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L-Alanine Sodium Nitrate (ASN), NLO Material: Growth and Characterization

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ABSTRACT

Single crystal of alanine sodium nitrate (ASN), a nonlinear optical material has been grown from solution by slow evaporation method. The isoelectric point of the alanine is 6(1). So, the growth of crystals has been carried out at pH 6. The grown crystals have been subjected to powder X-ray diffraction studies to identify the crystalline nature. Single crystal X-ray diffractometer was utilized to measure the cell parameters and morphology of the grown crystals. The FTIR spectra taken for the crystals grown at pH values show the peak intensity. The mechanical properties of the grown crystals are studied using Vickers micro hardness measurement. 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, Vickers micro hardness tester.

INTRODUCTION

Non-linear optics is a very useful technology because it extends the usefulness of lasers by increasing the number of wavelengths available both longer and shorter than the original can be produced by non-linear optics. Some materials change light passing through them, depending upon orientation, temperature, light wavelength etc.(red light, lower wavelength) releasing one photon of accumulated higher energy (blue and green light higher wavelength). The important nonlinear optical materials from the device point of view are generally in the form of single crystals and must need a wide variety of ancillary materials requirement for optical use. In general, they will require extra ordinary stability with regard to room temperature and high intensity light source.

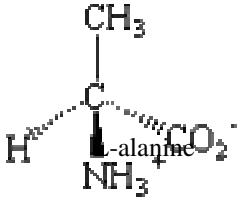
MATERIALS AND METHODS

SYNTHESIS AND CRYSTAL GROWTH

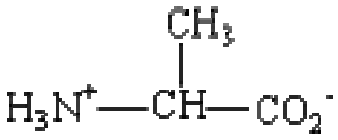
The solution was prepared using 4.995gms of alanine and 4.765gms of sodium nitrate dissolved in 30ml of distilled water. The pH value is low, so to increase the pH value. The pH of the solution is adjusted to 6 by adding 10 drops of NAOH [5].The above solution is filtered in the filter paper and transferred to a petri dish. The petri dish is 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



Further its linear zwitter ionic structure is



The crystal structure of L-alanine is orthorhombic [8]. Its cell parameters are
a = 6.032 A°, b= 12.343 A°, c = 5.784 A°.
α=β=γ=90°

CHARACTERIZATION

Powder XRD(Fig.1)
The powder XRD of alanine sodium nitrate (ASN) is shown in the fig. The peaks in the fig show the crystalline nature of ASN. Further peaks are indexed [6].
The grown crystals have been characterized by powder X-ray diffractometer. The lattice parameter values of alanine sodium nitrate taken from the values are-

a=10.83439 A°, b=15.39174 A°, c=6.19978 A°
α=90.7995° β =94.7954° γ =76.8417°
Cell volume V=1003.19 A°3.
The crystal crystal system is Triclinic.

FT-IR Analysis (Fig.2)
The FTIR spectrum of alanine sodium nitrate (ASN) was recorded using FTIR spectrometer in the region 2500-500cm^{-1} by KBr technique to confirm the presence of different organic groups along with the inorganic materials presence in the table[1]. The region 3448cm^{-1} with strong intensity represents N-H stretching. 3212cm^{-1} with medium intensity refers N-H stretching. The region 3066cm^{-1} with weak (broad) represents OH stretching.2058cm^{-1} with medium intensity refers overtones & combination bands with prominent peaks near 2500 and 2000cm^{-1}.The peak 1637cm^{-1} with strong band represents C=O stretching.829cm^{-1} with weak intensity refers C-H out of plane bending[3].

Micro Hardness(Fig.3)
ASN crystal was subjected to Vickers micro hardness test with the load varying from 25 to 100g [1]. 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 increases 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 increases with increasing the load [4]. The value of the work hardening coefficient n was found to be 0.08. According to Onitsch,$1.0 \leq n \leq 0.08$ for hard materials and $n > 0.08$ for soft materials [7]. Hence, it is concluded that ASN belongs to the soft materials.

