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SYNTHESIS AND CHARACTERIZATION OF NOVEL TARTARIC ACID DERIVATIVE

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Abstract
A new organic non linear optical material L-Tartaric acid and KCl doped have been synthesized and grown from its aqueous solution by slow evaporation method. The UV-Visible absorption spectrum indicates that the lower cut of wavelength of the crystals is 255nm and 241nm.The different modes of vibration presents in the crystal are identified by the FTIR analysis. Single crystal X-ray diffraction analysis reveals that the crystal belongs to monoclinic system.

Keywords: Tartaric Acid, Potassium Chloride, XRD, FTIR, UV, TGA, DTA, Micro Hardness, Nlo Studies.

Introduction

Crystal is a solid composed of periodic arrangement of atoms or molecules. Now days, crystals are produced artificially to satisfy the needs of science, technology and jewelers. The ability to grow high quality crystals has become an essential criterion for the development of the nation. Natural crystals have often been formed at relatively low temperature by crystallization from solutions [1].

Crystal are unacknowledged pillars of modern technology, which is very much dependent upon, materials\crystals such as semiconductors polarizer ferrites, magnetic garnets, solid state lasers, non-linear optics, piezo-electric, acousto-optic and photo sensitive materials. All this involves research in crystal growth.

Crystal growth is an important field of material science, which involves controlled phase transformation. The solid state materials can be classified into two types; crystalline and amorphous materials, depending on the arrangement of the atoms or molecules or ions. An ideal crystal is an infinite lattice of atoms arranged in patterns which repeat in all three dimensions with repeat distances [10].

Experimental Work

The calculated amounts of the angler grade starting materials pure and Potassium chloride doped tartaric acid were dissolved in double distilled water and thoroughly mixed for about 3 hours using a magnetic stirrer to ensure homogeneous temperature and concentration throughout the volume of the solution. The saturated solution was filtered twice with wattman filter paper before it was subjected to evaporation. The solution was covered to avoid dust and kept undistributed for days together. Crystal of appreciable size of tartaric acid was obtained within 40 days. The KCl doped tartaric acid single crystal was obtained at the end of 45thday.The single crystal pure and KCl doped tartaric acid was subjected to various characterization viz, UV-Visible absorption, FTIR spectroscopy, single crystal X-ray diffraction. The as grown pure and KCl doped tartaric acid single crystals are shown in the figures.

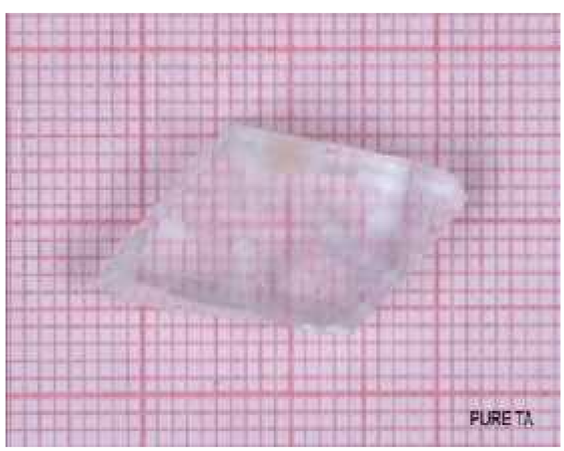

Fig.1 As grown pure Tartaric Acid single crystal

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Fig.2 As Grown Tartaric Acid + KCl Doped Single Crystal

Results and Discussion

UV-Visible Studies Of Pure and KCL Doped Tartaric Acid Single Crystals

UV-Visible studies of pure and KCl doped tartaric acid was carried out by using LAMBDA 35 model UV-Visible spectrometer in the range between 190 nm and 1100 nm. The absorbed UV-Visible absorption and transmittance spectra of grown materials are shown in fig.3 and 4.The UV cut off wavelength of the grown pure and KCl doped tartaric acid are seen around 255 nm and 241 nm respectively. The forbidden energy band gap of the material is calculated by using the following relation.

$$E_g = hc/\lambda$$

Where h is the planks constant, c is the speed of light and λ is observed UV cut off wavelength [11]. The estimated energy band gap of the grown pure and KCl doped Tartaric Acid single crystals are 4.871eV and 5.1543eV respectively. This results shows that the grown both pure and doped materials are belong to the category of insulating material. The optical transmission spectra give the valuable information about the structure of the molecules, because the absorption of UV and visible light involves proportion of electron in n and π* orbital from the ground state to higher energy state. From the device fabrication point of view, the transmission spectra are important since the grown crystal can be used only in the highly transparent region. The transmittance is uniformly high for light in both UV and visible region of the electromagnetic spectrum. Both UV-visible absorption and transmission spectra confirmed that the grown materials well suitable candidate
for device photonic fabrication [8].

