

Chitosan, a semi-synthetic polymer driven electrical and third-order nonlinear optical enhancement of SrTiO₃ perovskite

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Abstract

The chemical precipitation method was employed to synthesize SrTiO₃ (ST) and chitosan-loaded SrTiO₃ (CST) NPs. Cubic crystal structures with substantial (1 1 0) preferential development are seen in both ST and CST. The band gaps calculated were 3.19 and 3.07 eV, respectively, for ST and CST. The sheet resistance (R_{sh}), resistivity (ρ) values of ST and CST were $8.6 \times 10^4 \Omega/\text{cm}$, $3.56 \times 10^1 \Omega/\text{cm}$ and, $2.68 \times 10^2 \Omega/\text{cm}$, $1.95 \times 10^{-1} \Omega/\text{cm}$, respectively. From the Z-scan studies, the negative nonlinear refractive index that both samples display on closed aperture curves indicates that they are self-defocusing, with two-photon absorption, ST and CST's open aperture curves are reverse saturable. Chitosan loading enhanced SrTiO₃'s nonlinear refractive index from 4.85 to $5.88 \times 10^{-8} \text{ cm}^2/\text{W}$, absorption coefficient from 2.02 to $2.97 \times 10^{-6} \text{ cm/W}$, respectively. A high third-order nonlinear susceptibility ($3.99 \times 10^{-6} \text{ esu}$) realized for CST confirmed its utility in optical switching devices.

Keywords: Perovskite; chitosan; NLO properties; Z-scan; susceptibility

1. Introduction

Perovskite alkaline earth titanates (TiO₃, A=Ca, Ba, Mg, Sr, Zn, etc.,) with outstanding optical, electrical and mechanical properties act as building block materials for flexible electronic and photonics nowadays [1]. Perovskites are considered to contain more oxygen-containing membranes, due to the inherent defects of oxygen in their structure [2]. Among the perovskites, strontium titanate (SrTiO₃) has garnered attention as a practical electronic material due to its unique physical and chemical properties [3], which arises with the nature of the chemical bond between Sr²⁺, Ti⁴⁺ and O²⁻ ions. Due to

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the nature of bonding involved in SrTiO_3 , it is widely used in gas sensors [4], solar cells [5], UV detectors [6], capacitors [7], etc.

For high-speed optical devices, high nonlinear refractive index and absorption coefficient values are the prime requirements. SrTiO_3 , an n-type perovskite, exhibits suitable energy band levels, favourable crystalline structure, tunable morphologies and high stability, which make it promising for NLO devices. However, with a larger band gap (3.2 eV), SrTiO_3 responds only to UV light, restricting the efficiency of devices where it is used. To overcome this demerit, SrTiO_3 may be loaded with noble metals [8], doped with metal ions [9, 10], coupled with narrow band gap semiconductors [11], modified by ionic liquids [12], etc. Aside from these, composite creation using polymers with diverse optical properties may improve the optical response of SrTiO_3 . Unlike traditional polymers from limited fossil resources, biopolymers from renewable materials show improved material properties, enables straight forward and biomimetic reaction pathways, and possess natural biocompatibility [1].

In this work, chitosan, a semi-synthetic polymer derived from the deacetylation of chitin, is coupled with SrTiO_3 to improve its optical response as it exhibits hydrophilicity, light-absorbing behaviour, physiological compatibility and non-immunogenic nature [13]. Chitosan also contributes to the stabilization and surface functionalization of SrTiO_3 , improving its optoelectronic and nonlinear optical properties [14]. Chemical precipitation was used to synthesize SrTiO_3 and chitosan-loaded SrTiO_3 NPs, and their NLO characteristics were compared. In the literature, there exist no reports on the NLO properties for polymer-incorporated SrTiO_3 nanocomposites. Table 1 highlights the properties of pure and doped SrTiO_3 perovskite reported earlier. Researchers working on SrTiO_3 will find this work significant because studies on the third-order NLO characteristics of SrTiO_3 are sparse.

