International Journal of Engineering and Technical Research (IJETR)
ISSN: 2321-0869, Volume-2, Issue-8, August 2014

Growth and characterization of pure ADP crystals with and without the influence of Ultrasound

B. Kanickairaj, Dr. I. Johnson

***Abstract*— Pure Ammonium Dihydrogen Phosphate (ADP) crystals have been grown with and without the influence of Ultrasound by slow evaporation method. This work compares the crystal growth process and its properties due to the influence of Ultrasound. The obtained Nano crystal is having grain size of 150 – 300 nm. The grown crystals have been characterized by using XRD, FTIR, UV and Micro hardness. Remarkable changes in properties were observed due to irradiation of Ultrasound of frequency 12 MHz while growing the crystal.**

***Index Terms*— ADP, FTIR, UV, XRD.**

I. INTRODUCTION

Ammonium Dihydrogen Phosphate (ADP) is a powder with relative density 1.80, melting point 190℃ and refraction index of 1.525. **It** is stable in atmosphere and decomposed into ammonium metaphosphate (NH4PO3) while heated. **It** is slightly soluble in ethanol and easily soluble in water. ADP crystal is widely used in the field of optics due to its birefringence properties. It is in tetragonal crystal structure, therefore this material has negative uniaxial optical symmetry with typical refractive indices n_o =1.522 and n_e = 1.478 at optical wavelengths[1]. ADP crystals are piezoelectric, a property required in some active sonar transducers. Slow evaporation method is an easy one to grow ADP crystals[2]. The NLO properties of this crystal prove that it can be used in optical equipment like optical amplifier and modulator. The influence of ultrasound not only reduces the growing time but it improves properties like visibility and hardness[2]-[6].

II. METHODS AND MATERIALS

Ammonium Dihydrogen Phosphate was dissolved in 100 ml water little by little in terms of 0.5 g. and it is stirred with a magnetic stirrer. The saturated solution was obtained at 46.219 g and filtered. The filtered solution is divided in to two 50 ml parts in two beakers. One part of the solution is allowed to evaporate under room temperature by covering the top of the beaker with perforated foil. Ultrasonic wave of frequency 12 MHz from microcontroller based ultrasonic generator constructed by us was irradiated over the other part of the solution while allowed to evaporate until the crystal formed. The grown crystals were washed with distilled water and dried. The growing time is very much reduced in the solution under the ultrasonic wave. The crystal is grown in the solution with the exposure of the ultrasound within 3 days whereas it took 7 to 10 days without the exposure of ultrasound. The grown crystals have been characterised by XRD, FTIR, UV visible and Micro hardness.

A. *Characterization*

The mid Fourier transform infrared spectrum has been recorded in the range of 450 - 4000 cm^{-1}. Grown crystals were crushed and few mg of the powder was used for making pellets with KBr. The pellets were then exposed to IR radiation. The spectrum was recorded by using RXI.FTIR spectrometer. The UV spectrum was obtained using LAMBDA-35 UV visible spectrometer. The mechanical hardness was evaluated for crystals using Vicker's hardness tester with Vicker's diamond pyramidal indenter. Powder X-ray diffraction was performed using automated X-ray diffractometer with a CuKα radiation (λ = 1.5418Å) in a range of 2θ from 10° to 80°.

III. RESULTS AND DISCUSSION

A. *FTIR analysis*

The FTIR spectra were taken from the Perkin Elmer RXI.FTIR spectrometer in the range of 450 – 4000 cm^{-1}. The spectrographs are shown in fig. 1. and fig.2.

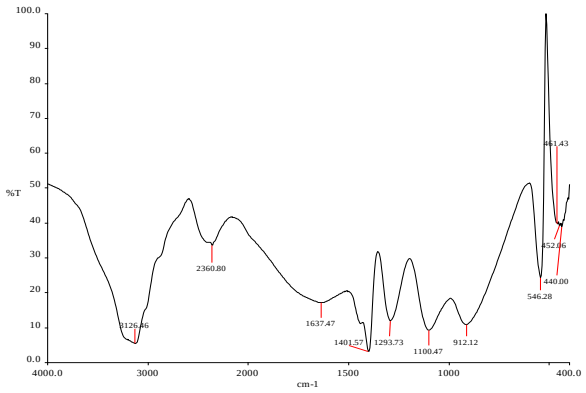

Fig.1. FTIR spectrum of Pure ADP with ultrasonic ittadiation

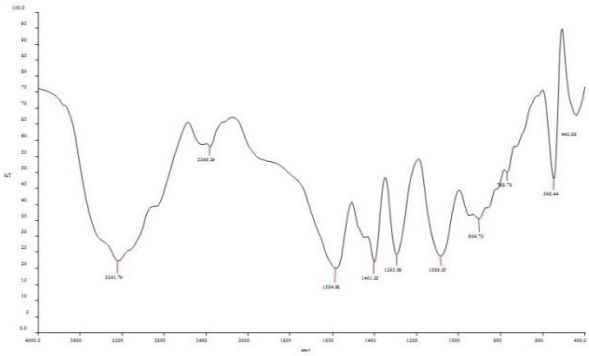

Fig.2. FTIR spectrum of Pure ADP without ultrasonic irradiation

Manuscript received August 21, 2014.

