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# Influence of Metal on Vibrational and Optical Properties of Mixed Tartrate Crystal in Silica Gel Medium

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## ABSTRACT

Single crystals of Potassium Strontium Tartrate (KSTAR) and Potassium Calcium Tartrate (KCTAR) were grown by single diffusion method at ambient temperature. Silica gel was used as the growth medium with test tube as crystallization vessels. The grown crystals were characterized by FTIR and UV-Visible spectroscopy. The FTIR analysis exhibited presence of varied bonds related to molecular structure of KSTAR crystals. UV-Vis-NIR transmission spectrum was recorded to study the optical transparency of the grown crystals. The metal which is present in the composition helps to enhance the size and transparency of the crystals. The growth conditions were optimized by varying the parameters such as pH, concentration of the gel, gel setting time and concentration of the reactants. The obtained results are deliberated in details.

**Key Words:** Silica gel medium, Single diffusion method, FTIR, UV-Vis-NIR

## INTRODUCTION

A series of pure and mixed crystals have been grown by several investigators with the aim of identifying new materials for practical and industrial purposes[1-5]. Single crystals are the pillar of the modern technological revolution. The impact of single crystal, is clearly visible in industries like semiconductors, optics etc. This type of crystal inventions of LASER, the field of the nonlinear optical properties and the practical implementations was possible with the applications of nonlinear optical crystal[6,7].

The gel technique is in fact a suitable method for the growth of crystals with low aqueous solubility and thermal decomposition behavior. A large variety of tartrate crystals have thus been grown by gel technique[8].Though the tartrate compounds are insoluble in water and decompose before melting. Hence single crystals of such compounds cannot be grown by either slow evaporation or melt technique. In this situation gel growth technique is the most appropriate one for its single crystals growth.

The aim of this work is to describe a detailed survey on the growth of alkaline($KCl_2$) with alkaline earth metals($SrCl_2$, $CaCl_2$), namely Potassium Strontium Tartrate(KSTAR), Potassium Calcium Tartrate(KCTAR) crystals. Mixed tartrate crystal using the gel method has been narrowly studied using the gel method have enough scope for preparing new materials. The gel method is effectively utilized to grow the KSTAR and KCTAR crystals. The vibrational and optical properties of these materials are analyzed. In the present investigation the influence of tartrate on alkaline and alkaline earth compounds have been elucidated.

## MATERIALS AND METHODS

Finely powdered Sodium Meta Silicate was dissolved in double distilled water. In the growth process, for a particular density of 1.08g/cm3 of Sodium Meta Silicate solution is taken in a test tube. Sodium meta silicate solution of desired can be prepared from the stock solution by using the following equation.

$$V_s d_s + V_w d_w = d_f V_f \qquad (1)$$

where,

$V_f$ is the final required volume (100cc) of gel solution of desired density

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$d_f$ is the final desired density of the gel solution (ie 1.04 g/cm$^3$)
$V_s$ is the volume of stock solution to be taken
$d_s$ is the density of stock solution
$V_w$ is the volume of the water to be added to prepare the desired density (ie 100-vs) cc
$d_w$ is the density of water (1.0g/ cm$^3$)

From the above equation

$$d_sV_s + d_wV_w = d_fV_f \quad (2)$$
$$d_sV_s + d_w(100-V_s) = d_fV_f \quad (3)$$
since ($V_w$ =100-$V_s$)

Volume of stock solution,

$$V_s = V_fd_f - 100d_w \quad (4)(d_s-d_w)$$

By adding 85 ML of tartaric acid into the solution, the pH of the gel set lies between 3 to 5. The test tube was kept in the undisturbed place for 48 hours. After the gel set, the desired top solution (0.5K.$Cl_2$ + 0.5Sr.$Cl_2$, 0.5K.$Cl_2$ + 0.5Ca.$Cl_2$) has been mixed into the stock solution. After a span of time, well grown white coloured crystals have been harvested. Optimum growth conditions were determined by varying gel concentration, pH, gel density, gel setting time, growth period and shape of the crystal with varying concentration of reactants. The effect of pH of the gel on the growth process and the quality of the grown crystals were examined by the varying the pH of the gel medium. Similar to the density of the gel, the pH variation also affects the transparency of the gel. It was found that advancement of the crystallization zone inversely varies with the pH of the gel.

