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Archives of Physics Research, 2014, 5 (1):31-36
(http://scholarsresearchlibrary.com/archive.html)

Archives of Physics Research
Scholars Research Library
ISSN : 0976-0970
CODEN (USA): APRRC7

Structural and optical properties of copper doped barium tartarate crystals by silica gel technique

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ABSTRACT

Single crystals of Copper doped Barium Tartarate crystals (CBT) have synthesized by gel technique. Gel acts as a growth medium for the crystals. The crystalline nature of grown crystal was confirmed using powder X-ray diffraction techniques. The functional groups present in the crystals were identified using Fourier Transform Infrared spectral analysis.

Keywords: Gel technique, XRD, FTIR

INTRODUCTION

A simple and inexpensive method to grow single crystals is an alternative technique to a solution growth with controlled diffusion. This is purifying process, free from thermal strain [1-6].

Crystal habits of various crystals, grown under different conditions and also by different methods were described by Buckley [7], Hartman [8], Kern [9], Chernor [10], Burton [11] and Mullin [12]. A number of factors such as degree of saturation, type of solvent [13], pH of the gel media [14,15], presence of impurities [16] and the change in growth temperature also presumably affect significantly the morphology of the crystal [17]. The crystals, which can't satisfactorily grow from melt and vapor, are grown successfully by using this method [18,19,20].

Henisch explained the use of gel to grow the crystals [21].This technique has become more popular because of simplicity of the process [22,23], crystals having low solubility [24] and its unique advantage in terms of crystal product [25,26]. Many investigators have grown the single crystals of Tartrate compounds by gel method [27,28,29]. The rare earth Tartrates [30,31], the rare earth oxalates [32,33,34], the transition metal oxalates [35], alkaline earth metal [36], Barium Copper oxalates [37] and Barium oxalates were grown by using this method [38]. Now a day, this method has applied to the study the crystal formation in human system such as cholesterol stones [39,40], cholesterol monohydrates crystal in gel medium. In urinary stones, Calcium Hydrogen Phosphate Dehydrates (CHPD) crystals were found. These CHPD crystals were successfully synthesized by this gel technique [41]. Recently, the crystals of biological macromolecules were also grown by this technique.

In the present research work, single crystals of Cu-doped BaTr crystals were grown by a simple gel technique using diffusion method. The optimum growth conditions for crystals were determined. Optimum conditions were

established by varying various parameters such as pH, concentration gel solution, setting time of the gel solution and concentration of the reactance.

MATERIALS AND MEHTODS

The test tubes were used as crystallization vessels while silica gel as a growth media. The gel was prepared by using CH_3COOH (Acetic Acid) and Na_2SiO_3 (Sodium Metasilicate) having different pH values varying from 4.0 to 4.3. The chemicals used for growth of single crystal were CH_3COOH of 1M, Na_2SiO_3 and $BaCl_2$ (Barium Chloride) of 1M both, $CuCl_2$ (Copper Chloride) of 0.1M with AR grade. Different molar mass were tried to determine the optimum growth condition. Gel was prepared by mixing the solutions of acetic CH_3COOH, Na_2SiO_3, $BaCl_2$, $CuCl_2$, and transferred in glass tube of diameter 2.5cm and 15cm in length. The mouth of tube is covered by cotton plug and kept for the setting. After setting the gel, it was left for aging. After two days the supernatant Tartaric Acid of 1M concentration was poured slowly over the set gel by using pipette and kept undisturbed by covering the cotton plug on the mouth of tubes.

Experiments were carried out by changing different concentrations of the reactants. The chemical reaction inside the gel can be expressed as:

$2CH_3COOH + Na_2SiO_3 \rightarrow 2CH_3COONa\downarrow + SiO + H_2O$

$4CH_3COONa + BaCl_2 + CuCl2 \rightarrow (CH_3COO)_2\,BaCu\downarrow + 4NaCl$

$(CH_3COO)_4\,BaCu + C_4H_6O_6 \rightarrow C_4H_6BaCuO_6\downarrow + 4\,CH_3COOH$

Fig.1 shows transparent crystals of $C_4H_4O_6BaCu$ under synthesis are attached themselves and forming a thick layer at the interface while fig. 2 represents a few crystals of $C_4H_4O_6BaCu$ having different habit.

