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Growth and Characterization of NLO Material: l-Alanine Sodium Chloride

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ABSTRACT
Single crystal of alanine sodium chloride (ASC), a nonlinear optical material has been grown from solution by slow evaporation method. The iso-electric 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 is utilized to measure the cell parameters and morphology of the grown crystals. The FTIR spectra taken for the crystals grown at a pH value of 6 show different peaks intensity which reveals the structure of the ASC crystal. 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 is studied by UV-Visible spectrometer.
Keywords: Characterization, X-ray diffraction, Slow evaporation, Sem analysis, Vickers micro hardness tester.

INTRODUCTION

Non linear optical (NLO) crystals has emerged as one of the most attractive fields of current research in view of its vital applications in areas like optical modulation, optical switching, optical logic, frequency shifting and optical data storage for the developing technologies in telecommunications and in efficient signal processing[4]. Surface morphology was studied by using SEM studies. The development of highly efficient nonlinear optical (NLO) crystals for visible and ultraviolet region is extremely important for both laser spectroscopy and laser processing.

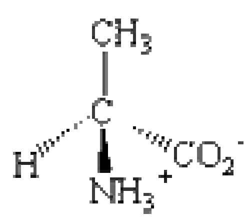
MATERIALS AND METHODS

Synthesis and crystal growth

The solution is prepared using sodium chloride and alanine in the molar ratio of 1:1[7]. The pH value is low, so to increase the pH value, the pH of the solution is adjusted to 6 by adding 4 drops of NaOH. The above solution is filtered using 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.

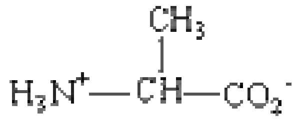
Structure of L-Alanine molecule

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 –



L-alanine

Further its linear zwitter ionic structure is



The crystal structure of L-alanine is orthorhombic [10]. Its cell parameters are
a = 6.032 A ^{o}, b= 12.343 A ^{o}, c = 5.784 A ^{o}.
$\alpha = \beta = \gamma = 90^{o}$

Characterization
The crystals are characterized by FTIR spectroscopy, powder XRD, UV-Visible spectroscopy, Vickers micro hardness tester and SEM. The FTIR spectra are recorded on Shimadzu 8400s FTIR. Powder XRD is obtained by Philips X-ray generator mode 1pw 1390 with a nickel filter. The visible absorption data is obtained using Jaso corp v570.

Powder XRD (Fig.1)
X-ray diffraction technique is a powerful tool to analyze the crystalline nature of the materials. If the material to be investigated is crystalline, well defined peaks will be observed. The peaks in the diffractogram are indexed by using powder X-ray software [5].

FT-IR Analysis (Fig.2)
The FTIR spectrum of alanine sodium chloride (ASC) is recorded using FTIR spectrometer in the region 2500-500cm^{-1}. T he region
3436cm^{-1} with strong intensity represents N-H stretching. 3085cm^{-1}with medium intensity refers C-H asymmetric stretching. The region 2929cm^{-1} with weak (broad) represents OH stretching. 2112cm^{-1} with weak intensity refers C=C stretching.1506cm^{-1} weak intensity represents N-H in lane bending. The peak 1412cm^{-1} with weak band represents C=H bending.1235cm^{-1} with variable refers C-O-C stretching. 539cm^{-1} with weak intensity refers S-S stretching. The peak 486cm^{-1} with weak intensity represents S-S stretching [3].

Micro hardness (Fig.3)
ASC crystal is 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 is 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 [11]. The value of the work hardening coefficient n is found to be 0.672. According to Onitsch, $1.0 \le n \le 0.672$ for hard materials and $n > 0.672$ for soft materials [6]. Hence, it is concluded that ASC belongs to the soft materials.

UV-Visible spectrometer analysis (Fig.4)
The optical absorption spectra of alanine sodium chloride crystals (ASC) are recorded in the range 190-2500nm using JASCO corp V-570 spectrometer [8]. It is seen from the absorption spectrum the crystal is transparent in the range 900-2500nm without any absorption peak, which is an essential parameter of NLO crystals.