Table 1: Literature results on the electrical and third-order nonlinear optical properties of pure and doped SrTiO_3

Sample	Form	Synthesis procedure	Results reported		Reference
			Electrical resistivity/ conductivity	Nonlinear optical	
SrTiO_3	Thin film	Sol-gel spin coating	$3.03 \times 10^{-4} \Omega/\text{m}$	---	[15]
SrTiO_3	Powder	Solid-state reaction	$7.8 \times 10^{-6} \text{ S/cm}$	---	[16]
Nb-doped SrTiO_3	Thin film	Pulsed laser deposition	$3.84 \times 10^4 \text{ S/cm}$	---	[17]
SrTiO_3	Thin film	Pulsed laser deposition	---	$5.7 \times 10^{-10} \text{ cm}^2/\text{W}$	[18]
SrTiO_3	Powder	Solid-state reaction	$7.8 \times 10^{-6} \text{ S/cm}$	---	[19]

2. Experimental

SrTiO₃ (ST) and chitosan-loaded SrTiO₃ (CST) NPs are synthesized using strontium chloride, titanium dioxide, chitosan, HNO₃, liquid NH₃ and de-ionized water.

2.1. Procedure to synthesize STNPs and CST NPs

Strontium chloride (0.1 M), titanium dioxide (0.2 M), and nitric acid (13 mL) were liquefied with 117 mL de-ionized water. With the addition of liquid NH₃ (20mL), the above solution's pH was neutralized to 10 and matured for 4h. The precipitates were washed, calcined at 300°C for 2 h and crushed to yield ST NPs. CST NPs were synthesized by using the same precursor solution used to synthesize ST NPs with the addition of 2% of chitosan of the weight of SrCl₂ (optimized value).

2.2. Characterization

Structural, morphological, optical, and luminescent properties were investigated using a diffractometer (X'Pert PRO Analytical), a microscope (HITACH S-3000H), a spectrophotometer (LAMBDA-25), a Varian Cary Eclipse Fluorescence Spectrometer, and a two-probe setup, respectively.

2.3. Z-scan studies

A 532 nm diode-pumped CW Nd:YAG laser (Coherent Compass TM215M-50) is used to perform NLO studies of ST and CST NPs, which are dispersed in ethylene glycol. Table 2 outlines all Z-scan investigations' experimental parameters.

Table 2: Experimental parameters involved in the Z-scan studies

Laser wavelength	532 nm
Laser power	100 mW
Laser power intensity	8.49 kW/cm ²
Optical path length	724 nm
Radius of aperture	1.25 mm
Beam radius falling on the lens	3.1 mm
Sample thickness	1 mm
Focal length of lens	103 mm

3. Results and discussion

3.1. XRD

The diffraction peaks of ST and CST (Figure 1) reveal the cubic phase of SrTiO₃ with the *Pm3m* space group (JCPDS No. 36-734). Except for a minor change in SrTiO₃'s peak positions, CST showed no impurity peaks related to chitosan. The high strength of the peaks correlated with SrTiO₃ can explain the absence of chitosan's peak.

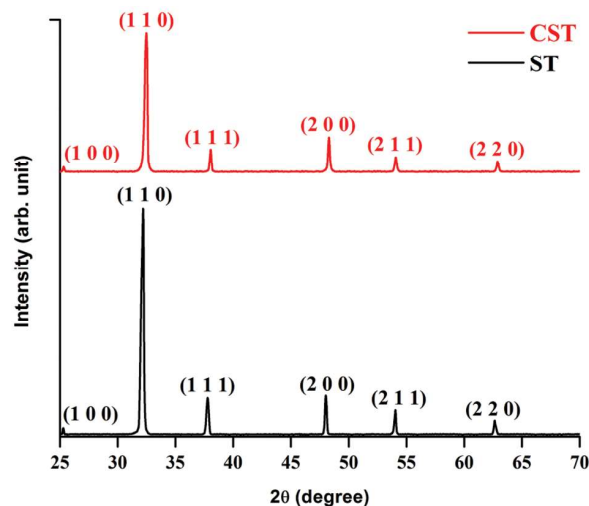


Figure 1: ST and CST's XRD patterns

Literature results showed that chitosan exhibits broad diffraction peak around 20.25° , by the amino acids and hydroxyl groups which form hydrogen bonds [20]. The minimal amount of chitosan added to the precursor solution for composite synthesis resulted in its peak's absence. Absence of chitosan's natural crystal structure in CST results from hydrogen bond (intermolecular) formation between SrTiO_3 and chitosan [21]. The crystallite sizes (D) of ST and CST calculated using the Scherrer equation, were 51 and 36 nm, respectively.

