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

A Review Of The Characterization Development Of Alkali And Transition Metal Element In Crystals Growths

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Abstract: **Crystals are the basis of a wide range of scientific and technological applications. The availability of high-quality crystals is critical to progress in these sectors. As a result, attempts have been made to improve crystal growth techniques. One of these is the gel growth technique. The current article offers a brief overview of the growth of many forms of crystals that can be grown by gel technique. The gel growth technique is straightforward and well-suited to the crystallization of chemicals that are only slightly soluble in water and disintegrate before melting. Without the use of costly instruments or high-temperature furnaces, this process can be set up in a laboratory with simple glassware. By carefully determining the gel's specific gravity, pH, and concentration of the reactants, good quality crystals can be grown at room temperature, Energy dispersive, x-ray photoelectron spectroscopy (XPS), UV-vis spectroscopy, Fourier transform infrared, photoluminescence (PL), electron paramagnetic resonance, and low-temperature magnetic investigations in the temperature range of 300-20 k characterize all researchers. The gel growth technique continues to entice diverse researchers even today.**

Keywords: **Gel growth, silica hydrogel, tartrate crystal, Low-temperature magnetic study, UV-VIS**

I. INTRODUCTION

Many researchers who have been interested in crystals produced in gel medium have written a good review study on gel growth strategies. Crystal growth plays an important role in all parts of materials science and in industries that depend upon the use of different materials(Patil and Patil 2015). Modern technology is widely demanded in materials for catalysis, semiconductors, pharmaceuticals, and optoelectronic devices due to the interesting properties of crystals such as piezoelectric, optical property, etc. The gel is a two-component system that is semi-solid, liquid-rich, and stable in shape. As well as possessing fine holes through which diffusion occurs. It is created when a gel-forming substance interacts with the solvating medium. The gel's distinguishing characteristics are that it includes a high percentage of solvent and little solid stuff. The gel structure is stabilized by the gel-forming substances and solvating solvents, although the gel may lose its solvent content during drying. Recently people are interested in growing pure as well as mixed crystals for the development of solid-state science. There is always a need for pure and defect-free crystals. Tartrate crystals are of considerable interest, particularly for basic studies of some of their interesting properties. The grown crystals are characterized by Fourier Transform Infrared spectroscopy, Energy Dispersive X-ray, UV-visible spectroscopy, and differential thermal analysis. The crystal structure has been studied by powder X-ray diffraction. Pure and doped crystals both possessed tetragonal structures. Tartrate compounds are used in a variety of medicinal, pharmaceutical, and industrial applications. Before melting, some of the tartrate compounds that are insoluble in water decay. Researchers were drawn to tartaric compound derivatives because of their practical applications in sectors such as sensors, lesser, optical filters, and non-linear optics, as well as features such as dielectric ferroelectric. Since the relevance of crystals, many researchers have been interested in crystal development in gel medium because it is a simple and effective method. Gel methods could be used to grow a variety of tartrates. Crystal growth in gel medium is a simple and efficient method. Some tartrate crystals can be grown by gel technique copper tartrate has been shown to stimulate the luteinizing hormone in vitro. The silica gel method of single diffusion-reaction technique, which was adopted by the majority of researchers, was capable of yielding crystals with high optical perfection and a wide morphology, as well as being quite suitable for the growth of fully developed and noticeably transparent crystals(*H.O.Jethva,andR.R.Hajiyani 2010*).

The copper's most important applications and a small investigation on stereoselective effects in the creation of complexes between the cooperation and optically active racemic tartrate. The researchers are noticing that crystals are of tremendous interest from the standpoints of both solid-state science and technology. Using the gel approach, various researchers have documented points of view. The gel growth technique appeared quite attractive for growing crystals, on account of its unique advantages in terms of crystals produced and the simplicity of the process. Moreover, the method is inexpensive and within the scope of the small laboratory(Arora et al. 2007).

