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RESEARCH ARTICLE

INVESTIGATIONS ON THE PROPERTIES OF TRIGLYCINE SULPHATE SINGLE CRYSTAL ADMIXTURED WITH PERCHLORIC ACID

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ABSTRACT

Triglycine sulphate crystal is a well known ferroelectric crystal which has ferroelectric domains at room temperature. The drawback of pure TGS crystal is its tendency to polarization reversal. To overcome this difficulty and to improve the ferroelectric properties of the TGS crystals, it has been doped with amino acids, metal ions etc. In the present work, growth, structural, mechanical, FTIR and optical characterization of TGS crystal admixtured with Perchloric acid. Ferroelectric crystals of pure and Perchloric acid-admixtured Tri Glycine Sulphate (TGS) single crystals were grown by slow evaporation technique. The cell parameters of the grown crystals are evaluated from single crystal XRD and the results are compared. UV-VIS spectra studies explain the transmission ability of the grown crystals in the visible range. FTIR studies are performed to identify the presence of various functional groups present in the grown crystals. The nonlinear optical (NLO) property of the grown crystal was confirmed by Kurtz-Perry powder technique and a study of its second harmonic generation efficiency in comparison with potassium dihydrogen phosphate KDP has been made. The mechanical strength of the crystal was estimated by Vickers hardness test.

INTRODUCTION

Crystals of amino acids and their complexes can be considered for a variety of applications. Among amino acids, glycine (NH_2CH_2COOH) is the simplest amino acid and has normally three polymeric crystalline forms viz. α-glycine, β-glycine and γ-glycine (Sun *et al*., 1999; Selvarajan *et al*., 1992; Alexandru and Berbecaru, 2002; Balamurugan *et al*., 2007; Krajewski and Breczewski, 1980; Newman and Budzier, 1992). The least stable form β-glycine is related to the other two polymorphs. It is always obtained from a water-alcohol mixed solvent and can transform rapidly to the α-form in the presence of water or upon heating. The α-form is the metastable form at ambient temperature, which spontaneously crystallizes from water, or may be obtained by evaporation of aqueous solutions. Some of the glycine complexes in single crystalline forms have been grown and studied by many researchers (Moravec *et al*., 1977; Banan *et al*., 1989; Brezina and Havrankova, 1985; Moravec and Novontny, 1972; Ravi *et al*., 1994; Lucia Rose *et al*., 2011). When glycine combines with sulphuric acid in the molar ratio 3:1, Triglycine Sulphate (TGS) crystal is formed and it is a suitable material for developing detectors of infrared radiation and target faces in vidicons based on the pyroelectric effect. Doping crystals with various kinds of dopants influences the solubility, growth rate, morphology, structural, electrical and other properties of the crystals (Arunmozhi *et al*., 2000; Theresita Shanthi *et al*., 2009; Kurtz and Perry, 1968; Dhanaraj *et al*., 2008; Mary Linet *et al*., 2011; Hameed *et al*., 2000; Muralidharan *et al*., 2002). Studies on various physical and chemical properties of undoped and divalent impurity doped TGS crystals have been reported in the literature (Berbacaru *et al*., 2005; Mani *et al*., 1988; Toshio Kikuta *et al*., 2007; Balamurugan *et al*., 2007; Krishnan *et al*., 2009; Dhanaraj *et al*., 2008). But there are no research papers found in the literature dealing with the properties of TGS crystals admixtured with Perchloric acid ($HClO_4$). I report here for the first time the investigations on growth, structural, optical mechanical, and FTIR characterization of Tri glycine sulphate crystal admixtured with Perchloric acid.

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Experimental Procedure

Synthesis and solubility

Triglycine sulphate (TGS) salt and Perchloric acid ($HClO_4$) were taken in 0.7: 0.3 molar ratio and the calculated reactants were dissolved in deionized water, stirred well, heated at 50 ^oC to get 30 mole % of Perchloric acid admixtured TGS salt. This solution was heated and kept for slow evaporation to dryness at room temperature. The purity of the salt was improved by successive re crystallization process (Nazarath Begum *et al*., 2013). Figure 1 shows that the solubility of Perchloric acid admixtured TGS sample was estimated for seven different temperatures and the curve shows a positive solubility gradient in water. It is observed that the solubility of Perchloric acid admixtured TGS samples in water is more compared to that of pure TGS sample.

