

Comparative X-ray Diffraction Studies on Gel Grown Crystals of Li-tartrate, Cu-tartrate and Mixed Li-Cu tartrate

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Abstract : Natural as well as Gel grown crystals plays an important role in modern technology development. Gel method for the growth of crystals which are insoluble or sparingly soluble in water is the best alternative for the growth of many crystals. Crystals grown by gel method are relatively perfect compared to the other methods. In the present investigation crystals of Li-tartrate, Cu-tartrate and Mixed crystals of Li-Cu tartrate have been grown by single diffusion gel technique. The x-ray diffraction studies of these crystals are carried out in this work. The x-ray diffraction studies on these crystals reveals that these crystals belongs to orthorhombic crystal structure system. The lattice parameters of these crystals are calculated and tabulated in this article.

IndexTerms – Gel method, XRD, Li-Cu tartrate.

I. INTRODUCTION

It is well established that there are extensive study on tartrate based crystal grown by gel technique, however, we have found that there are few reports on the lithium tartrate based crystal because of its chemical properties [1-4]. Therefore, in the present study, we have investigated the growth mechanism of lithium tartrate, copper tartrate and mixed lithium-copper tartrate crystals. All the three types of crystals were grown by gel method by using single diffusion techniques, the crystal growth procedures and various different parameters affecting the growth of the crystals are discussed. The present paper contains the comparative study of all crystals under investigation regarding their growth and X-ray diffraction study. All results obtained are put at a glance in present paper.

II. GROWTH OF CRYSTALS

The crystals of lithium tartrate, copper tartrate, and lithium-copper tartrate were grown by gel method by using single diffusion technique. Table 1 gives details regarding method and chemicals used, different habits of crystals obtained and their transparency etc. In the present work, we obtained semitransparent, shiny and star shaped lithium tartrate crystals. The copper tartrate crystals were of diamond shaped with bluish color, while the mixed lithium-copper tartrate crystals were whitish blue in color and having a cubic shape. The adopted single diffusion gel technique proved to be beneficial because of it only we successfully obtained well-shaped and good quality crystals. All the well-defined good quality crystals were found below 2 to 3 cm in the gel interface [5-8].

The optimum growth conditions for gel grown crystals established by varying the different parameters like pH of gel, gel setting time, gel density, room temperature etc. are reported in the Table 2 for the all these three crystals. The suitable value of gel density is found to be 1.04 gm/cm^3 and the pH value is 4 to 4.2.

Sr. No.	Type	Method	Chemical Used	Solvent	Quality	Size (mm)
1	Lithium Tartrate	Gel method using single diffusion techniques	Na_2SiO_3 , $\text{C}_4\text{H}_6\text{O}_6$ 2LiCl	Methanol or Ethanol	Opaque, Transparent Good	3 x 1 x 1
2	Copper tartrate	Gel method using single diffusion techniques	Na_2SiO_3 , $\text{C}_4\text{H}_6\text{O}_6$ CuCl_2	Distilled water	Opaque, bluish color	2.5 x 2 x 1
3	Lithium-copper Tartrate	Gel method using single diffusion techniques	Na_2SiO_3 , $\text{C}_4\text{H}_6\text{O}_6$ $\text{CuCl} + 2\text{LiCl}$	Methanol or Ethanol	Opaque, good	2 x 2 x 1.5

Table 1 Summary of lithium, copper and lithium-copper tartrate crystals grown by gel technique

Sr. No.	Parameter	Lithium tartrate	Copper tartrate	Copper – lithium tartrate
1	Concentration of tartaric acid	1M, 7ml	1 M, 7ml	1M, 5ml
2	pH of the mixture	4 to 4.2	4.2	3.8 to 4.2
3	Temperature	25 to 30°C	25 to 30°C	20 to 30°C
4	Gel setting time	120 hours	96 hours	96 hours
5	Density of sodium metasilicate solution	1.04 gm/cm ³	1.04 gm/cm ³	1.04 gm/cm ³
6	Period of growth	4 weeks	3-4 weeks	4 weeks
7	solvent	Ethanol or Methanol	Water	Ethanol or Methanol

