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Growth and Characterization of Lead Tartrate Crystals

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Lead tartrate crystals were grown using silica gel as the growth medium. Lead acetate was taken as the supernatant solution. The grown crystals were characterized by Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction analysis (XRD). The FT-IR spectra were recorded in 4000-400 cm^{-1} range. FT-IR spectra reveals the presence of water molecules, O-H bond, C-O and carbonyl (C=O) bonds. XRD reveals the crystalline nature and its various planes of reflection.

Key Words: Gel growth, Lead tartrate, FT-IR spectra.

INTRODUCTION

Tartrate crystals are of considerable interest, particularly for basic studies of some of their interesting physical properties[1-4]. Some crystals of this family are ferroelectric, some of others are piezoelectric and quite a few of them have been used for controlling laser emission. As tartrates are sparingly soluble in water and decompose before melting, the gel method is found to be more promising than the high temperature crystal growth methods. The growth of single crystals of calcium tartrate was reported[5]. Thermal behaviour of gel grown tartrates of yttrium and samarium was also reported[6].

Lead tartrate is orthorhombic with lattice parameters a = 7.99, b = 8.84 and c = 8.35. In the present study we have grown lead tertrate crystals using lead acetate as the supernatant solution. The grown crystals have been characterized by FTIR and XRD.

EXPERIMENTAL

The test tube diffusion method[7] was employed to grow lead tartrate crystals in the gel medium. 1 M sodium metasilicate ($Na_2SiO_3 \cdot 9H_2O$) was titrated with 1 M tartaric acid till the mixture attains the pH of 5.0-5.2. This gelling mixture was allowed to set in glass tubes of length 200 mm and diameter 25 mm. The gel was set in *ca.* 48 h. After a gel aging for 24 h, the supernatant solution was added over the set gel. The supernatant solution was lead acetate. The expected chemical reaction was

$$(CH_3CO_2)_2Pb \cdot 3H_2O + C_4H_6O_6 \rightarrow PbC_4H_4O_6 \cdot 3H_2O + 2CH_3COOH$$

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Small crystals appeared down the gel-solution interface in about 2 days and large crystals appeared down the gel column within a week. The crystals were harvested after a month. In the present investigation, the growth of lead tartrate crystals by gel method and its characterization by using XRD and FTIR are reported.

RESULTS AND DISCUSSION

The FTIR spectrum of the grown crystals was recorded in the wave number range 4000-400 cm^{-1}. Fig. 1 shows the spectrum of the grown crystals.

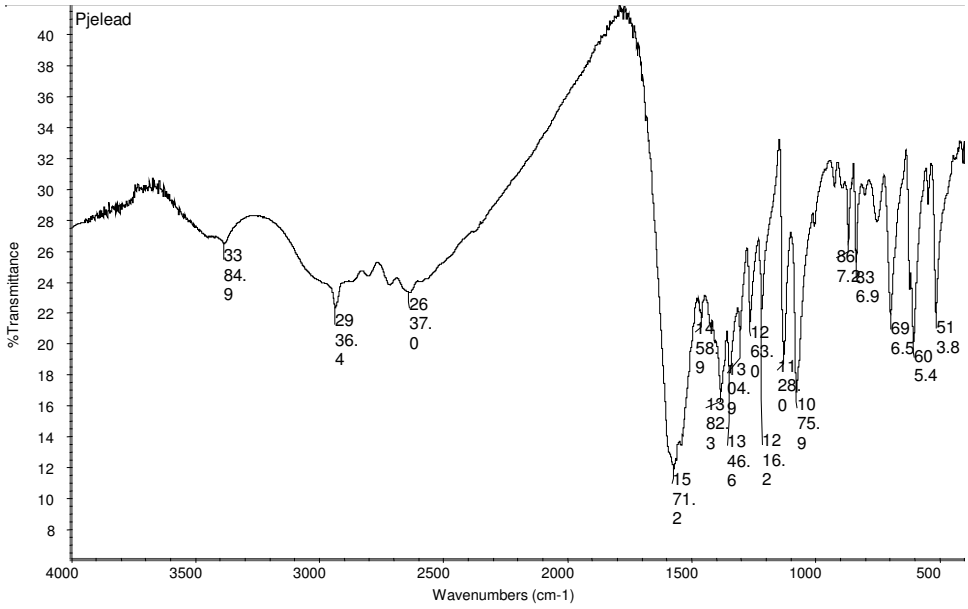

Fig. 1

TABLE-1
FREQUENCY (cm^{-1}) ASSIGNMENT FOR LEAD TARTRATE

Band (cm^{-1})	Assignment
3384	O-H stretching (alcoholic)
2637	O-H stretching (water)
2936	C-H stretching (alkane)
1571	C=O stretching (-COO-)
1382	C-H bending mode (alkane)
1128-1075	C-O stretching of (-COO-)
900-513	Pb-O mode

The peak at 3384 and 2637 cm^{-1} are due to OH stretching mode of alcoholic OH and OH (w.b.) (water), respectively. The band at 2936 cm^{-1} is attributed to the C-H stretching. The peak at 1571 cm^{-1} is assigned to C=O stretch of carbonyl group. The

peak at 1382 cm^{-1} is assigned to bending mode of alkane. The peak at 1128-1075 cm^{-1} is due to C-O stretching of (-COO-). The absorption between 900-513 cm^{-1} is due to formation of metal-oxygen (Pb-O) bond. Thus, FTIR spectrum revealed the presence of water molecules, O-H bond, C-O and carbonyl (C=O) bonds. The frequencies assignment is given in Table-1.