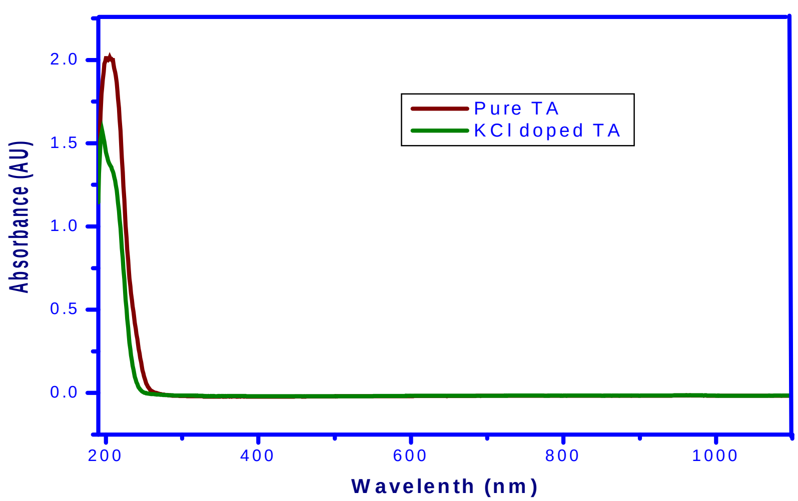

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Fig.3 Absorption spectrum of pure and KCl doped Tartaric acid single crystal

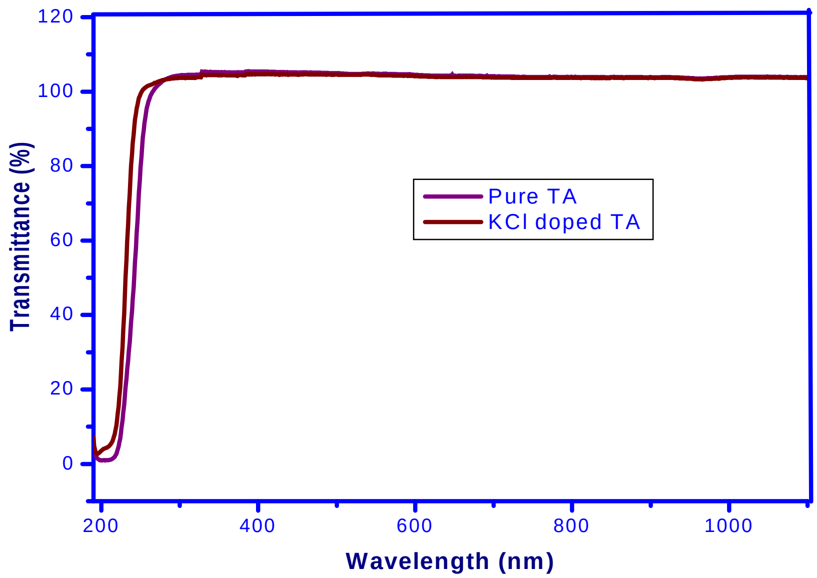

Fig .4 Transmittance spectrum of pure and KCl doped Tartaric acid single crystal.

