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# Synthesis, Electrical and Optical Properties of Nickel Sulphate Hexa Hydrate Single crystals Doped with L-Arginine

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**ABSTRACT**
L-Arginine doped Nickel Sulphate Hexa Hydrate (NSH) single crystals were grown by slow evaporation technique for different molar concentrations, viz., (0.2 to 1 mole% in steps of 0.2). The grown crystals were subjected to various studies. XRD data were collected from powder samples of the crystals. AC electrical measurements were carried out at various temperatures ranging from 40-75$^0$C. Results indicate an increase of the electrical parameters with the increase of temperature. The dielectric value suggests that the L-Arginine doped NSH single crystal is good for microelectronic application. The UV-Vis-NIR spectral studies were performed to analyze the optical absorption of the grown crystals in the range 200 – 1100nm. Results obtained were presented.
***Key words:*** Crystal growth, X-ray diffraction, Electrical and Optical property.

## I. INTRODUCTION

Now a days, the Scientists are in search for new conversion materials for various device applications and the recent interest is focused on the development of the new semi organic materials with improved properties. Single crystals are important materials for electronic, optical devices and laser crystals. The efficient NLO materials has resulted in the development of a new class of materials called semi-organics. These materials have the potential for combining the high optical nonlinearity and chemical flexibility of organic materials with the thermal stability of inorganic NLO materials. In the present investigation the single crystal of pure Nickel Sulphate Hexa Hydrate and L-Arginine doped Nickel Sulphate Hexa Hydrate was grown by slow evaporation method and the harvested crystals were characterized by X-ray diffraction studies. The title compound has good dielectric behavior and the results indicate an increase electrical parameter viz. $\sigma_{ac}$, $\varepsilon_r$ and tan $\delta$ with the increase of temperature. The optical transparency and optical constants were assessed employing UV–visible NIR studies in the range of 200–1100nm. The wide optical and gap of the grown crystal has been found.

## II. CRYSTAL GROWTH

The title compound was prepared by dissolving analar grade Nickel Sulphate Hexa Hydrate (NSH) by means of doping it with L-Arginine in different but definite molecular ratios. We have grown pure and L-Arginine doped crystals by the slow evaporation method at room temperature. NSH crystal was doped with L-Arginine in six NSH:L-Arginine molecular ratios, viz. 1:0.000, 1:0.002, 1:0.004, 1:0.006, 1:0.008 and 1:0.010.Approximate molar ratio of materials was taken using digital balance and dissolved in double distilled water. The solution of pH value 7 was stirred with magnetic stirrer and allowed to evaporate without disturbance. Optically good quality NSH single crystals have been grown within a period of 7 to 14 days. Initially very small crystals appeared then grew bigger in size. Out off grown crystals, best crystals were selected for further studies and are shown in Figure1.

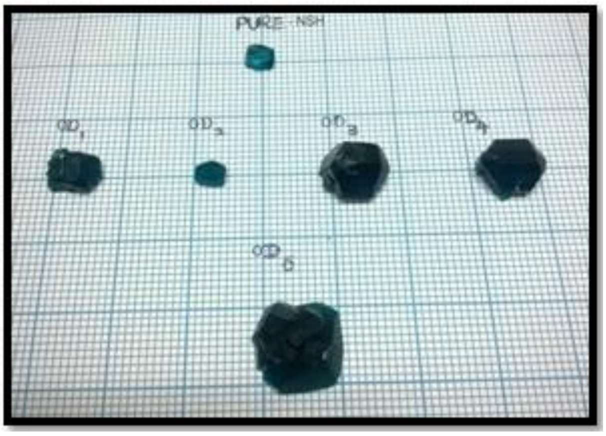



Fig1. *Photograph of the grown Pure and Doped NSH crystals*.

## III. CHARACTERIZATIONS

The PXRD data were collected from powdered sample for pure and doped NSH crystals using diffractrometer. The reflections were indexed. Analysis of the X-ray peaks was done by available methods and lattice parameters were determined [1, 2, 3].