The grown crystals have been characterized by powder X-ray diffractometer. Fig. 2 represents the powder X-ray pattern of the grown lead tartrate crystals.

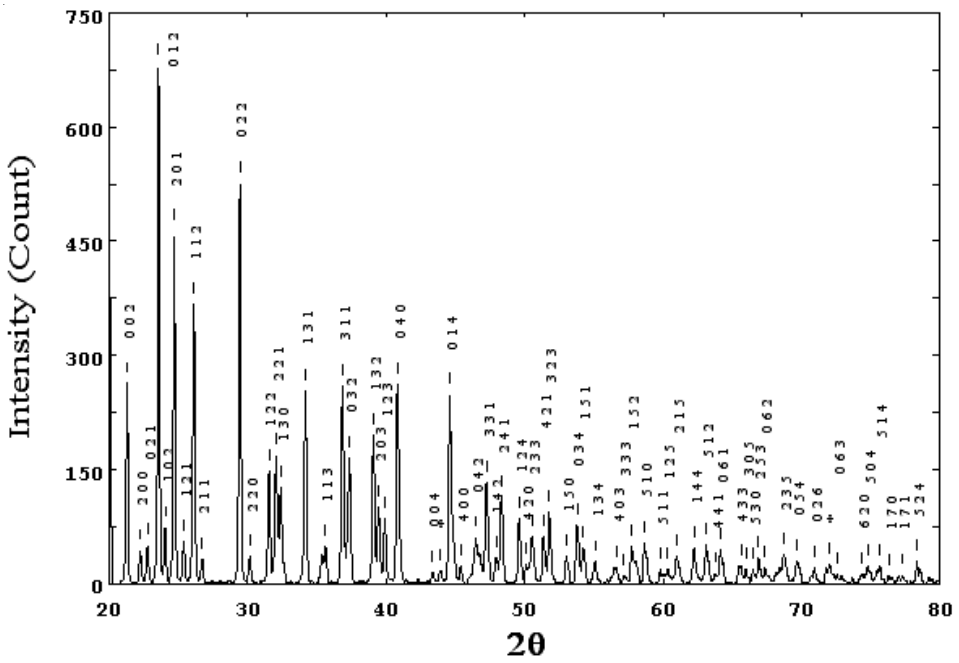

Fig. 2

TABLE-2
X-RAY DIFFRACTION RESULTS FOR LEAD TARTRATE CRYSTALS

2θ	d (Au)	Intensity	h	k	l
23.575	3.77370	680.94	0	1	2
29.446	3.03326	526.56	0	2	2
24.719	3.60154	463.31	2	0	1
26.129	3.41035	367.13	1	1	2
21.310	4.16939	262.80	0	0	2
34.182	2.62308	253.49	1	3	1
39.084	2.30460	195.12	1	3	2
32.063	2.79140	168.32	2	2	1
39.456	2.28373	100.61	2	0	3

The lattice parameter values of lead tartrate taken from the literature were used for the simulation of hkl values and corresponding d-values have been calculated. Table-2 shows X-ray diffraction results for lead tartrate crystals.

Conclusion

Lead tartrate crystals were grown in silica gel with lead acetate as supernatant solution. The grown crystals were characterized by FTIR spectroscopic and XRD. The FTIR spectrum of the grown crystals revealed the presence of O-H, C-O and C=O bonds. The presence of water molecules was detected. The XRD pattern confirmed the material of the grown crystal to be lead tartrate and the lattice parameters.

REFERENCES

1. V.S. Yadav and V.M. Padmanabhan, *Acta Crystallogr. B*, **29**, 493 (1973).
2. C.C. Desai and A.H. Patel, *J. Mater. Sci. Lett*., **6**, 1066 (1987).
3. H.B. Gon, *J. Cryst. Growth*, **102**, 501 (1990).
4. M.M. Abdel-Kader, F. El-Kabbany, S. Taha, M. Abosehly, K.K. Tahoon and A.A. El-Sharkawy, *J. Phys. Chem. Solids*, **52**, 655 (1991).
5. H.K. Henisch, J.I. Hanoka and J. Dennis, *J. Electrochem. Soc.*, **112**, 627 (1965).
6. A. Jain, S. Bhat, S. Pandita, M.L. Kaul and P.N. Kotru, *Bull. Mater. Sci*., **20**, 1089 (1997).
7. H.K. Henisch, Crystal Growth in Gels (New York: Dover Publications Inc.), p. 20 (1996).

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