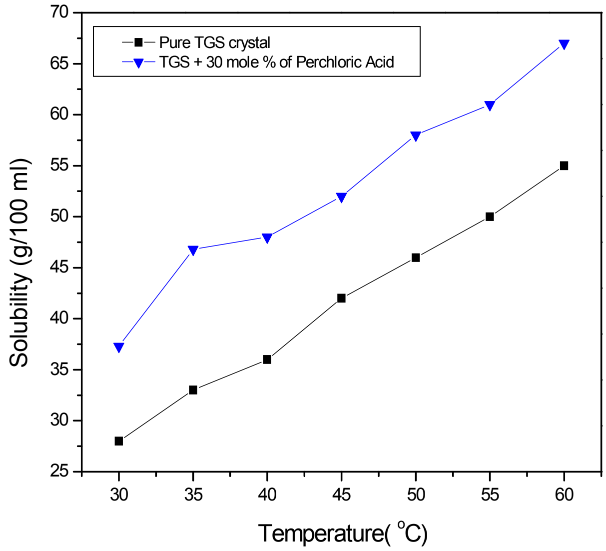

Figure 1. Solubility curves of pure and Perchloric acid admixture

TGS crystals

Growth of Perchloric acid admixtured TGS crystals

Single crystals of Perchloric acid admixtured TGS were grown by solution method using slow evaporation solution technique (SEST) at room temperature (31°C). Saturated solutions of the synthesized salts of Perchloric admixtured TGS salts were prepared separately based on the solubility data. The solutions were constantly stirred for about 3 hour using a magnetic stirrer and were filtered using high quality filter papers. Then the filtered solutions were taken in different beakers and covered with porous papers for controlled evaporation. Transparent, colour less single crystals were harvested within a period of 25-35 days. The harvested crystals are shown in Figure 2 and Figure 3. It is observed that the morphology of the crystals is altered when the concentration of Perchloric acid in the solutions is increased. This may be due to incorporation of Perchloric acid in the interstitial positions of the host TGS crystals.

Figure 2. Pure TGS crystal

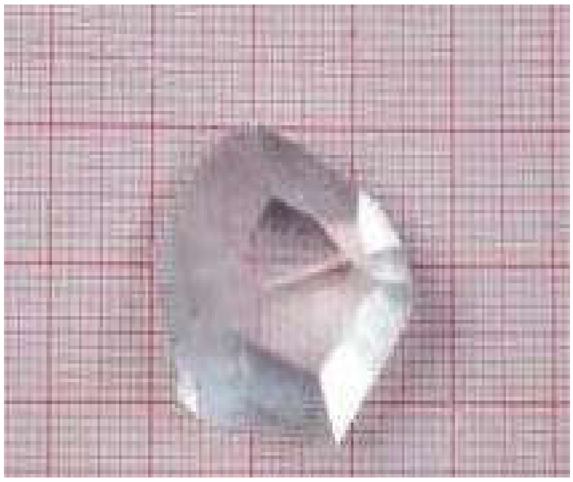

Figure 3. TGS crystal admixtured with Perchloric acid (30 mole %)

RESULTS AND DISCUSSION

Structural characterization

Structural characterization was carried out by XRD methods. Both single crystal and powder XRD methods are used to determine the unit cell parameters. An X-ray radiation of MoKα (λ= 0.71073 Å) with the help of a single crystal X-ray diffractometer was used to get the single crystal XRD data for the grown crystals of Perchloric acid admixtured TGS. It is observed that Perchloric acid admixtured TGS crystals crystallize in monoclinic system and the obtained data of unit cell parameters are listed in Table 1. The unit cell parameter values of Perchloric acid added TGS crystals are slightly different as compared to that of pure TGS crystal. The powder X-ray diffraction studies using PANalytical model nickel filtered CuK_{α} radiation (λ = 1.54056 Å) were performed to identify the structure and the diffraction planes. All the reflections of powder XRD patterns of the grown crystals were indexed using the INDEXING and TREOR software packages. The indexed powder X-ray diffraction patterns of the grown crystals are depicted in the Figure and Figure 5. From powder XRD patterns the numerous sharp peaks give a clear cut-proof of the crystalline nature the grown samples. Due to incorporation of Perchloric acid into TGS samples, some peaks are slightly shifted and a few diffraction peaks are more in the XRD patterns of Perchloric acid added TGS crystals compared to that of pure TGS crystal.

