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Experimental investigation of the structure, magnetic moment and decomposition process on heating in dipotassium tartarte crystals grown in chemical reaction gel method

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ABSTRACT

Single crystals of dipotassium tartrate were grown in gel by chemical reaction method. The various functional groups such as OH stretching, CH stretching, C—C modes, carbonyl modes and metal-oxygen stretching have been identified by using FTIR study. From the powder X-ray diffraction (XRD) studies, the unit cell dimensions and inter-axial angles were found to be $a = 9.8845$ Å, $b = 7.4420$ Å, $c = 8.8480$ Å; $\alpha = \beta = 90°$, $\gamma \neq 90°$. This reveals the monoclinic nature of the crystal. The magnetic susceptibility and magnetic moment of the grown crystals have been found to be 36.12×10^{-6} emu and 2.94 BM, respectively. The thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of the sample exhibit the decomposition process and the loss of weight during the process. The endothermic peak at 285.40 °C confirms the major stage of decomposition of the sample and there is no water of hydration.
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1. Introduction

A systematic study of crystallization in gels begins with Lissegong's famous discovery of periodic crystallization in gels. This method has gained considerable attention because of its simplicity and effectiveness in growing single crystal of certain compound. This method is an alternative technique to solution growth with controlled diffusion, free from convection and thermal strain. The various process parameters such as degree of saturation, type of solvent, pH of the gel medium, presence of impurities and the change in growth temperature also affect significantly the morphology of crystal.

Among various optical non-linear materials dipotassium tartrate ($K_2C_4H_4O_6$) has significant importance because of its high optical non-linearity [1–5]. Growth and characterization of various metallic tartrate crystals and mixed tartrate crystals using gel have been reported by several worker [6–12]. Crystal growth and characterization such as Laser Raman and Infrared spectra of solution grown dipotassium tartrate have been already done [13]. To the best of the authors knowledge, there is no report on gel growth by chemical reaction method and characterization of dipotassium tartrate.

2. Experimental method

Most of the work on crystal growth in gels has been done by the chemical reaction method. It has a special advantage of growing single crystals which are insoluble (or slightly soluble) in water and which decompose before reaching their melting point. The requirements to grow single crystals by this method are (i) the salts used here must be soluble in water and the product crystal must be relatively less soluble and (ii) the silica gel must be neutral and allow the reaction to take place between the two salt solutions. In this method, one of the salt solution (reactant (I)) is mixed with the gel and the gel is allowed to set. The other salt solution (reactant (II)) is introduced along the walls of the test tube over the set gel. This top salt solution goes through the pores of the gel and reacts with the salt solution which is already incorporated with the gel and thereby the product crystal of low solubility is grown. The process is highly controlled because the reactants combine due to the diffusion of ions through fine pores. The reaction can be represented as

$$\mathbf{aX} + \mathbf{bY} \rightarrow \mathbf{aY} + \mathbf{bX}$$

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aX is in the form of a solid, the gel surrounding it. The bY component is poured over the set gel. Controlled diffusion takes place in the gel media and the crystals are formed in the gel itself.

Crystals are grown by using single diffusion of salt solution through the gel medium. The equipment used for growing crystals consists of glass tube of 2.5 cm diameter and 14.0 cm length. The AR grade chemicals were used to grow the crystals. The gel was prepared by mixing 0.75 M L-tararic acid solution with sodium meta silicate ($Na_2SiO_3 \cdot 9H_2O$) solution of relative density 1.06 in such a manner that pH of the mixture could be maintained. This mixture has been poured into many glass test tubes. Depending on the gel pH and gel density, the gel sets at various timings. The following solutions were transferred gently on the set gels along the walls of the test tube:

Supernatant solutions:

$$1\,M, 5\,mlKCl + 1\,M, 5\,mlKNO_3 \quad (I)$$

$$1.5\,M, 5\,mlKCl + 1.5\,M, 5\,mlKNO_3 \quad (II)$$

$$2\,M, 5\,mlKCl + 2\,M, 5\,mlKNO_3 \quad (III)$$

The expected reaction to get the product crystal is shown below:

$$C_4H_6O_6 + KCl + KNO_3 \rightarrow K_2C_4H_4O_6 + HCl + HNO_3$$

The growth was completed within a month and transparent, monoclinic structured crystals were grown. The grown crystals were shown in Fig. 1. The various parameters which affect the formation of crystal are described in Table 1. The gel setting time is strongly dependent on pH as low pH gel takes more time to set than high pH gel.

