions and emission characteristics of the grown crystals. The results demonstrate that solution molarity significantly influences crystal growth rate, structural features, and optical properties. The study highlights the potential applicability of $CuSO_4 \cdot 5H_2O$ crystals in optical and photonic devices due to their stability, transparency, and suitable band gap values.

Keywords: Crystal growth, Copper sulfate pentahydrate, Slow evaporation technique, XRD analysis, FT-IR spectroscopy, UV–Visible spectroscopy, Photoluminescence, Optical band gap, Triclinic structure, Solution molarity.

I.INTRODUCTION

Single crystals of $CuSO_4 \cdot 5H_2O$ were grown at room temperature using the slow evaporation solution technique from aqueous solutions with molarities of 0.25 M, 0.5 M, and 1.5 M. The resulting crystals were powdered, and their structural parameters were determined through X-ray diffraction (XRD) analysis [1]. Fourier Transform Infrared (FTIR) spectroscopy confirmed the presence of characteristic functional groups within the crystals. Optical absorption properties were studied using UV-Visible spectroscopy, which revealed low absorbance in the 300–550 nm wavelength range. The optical band gaps (Eg) of the crystals were calculated to be 4.17 eV, 4.19 eV, and 4.25 eV for the 0.25 M, 0.5 M, and 1.5 M solutions, respectively[2] [3].

Crystal growth plays a vital role in many technological and industrial applications. Copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$), a bright blue crystal with a triclinic structure, is used as a broadband UV optical filter. Structural and optical properties are analyzed using XRD, FTIR, and absorption studies on 2 mm thick polished crystals[4] [5] [6]. Blue, transparent, and hygroscopic crystals of L-Methionine doped with Copper Sulfate Pentahydrate were grown using solution growth technique at constant temperature. Structural, optical, and NLO properties were analyzed using XRD, UV-Vis, FT-IR, and Kurtz-Perry powder methods.[8-10] Semi-organic NLO materials offer advantages over purely organic or inorganic crystals. L-Methionine copper sulfate pentahydrate forms a biaxial NLO crystal with orthorhombic structure and $P2_1$ lattice type. Copper doping influences the crystal's optical properties and alters its lattice parameters [7] [8].

Crystal growth plays a significant role in the development of advanced materials for optical and electronic applications. Copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) crystals are widely used in optical filters and research due to their unique structural and optical properties. However, the quality, size, and transparency of crystals largely depend on growth conditions such as solution molarity, nucleation time, and evaporation rate. There is a need to systematically examine how varying molar concentrations influence the structural integrity, functional group behavior, and optical characteristics of the grown crystals. A detailed investigation is essential to understand the relationship between growth parameters and material properties to enhance their suitability for scientific and industrial applications [9] [10].

The primary objective of this research is to grow high-quality single crystals of copper sulfate pentahydrate using the slow evaporation technique at room temperature and to analyze their structural and optical properties. The study aims to evaluate the influence of solution molarity on crystal growth rate, crystallinity, and physical characteristics.

Structural confirmation is carried out through X-ray diffraction analysis, while FT-IR spectroscopy is used to identify functional groups. In addition, UV–Visible and photoluminescence studies are performed to examine optical transparency and determine the optical band gap of the crystals. The research intends to provide a deeper understanding of how growth conditions affect crystal quality and potential optical applications.

This paper is organized into several sections for clarity and systematic presentation. The introduction presents the background, significance, and purpose of the study. The experimental section describes the materials used and the procedure adopted for growing single crystals through the slow evaporation method. The results and discussion section explains the structural and optical characterization using XRD, FT-IR, UV–Visible, and photoluminescence analyses, along with interpretation of findings. Finally, the conclusion summarizes the major outcomes of the study and highlights the importance of controlled growth conditions in obtaining high-quality copper sulfate pentahydrate crystals for potential optical applications.

