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Study of Growth and Characterization of Cobalt Tartrate Crystals

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Abstract — In present investigation, single crystals of cobalt tartrate were grown by using silica gel as a growth medium. These single crystals were grown by simple gel technique using diffusion method. The optimum growth conditions for these crystals were optimized by varying various parameters. The circular shaped, opaque and brown colored crystals were observed. The crystalline nature of grown crystal was confirmed by using powder X-ray diffraction technique which shows that cobalt tartrate hydrate has crystallized in orthorhombic structure. The functional groups present in the crystals were identified by using Fourier Transform Infrared Spectroscopy (FTIR) analysis which shows that the presence of O-H bond, C-H bond and metal–oxygen bond. The Scanning Electron Microscopy (SEM) study reveals that the crystals having flat, rectangular and orthorhombic shapes of different sizes and structures embedded in rock like structure. The analysis of X-Ray Spectroscopy (EDAX) has shown the presence of Cobalt and Oxygen. The values of energy gap and band gap energy were calculated from UV-visible absorption spectra and these values were determined as 8.45 eV and 5.27 eV respectively. The Differential Scanning Calorimetry (DSC) was done to find the thermal properties of the crystal which manifest the water of hydration in the crystal.

Keywords — *Gel Growth; Cobalt Tartrate; X-Ray Diffraction; Fourier Transform Infrared Spectroscopy; Scanning Electron Microscopy; X-Ray Spectroscopy; Differential Scanning Calorimetry.*

1. Introduction

Several researchers have grown materials of great interest by gel technique [1]. They have modified such materials by suitable substitution for determination of the effect of modification of the composition. They have focus on the characteristics of the modification of the composition [2-5]. This growth process is free from convection, a systematic study of crystallization in gels begins with Lissegang's famous discovery of periodic crystallization in gel. This method has received a lot of attention because of its simplicity and efficiency in growing a single crystal of a particular compound. This process is one way to address growth through controlled distribution. This growth process has no convection [6-9].

Crystal habit of various crystals, grown under different conditions and also by different methods such as, melt growth, vapour phase growth, solution growth and gel growth were described by H.E. Buckley [10], P. Hartman [11], K. Kern [12], A. A. Chernov [13],W. K. Burton [14] and J. Mullin [15]. A number of factors such as concentration of reactants, pH of gel, impurities in the solvent, gel setting time, gel aging time etc. have considerable effect on growth rate. Growth and Characterization of some tartrate crystals were reported by Henisch and Henisch et al [16], Patel and Rao [17]. Growth of some crystals of tartrate compounds like calcium tartrate [18], barium tartrate [19], ammonium tartrate [20], zinc tartrate [21], sodium tartrate [22], and cadmium tartrate [23] were reported by earlier researchers. The compounds of tartaric acid find numerous applications in semiconductor and optics industries with the invention of lasers. The tartrates find many applications in science and technology such as ferroelectric applications, ferroelectric-Ferro elastic applications and dielectric applications. Some tartrate compounds are used as tracers for military purposes [24-27]. They are also used for construction of transducers, many linear and non-linear mechanical devices [28, 29].

In the present investigation, single crystals of cobalt tartrate were grown by a simple gel technique using diffusion method. The optimum conditions were established by varying various parameters such as pH, concentration gel solution, setting time of the gel solution and concentration of the reactance. The optimum growth conditions for these crystals were determined. These crystals were characterized by using XRD, FTIR, SEM, EDAX, UV-visible and DSC.

2. Experimental Analysis

Cobalt tartrate crystals were grown by single diffusion method in silica gel medium at room temperature. The Sodium Meta Silicate (Na_2SiO_3) solution and acetic acid (CH_3COOH) was prepared by dissolving 22gm (Na_2SiO_3) into the 250ml distilled water and 15ml (CH_3COOH) dissolving into 250ml distilled water respectively. Then (Na_2SiO_3) was added into 6ml (CH_3COOH) drop by drop by maintaining the pH 4.2 with continues till the solution becomes milky. After that 15ml solution of Cobalt chloride ($CoCl_2$) with 1M added into the gel solution. This mixture was then transferred to the test tube of 15 cm × 2.5 cm

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dimension. The open end of the tub was sealed with cotton, preventing evaporation and contamination of the exposed area and stored tubes at room temperature.

