International Journal of Scientific Research in Research Paper
Physics and Applied Sciences
Vol.7, Issue.3, pp.156-166, June (2019) E-ISSN: 2348-3423
DOI: https://doi.org/10.26438/ijsrpas/v7i3.156166

Growth and Characterization Studies on Nicotinic Acid Doped Kdp Crystals

M. Navina[1], M. Sornambol[2], S. Gowri[3], J. Aarthi[4], P. Evangelin Teresa[5]

[1,2,5]Department of physics, Seethalakshmi Ramaswami College, Trichy-620 002, Tamilnadu
[3,4]Department of physics, Cauvery College for Women, Trichy-620 018, Tamilnadu

Correspondence Author: gow.1976@gmail.com

Available online at: www.isroset.org

Received: 18/Apr/2019, Accepted: 12/May/2019, Online: 30/Jun/2019

Abstract- In the present work the growth of potassium dihydrogen phosphate crystal doped with nicotinic acid at various concentrations is reported. KDP is a well –known optical material having different applications in electro-optics and laser technology. Nicotinic acid doped KDP crystal is grown using slow solvent evaporation technique. The doping of nicotinic acid at various concentrations was successively achieved in KDP crystal and confirmed by Fourier transform infrared spectroscopy analysis. The modification of optical transmittance and other optical constants of KDP crystal due to the doping of nicotinic acid have been investigated by means of UV-visible spectral analysis. The dielectric behavior of the sample has been studied. The variation of the dielectric loss with frequency of the applied field is reported.

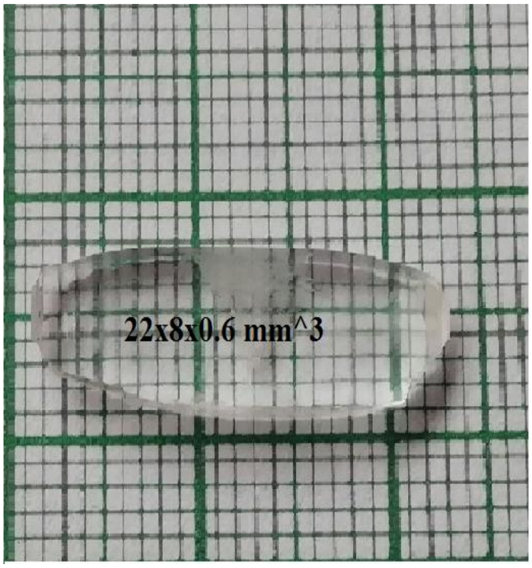
Keywords: FTIR, dielectric studies, antimicrobial studies UV-visible spectral analysis

I. INTRODUCTION

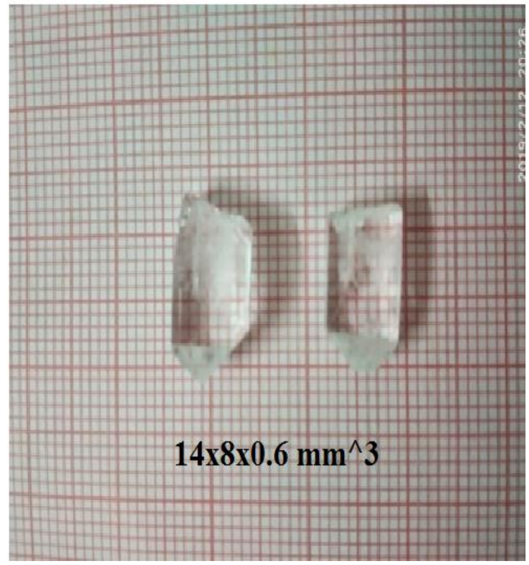
Materials can be classified as single crystals, poly crystals and amorphous materials depending upon the arrangement of constituent molecules, atoms or ions. The single crystals are solids in uniform condition that forms the basis for most of the applications of the crystals. The growth of single crystal with advanced properties plays a vital role in the modern scientific world [1]. Crystal growth is one of the significant fields with controlled phase transformation. The crystal growth process is important in the field of technological applications. The researchers are focused to find suitable materials which exhibit excellent second order nonlinear [2] optical properties for potential applications such as optoelectronics, telecommunication, and optical storage device [3]. The semi organic crystals attract great attention in the field of non linear optics due to their optical non linearity. Potassium dihydrogen phosphate (KDP) crystal is a well known inorganic NLO material which has good ferroelectric, piezoelectric and electro-optic properties. KDP exhibits good homogeneity over large volumes and has a high damage threshold [4]. It is an effective angle turned dielectric medium for optical harmonic generation in near infra red region [5].The bulk size KDP crystals are suitable for device applications [6]. The growth and characterization of KDP doped with nicotinic acid have been reported earlier [7]. Nicotinic acid is an organic compound and a form of vitamin B3. It belongs to the group of pyridine -3-carboxylic acid. It is also known as niacin [8]. The present work reports growth of single crystals of nicotinic acid doped KDP which has been grown by slow evaporation technique with the doping concentration of nicotinic acid as 3% and 4%. The FTIR study was carried out to recognize the modified functional groups of KDP as the consequence of doping. The dielectric studies were carried out to investigate the conductivity of the crystals

II. GROWTH PROCEDURE

Calculated amount of analytical grade (AR) samples of KDP and nicotinic acid were mixed and added slowly into double distilled water as a solvent accompanied by constant stirring with a magnetic stirrer to ensure uniform and homogeneous solution. Upon reaching super saturation, the solution was filtered and transferred to the Petri dish, covered with the thick paper with fine pores in order to minimize the rate of evaporation. Upon, complete evaporation of solvent, single crystals of size 22x8x0.6mm^3 and 14x8x0.6mm^3 were harvested within seven days.



3% of NKDP crystal



4% of NKDP crystal

Fig. 1 Photograph of NKDP crystals

III. RESULTS AND DISCUSSION

3.1 FOURIER TRANSFORM SPECTROSCOPY ANALYSIS

The FTIR spectrum is an applicable tool for the identification of functional groups in the grown crystal. The FTIR spectra of doped KDP were recorded in the range of 4000-400cm-1 using PerkinElmer RXI spectrophotometer. The spectra of both pure and doped crystals were compared and analyzed. The broad band which appears in the range of 3712 to 2753cm-1 [9] depicts the free O-H stretching of water molecules. There is a shift in vibrational assignment at wave number 2752 & 2753cm-1 in the doped crystals which identifies the inclusion of dopants into the parent compound. The P-O-H stretching vibrations can be observed in pure KDP at 2439cm-1 and in the doped crystals at 2434 and 2442cm-1. It may be due to the lattice strain evolved in the crystal. The peak in pure KDP at 1670cm-1 and in the doped crystals at 1659 and 1680cm-1 indicates C=O stretching. In pure KDP, the P=O stretching is observed at 1297cm-1 and it is absent in the doped crystals. The PO_2 symmetric and asymmetric vibrations can be observed at 1103cm-1 in the doped crystals which is absent in the pure.