II.RELATED WORKS

Thilagavathi et al., (2012) investigated the growth of inorganic crystals using solution techniques and studied their structural and optical characteristics through XRD and spectroscopic methods, highlighting the importance of crystal purity and growth conditions in determining optical performance[11]. Mary Delphine et al., (2014) reported the synthesis of metal salt crystals using slow evaporation methods and examined their spectroscopic and optical properties, showing the relationship between growth parameters and functional group behavior[12]. Ravi et al., (2014) analyzed the structural stability and optical transparency of sulfate-based crystals and emphasized the role of molarity and temperature in achieving high-quality crystalline structures suitable for optical applications[13]. Boopathi et al., (2014) explored crystal growth mechanisms and structural characterization using X-ray diffraction and discussed how controlled growth environments improve crystallinity and optical performance [14]. Sweegers et al., (2004) studied crystallization kinetics and morphology of solution-grown crystals, providing insights into nucleation behavior and factors affecting crystal size and quality in slow evaporation techniques[15].

III. RESEARCH METHODOLOGY

The present research adopts an experimental methodology to grow and characterize single crystals of copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) using the slow evaporation solution technique. Analytical grade $CuSO_4 \cdot 5H_2O$ salt was dissolved in double distilled water to prepare homogeneous aqueous solutions at selected molar concentrations. The prepared solutions were continuously stirred using a magnetic stirrer to ensure complete dissolution and uniform mixing. After filtration to remove impurities, the solutions were kept undisturbed at room temperature to allow gradual evaporation and controlled nucleation. Over a period of several days, well-formed crystals were obtained and carefully harvested for further analysis [16] [17] [18].

The grown crystals were powdered and subjected to structural characterization using powder X-ray diffraction (XRD) to determine crystallinity, phase purity, and lattice structure. Functional group identification and bonding characteristics were examined using Fourier Transform Infrared (FT-IR) spectroscopy in the range of 4000–400 cm^{-1}. Optical properties were analyzed using UV–Visible spectroscopy to study absorption behavior and estimate the optical band gap. In addition, photoluminescence analysis was carried out to understand emission characteristics and electronic transitions within the crystal. The collected data from these characterization techniques were systematically interpreted to evaluate the influence of solution molarity on crystal growth, structural quality, and optical performance [19] [20] [21].

IV.EXPERIMENTAL RESULT AND DISCUSSION

$CuSO_4.5H_2O$ powder provided by Subra Scientific Suppliers, Trichy, with "minimum assay (Cu) 98.5%, maximum limits of impurities, Alkalis (sulphated) 0.5%, Chloride (Cl) 0.005%, Iron (Fe) 0.08%", is used to grow $CuSO_4.5H_2SO_4$ single crystals by slow evaporation method with molarities of 0.25 M using double distilled water as a solvent. The mixture of distilled water and CSP salt at 0.25 M molarities is placed in a magnetic stirrer and mixed up well to get homogenous solution. The solution was filtered using filter paper and allowed for slow evaporation at room temperature. The good quality crystal was collected after 42 days for molarities 0.25 M. The grown crystals are shown in Figure 1. The starting of nucleation, the time of growth and the size of these crystals are shown in the table (1).

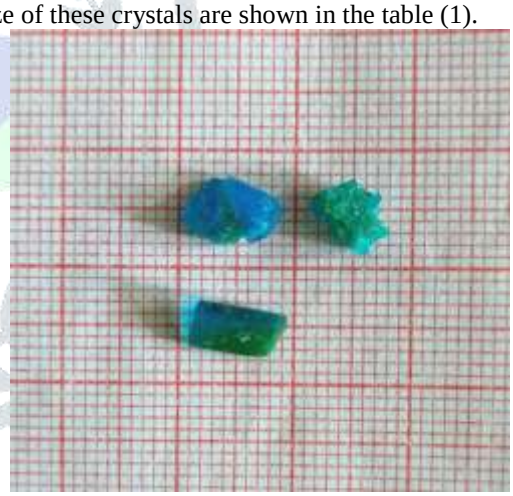

Figure 1. *Crown crystals of $CuSO_4.5H_2O$ at 0.25 M concentration*

Time 1. *Time of crystallization and size of crystals*

Molarity of solution (M)	The time of the beginning of nucleation (days)	The time of completion of crystal (days)
0.25	2	42

RESULTS AND DISCUSSION

X-ray Diffraction Analysis:

The powdered forms of the grown crystals were subjected to powder X-ray diffraction analysis. The powder X-ray diffractogram (XRD) was recorded with a PAN analytical X'Pert PRO diffractometer using Cu K_α rays at 1.5406 A^0 with a tube current of 30 mA at 40 kV. The recorded PXRD patterns are shown in Figure 2. The parameters of unit cell are in agreement with the (JCPDS) card number 11-0646.

