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Synthesis, Growth, Solubility and Characterisation of Pure L-Asparagine and Its Based Crystal LABP

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Abstract-- The synthesis of pure L-asparagine and its based crystal such as L-asparagine admixture with potassium bromide using de-ionized water as solvent (LAPB)was carried out by taking L-asparagine and potassium bromide in the molar ratio 2:1.The solubility of the grown crystals were carried out by gravimetrical method. Using the powder X-ray diffraction study the crystalline of pure L-asparagine and LAPB crystals were confirmed. The various functional groups of the grown crystals were assigned from FTIR study. The optical transmittance range obtained from UV-visible transmittance. The second harmonic generation (SHG) test confirms the NLO property of the grown crystals by kurtz and Perry and it was compared with KDP. The microhardness measurements were estimated by Vickers hardness test.

Keywords-- *Solubility, P-XRD, FTIR, UV-Vis, Microhardness.*

I. INTRODUCTION

Crystals are the pillars of modern technology [1]. The word crystals originates from the Greek word ‘krystallos’ meaning ‘clear ice’ [2]. Quartz found in Swiss Alps were first named as crystals and the most remarkable feature of matter in the solid state is the tendency of the constituent atoms to arrange themselves on orderly periodic extends throughout the material that is it is having perfect order [3]. Crystals are used in electronic industry, photonic industry, fiber optic communication, which depend on material such as semiconductors, superconductors, polarizers, transducers, radiation detectors ultrasonic amplifiers, magnetic garnets, solid state lasers, non-linear optics, piezo-elecrtic, electro-optic, acoustic-optic, photosensitive, refractory of different grades, crystalline films for microelectronics and computer industries.

Amino acids are interesting materials for NLO applications as they contain a proton donor carboxyl acid (COO^-) group and a proton acceptor amino (NH^{+2}) group in them. Amino acids are the molecular building blocks of peptides and proteins. L-Asparagine monohydrate (LAM) is an interesting material from amino acid family to investigate because it crystallizes in a structure exhibiting a complex network of hydrogen bonds among asparagines molecules and between asparagines and water molecules [4]. In the present study, L-asparagine admixture with Potassium bromide(LAPB) was grown from aqueous solution using slow evaporation technique. The grown crystal subjected to various characterization technique such as powder XRD, FTIR, UV-VIS and microhardness test.

II. SYNTHESIS, SOLUBILITY AND GROWTH

The synthesis of pure L-asparagine [5] based crystals such as L-asparagine admixture with potassium bromide using de-ionized water as solvent (LAPB) was carried out by taking L-asparagine and potassium bromide in the molar ratio 2:1 and sodium chloride solution (1 mol%) as solvent. Initially, the temperature was maintained at 30^0 C. The sample was added step by step to 50 ml of de-ionized water in an air-tight container kept on the hot-plate magnetic stirrer and stirring was continued till a small precipitate was formed. This gave configuration of supersaturated condition of the solution. Then, 25 ml of the solution was pipetted out and taken in a petri dish and it was warmed up at 40^0C till the solvent was evaporated out. By measuring the amount of salt present in the petri dish, the solubility (in g /100ml) of the sample in de-ionized water was determined by gravimetrical method. Similarly, the solubility for various other temperature can be found. The solubility diagram for pure L-asparagine and LAPB crystal are shown in figure 1.

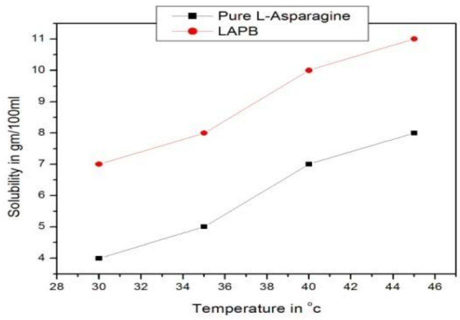


Figure 1: Solubility of pure L-Asparagine and LAPB Crystal

To grow L-asparagine based crystals, the synthesized salts of LAPB was used. The solutions were stirred well for about 2 hours and filtered separately. Then they were allowed to slow evaporation for the growth. The photograph of the grown pure L-asparagine and their based crystals are presented in the figure 2.