VIBRATIONAL ASSIGNMENTS

Pure kdp	**Intensity**	**3%nico acid doped kdp crystal**	**Intensity**	**4% nicotinic acid doped KDP crystal**	**Intensity**	**Assignments**
2762	S	2752	S	2753	S	O-H stretching vibration
2439	S	2434	S	2442	S	P-O-H bending
1670	S	1659	S	1680	S	C=O stretching of COOH
1297		1300	VS	1300	VS	P=O stretching
-		1103	VS	1103	VS	PO_2 symmetric and asymmetric stretching
904	VS	910	VS	908	VS	O-P-O symmetric stretching
544	VS	540	VS	538	VS	HO-P-OH bending

VS - very strong, S – strong

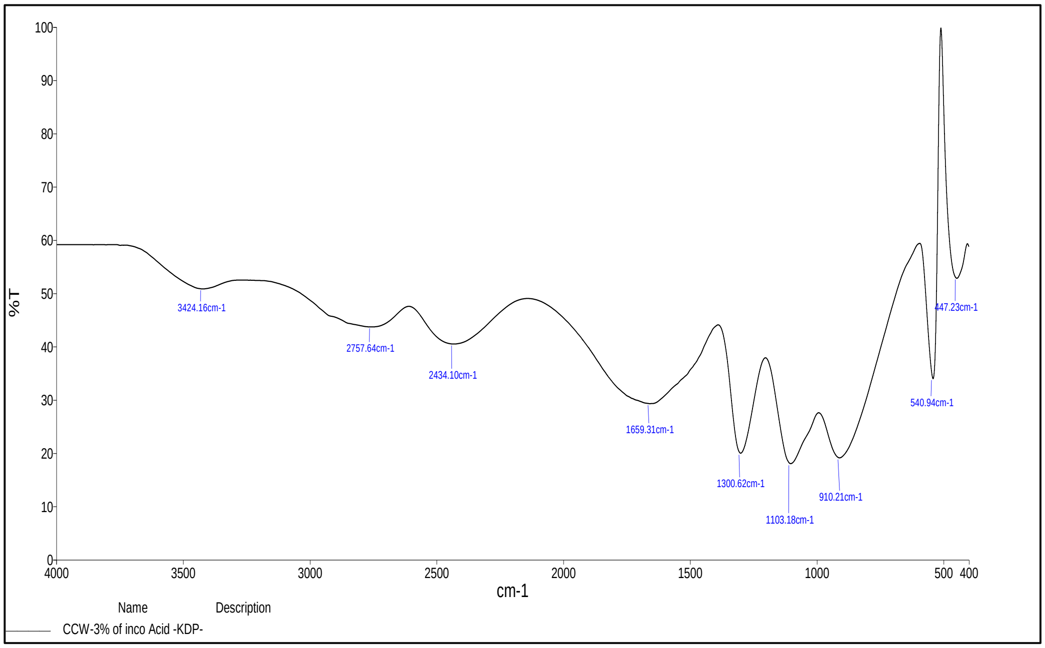


Fig3.1 (a) 3%NKDP

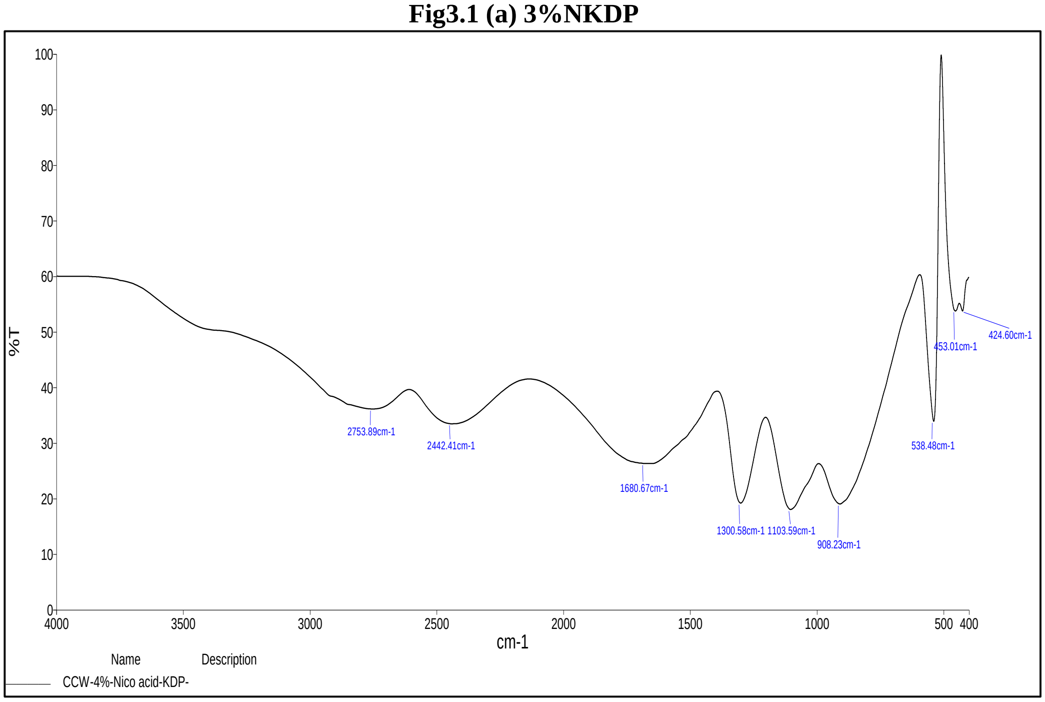


Fig3.1 (b)4%NKDP

3.2 UV-Vis SPECTROSCOPY STUDIES

The UV-Vis study was undertaken with the help of Perkin Elmer lambda (35) instrument within the wavelength range 200-1100nm. A good optical transmittance is a desirable property of NLO crystal. From the UV spectra, the 3% NKDP crystal is found to have 77.95% of transmittance. As for the 4% NKDP crystal, the transmittance is increased to 92.46%.The absorption spectra of both the doped crystals are shown Fig 5.2b and Fig 5.2d [10]. The narrow absorption regions are formed in higher wavelength due to excitation of vibration [11]. The measurement of the band gap is determined from the UV absorption spectrum. As the value of band gap energy is 4.1eV, it behaves apparently equal to the semiconductors.