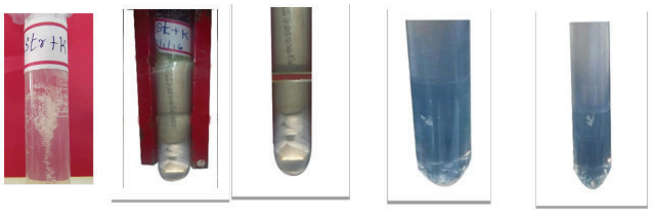

**Figure 1:** Growth system of KSTAR & KCTAR with stoichiometry.

**Table 1: Optimum conditions for the growth of mixed tartrate crystals**

| Various Parameters | KSTAR& KCTAR |
|---|---|
| Sodium Meta Silicate | 72 ML |
| Tartaric acid | 85 ML |
| Gel density | 1.04g/cc |
| Temperature | Room temperature |
| pH value | 4.7 |
| Concentration | 0.5 M |
| Gel setting time | 12 days |
| Growth period | 10 days |
| Inner reactant | Tartaric acid |
| Outer reactant Potassium chloride with | a)SrCl2 b)CaCl2 |

## RESULTS AND DISCUSSION

### Growth kinetics of crystals

Average temperature = 28°C

Growth apparatus = Single diffusion method [diameter =1.5 cm, length = 15 cm]

Inner electrolyte = O.5M/ltre Tartaric acid for both crystals

Upper electrolyte= Equal volumes of Potassium Chloride with

a) Strontium Chloride for KSTAR
b) Calcium Chloride for KCTAR

**Table 2: General properties of crystals**

| Order reacts | pH | General characteristics | Average size (mm) |
|---|---|---|---|
| 0.5% KCl2+ 0.5% SrCl2 | 4.7<br>4.5 | Small shining transparent crystals. Well developed facets. Quality improved. Size reduced. | 2.0<br>1.6 |
| 0.5% KCl2+<br>0.5% CaCl2 | 4.7<br>4.5 | Well developed facets. No interface growth<br>Good crystals seen at the bottom end | 2.5<br>2.2 |

### Vibrational analysis of KSTAR

The FTIR analysis is a technique that provides information about the chemical bonding or molecular structure of materials. The FTIR spectrum of the grown crystals is shown in Figure 3. The relations of the molecular group vibrations and the characteristic absorption bands were assigned according to the theories of infrared spectra. In the FTIR spectrum, the absorption bands in the region 3568-2600 cm$^{-1}$ are due to OH stretching mode, water and C-H stretch[9]. The band at 1593 cm$^{-1}$ is attributed to the C=O stretch of carbonyl group. The strong peak at 1390 cm$^{-1}$ is assigned to C=O symmetric and δ(O-C=O) mode. The peaks at 1329 cm$^{-1}$ is due to -OCH stretching mode. The peaks at 1060 and 965 cm$^{-1}$ are assigned to C-O stretching and C-C stretching modes. The peak at 712 cm$^{-1}$ is due to the deformation vibrations of $CO_2$. The peak at 533 cm$^{-1}$ is attributed to metal-oxygen bond. The presence of O-H bond, C-O bond, water of hydration and carbonyl C=O bond are established from the spectra.

