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Growth and Characterization of Potassium Nitrate Doped L-Arginine Single Crystals

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Abstract: Single crystals of potassium nitrate doped L arginine were grown by slow evaporation process at room temperature. The grown compound was characterized by powder XRD analysis to confirm the crystalline nature. The cell parameters and structure of grown crystals were identified through single crystal XRD analysis. The structure of the grown crystals is Orthorhombic. The suitability of the crystals for optical applications was studied by UV–Vis Spectroscopy which proved that the transparency of the grown crystals is high. Fourier Transform infrared (FT-IR) spectral analysis confirmed the presence of various functional groups in the grown crystals. The mechanical property of the grown crystals was determined by Vicker's micro hardness test and it proved the soft nature of the grown crystal.

Keywords: Crystal growth, slow evaporation; powder XRD, single crystal XRD, UV–Vis, FT-IR, Vicker's micro hardness.

1. INTRODUCTION

The present fascinating field of research is to synthesize, grow and characterize semi organic NLO crystals [1]. Non-linear optical (NLO) 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 [2]. Due to this fact, the rapid development of optical communication systems has led to a demand for non linear optical materials of high structural and optical quality.Amino acid families of crystals are under extensive investigations in recent times owing to their favorable NLO properties[3]. Many number of natural amino acids are individually exhibiting non linear optical properties because they have a donor NH_2 and acceptor COOH and also inter molecular charge transfer is possible [4]. In the present study we have made an attempt to semi organic non linear crystals of Potassium Nitrate with L-arginine in different ratios and the grown crystals were subjected to various characterization techniques.

2. MATERIALS AND METHODS

Slow evaporation is the direct conversation of material from liquid phase to solid phase. This process is effectively used for the materials having very low temperature co-efficient of solubility. In slow evaporation the vapour pressure of the solvent solution is higher than the solvent. Evaporation more rapidly occurs and the solution attains super saturation. In the present work, the KNO_3 doped L-arginine single crystals are grown by slow evaporation method.

2.1. Experimental Method

High purity (AR grade 99% purity) Merck company salts were used for the preparation. Saturated solutions of KNO_3 (57g) and L-arginine (36g) were prepared separately by adding the salts gradually and stirred by using magnetic stirrer (Remi-1 MLH) and then filtered twice using Whatman no:1 filter paper. The above prepared saturated solutions of KNO_3 and L-arginine were mixed in the ratios (1:3), (1:1) and (3:1) and taken in three separate beakers. These solutions were stirred well for two hours to get the homogeneity. Then the beakers were closed with perforated aluminium foil and kept in a dust free and vibration free environment and observed periodically. After a time span of 69, 17 and 17 days the KNO_3 doped L-arginine single crystals in the ratios (1:3), (1:1) and (3:1) were harvested successfully. The photographs of the grown crystals are shown in figures 1.a, 1.b and 1.c. respectively.

Figure 1.a. KNO_3 doped L-arginine single crystal (1:3)

Figure 1.b. KNO_3 doped L-arginine single crystal (1:1)

Figure 1.c. KNO_3 doped L-arginine single crystal (3:1)

3 .RESULTS AND DISCUSSION

3.1 . Powder XRD analysis

The grown compounds were subjected to powder X-ray diffraction analysis to confirm the crystalline nature. Powder X-ray diffraction pattern was recorded using a Rich Seifert diffractometer with Cu Kα radiation (λ=1.5418Å). The sharp peaks indicate the crystalline nature of the sample[5]. The indexed PXRD pattern of the single crystals of KNO_3 doped L-arginine in the ratios (1:3), (1:1) and (3:1) are shown in the figures2.a, 2.b and 2.c respectively[6].

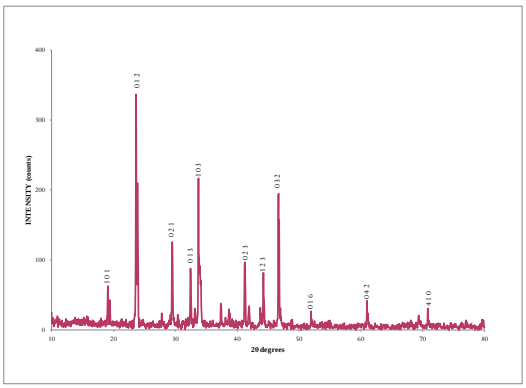

Figure: 2.a. PXRD pattern of KNO_3 doped L-arginine (1:3)