The dielectric constant of the material can be measured using two probe method. The samples were cut and polished, opposite faces of the crystals were coated with good quality graphite to obtain a good ohmic contact with the electrodes. The capacitance and dielectric loss measurements were carried out for various temperatures ranging from $40^0$C to $75^0$C [4,5,6] using an Agilent 4284ALCR meter for various frequencies 20Hz,100Hz,1KHz,10KHz,100KHz,1MHz.

The dimension of the crystal was measured using a microscope. Air capacitances were also measured for the dimensions equal to that of the crystals [7, 8, 9].

The UV-Vis-NIR absorption spectra were recorded in the wavelength range 200-1100nm for all the six crystals grown by using Double beam spectrophotometer 2202.

## IV. RESULTS AND DISCUSSION

### 4.1 POWDER XRD

X-ray diffraction studies of solution grown NSH crystals was carried on XPERT-PRO using X-ray CuKα radiation (1.54059340) was used. The samples were scanned in the 2ϴ range of $10^0$-$70^0$ X-ray diffract to gram is shown in figure2. The technique is based on observing the scattered intensity when X-ray beam is incident on a sample. It is a function of incident and scattered angle, polarization, and wavelength or energy[10]. The figure2 shows the powder X-ray diffraction pattern, the presence of prominent Bragg's peak 2ϴ angle confirms the perfect crystal line structure. The diffraction data almost matches with JCPDS data for pure NSH crystals. Table1 indicates the unit cell parameters satisfy the condition for Tetragonal system i.e., $a=b\neq c$ and $\alpha=\beta=\gamma=90^0$ from the above data and it may be concluded that the grown crystals of NSH have tetragonal system with very slight changes in the peak positions, slight change in the relative intensities, cell volume and lattice parameters and these slight changes are due to the doping of L-Arginine in NHS crystal.