II. LITERATURE SURVEY

(Mathivanan and Haris 2013) In copper–doped iron tartrate crystal grown by silica gel method Single crystals of pure and copper-doped iron tartrate were successfully produced from the gel using the single diffusion method. The dopant Cu2+ had infiltrated the lattice of the iron tartrate crystals, according to the EDAX analysis. The occurrence of O–H, C-H FTIR spectroscopy validated the C–O, C–H, and metal-oxygen bonds. XRD (from XRD) studies the unit cell capacity of pure iron tartrate was determined to be 650.863 A^0 and 702.073A^0.copper-doped iron tartrate was discovered. The pure magnetic moment, the formation of pure and mixed crystals of copper-iron tartrate in silica gel is described in this paper. The characterization of these mixed crystals is carried out for the first time in this study. The following reaction was expected to occur:

$FeSO_4 + C_4H_6O_6 \rightarrow FeC_4H_4O_6 + H_2SO_4$ (Pure iron tartrate)(1 − x) $FeSO_4$ + x $CuSO_4$+$C_4H_6O_6$ → CUXFE (1−x) $C_4H_4O_6 + H_2SO_4$.

(H.O.Jethva et al 2016)The crystals of copper Levo-tartrate and copper Dextro tartrate were produced using copper sulfate as a supernatant solution, and the following reaction is expected to occur in the creation of copper Levo-tartrate and copper Dextro tartrate crystals.

$CuSO_4\ nH_2O + H_2C_4H_4O_6 \rightarrow CuC_4H_4O_6.mH_2O + H_2SO_4 +$
(n-m) H_2O.

The FTIR spectra of copper Levo-tartrate and copper dextro-tartrate crystals were nearly the same and confirmed the presence of O-H, C=O, and C-H functional groups,(2)in this research researcher gives the difference in the properties can be explained based on the bi-nuclear nature of copper tartrate and different symmetry environment.

Single crystals of copper doped zinc mercury thiocyanate ZnHg (SCN) 4 have been successfully produced in a silica gel medium at a pH of 2.8 using the diffusion technique. X-ray diffraction, Fourier transform infrared spectroscopy, UV optical studies, SHG studies, EDAX, and thermogravimetric analysis were used to describe the crystals. The crystal system was found as a modified tetragonal-disphenoidal class with the I 4 space group in the ZMTC crystal family. When compared to pure ZMTC, the copper-doped ZMTC single crystal has improved nonlinear optical characteristics. Single crystal X-ray diffraction validated the generated crystal's tetragonal shape. The solid morphology diagram indexes all of the crystal's faces. FTIR research revealed the existence of several functional groups. With a cutoff of 260 nm, UV spectral analyses show a high percentage of uniform transmission. Kurtz powder tests revealed a greater efficiency compared to pure ZMTC, suggesting that the formed crystal could be used in nonlinear optical applications. The relative constituent composition and weight present in the crystal were determined using EDAX analysis. (Jagdish and Rajesh 2012)

(Dr. C. Balalakshmi., et al.2017)Innovations can be seen in this investigation. The bandgap was discovered using UV-VIS and Photoluminescence spectroscopic experimental approaches for copper tartrate crystal. Expected reactions are followed as

$CuSO_4.nH_2O+H_2C_4H_4O_6 \rightarrow CuC_4H_4O_6.mH_2O+H_2SO_4+(n\text{-}m)$ H_2O. Where $n \geq m$

In this study observed photoluminescence spectrum has two bands, one at 508.6nm and the other at 764.7nm, with the smaller peak at 508.6nm.

Authors have attempted to develop copper doped iron tartrate crystals in gel media as part of their characterization procedures for copper doped iron tartrate crystals

(Dhikale and Shitole 2018). Expected reaction for growth copper tartrate crystal. The reaction to growing copper doped iron tartrate crystals is –

(1 − x) $FeSO_4$+ x $CuSO_4$+$C_4H_4O_6$→ Cu(x) Fe (1-x) CH_4O_6+ H_2SO_4

According to the UV-VI analysis, the energy bandgap of the following tartrate crystal is 3.19 eV, indicating that the crystal is an insulator and the grown crystal shows uniform morphology.

(Ariponnammal and Velvizhi 2019)examined the paramagnetic character of copper tartrate crystals at various temperatures ranging from 333 - 77 K to 20 K to confirm the paramagnetic and weak ferromagnetic signal.

