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CRYSTAL GROWTH, PXRD, UV VIS AND FTIR STUDIES OF ADP SINGLE CRYSTALS

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ABSTRACT

Pure ammonium dihydrogen orthophosphate (ADP) single crystals have been grown at room temperature by the slow evaporation method. The grown crystals have been characterized structurally, spectral and optically using the available standard methods. The powder X-ray diffraction measurement confirms the crystal structures. UU-Vis and FTIR studies shows that the crystal has low cutoff wavelength and presence of various band assignments in the sample.

Keywords: ADP crystal, Crystal growth, Doped crystals, Physical properties, X-ray diffraction.

1.INTRODUCTION

Ammonium di-hydrogen phosphate (ADP) is a well known NLO material which also has many interesting Ferroelectric and Ant ferroelectric properties. ADP was one among the earliest materials which were exploited for their NLO properties. They are still widely used as nonlinear optic devices and one choicest electro-optic materials having wide practical applications. The nuclear fusion experiment require a NLO crystal of large second harmonic generation (SHG) efficiency, thermal and mechanical stability, high laser damage. Lot of research work has been devoted to improve the properties of ADP by adding different impurities or altering growth conditions like change of pH and lowering temperature with different rates etc. Presence of small amount of impurities in the form of anionic dopant K + and Na + plays a vital role in the growth rate, habit of the crystal and its properties of crystals. ADP and KCl doped ADP crystals were grown by solution growth technique by slow evaporation technique at 32^0C, KCl doped as 1,2,3, and 4 M% in ADP crystals. The grown crystals were subjected to different types of characterizations.

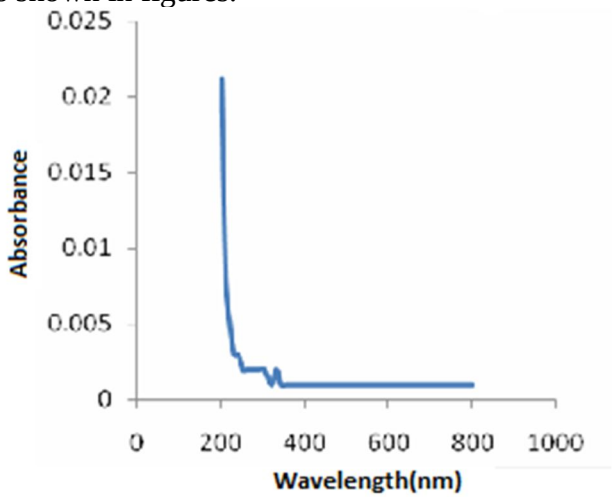
Synthesis of ADP crystal

Analytical reagent grade samples of ADP with double distilled water were used for the growth of single crystals. 8 gm of ADP is dissolved in 30 ml double distilled water. The solutions are mixed to get ADP 1M% solution. The solution is stirred using magnetic stirrer at 33^0C for four hours to get homogenous solution. The solution was filtered it using whatman filter paper kept beaker of solution in a constant temperature bath

by slow evaporation technique at 32^0C in about 20 days.

UV-Visible spectroscopy

The UV- Visible spectrum of KCl doped ADP crystals was recorded using UV-Visible spectrometer using SHIMADZU UV-160. The transparent behaviour of ADP in the entire UV-Visible spectrum shown in figure. Good transparency in UV-visible region is due to the delocalization of electrons of bonded oxygen long P=O which is expected to largely destroy the double bond character. This behavior may enhance three dimensional bonding interaction of phosphate with neighbouring units in the crystal. The less electronegativity of potassium is also important in strengthening such interactions. The recorded optical absorption spectrum of the grown crystals in wavelength range (200-400nm) is shown in figures.