Table 1. Unit cell parameters of pure and Perchloric acid admixtured TGS crystals

S.No.	Sample	Cell parameters	Volume of Unit cell $(Å)^3$
1.	Pure TGS crystal	a = 9.392 (2) Å b = 12.655(3) Å c = 5.727(2) Å α = γ= 90° β = 110.39° (2)	638.63
2.	TGS crystal added with 30 mole % of Perchloric acid	a = 9.447(4) Å b = 12.745(3) Å c = 6.031(1) Å α = γ= 90° β = 106.30° (3)	694.87

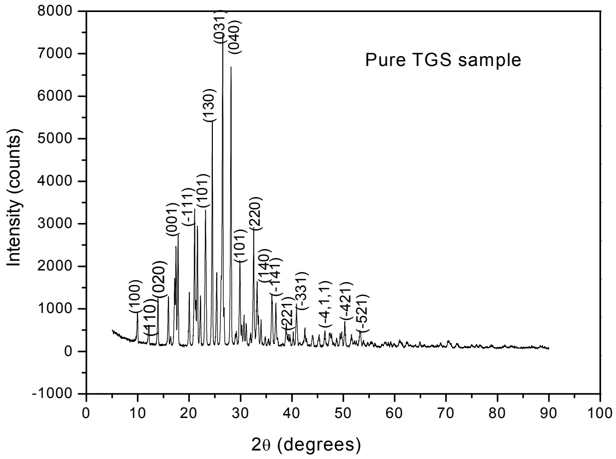

Figure 4. Powder XRD pattern of pure TGS crystal

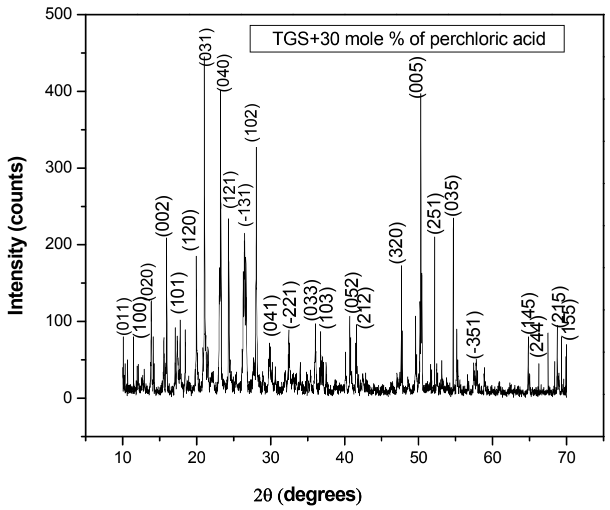

Figure 5. Powder XRD pattern of TGS crystal admixtured with Perchloric acid

Determination of hardness number, work hardening coefficient, yield Strength and stiffness constant

Vickers microhardness number (H_v) is calculated using the relation $H_v = 1.8544\ P / d^2$ kg/ mm^2 where P is the load in kilograms, d is the diagonal length of indentation impression in millimeters (mm). The values of 'd' are measured using a Vickers microhardness tester and the values of microhardness number is calculated (Nazarath Begum *et al.*, 2014). Figure 6 shows the variation of Vickers microhardness number (H_v) with applied load for pure and Perchloric acid admixtured TGS crystals. For pure and Perchloric acid added TGS samples, the hardness number increases with increase in load obeying the reverse indentation size effect.

The Mayer's relation $P = ad^n$ was used to determine the work hardening coefficient. The graphs Figure 7 and Figure 8 are drawn by taking log *d* versus log *P* and the values of slope of the straight lines are equal to the values of work hardening coefficient (n). The calculated values of work hardening coefficient are given in the Table 2. According to Onitsch's theory, if n is greater than 1.6, the materials are said to be soft materials (Onitsch, 1947). Hence the Perchloric acid admixtured TGS crystals belong to the category of soft materials.

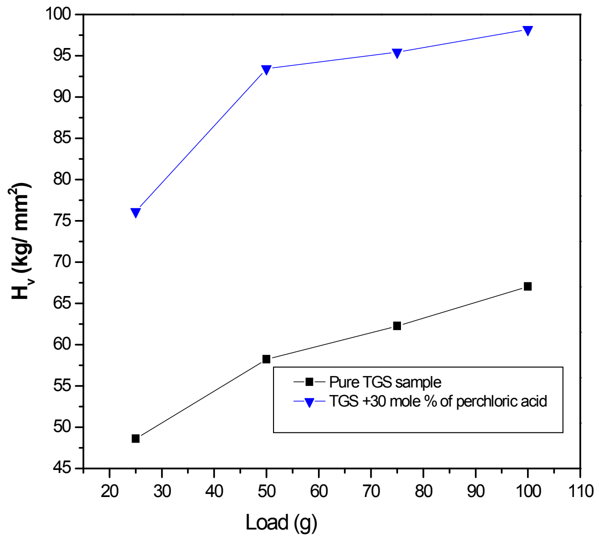

Figure 6. Dependence of hardness number (H_v) with loads in grams for pure TGS and TGS admixtured with Perchloric acid