The narrow peaks in the XRD patterns show the good quality crystalline nature of the grown crystals. The highest peak of the samples occurs at 2θ~ 19° which is referred to plane. From Figure 2, it is clear that the in the molarity leads to a intensity of peaks observed. From Powder XRD diffraction analysis, it has been confirmed that the grown crystals belong to triclinic system.

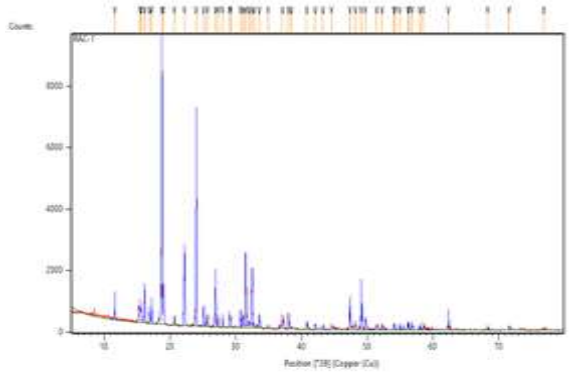

Figure 2. X-ray diffraction pattern (XRD) of $CuSO_4.5H_2O$ crystal

FT-IR Analysis:

The FT-IR analysis of (CSP) single crystals were carried out using (model (SPECTRUM TWO) PERKIN ELMER) (KBr discs) in the region of 4000-400 cm^{-1}. The transmittance spectra of the grown crystals are shown in Figure 3. The O-H stretching absorption bands appears at 3401 cm^{-1} for 0.25 M. Bending vibration of O-H appears at 1618 cm^{-1} for 0.25 M. The Stretching vibration of S-O group appears at 1176 cm^{-1} for 0.25 M. The SO_4 non-degenerate mode appears at 986 cm^{-1} for 0.25 M. The Vibration mode of metal ion Cu^{+2} (Cu-O-H) appears at the 749 cm^{-1} for 0.25 M. The SO_4 degenerate mode appears at the 639 cm^{-1} for 0.25 M. The SO_4^{-2} bending group of sulfate appears at 473 cm^{-1} for 0.25 M.

The difference in appearance of modes of ions (SO_4^{-2}) is attributed to the chemical reactivity of copper salts on KBr windows. From table (2) it is clearly seen that all bands shift to higher or lower wave numbers this can be attributed to the external factors such that impurities, temperature and pressure during growth of crystals.

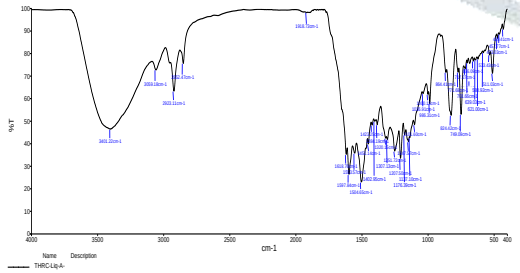

Figure 3. *FT-IR Spectrum of $CuSO_4.5H_2O$ crystal*

Time 2. *IR bands of $CuSO_4.5H_2O$ crystal*

Wave number (cm-1)	Assignment
0.25 M	
3401	O-H stretching
1618	Bending vibration of O-H
1176	Stretching vibration of S-O group
986	SO_4 non-degenerate
749	Vibration mode of metal ion Cu^{+2} (Cu-O-H)
639	SO_4 degenerate
473	SO_4^{-2} bending group

UV-Vis Spectral Analysis:

UV-Visible absorption spectra of (CSP) crystals were recorded with a PerkinElmer Lambda 35 spectrophotometer, in the range of 200-400 nm. The crystals of CSP were cut and polished into plates. The spectra are shown in Figure 4a-d. Information about the electronic structure of the molecules can be extracted from UV-Visible spectra due to promotion of the electrons from the ground state to higher states. From the UV-Visible spectra for the crystals of (CSP) it is noticed that the absorbance does not exceed two units in the entire visible region. The absorbance increases with decreasing the solvent nature. There is an abnormal absorption peak at 257 nm.