Fig1 doped crystals of $C_4H_4O_6CuBa$

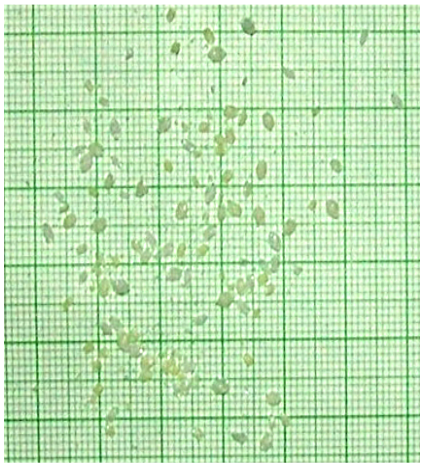

Fig 2 Few crystals of $C_4H_4O_6CuBa$

The parameters required for crystal growth was optimized and maintain constant throughout the investigation was as given in the table 1.

Table 1 Details of Experiment for the growth of $C_4H_4O_6CuBa$ crystals

Precursor used	Molarity	pH	Results
CH3COOH + Na_2SiO_3	1M + 1.04gm/c	4.0 to 4.3	For 4.3pH the semitransparent light pink Colures, platy shape and spherulitic crystals were observed in the interface and interstitial of the gel column. Size of the crystal 3.5mm×2.5mm× 2.5
$BaCl_2$+$CuCl_2$	1M +0.1M		
$C_4H_6O_6$ Tartaric acid	1M		

RESULTS AND DISCUSSION

The grown CBT crystals were subjected to the single crystals X-ray diffraction. The crystalline structure and lattice parameters are obtained from X Ray Difractometer (Bruker D-8) having wavelength of λ = 1.54060 Å at 40 KV and 40 mA. The sample was scanned over the required range for 2θ values (20^0-80^0). The crystalline phase of the sample was identified form crystallographic parameters such as 2θ, relative intensity and hkl values. The Fourier Transform Infra-Red spectrum was recorded for the powder sample in the range 450-4000 cm^{-1} using a Perkin Elmer Fourier transforms infrared spectrometer.

Powder X- Ray Diffraction Analysis

The crystal structure of the sample compound was studied by powder X-ray diffraction method. The recorded diffraction pattern of the $CuBaC_4H_4O_6$ crystals is shown in fig. 3.

The preferential orientation is found to be (220), (230), (013), (331), (350), (111), (200), and (2112) appears at 24.3°, 26°, 36°, 39.10°, 40.5°, 43.5°, 50.2° and 54.5° which matches with the standard JCPDS data card (26-0192 and 04-0836). The presence of large number of sharp peaks in the XRD spectrum confirms that crystalline nature having orthorhombic structure. The crystalline grains mainly oriented along the (013) plane. The crystal size evaluated as 60.98 nm by using the Debey-Scherrer formula.

$$d = \frac{0.9\,\lambda}{\beta \cos\theta} \qquad (1)$$

Where λ is the wavelength of X-Ray source, θ is the diffraction angle, β is Full width at half maximum of diffraction line.