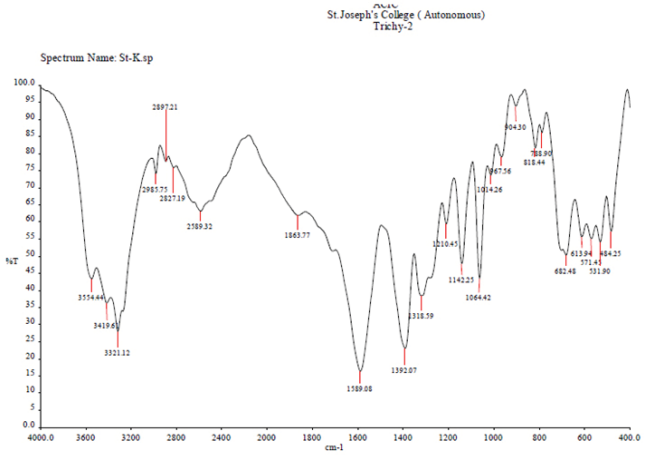


**Figure 2:** FTIR spectrum of KSTAR

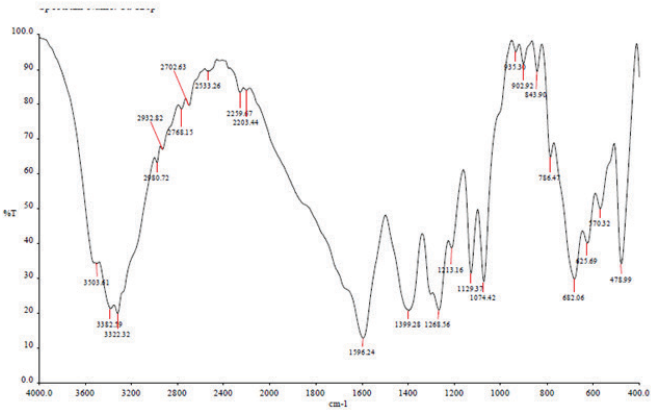


**Figure 3:** FTIR spectrum of KCTAR crystals.

**Table 3: Vibrational assignments of FTIR of KSTAR crystals**

| FT-IR value of parent compound (cm-1) [9] | FT-IR value of parent compound (cm-1) [12] | Present work (cm-1) | Assignment |
|---|---|---|---|
| 533 | - | 531.90 | Metal oxygen bond |
| 712 | - | 788.90 | Vibrations of Co2 |
| - | 968 | 967.56 | =C-H bonding |
| 1046 | 1134.19 | 1142.25 | C-O stretching |
| 1064 | 1072.46 | 1064.42 | C-O stretching |
| 1329 | - | 1318.59 | -OCH stretching mode |
| 1390 | 1392 | 1392.07 | C=O symmetric and δ(O-C=O) mode |
| 1593 | 1553.60 | 1589.08 | C=O stretch of carbonyl group |

## Vibrational analysis of KCTAR

The two strong peaks at 3571.32 cm-1 and 3423.76 $cm^{-1}$ are due to O – H stretching mode and water. The peak at 2987.84 cm-1 is due to the C- H stretching vibration. The band occurs at 1587.47cm–1 is due to the C=O stretching of carbonyl group. The strong peak at 1385.90 $cm^{-1}$is assigned to C=O symmetric stretching[10]. The peak at 1147.68$cm^{-1}$is attributed to C–H vibrational modes. The peak observed at 1282.71 $cm^{-1}$is assigned to OH plane bending[11]. The peaks of various intensities at 1061.85 $cm^{-1}$ and 1011.70 $cm^{-1}$are due to out of plane O–H deformation and C–O stretching. The absorption between 963.48$cm^{-1}$and 534.30 $cm^{-1}$is due to metal oxygen bonding (Ca-O)[12].