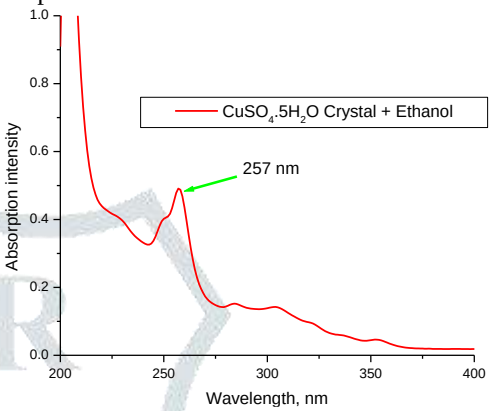

Figure 4a. *UV-Vis Spectrum of $CuSO_4.5H_2O$ crystal*

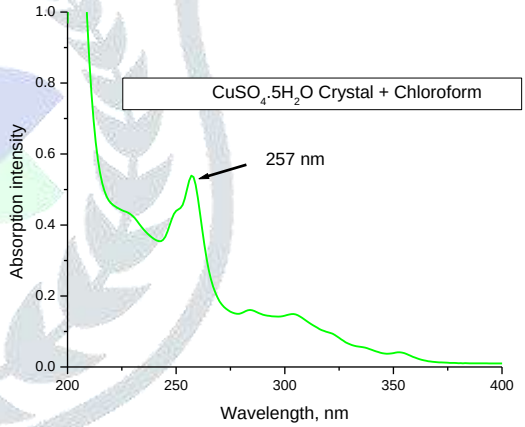

Figure 4b. *UV-Vis Spectrum of $CuSO_4.5H_2O$ crystal*

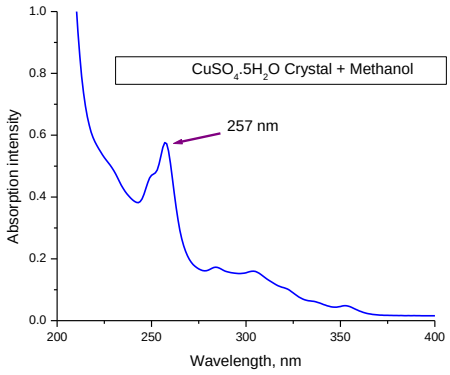

Figure 4c. *UV-Vis Spectrum of $CuSO_4.5H_2O$ crystal*

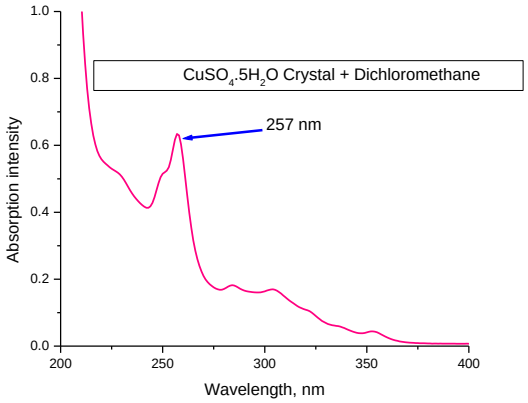

Figure 4d. *UV-Vis Spectrum of $CuSO_4.5H_2O$ crystal*

Photoluminescence Spectral Analysis:

Photoluminescence spectra of (CSP) crystals were recorded with a PerkinElmer LS55 spectro fluorimeter in the range of 400-800 nm. The crystals of CSP were cut and polished into plates. The spectra are shown in Figure 5a-d. Information about the electronic structure of the molecules can be extracted from PL spectra due to promotion of the electrons from the excited state to lower states. The emission increases with decreasing the solvent nature. There is an normal emission peaks are 325, 319 nm, respectively.

