International Journal of Multidisciplinary Research and Development

IJMRD 2014; 1(8): 241-243
www.allsubjectjournal.com
Received: 19-11-2014
Accepted: 07-12-2014
e-ISSN: 2349-4182
p-ISSN: 2349-5979

H. O. Jethva
Department of Physics, Maharaja Shree Mahendrasinhji Science College, Morbi, Gujarat, India.

Correspondence:
H. O. Jethva
Department of Physics, Maharaja Shree Mahendrasinhji Science College, Morbi, Gujarat, India.

FTIR Spectroscopic and XRD study of cobalt levo-tartrate crystals

H. O. Jethva

Abstract
Several metallic tartrates deserve special attention due to their various applications. In the present study, cobalt levo-tartrate crystals were grown using silica hydro-gel as growth medium. Spherulitic reddish crystals were obtained. The crystals were characterized by FTIR spectroscopy and powder XRD. The FTIR spectrum revealed the presence of O-H, C-H, C-O and C=O functional groups. The powder XRD study suggested that the crystals belong to orthorhombic system.

Keywords: Cobalt levo-tartrate crystals, gel growth, FTIR, Powder XRD.

1. Introduction
The growth of pure and mixed crystals in gel medium has attracted the attention of many investigators [1–11]. The principle of growth is based on the slow migration of crystal constituents through silica gel so that a very slow reaction occurs with the formation of a sparingly soluble compound. When the concentration of this compound exceeds the solubility limits, crystals will be formed. The main function of the gel is to control the flow of reacting ions. Most of the tartrate compounds are insoluble in water and decompose before melting. Hence, single crystals of such type of compounds cannot be grown by either slow evaporation or melt technique. In this situation gel method is the appropriate one for their growth. Gel acts as a 3 dimensional crucible which supports the growing crystal and at the same time yields to its growth without exerting major forces on it. In the present article, the pure crystals of cobalt levo-tartrate were grown by gel method and characterized by FTIR spectroscopic and powder XRD.

2. Materials and Method
In the present study, the silica hydro gel was used as a growth medium. To prepare the gel, a solution of sodium metasilicate of 1.05 specific gravity and 1 M solution of levo tartaric acid were mixed in such a manner that the pH of the mixture was set at 4.5. The gel solution was poured in to glass test tubes of 15 cm length and 2.5 cm diameter and allowed to set in the gel form. The supernatant solution containing cobalt nitrate hexahydrate solutions was poured on the set gel carefully without damaging the gel.
All the chemicals were of AR grade and obtained from Sigma Aldrich. The following reaction is expected to take place in the formation of cobalt levo-tartrate crystals.

$$Co(NO3)2{\cdot}6H2O + H2C4H4O6 + nH2O \rightarrow CoC4H4O6{\cdot}\ nH2O + 2HNO3 + 6H2O \quad (1)$$

The amount of HNO3 produced is very less in comparison to the nutrients being supplied to the growing crystals and hence no major limitation is imposed [6-11].
Spherulitic reddish crystals with dimensions of nearly 2 mm to 3 mm can be grown in a period of 3 to 4 weeks. Photograph of the grown crystals is shown in the figure 1.

Fig 1: Spherulitic crystals of cobalt levo-tartrate

3. Characterization Technique

The grown crystals were characterized by FTIR and powder XRD. The FTIR spectrum was recorded on Perkin Elmer Spectrum GX spectrophotometer in the range from 400 – 4000 cm^{-1}in KBr medium. The powder XRD pattern was recorded on Philips X'pert MPD by using Cu K_α radiation (1.5406 Å) and the data were analyzed by software powder-X.

4. Results & Discussion

4.1 FTIR Study

Infrared spectroscopy is useful for the identification of the structure of molecule. The FTIR spectrum for a particular chemical compound is a unique characteristic of that compound alone. Reflecting as it does the geometry, bond strength and atomic masses of the substance. There are reports available in literature on FTIR study on pure and mixed metal tartrate systems [6-11]. Infrared spectroscope of gel grown cobalt levo-tartrate sample is shown in figure 2.

