

Comparative Thermal Analysis Studies on Gel Grown Crystals of Li, Cu and Mixed Li-Cu tartrate

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Abstract: The 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 Thermal Analysis studies of these crystals are carried out in this work. The Thermal Analysis studies on these crystals.

Index Terms: Gel method, Thermal Analysis, Li, Cu and mixed tartrate.

I. INTRODUCTION

It is well established that there is 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 (Henisch H.,1970; Henisch H.,1986; Sawant D., et al., 2011; Patil H., et al.,2012). 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 Thermal Analysis study. All results obtained are put at a glance in present paper.

II. GROWTH OF CRYSTALS

In 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 (Krishnakumar V., et al.,2009; Sawant. D.,2012; Sonawane S.,2015; Ahmad N.,2014).

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 /cm³ and the pH value is 4 to 4.2.

III. THERMAL ANALYSIS

Thermal analysis is the measurement of how specific physical or chemical properties of a substance changes with temperature. It measures the change in weight of the substance with respect to applied temperature. In present work, thermogravimetric analysis of lithium tartrate, copper tartrate and mixed lithium-copper tartrate crystals was done. It was noticed that the pure lithium tartrate crystal was more stable at high temperature than the copper tartrate and mixed lithium-copper tartrate crystals. We observed 60 % weight loss in the temperature range of 200-212 °C for copper tartrate crystals, whereas for

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lithium tartrate crystal, weight loss is only about 28% in the same range of temperature. (Nandre S.,2013; Sawant D., et al.,2011; Yanes A., et al.,1996; Lopez T., et al.,1995). The details of weight loss with respect to temperature for all three the details of weight crystals is summarized in Table 3.1.

loss with respect to temperature for all three Similarly, for mixed lithium-copper crystals the total loss of weight was around 68 % in the temperature range of 0-310 °C.

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

Sr.No.	Type	Method	Chemicals Used	Solvent	Quality	Size (mm)
1	Lithium Tartrate	Gel method using single diffusion techniques	Na ₂ SiO ₃ , C ₄ H ₆ O ₆ LiCl	Methanol or Ethanol	Opaque, Transparent Good	3 x 1 x 1
2	Copper tartrate	Gel method using single diffusion techniques	Na ₂ SiO ₃ , C ₄ H ₆ 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 ₂ SiO ₃ , C ₄ H ₆ O ₆ CuCl + 2LiCl	Methanol or Ethanol	Opaque, good	2 x 2 x 1.5

Table 2. Optimum growth conditions for gel grown tartrate crystals

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	Water	Ethanol or Methanol

Table.3. Kinetic data of TGA analysis for lithium tartrate, copper tartrate and lithium-copper tartrate crystals.

Compound	Steps	Temp. range °C	Observed Weight loss %	Calculated weight loss %	Loss of Molecule
Lithium tartrate	I	25-95	18	18.18	2H ₂ O
	II	95-260	10	11.90	2H ₂ O
	III	260-750	15	15.50	2CO
	IV	750-950	30	32.10	2CO ₂
Copper tartrate	I	25-210	2.15	2.50	5H ₂ O
	II	210-240	65.80	66.15	2H ₂ O
Mixed Lithium-Copper Tartrate	I	30-100	18	19.8	3H ₂ O
	II	100-210	1.5	2.0	CO
	III	210-320	49.5	50.42	CO ₂ , 2H ₂ O

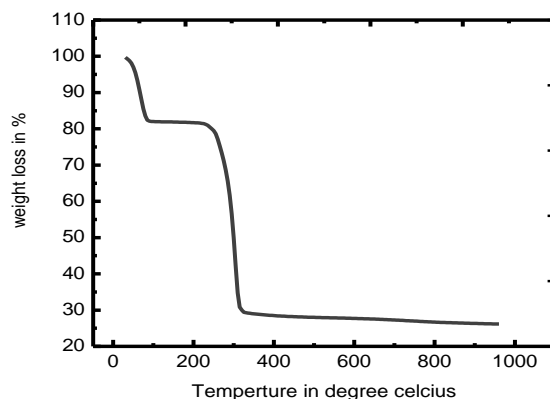
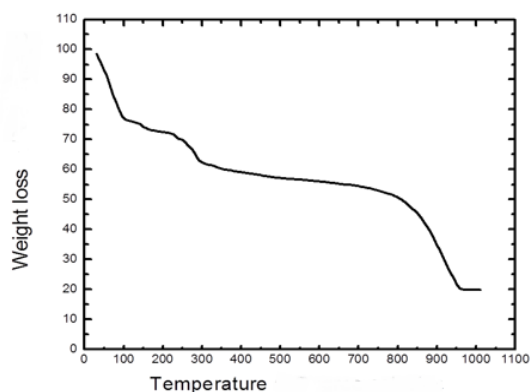


Fig. 1 TGA spectrum of lithium tartrate crystal.

Fig.2 TGA-DTA graph depicting the decomposition of lithium – copper tartrate crystal with respect to temperature

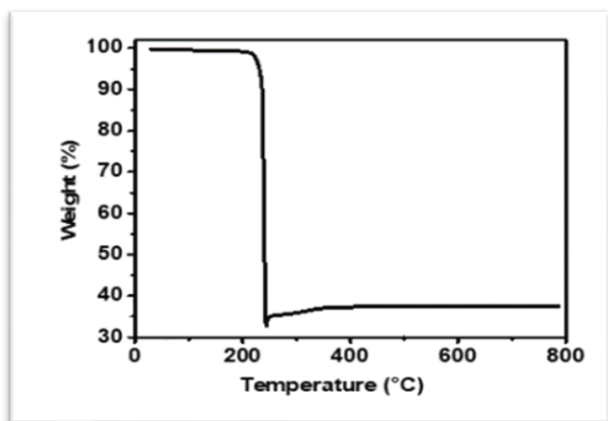


Fig. 3 TGA -DTA graph depicting the decomposition of lithium – copper tartrate crystal with respect to temperature

CONCLUSIONS

The Thermal Analysis revealed the crystallinity of the grown crystals. Thermal analysis is the measurement of how specific physical or chemical properties of a substance changes with temperature. It measures the change in weight of the substance with respect to applied temperature. In present work, thermogravimetric analysis of lithium tartrate, copper tartrate and mixed lithium-copper tartrate crystals was done.

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