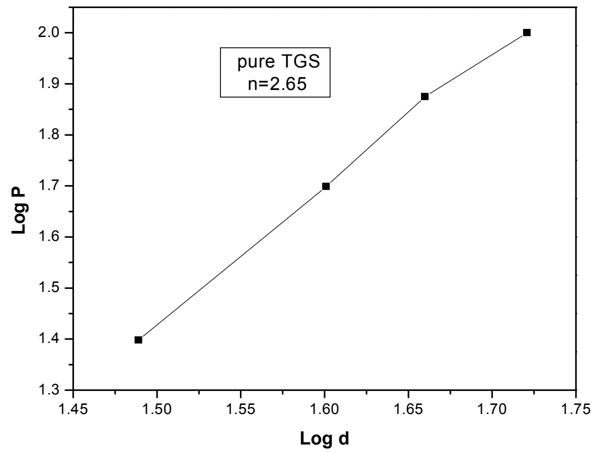

Figure 7. Plot of log P versus log d for pure TGS crystal

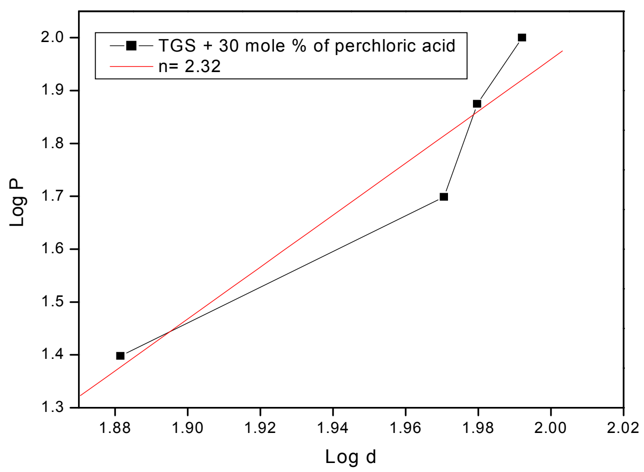

Figure 8. Plot of log P versus log d for TGS crystal admixture with Perchloric acid

Table 2. Work hardening coefficients (n) of pure TGS and Perchloric acid admixtured TGS crystals

Sample	Work hardening coefficient (n)	Materia l type
Pure TGS crystal	2.65	Soft
TGS +30 mole % of Perchloric acid	2.32	Soft

The elastic stiffness constant and yield strength for pure and Perchloric acid admixtured TGS crystals are calculated using the relations: Stiffness constant $C_{11}=(H_v)^{7/4}$ Pascal and yield strength $\sigma_y= H_v/3$ Pascal (Wooster, 1953). The calculated stiffness constant and yield strength for different loads for Perchloric acid admixtured TGS crystals are tabulated in Table 3 and Table 4.

Table 3. Yield strength and Stiffness constant for pure TGS crystal

Sample	Load (grams)	Yield strength (σ_y) x10^6 Pa	Stiffness constant x 10^{15} Pa
Pure TGS crystal	25	158.760	1.535
	50	190.218	2.106
	75	202.990	2.360
	100	218.964	2.695

Table 4. Yield strength and Stiffness constant for TGS crystals admixture With Perchloric acid

Sample	Load(g)	Yield Strength (σ_y) x10^6 Pa	Stiffness Constant x 10^{15} Pa
TGS + 30 mole % of Perchloric acid	25	248.65	3.367
	50	305.27	4.821
	75	311.80	5.063
	100	320.78	5.258