After setting the gel in 4-5 days, the 10ml tartaric acid ($C_4H_6O_6$) with 1M was allow to fall steadily along the wall of the tube above the set gel, after two days the small nucleation growth was observed at below the interface of gel. This nucleation growth was increased in size. The chemical reaction inside the gel can be expressed as follows:

$$2CH_3COOH + Na_2SiO_3 \rightarrow 2CH_3COONa\downarrow + SiO + H_2O$$
$$2CH_3COONa + CoCl_2 \rightarrow (CH_3COO)_4Co\downarrow + 4NaCl$$
$$(CH_3COO)_4Co + C_4H_6O_6 \rightarrow C_4H_4CoO_6\downarrow + 4CH_3COOH$$

Good quality circular shaped opaque brown coloured cobalt tartrate crystals were grown within28 days. The crystalline nature of grown crystal was confirmed by using powder X-ray diffraction technique. The functional groups present in the single crystals were identified using Fourier Transform Infrared spectroscopy (FTIR) analysis. The SEM was done to find the structural morphology of the grown crystals. The presence of Cobalt and Oxygen was confirmed by EDAX analysis. The values of energy gap and band gap energy were calculated from UV-visible absorption spectra. The Differential Scanning Calorimetry (DSC) was done to find the thermal properties of the crystal.

3. Results and Discussion

The figure 1 shows the crystals of cobalt tartrate attached themselves and forming a thick layer at the interface. Fig.2 shows a few grown crystals of cobalt tartrate having different habits with their scaling on a graph paper. The grown crystals are of the 2 mm × 2 mm × 1.5 mm size. The various optimum conditions for growing crystals were established and are given in the Table 1. For pH 4.2 the circular shaped opaque, brown colored crystals were observed in the interface of the gel column [30].

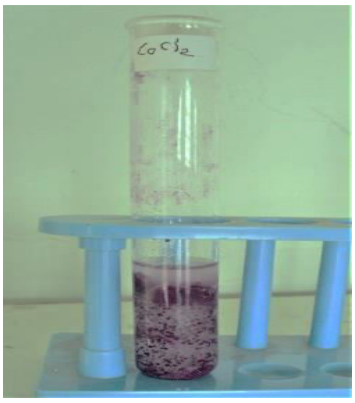

Fig. 1: Grown ($C_4H_4CoO_6$) crystals

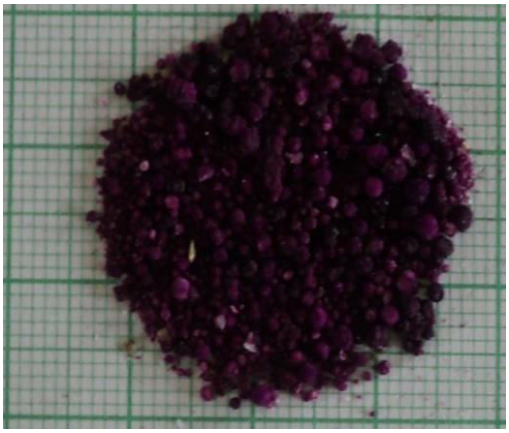

Fig. 2: Brown circular shaped opaque ($C_4H_4CoO_6$) crystals

Table 1. Optimum condition for growth of cobalt tartrate ($C_4H_4CoO_6$) crystals

Sr. No.	Conditions for growth of ($C_4H_4CoO_6$) crystals	Single diffusion
01	Density of sodium meta silicate (Na_2SiO_3)	1.05 gm/cm^3
02	Amount of 1M acetic acid (CH_3COOH)	6 ml
03	pH of the gel	4.2
04	Temperature	Room Temperature
05	Concentration of Cobalt Chloride ($CoCl_2$)	15 ml
06	Concentration of 1M Tartaric acid ($C_4H_6O_6$)	10 ml
07	Gel setting period	4-5 days
08	Gel aging period	24 Hrs
09	Growth of period	28 days
10	Quality	Circular shaped, Opaque and brown coloured crystals
11	Size of Crystals	2 mm × 2 mm × 1.5mm

3.1 Powder X-ray Diffraction Analysis of Cobalt Tartrate Crystals

Powder X-ray diffraction technique is used to investigate the crystal structure of the sample compound. The X-ray diffraction was recorded using Bruker-D8 Advance, Germany (2θ from 5° to 80°) with CuKα radiation of wavelength λ=1.54060Å. The recorded X-ray diffraction of the cobalt tartrate crystals is shown in the Fig.3.

