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Studies On Growth And Characterization Of Potassium Diammonium Chloride (PADC) Single Crystal By Solution Growth Technique

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Abstract:- *Potassium diammonium chloride (PDAC), an inorganic single crystal with dimensions of 3 x 3 x 2 mm^3 has been grown by slow solvent evaporation technique using water as a solvent. FTIR analysis explains the various functional groups present in the sample. The mechanical property of grown crystal was studied by Vicker's hardness test. The EDAX spectrum was used to investigate the presence of element in the title compound. The dielectric measurements on the PDAC crystal were carried out and the variation of dielectric constant and dielectric loss at different frequencies of the applied field has been studied. The results are discussed in elaborate.*

Keywords: *Slow evaporation technique, FTIR analysis, Vicker's microhardness test, dielectric measurement.*

I. Introduction

Nonlinear optical materials will be the key elements for future photonic technologies based on the fact that photons are capable of processing information with the speed of light. Inorganic materials are much more matured in their application to second order NLO materials than organics. Most commercial materials are inorganics especially, for high power use. In the present work, investigates the crystal growth and characterization of PDAC crystals. The grown crystal has been subjected to FTIR analysis, microhardness test, elemental and dielectric studies.

II. Experimental Procedure

Potassium Diammonium Chloride (PDAC) single crystal was grown by the solution growth method with slow solvent evaporation technique. Analar grade potassium hydroxide and ammonium chloride in stoichiometric ratio 1:2 in double distilled water and stirred well at room temperature for about 3 hours using magnetic stirrer to yield a homogeneous mixture of solution. The adduct is formed according to the reaction,

$$KOH + 2NH_4Cl \rightarrow (NH_2OH)_2\, KCl + HCl + 2H^+.$$

The prepared solution was filtered using WHATMAN filter paper to the impurities. The filtered solution was taken in vessel and closed with perforated covers and kept in dust free atmosphere. A good quality of optically transparent with clear morphology crystal of size 3 x 3 x 2 mm^3 was harvested in the growth period of 27 days and is shown in the fig.1.

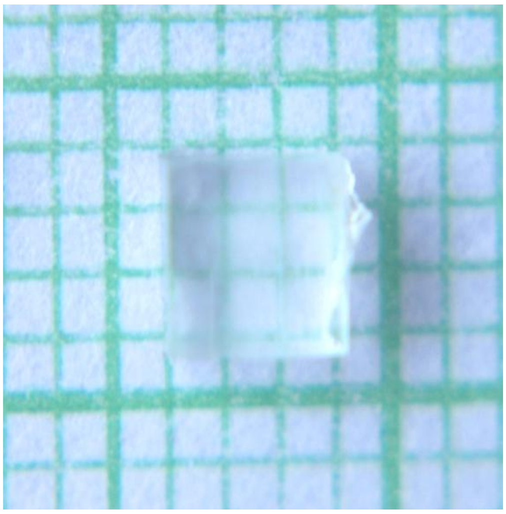

Fig.1: Photograph of PDAC crystal

III. Result and Discussions

3.1 Fourier Transform Infrared (FTIR) Analysis

FTIR is most useful for identifying chemicals that are either organic or inorganic. It can be utilized to quantify some components of an unknown mixture. In this study, FTIR spectrum was recorded in the region 4000 – 400 cm^{-1} by using PERKIN ELMER FTIR spectrometer with KBr pellet technique. The recorded FTIR spectrum of PDAC is shown in the fig.2. The peak observed at 2895 cm^{-1} due to O-H stretching mode vibration.. The band at 1700 cm^{-1} and 1363 cm^{-1} corresponds to the N-H stretching mode in the group of amine salts of ammonium and the aromatic compounds are present in the sample. The N-O stretching is observed in the wave number of 1220 cm^{-1}. The observed band at 680 cm^{-1} is assigned to the N-H out of plane bending vibration of amine group [1,2].