UV-Visible Spectrometer Analysis(Fig.4)
The optical absorption spectra of alanine potassium chloride crystals (ASN) were recorded in the range 190-2500nm using JASCO corp V-570 spectrometer [6]. The UV-Visible transmission spectrum of alanine sodium nitrate crystal is shown in the Figure..

Crystal surface analysis by SEM(Fig.5)
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 magnification of 330 and at a scale of 50 micro meter we observe the crystals have smoothed needle surfaces. 2. At a magnification of 1500 and 1 micro meter scale can observe that the crystals have an average thickness of 421nm [7].

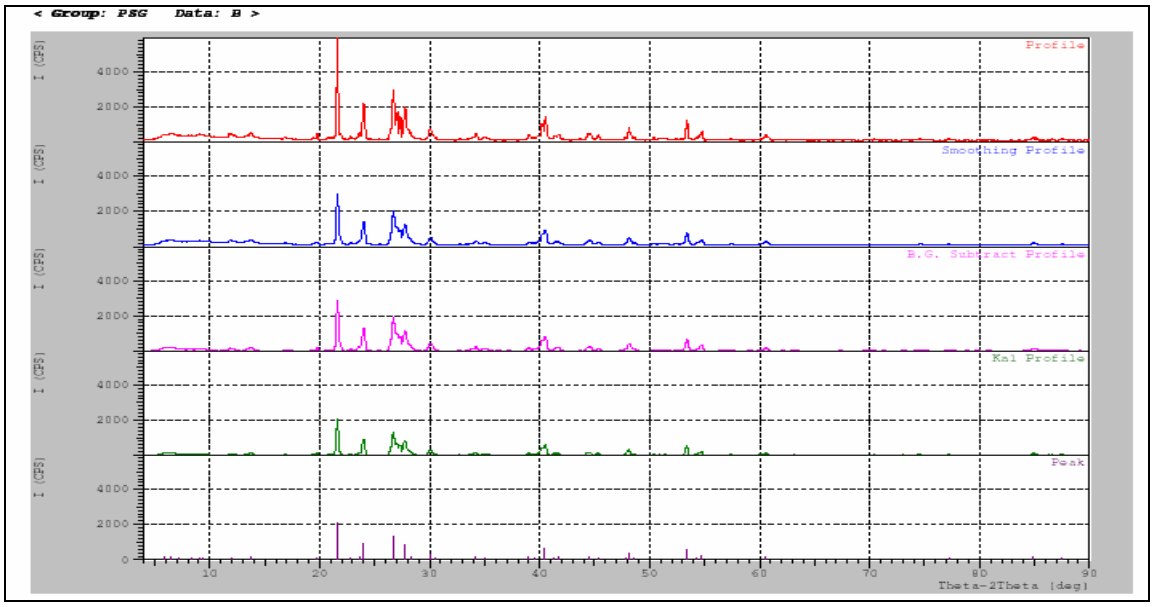


Fig.-1: Powder XRD pattern of Alanine Sodium Nitrate

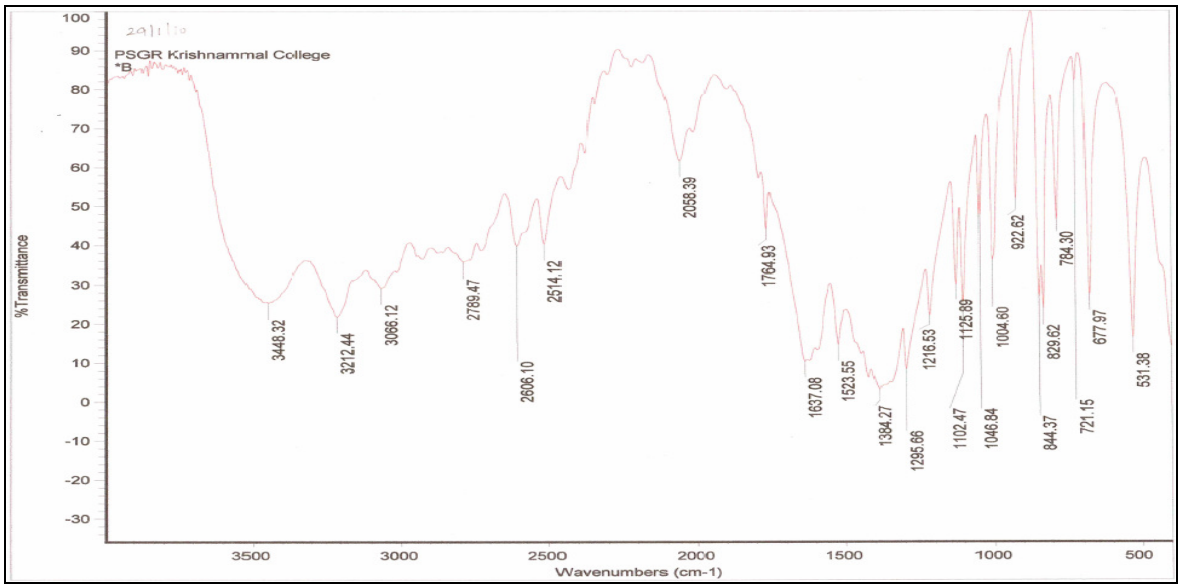


Fig.-2:FTIR Spectrum of Alanine Sodium Nitrate

RESULTS AND DISCUSSION

From the above experimental and characterization of alanine sodium nitrate crystal the following results and the discussion are significant.

1. Single crystals of ASN are successfully grown at a pH of 6. The grown crystals are larger in size having an average size of 1.5cmx11.5cm.

2. The grown crystals are characterized by using powder X-ray diffraction.
3. From this we confirm the structure of the crystal to be triclinic and the cell parameters: a=10.83439 A^{o} b=15.39174 A^{o} c=6.19978 A^{o}
4. From the FTIR spectrum we can confirm the structure of the ASN to have both the alanine and soiumnitrate molecules. These are arranged in alternate layers in the crystal. This is evident from the non damage of alanine structure.
5. From the Vickers' micro hardness test we find the micro hardness number increases with increasing the load. Further the value of the work hardening coefficient is found to be 0.08. From this result we conclude that the crystal is soft.
6. From the UV-Visible spectrum we find the crystal is transparent in the range 900-2500nm.
7. From the SEM analysis we conclude that the crystal formation inmicro range is 421nm. Further in the micro level the crystal surface is very smooth which shows that it can add more molecules to into a large crystal.