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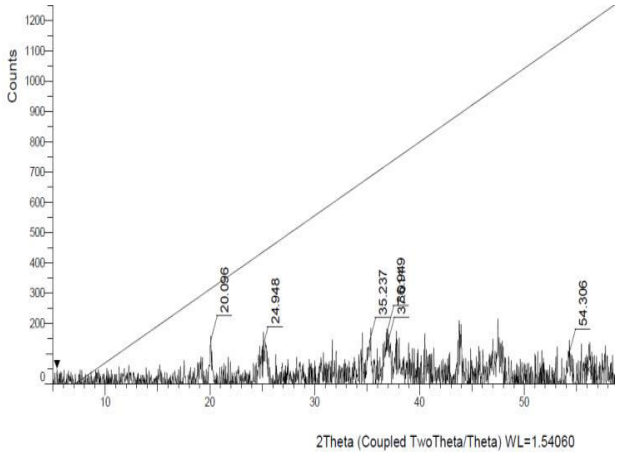

Fig. 3: XRD pattern of cobalt tartrate crystals

The analysis of different diffraction peaks indicates the formation of orthorhombic structure [31, 32]. The crystalline phases and d-values obtained from the XRD have been compared with the JCPDS data from X'Pert High Score PANalytical software. Fig.4 shows the plot identified phases of cobalt tartrate hydrate ($C_4H_4CoO_6$ $2.5H_2O$) crystal with the JCPDS files 00-023-0187. The grain size is determined by measuring the width of the line with each intensity peaks and then the average grain size have been obtained from the XRD pattern using the Scherrer's calculator from X'Pert High Score software. [33] The obtained average grain size of cobalt tartrate hydrate crystals is 16.16 nm. The values of 2θ, d-values, intensity ratio and their corresponding crystallite size in nanometer are shown in the Table 2.

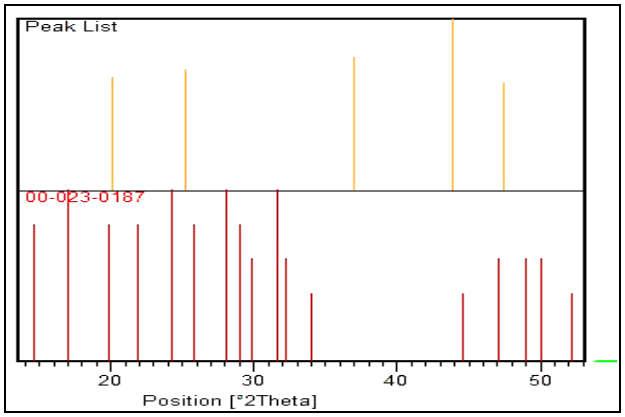

Fig. 4: Plot identified phases of $C_4H_4Co\ O_6$ 2.5 H_2O crystal by X'Pert High Score PANalytical Software

Table 2. Powder X-ray diffraction data of ($C_4H_4CoO_6 2.5H_2O$) crystals (λ =1.54060Å)

Observed data values			Standard data values			Crystallite size nm	Matched by X'Pert HighScore PANalytical Software
2θ	d-value	Intensity	2θ	d-value	Rel. Intensity		
20.096	4.41509	147	20.1115	4.41529	66.39	22.8	00-023-0187
24.948	3.56631	110	25.2281	3.53021	70.74	8.6	00-023-0187
36.949	2.43089	179	36.9653	2.43185	77.67	17.7	00-023-0187
37.011	2.42694	142	43.8438	2.06496	100.00	24.2	00-023-0187
54.306	1.68790	103	47.4938	1.91285	63.07	7.5	00-023-0187