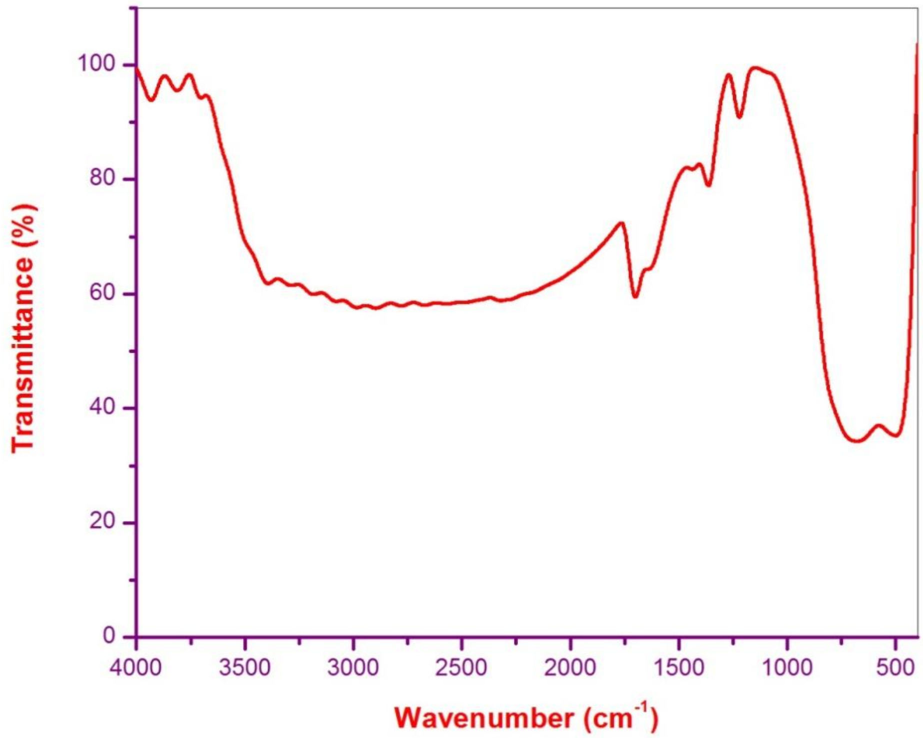

Fig.2. FTIR spectrum of PDAC crystal

3.2 Vickers Hardness Test

Hardness is a measure of materials resistance to localized plastic deformation. It plays key role in device fabrication [3]. The mechanical strength of the grown crystal was studied by using HMV 2T Vickers microhardness tester.For a varying load of 25, 50 and 100 g, the respective Vickers hardness number was measured. The Vickers hardness number (H_V) of PDAC crystal is determined using the relation,

$$H_V = \frac{1.854\,P}{d^2} \quad \text{Kg / mm}^2.$$

Where, P is applied load in Kg and‘d’ is applied length of diagonals in mm.

The calculated Vickers hardness number of PDAC crystal as a function of load was shown in fig.3. From the Fig.3, it is observed that hardness increases with the increase of load. The maximum hardness obtained in this material is 98 Kg / mm^2. In order to find the work hardening coefficient ‘n’ of the grown crystal, another graph was drawn between log P and log d as shown in fig.4. The slope of the graph equals the work hardening coefficient n = 1.15. According to Onitsch [4], n lies between 1 and 1.6 for hard materials and more than 1.6 for soft materials. The work hardening coefficient of grown crystal is 1.15 suggested that it comes under the category of hard material.