Scanning electron microscope (SEM) (Fig.5)
The quality of the grown crystals can be identified to some extent by identifying the surface morphology of crystal face is observed by optical microscope in reflection mode using JSM 6360 JEOL/EO. From the SEM photograph shown in figure, the following observations are evident: 1. at a magnification of 850 and at a scale of 20 micrometer we observe the crystals have smoothed, rectangular surfaces. 2. At a magnification of 15,000 and 1 micrometer scale we can observe that the crystals have an average thickness of 200µm [9].

RESULTS AND DISCUSSION

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

1. Single crystals of alanine sodium chloride (ASC) are successfully grown at a pH of 6.The grown crystals are larger in size having an average size of 3cmx4cm.
2. The grown crystals are characterized by using powder X-ray diffraction.
3. From the FTIR spectrum we can confirm the structure of the ASC to have both the alanine and sodium chloride molecules. These are arranged in alternate layers in the crystal. This is evident from the non damage of alanine structure.
4. From the SEM analysis we conclude that the crystal formation size in micro range is 200 micrometers. Further in the micro level the crystal surface is very smooth which shows that it can add more molecules to grow into a large crystal.
5. From the Vickers' micro hardness test we find the micro hardness number increases with load. Further the value of the work hardening coefficient is found to be 0.672. From this result we conclude that the crystal is soft.
6. From the UV visible spectrum we find that the crystal is transparent in the range 900-2500nm without any absorption peak.
7. The NLO test using Nd-YAG laser confirms that the crystal has NLO property.

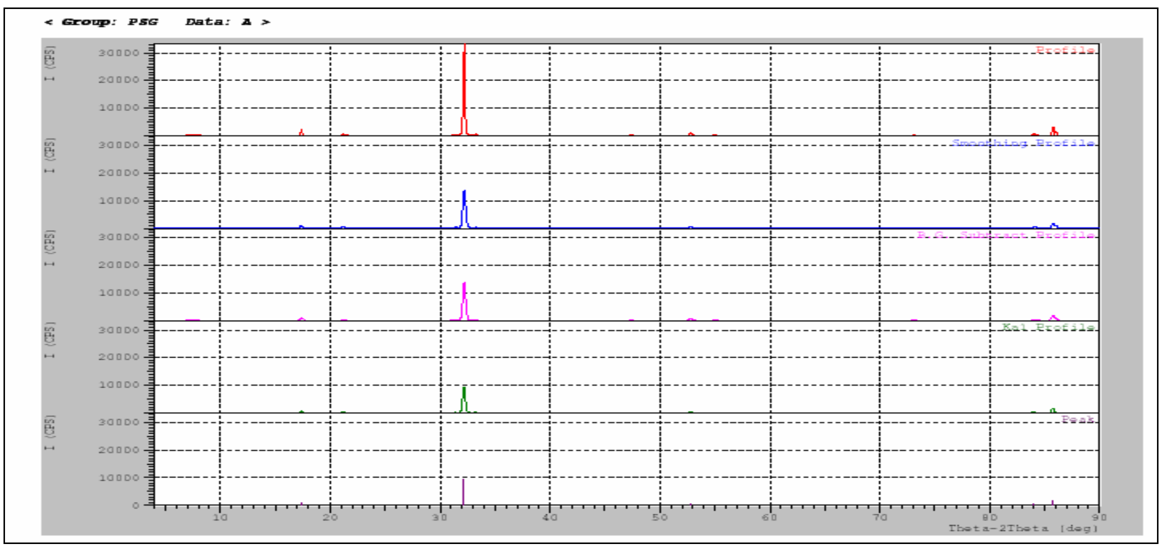


Fig.-1: Powder XRD pattern of Alanine sodium chloride (ASC)