**Table 4: The assignments of FTIR bands of KCTAR crystals**

| FT-IR value of parent compound ($cm^{-1}$) [13] | FT-IR value of parent compound ($cm^{-1}$) [14] | Present work ($cm^{-1}$) | Assignment |
|---|---|---|---|
| 534.30 | - | 570.32 | Vibrations of Co2 |
| 963.48 | 1072.46 | 935.30 | -OCH stretching mode |
| 1011.70 | 1134.19 | 1074.42 | C=O symmetric and δ(O-C=O) mode |
| 1147.68 | - | 1129.37 | C-O stretch of carbonyl group |
| - | 1392 | 1213.16 | C-O stretching |
| 1282.71 | 1553.60 | 1268.56 | C-H stretching |
| 1385.90 | - | 1399.28 | C=O symmetric and δ(O-C=O) mode |
| 1587.47 | - | 1596.24 | C=O stretch of carbonyl group |
| - | 2961.61 | 2259.67 | C-H stretching |
| 3571.32 | - | 3503.61 | O-H stretching |

## Optical analysis of KSTAR

The UV spectrum was recorded in the spectral range of 190 - 1200nm. The UV-Visible transmittance spectrum is shown in Figure 4a.For optical applications, the material considered must be transparent in the wavelength region. The grown crystals were 96% transparent in the UV region. The value of energy gap was calculated from UV-Vis absorption spectra.

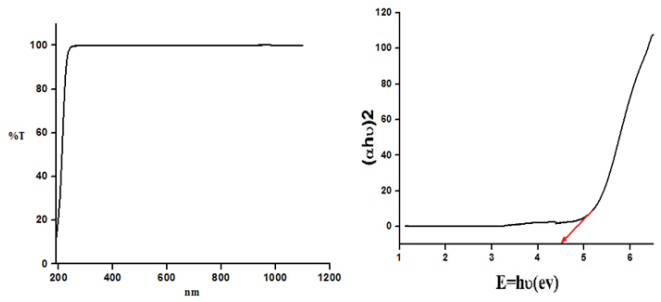


**Figure 4:** a) Optical transmission spectrum and b) band gap energy of KSTAR crystals

## Optical analysis of KCTAR

The UV-Visible study of KCTAR are carried out by varian, car 5000 model, UV visible spectrometer in the spectral range 190 nm to 1100 nm. Figure 5a shows UV transmission spectra of Potassium Calcium tartrate crystals. From the spectrum, it has been inferred that KCTAR crystals have sufficient transmission in the entire visible and IR region. The lower cut-off wavelength is 328.17 nm

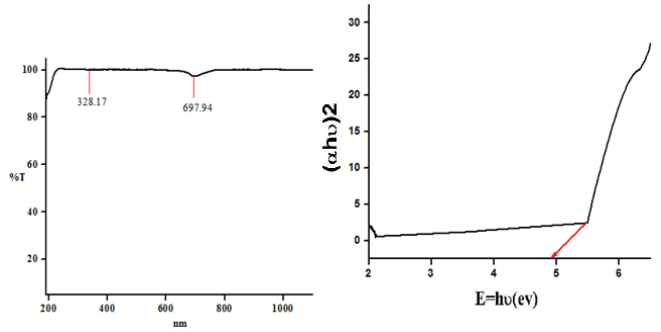


**Figure 5:** a) Optical transmission spectrum and b) band gap energy of KCTAR crystals

**Table 5: Band gap energy of KSTAR & KCTAR crystals**

| Sample name | Direct band gap energy (eV) | Literature review(eV) [15,16] |
|---|---|---|
| KSTAR | 4.5 | 4.055 |
| KCTAR | 4.9 | 5.13 |

## CONCLUSION

A well defined transparent single crystals were obtained by using gel technique and it is best suitable for growing crystals like KSTAR and KCTAR in silica gel used as the growth medium. Different habits of KSTAR & KCTAR have been analyzed by changing parameters like gel density, gel aging, pH of gel, concentration of reactants and the growth patterns of the crystals were improved by varying these parameters. The presence of various functional groups of the grown crystals have been identified by FTIR spectral analysis. Optical absorption studies were carried out using UV-Vis-NIR spectrophotometer and the optical band gap energy was verified with reported values and it plays a key role for technical related applications**.** The well grown KSTAR & KCTAR have received greater attention on account of their suitable growth, non linear optical and spectral characteristics.

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