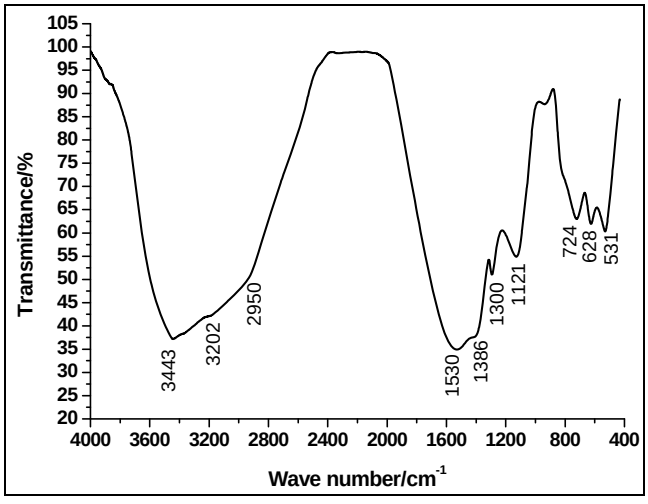

Fig 2: FTIR spectrum for cobalt levo-tartrate crystals

The IR spectrum absorptions at 3443 cm^{-1} and 3202 cm^{-1} are due to free O-H stretching and water of crystallization, which confirms the hydrous nature of the compound. Band at 2950 cm^{-1} is assigned to asymmetrical C-H stretching vibrations. A band at 1530 cm^{-1} is attributed to C=O stretching vibrations. The absorptions at 1386 cm^{-1} and 1300 cm^{-1} are due to C-H banding of alkane. The band at 1121 cm^{-1} is due to C-O stretching vibrations. The absorption at 724 cm^{-1} is assigned to C-C banding vibrations. The absorption band found between 530-630 cm^{-1} are due to the metal-oxygen bonding vibrations.

4.2 Powder XRD Study

There are reports available in literature on powder XRD study on pure and mixed metal tartrate systems [6-11]. The powder XRD pattern for gel grown cobalt levo-tartrate crystals is shown in figure 3.