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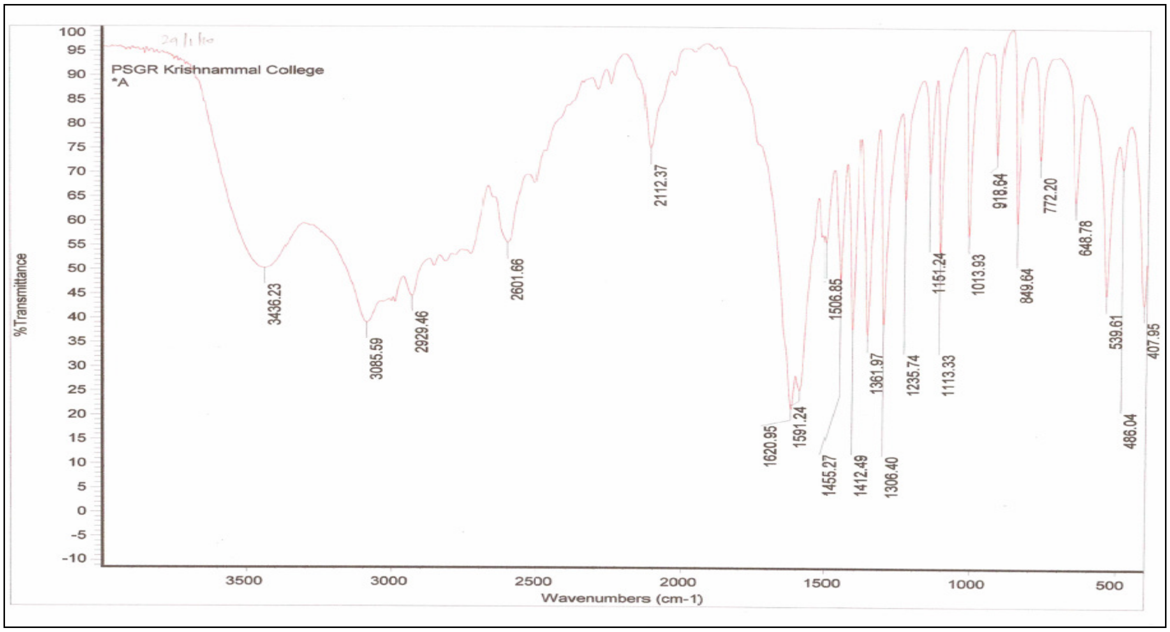

Fig.-2: FTIR sopectrum of Alanine sodium chloride (ASC)

