

Thermal, FTIR and NLO Studies on L-Alanine Doped KDP Crystal

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Abstract:- Potassium Dihydrogen Phosphate (KDP) is an excellent inorganic nonlinear optical (NLO) material with different device applications. Most of amino acids possess NLO property; therefore it is doped in KDP crystals. Nonlinear optical (NLO) materials are capable of producing higher values of the original frequency and hence, this phenomenon can find applications in optical modulation, fiber optic communication and opto-electronics. In the present study, amino acid ALANINE doped KDP crystals were grown by slow aqueous solvent evaporation technique. The doping of ALANINE was confirmed by the UV-Vis spectroscopy and the FT-IR spectroscopy. The powder XRD was carried out to study the single phase nature of the samples. The effect of doping on thermal stability of the crystals was studied by TGA,

Keywords: Nonlinear optical crystal, FTIR, TGA, powder XRD,

INTRODUCTION

Inorganic NLO material such as potassium-Dihydrogen Phosphate (KDP) are known to exhibit second harmonic generation effect (SHG). Non linear optic is playing a major role in the emerging photonic and optoelectronic technologies. New nonlinear optical material (NLO) have an impact on laser technology, optical communication & data storage^[1]

(KDP) Potassium dihydrogen phosphate material possesses important piezoelectric, electro optic & nonlinear properties. They have attracted the interest of many theoretical & experimental researchers probably because of their comparatively simple structure & very fascinating properties associated with hydrogenatively bond system involving large isotopic effect, broad transparency range, a high laser damage threshold & relatively low production cost^[2]

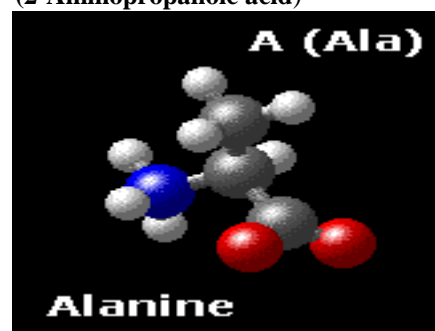
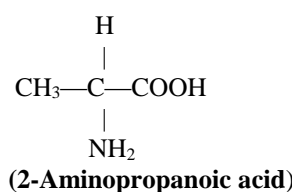
NLO organic crystal must possess high second order optically non linearity, lower laser damage threshold power, transparency in UV region easy growth with larger dimension^[3]. S.S Husaini^[4] have grown Glycine doped KDP and it was observed that the SHG efficiency is enhanced by that of pure KDP that means that improved NLO properties.

Kumaresan et al^[5] have grown KDP with L-glutamic acid, L-Histidine and L-Valine as dopant. They report the growth habit modification with P^H variation, improved

optical transmission and NLO property. They also observed an increase in the mechanical hardness with respect to the dopant concentration.

Parikh et al^[6] have grown L-Lysine doped KDP crystals with increasing NLO properties and optical transmission but with decreased thermal stability.

In the present investigation, the ALANINE doped KDP crystals have been grown and characterized by FT-IR, powder XRD, TGA-DTA, to identify the improvement in various properties of the grown crystals due to doping. The chemical structure of L-Alanine is given as follows,



EXPERIMENTAL

CRYSTAL GROWTH:

Single crystals were grown by solution growth technique under optimized identical experimental conditions (such as solvent growth method and growth temperature). The doping of L-Alanine (GR-grade) was carried out by adding 0.2 weight percent powder form of Alanine into a 100 ml solution of double distilled water. The mixture was thoroughly stirred for 6 to 8 hours using a magnetic stirrer for homogenization. The purity of the crystals was improved by successive recrystallization. The grown crystals were found to be colorless and transparent. Fig 1 shows the photographs of 0.2 wt % Alanine doped KDP crystal.



Fig. 1: 0.2 wt % L-alanine doped KDP crystal

CHARACTERIZATION

The grown Alanine doped KDP crystal was subjected to various characterization viz. powder & single crystal X-ray diffraction, TGA (Thermo gravimetric analysis), FTIR Analysis Dielectric studies etc. and the result we compared with that of pure KDP .

RESULT AND DISCUSSION

1. FTIR ANALYSIS: FTIR spectra of powdered samples were studied in the range of 400-4000 cm⁻¹. The FTIR spectrum of 0.2 wt % of L-ALANINE doped KDP crystal as shown in fig 2. The assignments confirms the presence of various functional group present in the material given in table below. The recorded FTIR spectra we compared with the standard spectra of functional group [7]

All the absorption frequencies & there tentative assignment are listed in below table -

SR NO	WAVE NUMBER	FUNCTIONAL GROUP
1	2723.30	OH-STRETCHING
2	2675	NH3-STRECHING
3	2171.70	OH-SYMETRIC DEFORMATION
4	1747.39	NH3 ⁺ ASYMETRIC DEFORMATION
5	1645.17	NH3 ⁺ ASYMETRIC DEFORMATION
6	1568.2	NH-DEFORMATION
7	1531.37	NH3 ⁺ SYMETRIC STRECHING
8	1487.01	CH3 ASSYMETRIC DEFORMATION
9	1427.23	C-O STRECHING
10	1299.93	CH3 DEFORMATION
11	1099.35	C-N STRECHING
12	902.62	C-C STRECHING
13	889.12	C-C-N STRECHING
14	534.25	C-C DEFORMATION

