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GROWTH AND CHARACTERIZATION OF NLO MATERIAL: L-ALANINE POTASSIUM CHLORIDE

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ABSTRACT

Single crystal of alanine potassium chloride (APC), a semi organic nonlinear optical material has been grown from solution by slow evaporation at ambient temperature. The isoelectric point of L-alanine is 6(1). So, the growth of crystals has been carried out at pH 6. The chemical composition of the grown crystals was determined by the FTIR spectra. The crystalline nature and its various planes of reflections were observed by the powder XRD. The structure is built from alternate layers of alanine organic molecules and inorganic layers consisting of K^+ ions and Cl^- ions. The Hardness of the crystals was studied by Vickers micro hardness tester. Surface morphology was studied by SEM analysis. Using Nd-YAG laser the NLO property of the crystal is studied.The transmittance and absorption of the crystal was studied by UV-Visible spectrometer.

Keywords: Characterization, X-ray diffraction, Slow evaporation, SEM analysis.

INTRODUCTION

Non – linear optical materials (NLO) have wide applications in the area of laser technology, optical communication and in storage technology.

In recent years, organo – inorganic hybrid materials have attracted considerable attention. In particular, the inorganic derivatives of protein amino acids are often attributed to symmetric groups without an inversion center mostly to polar symmetric groups without an inversion center mostly to polar acids are well defined; the structures of the derivatives of the protein amino acids with inorganic components are not[2]. This paper defines the crystal structure of alanine potassium chloride (APC). This has been investigated by the FTIR studies, its crystalline nature is studied by the powder XRD, the transmittance and absorbance of electromagnetic radiation is studied through UV-Visible spectrum, Surface morphology was studied by using SEM studies.

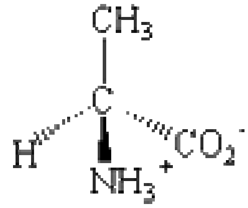
EXPERIMENTAL

Synthesis and crystal growth

The solution is prepared using L-alanine and potassium chloride in the molar ratio of 1:1. The pH value was high, so, to reduce the pH value. The pH of the solution was adjusted to 6 by adding 4 drops concentrated hydrochloric acid[3]. The above solution was filtered using the filter paper and transferred to a Petri dish. The Petri dish was covered with a filter paper with small hole, tied on top with rubber band to facilitate evaporation and crystal growth.

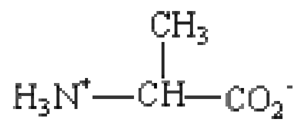
Structure of L-Alanine molecule

The α-carbon atom of L-alanine is bound with a methyl group ($-CH_3$), making it one of the simplest α-amino acids with respect to molecular structure and also resulting in L-alanine being classified as an aliphatic amino acid. The methyl group of L-alanine is non-reactive and is thus almost never directly involved in protein function. The structure of L-alanine is



L-alanine

Further its linear zwitter ionic structure is



The crystal structure of L-alanine is orthorhombic[4]. Its cell parameters are

a = 6.032 A^o, b= 12.343 A^o, c = 5.784 A^o
α=β= γ =90^o

Charatcerization

FT-IR Analysis(Fig.1)

The FT-IR Spectrum of alanine potassium chloride reveals the following fetch. 3085cm^{-1} with medium intensity represents C-H asymmetric stretching. 2934cm^{-1} with weak intensity shows OH stretching. The peak at 1620cm^{-1} with strong intensity represents C=O stretching.1455cm^{-1} with medium intensity represents C-H in plane bending[5]. The peak 1014cm^{-1} with variable represents C-CHO stretching. 772cm^{-1} strong band refers to C-H out of plane bending[6].

Powder XRD analysis(Fig.2)

The powder XRD of alanine potassium chloride is shown in the fig. The peaks in the fig show the crystalline nature of alanine potassium chloride. Further the peaks are indexed[7]. The cell parameters of alanine potassium chloride (APC) is

a=11.52746 A^o
b=15.70642 A^o
c=4.76734 A^o
V (volume of the crystal) = 841.44A^{o3}
α=96.6299^o β=98.6734^o γ =95.7711^o
The crystal system is Triclinic.

UV-Visible Spectrometer analysis (Fig.3)

The light of wavelength 193nm is absorbed. This is the cut of point. Then the crystal is highly transparent to the wavelengths above 193nm to 1500nm[8].

