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Growth and Characterization of Cobalt Oxalate Crystal by Ager-Ager Gel Method

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Abstract

We have grown the cobalt oxalate crystals by adopting single diffusion technique via agar-agar gel. The tendency of cobalt oxalate crystals to form splices, twins, spherulites and dendrites was demonstrated. The growth dynamic of cobalt oxalate was studied by controlling the parameters like, concentration of gel, concentration of reactants, aging period and reversing of reactants. Physical properties of the grown crystals were analyzed by XRD, and FTIR techniques and the results are discussed.

KEYWORDS: Gel, Crystal, Gel Growth, Crystal Growth, XRD, and FTIR

Introduction

Crystals grown by the gel method has gained interest in the research community because it is cheap and easy to grow single crystals of alkaline-earth metal oxalates[1] and transition metal oxalates [2]. These materials have interesting properties like low solubility in water [3], decomposition before freezing point [4], interesting optoelectronic properties. Their role in analytical chemistry and subsequently in industries [5, 6] has created an opportunity for the researcher to investigate every scientific aspect of these materials. Therefore, efforts are being made to investigate and study the physical and chemical properties of these materials. Recently, there are reports on the growth of mixed-ligand complex formation using cadmium oxalate [7]. In the present study, we have presented the optimization of growth parameters to grow the cobalt oxalate single crystals using the agar gel method.

Materials and Methods

Materials used to grow the cobalt oxalate crystals are cobalt chloride, oxalic acid, and agar-agar gel. All the chemicals used for the experiment were used without any further purification. Sodium silicate glass test-tubes were used as crystallizing vessels. The test-tubes were filled with the first reactant (cobalt chloride) of desired volume and morality. The second reactant, oxalic acid having a concentration range of 0.5 to 1.0 M, was poured along the walls of the test-tube into the set-gel, and allowed to diffuse into the gel medium. The open end of the tube was closed with cotton plugs and kept undisturbed. The said procedure was carried out at room temperature. The ions of the supernatant solution reacted with ions of the first reactant via capillaries formed in gel medium. After six to seven days, nucleation kick-started at the gel-solution interface. The chemical reaction that occurred between the two reactants is given as follows:

$$CoCl_2 + H_2C_2O_4 \longrightarrow CoC_2O_4 + 2HCL$$

The diamond-shaped opaque crystals were obtained in the test-tube. The crystals were harvested by washing them carefully with acetone and collected for further characterization. Table 1 shows the optimized crystal growth parameters for the cobalt oxalate crystals.

Sr.No	Condition Single Diffusion	Condition Single Diffusion
1	Percentage of gel	2.0 %
2	Concentration of cobalt chloride	1.0M
3	Concentration of oxalic acid	1.0M
4	Volume of cobalt chloride	5.0 ml
5	Volume of oxalic acid	15 ml
6	Gel setting period	34 Hours
7	Gel aging period	4 days