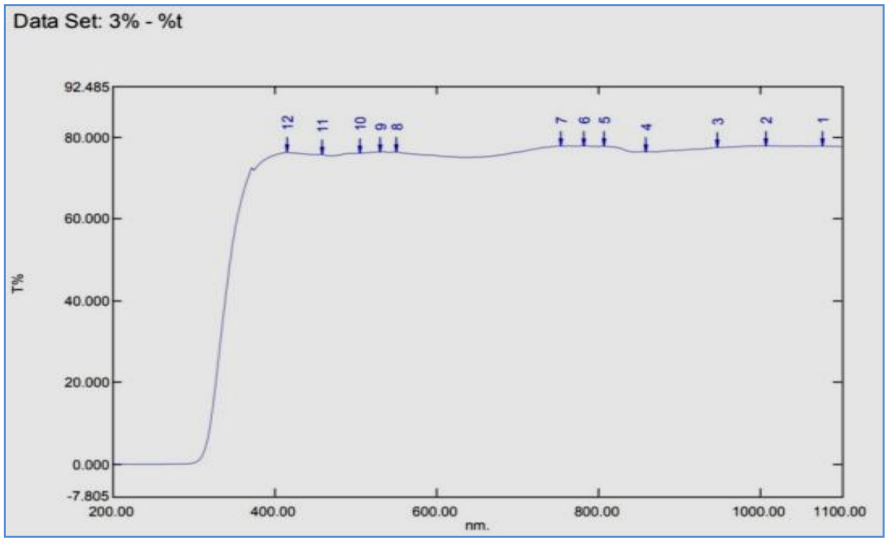


Fig 3.2 a

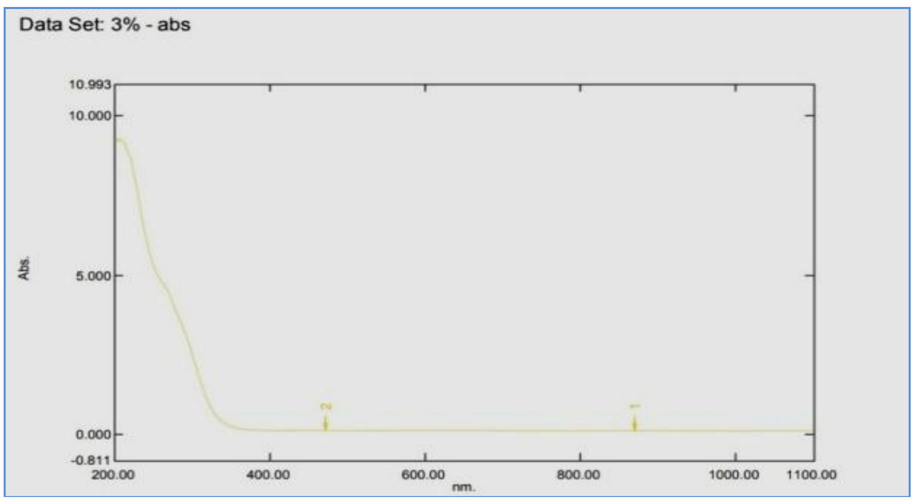


fig 3.2b

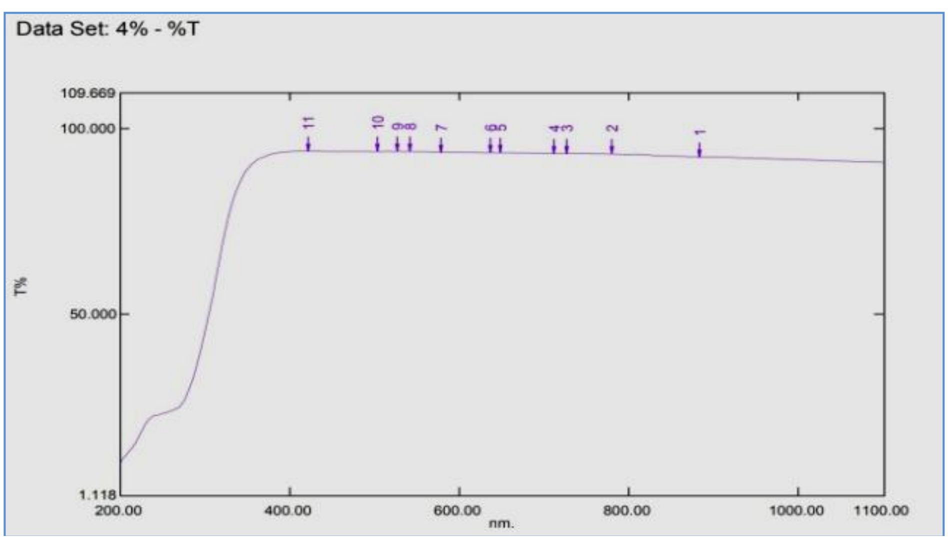


Fig 3.2 c

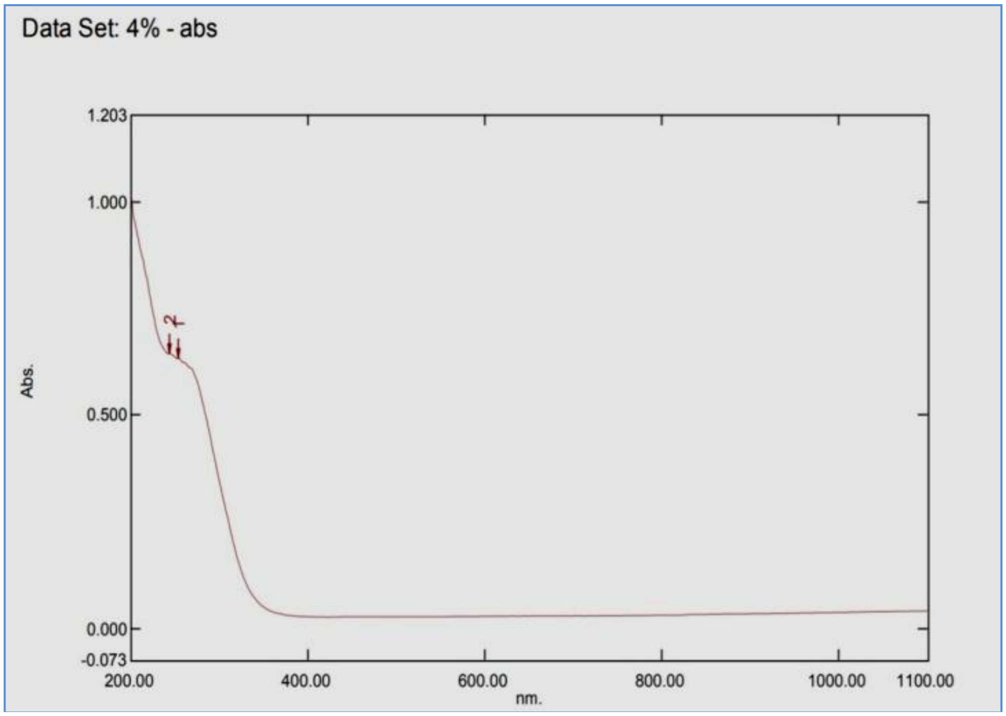


fig 3.2 d

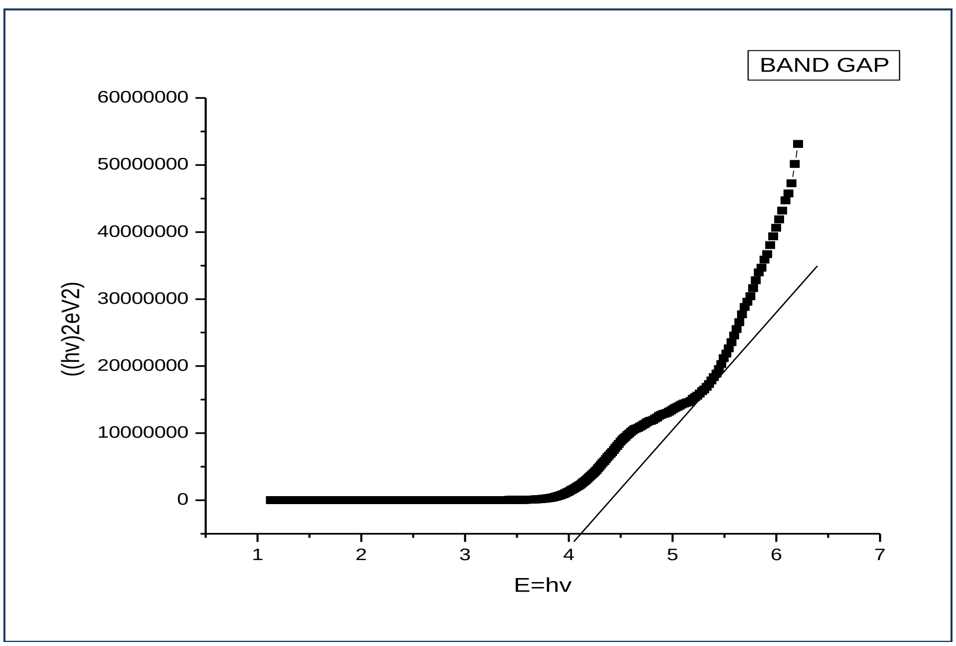


fig 3.2e

3.3 FLUORESCENCE STUDY

The fluorescence studies provide the vital information about the quality, electronic states and influence of intrinsic impurities of the grown crystals. The fluorescence spectrum of synthesized nicotinic acid doped KDP crystal was recorded using Perkin Elmer (LS45) instrument within the range 260-800nm. From the emission spectrum, the excitation wavelength of the grown crystal was found to be 266.59nm (for 3% NKDP) and 384.21nm (for 4%NKDP), and the sharp emission peak was observed at 489.30nm (for 3%NKDP) and at 489.06nm (for 4%NKDP).The recorded spectra confirms that both the grown crystals belongs to blue fluorescence spectrum [12].

