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Study on Mechanical and Dielectric Properties of 1, 3, Dimethyl Urea Doped L-Arginine Phosphate Monohydrate Single Crystals

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ABSTRACT

Single crystals of pure and 1mol% 1,3-dimethyl urea doped L-arginine phosphate monohydrate were grown from solution by a slow solvent evaporation method. Grown crystals were subjected to the dielectric measurement study in the frequency range 1 KHz-1MHz at various temperatures. The dielectric constants and dielectric losses have been determined. The temperature and frequency dependence of dielectric constant and dielectric loss has been discussed. Other parameters like ac conductivity has been estimated and presented in present work.Hardness study confirms the doped LAP crystal have moderate hardness value.

Keywords : Dielectric Constant, Mechanical, Ac Conductivity.

I. INTRODUCTION

L-arginine phosphate monohydrate (LAP) is a renowned biaxial nonlinear optical (NLO) crystal. It crystallized into the monoclinic crystal structure with $P2_1$ space group. It shows excellent NLO properties such as optical damage threshold and second harmonic generation (SHG) efficiency [1]. A microbes has been develop into the LAP solution during crystal growth and an attempts have been made to suppress the development of microbial growth [2-4]. Since 1983, many researchers have investigated the effect of various dopants on the physio-chemical properties of the LAP crystal [5-10].

Urea (H_2NCONH_2) and its derivatives are renowned organic compound. They show interesting NLO properties. Other urea derivatives like monomethylurea ($H_2NCONHCH_3$), 1,1-dimethylurea ($H_2NCON(CH_3)_2$), 1,3-dimethylurea ($CH_3HNCONHCH_3$) and phenylurea ($H_2NCONHCH_5$) have been investigated in the light of NLO applications and few of them found suitable for SHG applications [11]. 1,3-dimethyl urea is derived from urea by replacing two hydrogen atoms of two $-NH_2$ groups by $-CH_3$ groups. It has one H-donor site while two acceptor sites. It is capable of forming multiple hydrogen bonds. It was difficult to crystallized it and handle too, as it is hygroscopic. NLO properties of 1,3-dimethylurea have been studied in details [12-13].

In the our previous communication, we reported the effect of 1,3-dimethyl urea doping on the linear and nonlinear optical properties of LAP crystal [8]. It is evident that, doping of 1,3-dimethyl urea improves optical properties of the host LAP crystal.

Now, we are presenting the dielectric study of Pure and (1mole%) 1,3-dimethyl urea doped LAP crystals grown by slow evaporation of solution growth method at a constant temperature. The effect of dopant on the dielectric constant, dielectric loss, ac conductivity were studied.

II. METHODS AND MATERIAL

Material synthesis and Crystal growth

Organic L-arginine and inorganic phosphoric acid were taken in equimolar ratios in double distilled water. The semiorganic compound LAP was synthesized as per reaction mentioned elsewhere [9]. The solution of LAP was continuously stirred, filtered and dried optically to get a title compound. The purity of the LAP compound was further enhanced by repeated recrystallization from aqueous solution. The crystals of pure and 1,3-dimethyl urea doped LAP were grown by mixing 1mole% 1,3-dimethyl urea in LAP and designated as LAP (pure) and LDMU2 respectively. The good quality single crystals were grown within 25 days by allowing slow evaporation of solvent at a constant temperature.

III. RESULTS AND DISCUSSION

3.1 Dielectric Study

The crystals of pure LAP and LDMU2 having dimensions 1.08×3.2×4.3mm^3 and 24.44×1.95×3.85mm^3, respectively were used to study the electric properties. The dielectric constants and dielectric loss have been calculated using the formulae as given in equations $\varepsilon_r = \frac{Ct}{\varepsilon_0 A}$ and $\varepsilon'' = \varepsilon_r D$, Where, C is the capacitance, d is the thickness of the crystal and A is the silver-pasted area of the crystal sample. The dielectric loss (ε") was calculated using relation [8]. The variation of dielectric constant with applied frequency for pure LAP and LDMU2 crystals are shown in Fig.1 (a and b), respectively. It has been observed from the nature of graphs that, the dielectric constant of all crystals decreases with increase in frequency and reaches a constant low value at high frequencies at all temperatures. The high values of dielectric constant at low frequencies may be due to the contributions of total polarization by excitation of bound electrons, lattice vibrations, dipole orientation and space charge polarization. Space charge polarization contributes significantly in lower frequency region. The contribution of above-mentioned polarization decreases gradually as the frequency increases. This may be due to dipole unable to synchronize with the applied alternating electric field as frequency increases [14]. The dielectric constant of all crystals is almost temperature independent in high frequency region.The dielectric constants of doped crystal in high frequency region are larger than pure LAP crystal.

The crystals with high dielectric constant lead to more power dissipation. According to Miller rule, the lower value of dielectric constant at higher frequencies is a suitable parameter for the enhancement of SHG coefficient [16].

