ISSN: 2319-8753

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 3, Issue 2, February 2014

Molecular Spectroscopic Studies OF TGS, TGSP and TGSZC Crystals

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Abstract: TGS crystals are important ferroelectric materials. TGS plays a vital role in Infrared detector and FT-IR instrumentation and most widely used in Capacitors, RAM, Earth exploration monitoring, Pyroelectric vidicon tubes etc. In present work TGS, TGSP and TGSZC crystals are grown by slow evaporation method and then the grown crystals are subjected to UV, IR and Raman spectral studies. From UV – Vis spectral study optical transparency of the grown crystals is determined. From FT-IR and Raman spectral studies the presence of characteristic functional groups are confirmed. Powder XRD study is carried out to determine the crystal structure of grown crystals. Ferroelectric hysteresis study is carried out using homemade Sawyer-Tower circuit to investigate the ferroelectric behavior of grown crystals. Spontaneous polarization values were obtained. Electrical conductivity measurements are carried out for grown crystals. NLO property of grown crystals is tested using Kurtz-Perry powder technique.

Keywords: Growth from solution, ferroelectric materials, Characterization, Zwitter ion, glycinium ion.

I.INTRODUCTION

Triglycine sulfate is a good ferroelectric material for fabrication of Infrared detector. TGS has attractive pyroelectric coefficient and dielectric constant values at room temperature. It exhibits second order phase transition at the Curie point 49^0C. It provides large spontaneous electric polarization below its Curie point. It has basic pyroelectric figure of merit P/K = 1.1x 10^{-5} C/m^{2}K[1]. The crystal structure of TGS was reported by Hoshino et al [2]. The unit cell of TGS contains three types of glycines GI, GII, and GIII. Glycine I is in the form of Zwitter ion. GII and GIII are two planar glycines. They have protonated Carboxyl groups which have taken protons from the sulfuric acid. They form chain like system with SO_4^{2-} group. They have protonated Carboxyl groups which have taken protons from the sulfuric acid. They form chain like system with SO_4^{2-} group. The neighbouring chains are connected by SO_4^{2-} groups of adjacent chains and GI group. Such configuration of TGS is regarded as particularly important for the ferroelectric behavior of TGS crystal. The orientation of positively charged NH_3 groups determines the direction of spontaneous polarization in TGS. The spontaneous polarization reversal in TGS is due to the proton transfer between glycine and glycinium ions [3]. TGS exhibits order-disorder phase transition at the Curie point. Above Curie temperature it is in para electric phase with space group symmetry $P2_1/m$ and below Curie it is in ferroelectric phase with space group symmetry $P2_1$ with two formula units per unit cell[4]. The unit cell parameters of TGS are a = 9.15A˚, b = 12.6445A˚, c = 5.725A˚ and β = 105.53˚ [5]. TGS provides rich lattice vibration spectrum below 300 cm^{-1}[6]. The main disadvantage of TGS is its lower Curie temperature and easy depolarization by time, electrical, mechanical and thermal means. Because glycine has no asymmetric carbon and it is optically inactive. It is believed that doping of TGS with an optically active molecule will keep permanent polarization in TGS lattice.