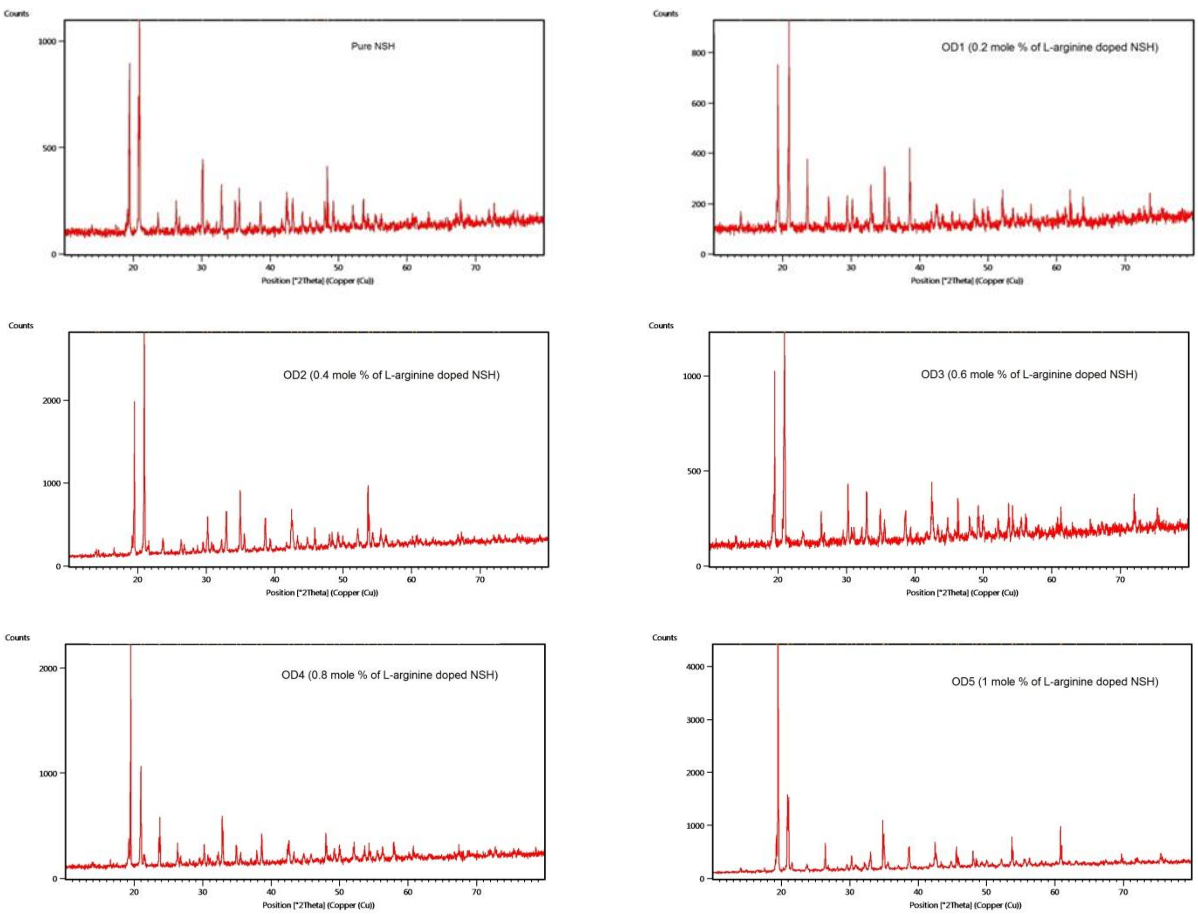



Fig2. *Indexed Powder XRD pattern of pure and doped NSH crystal*

Table 1 – *Calculated Lattice parameter for pure and doped NSH single crystals.*

| Samples | Crystal system | Unit cell parameters | | | Volume |
|---|---|---|---|---|---|
| | | a=b | c | | |
| Pure NSH | Tetragonal | 6.782 | 18.266 | $\alpha=\beta=\gamma=90^0$ | 840.130 |
| OD1(0.2 mole % of L-Arginine doped NSH) | | 6.770 | 18.295 | | 838.505 |
| OD2(0.4 mole % of L-Arginine doped NSH) | | 6.782 | 18.266 | | 840.130 |
| OD3(0.6 mole % of L-Arginine doped NSH) | | 6.774 | 18.122 | | 831.563 |
| OD4(0.8 mole % of L-Arginine doped NSH) | | 6.746 | 18.314 | | 833.413 |
| OD5(1 mole % of L-Arginine doped NSH) | | 6.761 | 18.307 | | 836.901 |

### 4.2 DIELECTRIC STUDIES

The AC electrical conductivity ($\sigma$) was calculated using the relation.

$$\sigma = \varepsilon_0\, \varepsilon_r\, \omega \tan \delta$$

Where $\varepsilon_0$ is the permittivity of free space ($8.85\times10^{-12}$ $C^2$ $N^{-1}$ $m^{-2}$) and $\omega$ is the angular frequency ($\omega = 2\pi f$; f is the frequency) [11].

The dielectric constant ($\varepsilon_r$) of the crystal was calculated using this relation

$$\varepsilon_r = \left(\frac{A_{air}}{A_{crys}}\right)\left(\frac{C_{crys} - C_{air}\left(1 - \frac{A_{crys}}{A_{air}}\right)}{C_{air}}\right)$$

Where $A_{cry}$ is the area of the crystal touching the electrode and $A_{air}$ is the area of the electrode. The dielectric losses were measured for all the grown crystals by a method adopted by previous authors [12, 13]. The $\varepsilon_r$, tan$\delta$ and $\sigma_{ac}$ values obtained in the present study with different frequencies are shown in Figures 3-5.The $\varepsilon_r$, tan$\delta$ and $\sigma_{ac}$ values obtained at $50^0$C with all the frequencies are provided in Table2. It has been found that the $\varepsilon_r$, tan$\delta$ and $\sigma_{ac}$ values increase with the increase in temperature. This is a normal dielectric behavior.

