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"Growth and Characterization of KCl doped ADP single crystal"

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Abstract: *Crystals are the unacknowledged pillars of modern technology. It is an interdisciplinary subject covering physics, chemistry, materials science, chemical engineering, metallurgy, crystallography, mineralogy etc. In the past few decades, there has been a growing interest on crystal growth processes, particularly in view of the increasing demand of materials for technological applications. Among different crystals ammonium dihydrogen phosphate (ADP) has gained considerable importance because of its nonlinear, ferroelectric, piezoelectric and electro-optical properties. In this research work, study the effect of KCl on ADP single crystals both pure and KCl doped (dopant 0.10M, 0.15M) ADP single crystals are grown successfully from aqueous solution by slow evaporation technique. The optical properties of both pure and KCl doped ADP are measured by UV-Visible transmittance spectra. The thermal properties of both pure and KCl doped ADP crystals are investigated by thermo gravimetric analysis (TGA) from temperature of 22^0C to 600^0C.The grown crystals are found to be stable and transparent. From UV-Visible spectra of pure and KCl doped crystals it is found the doped crystals have enhanced the transmittance. The UV cut off wavelength of all crystals are found to be about 360 nm. TGA spectra indicate that the materials exhibit multi-stage weight loss starting before 194.02°C, 184.55°C, 169.96°C for pure, 0.10 mole%, 0.15 mole% KCL doped ADP crystals, respectively.*

Key words: *Slow evaporation, Crystallization, UV-Visible spectra and TGA.*

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I. Introduction

ADP is widely used as the second, third and fourth harmonic generator of Nd: YAG and Nd: YLF lasers. Due to its good structural quality and UV transmission, mechanical properties, high mechanical strength [1-5] and laser damage threshold, fast response time and wide phase match able angle. The crystal is widely used for electro-optical applications such as Q- switch for Nd: YAG, Nd: YLF and Ti-sapphire lasers as well as for acoustic-optical applications [6-8] Single crystals of ADP are used for frequency doubling and tripling of laser system [6]. Nowadays, crystals are produced artificially to satisfy the needs of science, technology and jewellery. The ability to grow high quality crystals has become an essential criterion for the competitiveness of nations. Crystal laboratories operate in large numbers to satisfy the needs of research and technology for high-quality, tailor-made crystals of all kinds. Electronic industry, photonic industry, fiber optic communications depend on materials/crystals such as semiconductors, superconductors, polarizers, transducers, radiation detectors, ultrasonic amplifiers, ferrites, magnetic garnets, solid state lasers, non-linear optics, piezo-electric, electro-optic, acousto-optic, photosensitive, refractory of different grades, crystalline films for microelectronics and computer industries. Hence, in order to achieve high performance from the device, good quality single crystals are needed. The reason for growing single crystals is, many physical properties of solids are obscured or complicated by the effect of grain boundaries. The chief advantages are the anisotropy, uniformity of composition and the absence of boundaries between individual grains, which are inevitably present in polycrystalline materials. Growth of single crystals and their characterization towards device fabrication have assumed great impetus due to their importance for both academic as well as applied research. An attempt has been made modify ADP crystal by controlling crystal growth parameter like temperature and growth rate etc. A simple localized- bond charge model for the calculation of nonlinear optical susceptibility is presented as per it there are three important contribution to nonlinearity namely the bond ionicity, the difference in atomic radii of the bonded atoms and d-electrons contributions [9], e.g. ZnS, ZnOBeO, CuCl, $AlPO_4$, NH_4PO_4 and KH_2PO_4 these crystals are highly anisotropic. Higher efficiency of frequency conversion can be achieved by growing good quality crystal and increasing the intensity of the input signal.