ISSN: 2319-8753

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

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II RELATED WORKS

There are a large number of researches going on TGS to minimize the depolarization effect and to keep permanent polarization in TGS lattice. There is no change in crystal structure for Nd doped TGS and it is reported that Nd is coordinated with glycine[3]. But in the case of ADP and L-Asparagine doped samples change in crystal morphology is observed[7,8]. Tc value is higher for l-threonine, dl-threonine , l-methionine doped crystals and TGSP crystals[1,9]. It is observed that there is no low temperature phase transition in Lu-TGS[10]. No internal bias field is created for Pd doped TGS crystals and Ec is same as pure TGS crystals[11]. Spectral investigation of TGS doped with ADP, L-lysine, L-tryptophan, L-Cystine, Phosphoric acid reveals that doped samples have lower Spontaneous polarization values Ps and higher coercive field values compared to pure TGS. Higher Coercive field value implies that the crystal is in mono domain state [7,12-14,9]. Pyroelctric coefficient is increased for Thiourea, L-lysine , L-alanine and DL-alanine doped samples. [15-17]. Incorporation of Nitric acid and EDTA into TGS crystal increases the dielectric constant value.[18,19]. The substitution of amino acids l-threonine, dl-threonine , l-methionine and L-cystine results in the decrease of dielectric constant value compared to pure TGS crystal [1,14]. Zinc chloride and Urea doped samples exhibit Non-linear optical property and have S.H.G efficiency very much greater than standard KDP crystal.[20,21]. Incase of LGLM, La, Ce, Nd, L-lysine doped TGS crystal strong internal bias field is created indicates that the dopant is reduced the depolarizing effects in TGS crystals. The internal bias field fixes the polarization in a preferential direction with minimum possibility of polarization reversal [22,23,16]. Doping of TGS with metal ions Fe^{3+}, Cr^{3+} and Co^{2+} decreased the indirect band gap[24]. Investigation of TGS samples previously influenced by electric field E perpendicular to ferroelectric b axis reveals that there are rigid stripped domains parallel to c axis[25]. In present work pure and phosphoric acid, Zinc chloride admixtured TGS crystals are grown from solution.

III CRYSTAL GROWTH AND CHARACTERIZATION

Pure and Phosphoric acid, Zinc chloride admixtured TGS crystals were grown by solution growth method[5]. Grown crystals are shown in Fig. 1. UV-Vis absorption spectra of grown crystals are recorded in the wavelength range from 200-800nm using UV-Vis 2450 Make Shimadzu model spectrophotometer. FT-IR spectra are recorded in the frequency range from 400-4000cm^{-1} using IR Affinity Make Shimadzu model spectrophotometer. FT-Raman spectra were recorded in the frequency range from 0-3500cm^{-1} using the excitation radiation of 5145A˚ using Lab Ram HR 800 model spectrophotometer. Powder XRD pattern is obtained from BRUKER Diffractometer with CuKα radiation $\lambda = 1.054$ A^0. Ferroelectric hysteresis study is carried out using homemade Sawyer-Tower circuit [26]. Electrical conductivity of grown crystals is tested using IMPEDENCE ANALYZER IM3570. NLO Study is carried out using Q-switched Nd:YAG Laser.

a b c

Fig. 1 Grown crystals of a) TGS, b) TGSP and c) TGSZC

ISSN: 2319-8753

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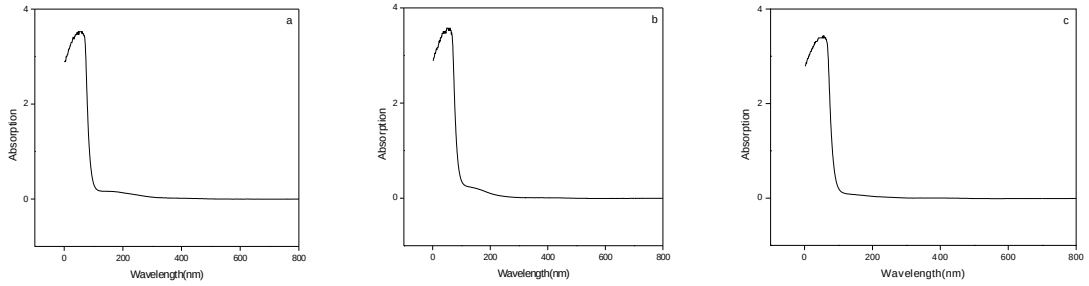
IV RESULTS AND DISCUSSION