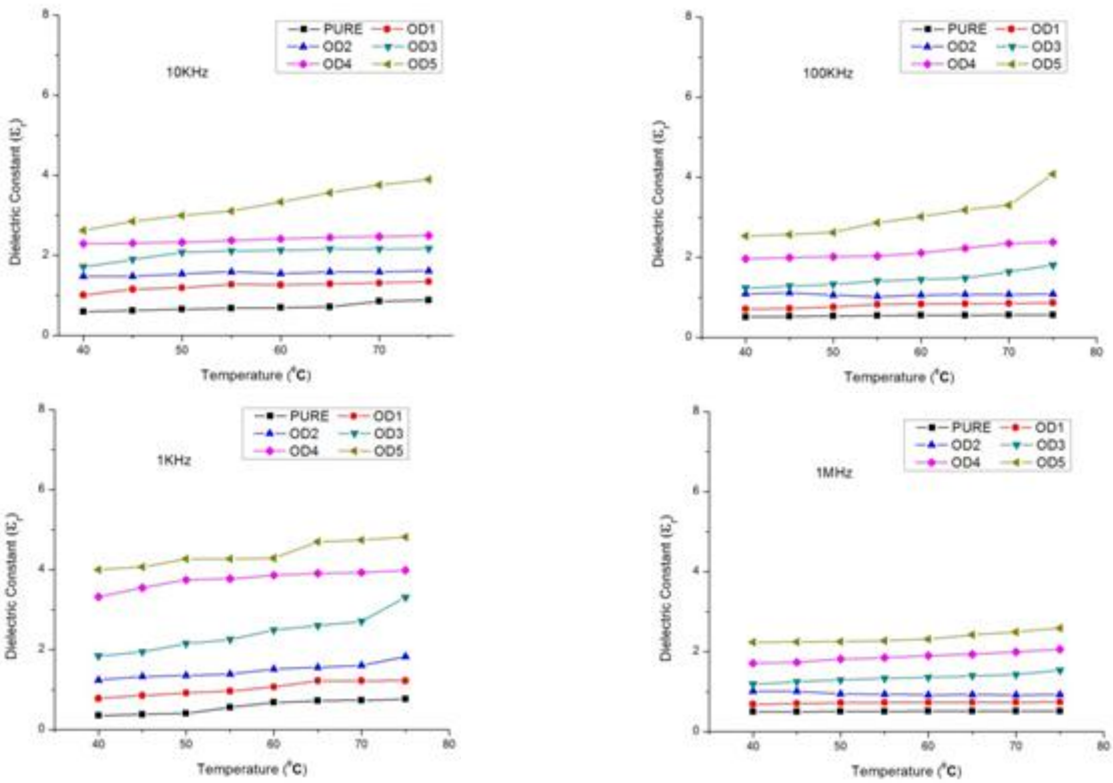



Fig3. *The dielectric constants for pure and doped NSH*