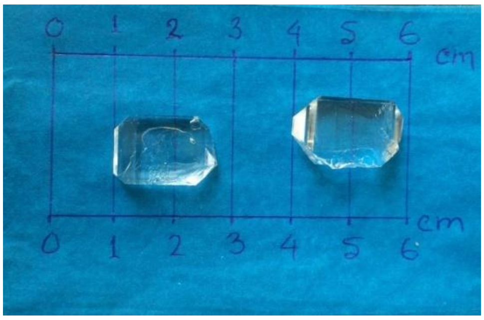
In this research an attempt has been made to grow large size optically transparent and mechanically strong and well faced shape of the pure and KCl doped ADP crystals by slow evaporation solution growth technique and to see the effects of KCl into the pure ADP crystals. In order to improve their optical properties of ADP for optoelectronic devices, KCl is added in different concentrations (0.10 mole%, 0.15 mole %). The grown crystals are characterized by UV-Visible and TGA. Then the influence of KCl on the growth kinetics as well as on structural, optical, mechanical characteristics are investigated and all these results are compared with pure ADP crystal.

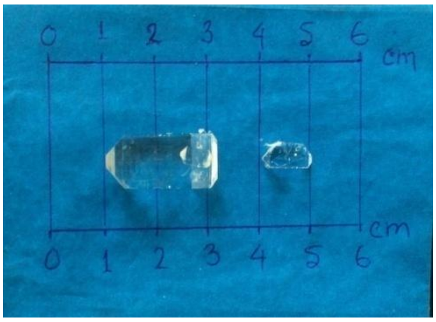
II. Materials and Methods

A Single crystal of pure ADP was grown from the saturated solution at room temperature by slow evaporation method using AR grade samples of ADP. Solution is stirred for one hour using magnetic stirrer and filtered using Whitman filter paper. The filtered solution was transferred to borosil glass beaker; it was kept opened and was kept in a constant temperature bath in a dust free atmosphere. In similar procedure 0.1 mole% and 0.15 mole% KCl doped ADP solution was prepared. After fifteen days good quality single crystals were obtained. All the crystals were colorless, stable and transparent.

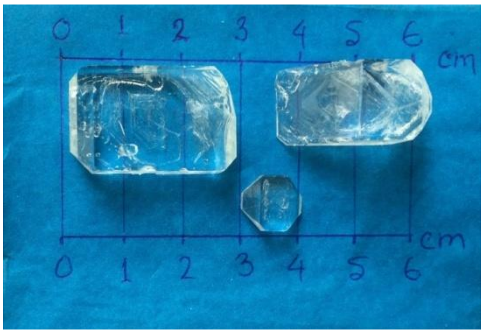
III. Results and Discussion

Good quality single crystals of pure and doped ADP are grown from solution. The grown crystals are found to be transparent for low concentrations (0.10 mole %, 0.15 mole %). All the grown crystals have been observed to be tetragonal shape. The photographs of these crystals are taken by putting the crystals on the colored paper which are shown in Figure.





(a) (b)



(c)

Photographs of (a) pure ADP crystal (b) 0.10 mole% (c) 0.15 mole% KCl doped ADP crystals.

3.1 UV-Vis spectra of sample crystals

The optical transmission spectra are recorded for both pure and KCl doped ADP using a spectrophotometer with a medium scan speed having a single scan mode and sampling interval 20 in the wavelength range 200 to 1100 nm. Samples of 1.5 mm thickness are used for the measurement.

The transmittance spectra obtained for both pure and KCl doped ADP crystals (of different concentrations) in the wavelength range 200 -1100 nm are represented in the following Figure-1.