FTIR Spectral Investigation of Pure KCL Doped Tartaric Acid Single Crystals

The FTIR spectroscopy was used to analyze qualitatively the presents of fundamental groups in grown crystal. The FTIR spectra of grown pure and KCl doped tartaric acid single crystal was recorded using perkin Elmer spectrum FTIR spectrometer by KBr pellet technique in the range of 400cm^{-1} to 4000cm^{-1}.which are shown in figure 5. The peak observed at 3359cm^{-1}, 3412cm^{-1}, 3360cm^{-1} are assigned to be N-H stretching vibration. The peak 1401cm^{-1} can be assigned to the C=S asymmetric stretching vibration. The absorption peak observed at around 1617cm^{-1} corresponds to N-C-N symmetric stretching vibration. The absorption bond observed at 1617cm^{-1}, 1741cm^{-1} in the spectrum can be assigned to the NH_2 asymmetric bonding vibration. The strong bonds at 486cm^{-1} can be assigned to N-C-N symmetric bending vibration[8].The peak observed KCl doped tartaric acid of single crystal spectra is slightly shifted due to the incorporation of KCl ions in tartaric acid single crystal.

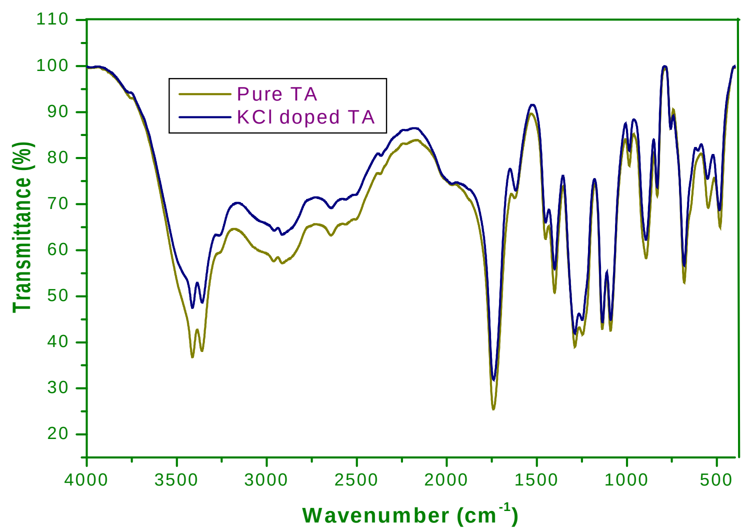

Fig. 5 FTIR spectra of pure and KCl doped Tartaric acid single crystals

Single Crystal X-Ray Diffraction

Single crystal X-ray diffraction studies were carried by using ENRAF NONLUS CAD_4 single crystal X- ray diffractrometer. The estimated lattice parameters of the grown pure and KCl doped tartaric acid crystals are shown in table 1 this results confirmed that the grown both pure and KCl doped tartaric acid crystal belongs to the category of monoclinic system [12]. The doped tartaric acid lattice parameters are slightly changed due to incorporation of the K^+ ion in the pure tartaric acid.

Table: 1 Lattice Parameters of Pure And KCL Doped Tartaric Acid Single Crystal

S.No	**Lattice Paramaters**	**Materials**	a(Å)	b (Å)	c(Å)	V(Å^3)
1	$\alpha = 90^o$ C, $\gamma = 90^o$ C	Pure tartaric acid	4.866	8.046	9.140	357.84
2	$\beta = 109^o$ C	KCl doped tartaric acid	4.868	8.046	9.142	358.073

Vickers Micro hardness Analysis of Pure Tartaric Acid and KCL Doped Tartaric Acid Single Crystals

The Vickers micro hardness studies of a single crystal is influenced by various parameters such as lattice energy, Debye temperature, molecular bonding, yield strength, heat of formation and inter atomic spacing. Micro hardness measurements was carried out by selecting smooth face and free from defects of the grown tartaric acid and KCl doped tartaric acid were selected. In the crystals were measured by applying different loads like 25gm,50 gm and 100gm.The micro cracks were observed on the surface of grown materials due to the mechanical stress when the load of added beyond 100gm.The graph was plotted between load P and hardness number which gives straight line which is shown in fig 6 and 7. The graph was also plotted between log P and log d in fig.6.

The hardness numbers of the both pure and KCl doped tartaric acid crystal were increased while increasing the load value in gm. From the work hardening co-efficient of materials was calculated by taking slope. The calculated work hardening co-efficient of pure and KCl doped tartaric acid single crystals are 1.6199 and 3.7672 respectively. According to the concept of Onitosch the work hardening co-efficient values for hard materials lies between 1 and 1.6 and for soft material is ≥1.6[20].The obtained values of n shows that the grown both pure and KCl doped materials belong to the category of soft class in nature. The work hardening co-efficient of doped material is increased due to the addition of K^+ ion in tartaric acid single crystals. Therefore, both the materials are well suitable candidates for applying the photonic device.