3.2 FTI Ranalysis of cobalt tartrate crystals

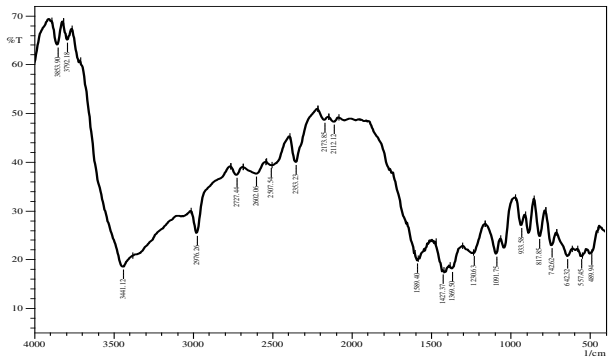

Fig.5: FTIR spectrum of ($C_4H_4CoO_6$) crystals (pH 4.2)

The Fig.5 shows the FTIR spectrum of the cobalt tartrate crystals. The FTIR spectrum was recorded by using Shimadzu FTIR-8400, Japan (400cm^{-1} to 4000cm^{-1}). The absorption peaks positioned in between 3853.90 - 3441.12cm^{-1} and 2976.26 - 2112.12cm^{-1} corresponds to symmetric and asymmetric stretching vibrations of O–H bond due to water of crystallization. The peak at 1589.40cm^{-1} is the band corresponding to carbonyl C=OH stretching vibrations. The peak at 1427.37cm^{-1} is the band corresponding to OH in plane bending. The band at 1369.50cm^{-1} is the band corresponding C–O stretching vibration and OH in plane bending. The band at 1230.63 - 1091.75cm^{-1} is the band corresponding C–O stretching vibrations. The band at 933.58 - 817.85cm^{-1} is the bands corresponding to C-H bending (out of plane) and O-H bending (out of plane).The band at 742.62cm^{-1} is the bands corresponding to C-H bending. The band at 642.32cm^{-1} is the bands corresponding to C-H bending and O-H bending.

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The band observed at 557.45 - 489.94cm^{-1} corresponds to metal oxygen bonding [34-37]. Table 3shows the FTIR spectral analysis of cobalt tartrate crystal.

Table 3. FTIR spectral analysis of ($C_4H_4CoO_6$) crystals (pH 4.2)

Observed data values			Standard data values			Crystallite size nm	Matched by X'Pert HighScore PANalytical Software
2θ	d-value	Intensity	2θ	d-value	Rel. Intensity		
20.096	4.41509	147	20.1115	4.41529	66.39	22.8	00-023-0187
24.948	3.56631	110	25.2281	3.53021	70.74	8.6	00-023-0187
36.949	2.43089	179	36.9653	2.43185	77.67	17.7	00-023-0187
37.011	2.42694	142	43.8438	2.06496	100.00	24.2	00-023-0187
54.306	1.68790	103	47.4938	1.91285	63.07	7.5	00-023-0187

3.3 SEM analysis of cobalt tartrate crystals

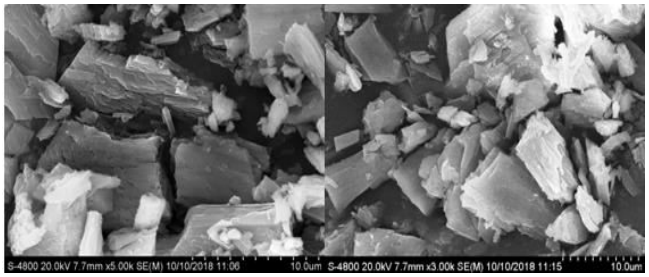

Fig. 6: Shows the SEM images of cobalt tartrate crystals with two different magnifications

Fig.6 shows the SEM images of cobalt tartrate crystals with two different magnifications. The powder sample of cobalt tartrate crystals were examined by using FESEM Hitachi S8400 instrument. The scanning electron microscopy reveals that the crystals having flat, rectangular and or thorhombic shapes of different sizes and structures embedded in rock like structures.