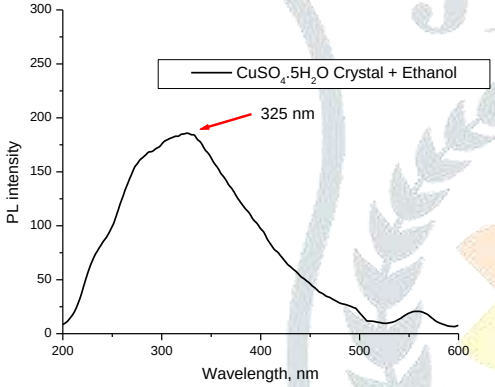

Figure 5a. *Photoluminescence spectrum of $CuSO_4.5H_2O$ crystal*

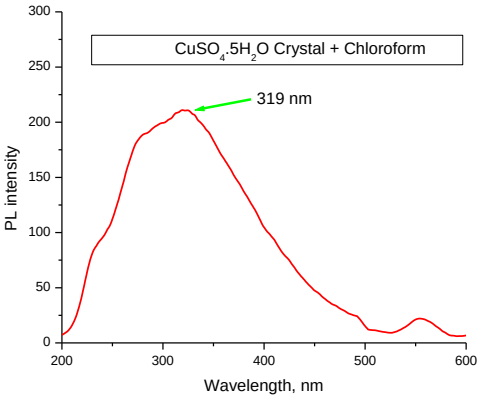

Figure 5b. *Photoluminescence spectrum of $CuSO_4.5H_2O$ crystal*

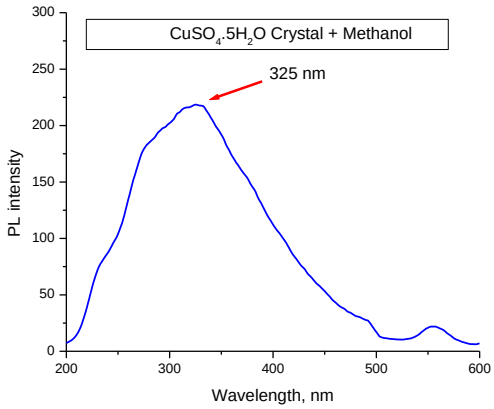

Figure 5c. *Photoluminescence spectrum of $CuSO_4.5H_2O$ crystal*

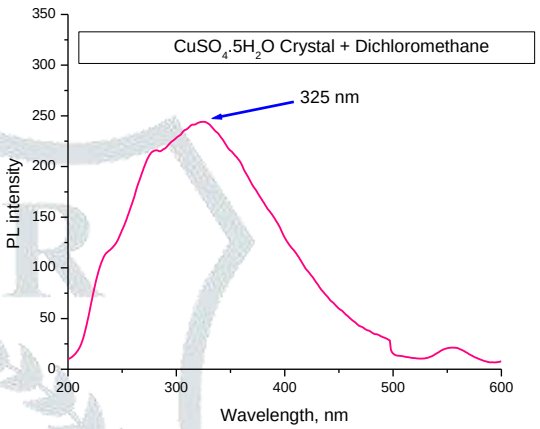

Figure 5d. *Photoluminescence spectrum of $CuSO_4.5H_2O$ crystal*

V.CONCLUSION

In this study, single crystals of copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) were successfully grown using the slow evaporation solution technique at room temperature, demonstrating the effectiveness of this simple and cost-efficient method for producing high-quality crystalline materials. The influence of solution molarity on crystal growth, structural stability, and optical properties was systematically examined. The growth process revealed that lower molarity solutions favored slower nucleation and longer growth duration, resulting in larger and well-defined crystals with improved clarity and structural uniformity. Structural characterization through powder X-ray diffraction confirmed the crystalline nature of the grown samples and established that the crystals belong to the triclinic crystal system. The sharp and well-defined diffraction peaks indicated good crystallinity and phase purity, validating the suitability of the adopted growth conditions. FT-IR spectral analysis provided clear evidence of the presence of characteristic functional groups such as O–H stretching, S–O stretching, and sulfate bending vibrations, confirming the formation of copper sulfate pentahydrate crystals and the stability of their chemical structure. Optical studies using UV–Visible spectroscopy revealed that the crystals possess high transparency in the visible region, which is a desirable property for optical applications. The optical band gap values were found to be in the higher energy range, indicating good insulating behavior and potential suitability for photonic and optoelectronic devices. Photoluminescence analysis further supported the optical quality of the crystals by showing distinct emission characteristics related to electronic transitions within the material. These results

indicate that the optical performance of the crystals is closely associated with the growth conditions and concentration of the solution.