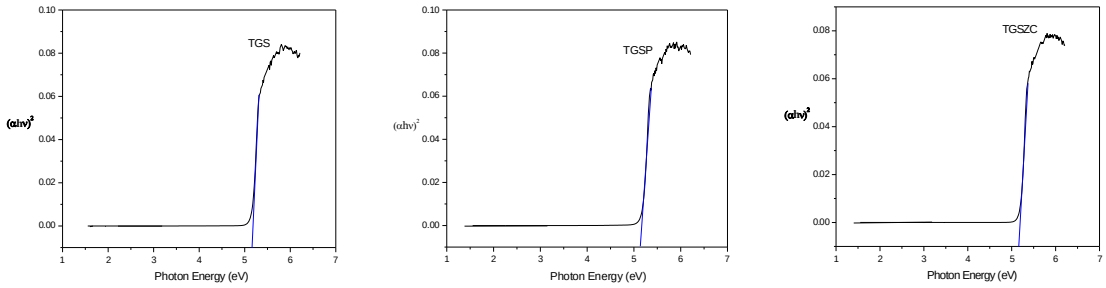
UV-Vis spectral investigation of grown crystals

Fig. 2 shows UV-Vis absorption spectra of TGS, TGSP and TGSZC crystals. For pure TGS wavelength of maximum absorption (λmax) occurred at around 233.5nm. For TGSP crystal λmax = 233nm. For TGSZC crystal, λmax = 232.5nm. Absorption at lower wavelength reveals that there must be higher energy transition corresponding to C = C – NO_2 group. It is observed that all these crystals have transmission percentage of above 90%. Energy band gap values were found out using the relation E = 1240/λmax eV [27]. Also Energy band gap values are calculated using Urbach Plot and results are reported in Table 1.



a b c

Fig. 2 UV-Vis absorption spectra of a)TGS b) TGSP c) TGSZC crystals.



a b c

Fig. 3 Urbach plot of a) TGS b) TGSP c) TGSZC crystals

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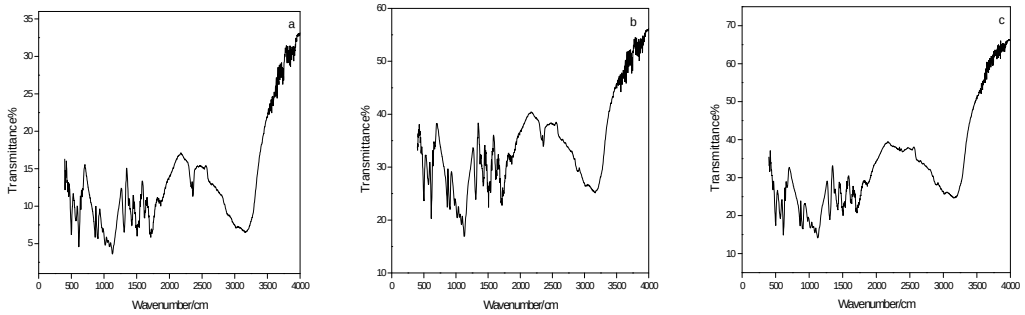
Table 1 Energy band gap values of grown crystals

Crystal	Eg (calculated from Eg = 1240/λ_{max} eV) eV	Eg (obtained from Urbach Plot) eV
TGS	5.31	5.16
TGSP	5.32	5.1496
TGSZC	5.33	5.1606

FT-IR spectral analysis

Fig. 4 shows FT-IR spectra TGS, TGSP, and TGSZC crystals. FT-IR spectrum of pure TGS crystal matches very well with the earlier reported values[18]. All expected characteristic vibrations are observed and assignments were tabulated in Table 2. IR band at 1425 cm^{-1} and 1624 cm^{-1} corresponding to symmetric and asymmetric stretching vibrations of COO^- indicate the Zwitter ion configuration of glycine [18]. IR bands observed in the region between 1716 cm^{-1} to 1869 cm^{-1} corresponding to stretching vibration of C = O indicate the presence of glycinium ion configuration [18]. Degenerate modes of NH_3 bending and C = O, NH_4 , C – H, O – H stretching vibrations are observed. FT-IR spectra of Phosphoric acid and Zinc chloride admixtured samples provide very similar features as that of pure TGS. More bands were located at same positions as that of pure TGS. There is a very slight shift observed in band positions compared to pure TGS. But doped samples provide less resolution of bands. Some bands are broadened and some are narrowed. Degeneracy is more for doped samples than that of pure TGS.