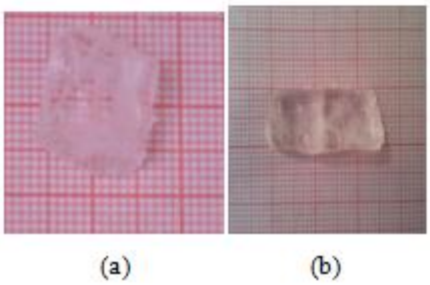


Figure 2: Photography of (a) L-Asparagine (b) LAPB

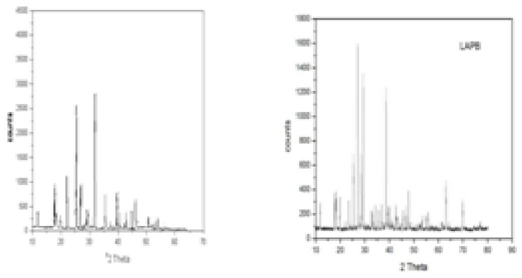
Table 1: Size and duration of grown crystals:

Crystal Type	Size (dimension) in mm^3	Duration in days
Pure L-asparagine LAPB Crystal	19x14x6 26x11x6	30-35 30-35

III. RESULT AND DISCUSSION

A. Powder X-ray diffraction

The powder X-ray diffraction data recorded for grown pure L-asparagine and LAPB crystals were finely powered and subjected to powder XRD analysis using the X'PERT-PRO Diffractometer system. The samples were examined with CuK_{α} radiation (λ = 1.54056 A^{O}) in a 2θ range of 10^{O} -80^{O} at a scan rate of 2^{O}/ min and in step size [2^{O} Th.] 0.0170. From the X-ray diffraction spectra, the (2θ) values were read directly and relative intensities of the diffraction peaks were estimated. The powder X-ray diffraction pattern of the synthesized compounds are shown in Figure 3(a)-3(b).



(a) (b)

Figure 3: Powder XRD Pattern of (a) pure L-Asparagine (b) LAPB crystal

B. FTIR study

Fourier Transform Infrared Spectroscopy (FTIR) is an analytical technique used to identify mainly the functional groups present in organic materials. FTIR analysis provides information about the chemical bonds and molecular structure of a material. In order to qualitatively analyze the presence of functional groups in the grown crystals, the FT-IR spectrum were recorded in the range 400-4000 cm^{-1} using a KBr pellet on SHIMADZU-FTIR-8400S spectrometer and the recorded spectra were shown as Fig.4(a) and 4(b).and the various observed peaks are assigned[6-8] The Fourier transform infrared analysis is a technique in which almost all the functional groups in a molecule absorb characteristic frequencies.

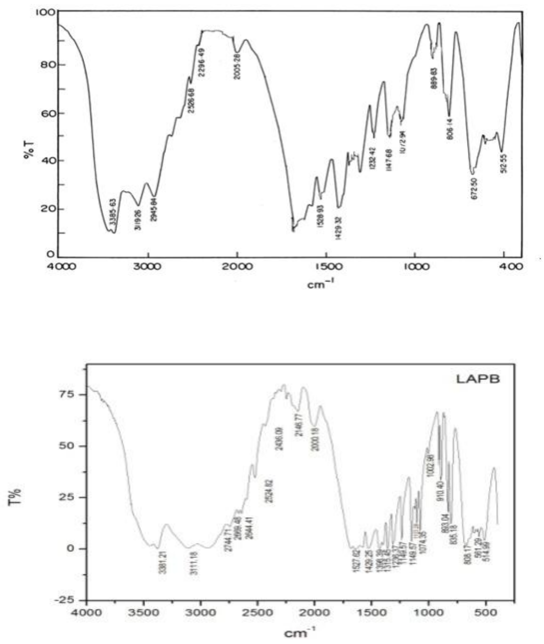
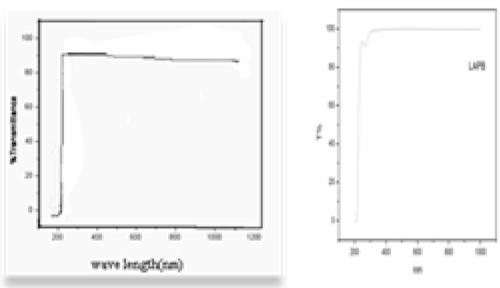


Figure 4: FTIR Pattern of pure L-Asparagine and LAPB Crystal

C. UV-Visible spectroscopy analysis

The UV-Vis transmission spectrum of pure L-Asparagine and based single crystal was recorded between 190 nm and 1200 nm using Perkin Elmer Lambda 35 UV-V is Spectrometer. The recorded spectrum is shown in Figs. 5(a) and 5(b).