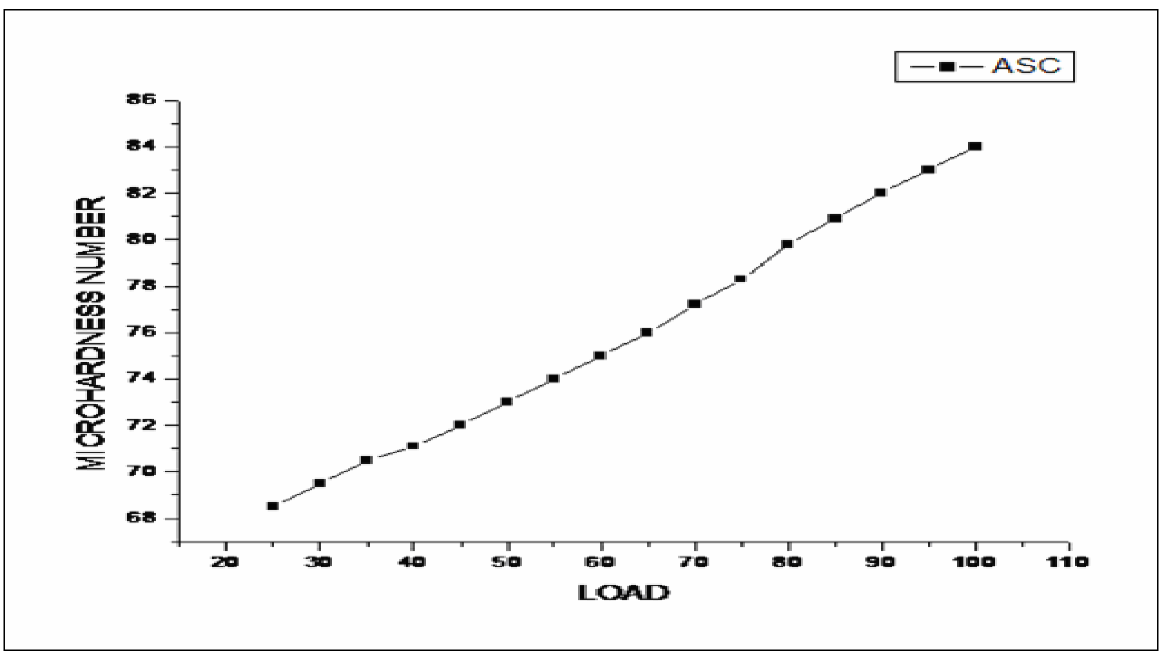

Fig.-3A: Variation of micro hardness number with load

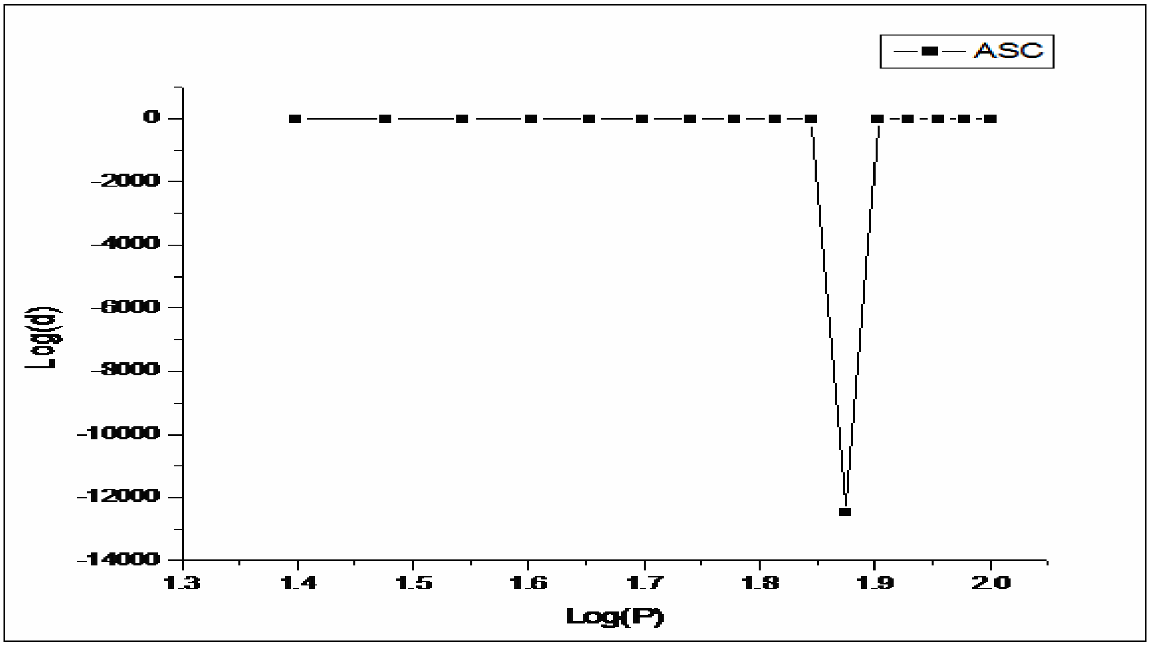

Fig.-3B: Variation of log(D) with log(P)