a **b** **c**

Fig. 4 FT-IR spectra of a)TGS b) TGSP c) TGSZC crystals

Table 2 FTIR ANALYSIS

TGS	TGSP	TGSZC	ASSIGNMENT
401w – 470w (7 lines)	401w - 472w (8 lines)	412w – 459w (5 lines)	$\nu(PO_4)$, $\nu(SO_4)$
497w	503w	501w	τ(C-N)

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572w	572w	570w	$\nu(SO_4^-)$
615m 646w	615m 646w	615m 648w	δ(C-N), NH_3^+ oscillation
669m	669m	667m, 675w	ν(S-H)
866m	866m	866m 898m	ν(C-C)
906w	908w	908w	ν(P-O-H), $\rho(NH_3)$
979w	979w	977w	ν(C-C-N),$\nu(SO_4^-)$
1016w	1018w	1016w	$\omega(CH_2)$, δ(C-C), ν(C-N)
1051w	1051w	1051w	ν(P-O-H)
1080w	1083w	1085w	ν(P-O-H), ν(C=O)
1128S	1111m 1124S	1128S	$\rho(CH_2)$, ν(C-C), $\nu(SO_4)$
1307m	1308m	1309m	$\delta(CH_2)$ of Glycine ,ν(C-C)
-	1338w	-	$\nu(NO_2)$
-	1363w	-	ν(C-N)
1375m 1377	1375m	1379m	$\delta(CH_2)$
	1386w	-	$V(NH_4)$
	1398w	-	ν(C=O), $\nu(NH_4)$
	1404w	-	$\delta(NH_4)$
	1409w	-	$V_{as}(COO^-)$, $\delta(NH_4)$
1425m	1417m 1425m	1429m	$\nu(COO^-)$, $\delta(CH_2)$

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	1435w		$\delta(NH_3)$
1456m 1473w	1448w-1498w (6 lines)	1456m – 1494w (5 lines)	v(NH4), v(COO-)
1506m – 1558w (3 lines)	1506m – 1558w (5 lines)	1506m – 1558w (6 lines)	$\delta(NH_3)$
1570m 1575w	1570m – 1595w (3 lines)	1568m 1573w	$v(COO^-)$
1618w 1624w	1616w 1622w	1616w - 1627w (3 lines)	$V_{as}(COO^-)$, $\delta(NH_3)$
1635w – 1653w (3 lines)	1635w - 1653w (3 lines)	1633 - 1651w (3 lines)	v(O=P-OH), v(C=C)
1668w	1662w 1670w	1660w - 1674w (3 lines)	$\delta(H_2O)$, v(C=C)
1716w - 1869w (4 lines)	1683w – 1869w (12 lines)	1683w – 1869w (16 lines)	v(C=O), $v(C=NH_4)$
2362w	1942w – 2360w (5 lines)	2355w	Overtone and combination bands.
3527w ,3545w	3502w – 3547w (5 lines)	3500w – 3558w (4 lines)	v(O-H), v(N-H)
3566w – 3689w (7 lines)	3566w – 3701w (11 lines)	3564w – 3697w (15 lines)	v(O-H), v(C-H), v(C=O), δ(P-O-H)
3709w – 3917w (15 lines)	3711w – 3957w (24 lines)	3701w – 3956w (31 lines)	v(O-H) of water

ISSN: 2319-8753

International Journal of Innovative Research in Science, Engineering and Technology