Crystal surface analysis by SEM (Fig.4)

Surface analysis of alanine potassium chloride is carried out through JSM 6360 JEOL/EO make. The surface of the crystal was coated with a thin of carbon to make the sample conducting. From the figure, the following observations are evident: 1.At a magnifications of 85 and at a scale of 200 micrometer we observe the crystals have smoothed rectangular surfaces. 2. At a magnifications of 2300 and 10 micrometer scale we can observe that the crystals have smoothed surfaces.3. At a magnifications of 3500 and at a scale of 5 micrometer. 4. At a magnification of 5500 and 2 micrometer scale we can observe that the crystals have an average thickness of 293.17nm[9].

Micro hardness(Fig.5)

APC crystal was subjected to Vickers micro hardness test with the load varying from 25 to 100g[10]. Hardness number of the crystal is calculated using the relation

$$Hv = 1.8544\ P/d^2\ Kg/mm^2$$

Vickers micro hardness profile as a function of the applied test loads is illustrated by fig. It is evident from the plot that the micro hardness of the crystal decreases with increasing the load. The value of the work hardening coefficient n was estimated from the plot of log p versus log d drawn by the least square fit method. It is observed that the Vickers hardness number of the crystal decreases with increasing the load[11]. The value of the work hardening coefficient n was found to be 0.08. According to Onitsch,$1.0 \le n \le 1.032$ for hard materials and $n > 1.032$ for soft materials[12]. Hence, it is concluded that APN belongs to the soft materials.

RESULTS AND DISCUSSION

The seed crystals of alanine potassium chloride (APC) are grown from the solution prepared from the raw materials L-alanine and potassium chloride of 99.99 percentage of purity. These crystals are characterized and the following results are obtained.

Single crystals of alanine potassium chloride are successfully grown at a pH of 6. The grown crystals are larger in size having an average size of 3cmx5cm.

1. From the FTIR spectrum we confirm the structure of the APC to have both the alanine and potassium chloride molecules. These arrangement in alternate layers in the crystal. This is evident from the non damage of alanine structure.
2. The grown crystals are characterized by using powder XRD diffraction. From this we confirm the structure of the crystal to be triclinic and the cell parameters: a=11.52746A^o b=15.70642A^o c=4.76734A^o .
3. From the UV-Visible spectrum we find the crystal is transparent in the range of 900-2500nm without any absorption peak.
4. From the SEM analysis we conclude that the crystal formation size in micro range is 293.17. Further in the micro level the crystal surface is very smooth which shows that it can add more molecules to grow in to a large crystal.
5. From the Vickers' micro hardness test we find the micro hardness number decreases with increasing the load. Further the value of the work hardening coefficient is found to be 1.032. From this result we conclude that the crystal is soft.
6. The NLO test using Nd-YAG laser confirms that the crystal has NLO property.