B. Kanickairaj, Research Scholar, Department of Physics, St. Joseph's College (Autonomous), Trichy, India.

Dr. I. Johnson, Department of Physics, St. Joseph's College (Autonomous), Trichy, India.

By comparing the peaks obtained from the spectrum with standard values, it is clear that 1637.47, 1100.47, 1584.98, 1086.55 correspond to the peaks of ADP[6,7]. It also closely matches with literature values. Therefore it confirms the presence of ADP.

B. Optical studies

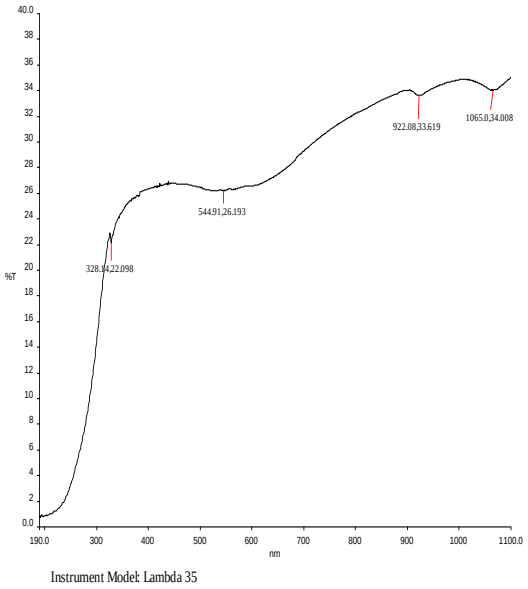

Fig.3. UV spectrum of Pure ADP with Ultrasound

The UV spectra were taken by using LAMBDA 35 UV spectrometer.

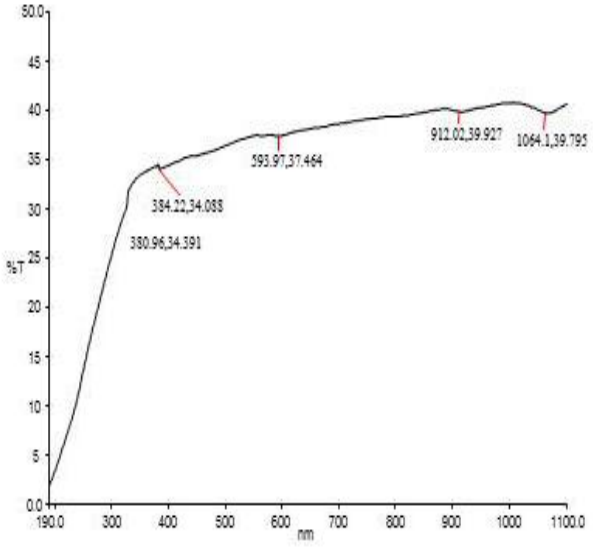

Fig.4. UV spectrum of Pure ADP without Ultrasound

From the above graphs it is evident that the crystal grown under the influence of ultrasound has the UV transparency above 328.14 - 1100 nm whereas the other above 384.22 – 1100 nm. The peek absorption is observed at 196.33 nm. The energy band gap of the crystal grown under the influence of ultrasound is 3.78eV and of the crystal without the influence of ultrasound is 3,23 eV, which is calculated using simple equation E = hv at transparency begins.

C. Micro Hardness Studies

The mechanical strength of the crystal was studied with different loads. The hardness values were recorded.

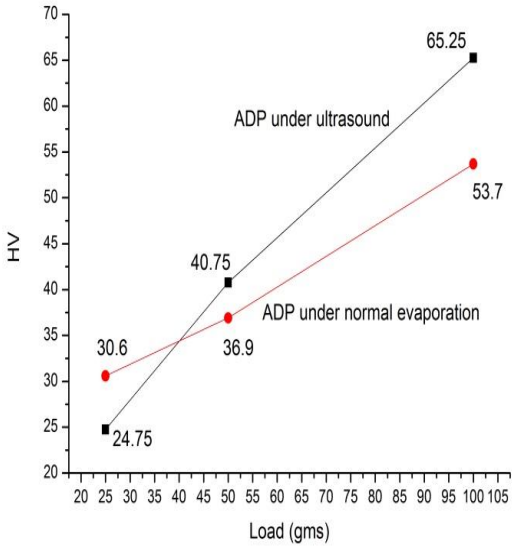

Fig.5. Micro hardness graph

From the above graph it is clear that the hardness value (HV) increases with load which confirms that the crystals are of hard nature. But for the crystal grown under the influence of ultrasound, the HV varies between 24.75 – 65.25 implies stronger. For low values of load the influence of ultrasound makes the crystal soften while it makes the crystal harder with increase in load (especially after 38 gms)