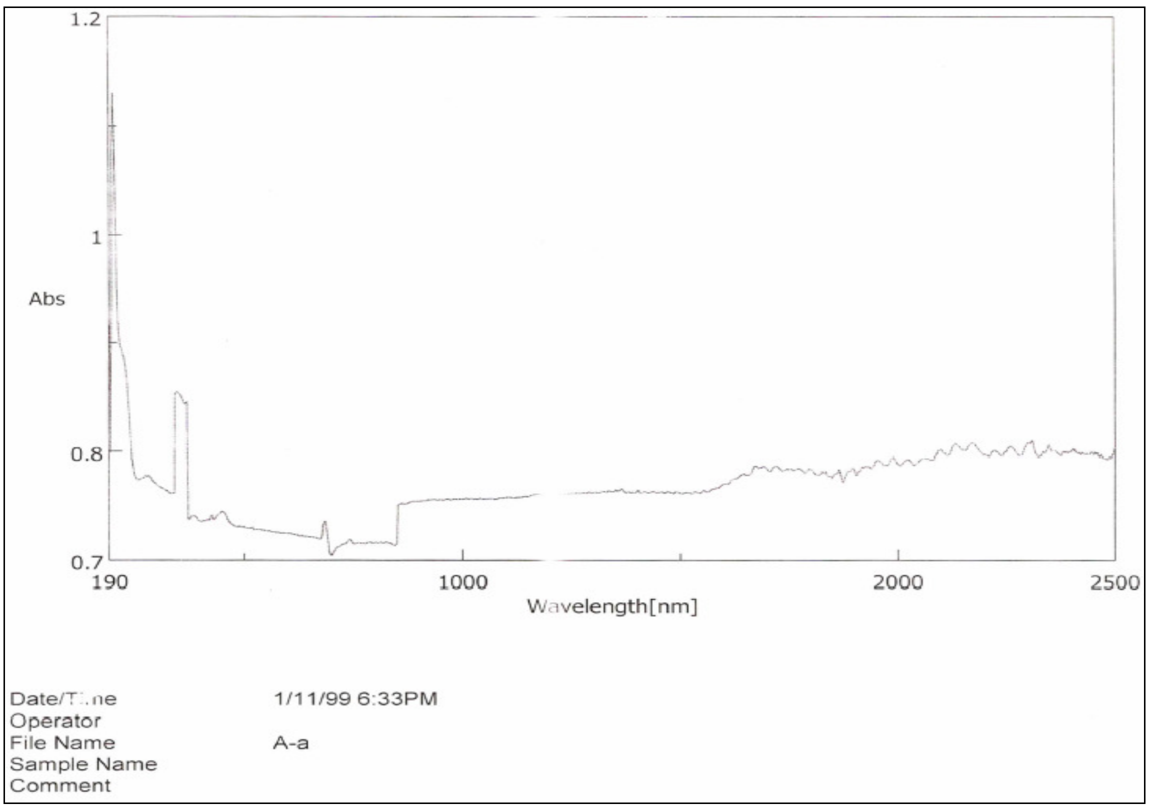

Fig.-4A: UV-Visible spectrum of Alanine sodium chloride (ASC)