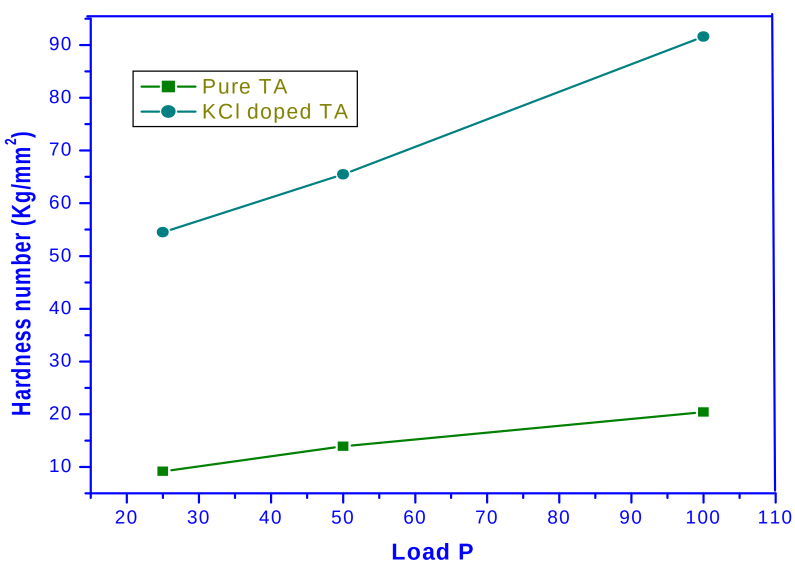

Fig.6 load P Vs Hardness number pure and KCL doped Tartaric acid single crystal

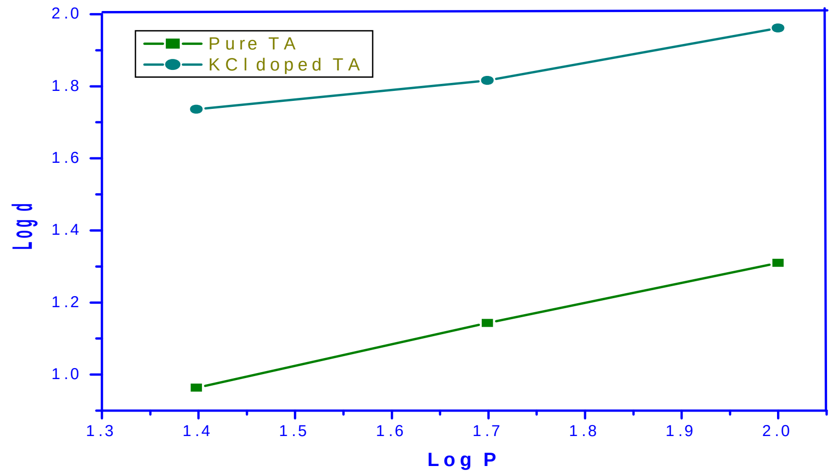

Fig.7 log P Vs Log d pure and KCL doped Tartaric acid single crystal

Thermo Gravimetric Analysis and Differential Thermal Analysis of Pure and KCL Doped Tartaric Acid Single Crystals

Thermo gravimetric analysis and differential thermo gravimetric analysis give more information about different stages of decomposition and phase transition of the grown materials. Thermo gravimetric analysis and differential thermo gravimetric analysis of pure and KCl doped tartaric acid were carried out with a heating rate of 20^{0}C per minute in the air atmosphere from 28^{0}C to 508^{0}C by using simultaneous thermo gravimetric analysis unit of size. The thermo gravimetric analysis and differential thermal analysis traces of pure and KCl doped tartaric acid are shown in figs (8 – 11). There is no loss of weight observed around 100^{0}C indicating the absents of water molecules in the sample. Both pure and KCl doped tartaric acid are thermally stable upto 200^{0}C and 219^{0}C respectively. From the thermo gravimetric analysis, curve of grown materials shows that the decomposition states at 200^{0}C and 219^{0}C. The maximum weight losses observed for both pure and KCl doped materials are 48% and 46% between the temperatures 200^{0}C to 202^{0}C and 219^{0}C to 262^{0}C may be due to the discharge of volatile substances CO_2.The resulting resides of 52% and 8% are stable up to 508^{0}C.This crystal can be used for any application up to 200^{0}C and 219^{0}C.The endothermic peaks of differential thermal analysis curve shows that the melting point of the material. The melting point of grown pure and KCl doped tartaric acid are 220^{0}C and 229^{0}C respectively. The sharp endothermic peaks shows that the grown both pure and KCl doped tartaric acid materials having high crystalline nature. The grown materials are suitable one to apply in photonic application [14].