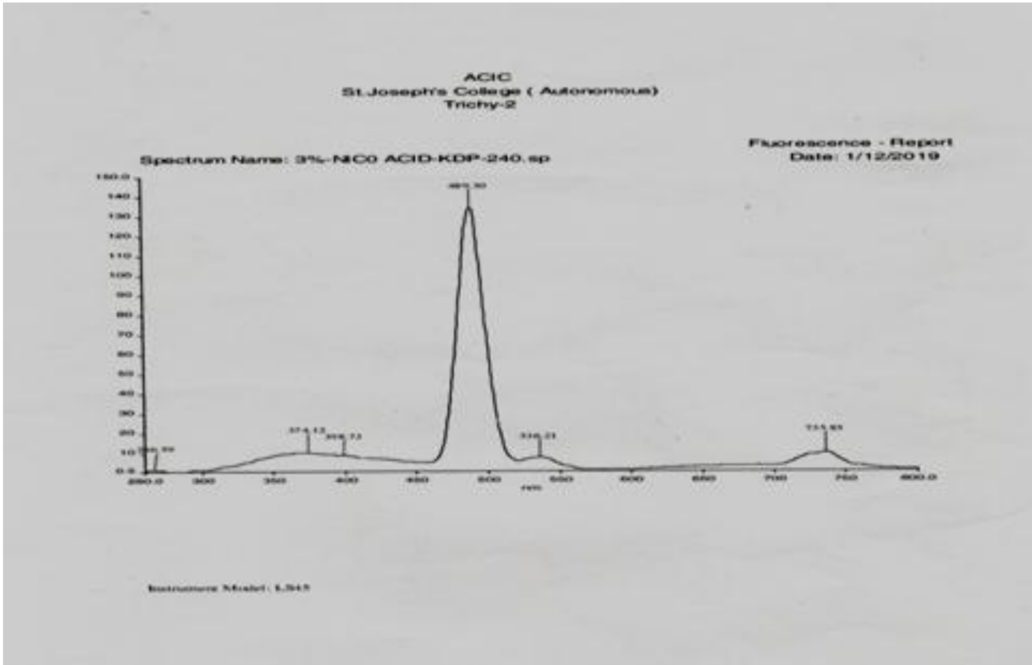


Fig 3.3 a

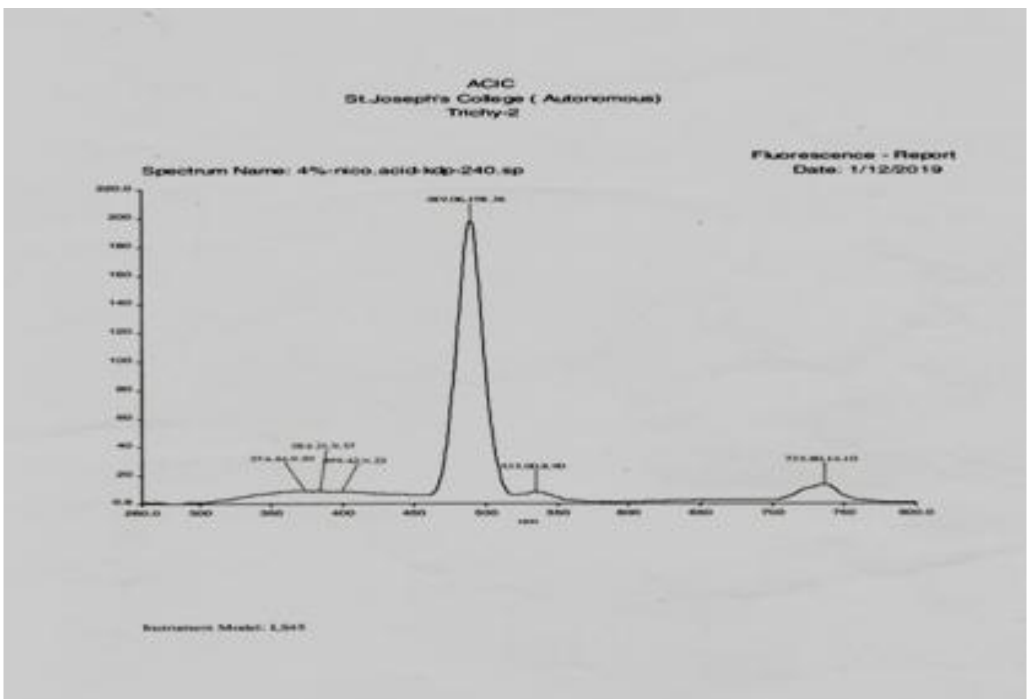


Fig 3.3 b

3.4 PHOTOLUMINESCENCE STUDIES

The photoluminescence studies furnished the quality information regarding the electronic states of the material. The NKDP crystal is photo excited with the energy of wavelength 700nm. The GSA materials with near infrared emission might be suitable of NIR optical imaging and sensing targets [13-14].

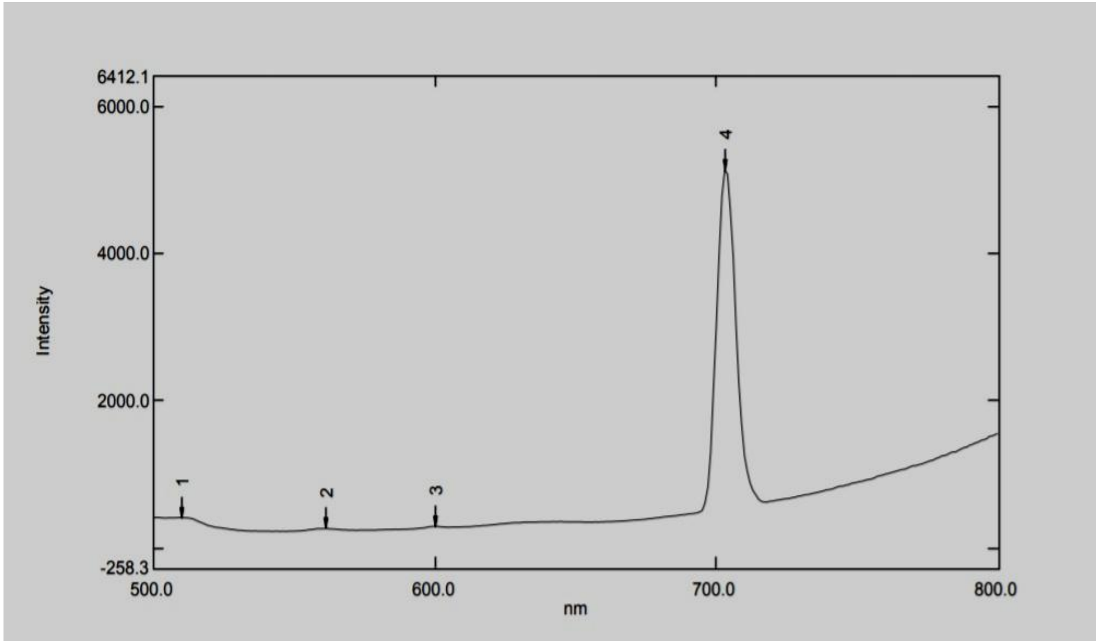


Fig a 3% NKDP

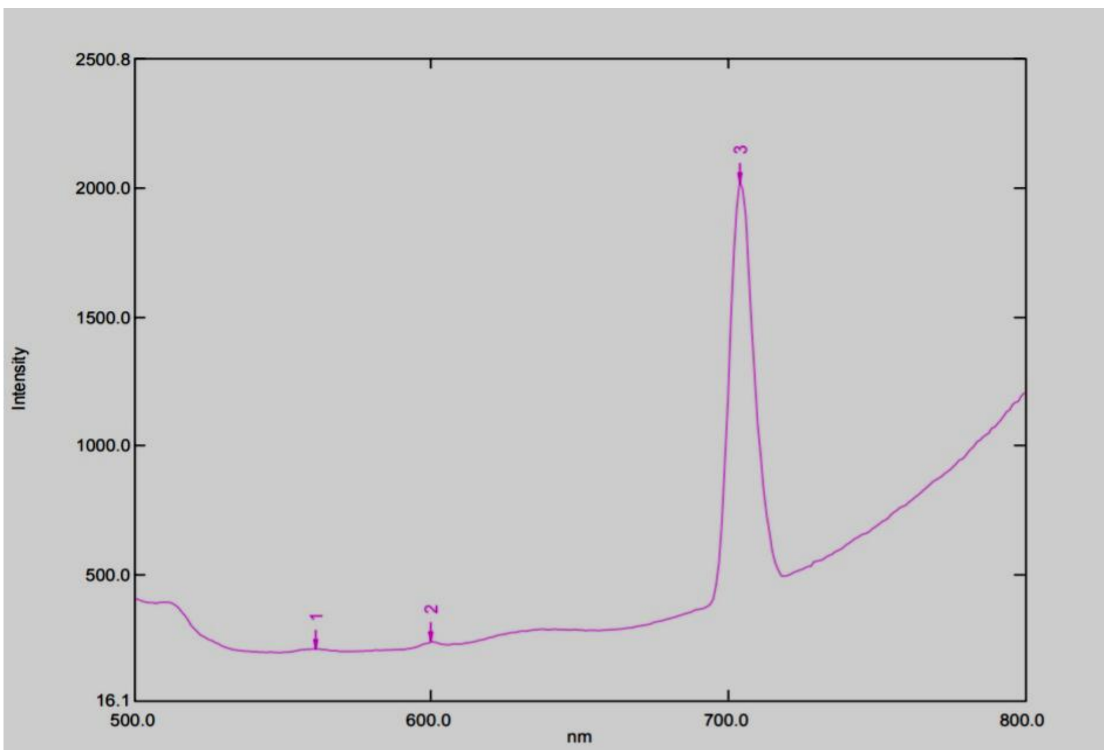


Fig b 4%NKDP

3.5 DIELECTRIC STUDIES

The electrical parameters of NKDP single crystals were measured using the instrument JOGNIC 'S 2816B LCRZ within the frequency range 50Hz to 200 KHz. The sample crystal having silver coating on opposite faces was placed between two copper electrodes to form a parallel plate capacitor. The dielectric constant is calculated using the relation $\in' = c\ d / \in_0$ where C is the capacitance in pF, d is the thickness in m, A is the area in m^2 and $\in_0$ is the permittivity of the free space (8.85×10^{-12} F/m).