(a) (b)

Figure 5: UV-Visible Transmittance Spectra of (a) pure L-Asparagine (b) LAPB Crystal

D. Mechanical study

Micro hardness is an important for good quality crystals along with good optical performance [9]. The Vickers hardness (Hv) numbers at different loads were calculated using the relation,

Hv = 1.8554 P/d^2 $kd\text{-}mm^{-2}$

Where P is the applied load in kilogram and d is the average diagonal length of the indentation marks in millimeter and 1.8554 is a constant of a geometrical factor for the diamond pyramid. The Vickers microhardness number as a function of the applied test load shown in figure 6(a) and 6(b).

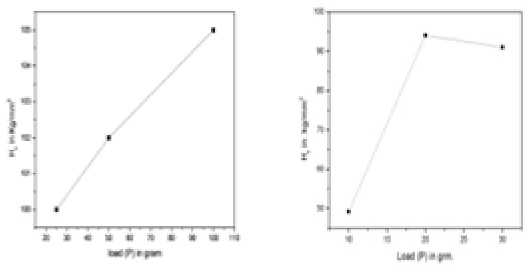


Figure 6: Plots between hardness number (H_v) and applied load for(a) Pure L-asparagine (b) LAPB Crystal

a. Estimation of Work hardening coefficient (n):

The load variation can be interpreted using Meyer's law, P = ad^n, where P is the load applied, d is the diagonal length of impression, a is a constant and n is the Meyer's index or work hardening co-efficient. The work hardening coefficient can be estimated from the slope of log P versus log d plot shown in Figures 7(a) and 7(b).

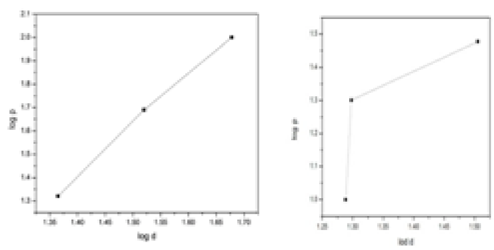


Figure 7: plot of log P versus log d for pure L-Asparagine and LAPB crystal

The work hardening coefficient (n) for all the grown crystals are estimated from the Fig.7 using least square fit method and the obtained values are reported in Table 2. It can be seen that log P versus log d is linear for both the crystals and the slope of the straight line gives the work hardening coefficient (n).

Table 2: Work hardening coefficient (n) of Pure L-Asparagine and it is based crystal

Samples	Work hardening coefficients (n)
Pure L-Asparagine	2.1749
LAPB Crystal	1.621

According to Onitsch[10] and Hanneman [11], if n is greater than 1.6, the material belongs to the category of soft materials [12]. In the present study, the work hardening coefficients of all the grown crystals are greater than 1.6. Thus, the present crystals under study belongs to soft material category. Because of the high mechanical strength, both crystals can be very well used for device fabrication.

CONCLUSION

Growth of pure L-asparagine and its based crystal are carried out by slow evaporation technique. The more transparent crystal can be obtained using recrystallization process. The powder XRD patterns of L-asparagine and its based samples are compared. However, a slight variation in intensity is observed as a result of doping. The most prominent peaks with maximum intensity of the XRD patterns of pure and doped specimens are quite different. FTIR analysis provides information about the chemical bonds and molecular structure of a material. Hence, the presence of various functional groups was confirmed from the band assignment. The transparency was checked by carrying out UV-visible spectral studies for both pure L-asparagine and its based crystals. The optical band gap is slightly decreased for LAPB compared to that of pure L-asparagine. The cut –off wavelength values for pure L-asparagine and its based samples were found to be 220 and 230 nm and corresponding energy gap values are 5.46 and 5.27 eV respectively.From the mechanical results, it is noticed that the hardness number increases with the applied load and this is due to reverse indentation size effect. In the present study, the work hardening coefficients of all the grown crystals are greater than 1.6. Thus, the present crystals under study belong to soft material category.

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