D. XRD Analysis

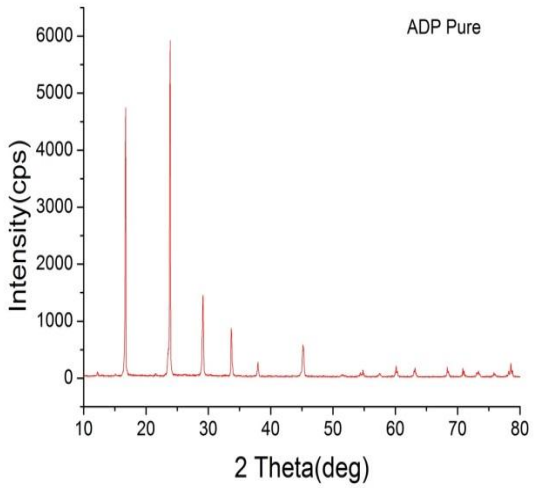

Fig.6. XRD pattern of Pure ADP with ultrasonic irradiation

X-ray diffraction spectra using CuKα wavelength (λ=1.54059 Å) were recorded and is shown in fig 6. and Fig.7. From both graphs the peaks are obtained at almost same 2θ (deg). The unit cell parameters are a=b=10.629 Å, c=7.500 Å. Since a=b ≠ c it proves that the crystal structure is tetragonal. The sharp peeks in fig.6. proves that the grown material is a crystal.

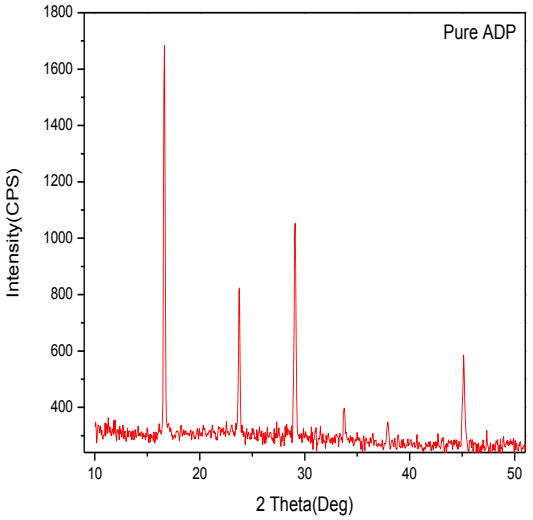

Fig.7. XRD pattern of Pure ADP without ultrasonic irradiation

IV. CONCLUSION

Pure ADP crystals were grown successfully with and without the influence of ultrasonic waves of frequency 12MHz. From the FTIR analysis the presence of ADP is confirmed. The UV spectrum shows transparency over entire visible spectrum. The vicker's hardness test proves that they are strong. So the influence of ultrasound tailors the properties of the crystal.

REFERENCES

[1] Amnon Yariv, Pochi Yeh (1984). *Optical Waves in Crystals*. Wiley, Inc.
[2] "Influence of ultrasonic vibrations on the growth of semiconductor single crystals", Original Research Article Ultrasonics, Volume 35, Issue 8, January 1998, Pages 599-604
[3] "Effects of ultrasonic waves on crystal growth", Original Research Article Journal of Crystal Growth, Volume 62, Issue 3, August 1983, Pages 458-464
[4] "Influence of Ultrasound on Crystal Growth from Solution and Related Flow Visualization Crystal Growth & Design", 2006, 6 (10), pp 2412–2416 DOI: 10.1021/cg0499313, Publication Date (Web): September 16, 2006 Copyright © 2006 American Chemical Society
[5] G. N. Kozhemyakin, "Effect of ultrasound on the growth of semiconductor single crystals", Crystallography Reports Volume 49, Number 2, 318-323, DOI: 10.1134/1.1690438
[6] Yoshiharu Murata, Kaoru Wada and Masakuni Matsuoka, "Effects of ultrasonic waves on crystal growth Department of Chemical Engineering", Tokyo University of Agriculture and Technology, Koganei, Tokyo 184, Japan Received 25 November 1982; accepted 22 February 1983. Available online 30 July 2002.
[7] Russel S Drago, "Physical Method in Inorganic Chemistry"(Chapter 7), East-West Press Pvt. Ltd, New Delhi (1968).

First Author is a Research Scholar doing PhD in design of an automated device for Nano crystal powder.

Second Author is Research adviser in Physics, St. Joseph's College (Autonomous), Trichy. He has published more than 50 research papers