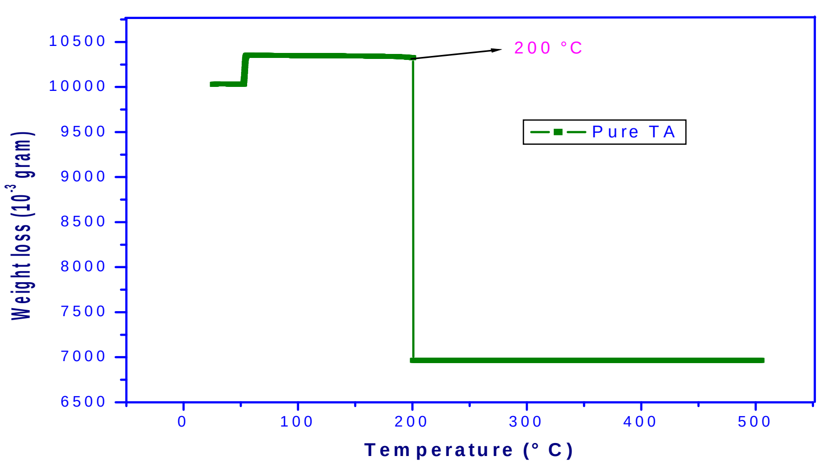

Fig.8 Thermo gravimetric analysis for pure Tartaric acid single crystal

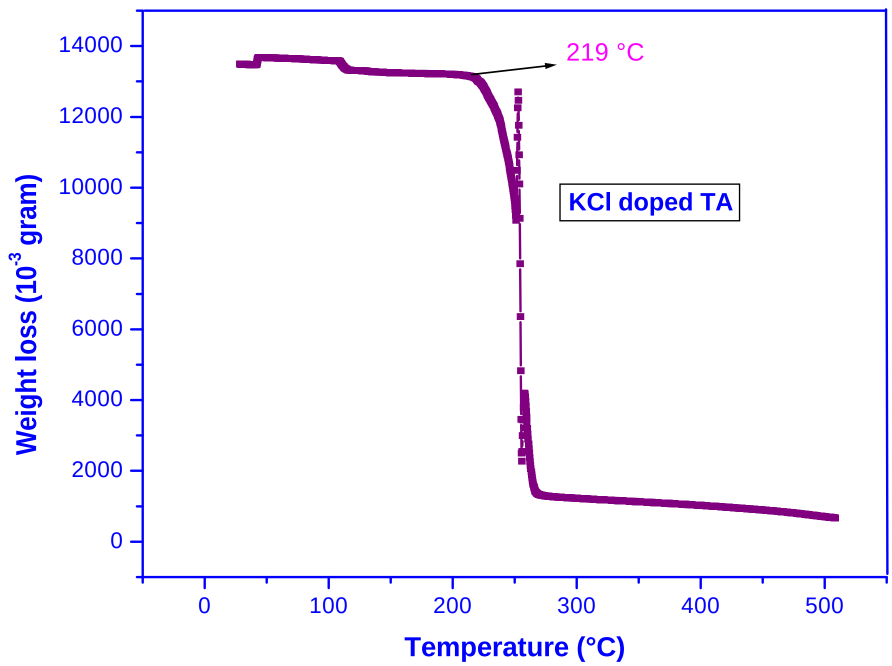

Fig.9 Thermo gravimetric analysis for KCl doped Tartaric acid single crystal

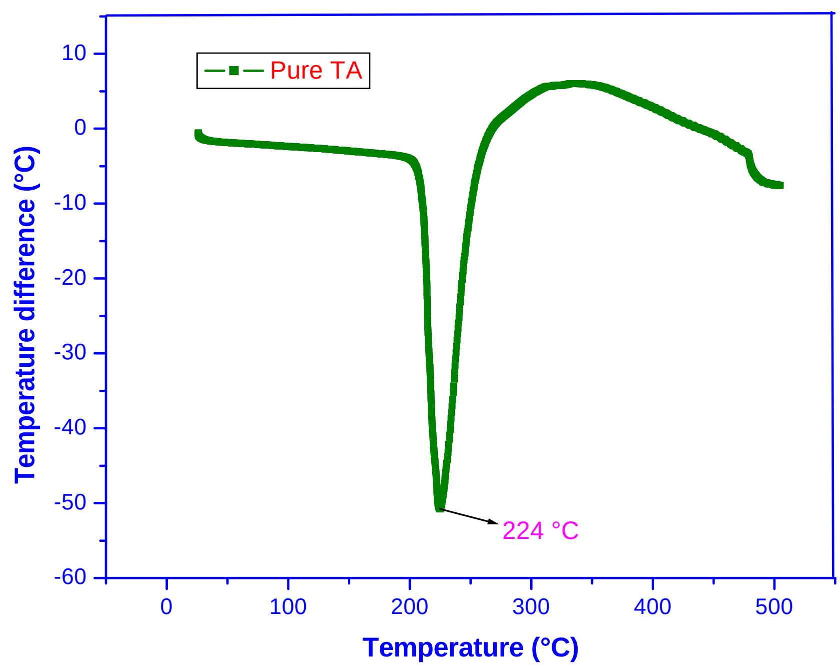

Fig.10 Differential thermal analysis for pure Tartaric acid single crystal

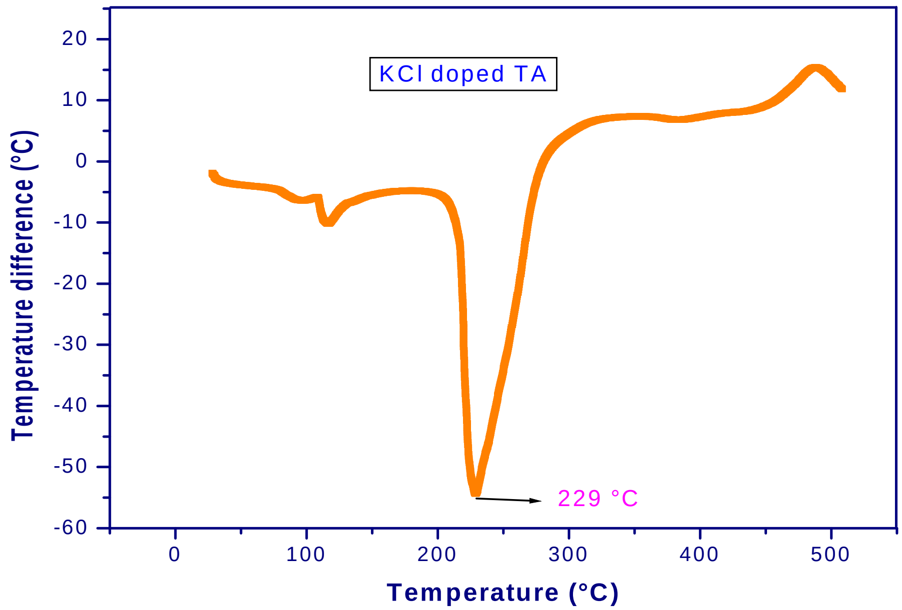

Fig.11 Differential thermal analysis for KCl doped Tartaric acid single crystal

Non Linear Optical Studies For Pure And Kcl Doped Tartaric Acid Single Crystal

The second harmonic generation conversion efficiency was tested by using a modified set up of KURTZ and PERRY technique with the wavelength of 1064 nm [5]. The input power of 70m Joule and pulse with 10 second was passed through fine powdered pure and KCL doped tartaric acid crystal. The output from the sample was collected by using photomultiplier tube. The measured output intensity from the sample was obtained by using a photomultiplier tube [6].The second harmonic generation of the grown material was confirmed by the emission of green radiation with wavelength of 532 nm from the sample. The second harmonic signals of pure and KCL doped crystals are 4mV and 5.5mV respectively. But the standered KDP crystals gave a second harmonic signal of 10mV/pulse for the same input energy [8].The obtained second harmonic efficiency results shows that the pure and KCL doped material have higher value than that of KDP crystals.