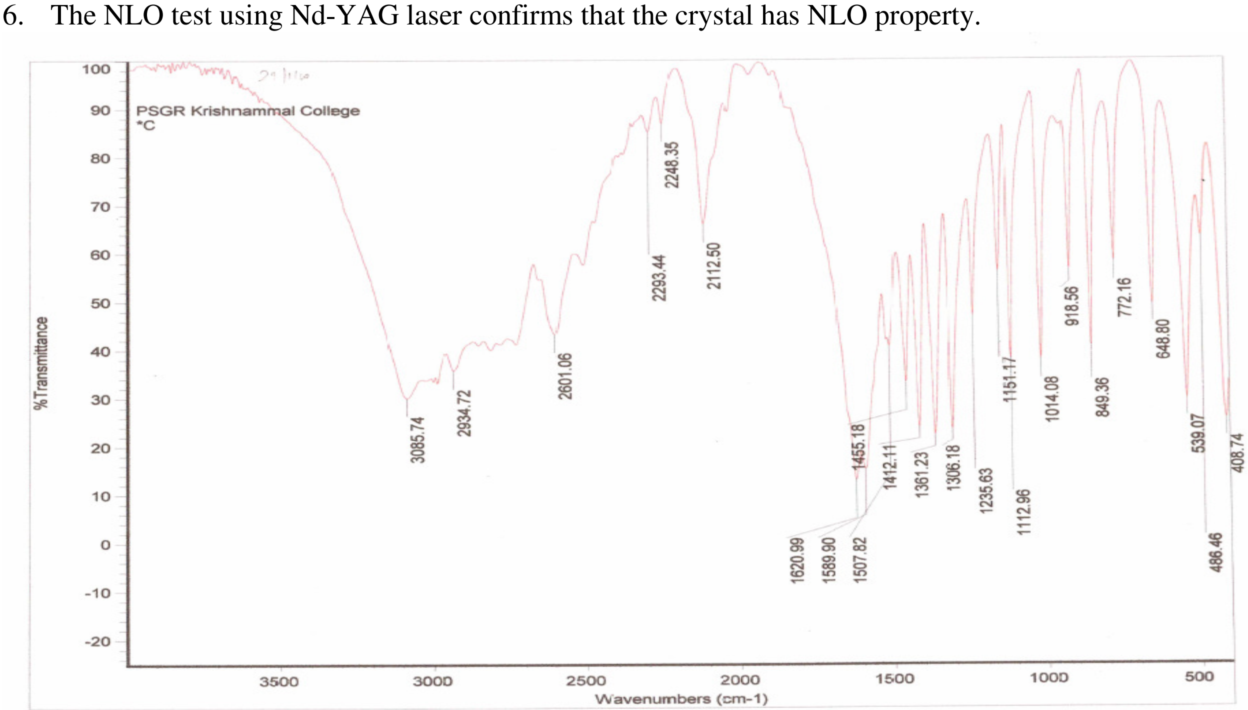


Fig.-1: FTIR Spectrum of Alanine Potassium Chloride (APC)

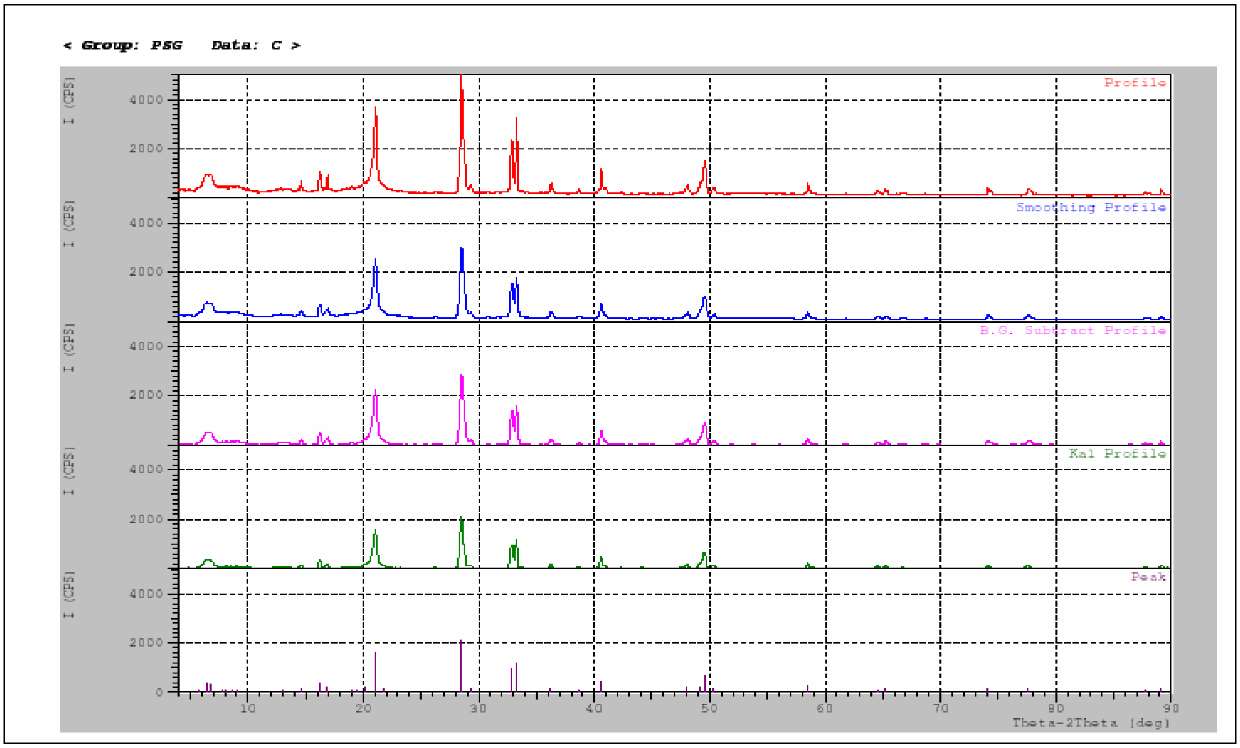


Fig.-2: Powder XRD pattern of Alanine Potassium Chloride (APC)

