STUDIES OF THE CRYSTAL GROWTH AND CHARACTERIZATION OF KDP DOPED CRYSTALS

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***Abstract*— Nonlinear optical single crystals of potassium dihydrogen phosphate doped Congo red dye were grown by slow evaporation method by suitable solvent. The good quality single crystals have been harvested in the period of 23 days. The lattice parameters of the crystal were confirmed by single crystal X-Ray diffraction analysis. The optical transmission observed from the specimen, FTIR confirms the presence of functional groups in the grown crystal. The solidity of the crystal has been studied by Vicker's micro hardness values. The relative second harmonic generation ability has been tested by Kurtz Perry powder technique. The thermal studies of the crystal has be**en studied by TGA/DTA analysis.

Index terms- **Congo red dye, NLO, micro hardness, XRD**

I. INTRODUCTION

The new materials inspected for nonlinear optical applications had always been inorganic. Many inorganic crystals are well examined in terms of their physical properties. Since these materials are mostly ionic bonded and it is easier to synthesize. Inorganic materials often have high melting point and high degree of chemical inertness. High temperature oxide materials are well studied for diverse applications like piezo-electricity,pyro-electricity, Ferro electricity and opto electronics. Hence when the search for new materials began in NLO scientists often trusted their intuition,screened the known materials and were fairly successful

KDP is a reviewed system for nonlinear optical device application. It is an effectual angle tuned dielectric medium for second harmonic generation in the visible region.The material offers high transmission in the range of visible spectrum [1-4].

Improvement in the quality of KDP crystals and the performance of KDP based devices can be realized with suitable dopants[5-7]. To analyse the dye based dopants on the NLO property of the KDP crystals for which efforts were made to dope KDP with Congored dye has been studied the dye doped KDP crystals were grown by slow evaporation method at room temperature. The crystallinity of the material were characterized by single crystal x ray diffraction analysis, the frequency assignments of different functional group of doped KDP was determined by FTIR analysis and the optical properties were analyzed by UV- Vis .The thermal stability were studied by TGA and DTA analysis. The relative second harmonic generation efficiency has been approved by Kurtz Perry powder technique.

II. SAMPLE PREPARATION

Reasonable size favorable qualities of single crystal are crucial for the practical applications. In the existent work, the single crystal of potassium dihydrogen phosphate doped Congo red dye was grown using double distilled water as the convenient solvent by solution evaporation method at constant temperature[9-11]. The supersaturated solution was blended well to attain the homogenous solution. The solution was kept for evaporation to dry at room temperature. The purity of crystal was elevated by successive recrystallization process which was free from macro defects by self-nucleation of saturated solution. Single crystals of KDP doped Congo red dye have been harvested in the period of 23 days were shown in figure 1.

Figure 1 grown crystal image KDP doped Congo red dye

III. RESULTS AND DISCUSSION

A. STRUCTURE OF THE CRYSTAL

To confirm the crystallinity of the grown crystals and the unit cell parameters of potassium dihydrogen phosphate doped Congo red dye, X-Ray diffraction analysis carried out with the help of single crystal X-Ray diffractometer. It was found that KDP doped Congo red dye having triclinic system. The lattice parameter values were obtained in Table 1.

Table-

Lattice parameters	Values
a	6.307Å
B	6.317Å
c	6.309Å
A	107.54°
B	107.70°
Γ	113.06°
Volume	193.2Å³
System	Triclinic

B. OPTICAL STUDIES

To determine the transmission range of the crystal which is used to know the suitability of KDP doped Congo red dye crystals for optical applications. The wavelength range between 190nm and 1100nm was made by UV-Vis spectrometer (BERKIN ELMEP LAMBDA 35)[11-13].The absorbance is not enrolled in the wavelength range starting from 200nm to 1100nm is an advantage for materials having NLO properties.

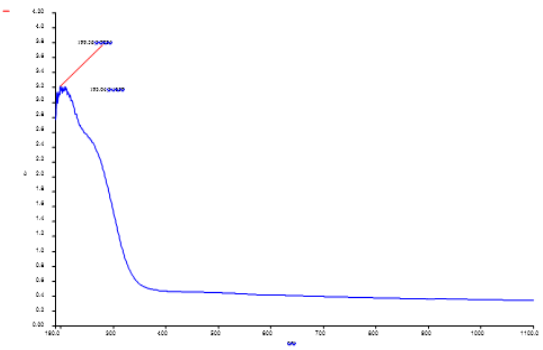
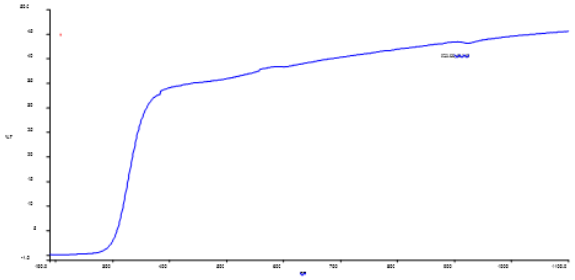


Figure 2: UV Absorbtion graph of KDP doped congo red dye



C. SHG EFFICIENCY

The NLO property of potassium dihydrogen phosphate doped Congo red dye crystal was performed by Kurtz Perry powder technique[14-15]. The crystal was grained into powder and densely packed in the microcapillary tube of uniform diameter. A quanta Ray Spectra physics ND:YAG laser producing pulses with a width of 8ns and a reaction rate of 10Hz was used. The laser was focused to fall on the powder sample. SHG was confirmed by the emission of green radiation (532nm) and the optical signal was controlled by photomultiplier tube (PMT) and converted into voltage output in CRO.[16]. The powder SHG efficiency of KDP doped Congo red dye was found to be 0.68 times that of the standard KDP crystal.