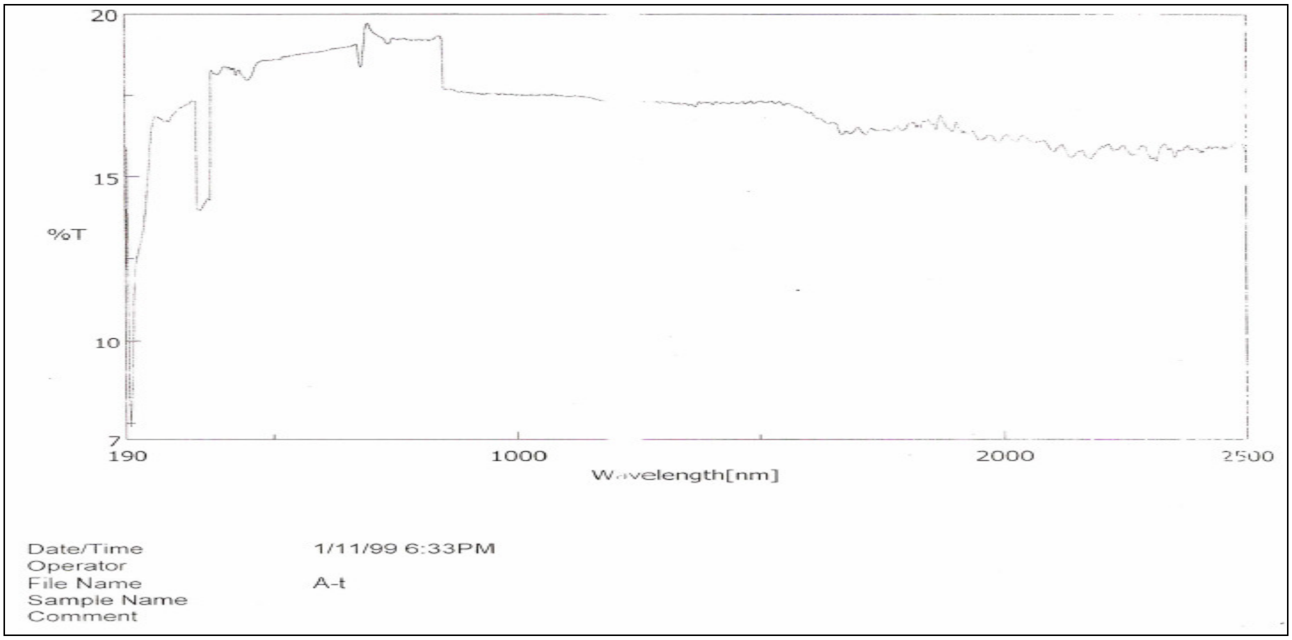

Fig.-4B: UV-Visible spectrum of Alanine sodium chloride (ASC)

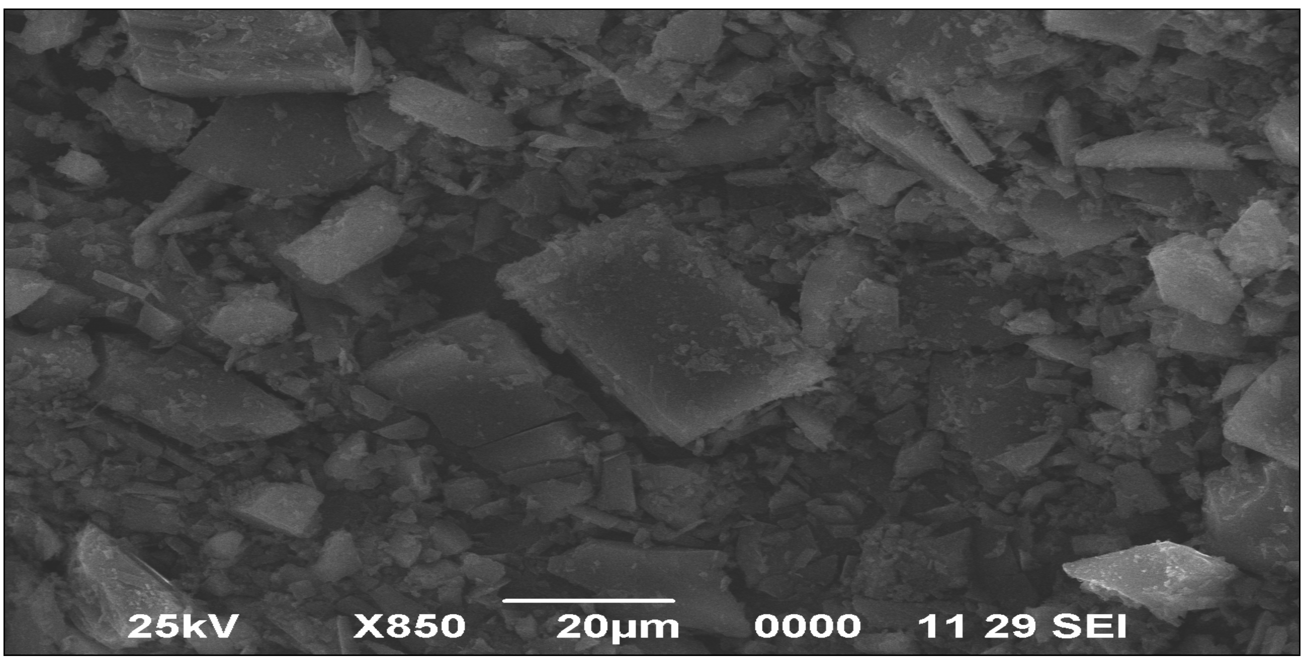

Fig.-5A: SEM Photograph of Alanine sodium chloride (ASC)