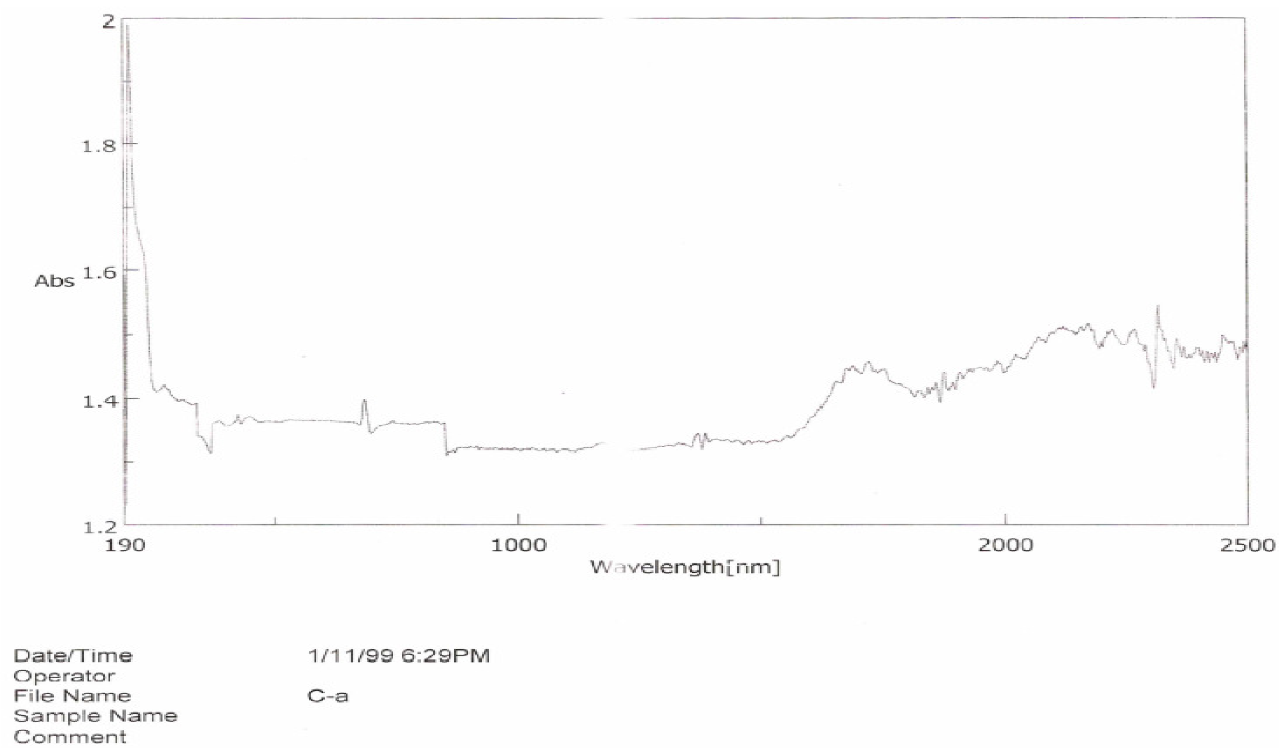


Fig.-3A: UV-Visible spectra of Alanine Potassium Chloride (APC)