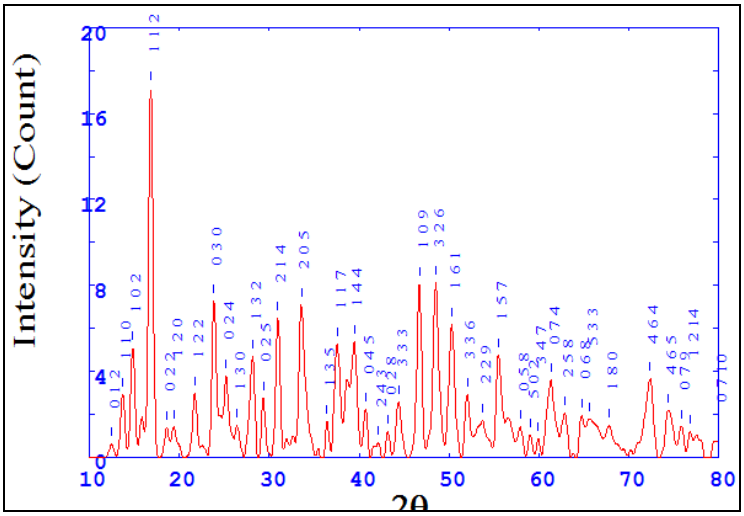

Fig 3: Powder XRD pattern for cobalt levo-tartrate crystals

The crystallinity of the crystal is quite clear from the diffractogram because of the occurrence of sharp peaks at specific Bragg's angles [12]. Each peak is indexed by its related hkl parameters. Calculation of cell parameters reveals that the crystal belongs to orthorhombic crystal system having space group $P2_12_12_1$. The unit cell dimensions of the cobalt levo-tartrate crystals are found to be a = 7.95 Å; b = 11.24 Å; c = 18.050 Å, V = 1612.91 $Å^3$ and the interaxial angles are $\alpha = \beta = \gamma = 90^\circ$. These values are in good agreement with the reported values [13]. The orthorhombicity is calculated by using the formula [b – a / b + a] × 100, where a and b are the lattice parameters. The value orthorhombicity is estimated to be 17.14. The X-ray density (d_x) is calculated by the relation, $d_x = 8M/N_a$ abc, where, M = Molecular weight, N_a = Avogadro number and abc = cell volume. The value of X-ray density was estimated to be 2.090 gm/cm^3 for cobalt levo-tartrate crystals. The grain size (i.e. the diameter of the crystal particle in the material) is determined by measuring the width of the line with highest intensity peak. The grain size can be calculated by using the formula

Grain size $D = 0.9\,\lambda / \beta_{1/2} \cos\theta$
$= 0.9 \times 1.54178$ Å $/ 0.4647 \times \cos(16.854)^\circ$
$= 1.387602 / 0.007758235$
$= 170.85$ Å
$= 17.085$ nm

where, $\beta_{1/2}$ is full width of half maxima (FWHM) in radian.

5. Conclusion

Pure cobalt levo-tartrate crystals were grown in silica gel by using 1 M cobalt nitrate solution as supernatant solution. The FTIR spectrum of the grown crystals indicated the presence of O-H, C-H, C-O, C=O functional groups with metal-oxygen

vibrations. The powder XRD suggested the orthorhombic crystal structure. The orthorhombicity and grain size of the crystals is calculated and found to be 17.14 and 17.085 nm, respectively.

6. Acknowledgments
The author (Harshkant Jethva) is thankful to the Principal and the Management of Maharaja Shree Mahendrasinhji Science College, Morbi for encouragements.

7. References

1. Henisch HK. Crystal growth in gels. University Park, Pennsylvania State University Press, 1970.
2. Garud SL, Saraf KB. Growth and study of mixed crystals of Ca–Cd iodate. Bull Mater Sci 2008; 31(4):639–43.
3. Girase KD, Girase ND, Patil TK, Bhavasr DS. Crystal growth and photo luminescent property of the lead iodate $pb(IO3)_2$. Asian J Chem Env Res 2010; 3(4):67–9.
4. Patil TK, Saraf KB. Growth and study of gel grown crystals of bismuth iodate Bi(IO3)3. Asian J Chem Env Res 2010; 3(4):78–83.
5. Patil HM, Sawant DK, Patil JH, Bhavsar DS. Spherulitic growth of neodymium tartrate crystals in silica gel. J Sci Rev 2010; 2(2):108–13.
6. Jethva HO, Parsania MV. Growth and characterization of lead tartrate crystals. Asian J Chem 2010; 22(8):6317.
7. Joshi SJ, Tank KP, Parekh BB, Joshi MJ. Characterization of gel grown iron-manganese-cobalt ternary levo tartrate crystals. Cryst Res Technol 2010; 45:303.
8. Joshi SJ, Tank KP, Parekh BB, Joshi MJ. FTIR and thermal studies of iron-nickel-manganese ternary levo tartrate crystals. J Therm Anal Calorim, 2012.
9. Jethva HO, Vyas PM, Tank KP, Joshi MJ. FTIR and thermal studies of gel grown lead-cadmium mixed levo tartrate crystals. J Therm Anal Calorim, 2014.
10. Jethva HO, Joshi MJ. FTIR and thermal studies of gel grown lead-iron mixed levo tartrate crystals. IJEIT 2014; 4(1):121-127.
11. Jethva HO, Joshi MJ. FTIR and thermal studies of gel grown lead-cobalt mixed levo tartrate crystals. IJIRSET 2014; 3(9):16517-16526.
12. Jesu Rethianam F, Arivu Oli D, Ramasamy S, Ramasamy P. Growth and characterisation of pure and nickel-doped strontium tartrate tetrahydrate single crystals. Cryst Res Technol 2006; 28(6):861-865.
13. Ariponnammal S, Srinivasan T. Growth and spectroscopic characterization of cobalt tartrate crystals. Res J Recent 2014; 3:63-66.