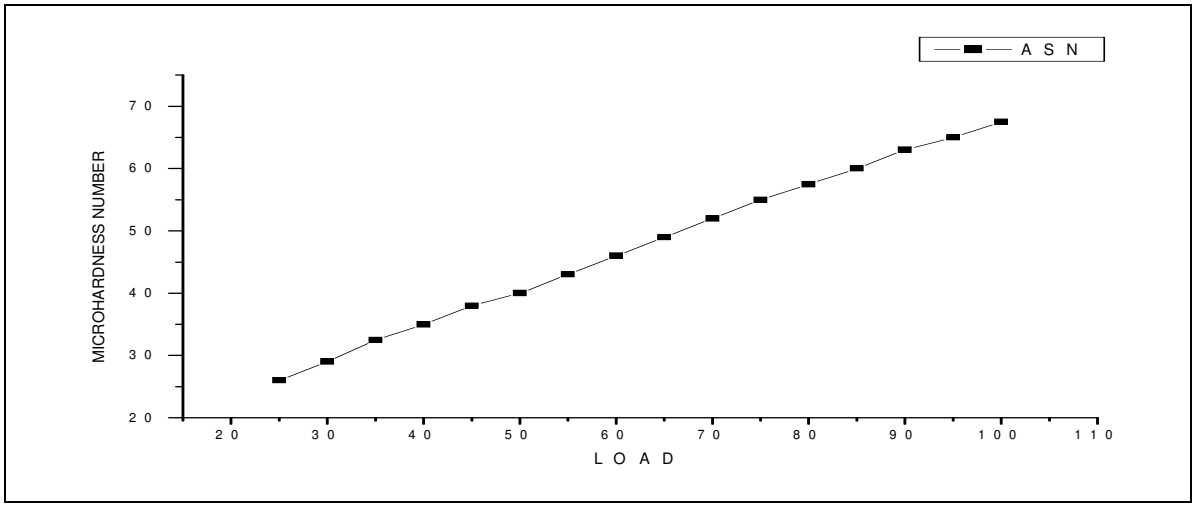


Fig.3A: variation of Micro hardness number with Load

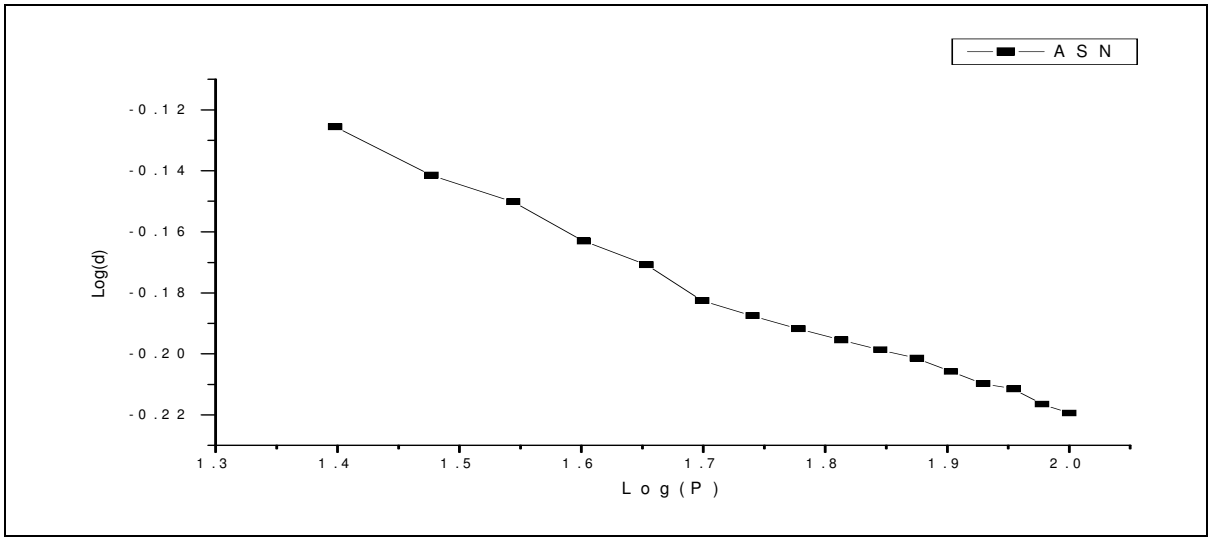


Fig.-3B: variation of log(D) with log(P)