$CuCl_2 + C_4H_6O_6 \rightarrow CuC_4H_4O_6 + 2HCl$

The magnetic properties of the M-H curve were investigated, and it proved its paramagnetic behavior up to 77K. Below 77K, at 20K, it revealed a weak indication of interesting ferromagnetic behavior. Low temperature combined with a magnetic field causes the crystal to become more ordered.

The majority of tartrate composites do not dissolve in water and must be molded before melting, according to the circumstances of copper single crystals discovered under varied environmental conditions. In his dissertation, he exhibits the optimum condition of CoCT crystals. Energy-dispersive X-ray spectroscopy confirms the presence of cobalt, copper, carbon, and oxygen (EDX). Depending on the growth and environmental circumstances, the crystals obtained are regular or elongated in shape. He eventually concluded that cobalt copper tartrate single crystals can be produced at room temperature if more laser medium is used than other lights. The nucleation rate of CoCT single crystals is reduced if more laser medium is used than other lights. FTIR and PXRD are used to characterize the produced crystals. The temperature behavior of the formed CoCT crystal is explained by TGA tests. The energy gap was determined and the optical transparency of the produced crystals was studied using UV-Vis-NIR transmission spectroscopy analysis. (K. V. Pradeepkumar, Jagannatha, and Rohith 2020a)

(Rohith, Jagannatha, and Pradeep Kumar 2019)explored magnesium doped copper cadmium oxalate single crystals, with such a special emphasis on the growth of pure and magnesium doped copper cadmium oxalate crystals in a single diffusion techniques and use the procedure described below.

$$H_4 SiO_4 + 2NaOHR \rightarrow Na_2 SiO_3 + 3H_2O$$

This sodium hydroxide is expected to react with oxalic acid and generate magnesium doped copper mixed cadmium oxalate (MDCCO) crystals in a gel from the supernatant solution.

The objective of (Nandre, Shitole, and Ahire 2012)paper is to show how various parameters affect the growth mechanism of zinc tartrate crystals in silica gel at room temperature. The following reaction was expected to take place inside the gel to generate a crystal:

$$C_4O_6H_6 + Zncl_2 \rightarrow ZnC_4H_4O_6 + 2HCl$$

This paper illustrates the straightforward conversion equation of Bandgap energy (eV) = 1240 / wavelength (nm)At a wavelength of 285 nm, the bandgap energy of Zinc tartrate crystal is found to be 4.35 eV.

(S.J. Nandre, et al 2013) Research on gel-grown cobalt tartrate crystals describes the growth of cobalt tartrate crystals grown in silica gel and their characterization using FT-IR, TGA, and various thermal analyses as well as UV absorption spectroscopy. He explained how to react with tartrate ions by diffusing cobalt ions via the narrow ports of the gel. To generate a crystal, the following reactions take place in gel media.

$$CoCl_2 + C_4H_6O_6 \rightarrow CoClC_4H_6O_6 + 2HCl\ .$$

They experimented with other factors such as reactant concentrations, gel pH, solvent contaminants, gel setting time. etc. The single crystal of cobalt tartrate was grown by the gel grown technique through this research, and all characterizations show the formation of cobalt tartrate crystals.

To change the PH of the solution in this study, a mixture of two acids was used. At the ideal temperature, single crystals of cadmium tartrate oxalate were produced utilizing a gel approach and a single diffusion method. The effect of changing several process factors on crystal formation, such as gel PH, gel setting time, and gel concentration of the reactance, was investigated. Sodium metasilicate (Na_2SiO_3) is combined with a mixed solution of oxalic acid ($C_2H_2O_4$) and tartaric acid ($C_4H_6O_6$) in the necessary mole fraction during the gel formation process. X-ray powder Diffractogram, Fourier Transform Infrared Spectroscopy, quantitative elemental analysis of EDAX, and scanning electron microscopy were used to describe the harvested crystals. The polycrystalline character of these materials is revealed by powder XRD findings. All of the bands expected from the metal tartrate oxalate with water of crystallization are visible in the FTIR spectrum of these crystals. EDAX also confirms the existence of cadmium, carbon, and oxygen. The structure in the shape of flat plates is seen in SEM pictures. However, the primary goal of this research is to investigate 1) the formation of pure and magnesium doped copper cadmium oxalate (MDCCO) crystals in a single diffusion process, and 2) the thermal and optical properties of MDCCO crystals(D.Arumugam and A.Krishnan 2013).