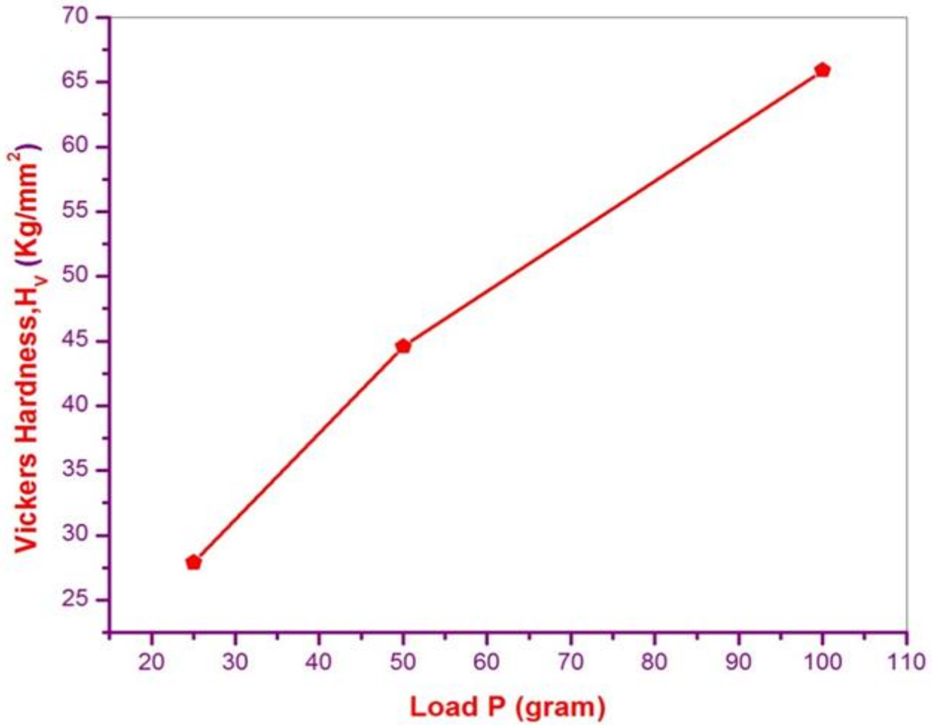

Fig.3. Load P vs Hardness Number (H_V)

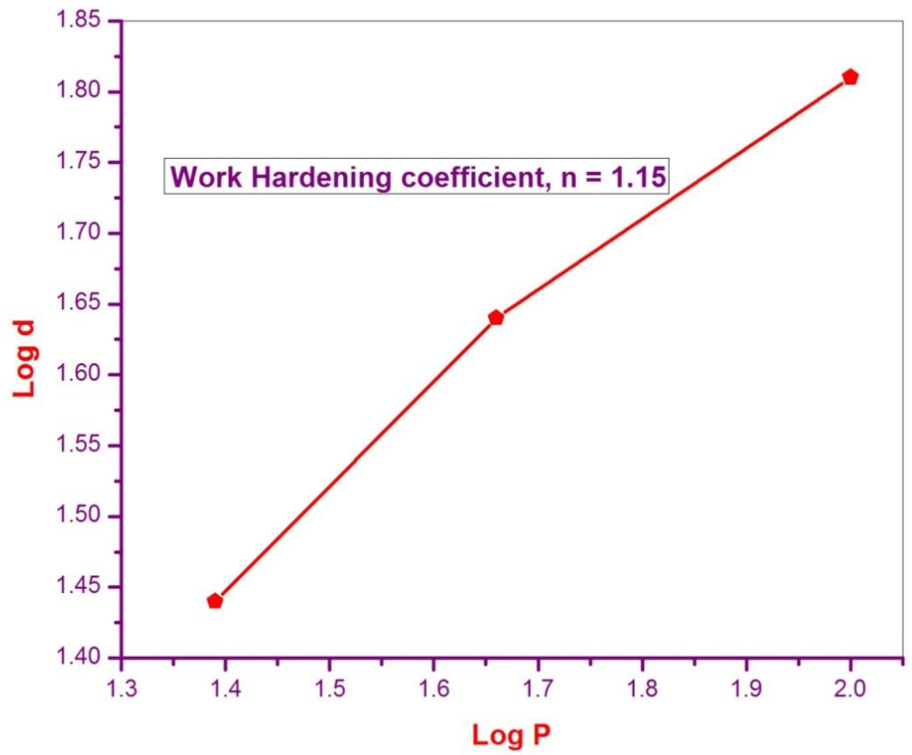

Fig.4. Plot of Log P Vs Log d

3.3 EDAX Analysis

The elemental analysis was carried out by using JEOL model JSD – 5610 LV Energy Dispersive X-ray Spectrometer with an accelerating voltage of 20 kV. The EDAX analysis of grown crystal is shown in fig.5. From the EDAX spectrum confirms the presence of potassium, nitrogen, oxygen and chlorine within the potassium diammonium chloride crystal. The elemental analysis data is given in the table. 1

Table.1. EDAX data of PDAC crystal

Element	Weight (%)	Atomic (%)
Cl	22.43	46.35
O	00.18	00.22
N	10.12	11.13
K	67.27	53.30
Total	**100.00**	**100.00**