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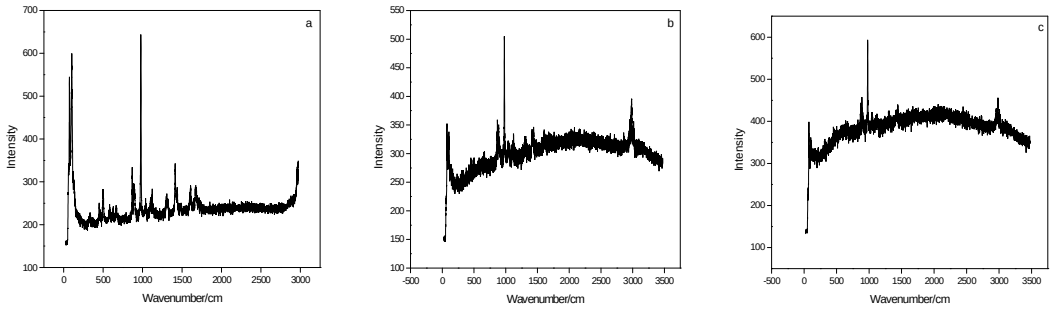
υ_s – Symmetric stretching, υ_{as} – asymmetric stretching, δ – bending, ρ – rocking, ω – wagging, τ – torsional vibrations.

FT-Raman spectral analysis

Fig. 5 shows FT-Raman spectra of pure and doped TGS crystals. FT-Raman spectrum of pure TGS matches very well with the earlier reported values [3,18]. Obtained Raman bands and their assignments are tabulated in Table 3. Raman band observed at 1413cm^{-1} and 1609 cm^{-1} corresponding to symmetric stretching of COO^- group confirms the Zwitter ion configuration of TGS crystal [3,18]. Band at 1681cm^{-1} corresponding to symmetric stretching of C = O group confirms the presence of glycinium ion configuration of TGS crystal [18]. In FT-Raman spectra of Phosphoric acid and Zinc chloride admixtured samples some peaks were shifted to a considerable range compared to pure TGS. There is a change in intensity of all peaks were observed. The amount of polarisability change will determine the Raman scattering intensity. So it can be concluded that change in intensity of peaks may be due to incorporation of dopants.



a b c

Fig. 5 FT-Raman spectra of a)TGS b) TGSP c) TGSZC crystals

Table 3 FT-RAMAN ANALYSIS

TGS	TGSP	TGSZC	ASSIGNMENT
73m 103m 336m	74m 103m	74m 110m	Lattice mode vibration of Glycine
450w	-	-	Lattice mode vibration of SO_4, δ (C-N) out of plane, $\nu(PO_4)$
503w	-	-	τ(C-N),$\nu(PO_4)$,

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582w			δ(C-CO), ν(SO_4^-)
630w	633w 634w	639w	ν(SO_4), δ(C-N) in plane, NH_3^+ oscillation
672w	-	-	ν(C-C)
868m	-	870m	ν(C-C)
890m	869m 889m	889m	ρ(CH_2), ρ(NH_3), ν(C-C), ν(P-O-H)
977s	978s	978s	ν(C-C-N),ν(SO_4^-)
1038w	1051w	1044w	ω(CH_2) , δ(C-C), ν(C-N), ν(C-N-H), ν(P-O-H), υ(SO_4)
1117w 1122m	1114w 1122w	1124w	ρ(CH_2), ν(C-C), ν(SO_4)
1307m	1304w 1314w	1312w	δ(C-H), ν(C-C)
1413m	1414w	-	ν_s(COO^-), δ(CH_2)
1438w 1492w	1439w	1441w	ν(NH_4), δ(CH_2)
1609m	1605w	-	ν (COO^-),ν (NH_4), ν (P-O-H),ν (C=C)
1681m	-	1678w	ν(C=O), ν(O-H-O)
-	-	2063w	Overtone and combination bands
2759m	2648m	2726m	δ(H_2O), ν(COOH), υ(P-

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International Journal of Innovative Research in Science, Engineering and Technology

