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Electrical measurements on $ZnSO_4$ doped L-Threonine single crystals

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Abstract: *Growing organic NLO material is a fascinating field of research today due to its high SHG efficiency. In the present work $ZnSO_4$ doped L-Threonine crystals were grown by slow evaporation technique. The grown crystals were electrically characterized by measuring the capacitance values for various temperatures at various frequencies. The electricalparameters like dielectric constant, dielectric loss, a.c. conductivity and a.c .activation energy were calculated . All the parameters found to vary non linearly with dopant concentration.*

Keywords: *[Dielectric parameters, Dielectric Constant, Dielectric loss , a.c. conductivity, a.c. activation energy]*

I. Introduction

Growing organic NLO materials is a fascinating field of research today due to its high SHG efficiency, even though the material have low mechanical strength. Of the several organic NLO materials L-Threonine is a important and attractive organic NLO material. It is a polar amino acid which shows higher SHG efficiency than other amino acids [1]. L-Threonine belongs to orthorhombic crystal system with space group $p_{2_1}\ p_{2_1}p_{2_1}$ and lattice parameters a=13.611$Å$, b=7.738Å and c=5.1444Å [2]. The SHG efficiency of a material was found to be comparable to that of KDP crystal which is frequently used in commercial devices. In the resent study, the zinc doped L-Threonine crystals were grown from the aqueous solution by slow evaporation technique and electric measurements were made for different temperatures at different frequencies. The electrical parameters like dielectric constant ,dielectric loss, a.cconductivity and a.c.activation energy were calculated. The results obtained are discussed below.

II. Experimental details

Commercially available AR grade L-Threonine, $ZnSO_4$ and doubly distilled water were used to prepare the solution. Saturated solution of L-Threonine was prepared at 35℃ according to the solubility data available in the literature [3]. Pure and $ZnSO_4$ doped L-Threonine single crystals were grown from aqueous solution by slow evaporation technique for the various dopant concentration ratio viz.1:0.002, 1:0.004, 1:0.006, 1:0008 and 1:0.01. In the present study, totally six crystals were grown.

The dielectric constant and loss factors of all the grown crystals were determined by the method followed by several authors [4-8]. The capacitance (C) and dielectric loss factor (tan δ) measurements were carried out to an accuracy of ±1 % with Agilant 4284 ALCR meter in the temperature range of 40° to 130℃ with five different frequencies viz. 1KHz, 10KHz, 100KHz, 1MHz and 2MHz. The observations were made while cooling the sample. Temperature was controlled to an accuracy of ±0.5ºC. Air capacitance (C_{air}) was also measured for thickness equal to that of the sample crystal. The grown crystals were polished and opposite faces of the sample crystals were coated with good quality graphite to obtain a good conductive surface layer. They were annealed for two hours at ∼ 150℃ to remove moisture content if present

The dimension of the sample crystals were measured using travelling microscope (LC=0.001cm). The dielectric constant (ε_r) of the crystal was calculated using the relation

$$\varepsilon_r=\left[\frac{C_{crys}-C_{air}\left[1-\frac{A_{crys}}{A_{air}}\right]}{C_{air}}\right]\left[\frac{A_{air}}{A_{crys}}\right],$$

Where, C_{crys} is the capacitance with crystal (including air),
C_{air} is the capacitance of air,
A_{air} is the area of electrode.
A_{crys} is the area of surface of the crystal in contact with the electrodes
The A.C conductivity (σ_{ac}) was calculated using the relation

$$\sigma_{ac} = \varepsilon_0\varepsilon_r\omega \tan\delta$$

Where, ε_0 is the permittivity of free space ($8.85\times10^{-12}\ C^2N^{-1}m^{-2}$) and
ω is the angular frequency ($\omega = 2\pi f$),

f is the frequency of a.c.
The values were fitted into the equation

$$\sigma_{ac} = \sigma_0 \exp\left(\frac{-E_{ac}}{kT}\right)$$

Here k is the Boltzmann's constant,
T is the absolute temperature and
σ_0 is a constant depending on the material.
By plotting a graph between 1000/T vs ln σ_{ac} ,from the slope of the line of best fit $E_{a.c}$ can be determined

III. Results and Discussion

3.1. Growth procedure

The photograph of all the pure and doped crystals of L-Threonine are shown in figure 1. All the crystals grown in the present study are transparent and good quality crystals. They are more hard and stable All the crystals grown in c- direction, needle shaped and about 20 mm length

Fig 1: Photograph of all the grown crystals

Analysis of X-ray diffraction peaks by the available method [2] shows that the peaks were indexed with orthorhombic system. The lattice parameters determined for all the six crystals in the present study are provided in the table 1. The unit cell parameters determined for pure L-Threonine from SXRD also provided in table 1 for comparison purpose. The lattice parameter already reported in the literature are given in bracket. This shows that lattice parameters calculated in the present study well agreed with the reported as well as single crystal XRD data.