3.5.1 Variation of dielectric constant with frequency

The variation of dielectric constant with applied frequency for both the doped crystals is shown in the graphs (3.5.1a and 3.5.1b). It is seen from the plots that the dielectric constant decreases as the frequency increases at all temperatures. The constancy of dielectric constant at particular high frequencies is due to the diminishing of space charge polarizations [15].

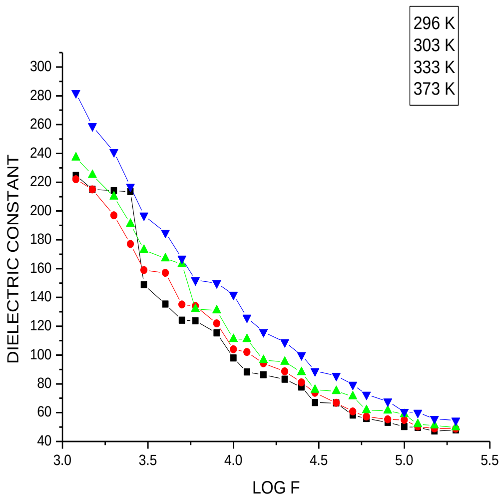


Fig3.5.1 a 3% NKDP

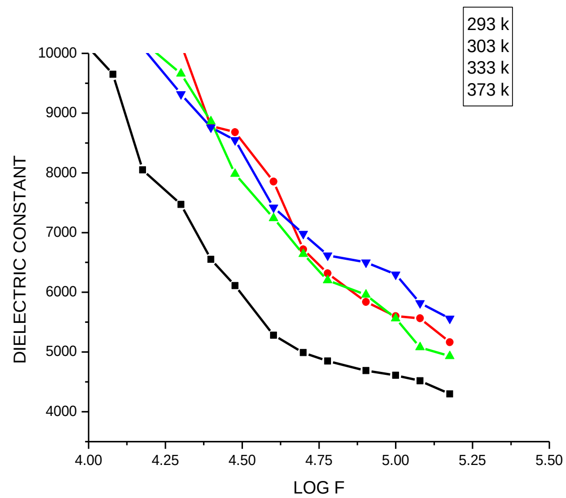


Fig3.5.1 b 4% NKDP

3.5.2 Variation of dielectric loss with frequency

Similarly, the variation of dielectric loss as a function of frequency is shown in the fig (3.5.2a and 3.5.2b). It is seen from the plots that the dielectric loss decreases with increases in frequency for all the temperatures. This suggests that dielectric loss is strongly depending on the frequency of the applied field. The higher values of dielectric loss at low frequencies originate from space charge polarization mechanism dipoles. The characteristic of low dielectric loss at high frequencies enhances the optical quality of the grown sample with lesser defects [16].

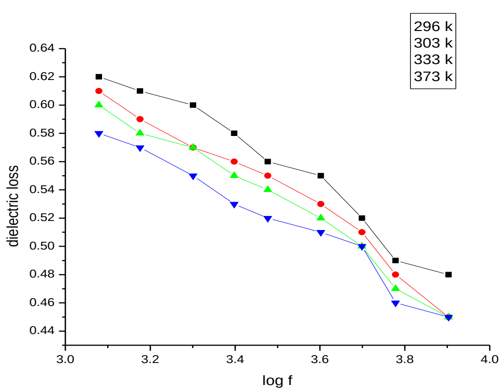


Fig3.5.2 a 3%NKDP

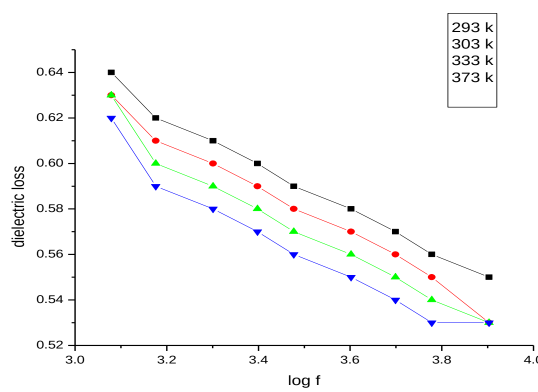


Fig3.5.2 b 4% NKDP

3.5.3 Variation of ac conductivity with frequency

AC conductivity studies are concerned with the mobility and production of lattice defects. The bulk resistance of the crystalline sample is also firm from the study. The graphs shows increase in AC conductivity with increasing frequency. The ac conductivity of doped KDP is higher compared to that of pure KDP [17].