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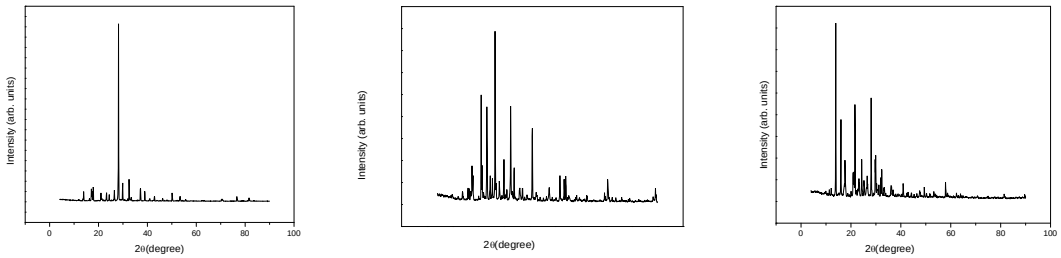
			O-H), $\upsilon(CH_2)$
3007w	2982m	2982s	ν (C-H), υ(O-H-O}
3014w 3057w	3014w 3057w	-	$\nu(NH_4)$

υ_s – Symmetric stretching, υ_{as} – asymmetric stretching, δ – bending, ρ – rocking, ω – wagging, τ – torsional vibrations.

Powder XRD study

Fig. 6 shows powder XRD pattern TGS, TGSP, TGSZC crystals. Sharp and well defined peaks are obtained for all crystals and matches very well with JCPDS card No. 14-0873. All crystals belong to Monoclinic Structure. XRD pattern of grown crystals differ from each other in intensity of reflection. Sharp and high intensity of reflection planes revealed that all crystals have good crystalline nature. Changes in Intensity of peaks for Phosphoric acid and Zinc chloride admixture samples compared to pure TGS revealed that there must be some changes in electron density in reflection planes due to the incorporation of dopants. Small change in 2θ value is observed for TGSP and TGSZC crystals. So pure and doped samples have different morphologies. Lattice parameter values are shown in Table 4. Obtained lattice parameter values for pure and doped crystals slightly differ from each other. Because of doping there may be some defects or strains in grown crystals.



a b c

Fig. 6 Powder XRD pattern of a) TGS b) TGSP c) TGSZC crystals

Table 4 Lattice parameter values of grown crystals

Crystal	a (nm)	b (nm)	c (nm)	β (deg)
TGS	9.4365	12.6584	5.7316	110.36
TGSP	9.4017	12.6449	5.7734	110.36
TGSZC	9.4396	12.7281	5.73999	110.36

ISSN: 2319-8753

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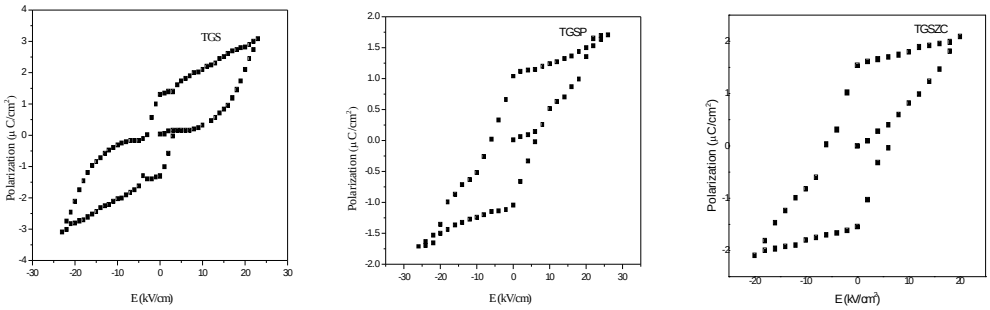
Ferroelectric hysteresis study