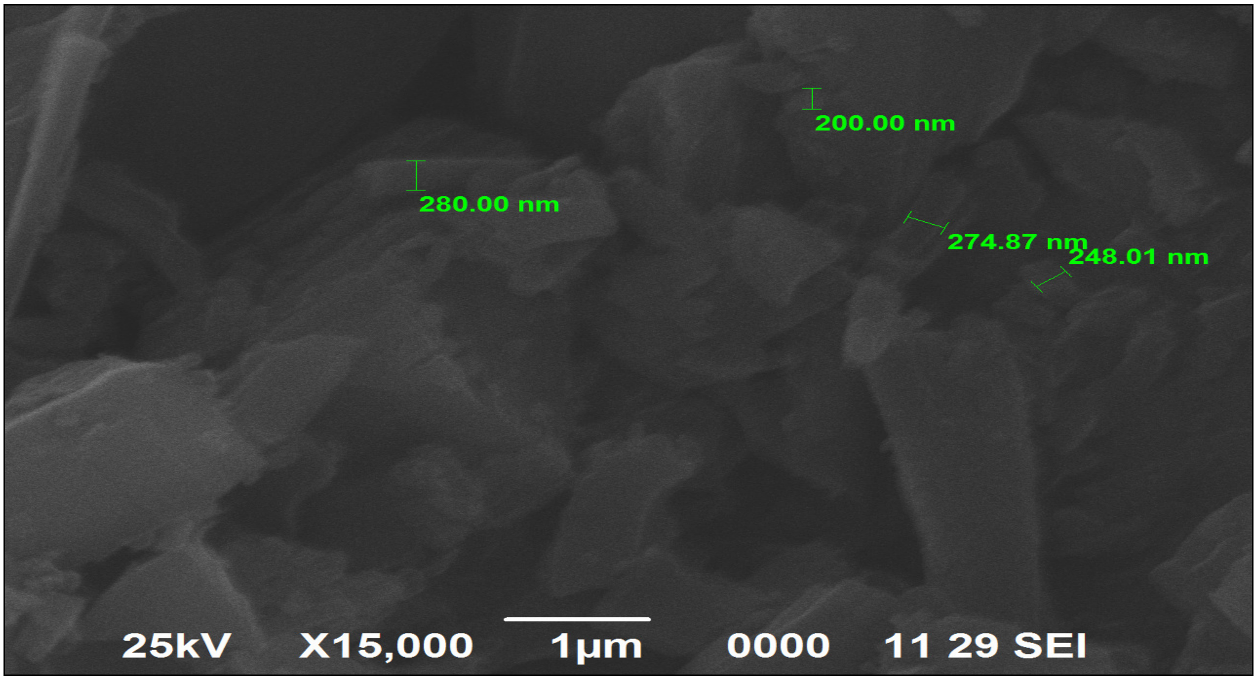

Fig.-5B: SEM Photograph of Alanine sodium chloride (ASC)

Table-1: Charcteristic Absorption frequencies of various functional groups

S. No	Frequency Range	Intensity	Mode of vibration
1	3436	s	N-H stretching
2	3085	m	C-H asym.stretching
3	2929	w (broad)	OH stretching
4	2601	w (broad)	OH stretching
5	2112	w	C≡O stretching
6	1620	s	C=O stretching
7	1591	s	C=O stretching
8	1506	w	N-H in plane bending
9	1455	m	C-H in plane bending
10	1412	w	C-H bending (in-Plane)
11	1361	w	C-H bending (in-Plane)
12	1306	w	C-H bending (in-Plane)
13	1235	variable	Asym.C-O=C stretching
14	1151	s	Asym.C-O=C stretching
15	1113	m	Sym. C-O=C stretching
16	1013	variable	C-CHO stretching
17	918	m (broad)	OH bending (out of plane bending)
18	849	w	C-H out of plane bending
19	772	s	C-H out of plane bending
20	648	s	C-H bending (out of plane)
21	539	w	S-S stretching
22	486	w	S-S stretching