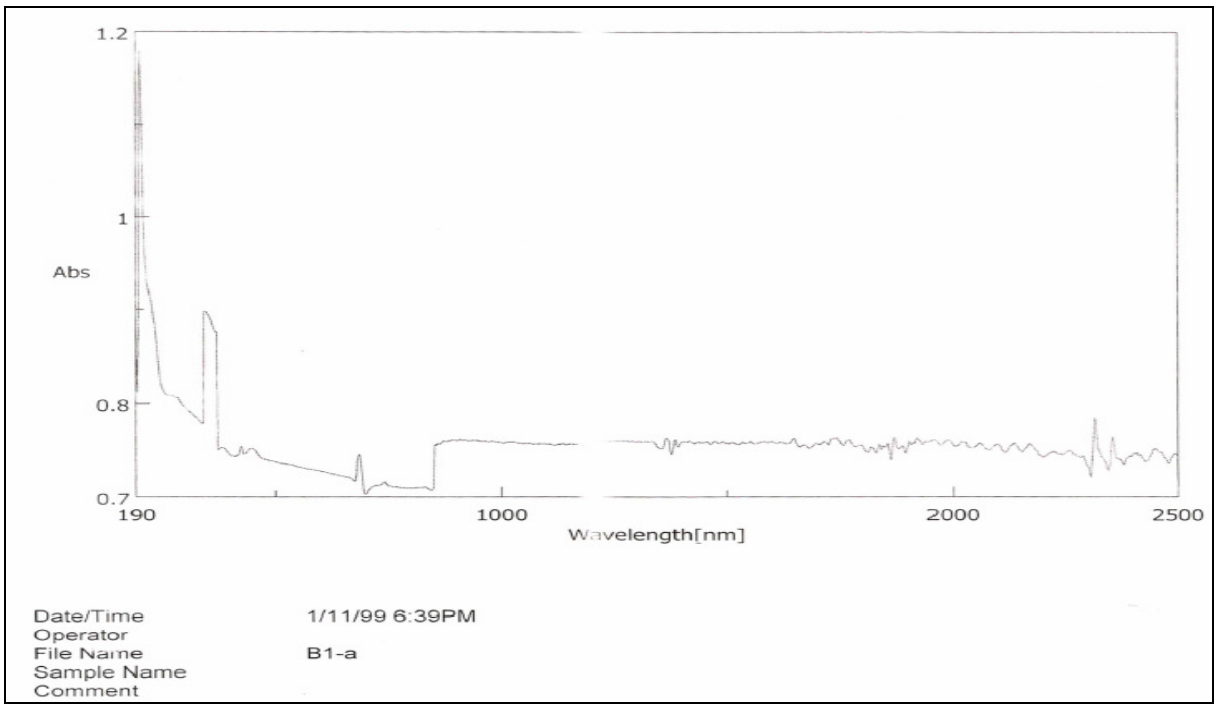


Fig.-4A: UV-Visible spectra of Alanine Sodium Nitrate

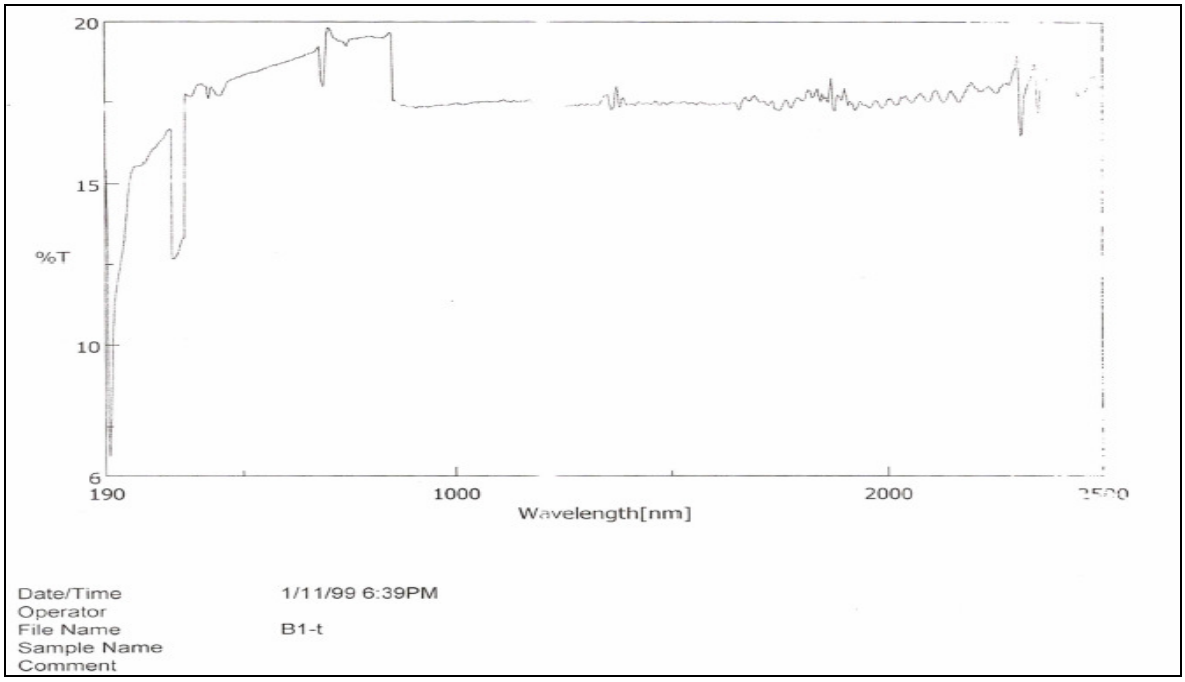


Fig.-4B: UV-Visible spectra of Alanine Sodium Nitrate

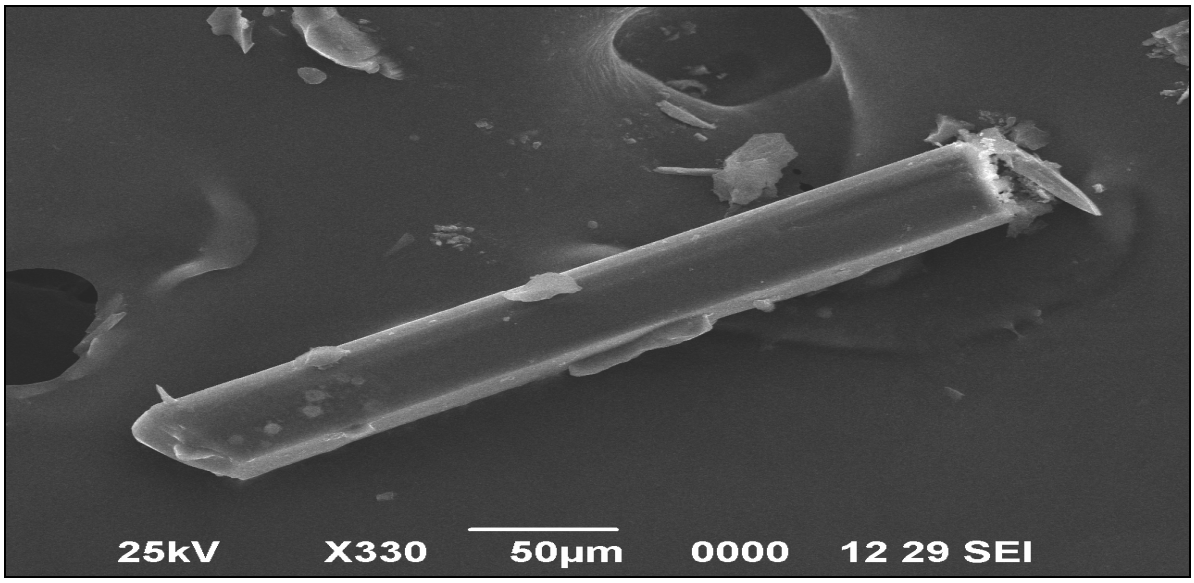


Fig.-5A: SEM photograph of Alanine Sodium Nitrate

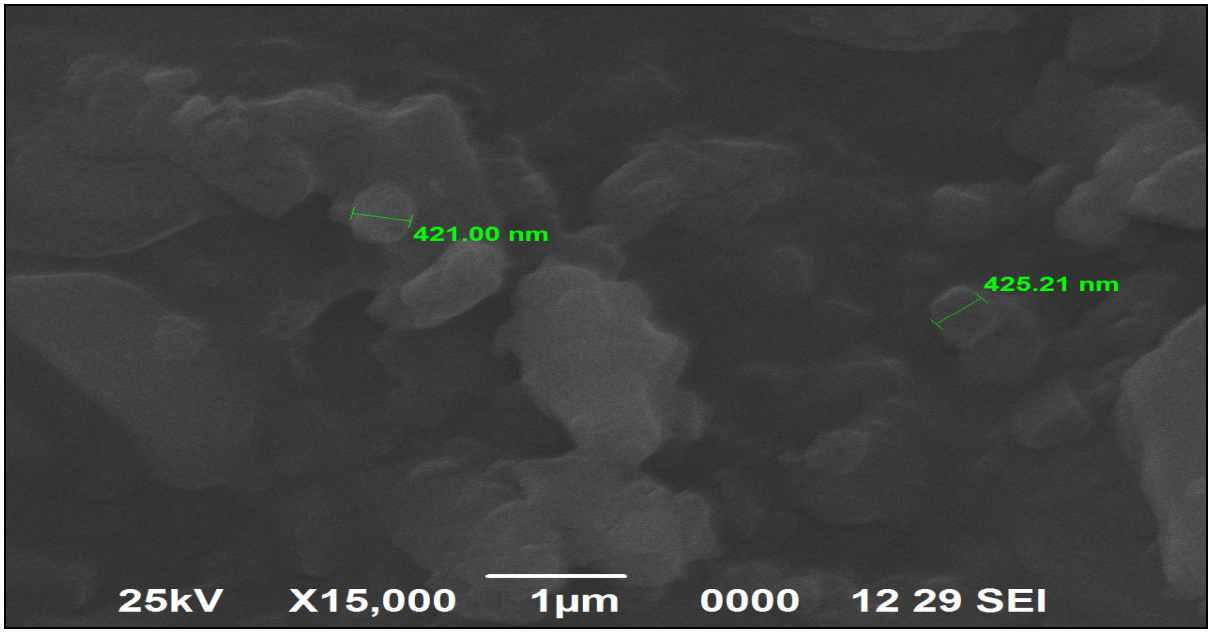


Fig.-5B: SEM photograph of Alanine Sodium Nitrate

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Table-1: Characteristic Absorption Frequencies of Various Functional Groups