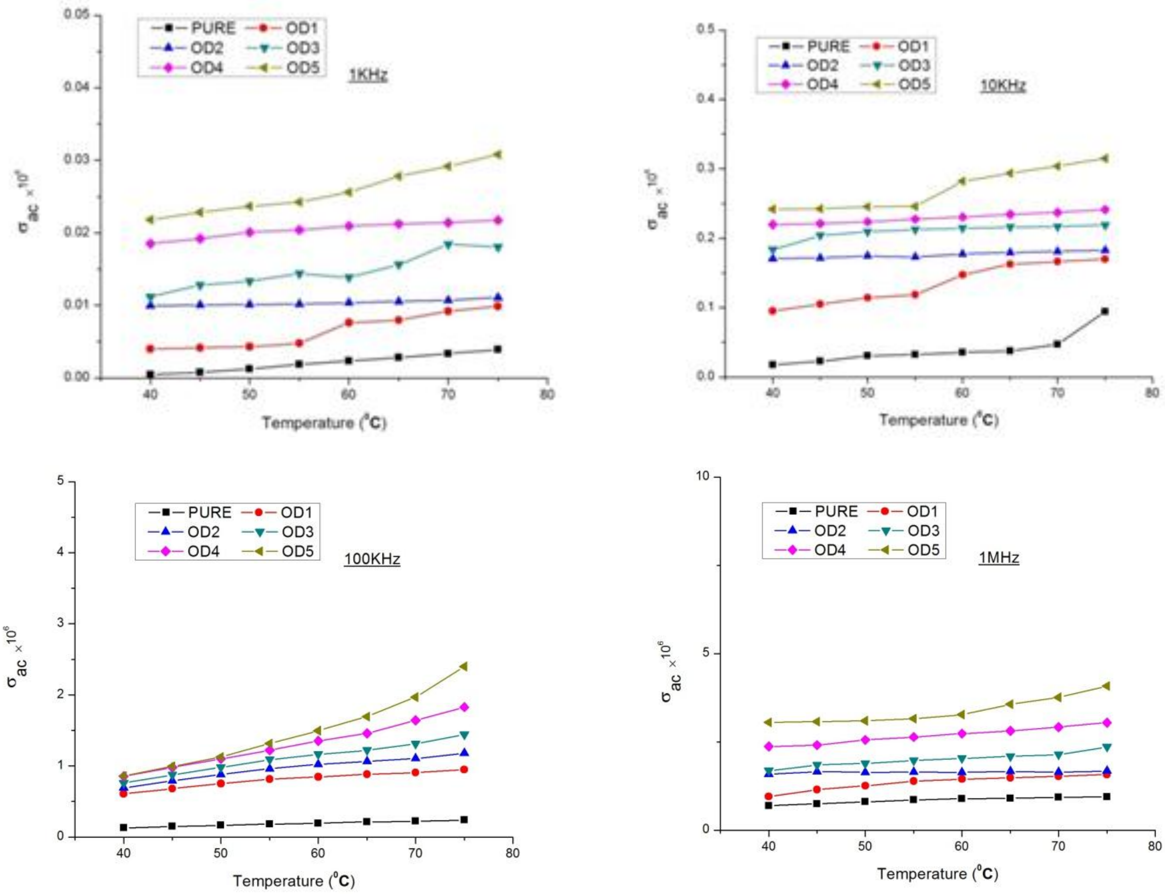
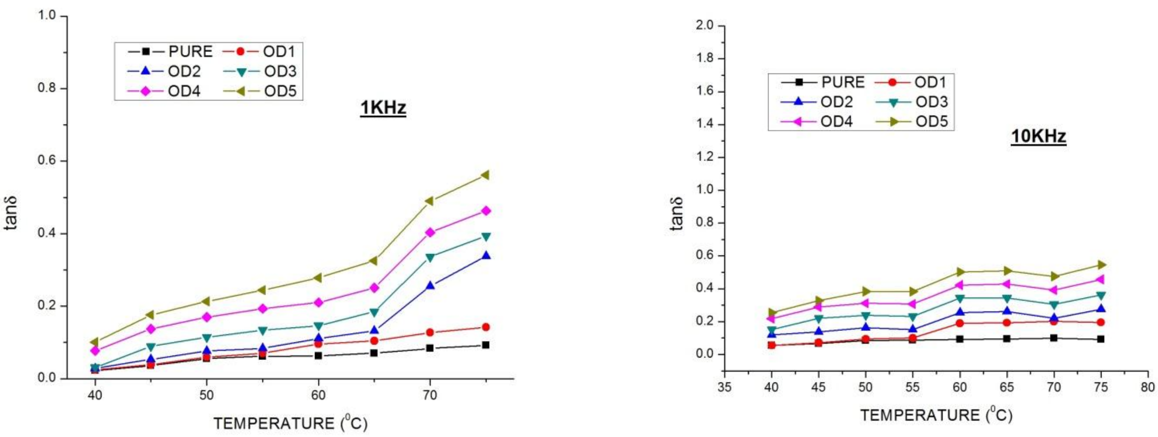