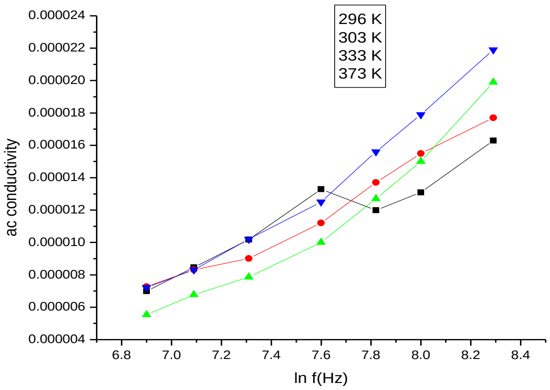


Fig3.5.3 a 3%NKDP

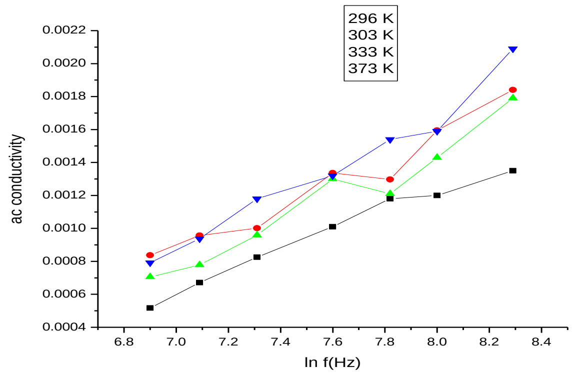
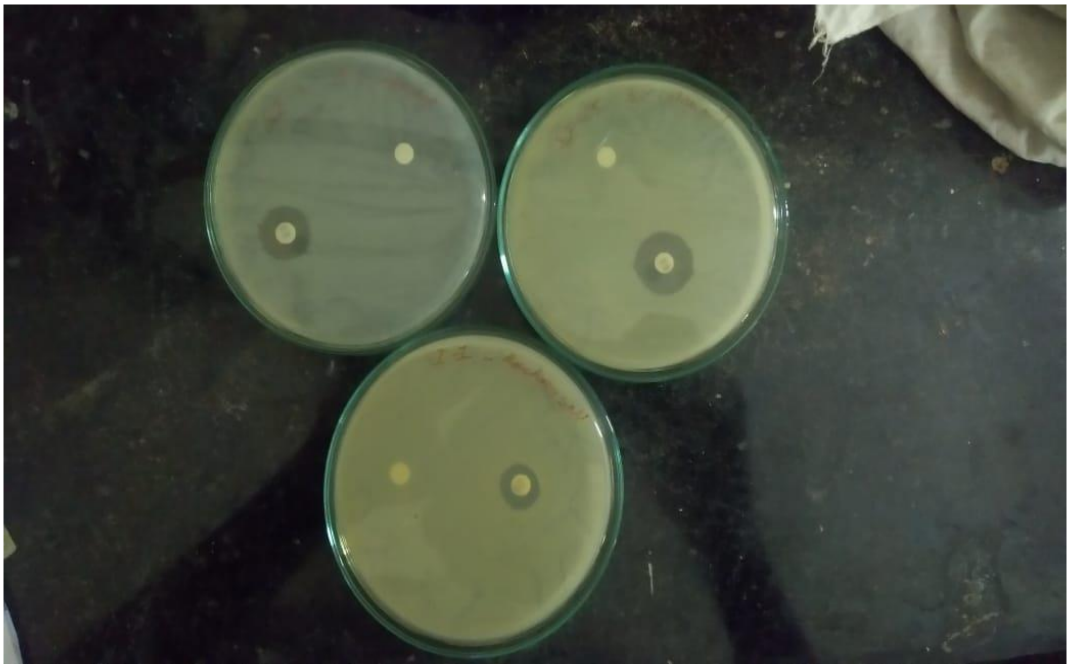


Fig 3.5.3b 4%NKDP

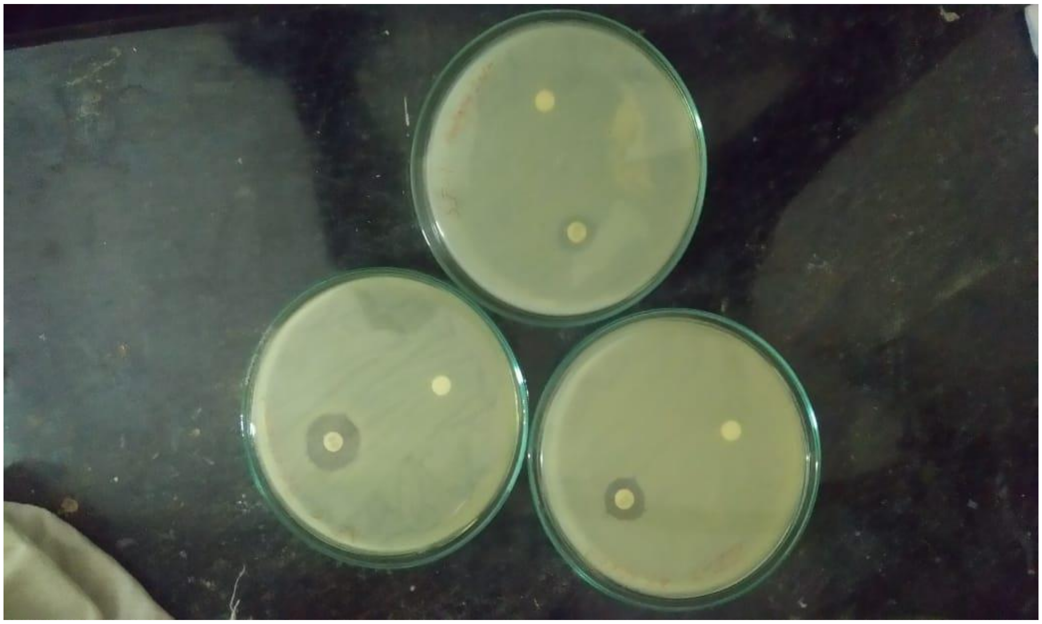
3.6 ANTI MICROBIAL STUDY

The test organisms used in the study were Escherichia coli, proteus vulgaris, klebsiella pneumonia .The cultures were maintained 4℃ on nutrient agar (Hi Media) slants. The antimicrobial activity was assessed by minimum inhibitory concentration (MIC) by means of tetra cyclic disc diffusion method. Twenty ml of sterile, Muller Hinton agar medium (seed agar) was seeded with organism in semi hot conditions and was poured uniformly on the base agar. Standard sterile disc was inoculated with 300µl of different sample preparation were added to respectively and it was repeated for 3 times. These sampled discs were placed in middle of the plate. The plates were incubated at 37℃ for 24 hour and zone of inhibition was measured.

Bacteria	Zone of inhibition		
	3%nico acid doped KDP crystal	4%nico acid doped KDP crystal	Pure KDP
Escherichia coli	8	14	10
Proteus vulgaris	12	15	11
Klebsiella pneumonia	18	12	12