Table 1: Values of lattice parameters of pure and doped L-Threonine

System	Lattice Parameters			Volume
	a Å	**b Å**	**C Å**	**$Å^3$**
SXRD for Pure L-Threonine	5.140 [5.139]	7.738 [7.723]	13.546 [13.579]	538.769 [538.9][9]
PXRD for Doped L-Threonine				
Pure	5.123	7.747	13.680	528.642
1:0.002Zn	5.042	7.784	13.680	536.928
1:0.004Zn	4.914	7.785	13.414	513.151
1:0.006Zn	4.918	7.760	14.113	538.626
1:0.008Zn	4.815	7.880	13.316	506.910
1:0.010Zn	4.941	7.748	13.556	518.977

3.2 Dielectric constant

The variation of dielectric constant with temperature and a.c. frequency for all the grown crystals is shown in the figure 2 and figure3 respectively. .The dielectric constant determined in the present study for all the samples increase with increase in temperature and decreases with increase in frequency.

Variation of ε_r with temperature is generally attributed to the crystal expansion of the electronic and ionic polarization and the presence of impurities and crystal defectes. At low temperature, the variation of ε_r is mainly due to expansion and electronic and ionic polarization. At high temperature the increase is mainly attributed to the thermally generated charge carriers and impurity dipoles. Varotsos [11] has shown that out of the two contributions, the electronic polarization practically remains constant. The increase in dielectric constant with temperature is essentially due to the temperature variation of ionic polarizability. Varotsos [11] and Owen's [12] have proposed simple methods for the calculation of dielectric constants at different temperatures. Their relation also shows that the dielectric constant increases with increase in temperature.

The decrease in dielectric constant with increase in a.c frequency is attributed to the interfacial polarization in which mobile charge carriers are impeded by a physical barrier that inhibits producing a localized polarization of the material. At high frequencies, the dielectric constant becomes almost constant. The dielectric constant vary nonlinearly with dopant concentration. The dielectric constants of all the Zn doped crystals are found to be less than that of the pure L-Threonine. This implies that the dopant addition decreases the dielectric constant value. It is found that the dielectric constant of the doped crystal of concentration 1:0.01Zn and 1:0.006Zn crystals are lower than that of pure and other doped crystals.

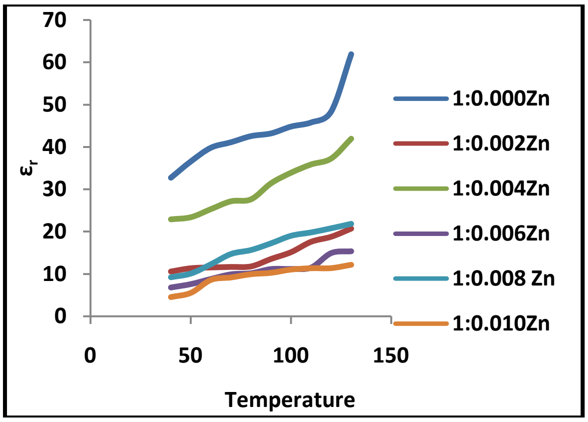

Fig 2: Variation of dielectric with temperature for all the grown crystals at constant frequency 1KHz

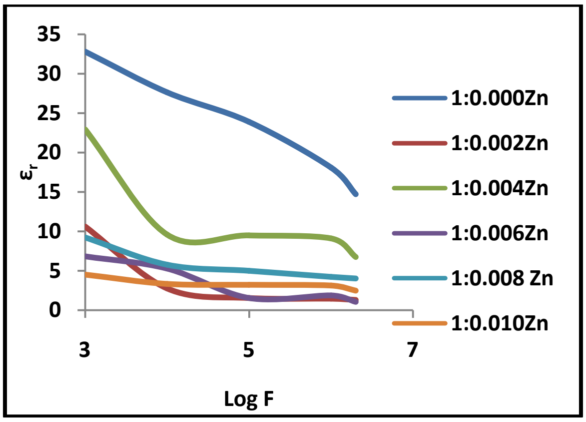

Fig 3: Variation of dielectric with log f for all the grown crystals at constant temperature 40℃

3.3. Dielectric loss

The variations of dielectric loss with temperature and a.c. frequency are shown in figure: 4.and figure 5. The dielectric loss factor is found to be increased with temperature and decrease with increase in applied a.c. frequency like the dielectric constant values. The low dielectric loss with high a.c.frequency proves that these materials possess enhanced optical quality with lesser defects. This characteristic is an important parameter for nonlinear optical materials in their applications [10]. The dielectric loss for Zn doped crystals are found to be less than that of pure L-Threonine except for the concentration 1:0.002 Zn and 1:0.006 Zn. A wide shift in the loss is observed for the sample 1.0.006Zn compared to the others.