3.4 EDAX Analysis of Cobalt Tartrate Crystals

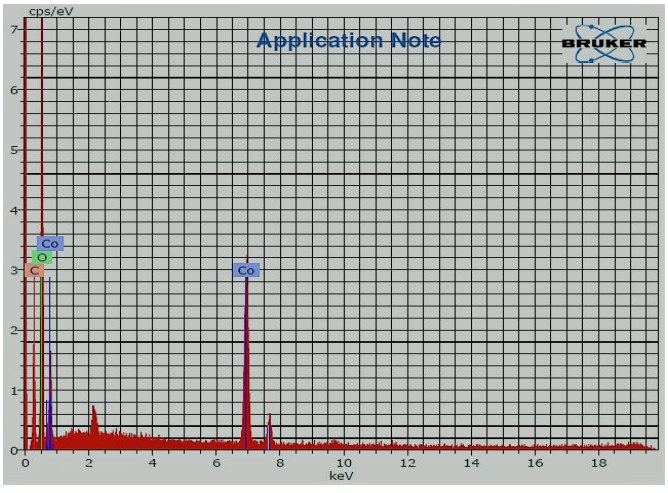

Fig.7: Shows the EDAX pattern of cobalt tartrate crystals

Fig.7 Shows the EDAX pattern of cobalt tartrate crystals. The elemental analysis of gel grown cobalt tartrate crystals was carried out by using X-Flash -60 Bruker instrument. Cobalt tartrate is one of the most complex systems with many phases and unique chemical composition as it is amplified between silica gel by the distribution of cobalt chloride in the gel. The diversity in the stoichiometry of cobalt tartrate poses a challenge for the control of size and shape. The energy dispersive x-ray spectrum has shown that the Co and O are present in the atomic percentage of 10.81 and 56.99 and weight percentage of 32.92 and 47.10respectively. The analysis of EDAX has confirmed the hydrous nature of the compound. That is, cobalt tartrate with dehydrate ($CoC_4H_4O_6.2H_2O$) may be formed.

3.5 UV-visible study of Cobalt Tartrate Crystals

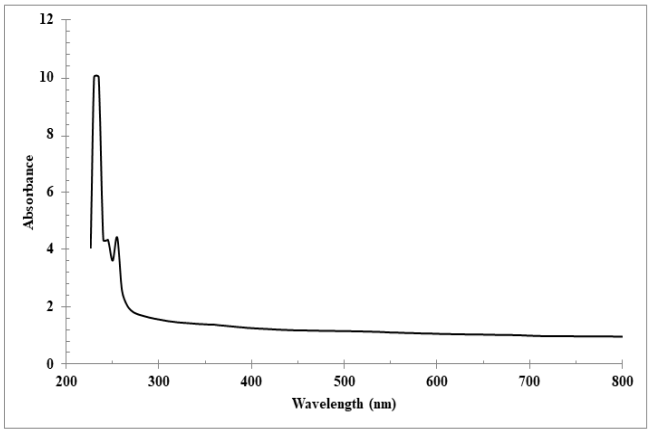

Fig.8: UV-visible absorption spectrum of cobalt tartrate crystal

Fig.8 shows the UV-visible absorption spectrum of cobalt tartrate crystal. The UV-visible spectrum of cobalt tartrate was recorded in the spectral range of 200-800nm by using Agilent Cary-60 UV-Vis Spectrophotometer (Agilent Technologies India Pvt. Ltd, New Delhi). For the optical applications, the material considered must be transparent in the wavelength region. In case of cobalt

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tartrate the absorption coefficient is high at lower wavelength region and wide transparency is high at higher wavelength region suggesting their suitability for second and third harmonic generations of the radiation [38-40]. The value of energy gap was calculated from UV-visible absorption spectra. The maximum absorption was observed at 234.98nm. The energy gap of the material was calculated by using the formula,

$$E = \frac{hc}{\lambda}$$

Where,
E-is energy band gap, h-is Plank's constant, c-is velocity of light and λ-is the wavelength

$$\therefore E = \frac{6.62607015 \times 10^{-34}\,\text{Joule.sec} \times 300000\ \text{km/s}}{234.9821777\,\text{nm}}$$

$$\therefore E = \frac{1.98782 \times 10^{-28}}{234.9821777}$$

$$\therefore E = 8.45945 \times 10^{-31}\,\text{eV}$$

$$\therefore E = 8.45\ \text{eV}$$

Therefore, the energy gap of the cobalt tartrate crystal is 8.45 eV.