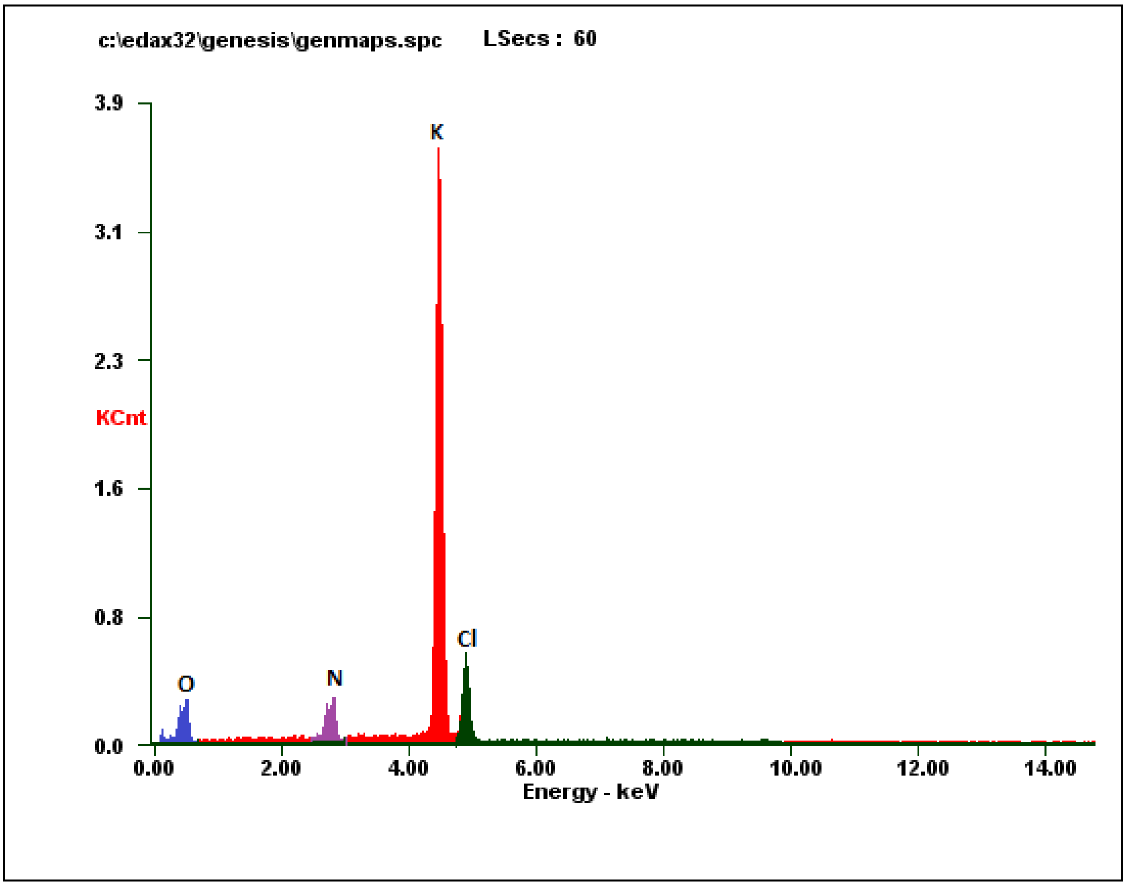

Fig.5 EDAX spectrum of PDAC crystal

3.4 Dielectric Studies

Potassium Diammonium chloride crystal was subjected to dielectric studies in the frequency range from 50 Hz to 5 MHz with a temperature range from 313 K to 393 K using LCR HIOKI 3532 HI TESTER which is capable of measuring different parameters such as capacitance and dissipation factor. The dielectric constant ε_r has been calculated using the equation,

$$\varepsilon_r = \frac{Ct}{A\varepsilon_0}$$

Where, $\varepsilon_0 = 8.854 \times 10^{-12}$ F/m is the permittivity of free space, C is the capacitance, t is the thickness of the sample and A is the area of the sample.

Fig.6 shows that the plots of dielectric constant versus log frequency for PDAC crystal from the graph it is observe that as the frequency increases the dielectric constant decreases and for high frequency region it remains almost constant for PDAC crystal. The high value of dielectric constant at low frequencies may be due to the contributions from all the four types of polarization namely electronic, ionic, orientational and space charge polarization and its low value at higher frequencies may be due to the loss of significance of these polarizations gradually[5,6].

The variation of dielectric loss with log frequency is shown in fig.7. From the plot it is observed that the value of dielectric loss decreases when increases in frequency. The characteristics of low dielectric loss with high frequency for a crystal suggested that the sample posses enhanced optical quality with lesser difference and this parameter is of vital importance in the field of opto- electronic devices[7,8].