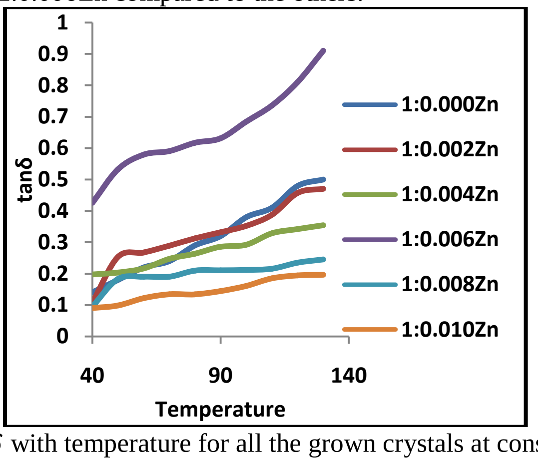

Fig 4: Variation of tanδ with temperature for all the grown crystals at constant a.c.frequency 1KHz

3.3 a.c. Conductivity

The variation of A.C conductivity (σ_{ac}) with temperature and a.c frequency are respectively shown in the fig. 6 and fig. 7. It is observed from the graphs that the conductivity values increases with increase in both temperature and applied frequency. The conductivity of the doped crystals are found to be less than that of the pure L-Threonine crystal and it varies non-linearly with dopant concentration. The conductivity of the concentration 1:0.006 Zn sample is greater than other doped crystals.

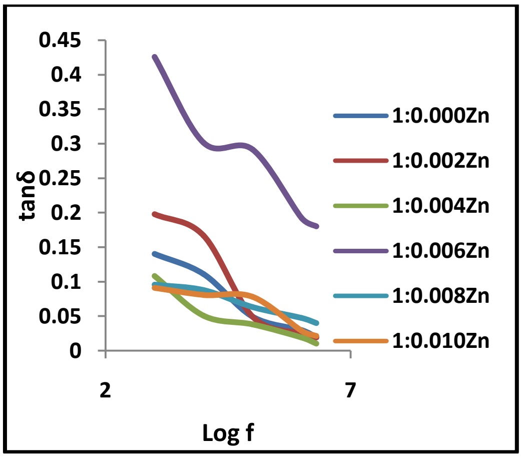

Fig 5; Variation of tan δ with log f for all the grown crystals at constant temperature 40℃

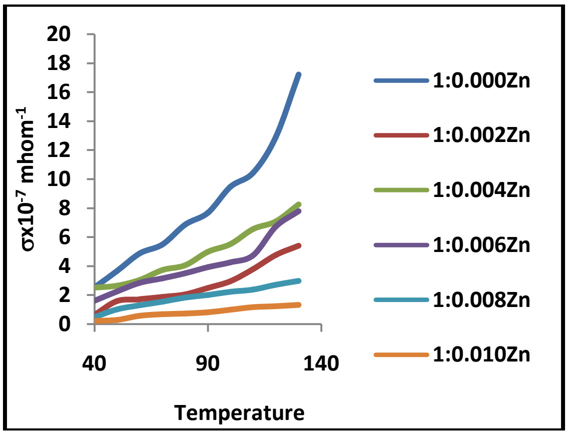

Fig 6: Variation of conductivity with temperature for all the grown crystals at constant frequency 1KHz

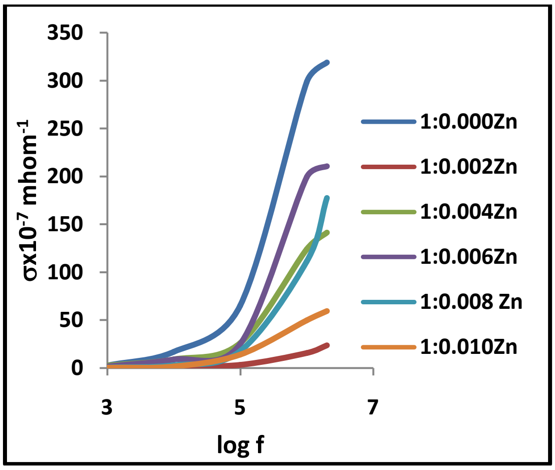

Fig 7: Variation of conductivity with log f for all the grown crystals at constant temperature 40℃

3.4. A.C. activation energy

The activation energy for all the grown crystal are provided in table 2. It is observed that the a.c. activation energy varies non-linearly with dopant concentration

Table 2: Values of a.c. activation energy of all grown crystals

System	a.c Activation Energy (eV)
Pure	0.209
1:0.002 Zn	0.15003
1:0.004 Zn	0.2254
1:0.006 Zn	0.205
1:0.008 Zn	0.1669
1:0.010 Zn	0.1839

IV. Conclusion

The dielectric constant and dielectric loss increases with temperature and decreases with frequency. But the a.c.conductivity increases both in temperature and frequency. The electrical parameters like dielectric constant, dielectric loss, a.c.conductivity and a.c.activation energy varies non-linearly with dopant concentration

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