Homemade Sawyer- Tower circuit is constructed [26]. Sample capacitors were prepared by using aluminium foils and the pure and doped TGS samples as dielectric in between the aluminium foils [28].Spontaneous polarization values (Ps) for all samples were obtained by using the equation P = Q/A micro coulomb/cm^2 . Here Q = Charge measured on sample capacitor (coulomb). A = Area of capacitor plate (cm^2) [28]. Results are shown in Table 5. Obtained Ferroelectric hysteresis loops for grown crystals are shown in Fig.7.



a b c

Fig. 7 Ferroelectric hysteresis loops of a) TGS b) TGSP c)TGSZC crystals

Table 5 Ferroelectric hysteresis loop Analysis

Crystal	Spontaneous Polarization Ps (μC/cm^2)	Remnant Polarization Pr (μC/cm^2)	Coercive field value (kV/cm)
TGS	3.08	1.3	3
TGSP	1.712	1.04	6
TGSZC	2.1230	1.541	6

Electrical Measurement

Grown crystals are subjected to electrical characterization using IMPEDENCE ANALYSER IM3570. All crystals conduct electricity linearly. TGSZC crystal has higher electrical conductivity than pure TGS crystal. Results are shown in Table 6. Fig 8 shows electrical conductivity graphs of grown crystals. Fig 9 shows Impedance graphs of grown crystals. Electrical

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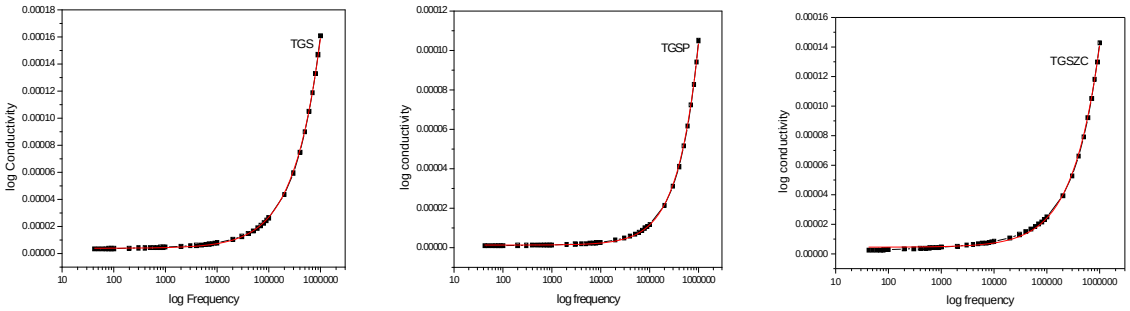
conductivity graphs of TGS, TGSP and TGSZC crystals contain two regions. Frequency independent Ac conductivity region in low frequency range and frequency dependent dc conductivity region in high frequency range. Conductivity graphs obey Arhenius relation and Jonscher's power law [29,30,31,32]. For all grown crystals dc conductivity linearly increases with increase in frequency. This indicates that electrical conductivity of these crystals is due to hopping mechanism. Electrical conductivity σ_{dc} is obtained by non linear fitting for the conductivity graphs. Then hopping frequency ω_p is obtained by using the relation $\omega_p = (\sigma_{dc}/A)^{1/n}$. Here n is frequency exponent. A is temperature dependent parameter. Chargre carrier concentration is obtained by $N = \sigma_{dc}T/\omega_p$. Mobility of charge carriers is obtained by $\mu = \sigma_{dc}/Ne$. Here e is charge of electron [31]. From electrical conductivity analysis TGSZC crystal has maximum dc electrical conductivity, Hopping frequency and Mobility of charge carrier values.



