

Growth and Characterization of Gel Grown Cobalt Iodate Crystals

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Abstract. Cobalt iodate crystals have been grown by gel aided technique employing sodium metasilicate. The grown crystals were analyzed XRD, FESEM, EDAX and FTIR. X-ray diffraction studies of the powdered sample reveal the hexagonal structure. Fourier transform Infra-red studies confirm the formation of iodate group with water of crystallization, while energy dispersive X-ray analysis established the presence of cobalt, iodine and oxygen in the sample.

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1 Introduction

A search for new nonlinear optical materials among the transition metal iodates has led to grow and characterization of copper iodates. Gel growth technique has been used for the preparation of single crystals of alkaline earth metal iodate crystals. The gel medium prevents clutter and being chemically inert, it provides a three dimensional crucible which allows the reagents to diffuse at a required controlled rate. Its softness and uniform nature of constraining forces that it exerts upon the growing crystals encourages orderly growth [1,2].

Nonlinear optical phenomena have found vast applications in many areas of modern science, technology and engineering. The nonlinear devices find large applications in optical communication, image processing and wave-guide coupling [3].

In this paper we report a method of growing cobalt iodate crystals by controlled diffusion of chemical reaction in silica gel medium. The aim of the present work is to study the structural property of cobalt iodate crystals.

2 Experimental

In the present work, to grow the crystals of cobalt iodate, single diffusion method was used [4]. In actual procedure, 7cc, 2N acetic acid was taken in a small beaker, to which sodium meta silicate solution of density 1.04 gm/cc was added drop by drop with constant stirring by using magnetic stirrer, till pH of the solution reaches a value of 4.2. Continuous stirring process avoids excessive ion concentration which otherwise causes premature local gelling and makes the final medium inhomogeneous and turbid. To this mixture, 5cc of 0.1M cobalt nitrate solution (one of the reactants) was added with constant stirring. The resulting solution is allowed to set in test tubes of length 150 mm and internal diameter 15 mm. The mouth of test tube was covered by cotton plug to avoid contamination of the exposed surface with atmospheric impurities and to keep the gel at atmospheric conditions. Initially the mixture appeared to be quite translucent. However, with the lapse of time, its color changed and became milky white when the gel was completely set. At a pH of 4.2 gelling is completed within four days. Over the fully set gel, supernatant solution of 0.1M potassium iodate was slowly added. The nucleation starts as the anions of potassium iodate slowly diffused into the gel column containing cobalt nitrate and react together to form the expected crystals. The experiment was carried out at an ambient temperature of about 30°C. The fully grown crystals are shown in Figure 1. Then these crystals were removed from the crystallizing tube and washed in distilled water.

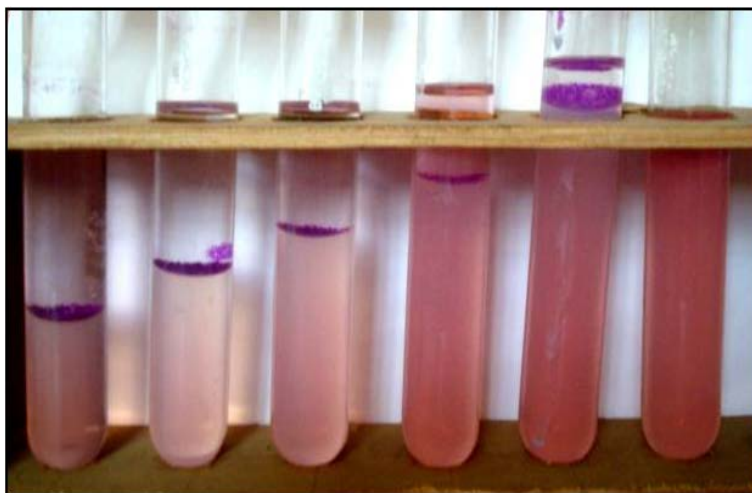
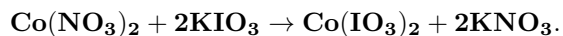


Figure 1. (Colored on-line) Growth of cobalt iodate crystals for different pH.