UV-visible spectral studies

The linear optical constants such linear absorption coefficient, extinction coefficient, refractive index and reflectance etc have been determined from UV-visible transmittance spectrum of pure TGS and Perchloric acid admixtured TGS crystals. The optical transmission spectrum of pure TGS and Perchloric acid admixtured TGS crystals were recorded using a UV-vis-NIR spectrophotometer (Lamda 35 model) in the range of 190-1100 nm. A good quality crystal of pure TGS and Perchloric acid admixtured TGS crystals with a thickness of 2 mm were used in this study. The obtained transmittance spectrum of pure and Perchloric acid admixtured TGS crystal is shown in Figure 9. This study helps us to find the suitability of materials in optical device applications. The optical property of the material gives information regarding the composition, nature and the quality of the crystal. From the results, it is noticed that the grown crystals have good transmittance in the visible region. It is observed that the percentage of transmission gets decreasing with concentration of dopants in the host TGS crystals. Due to the presence of impurities in the host crystals, the absorption in UV-visible region decreases. A strong absorption is observed at 236 nm for pure TGS crystal and the strong absorption is noticed at 241 nm for the percholoric acid added TGS crystals. The optical absorption coefficient of photon energy helps to study the band structure and explain the type of transition of electrons. The optical absorption coefficient (α) was calculated using the following formula (Jeyaprakash Manoharan *et al.*, 2011).

$$\alpha = \frac{2.303 \, log \, (\frac{1}{T})}{d}$$

where T is the transmittance and d is the thickness of the crystal in mm.

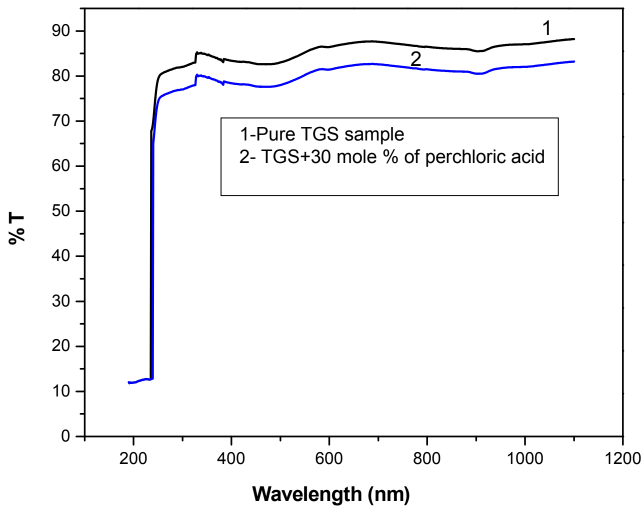

Figure 9. UV-visible transmittance spectra for pure and Perchloric acid admixtured TGS crystals

The extinction coefficient (K) and reflectance (R) are calculated using the following relations (Shanthi *et al.*, 2013; Anbazhagan *et al.*, 2013)

$$K= \frac{\lambda\alpha}{4\pi}$$

$$R = \frac{1 \pm \sqrt{1 - e^{(-\alpha d)} + e^{(\alpha d)}}}{1 + e^{(-\alpha d)}}$$

The relation between linear refractive index (n) and reflectance (R) is given by

$$n = \frac{-(R+1) \pm \sqrt{(-3R^2+10R-3)}}{2\,(1-R)}$$

The energy dependence of absorption coefficient suggests the occurrence of direct band gap of the crystal obeying the following relation

$$\propto h\nu = A\sqrt{(h\nu - E_g)}$$

where E_g is the optical band gap energy of the crystal, h is the Planck's constant, γ is the frequency and A is a constant. Tauc's plots are drawn to determine the optical band gap for the grown crystals. The plots of variation of $(\alpha h\upsilon)^2$ verses $h\upsilon$ are known as Tauc's plots and they are shown in the figure 10, and the band gap energy is calculated by extrapolation of linear part. The band gap values are tabulated in Table 5. Using the above equations the values of absorbtion coefficient, extinction coefficient, reflectance and refractive index for pure and Perchloric acid admixtured TGS crystal were determined. The variations of extinction coefficient, absorbtion, reflectance and refractive index with wavelength are shown in the Figures 11, 12, 13 and 14.

Table 5. Values of cut-off wavelength and band gap for Perchloric acid admixtured TGS crystals

Sample	Cut- off wavelength (nm)	Band gap (eV)
Pure TGS crystal	236	5.25
TGS+30 mole % of Perchloric acid	241	5.14