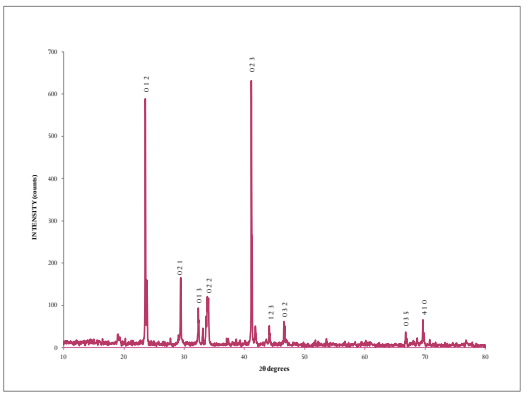

Figure: 2.b. PXRD pattern of KNO_3 doped L-arginine (1:1)

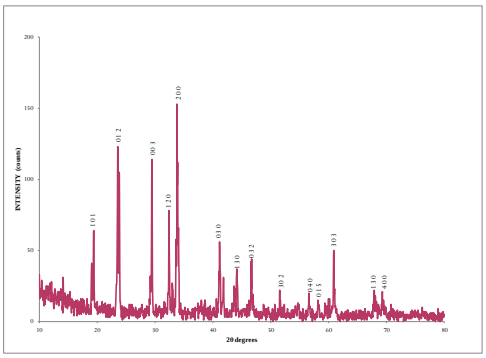

Figure: 2.c. PXRD pattern of KNO_3 doped L-arginine (3:1)

3.2 .SINGLE XRD ANALYSIS

Single crystal X-ray Diffraction is a non-destructive analytical technique which provides detailed information about the internal lattice of crystalline substances, including unit cell dimensions, bond-lengths, bond-angles and details of site-ordering. The single crystal XRD data was recorded using Bruker kappa Apex II diffractometer.

The single crystal XRD data of potassium nitrate doped L-arginine crystals indicate that they crystallize in orthorhombic system. The unit cell parameters are listed in Table1. The single crystal XRD results are in good agreement with the reported values.

Table 1. Unit cell parameters of KNO_3 doped L-arginine

Crystals	**KNO_3**	**L-arginine**	**KNO_3 - L Arginine (1:3)**	**KNO_3 - L arginine (3:1)**
a (Å)	5.414	11.36	5.424	5.398
b (Å)	9.164	16.10	6.417	6.526
c (Å)	6.431	5.565	9.163	9.047
α,β,γ	α=β=γ =90º	α=γ =90º β=94º	α=β=γ =90º	α=β=γ =90º
Volume ($Å^3$)	319.07	1016.05	318.9	318.7
Crystal System	Ortho rhombic	Mono clinic	Ortho rhombic	Ortho rhombic

KNO_3 crystals had orthorhombic crystal system. L-arginine crystals had monoclinic crystal system. The grown crystals had orthorhombic crystal system. This proved that KNO_3 acted as the parent material and even though the doping with L-arginine did not alter the structure, change in lattice parameters was observed.

3.3 . FTIR ANALYSIS

FTIR spectroscopy can be used to identify the functional groups present in the compounds. FTIR spectrum of KNO_3 doped L-arginine crystals in the ratios (1:3), (1:1) and (3:1) were recorded by using BRUKER 66 VFT-IR spectrometer in the range 400-4000 cm^{-1} and are shown in figures3.a, 3.b, and 3.c respectively. Band assignments are given in table 2. NO bending is represented by the peak 825cm^{-1}. The peak at 987cm^{-1} is due to stretching of N-H [7]. The presence of carboxyl group [7] is confirmed by the peak at 1381cm^{-1}. The peak at 2924cm^{-1} is due to CH stretching vibration [8]. NH_2 stretching vibration [9,10] is confirmed by the peak 3363cm^{-1}.