Table 2 Optimum growth conditions for gel grown tartrate crystals

III. XRD ANALYSIS

The crystals of lithium tartrate, copper tartrate, and mix lithium – copper tartrate were characterized by XRD analysis. From these diffractogram, 'd' values were computed [9]. Figure 1 shows comparative X-ray diffraction pattern of lithium-copper tartrate crystals. From the XRD pattern it is noticed that the peaks obtained at 11.00, 14.80, 25.00, 31.95, and 43.35° are corresponds to the (020), (220), (221), (620),

and (312) lattice planes of the copper tartrate crystals, respectively. We also found (101), (021), and (102) lattice planes of lithium tartrate crystals at 2θ of 21.40, 28.81 and 36.51°, respectively. Moreover, apart from the individual lattice planes, we also obtained new peaks at 18.83, 23.11, 26.53, 34.51, 40.78, 48.19, 51.90 and 55.03 degree (2θ) exhibiting the planes (201), (141), (151), (171), (352), (313), (211) and (850), respectively, confirming the formation of lithium-copper tartrate crystals [10-11]. Calculated (hkl) and 'd' values indicate orthorhombic crystals structure of lithium-copper tartrate crystals and having lattice parameters of $a = 14.5289 \text{ \AA}$, $b = 20.8745 \text{ \AA}$ and $c = 6.1806 \text{ \AA}$ and volume of unit cell, $V = 1874.474 (\text{ \AA})^3$, which are summarized in Table 3[12]. The details of the interplaner distance corresponding to each plane is tabulated in Table 4,5 and 6.

Material	Chemical formula	System	Lattice parameters $a, b, c, \alpha, \beta, \gamma$	Volume ($\text{ \AA})^3$
Lithium tartrate	$\text{Li}_2\text{C}_4\text{H}_4\text{O}_6$	Orthorhombic	$a = 6.7942 \text{ \AA}$ $b = 7.9804 \text{ \AA}$ $c = 5.2386 \text{ \AA}$ $\alpha = \beta = \gamma = 90^\circ$	284.05
Copper tartrate	$\text{CuC}_4\text{H}_4\text{O}_6$	Orthorhombic	$a = 17.9058 \text{ \AA}$ $b = 16.0728 \text{ \AA}$ $c = 4.4232 \text{ \AA}$ $\alpha = \beta = \gamma = 90^\circ$	1272.97
Lithium-copper tartrate	$\text{LiCuC}_4\text{H}_4\text{O}_6$	Orthorhombic	$a = 14.5289 \text{ \AA}$ $b = 20.8745 \text{ \AA}$ $c = 6.1806 \text{ \AA}$ $\alpha = \beta = \gamma = 90^\circ$	1874.47