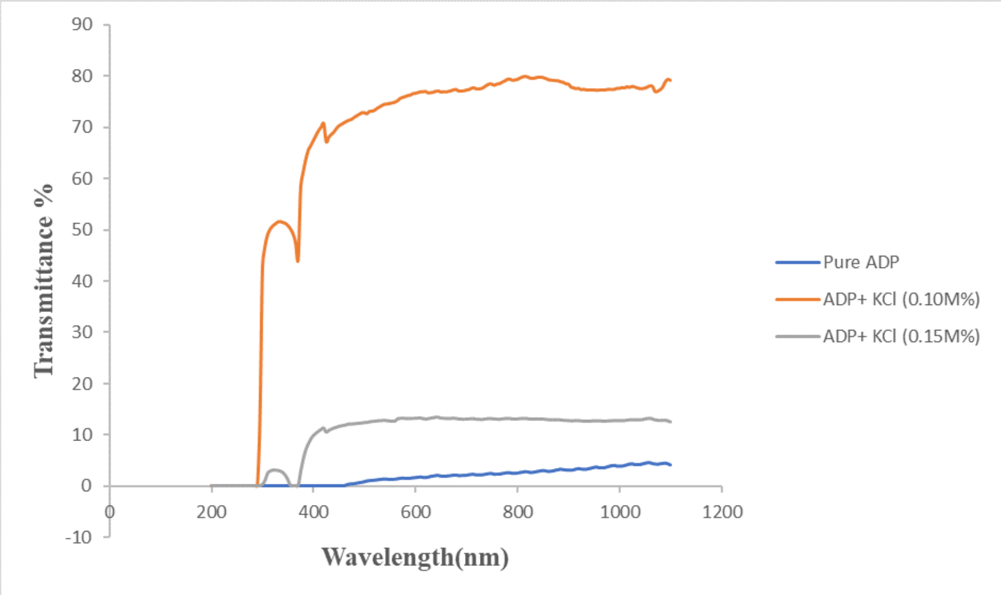


Figure-1: UV-Vis transmission spectra of both pure and KCl doped ADP single crystals.

UV-Vis spectra of pure ADP and KCl (0.10M%) doped crystals have found sufficient transmission in the entire visible region. KCl doped crystals enhance the transmittance about 75% from visible to infrared region. For ADP and KCl (0.15M%) drastically due to more molecules in the crystal. As a result more light is blocked. Around 360 nm a sharp fall in the transmittance to a very low value is observed for all the crystals. Doping of the crystals with KCl does not shift the lower cutoff value. The lower cut-off wavelength is around 360 nm. The band gap is found to be 3.4531eV by using formula, $E=\frac{hc}{\lambda}$, where λ is the lower cut-off wavelength. For wide band gap, the grown crystal has large transmittance in the visible region.

3.2 TGA measurement of sample crystals

The thermo gravimetric analysis (TGA) are performed for both pure and KCl doped ADP crystals using a TGA-50H thermal analyzer under nitrogen atmosphere heated from 21ºC to 600ºC. Aluminium cell was used and the analyses were performed at a temperature rate 10ºC per minute. The initial mass of pure ADP subjected to analyses was 8.295mg, for 0.10 mole% and 0.15 mole% the mass of KCl doped ADP subjected to analyses was 7.24 mg and 3.114 mg.

The TGA spectra recorded for both the pure and KCl doped ADP at different concentrations are shown in the following:

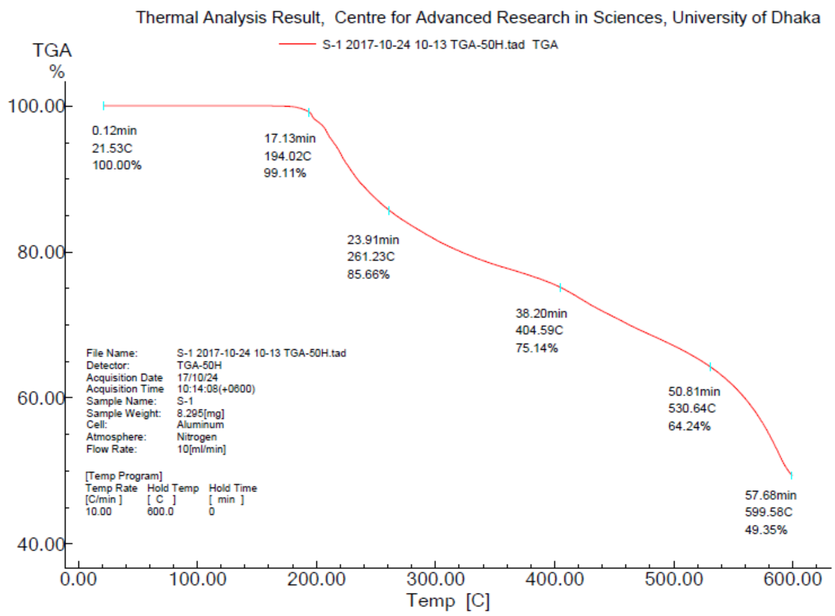


Figure A

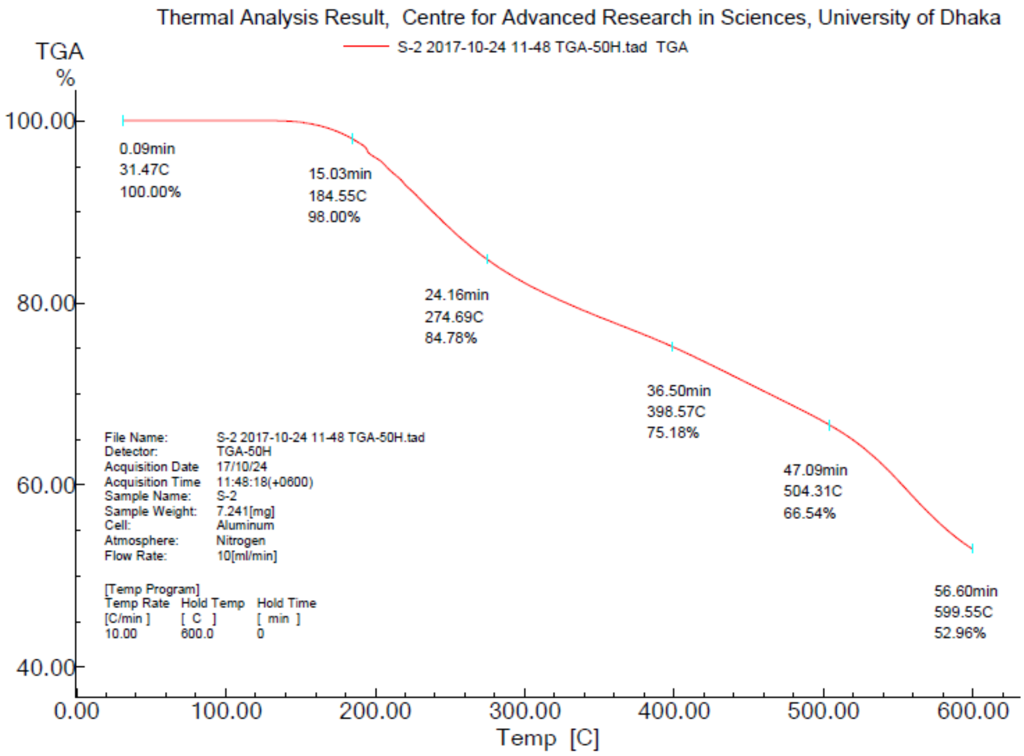


Figure B

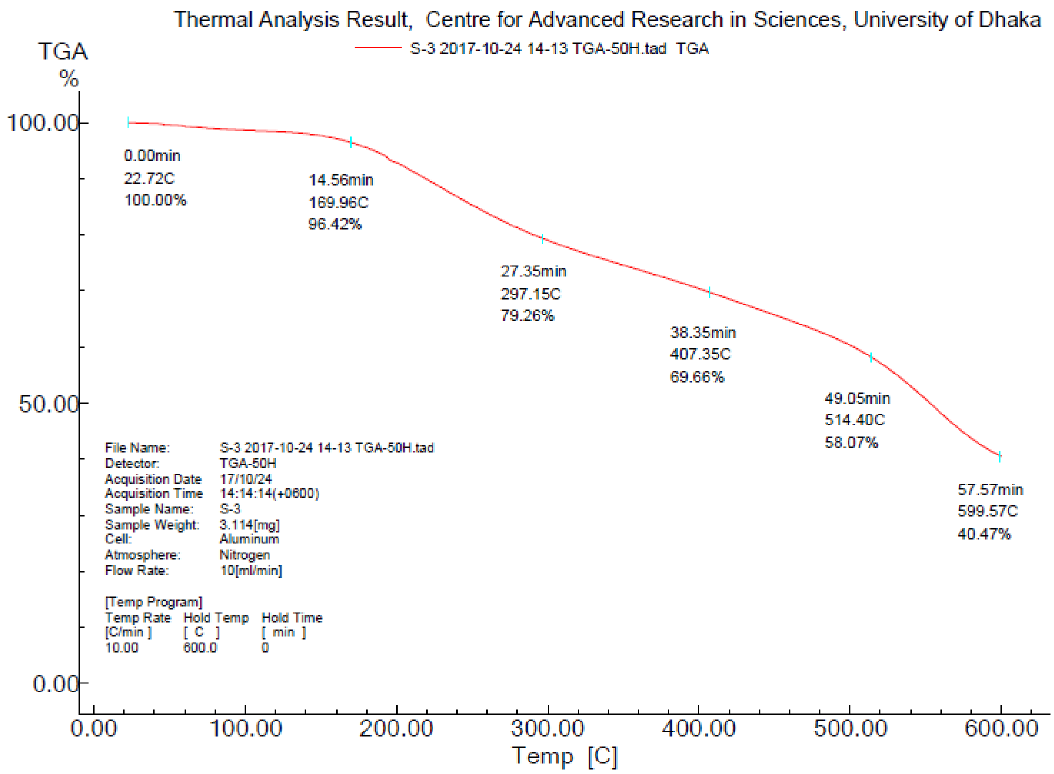


Figure C

Figure-2: TGA curves of (A) pure ADP (B) ADP+0.10% KCl (C) ADP+0.15% KCl

The thermo gravimetric analysis (TGA) indicates that the materials exhibits multi-stage weight loss for both the pure and KCl doped ADP crystals. Figure 2(A), 2(B) and 2(C) indicates no weight loss for pure ADP, ADP+10% KCl and ADP+15% KCl until 194.02^{0} C, 184.55°C and 169.96°C respectively. It proves that there is no insertion of water in the crystal lattice. It is also seen that the major weight loss starts at the same temperature for all crystals mentioned above. The result agreed with Jegatheesan Amirthalingam et al. [10] who investigated TGA of Ammonium Dihydrogen Phosphate (ADP) single crystal. All TGA curves show four distinct weight losses which is due to decomposition of the material.

IV. Conclusions

Ammonium dihydrogen phosphate (ADP) is an excellent inorganic nonlinear optical material with different device applications. With an aim of discovering new useful materials for academic and industrial uses, an attempt has been made to grow ADP crystals by doping KCl with two different molar concentrations. Single crystal of pure ADP crystal has been grown from the aqueous solution by slow evaporation technique. It is observed that the grown crystals are stable and optically transparent. The grown crystals have been subjected to various characterizations studies like UVvisible, thermo gravimetric analysis (TGA).The UV-Vis spectra measurements point out that KCl doping increase the transmittance in the visible range. Moreover, the UV cut off wavelength of all crystals are found to be about 360 nm. In thermo gravimetric analysis (TGA) the materials exhibits multi-stage weight loss because of the decomposition of the material for both the pure and KCl doped ADP crystals. Thus, the outcomes of this research will be helpful in future to grow large single crystals for industrial and research purposes.

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