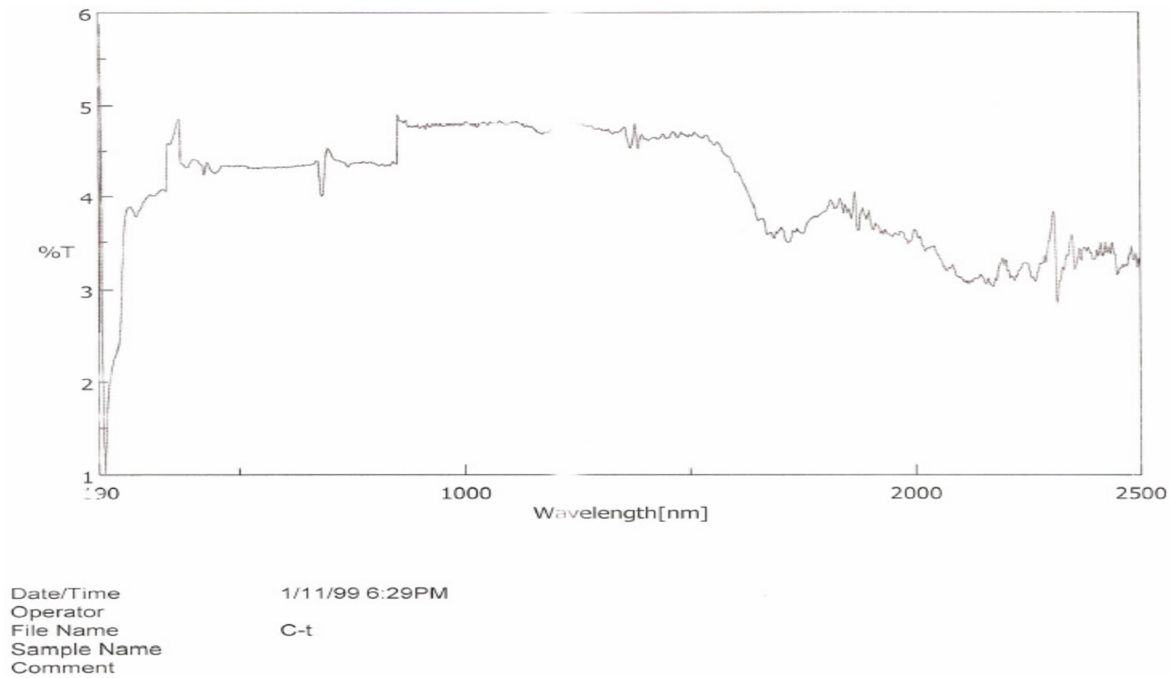


Fig.-3B: UV-Visible Spectra of Alanine Potassium Chloride (APC)

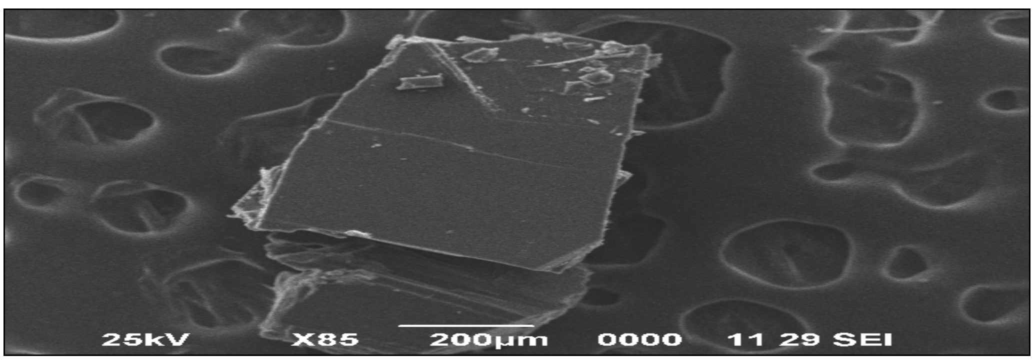


Fig.-4A: SEM Photograph of Alanine Potassium Chloride (APC)

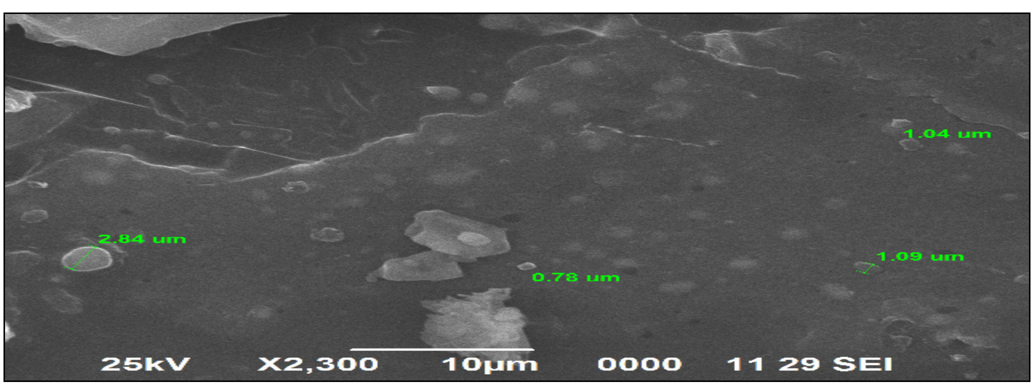


Fig.-4B: SEM Photograph of Alanine Potassium Chloride (APC)