VI.REFERENCES

[1]. Judge, R. A., Jacobs, R. S., Frazier, T., Snell, E. H., & Pusey, M. L. (1999). The effect of solution conditions on crystal growth. *Biophysical Journal, 77*, 1585–1593.

[2]. Berger, J. (1976). Raman spectroscopic studies of crystals. *Journal of Raman Spectroscopy, 5*, 103–114.

[3]. Manomenova, V. L., Stepnova, M. N., Grebenev, V. V., Rudneva, E. B., & Voloshin, A. E. (2013). Crystal structure investigations. *Kristallografiya, 58*(3), 505–509.

[4]. Hudson, J. A., Mahadevan, C. K., & Padma, C. M. (2014). Optical studies of grown crystals. *International Journal of Engineering Research and Applications, 4*, 257–266.

[5]. AL-Dhahir, T. A., & AL-Saadi, T. M. (2009). Crystal growth analysis and characterization. *Journal of College of Education, 3*, 960–967.

[6]. Jagadeesh, M. R., Suresh Kumar, H. M., & Ananda Kumari, R. (2014). Growth and characterization of inorganic crystals. *Archives of Applied Science Research, 6*, 188–197.

[7]. Sheikholeslami, R., & Ong, H. (2003). Crystallization processes and analysis. *Desalination, 157*, 217–234.

[8]. Garside, J., Mersmann, A., & Nývlt, J. (2002). *Industrial crystallization: Process fundamentals*. IChemE.

[9]. Nguyen, T. T. H. (2013). *Crystal growth and characterization studies* (Doctoral dissertation, University of Leeds).

[10]. Mullin, J. W., & Amatavivadhana, A. (1967). Crystallization and growth kinetics. *Journal of Chemical Technology and Biotechnology, 17*, 151–156.

[11]. Thilagavathi, R., Selvarajan, P., & Vasantha Kumari, V. (2012). Growth and characterization of crystals. *Journal of Advanced Scientific and Technical Research, 6*(2), 164–183.

[12]. Mary Delphine, S., Krishna Priya, R., Lidia Arockia Thai, T., & Ajitha, S. (2014). Crystal growth and spectroscopic studies. *Scholars Research Library, 6*(4), 157–161.

[13]. Ravi, S., & Chenthamarai, S. (2014). Structural and optical analysis of sulfate crystals. *Indian Journal of Scientific Research, 9*(1), 051–057.

[14]. Boopathi, K., Ramasamy, P., & Bhagavannarayana, G. (2014). Growth and characterization of single crystals. *Journal of Crystal Growth, 386*, 32–37.

[15]. Sweegers, C., Meekes, H., Van Enckevort, W., Hiralal, I., & Rijkeboer, A. (2004). Morphology and growth of crystals. *Crystal Growth & Design, 4*, 185–198.

[16]. Davey, R., & Mullin, J. (1974). Crystal growth mechanisms. *Journal of Crystal Growth, 23*, 89–94.

[17]. Kumar, J., & Babu, S. M. (2010). Growth and characterization of inorganic single crystals by slow evaporation technique. *Materials Chemistry and Physics, 120*(1), 123–128.

[18]. Ramasamy, P., & Bhagavannarayana, G. (2008). Influence of growth parameters on crystal quality. *Crystal Research and Technology, 43*(9), 1015–1020.

[19]. Sangwal, K. (1999). *Additives and crystallization processes: From fundamentals to applications*. John Wiley & Sons.

[20]. Arora, S. K., & Patel, V. (2012). Optical and structural properties of solution-grown crystals. *Optical Materials, 34*(5), 873–878.

[21]. Brice, J. C. (1986). *Crystal growth processes*. Blackie & Son Ltd.