D. MICROHARDNESS MEASUREMENT

To find the mechanical hardness of the grown crystal which was also the deciding properties particularly for post-growth process and device fabrication. Microhardness was measured using Vickers micro hardness tester. The applied load was varied from 25g to 100g for constant indentation period. The Vickers hardness number Hv was calculated using the relation

$Hv = 1.854\ p/d^2 kg/mm^2$

Where p is the indenter load in kg and d is the diagonal length of the impression in mm [16]. The graph shows the variation of Hv with applied load reveals that the hardness increases with the increase of load in figure 4.

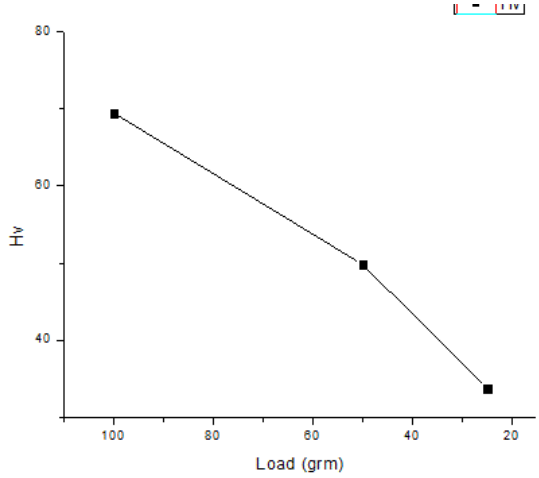


Figure 4: hardness graph for KDP DOPED CONGO RED DYE

E. FTIR ANALYSIS

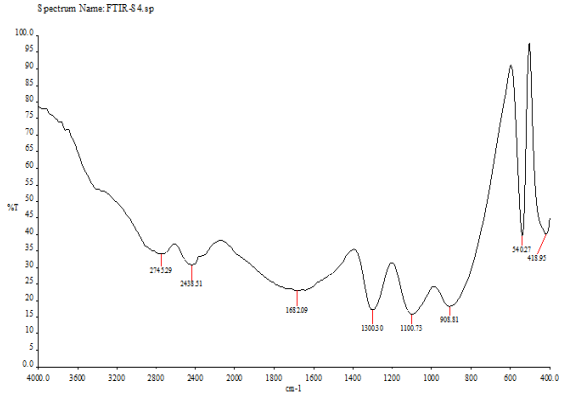


Figure 5 FTIR graph for KDP DOPED CONGO RED DYE

The FTIR was recorded in the frequency range of 400 - 4000 cm^{-1} to identify the functional groups in KDP doped Congo red dye was shown in figure 5. The KBr pellet method was used to analyze the sample. The various functional groups present in the grown crystal were identified. The observations of 1300 cm^{-1} and 908 cm^{-1} confirms that P=O stretching , the peak 1682cm^{-1} due to N-H bending of the dopant and 540 cm^{-1} due to P-O-H stretching of the grown crystal.[17-20]

F. THERMAL ANALYSIS

The thermogravimetric analysis(TGA) and differential thermal analysis (DTA) were carried out at heating 20ºC/min in air to determine the thermal stability of the compound.[21-22]. The thermogram curve of KDP doped Congo red dye shows that the weight loss of about 4.7% takes place at 150ºC and 93.5% at 200ºC. There is no weight loss up to 150ºC. In the DTA curve the first endothermic peak has been observed at 188ºC. There is no endothermic or exothermic peak up to 350ºC in the DTA curve.[23].

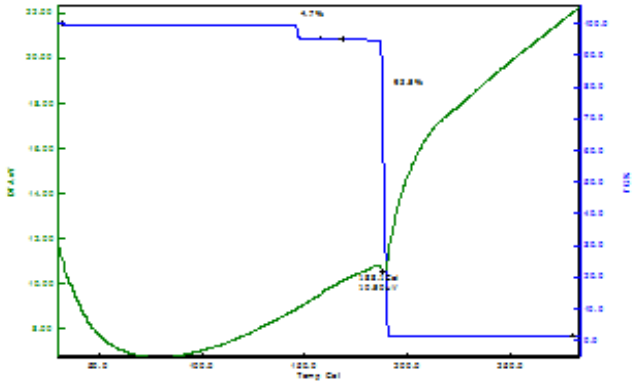


Figure 6: TGA/DTA graph of KDP doped Congo red dye

IV. CONCLUSION

Potassium dihydrogen Phosphate doped Congo red dye crystal was grown by solution evaporation technique. From the results of single crystal XRD, the cell parameters are observed that the grown crystal belongs to triclinic structure. The Fourier transforminfra-red spectroscopy studies carried out it confirms the functional groups of dopant present in the grown crystal. The UV-Vis studies reveals that the grown crystals having transmission in the visible range. Then the grown crystals subjected to Kurtz Perry powder method to test the efficiency of the relative second harmonic generation it reveals the NLO property of the grown crystal and the results compared to standard KDP. The micro hardness of the crystal was tested by Vickers hardness tester it shows the crystal hardness increased for various loads. The TGA/DTA reveals that this compound is stable up to its melting point.

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