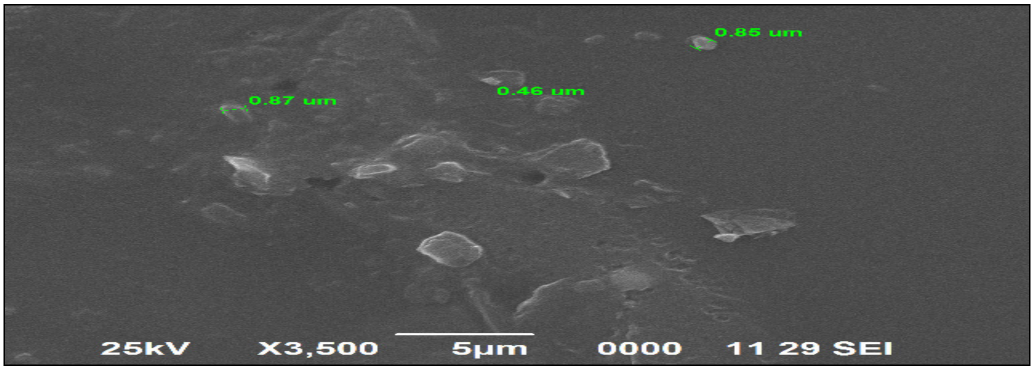


Fig.-4C: SEM Photograph of Alanine Potassium Chloride (APC)

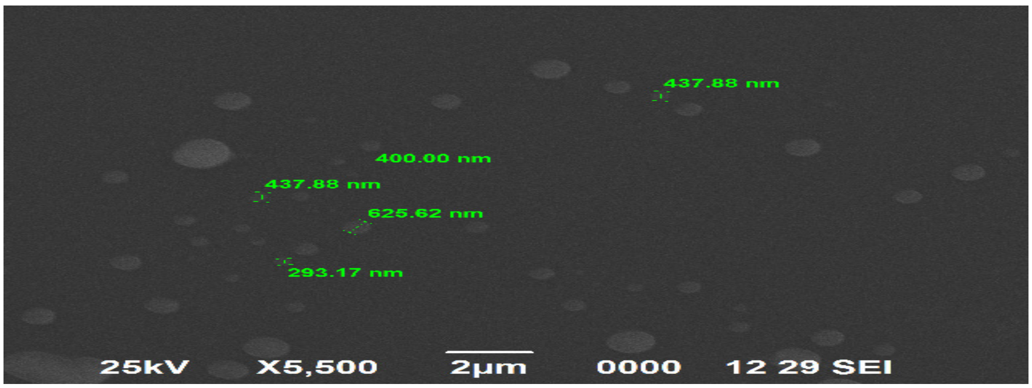


Fig.-4D: SEM Photograph of Alanine Potassium Chloride (APC)