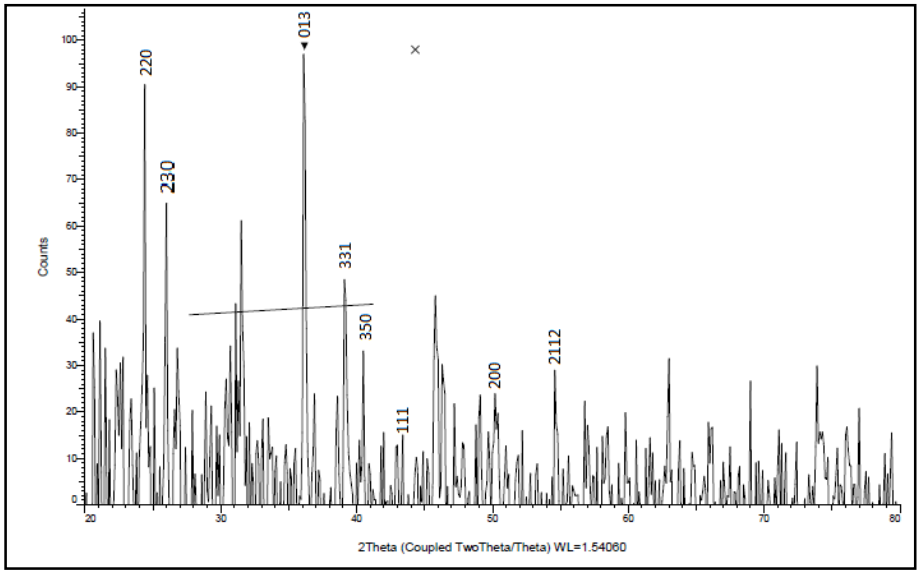

Fig. 3 X-ray Spectrum of $CuBaC_4H_4O_6$ Crystal

Fourier Transform Infrared Spectral (FTIR) Analysis

The FTIR analysis provides information about the chemical bonding or molecular structure of materials. The fig. 4 depicts the FTIR spectrum of the grown crystals. The absorption peaks correspond to the molecular group vibrations. The relation of the molecular group vibrations and the characteristic absorption bands were assigned according to the theories of infrared spectra [42]. The FTIR spectrum recorded for $BaCuC_4H_4O_6$ crystal with observed band as shown in the fig. 4. The spectrum is scanned in the region 450 to 4000 cm^{-1} using "Perkin Elmer model 783". The bands around 3099 to 2853 cm^{-1} are attributed to asymmetric and symmetric–OH stretching of water [43]. The–OH stretching frequency of $CuBaC_4H_4O_6$ appeared at 2925 cm^{-1}.The moderate absorption around 3551 to 2853cm^{-1} is probably due to stretching vibration of alkali group. 2853 to 2925 cm^{-1} may be attributed to hydrogen bonding. The presence of the –C–O– group is indicated by the occurrence the sharp and intense band at 1598cm^{-1} and 1456cm^{-1} indicate asymmetric –C–H bending. The peaks at 1345 cm^{-1} is due to –OCH stretching mode. These bands may be assigned respectively to –C–O– asymmetric and symmetric stretching of –C–O–. The bonding mode of water of crystallization overlaps with the new asymmetric –C–O– frequency band. i.e. the region of for the broadness of the absorption around 1598 cm^{-1}.

The inplane bonding of –C–H is assigned by the peak at 1456 cm^{-1}. The absorption at 1136 cm^{-1} are probably due to –O–H bending while –C–OH stretching vibration respectively which represents the co-ordinate –C–OH group. With the help of the assignment made above, 1080 represents C-O stretching shows sharp peaks absorption at 1080 cm^{-1} indicate that C-O bond stretching in alcohol –C-OH group. 836 These band indicate that O-H out of plane band 753 cm^{-1}. These bands represent that C-H binding out of plane. This is jointed to –C-OH. The probable structure of $BaCuC_4H_4O_6$ crystal may be of the type as indicated.

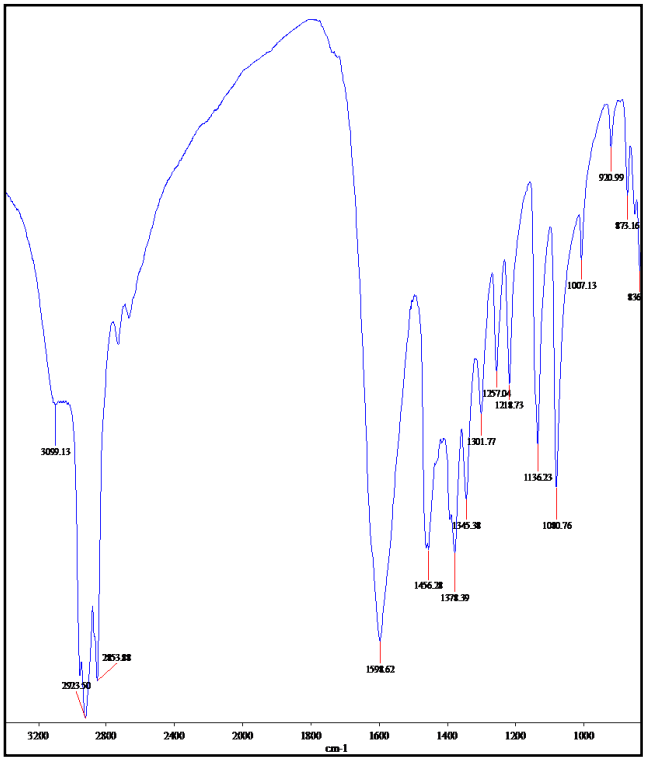

Fig 4. The FTIR spectrum recorded for $BaCuC_4H_4O_6$ crystal

CONCLUSION

The dope crystals of Copper Barium Tartarate were successes fully grown in silica gel. Structural characterization of the grown crystals was carried out by single crystals and powder X-ray diffraction studies, and lattice parameters have been evaluated. FTIR spectrum of Copper Barium tartarate crystals reveals the presence of various functional groups present in the crystal.

Acknowledgement
We are thankful to principal Mr. Dr. L. P. Deshmukh Arts, Commerce and Science College, Varangaon for constant support in all the fields. We are also thankful to Dr. B. K. Sonawane Head, Department of Physics, Varangaon College for his inspired suggestion.

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