Fig4. *The AC electrical conductivities (x10$^{-6}$mho/m) for pure and doped NSH crystal*



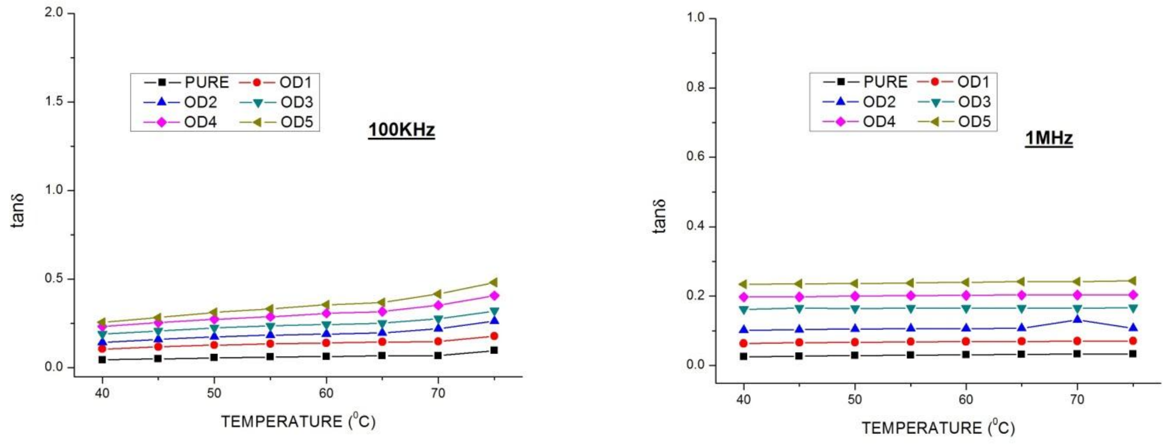



Fig5. *The dielectric loss factors for pure and doped NSH crystals*

Table 2 – The $\varepsilon_r$, tanδ and $\sigma_{ac}$ ($x10^{-6}$ mho/m) values at $50^0$C for pure and doped NSH.

| Samples | Parameter | With a frequency of | | | |
|---|---|---|---|---|---|
| | | 1KHz | 10KHz | 100KHz | 1MHz |
| Pure NSH | $\varepsilon_r$ | 22.190 | 27.147 | 14.324 | 12.342 |
| | Tanδ | 0.055 | 0.085 | 0.056 | 0.029 |
| | $\sigma_{ac(x10^{-6}\ mho/m)}$ | 0.068 | 1.278 | 4.442 | 19.857 |
| NSH+ OD1 | $\varepsilon_r$ | 88.667 | 100.568 | 46.320 | 33.278 |
| | tanδ | 0.004 | 0.059 | 0.073 | 0.037 |
| | $\sigma_{ac(x10^{-6}\ mho/m)}$ | 0.021 | 3.345 | 18.690 | 68.431 |
| NSH+ OD2 | $\varepsilon_r$ | 37.010 | 51.476 | 35.346 | 94.540 |
| | tanδ | 0.017 | 0.068 | 0.046 | 0.023 |
| | $\sigma_{ac(x10^{-6}\ mho/m)}$ | 0.0351 | 1.937 | 9.017 | 120.329 |
| NSH+ OD3 | $\varepsilon_r$ | 107.092 | 121.564 | 78.581 | 94.838 |
| | tanδ | 0.038 | 0.076 | 0.051 | 0.029 |
| | $\sigma_{ac(x10^{-6}\ mho/m)}$ | 0.226 | 5.157 | 22.220 | 152.061 |
| NSH+ OD4 | $\varepsilon_r$ | 54.451 | 27.249 | 34.182 | 16.060 |
| | tanδ | 0.055 | 0.073 | 0.049 | 0.026 |
| | $\sigma_{ac(x10^{-6}\ mho/m)}$ | 0.168 | 1.099 | 9.229 | 23.464 |
| NSH+ OD5 | $\varepsilon_r$ | 89.223 | 32.444 | 78.166 | 8.303 |
| | tanδ | 0.043 | 0.072 | 0.044 | 0.041 |
| | $\sigma_{ac(x10^{-6}\ mho/m)}$ | 0.2147 | 1.293 | 18.985 | 19.072 |