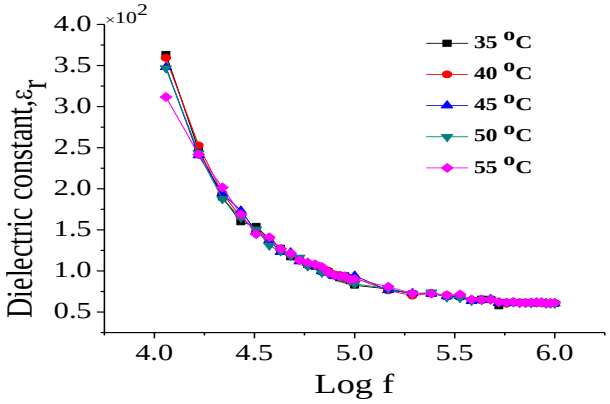

Fig. 1(a): Log f vs. dielectric constant of pure LAP crystal

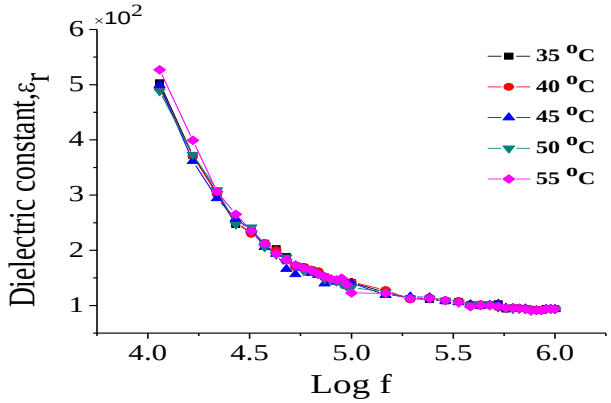

Fig. 1(b): Dielectric constant vs. logf for LDMU2.

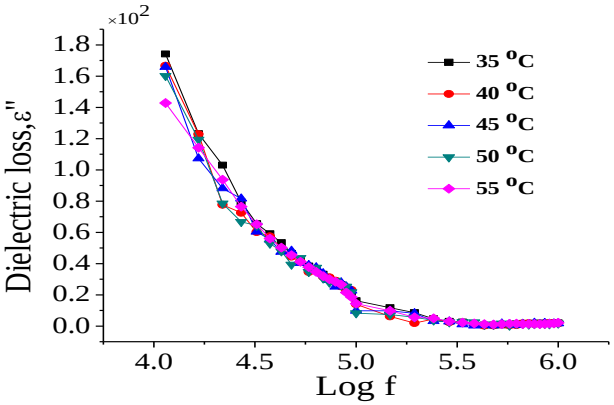

Fig.1(c): Log f vs. dielectric loss of pure LAP

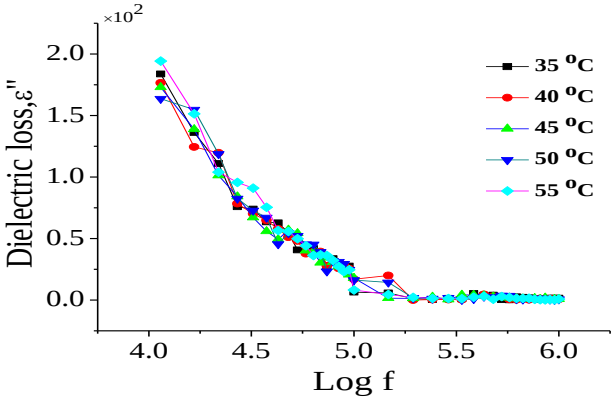

Fig.1(d): Dielectric loss vs. log f of LDMU2

The quality of the crystal can be judged from the dielectric loss study. Dielectric loss of the crystal is defect sensensitive. High dielectric loss value indicates the presence of large defect density in crystal and ultimately poor crystal quality. The variation of log f against dielectric loss at different temperatures for pure LAP and LDMU2 crystals are plotted in the graph shown in Fig.1(c) and 1(d), respectively. The dielectric loss decreases with increasing frequency and it is almost constant in high frequency region at all temperatures. The variation in dielectric loss results due to variation in polarization mechanism [16]. At high frequency, crystal show almost temperature independent dielectric loss for all crystals. The low value of dielectric loss suggests good optical quality of the crystal with fewer defects, which is the desirable property for electro-optic and other NLO applications [9].