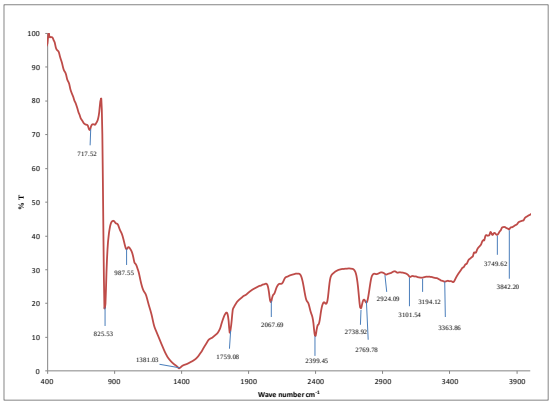

Figure: 3.a. FTIR spectrum of KNO_3 doped L-arginine (1:3)

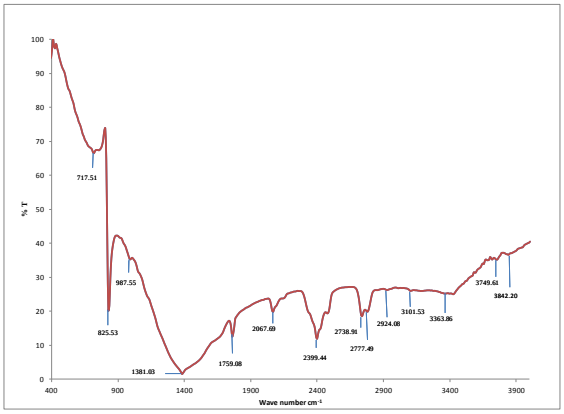

Figure:3.b. FTIR spectrum of KNO_3 doped L-arginine (1:1)

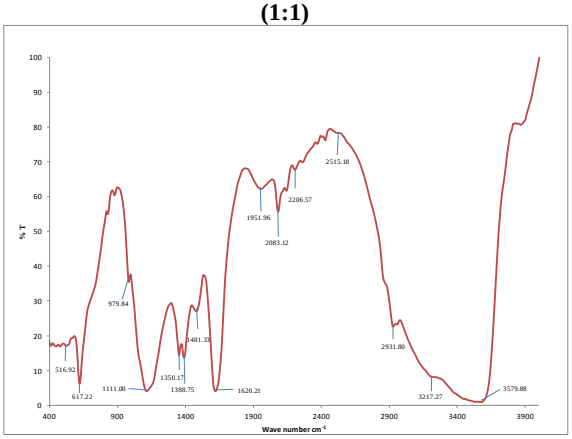

Figure: 3.c. FTIR spectrum of KNO_3 doped L-arginine (3:1)

Table 2. FTIR data table of KNO_3 doped L-arginine [2]

WAVE NUMBER (cm^{-1})			BAND ASSIGNMENT
(1:3)	(1:1)	(3:1)	
825.53	825.53	-	NO group bending of KNO_3
987.55	987.55	979.84	N-H stretching
1381.03	1381.03	1388.75	CH_2 bending
1381.03	1381.03	1388.75	Carboxyl group
2924.09	2924.08	2931.80	CH stretching vibration
3363.86	3363.86	3440.72	NH_2 stretching vibration

From the above observation it is noted that, for the doping ratios of (1:3) & (1:1) almost all the vibration frequencies remains same where as for the ratio (3:1) the value of vibration frequency is increased. This increase may be attributed due to doping.

3.4 . UV SPECTRAL ANALYSIS

To know the suitability of the grown single crystals for optical application [11], the optical transmission spectrum of grown crystals was recorded in the wavelength range 200-800 nm using a LAMBDA 35 Model UV-Vis spectrometer. The recorded spectrum of KNO_3 doped L-arginine in the ratios (1:3), (1:1) and (3:1) is shown in figure 4.

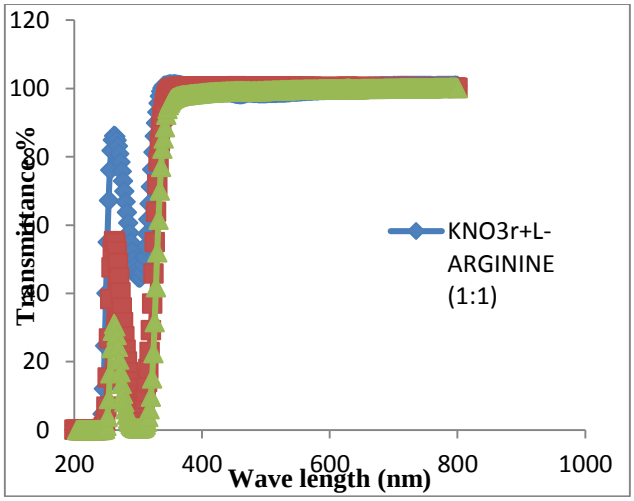

Figure.4. UV-Vis spectrum of KNO_3 doped L-arginine

The lower cut-off wave length for KNO_3 doped L-arginine in the ratios (1:3), (1:1) & (3:1) are 348 nm, 340 nm and 354 nm respectively. The transmittance of the crystals was found to be 98% in the entire visible region. This quality of the grown crystals makes them suitable for NLO applications. Band gap

energy values were calculated using the formula

$$Eg = hc/\lambda \min \quad eV$$

where, h is Plank's constant (Joule-sec), c is the speed of light (m/sec) and λ is the wavelength (m). The band gap values for KNO_3 doped L-arginine in the ratios (1:3), (1:1) and (3:1) are 3.56 eV, 3.65 eV and 3.5 eV respectively.