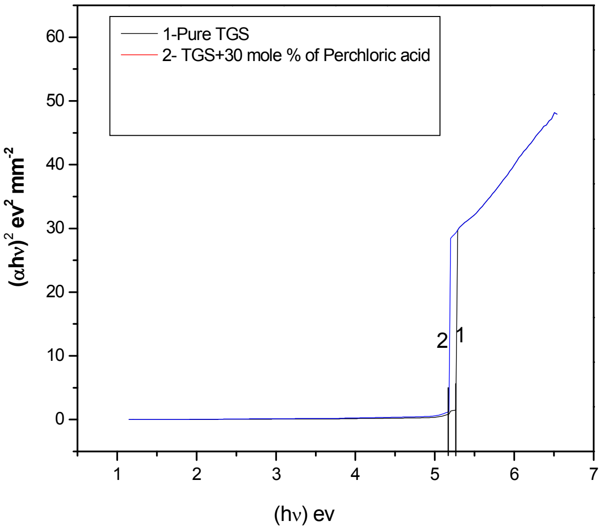

Figure 10. Plot of (αhv)^2 versus hυ for pure and 30 mole % of Perchloric acid admixtured TGS crystals

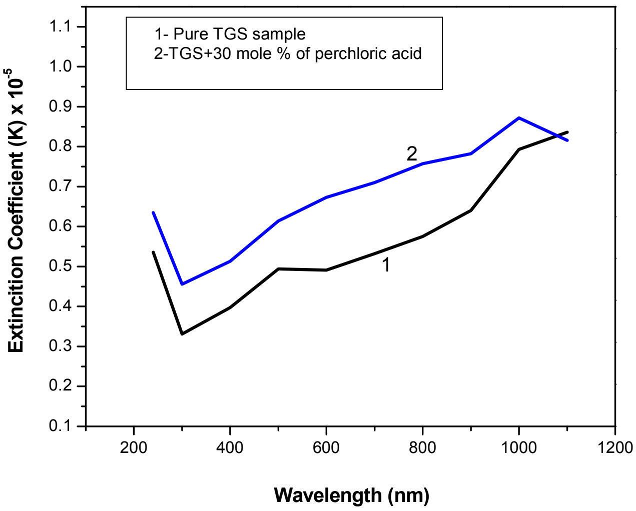

Figure 11. Variation of extinction coefficient with wavelength for pure and 30 mole % of Perchloric acid admixtured TGS crystals

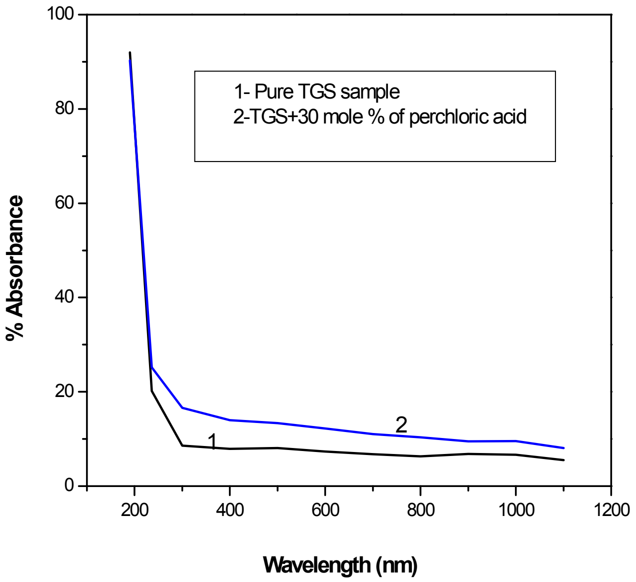

Figure 12. Plot of absorbance versus wavelength for pure and Perchloric acid admixtured TGS crystals

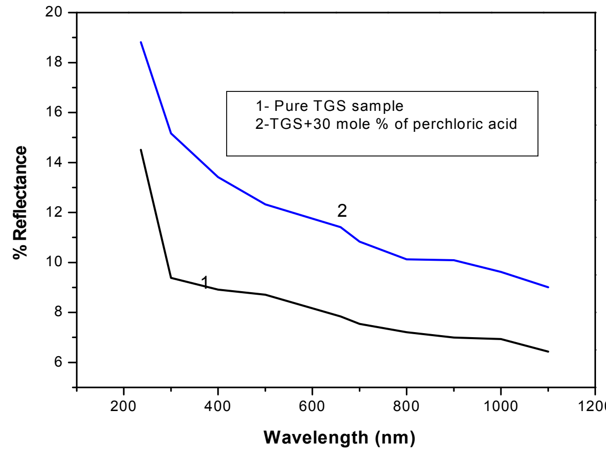

Figure 13. Plot of reflectance versus wavelength for pure and Perchloric acid admixtured TGS crystals