S. No	Frequency Range	Intensity	Mode of vibration
1	3448	s	N-H stretching
2	3212	m	N-H stretching
3	3066	w (broad)	OH stretching
4	2789	w (broad)	OH stretching
5	2606	w (broad)	OH stretching
6	2514	w (broad)	OH stretching
7	2058	m	Overtones & combination bands with prominent peaks near 2500 and 2000 cm^{-1}
8	1764	s	C=O stretching
9	1637	s	C=O stretching
10	1523	s	N-H in plane bending
11	1384	s	CO^{-}_{2} sym. stretching
12	1295	s	C-O stretching
13	1216	s	C-O stretching
14	1125	variable	C-CHO stretching
15	1102	variable	C-CHO stretching
16	1046	variable	C-CHO stretching
17	1004	variable	C-CHO stretching
18	922	w	C-H out of plane bending
19	844	w	C-H out of plane bending
20	829	w	C-H out of plane bending
21	721	m	N-H out of plane bending
22	677	m	OCN deformation
23	531	s	OCN deformation

Table-2: Crystalographic data of ASN Crystal

ALANINE SODIUM NITRATE	CRYSTAL DATA
Lattice parameters	a=10.83439 A^{o} b=15.39174 A^{o} c=6.19978 A^{o} α=90.7995 ^{o} β =94.7954 ^{o} γ =76.8417 ^{o}
Cell Volume (V)	Cell volume V=1003.19 $A^{o\,3}$.