3.5 . MICRO HARDNESS STUDY

Mechanical strength of the material plays a key role in device fabrication [12]. Micro hardness measurements were carried out by Vickers hardness test at room temperature. The hardness measurements were taken for applied load varying from 25 to 100gm. The Vickers hardness number (H_v) was calculated using the relation

$$H_v = 1.8344\ P/d^2\ kg/mm^2$$

where,

P - the applied load in grams

d - the diagonal length of the indentation impression in mm

The variation of H_v with the applied load P for KNO_3 doped L-arginine crystals in the ratios (1:3), (1:1) and (3:1) is shown in figure 5.a. It is evident from the plot that the hardness number of the grown crystals increases with the applied load.[13]

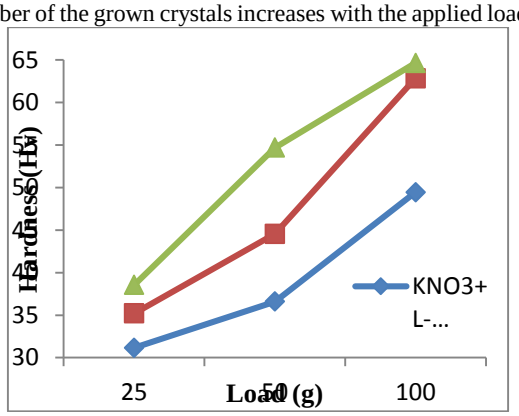

Figure:5a. The variation of H_v Vs load

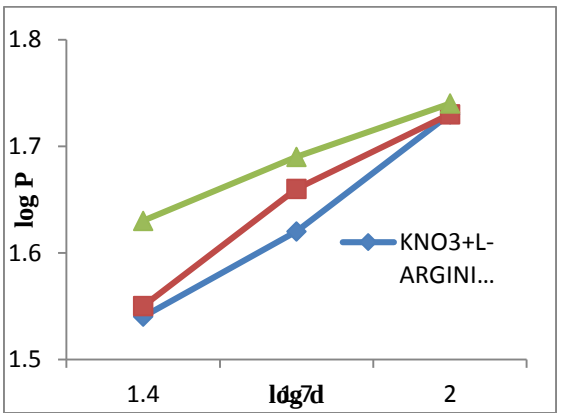

Figure5.b. Plot between log P Vs log d

Table 3. Hardening Index of KNO_3 doped L-arginine

CRYSTAL	Hardening Index (n)		
Ratio	(1:3)	(1:1)	(3:1)
KNO_3 L-arginine	3.7	4.2	6

A plot between log P Vs log d for KNO_3 doped L-arginine in the ratios (1:3), (1:1) and (3:1) are shown in figure 5.b. The values of hardening index were calculated and presented in Table 3. All the three crystals belong to soft category [14] as n > 1.6. It is also proved that the increase in doping percentage of KNO_3 in L-arginine decreases the hardness of the crystal.

4. CONCLUSION

Single crystals of Potassium Nitrate doped L-arginine in the ratios (1:3), (1:1) and (3:1) were successfully developed using slow evaporation technique. Crystalline nature of developed compounds was verified with PXRD analysis and reflections were duly indexed. The lattice parameters were found by single crystal XRD. The KNO_3 doped L-arginine crystals had orthorhombic crystal system. This proved that KNO_3 acted as the parent material and doping with L-arginine did not alter the structure .Various functional groups present in the crystals were identified through FTIR analysis. Optical behaviour of the crystals was analyzed with UV-Visible spectrum. It is found that the lower cut off wave length, transparency range and band gap energy are comparable for all the three ratios. In average the lower cut off wave length is around 347 nm, transparency range is 98% and band gap energy is 3.57 eV for KNO_3 doped L-arginine crystals. Mechanical part of the analysis says that the grown crystals are soft materials. From the above results, it is concluded that the grown crystals are suitable for NLO applications.

5. ACKNOWLEDGEMENTS

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