Zone of inhibition of 3% nicotinic acid doped KDP against the bacteria



Zone of inhibition of 4% of nicotinic acid doped KDP against the bacteria

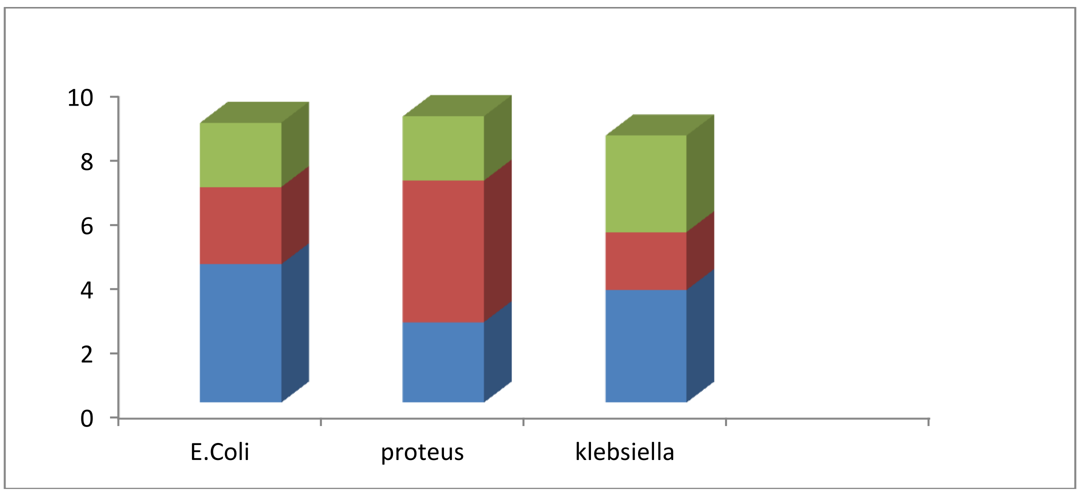


Fig 3.6 a

As we can seen from the table, the crystals of both concentrations shows good inhibitory activities. The 3% NKDP crystal shows good inhibitory activity against klebsiella bacterial strain compared to 4% NKDP. On the other hand, the 4% NKDP crystal shows good inhibitory activity against Escherichia coli and proteus organisms rather than 3% NKDP. The nature of coordination and nuclearity of the compounds in the crystal causes the antibacterial potent of the material [18-20]. The doped KDP crystal has higher antibacterial efficiency due to chelation formation, which reduces the polarity of the metal ion (k^+) to a greater extent. For potential application such as opto-electronic industries crystals with good bio compatibility are used, which is suitable for 3% and 4% NKDP crystals.

IV. CONCLUSION

Single crystal of nicotinic acid doped KDP crystal were grown by slow evaporation solution growth technique and characterized. FTIR analysis confirmed the inclusion of the dopant into KDP and identified the parent shifting assignments. From, UV –Vis spectrum, it is clear that the crystals have High transmittance window in visible region and the lower cut off wavelength is shifted to higher wavelength. The Fluorescence spectra revealed the emission and excitation peaks of both 3% and 4% NKDP crystals. Photoluminescence measurement identified the NIR optical imaging and sensing determination of the grown crystals. The plots of dielectric constant and dielectric loss against frequency proved the good optical quality and low value of dielectric loss at high frequencies. The study confirms that the crystal holds high NLO property. From the ac conductivity plots it was observed that the activation energy increases with increase in frequency. The antimicrobial studies concluded that the 3% NKDP crystal shows good inhibitory activity against klebsiella bacterial strain compared to 4% NKDP. On the other hand, the 4% NKDP crystal shows good inhibitory activity against Escherichia coli and proteus organisms rather than 3% NKDP.

REFERENCES

[1]. P.Santhana Raghavan, P.Ramaswamy 'Recent trends in crystal growth technology', PINSA , 68; No 3 may (2002).

[2]. R. Dillip, C.Madhukar Reddy, B.Deva Prasad Raju 'Growth and characterization of non linear optical material', J.Minerals and materials characterization and engineering,10-12, 1103-1110, (2002).

[3]. E.D.Yakushkin, E.P.Efremova, A.I.Baranova, 'Growth conditiond and electrical properties of KDP crystals: II Dielectric permittivity measurements', crystallography reports, 46-5,904-908, (2001).

[4]. T.Prasanyaa and M.Haris , 'Growth and chacterization of semi organic NL0 L-arginine trifluroacetate 9LATF added KDP single crystals', Archives of physics Research, 2(4), 60-66, (2011).

[5]. M.Iyanar, J.Thomas Joseph Prakash, C.Muthammichelvan and S.Ponnusamy, 'synthesis, growth, and characterization studies of the semi organic nonlinear optical single crystal of gamma glycine', Journel of physics sciences, 235-244, 13, (2009).

[6]. K.D.Parikh, B.B. Pakekh, M.J.Joshi, 'structural, SHG and dielectric study of L-tryptophan doped KDP crystals', Proc. International conference on materials and characterization techniques, VIT university, vellore 1559-1562, (2014).

[7]. B.Deepa and P.Philominathan , 'Enchanced NLO and antibacterial properties of nicotinic acid doped KDP crystals : synthesis, growth and characterization', Materials research innovation, (2016).

[8]. https://en.m.wikipedia.org>wiki> Niacin.

[9]. V.Sangeetha, K.Gayathiri, P. Krishnan, N.Sivakumar and N.Kanaganathan 'Growth, optical, thermal, dielectric and microhardness characterizations of melaminimum bis (trifluroacetate) trihydrate single crystal', J.cryst.growth, 389,30-38, (2014).

[10]. Z.Delci, D.shyamala, S.Karuna, and A.Thayumanavan. 'Optical characterization studies on boron doped KDP crystals', Archives of physics research, 3(5): 346-353, 2012.

[11]. H.Huang, X.Yang, Carbohyder.Res. 339, 574, (2004).

[12]. Salah Sedarous, J.Anand Subramony , Bart Kahr, 'Structure and optical characterization of Rochelle salt dye inclusion crystals and fluorescence detection of their phase transitions' ferroelectric, 191, 301-306, (1997).

[13]. A.Arunkumar, P. Ramaswamy, K. Vishnu, M.K. Jayaraj, ' Growth, structural, thermal, optical, and electrical properties of potassium succinate-succinic acid crystal', J.Mater. Sci. 49, 3598-3607 (2014).

[14]. C.L. Amiot, S. Xu, S. Liang, L.Pan, J.X. Zhao, ' Near –infrared fluorescent materials for sensing of biological targets', Sensors 8, 3082-3105 (2008).

[15]. V.Revathi, V.Rajendran, 'synthesis, Growth and characterization of semi organic zinc(tris) thiourea chloride crystals', Recent Research in science and technology., 4(2), 38-41, (2012).

[16]. U.Krunanithi, S.Arulmozhi and J.Madhavan, 'structural, optical and dielectric studies on pure and doped L-Alanimium Maleate single crystals', IOSR Journal of applied physics, 1(2), 19-23, (2012).

[17]. R.Anandhakumari and R.Chandramani, 'Electrical conductivity and dielectric measurements in Au+ doped /undoped KDP crystals with KCL and NaCl magnetic, as additives', Indian Journal of applied physics , 1(2), 19-23, (2012).

[18]. P.R.Modiya and C.N patel 'synthesis and screening of antibacterial and fungal activity of 5-chloro-1, 3-benzoxal-2(3h)-0ne derivatives org', med. Chem.lett.,2,29, (2012).

[19]. S.M.Roy, M.R.Sundarsanakumar, V.S.Dhanya, S.Suma and M.R.Prathapachandra kurup ' crystal growth, spectral, Antibacterial and antifungal studies of co(II)and Ni(II) complexes of 4-nitrobenzoic Acid',J.Korean chem. .Soc.,58,(3),258-266,(2014).

[20]. H.Liu, W.Yang, W.Zhou,Y.Xie and M.Li: 'Crystal structures and antimicrobial activities of copper (2) complexes of fluorine-containing thioureido ligands',Inorg.Chim. Acta.405, (24),387-394,(2013).