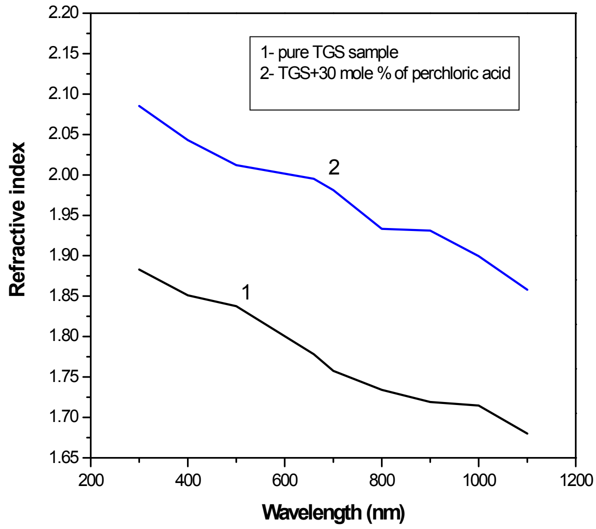

Figure 14. Variation of refractive index with wave length for pure and Perchloric acid admixtured TGS crystals

From results, compared to pure TGS crystal, due to impurity Perchloric acid admixtured TGS crystals have high reflectance, absorbance and extinction coefficient. The internal efficiency of the optical devices depends upon the absorption coefficient and reflectance. Hence by tailoring the absorption coefficient and tuning the band gap of the material, we can achieve the desired material which is suitable for fabricating various layers of the optoelectronic devices as per our requirements (Balasubramanian *et al*., 2010). From the Figure 14 the refractive index decreases with increasing wavelength. Due to presence of impurities, the admixtured TGS crystals have high refractive index than that of pure TGS crystal.

FTIR studies

FTIR absorption spectra of the grown pure and nitric acid admixtured TGS crystals in the infrared region 4500 cm^{-1} to 400 cm^{-1}were recorded on a spectrophotometer (Model: Perkin Elmer Spectrometer) using a KBr pellet technique. The recorded FTIR spectrum of pure TGS crystal is presented in the Figure 15. The present Vibrational spectroscopic study was carried out with a view of obtaining an insight into the structural aspects of glycine based crystals. In order to understand the existence of dopants and its bonding nature, the FTIR spectra of the admixtured TGS crystals are recorded and presented in the Figure 16. The spectra of the crystals are similar except small shifts in the peak positions and hence the crystals are expected to preserve nearly, the same interaction among the groups and ions. For the samples the broad band between 2200 and 3800 cm^{-1} in the spectra indicate stretching frequencies of superimposed O-H and NH_3^+ modes. Multiple combination and overtone bands of CH_2 have been observed in the region 2300-2500 cm^{-1}. The NH_2 asymmetric stretching vibrations appear between 3200 and 3500 cm^{-1} and NH_2 symmetric stretching vibrations occur between 2800-3200 cm^{-1}.The absorption in the region 1700-1650 cm^{-1} is assigned to C=O stretching of COOH group. The peaks between 1610 and 1450 cm^{-1} in the FTIR spectra can be assigned to COO^- vibrational mode. It is noticed here that some of NH_2 vibrations overlap with C-N and SO_4 vibrations. The strong peak region 1120-1150 cm^{-1} in the samples is attributed to C-N stretching vibrations. The peaks observed at 570 and 502 cm^{-1} are due to NH_3^+ oscillation. The observed vibrational wave numbers and their assignments for pure TGS and Perchloric acid admixtured TGS crystals are tabulated in Tables 6 -7. The assignments for the absorption bands of the FTIR spectra of the samples are provided as per the data reported in the literature (Meera *et al.*, 2005; Balasubramanian and Selvarajan, 2010). Comparing the absorption bands / peaks, it can be seen that FTIR spectra of pure and Perchloric acid admixtured TGS crystals are identical with some changes. For admixtured crystal the broad band covering 3886- 3100 cm^{-1} indicates the asymmetric stretching of NH_3^+ modes. The peak 1650- 1595 cm^{-1} corresponds NH_3^+ asymmetric bending. The strong peak region 650 cm^{-1} – 710 cm^{-1} can be assigned to C-Cl stretching vibration .The peak around 550 cm-1 – 620 cm-1 belongs to SO_4^{2-} scissor bending.