**4.3 UV-Vis-NIR SPECTROSCOPY**

The UV-Vis-NIR spectroscopy of the pure and doped NSH crystals was performed by using Double Beam Spectrophotometer. The crystals have a good optical transmission in the entire visible region and the lower cut off wavelength is observed. The band gap energies were calculated and presented in Table3 and Figures 6-7 showed the UV absorbance and Taue plots.

The single crystals are mainly used for optical applications. Thus the study of optical transmission range of grown crystal is important. The optical transmission spectrum was recorded using Double Beam spectrophotometer in the wavelength region 200 – 1100nm. The UV Vis NIR absorption spectra are observed in the present study. Efficient non-linear optical crystals have an optical transparency lower cut-off wavelengths between 200-400nm [14]. The lower cut off region lies in the range 384 nm. The low absorption in the visible and NIR regions along with low cut off wavelengths confirm the suitability of the grown crystals for NLO applications. The grown crystals has good transmission in UV as well as in visible regions.

Table 3 - *Results of Band Gap Energy*.

| Samples | Band gap energy $E_g$(eV) |
|---|---|
| Pure NSH | 5.02 |
| OD1(0.2 mole % of L-Arginine doped NSH) | 5.00 |
| OD2(0.4 mole % of L-Arginine doped NSH) | 5.42 |
| OD3(0.6 mole % of L-Arginine doped NSH) | 5.03 |
| OD4(0.8 mole % of L-Arginine doped NSH) | 5.01 |
| OD5(1 mole % of L-Arginine doped NSH) | 4.96 |