Figure 2. (Colored on-line) Few crystals of cobalt iodate.

Figure 2 shows a few violet color crystals of cobalt iodate. The following reaction is expected to take place in the gel medium.

3 Results and Discussion

3.1 X-Ray diffraction analysis

Cobalt iodate crystals were subjected to powder X-ray diffraction studies. The X-ray powder diffraction pattern of the grown crystals was obtained at room temperature on a Bruker Germany model no. D8 advance at School of chemical technology, N.M.U. Jalgaon. All diffraction patterns were obtained using $\text{CuK}\alpha$ radiation of wavelength 1.54060 \AA , at 40 kV and 40 mA. Measurements were made from $2\theta = 10^\circ$ to 80° with steps of 0.02° . The powder X-ray diffraction pattern of cobalt iodate crystals are shown in Figure 3. The well defined, sharp peaks in the XRD patterns signify the good crystalline and single phase nature of the cobalt iodate crystals. [5-7]. The recorded XRD peaks (black color) almost matching with the peaks (red color) of JCPDS data [8] for the cobalt iodate crystals and indicates that the crystal belongs to hexagonal crystal system with unit cell parameters: $a = 10.954 \text{ \AA}$ and $c = 5.0723 \text{ \AA}$.

3.2 Field emission scanning electron microscopy

Surface morphological study of the cobalt iodate crystals was carried out using Field Emission Scanning Electron Microscope make Hitachi Model S-4800. The FE-SEM micrographs of cobalt iodate crystals are shown in Figure 4. The

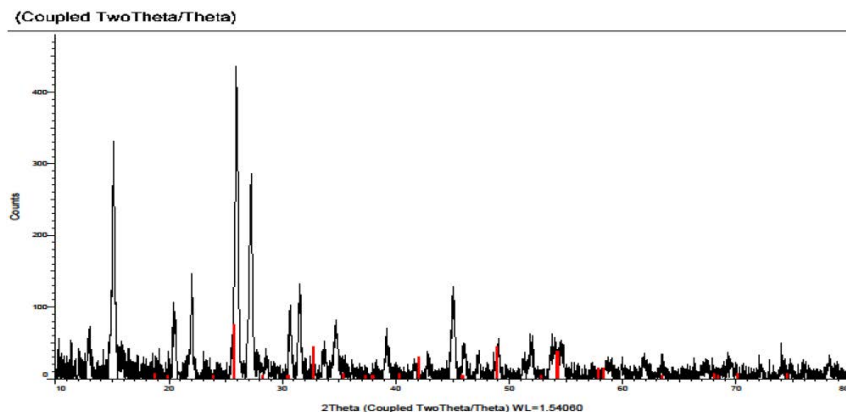


Figure 3. (Colored on-line) XRD pattern of the crystals of cobalt iodate.

FESEM images of the crystal reveal the well formation of the faces of the crystal and indicates that particles of powder were hexagonal platelets.

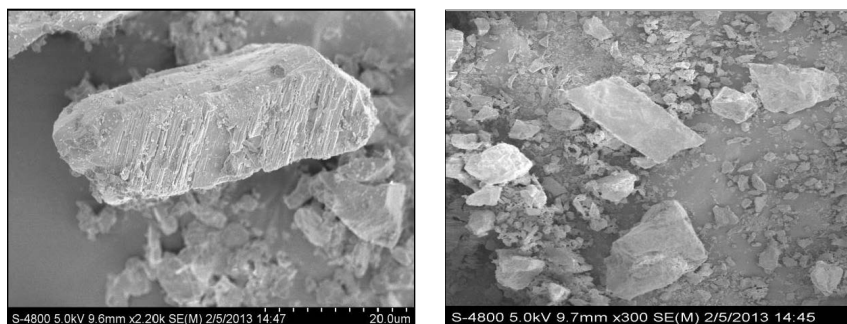


Figure 4. (Colored on-line) FE-SEM micrographs of cobalt iodate crystal.