Table 3 Comparison of Lattice parameters

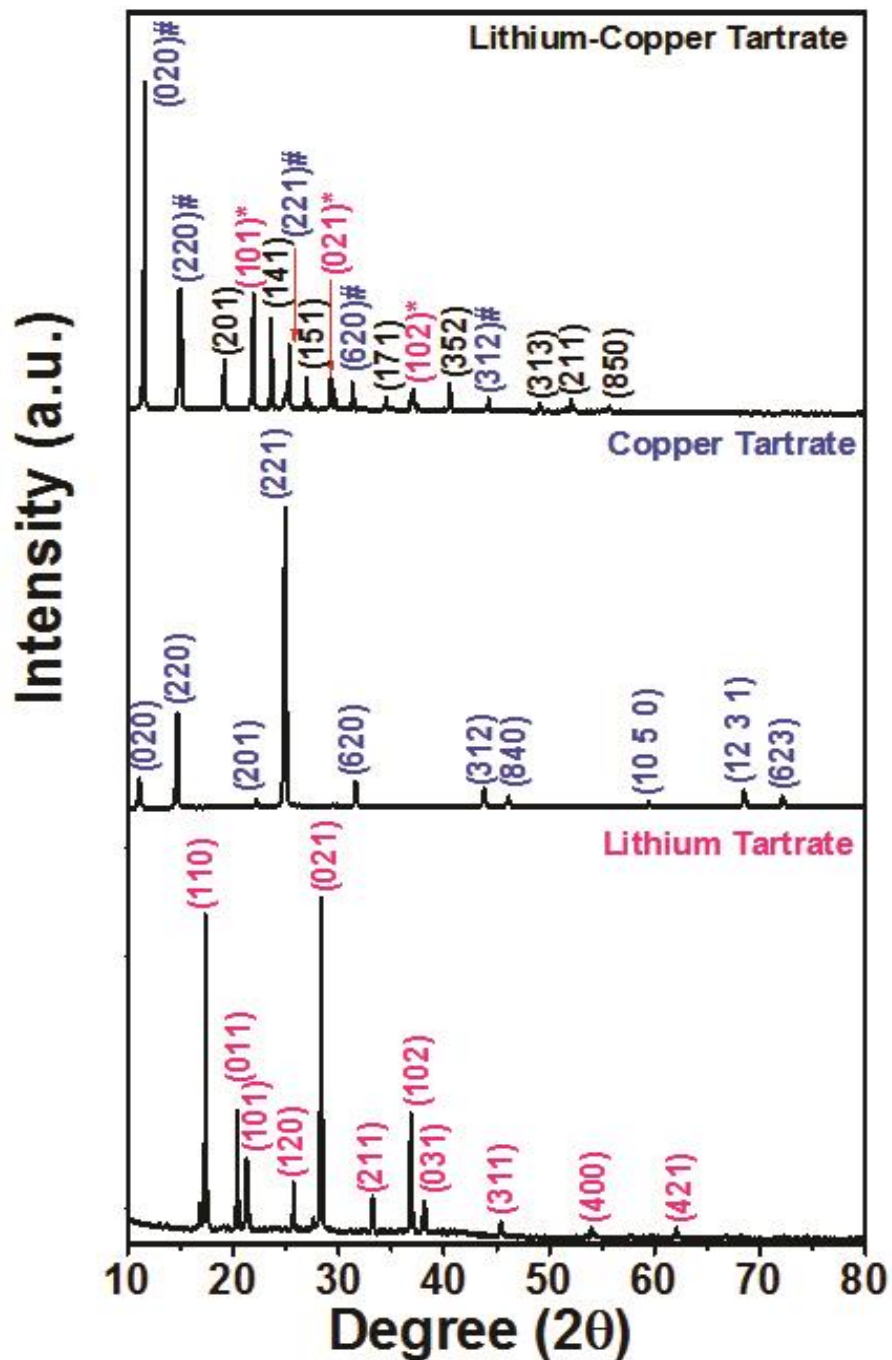


Figure 1 X-ray Diffraction patterns of Li-tartrate, Cu-tartrate and Mixed Li-Cu Tartrate crystals

Peak no.	d-Spacing		Indices (h k l)	2 θDegree	
	Obs.	Cal.		Obs.	Cal.
1	5.17	5.17	(1 1 0)	17.13	17.13
2	4.37	4.37	(0 1 1)	20.26	20.26
3	4.14	4.14	(1 0 1)	21.40	21.41

4	3.46	3.44	(1 2 0)	25.67	25.69
5	3.15	3.17	(0 2 1)	28.24	28.21
6	2.70	2.68	(2 1 1)	33.08	33.10
7	2.44	2.44	(1 0 2)	36.79	36.71
8	2.37	2.37	(0 3 1)	37.93	37.88
9	2.01	2.01	(3 1 1)	45.05	45.05
10	1.69	1.69	(4 0 0)	53.89	53.90
11	1.49	1.49	(4 2 1)	61.87	61.84

Table 4 Calculated and observed d-spacing of Li-tartrate

Peak	d-Spacing (Å)		Indices hkl	2θ Degree	
	Obs	Calc.		Obs	Calc.
1	8.0364	8.0364	(0 2 0)	11.00	11.00
2	5.9804	5.9804	(2 2 0)	14.80	14.80
3	3.9656	3.9656	(2 0 1)	22.40	22.40
4	3.5588	3.5562	(2 2 1)	25.00	25.02
5	2.7945	2.7976	(6 2 0)	32.00	31.96
6	2.0562	2.0567	(3 1 2)	44.00	43.99
7	1.9553	1.9553	(8 4 0)	46.40	46.40
8	1.5642	1.5643	(10 5 0)	59.00	59.00
9	1.3668	1.3671	(12 3 1)	68.60	68.59
10	1.3042	1.3043	(6 2 3)	72.40	72.39

Table 5 Calculated and observed d-spacing of Cu-tartrate

Peak	d-Spacing (Å)		Indices hkl	2θ Degree Obs
	Obs.	Cal.		
1	8.4767	8.4767	(0 2 0)	11.00
2	5.9624	5.9624	(2 2 0)	14.80
3	4.7074	4.7074	(2 0 1)	18.83
4	4.1486	4.1749	(1 0 1)	21.40
5	3.8453	3.8452	(1 4 1)	23.11
6	3.5842	3.5070	(221)	25.00

7	3.3569	3.3655	(1 5 1)	26.53
8	3.0962	3.0968	(0 2 1)	28.81
9	2.7945	2.7976	(6 2 0)	31.95
10	2.598	2.5601	(1 7 1)	34.51
11	2.4593	2.4598	(1 0 2)	36.51
12	2.2108	2.2101	(3 5 2)	40.78
13	2.0857	2.0853	(312)	43.35
14	1.8867	1.8880	(3 1 3)	48.19
15	1.7604	1.7601	(2 1 1)	51.90
16	1.6673	1.6654	(8 5 0)	55.03

Table 6 Calculated and observed d-spacing of Li- Cu-tartrate

CONCLUSIONS

The X-ray diffraction revealed the crystallinity of the grown crystals. The crystal structures of all three types of tartrate crystals were found to be orthorhombic. The volume of unit cell is of increasing order for Li, Cu and Li-Cu tartrate.

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