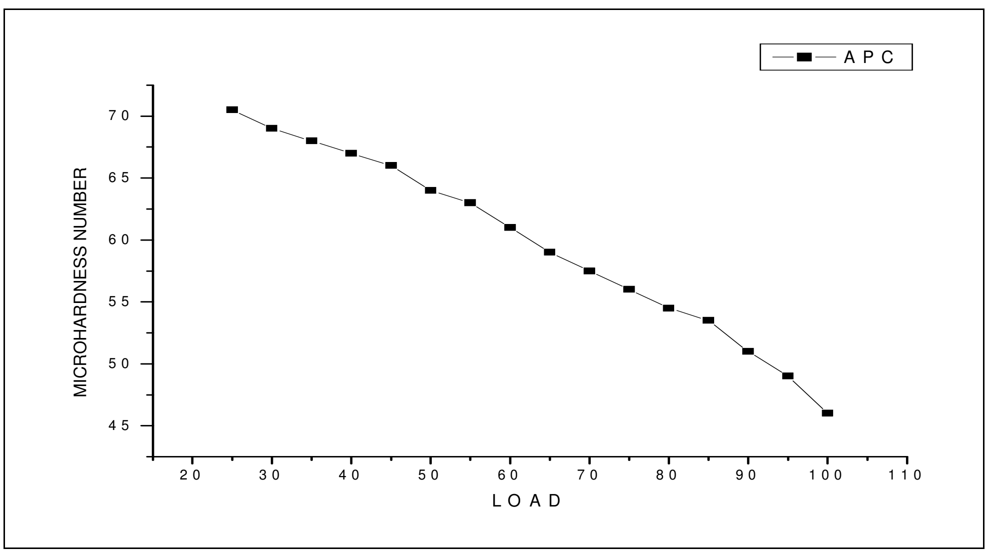


Fig.-5A: Variation of micro Hardness number with load

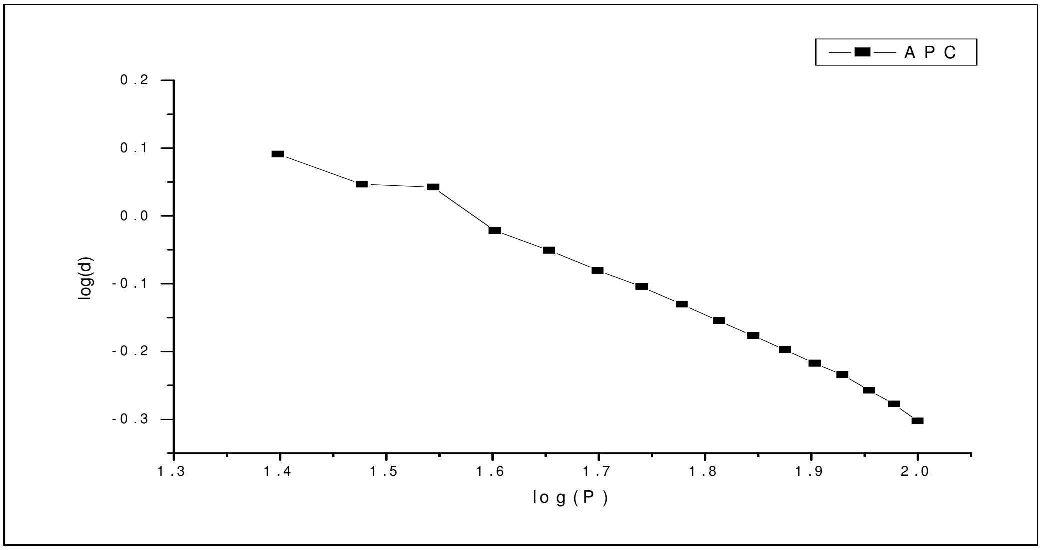


Fig.-5B: Variation of Log(D) with log(P)

Table-1: Characteristic absorption frequencies of various functional groups

S. No	Frequency Range	Intensity	Mode of vibration
1	3085	m	C-H asym. stretching
2	2934	w (broad)	OH stretching
3	2601	w (broad)	OH stretching
4	2293	m	Overtones & combination bands with prominent peaks near 2500 and 2000 cm^{-1}
5	2248	m	Overtones &combination bands with prominent peaks near 2500 and 2000 cm^{-1}
6	2112	m	Overtones &combination Bands with prominent peaks near 2500 and 2000 cm^{-1}
7	1620	s	C=O stretching
8	1589	s	C=O stretching
9	1507	s	NH $^{+}$3 sym. bending
10	1455	m	C-H in plane bending
11	1412	w	C-H bending (in plane)
12	1361	w	C-H bending (in plane)
13	1306	w	C-H bending (in plane)
14	1235	s	C-O-C stretching
15	1151	s	C-O-C stretching
16	1112	m	Sym. C-O-C stretching
17	1014	variable	C-CHO stretching

18	918	m (broad)	OH bending(out of plane)
19	849	s	C-H out of plane bending
20	772	s	C-H out of plane bending
21	648	s	C-H bending (out of plane)
22	539	s	OCN deformation
23	486	w	S-S stretching

Table-2: Crystallographic data of APC

ALANINE SODIUM NITRATE	CRYSTAL DATA
Lattice parameters	a=11.52746 A ^{o} b=15.70642 A ^{o} c= 4.76734 A ^{o} α= 96.6299^{o} β=98.6734^{o} γ =95.7711^{o}
Cell Volume (V)	Cell volume V=841.44A^{o3}

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