3.2 ac Conductivity

The ac conductivity of the pure LAP and LDMU2 single crystals with the applied frequency is calculated using equation $\sigma_{ac} = \frac{2\pi fct}{A}$, Where, C is the capacitance, t is the thickness, A is the area of the crystal and f is the frequency of the applied field [18]. The variation in ac conductivity of pure LAP and LDMU2 single crystals with frequency is shown in Fig. 3(a) and 3(b) respectively. ac conductivity of pure LAP and LDMU2 crystal increases exponentially with applied frequency. Larger value of ac conductivity in all crystals at higher frequency may be due to the presence of polaron hopping process and other defective process [17]. There has no major change in the ac conductivity observed with temperature in the investigating range of frequency for pure LAP and LDMU2 crystals. In ac conductivity, low temperature region is known as extrinsic region and it is defect sensitive region. Mostly in extrinsic region, ac conductivity takes place due to presence of internal defects in crystals or by addition of impurity creates defects or vacancy in crystals or modification in bondings [18]. L-defect (vacant hydrogen bonds) and D-defect (doubly hydrogen bonds) contribute in the ac conductivity. ac conductivity of the doped LAP crystal was found to be higher than the pure LAP. It may be due to the addition of impurity create impurity vacancies or defects contribute in the ac conductivity in low temperature region. An influence of temperature on ac conductivity for pure LAP crystal is observed in only lower frequency region. It

indicates that, there is no contribution of thermally generated vacancies in high frequency regions.

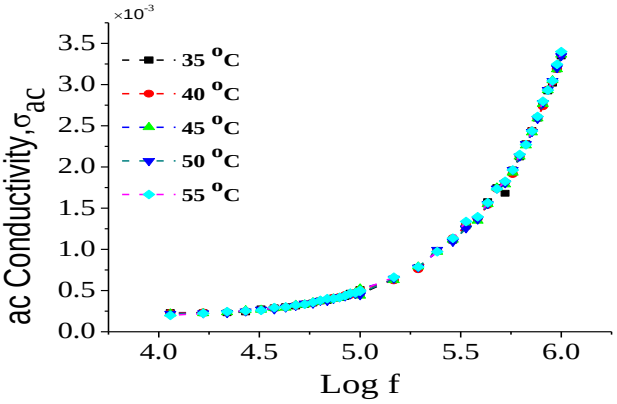

Fig. 3(a): Log f vs.ac conductivity for pure LAP single crystal

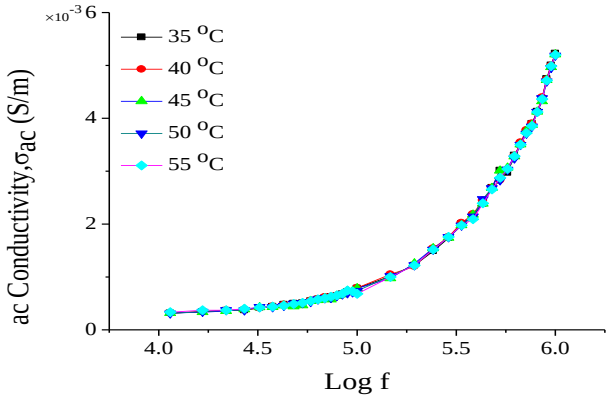

Fig.3(b): Log f vs. ac conductivity for LDMU2

3.3 Mechanical Study

The mechanical hardness study of the pure and doped crystals was carried out by Vicker's hardness test at room temperature for various dwell times (5, 10, and 15 s). Several indentations were made on the (1 0 0) face of pure and doped crystals for constant load of 5g. Hardness number was estimated on Future Tech Micro hardness Tester FM-700 hardness tester fitted with diamond indentor. Elastic stiffness constants (C_{11}) were also calculated using Wooster's empirical relation $C_{11} = [Hv]^{7/4}$ [19].The hardness number (Hv) and elastic stiffness constant (C_{11}) with variation in dwell time is presented in Table 1.

Table 1: Hardness number (Hv) and elastic stiffness constant (C_{11}) of pure and doped LAP single crystals.

Crystal	Dwell time (s)	5	10	15
LAP [19]	Hv (kg/mm²)	78.3	65.2	59.1
	C_{11}(×10¹⁴, Pa)	2061.02	1496.01	1259.73
LDMU2	Hv (kg/mm²)	80.0	58.0	52.6
	C_{11} (×10¹⁴, Pa)	2139.97	1218.99	1027.37

The hardness value decreases with increase in dwell time for all crystals. It indicates that, crystals cannot sustain the applied load for longer time period. This may be attribute to indentation energy transfer to crystal and utilized to generate the defects in the grown crystal by breaking multiple bonds. This makes the crystals softer and softer with increase in energy [20]. Overall, pure and doped LAP crystals have moderate hardness value.

IV. CONCLUSION

Pure and, urea and 1,3-Dimethyl urea doped LAP crystals are grown by solution growth method from aqueous solution at a constant temperature. Decrease in dielectric constant and dielectric loss is observed for doped crystals as compare to pure LAP. ac conductivity increases with increase in frequency. Temperature influence on conductivity for a lower frequency is observed for pure LAP crystal only. crystals cannot sustain the applied load for longer time period. So, 1,3-dimethyl urea doped LAP crystals may be the used for the NLO device applications at the place of pure LAP crystal.

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