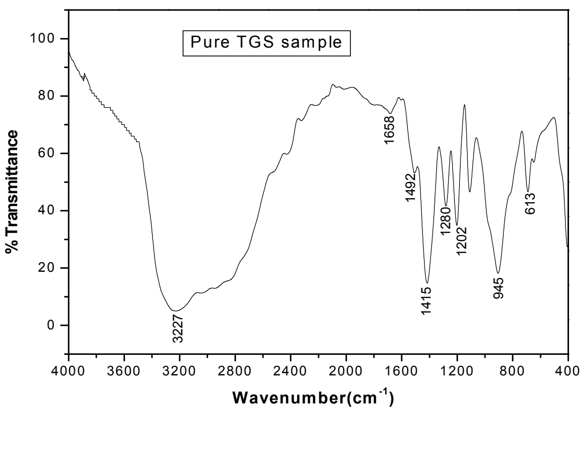

Figure 15. FTIR spectrum for pure TGS crystal

Table 6. FTIR spectral assignments for pure TGS crystals

Bands/Peaks (cm^{-1})	Assignments
3227	NH_3^+ asymmetric and OH stretching
1658	NH_3^+ asymmetric bending
1492	COO- symmetric stretching
1415	C-N stretching
1280	C-O stretching
1202	OH-bending
945	C-C stretching
613	SO_4 Scissor bending

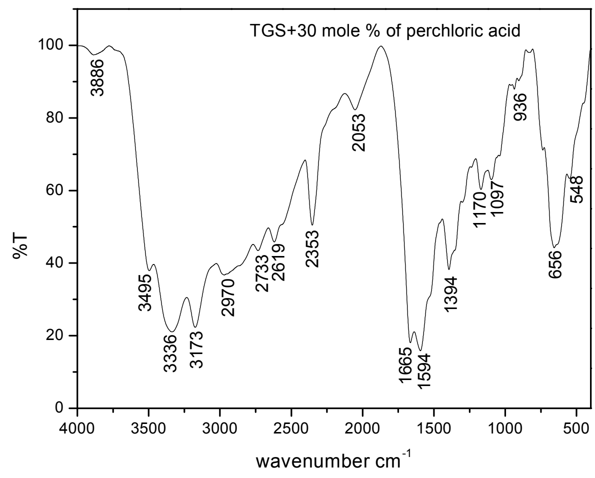

Figure 16. FTIR spectrum of TGS crystals admixtured with 30 mole % of Perchloric acid

Table 7. FTIR spectral assignments for TGS crystals admixtured with 30 mole % of Perchloric acid

Bands/Peaks (cm^{-1})	Assignments
3886	N-H stretching
3495	NH_3^+ asymmetric and OH stretching
3336	NH_3^+ asymmetric stretching, OH stretch of water
3173	NH_3^+ asymmetric stretching
2970	CH_2 stretching
2733	C-H stretching mode
2619	Combination band
2353	Stretching of CH_3 vibration
2053	Combination band of NH_3^+ degenerate mode and NH_3^+ torsion
1665	Amide
1594	NH_3^+ asymmetric bending
1394	C–C stretching
1170	OH bending
1097	NH_3^+ rocking
936	C-C-N stretching
656	C-Cl stretching vibration
548	SO_4 Scissor bending

Measurement of Second Harmonic Generation (SHG) efficiency

A high intensity was used to comport Kurtz-Perry test. The SHG was confirmed by the emission of green radiation (532 nm) which was detected and the values of relative SHG efficiency 30 mole % of Perchloric acid admixtured TGS crystal are found to be 0.85. Hence the Perchloric acid TGS crystals could be used as the second harmonic generators. Here it is to be mentioned that pure TGS crystal has no SHG efficiency. From the results, it is found that SHG efficiency increases with increase in concentration Perchloric acid in the host TGS crystals

Conclusion

Good quality single crystals of pure and Perchloric acid admixtured TGS were developed by slow evaporation technique. Positive temperature coefficient of solubility was found for the samples. Its cell parameters have been determined by the single crystal XRD and powder XRD analysis. The UV-vis-NIR transmittance spectrum shows a good optical transmittance. The mechanical property of the grown crystals shows that the crystal belongs to the soft material category. The mechanical strength of the grown crystals have been analyzed and yield strength, stiffness constant ,work hardening coefficient of the samples has been obtained. The Functional groups of the sample were obtained by the FTIR spectrum. SHG studies reveal that Perchloric acid admixtured TGS material is a promising material for NLO applications. The above results concluded that the grown crystals are good candidate for optoelectronic applications. Enhancement in the above properties of TGS sample shows that the Perchloric acid plays a vital role in the lattice of pure TGS sample to improve foe NLO applications.

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