In the FTIR spectra the broad band gives at 2171.70 cm⁻¹ & 2723.30cm⁻¹ represent OH stretching of COOH group. The prominent bands between 2675 & 2171 cm⁻¹ indicate combination of asymmetrical bending vibrations of NH3 group. Instance absorption is observed at 534.25 c m⁻¹

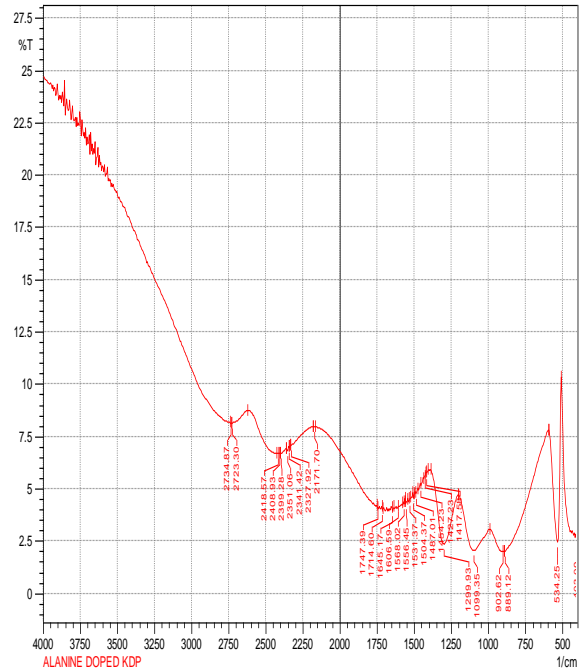


Fig. 2: FT-IR spectrum of 0.2 wt % L-alanine doped KDP crystal

2. TGA ANALYSIS:-The grown crystal was subjected to thermogravimetric analysis (TGA) at heating rate of 35^oc/min to 800^oc, to study the wt loss thermal stability of KDP crystal after addition of ALANINE. The thermogram of the endothermic peak was observed at 210.87^oc that means there is short wt loss takes place at 210.87^oc. The exothermic peak observed at 260.5^oc

The thermo grams & its differential thermogravimetric trace as shown in fig 3

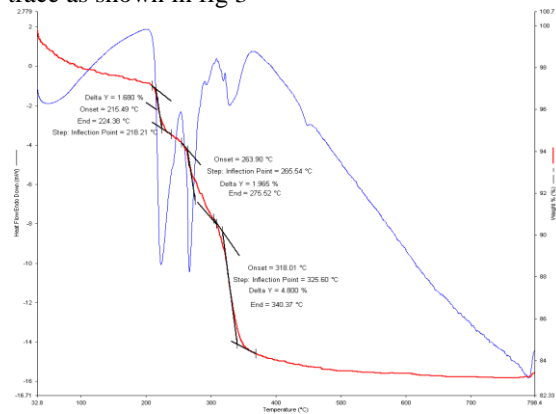


Fig. 3: TGA of ALANINE doped KDP

There is no wt loss between 100^oc to 200^oc this indicate that there is no inclusion of water in the crystal lattice .The major wt loss starts at 260.5^oc and it continues up to 340^oc .The nature of the weight loss indicate the decomposition point of the material. In the DTA spectrum, exothermic peak is observed around 350^oc corresponds to the decomposition temperature of the material.

XRD (X-RAY DIFFRACTION ANALYSIS)

Single crystal x-ray diffraction data of KDP crystal were collected using XPERT-PRO INSTRUMENT (XRDML) with CuK α radiation (K α =1.54060).The powder XRD study conducted to verify single phase nature of the sample .The sample was scanned in the range of 10 to 80 ^oc at the scan rate of 2^o/min .The finely powdered material of grown crystals were used for the analysis . XRD patterns ALANINE doped KDP crystal as shown in below figure .

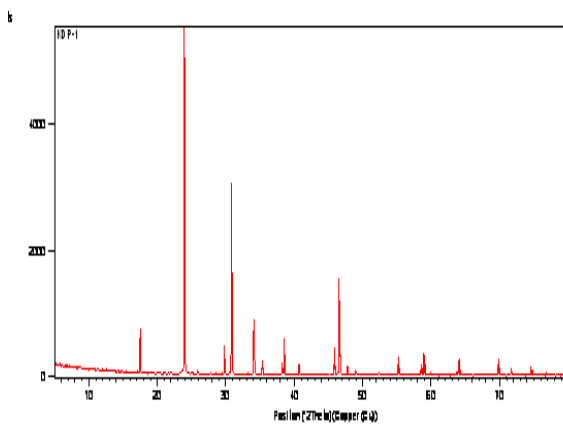


fig. 4: Powder XRD pattern of 0.2 mole % ALANINE doped KDP crystal

The prominent Bragg's peak at specific 2 θ angle reveals the high crystalline nature of the crystal .Powdered XRD software used for calculating d value and crystal plane^[8] .X-ray powder diffraction pattern of pure and doped KDP crystal are identical as shown in above fig KDP has tetragonal unit cell space group of 142d having dimension a=b=7.4523Å & c=6.3589 Å

CONCLUSION

Pure and ALANINE doped KDP crystal were grown by slow solvent evaporation technique .The FTIR spectra confirmed the presence of amino acid ALANINE in KDP crystals. All the functional group are present n the crystals and are confirmed by FTIR Spectrum. Thermo grams of pure and ALANINE doped KDP crystal suggested that as the doping increased the crystal became thermally less stable and it indicate that this crystal can be exploited for NLO application up to the temperature of 218.21^oc.The XRD spectrum shows the excellent nature of ALANINE doped KDP crystal.

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