(Sonawane and Ahire 2014)Single crystals of calcium tartrate (CaTr) were produced in this study using a simple gel approach and a single diffusion procedure. Variations in process factors such as gel concentration, pH of the gel, gel aging, reactant concentration, and so on were used to find the best growth conditions. The test tubes served as crystallization vessels, and the growth media was silica gel. The gel was made by combining a tartaric acid and sodium metasilicate solution. It was then left to age after the gel had been set. The supernatant calcium chloride of various molarities was poured over the set gel after two or three days. Small nucleation development was seen below the gel interface after 48 to 60 hours of supernatant pouring. In this study, a few prismatic, semitransparent crystals of calcium tartrate were produced, as well as some white crystals. EDAX, SEM, and UV spectroscopy were used to describe these crystals.

The formation of pure and mixed crystals of cobalt and cadmium levotartrate is achieved in this study using the gel method. It is possible to obtain spherulitic and dark brown crystals. EDAX calculates the metallic element content of the crystals. The existence of various functional groups is confirmed by FTIR spectroscopy data analysis. The mixed-phase character of the produced crystals is suggested by the powder XRD. The orthorhombic phase of cobalt Levo-tartrate and the monoclinic phase of cadmium Levo-tartrate were found using Rietveld analysis. The thermograms show that after water molecules are removed, crystals are converted to oxide form(Manani, Jethva, and Joshi 2020).

Single crystals of magnesium doped copper tartrate were effectively produced in the silica gel medium under various light conditions. By adjusting several parameters, the best conditions were discovered. To alter the nucleation, MgCT crystals are produced at room temperature using a compact fluorescent light (CFL) and a semiconductor laser. (Pradeep Kumar, Jagannatha, and Rohith 2020) It was discovered that the nucleation rate of MgCT crystals decreases more in the laser medium than in other light radiation and that without any irradiation medium, different lights interact and diminish nucleation while increasing crystal size, number, and transparency. Because of the variations in supersaturations, this is the case. The presence of Magnesium copper cation and tartrate group bond in these MgCT single crystals is confirmed by FTIR and EDX spectral investigations. Above 500oC, the produced crystals are extremely crystalline and thermally stable. UV-Visible spectroscopy experiments reveal that gel-produced MgCT single crystals have an insulating and transparent behavior characterized by spectral studies, allowing these materials to be used in optoelectronic devices.

The silica gel medium was used to successfully produce single crystals of magnesium doped Copper Tartrate in a variety of environments. By adjusting several parameters, the best conditions were discovered. MgCT crystals are produced at ambient temperature and exposed to a variety of light and laser sources. It was discovered that the rate of

MgCT crystal nucleation decreases more in the laser medium than in other light radiation and in the absence of any irradiation medium, which is related to supersaturating variation. The presence of magnesium, copper cation, and tartrate group bonds in this MgCT single crystal is confirmed by FTIR and EDX spectral analyses. UV-Visible indicates that gel-produced MgCT single crystals have an insulating and transferring behavior characterized by spectral measurements, allowing these materials to be used in optoelectronic devices. (K. V. Pradeepkumar, Jagannatha, and Rohith 2020b)

(Rajesh et al. 2019)Single crystals of metal ion-based Barium L-Tartrate and amino acid-based L-Alanine Tartrate were produced using a slow evaporation approach. Vicker's microhardness inventor was used to determine the mechanical properties of the crystals. The modest stress to the dislocation is confirmed by the slight shift in microhardness number HV with increasing load for both BaTr and LAT crystals. Both BaTr and LAT crystals are classified as hard materials. The brittleness of the BaTr crystal is superior to that of the LAT crystal. As a result, the BaTr crystal is a good elastomeric. In comparison to L-Alanine and Tartaric acid, the bonding between the atoms of barium and tartaric acid is quite strong. Overall, both crystals have good mechanical properties, indicating that they are suitable for material manufacturing and structural materials. The action of a metal ion (barium) on the tartrate ion in Barium L-Tartrate results in a material with improved hardness, bonding properties, and high elastic properties. As a result, we may conclude that BaTr crystal is a better choice for fabricating optoelectronic and photonic devices.