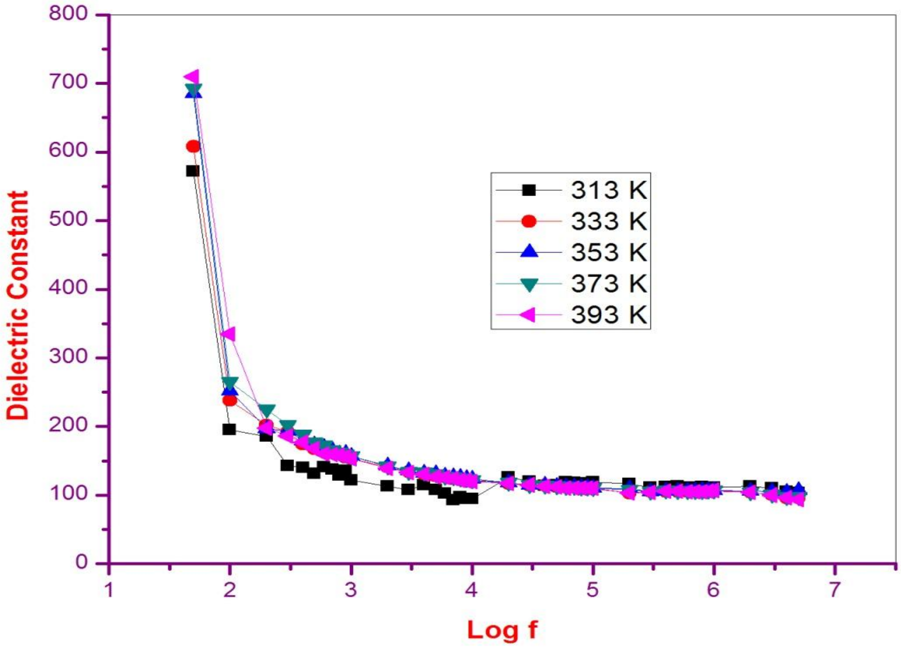

Fig.6. Variation of log frequency with Dielectric constant at different temperature

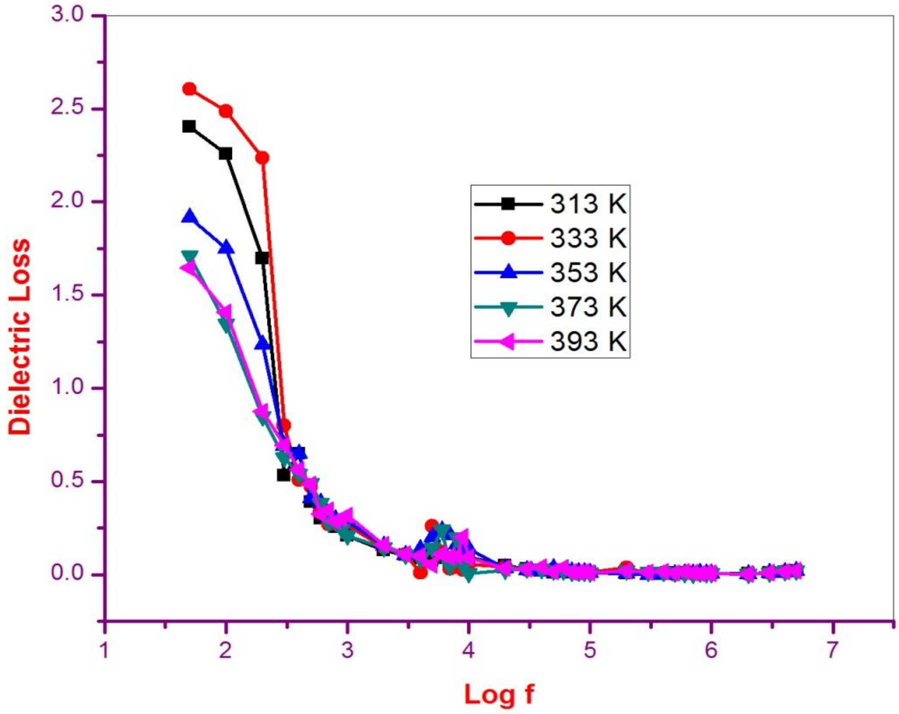

Fig.7. Variation of log frequency with Dielectric Loss at different temperature

CONCLUSION

PDAC single crystals have been grown from aqueous solution by slow evaporation technique. The bonding structure and molecular associations were analyzed by FTIR spectroscopy. Hardness study confirms that the grown material is harder one. The presence of potassium, nitrogen, oxygen and chloride within the specimen was evaluated using EDAX analysis. The low dielectric constant and dielectric loss of the PDAC crystal at high frequency region reveals the good optical quality. The above results shows that the potassium diammonium chloride crystal can be consider to a prospective material for nonlinear optical applications.

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