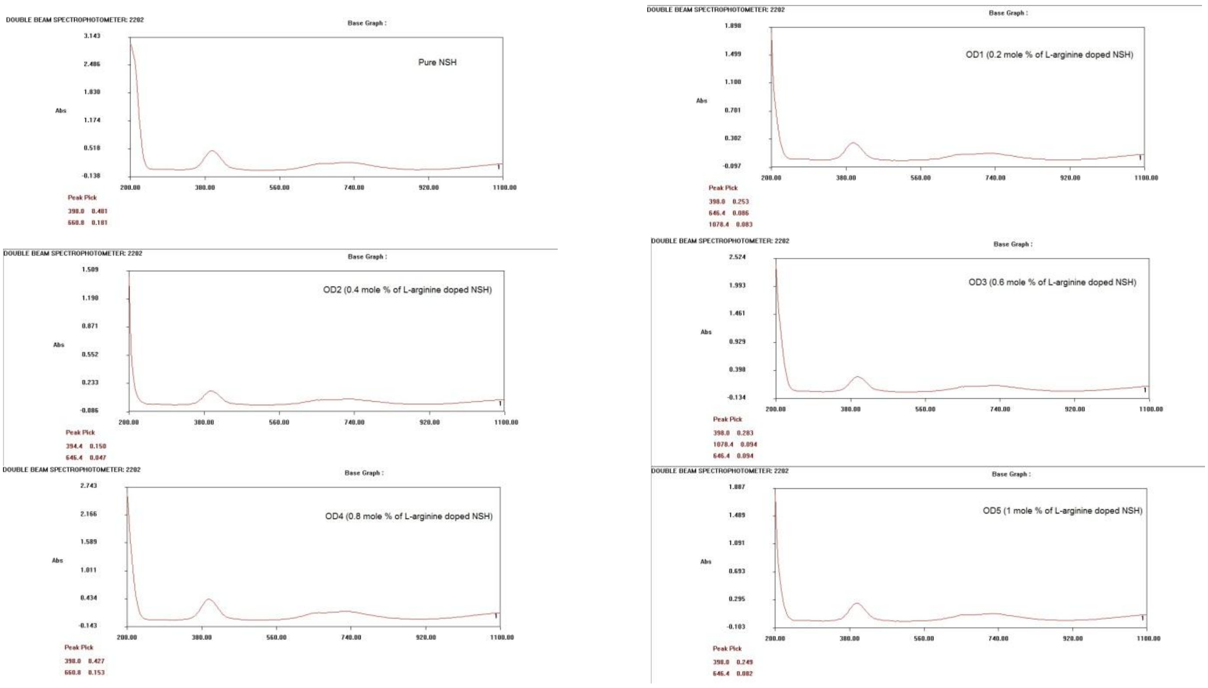



Fig6. *UV-Vis-NIR spectroscopy of the pure and doped NSH crystals.*

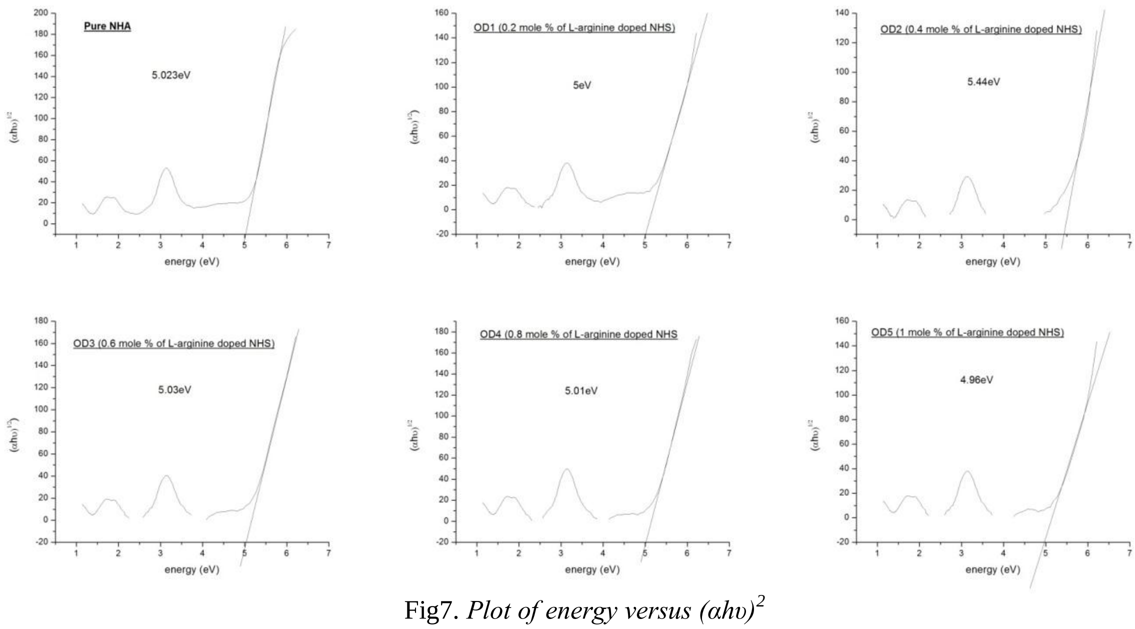



Fig7. *Plot of energy versus (αhυ)*$^2$

## V. CONCLUSION

Pure and doped NSH crystals were grown by slow evaporation method and characterized by PXRD, conductivity and optical (UV-Vis-NIR) measurements. The PXRD of spectra confirms the crystalline perfection of the grown crystals. Lattice parameters calculated from the XRD pattern of the pure and doped NSH crystals and determination of lattice volume indicate that the impurity molecules have entered in the crystal matrix of NSH. The dielectric constants were found to increase with increasing temperature. The increase of AC conductivity with increase of temperature has been understood as essentially due to temperature dependence. The UV-Vis-NIR spectral studies revealed that the band gap of pure NSH was altered due to doping of L-Arginine. The preset study indicates that dopant L-Arginine leads to the discovery of promising low value dielectric materials. So it is more interesting that doped crystals are useful in micro electronic industry.

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