a b c

Fig. 8 Frequency versus conductivity graphs of a) TGS b) TGSP c) TGSZC crystals

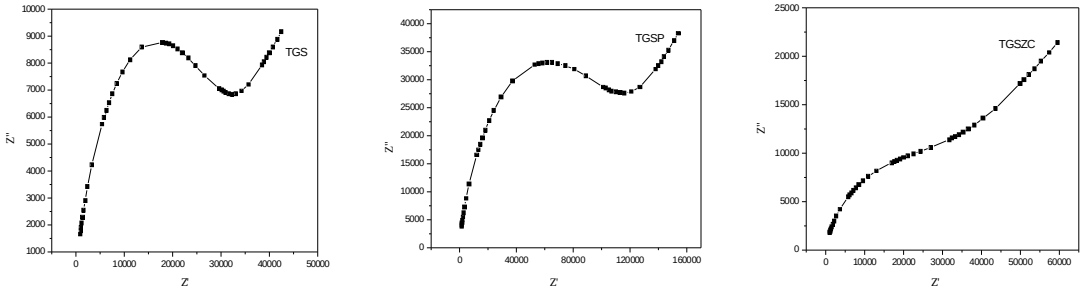


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Fig. 9 Impedance graphs of a) TGS b) TGSP c) TGSZC crystals

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Table 6 Electrical conductivity Analysis

Crystal	Electrical Conductivity Ec (Siemen/cm)	Hopping Frequency ω_p (Hz)	Charge Carrier Concentration N/cm^{-3}	Mobility μ (cm^2/Vs)
TGS	4.2032e-6	14036.19	0.9583e-8	27.414e+20
TGSP	1.3048e-6	12690.11	0.3290e-8	24.787e+20
TGSZC	4.5328e-6	17340.76	0.8365e-8	33.8673e+20

NLO Study

Non linear optical property of grown crystals is tested using Q-switched Nd:YAG laser (1064 nm, Quanta ray series). Input power is 0.68 J/s. NLO study confirms that all grown crystals exhibit second harmonic generation. Conversion efficiency of grown crystals compared to standard KDP crystal for second harmonic generation is listed in table 7.

Table 7 NLO Study

Grown Crystal	S.H.G Efficiency
TGS	0.818
TGSP	0.705
TGSZC	0.727

V CONCLUSION

From UV-Vis spectra of grown crystals it is confirmed that all these crystals have excellent optical quality.This property makes these crystals useful for applications in lasers, holographic recording, optical filters and Non-linear optical applications and electro- optic applications. From FT-IR and FT-Raman spectral investigations molecular structure of pure and Phosphoric acid, Zinc chloride admixtured TGS crystals are verified. Less resolution of peaks and change in intensity of peaks for doped samples compared to pure TGS are due to interaction between parent and dopants. It is concluded that Phosphoric acid and Zinc chloride were well incorporated into the lattice of TGS crystal. Ferroelectric hysteresis study reveals that for Phosphoric acid and Zinc chloride admixtured samples spontaneous polarization values were slightly decreased and coercive field values are increased compared to pure TGS. So doped crystals have improved Pyroectric behavior than pure TGS. So it can be concluded that Phosphoric acid and Zinc chloride admixtured TGS crystals are most suitable for Infrared detector applications. From Electrical conductivity graphs it is confirmed that grown crystals could be very useful for capacitor and other electrical applications. TGSZC crystals have higher electrical conductivity, hopping frequency and mobility of charge carrier values compared to pure TGS. From NLO Study it is confirmed that all crystals

ISSN: 2319-8753

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exhibit NLO property and pure TGS has maximum S.H.G efficiency compared to doped samples. So it is clear that all grown crystals are suitable for NLO and Opto electronic applications.

ACKNOWLEDGEMENT

Authors are thankful to the Management of Karpagam University for providing the fecilities for the work, and to the Head, Department of Nanoscience and Technology , Bharathiyar university, for recording the Raman spectra. Authors are thankful to Karunya University, Coimbatore for recording powder XRD pattern for grown crystals and to Materials Research Centre, Karunya University, Coimbatore for recording Electrical conductivity graphs for grown crystals. Authors are thankful to Department of Physics, B.S.Abdur Rahman University, Chennai for their help to carry out NLO study for all samples. Also thanks are due to the faculty and research scholars of the Department of Physics, Karpagam university, Coimbatore for their help during the course of the work.

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International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

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