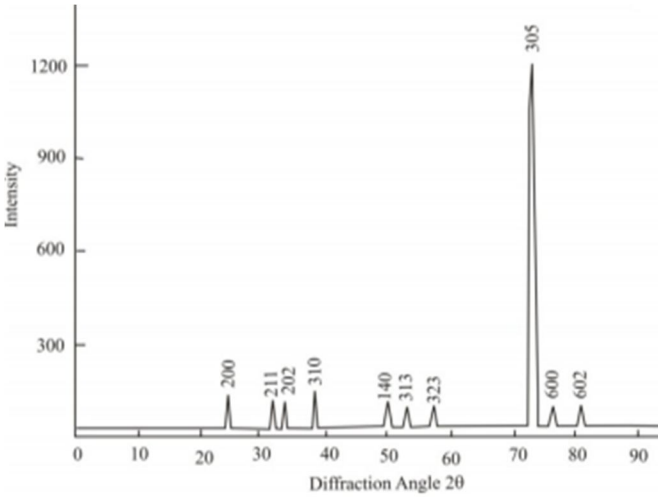


UV Vis spectrum of ADP crystal

It is inferred from the spectrum that the grown crystals have low absorbance in entire UV-Visible region considered and the cut off wavelength is around 230nm for 1M% . The presence of low cut off wavelength and the wide optical transmission window range are the most desirous properties of materials possessing NLO activity.

Powder XRD studies

Structural Studies (XRD) The crystallographic structure and lattice parameters of grown ADP single crystals were determined from the X-ray diffraction pattern obtained employing X-ray diffractometer. The diffraction peaks of the XRD patterns shown in Figure, could be indexed as those of the ADP with tetragonal structure (JCPDS Card No.37-1479). The XRD peaks were indexed and crystallographic lattice parameters were determined by powder-X software. The determined lattice parameters are a = 7.502 °A and c = 7.554 °A having space group 42 .The lattice parameters are in good agreement with the reported values.

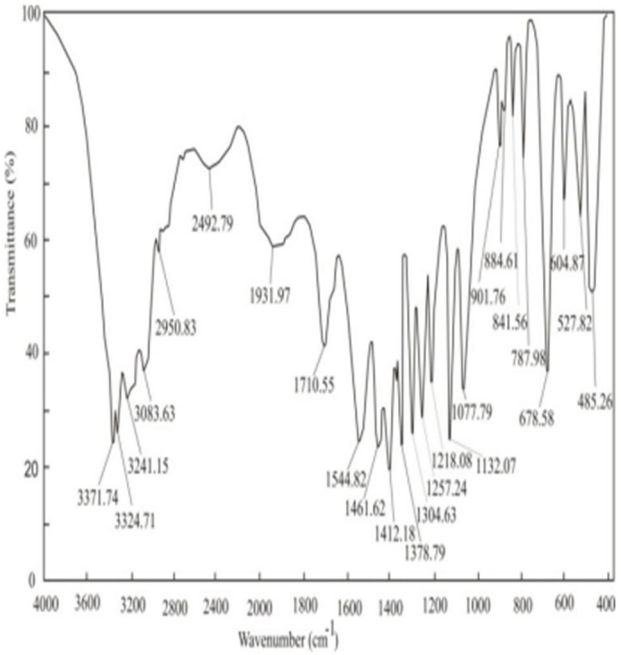


PXRD spectrum of ADP crystal

FTIR Studies

Fourier Transform Infrared Spectroscopy (FTIR) The functional groups of pure ADP crystals involved in vibration frequency have been identified using FTIR spectroscopy. Figure. FTIR spectrum of ADP single crystal The gel grown ADP FTIR spectrum was taken between wave-number 400 to 4000 cm-1, shown in Figure. The peaks between 485 to 902 cm-1 are due to the O-N=P and –ONO2 bond vibration in ADP crystal. Whereas P=O and O-H are responsible for the peaks in the wave-number

range, 1076 to 1544 cm-1. In the wave-number range from 2400 to 3371 cm-1, the numbers of peaks are lesser than the above two wave-number ranges. These peaks are due to O-H and N-H stretching.



FTIR Spectrum of ADP Crystal

3.CONCLUSION

The good quality of a non linear optical ADP crystal was grown by slow evaporation at ambient temperature. Colourless and transparent crystals were obtained. The crystals structure was found to be tetragonal from the powder XRD results and also calculated lattice parameters values are a = 7.502 °A and c = 7.554 °A . From the results of Fourier transform infrared spectrum various functional group present in the crystal were identified. From the UV-Vis spectrum the band energy of the doped ADP was calculated to be 4.7 eV suggesting a positive NLO activity. The crystal has a wide transmission window ranging from 300 to 1100 nm. This crystal is suitable candidate for non linear optics and optoelectronics field.

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