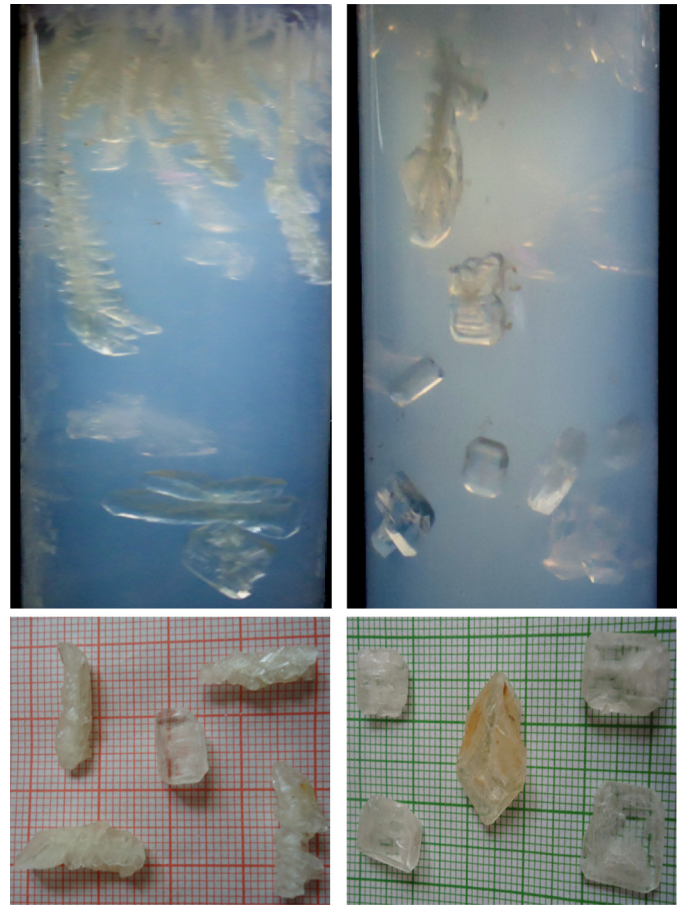

Fig. 1. Crystals of dipotassium tartrate grown in gel.

3. Results and discussion

3.1. FTIR spectroscopy

The FTIR spectrum of dipotassium tartrate crystals are shown in Fig. 2. The absorption bands for various vibrating molecules in coordination with their intensity are given in Table 2. Several authors have reported the vibrational spectra of metal tartrate crystals [14,15].

The peak observed at 3318.29 cm^{-1} is due to OH stretching mode. The band at 2975.85 cm^{-1} is attributed to CH stretching mode of tartaric acid. The bands at 1784.48 cm^{-1} and 1572.62 cm^{-1} that are unaffected by deuteration seem to be associated with γ^aCO and the band around 1415.93 cm^{-1} is due to γ^sCO. The absorption peaks at 1069.99 cm^{-1} is attributed to γC(OH). The two sharp peaks at 903.80 cm^{-1} and 878.64 cm^{-1} are due to γ_{CC} mode of vibrations. The band at 842.61 cm^{-1} is due to δCOO. The absorption peaks around 789.60 cm^{-1}, 680.21 cm^{-1} and 616.64 cm^{-1} are attributed to τCOO mode of vibrations. The absorption below 500 cm^{-1} are attributed to metal-oxygen stretching.

Table 2
FTIR spectrum assignments for dipotassium tartrate crystals.

Absorption in wavenumber (cm^{-1})	Assignments
3318.29	OH stretching
2975.85	CH stretching
1784.48	γ^aCO
1572.62	γ^aCO
1415.93	γ^sCO
1069.99	γC(OH)
903.80	γ_{CC}
878.64	γ_{CC}
842.61	δCOO
789.60	τCOO
680.21	τCOO
616.64	τCOO
Below 500	Metal–oxygen stretching

Table 1
Factors which determine the growth process of crystals.

S.No.	Gel density	Gel pH	Gel setting time (h)	Concentration of the reactants	Results
1	1.04	4.0	72	U.R.: 1 M, 5 ml KNO_3 + 1 M, 5 ml KCl L.R.: 0.5 M, $C_4H_6O_6$	Transparent crystals of dimensions 8 mm × 5 mm × 4 mm were obtained
2	1.05	4.5	48	U.R.: 1.5 M, 5 ml KNO_3 + 1.5 M, 5 ml KCl L.R.: 1.0 M, $C_4H_6O_6$	Transparent crystals of dimensions 12 mm × 7 mm × 5 mm were obtained
3	1.06	5.0	24	U.R.: 2 M, 5 ml KNO_3 + 2 M, 5 ml KCl L.R.: 1.5 M, $C_4H_6O_6$	Transparent crystals of dimensions 18 mm × 8 mm × 6 mm were obtained

U.R.: upper reactant L.R.: lower reactant.

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