3.3 Energy dispersive X-ray analysis

In order to confirm the presence of the elements of cobalt iodate crystals, the sample of grown crystals were subjected to Energy Dispersive X-ray analysis. Figure 5 shows the EDAX result of cobalt iodate crystals. EDAX pattern shows peaks corresponding to all the major elements (Cobalt, Iodine and Oxygen) present in the grown crystal. The experimental and theoretical calculated atomic and weight percentage of elements is given in the Table 1.

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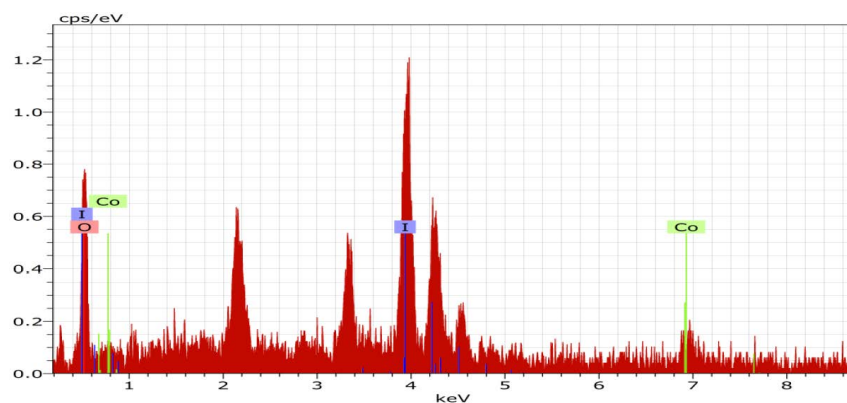


Figure 5. (Colored on-line) EDAX spectrum of cobalt iodate.

Table 1. Experimental and theoretical calculated composition obtained from EDAX of various constituent elements cobalt iodate crystal

Elements	Experimental values		Theoretical values	
	Mass [%]	Atomic [%]	Mass [%]	Atomic [%]
I	68.24	26.21	62.10	22.22
Co	12.45	10.65	14.42	11.11
O	19.31	63.14	23.49	66.66

3.4 FT-IR spectral analysis

The FTIR spectra of cobalt iodate crystals were recorded on SHIMADZU model no. 8400 transmission spectrophotometer with KBr pellet method over the wave number range $400\text{--}4000\text{ cm}^{-1}$ at Institute of Chemical Technology, N.M.U. Jal-

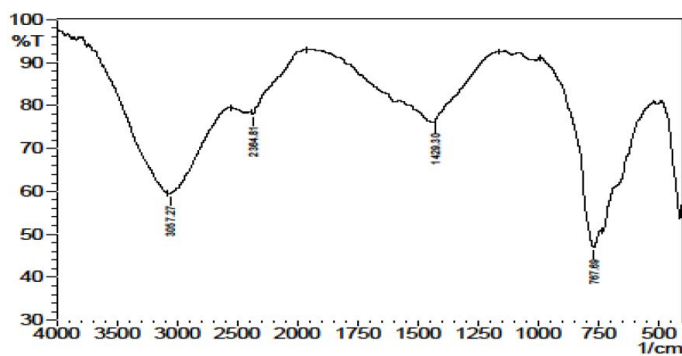


Figure 6. FTIR Spectrum of Cobalt iodate crystal.

gaon. The results of FTIR of cobalt iodate are shown in Figure 6. The FTIR spectrum of cobalt iodate shows the band at 2364.81 cm^{-1} may be due to CO_2 atmosphere. The stretching band in the region of $830\text{--}500\text{ cm}^{-1}$ indicates presence of iodate and metal oxide [9-11]. A band at 767.69 cm^{-1} is due to I—O bond. FTIR spectrum does not show band around 3500 cm^{-1} which indicates that cobalt iodate contains no water of crystallization.

4 Conclusion

From the above studies we conclude that, crystals of cobalt iodate have been successfully grown by single diffusion silica gel method. XRD studies reveal the hexagonal structure and the crystalline perfection of the grown crystal. The FESEM images show hexagonal platelets. The presence of major elements and functional group in the grown crystals was ascertained using EDAX technique and FTIR spectral analysis respectively.

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