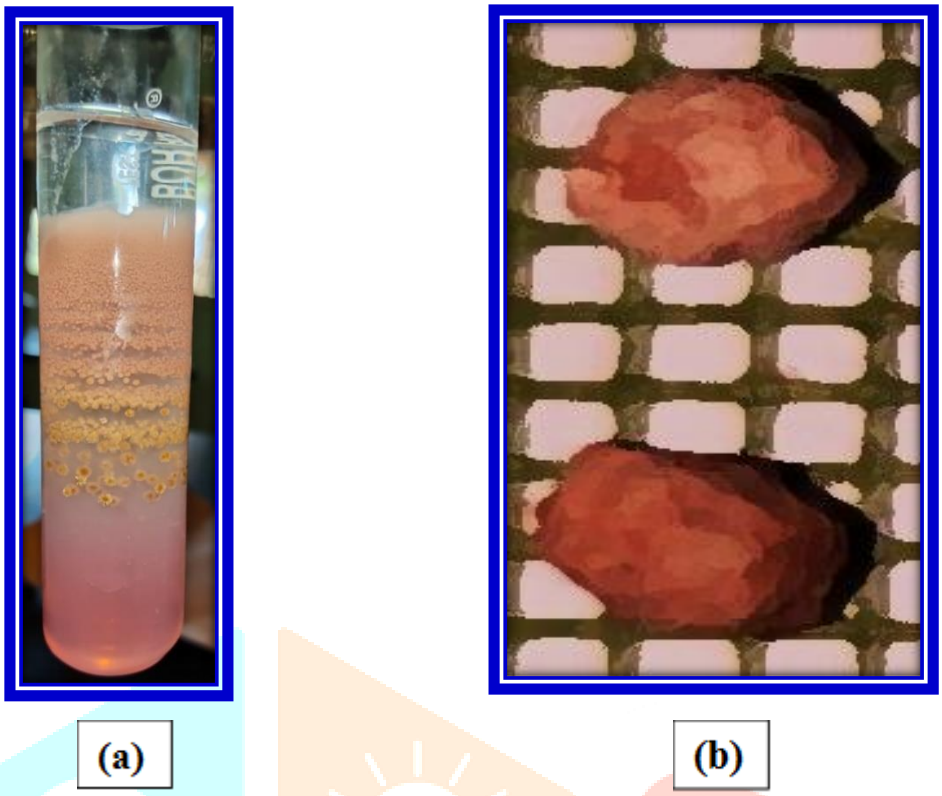

Figure 1:(a) in situo growth of Cobalt oxalate crystals in test-tube and (b) optical photograph of cobalt oxalate crystals

Result and discussion

The crystal structure analysis of the grown cobalt oxalate crystal was done via X-ray diffraction. X-ray pattern was recorded from the range of 10 to 80 degrees. The occurrence of highly resolved intense peaks at specific Bragg angles 2θ indicates the high crystallinity of the grown material and revealed monoclinic structure. The obtained crystal data has been compared with the JCPDS data and it closely matched with the reported JCPDS no. 037-0719. The unit cell parameters ('a' = 5.39820 Å, 'b' = 5.03100 Å, and 'c' = 5.73590Å) are close to the reported cell parameters of CoC2O4.2H2O, indicating the monoclinic phase of cobalt oxalate crystal. Comparative data is tabulated in Table 2 for the gel-grown cobalt oxalate crystal.

Table 2. Comparison of unit cell parameters of cobalt oxalate.

Parameters	Calculated	JCPDS data
System	Monoclinic (P)	Monoclinic
a	9·67638 Å	6·4534 Å
b	6·7156 Å	7·5009 Å
c	8·6822 Å	10·940 Å

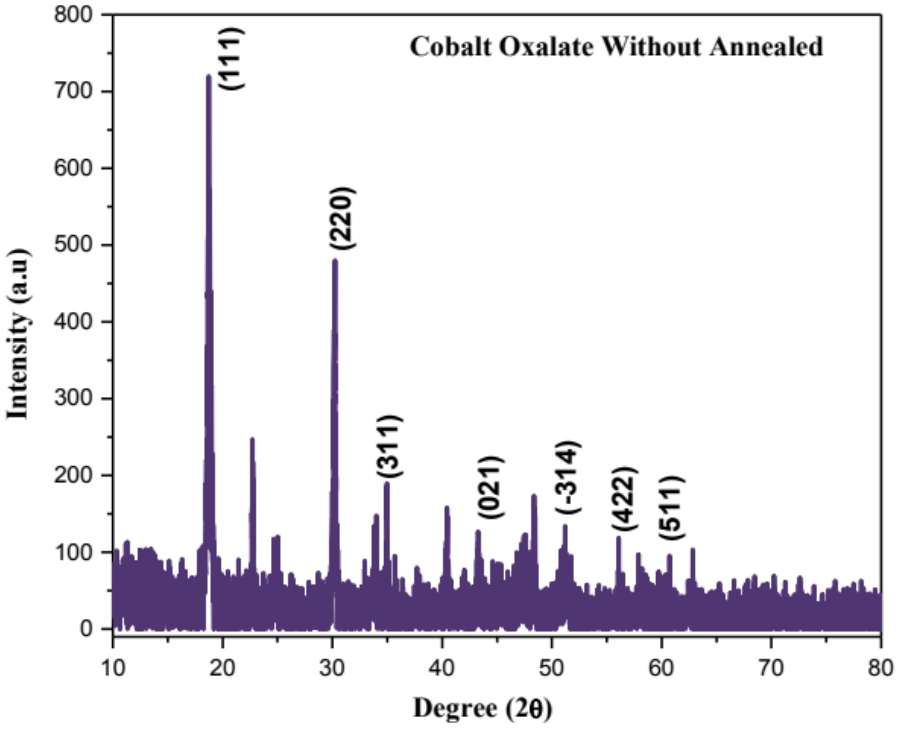

Figure 2: X-ray diffraction pattern of gel-grown cobalt oxalate crystal.