(K. V. Pradeepkumar, Jagannatha, and Rohith 2020a)Single crystals of Cobalt doped Copper Tartrate are produced at ambient temperature and various light sources, including compact fluorescent light and laser medium. Due to variations in supersaturating, the rate of CoCT single-crystal nucleation is lowered more in the laser medium than in other lights-exposed mediums and at room temperature. These findings are documented and compared to the stated figures. This demonstrated the impact of a produced crystal in various conditions. SEM investigation was also performed, revealing the surface shape of a single crystal of CoCT. TGA analysis records the breakdown temperature and percentage of weight loss of the formed crystal. PXRD is used to calculate CoCT lattice parameters and the structure of the unit cell.

The complexity of the 2p spectra arising from shake-up structures for Cu(II) species and overlapping binding energies for Cu metal and Cu(I) species makes X-ray photoelectron spectroscopic study of copper species difficult. For a wide spectrum of copper-containing compounds, this paper builds on and expands previously published X-ray photoelectron spectroscopy curve-fitting and data processing approaches. The following are some of the steps taken: (i)an examination of existing Cu $2p_{3/2}$ main peak and Cu $L_3M_{4,5}M_{4,5}$ Auger parameter literature data, (ii) analysis of a series of quality standard samples, (iii) curve-fitting procedures for both the Cu $2p_{3/2}$ and Cu $L_3M_{4,5}M_{4,5}$ spectra (as well as associated anions), (iv) calculations determining the amount of Cu(II) species in a mixed oxidation state system. Several solutions have been proposed to solve some of the challenges of XPS-based copper species assignment and quantification. A more comprehensive examination of copper-based materials has become possible with the use of a variety of methodologies and the incorporation of all relevant data. An examination of binding energies and Auger parameter values, realistic advanced curve-fitting approaches applied to the Cu $2p_{3/2}$ and Cu $L_3M_{4,5}M_{4,5}$ spectra, and measurements of mix oxide/hydroxide surface coating thickness are among the strategies given(Biesinger 2017).

(Rohith, Jagannatha, and Pradeep Kumar 2019)Single crystals of Pure (CCO) and Magnesium Doped Copper Cadmium Oxalate (MDCCO) with sizes of 0.300.150.10 were produced in silica gel medium at 23 C temperatures using the single-diffusion reaction method. Powder X-Ray Diffraction (PXRD), Fourier-Transform Infrared (FT-IR) Spectroscopy, UV-Visible spectrum investigations, Scanning Electron Microscopy-Energy Dispersive X-ray Analysis (SEM-EDAX), and Thermo Gravimetric Analysis /Differential thermal analysis (TGA/DTA) were used to describe the crystals. The gel method was used to grow pure and magnesium-doped Copper Cadmium Oxalate single crystals. By adjusting several parameters, the best conditions were discovered. The crystalline characteristics of as-grown crystals are revealed by powder XRD. The presence of predicted functional and metal-oxygen bound groups in the as-grown crystal is confirmed by FTIR and EDAX spectrum analyses. On the surface of the formed crystals, the SEM micrograph displays good perfection and minimal flaws. The MDCCO crystals were insulators and ideal for the manufacture of materials for optoelectronic devices, based on their bandgap energy and wide transparency. The TGA/DTA investigated the thermal stability of the material to support its use in the electronic industry.