The band gap energy of the cobalt tartrate crystals are calculated with the obtained wavelength using the following simple conversion equation.

$$\text{Band gap energy (eV)} = \frac{1240}{\text{Wavelength (nm)}}$$

$$\therefore \text{Band gap energy(eV)} = \frac{1240}{234.9821777}$$

$$\therefore \text{Band gap energy(eV)} = 5.27699594$$
$$\therefore \text{Band gap energy(eV)} = 5.27\ \text{eV}$$

Therefore, the band gap energy of the cobalt tartrate crystal is 5.27 eV [41]. Table 4 shows energy gap and band gap energy of cobalt tartrate crystal.

Table 4. Energy gap and Band gap energy of cobalt tartrate crystal

Crystal	λ (nm)	Energy gap (eV)	Band gap Energy (eV)
Cobalt tartrate	234.982177	8.45	5.27

3.6 DSC analysis of cobalt tartrate crystals

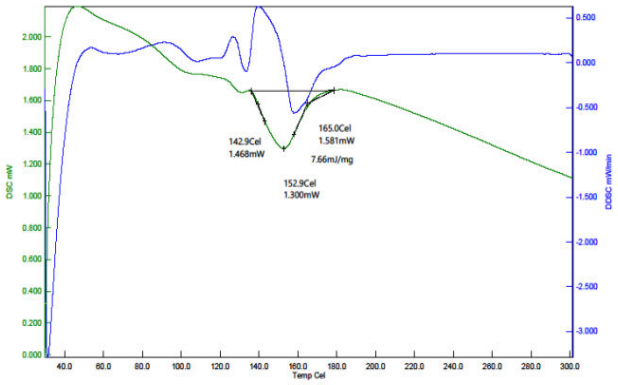

Fig. 9: DSC and DDSC curves of cobalt tartrate crystals

The fig. 9 shows DSC and DDSC curves of cobalt tartrate crystals. This Fig. illustrates the thermal stability of this sample, the heat flow (milli Watt) with the temperature in Celsius and the type (ΔH) exothermic or endothermic as well as the thermal transition. Since the instrument cannot go beyond 300℃, the sample was heated in between 30℃ to 300 ℃ in the nitrogen atmosphere using DSC- 60, Shimadzu, Japan instrument. The initial weight of the sample was 2.700mg and the heating rate was maintained at 20℃/min. The Table 5 shows complete endothermic peak recorded. The one stage of DSC curve under study is as follows,

Stage-I: The initial temperature is 142.9 ℃, the phase change is complete at peak end-down temperature 152.9℃. The temperature at which the sample and the reference come to thermal equilibrium by thermal diffusion, the peak appeared in the DSC curve at 165.0℃ indicates the phase transformation due to the loss of water molecules and formation of stable anhydrous $C_4H_4CoO_6$ crystals.

Stage-II: The heat area under the curve is 7.66mJ/mg.

Table 5. DSC data of $C_4H_4CoO_6$ crystals

Peaks	Temperature	On set	End set	Heat
Endothermic	142.9℃	152.9℃	165.0℃	7.66 mJ/mg

4. Conclusions

Gel method is found to be suitable for growing the cobalt tartrate crystals. Single diffusion method is convenient for the growth of these crystals. Nucleation control can be achieved by changing a various gel parameters such as pH of gel, density of gel and concentration of feed solutions. The good quality circular shaped, opaque and brown coloured crystals were observed. The crystalline phases and d-values obtained from the XRD have been compared with the JCPDS data from X'Pert High score PANalytical software. It indicates that unit cell parameter values and d values match very well

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with the JCPDS data and the sample consisted of cobalt tartrate hydrate ($C_4H_4CoO_6 2.5H_2O$) crystalline having orthorhombic structure. The particle size is determined as 16.16 nm. From the FTIR spectroscopic study, the presence of O–H, C=OH, C–O, C–H and metal–oxygen bonds were confirmed. The SEM study reveals that the crystals having flat, rectangular and orthorhombic shapes of different sizes and structures embedded in rock like structure. The analysis of EDAX has shown hydrous nature of the cobalt tartrate crystal with the presence of Cobalt and Oxygen. The value of energy gap 8.45 eV and band gap energy 5.27 eV were deduced from UV-visible absorption spectra which confirms the semiconducting nature of the crystal. DSC was taken to find the thermal properties of the crystal which showed the water of hydration in the crystal.

Acknowledgments

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