Conclusion

Pure and KCl doped tartaric acid single crystals have been successfully grown by slow evaporation method. Optical transmission study shows that the crystal has more than 99% of transmittance is visible and near infrared region which exhibits the good optical quality of the crystal and the optical band gap was found to be 4.871eV and 5.153eV. FTIR studies are clearly confirms that the doping produces shift in the peaks position and the prominence in absorption peaks. The grown crystals were confirmed by single crystal X-ray diffraction analysis which shows that the crystal belongs to Monoclinic system with space group $P2_1$ and lattice parameters are a = 7.718Å, b = 6.023Å,c = 6.206Å, β = 109° C. The micro hardness studies revealed that the grown pure and tartaric acid crystals belong to the category of soft in nature. Thermal analysis shows that the crystal to be stable up to 200°Cand 219°C. The melting point of pure and doped materials are 224° C 229° C respectively. The calculated relative SHG efficiency of the grown pure and KCl doped materials have greater than that of KDP crystals.

References

1. Santhanaraghavan D.N and Ramasamy.P, Crystal growth method and process, Krupublication, Kumbakonam,2001.
2. Puri.P.K, Bubbar.V.K, S.Chand Solid State Physics and S.ChandElectronics, New Delhi, 1997.
3. Lqbals.S.A, Physical Chemistry,Discovery Publishing House, New Delhi, 2005.
4. Arora.M.G, Solid State Chemistry, Anmal Publication Pvt. Ltd., New Delhi, 1997.
5. Bloembergan.N, Nonlinear Optics, World Scientific, Singapore, 1996.
6. Shen.Y.R, Principles of non linear optics, John Wiley and Sons, Newyork, 1967.
7. Franken, P. Hill, A. Peters, Weinreich.C, and Generation of Optical Harmonics. Physical review lestters, 118, 2004.

8. 8. Y.R. Sharma. Elementary Organic Spectroscopy, principles and chemical applications. Published by S.Chand, Multicolour edition.
9. Breitzar.J.G, Dlott.D.D, Iwaki.L.K, Kirkpatrick.S.Mand Rauchturs.T.B, Third –order Non linear optical properties of Sulfur-Rich Compounds, J.Phy.chem103(1999).pp 26-35.
10. 10.ReenaIttyachan ,Sagayaraj.P, J.crystal growth 249(2003).pp553-556.
11. 11.Aruna.S, Bhagavannarayana.G, Sagayaraj.P, Journal of crystal growth, 304(2007).pp184-190.
12. 12.Eugene P. Burtin, “ Principles and Practice of X – Ray Spectrometry Analysis” 2nd ed., Pknum Press, New York,NY,1984.
13. 13.K.V.Rajendran, D.Jayaraman, R.Jayavel, P.Ramasamy,crystal growth 255(2003)pp 361-368.
14. 14.J.Therm. Anal. Calorim., Thermoanalytical study of salts of 3d metal with D – tartaric acid 46(1996),pp.1403-1412.
15. 15.S.Gokul Raj, G.Ramesh Kumar, R.Mohan, Babu Varghese, R.Jayavel J.Molstr 825(2005)pp158-164.
16. 16.S. NaliniJayanthi, A. R. Prabhakaran, D. Subashini, T. Panchanathan and K. Thamizharasan,Archives of Applied Science Research, 5 (2013)pp241- 246.
17. 17.P.N. Prasad, D.J. Williams, Wiley-Interscience, New York, 1991.
18. 18.K.C. Bright, T.H. Freeda, Physica B: Condensed Matter 405 (2010) pp 38-57.
19. 19.M. Jiang, Q. Fang, Advanced Materials 11(1999)pp 1147.
20. 20.K. Kirubavathi, K. Selvaraju, R. Valluvan, N. Vijayan, S. Kumararaman, Spectrochimica Acta Part A 69(2008) pp 1283.