Using varying gel densities and variable quantities of orthophosphoric acid and supernatant solutions, pure and Calcium, Cadmium, Magnesium doped zinc hydrogen phosphate (MZHP) crystals develop in the silica gel medium using the single diffusion method. For crystal growth investigations, a sodium metasilicate gel with a significantly acidic cation exchange in the H-form was used. The effect of pH, gel concentration, and upper and lower reactant concentrations on crystal size, quality, and nucleation density is explored. To increase the crystal size, concentration programming and seeded growth procedures are applied. The pH range where HPO_4^{2-} ions predominate was investigated, which is required for the crystal formation of zinc hydrogen phosphate (ZHP), calcium doped zinc hydrogen phosphate (CAZHP), cadmium doped zinc hydrogen phosphate (CDZHP), and magnesium doped zinc hydrogen phosphate (MZHP). Different shapes and behaviors of crystals were obtained. Some of them were diamond-shaped translucent platelets, while others were twined. The effect Single diffusion was used to make pure zinc hydrogen phosphate ($ZnHPO_4$) and calcium, cadmium, and magnesium doped $ZnHPO_4$ crystals in the form of single crystals. In pure $ZnHPO_4$ and calcium doped $ZnHPO_4$ crystals, the absorption peaks produced in the FT-IR spectrum proved the water of crystallization, symmetric and asymmetric stretching, bending vibration of PO_4 units, and metal-oxygen bonds. The existence of water of hydration and other functional groups in both pure and doped zinc hydrogen is further supported by FTIR studies (T.Jayaprakash P.Kalugasalamb, G Rajeshkanna 2016)

(Ahmad, Ahmad, and Kotru 2015)LHT crystallizes as single crystals after a controlled reaction of lithium chloride with tartaric acid in a silica gel media. The crystals have a distinct shape and are beautifully facetted. Gel concentration, pH, age, reactant concentration, and temperature all have a significant impact on crystal count. The following conditions were found to be optimal for the development of single crystals of LHT: gel concentration 0.2-0.6 M, pH 4, gel age 96 hours, LR concentration 0.5 M, UR concentration 0.8M at a temperature of around 25^0C. The IR spectra of tartrate molecules coupled with water of hydration show all of the predicted peaks. Optical absorption tests have indicated that the optimum bandgap of LHT crystals is 4.83eV. These crystals have a wide bandgap and low absorption over the entire visible range, making them a good contender for optoelectronics. With the increasing frequency of the applied ac field, the dielectric constant of LHT drops steadily. With the increasing frequency of the applied ac field, the dielectric loss reduces slowly. The dependence of ac on frequency indicates that the conduction phenomenon is of the ac type, which could be attributed to charge carrier hopping.

(Rohith P S, Jagannatha N, and Pradeep Kumar K V 2018)The gel method was used to generate single crystals of Magnesium doped Copper Cadmium Oxalate. Single crystals of Magnesium (Mg2+) Doped Copper Cadmium Oxalate (MDCCO) were produced at ambient temperatures utilizing a single diffusion-reaction technique and silica gel. TGA/DTA stands for Thermo Gravimetric Analysis/Differential Thermal Analysis. The formed crystals' UV-visible absorption and transmission spectra were obtained. The findings were discussed and published. By adjusting several parameters, the best conditions were discovered. The presence of predicted functional and metal-oxygen bound groups in the as-grown crystal is confirmed by FTIR and EDAX spectrum analyses. On the surface of the formed crystals, the SEM micrograph displays good perfection and minimal flaws. The MDCCO crystals were insulators and ideal for the manufacture of materials for optoelectronic devices, based on their bandgap energy and wide transparency. The TGA/DTA investigated the thermal stability of the material to support its use in the electronic industry.

III. CONCLUSION

By using a single gel diffusion method in the silica gel that is sparingly soluble water, the gel technique is the simplest way to create various types of pure and mixed crystals. This method can also be used to grow different kinds of crystals. Different types of characterization can be used to confirm crystal structure. The wide transmission across the whole visible range in the above review makes it a good choice for optoelectronic applications. The pH of a mixture of sodium metasilicate and tartaric acid had a considerable influence on the crystal growth duration in all of the experiments. The morphology of gel-grown crystals is consistent, and the crystal particles have a smooth surface. The material's suitability for UV filters and optoelectronic applications was demonstrated by a UV-vis investigation. Room temperature, as well as compact fluorescent light and laser medium, are useful for forming crystals. Thermal stability is investigated using TGA/DTA, and crystal morphology is examined using SEM pictures. The optimal growths for an equimolar condition are obtained in all studies. Changing parameters such as gel density, gel aging, gel pH, and reactant concentration will provide various crystal characteristics. Finally, this explores the impact of a grown crystal in various environments.

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