4.2 Fourier transform infrared (FTIR) Spectra

The Fourier transform infrared (FTIR) spectrum of cobalt oxalate was recorded at room temperature in the spectral range 500 – 4500cm^{-1} by KBr pellet method using SHIMADZU spectrophotometer at the department of Physics, Shivaji University Kolhapur. Figure 3 shows the FTIR spectrum of cobalt oxalate. The spectrum shows various frequencies of vibrational modes which confirm the presence of oxalate in the crystal.

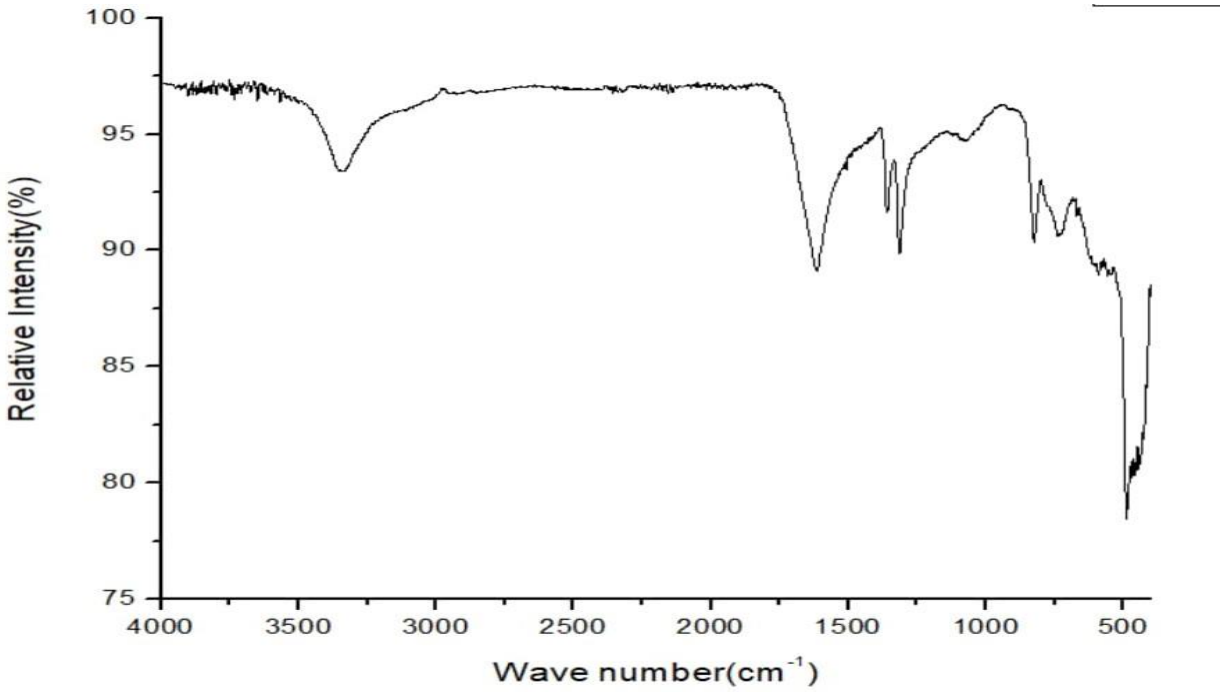

Figure 3: FTIR of Cobalt oxalate grown crystal

The sharp peak at 3300.18 $cm^{-1}$is attributed to the stretching of O−H group, indicating the presence of water of crystallization or water of hydration. However the peak at 1710. 00 cm^{-1} to 1665.00cm^{-1}correspond to α,β-unsaturated aldehydes, and ketones. The two identical sharp peaks around 1367.93 and 1327.43 cm^{-1} correspond to an asymmetric and symmetric stretch of C–H rock, respectively. Thus the FTIR spectroscopy confirmed the growth of cobalt oxalate crystals due to the presence of water of crystallization.

Conclusions

Cobalt oxalate crystals were grown by gel method using agar- agar gel in well size and shape. XRD powder diffraction patterns and analysis shows the crystalline nature of crystal with monoclinic phase. Different functional groups revealed by FTIR show the metal bond and different vibrations in the sample.

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