3.2 SEM & EDX

Figure 2, SEM images of ST and CST reveal nanosized granules. Grains with well-defined boundaries are seen in CST with chitosan coupling. ST's grain size decreased with chitosan loading. A few nanoneedles are also seen for CST. EDX spectra (Figure 3) confirmed Sr, Ti and O's existence. C peak's presence confirms the blending of chitosan on pure SrTiO_3 .

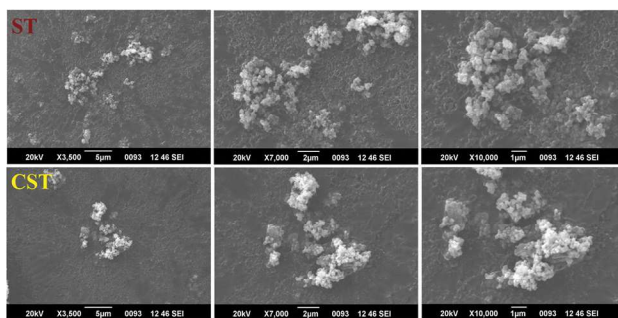


Figure 2: ST and CST's SEM images

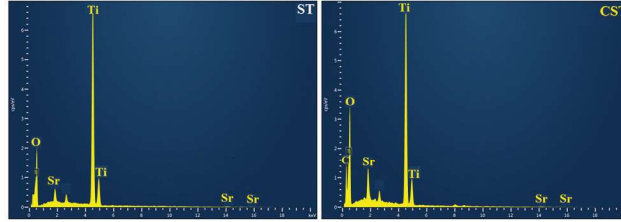


Figure 3: ST and CST's EDX spectra

3.3 Optical Analysis- Absorption study

Absorption edges occur at 389 and 404 nm for ST and CST (Figure 4 (a)). Red shift in SrTiO_3 's absorption edge resulted from the interfacial formation between chitosan and SrTiO_3 [22]. Due to the ST absorption edge's red shift with chitosan loading, CST exhibited decreased band gap (E_g) value, calculated from the Tauc plots (Figure 4(b)) for direct allowed transition, which abides with the formula: $(\alpha h\nu)^2 = A(h\nu - E_g)$, where α is the absorption coefficient. The E_g values calculated were 3.19 and 3.07 eV, respectively, for ST and CST. The reduced E_g value for CST is due to the inducement of more energy levels which shifted its absorption edge [23].

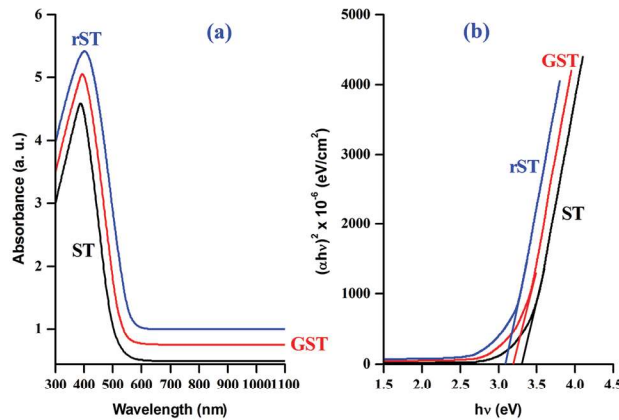


Figure 4: ST and CST's, a) absorption spectra and b) Tauc plots

3.4 Photoluminescence study

The PL peaks (Figure 5) centered at 363 and 377 nm are violet colored, the peaks at 409, 438 and 493 nm are blue coloured and the peaks at 506, 522 and 544 nm are green colored. High-level excitonic emissions connected to quantum confinement account for the peaks at 363 and 377nm [24]. The crystal structure's Sr deficit is the primary cause for the blue emission peaks [25]. The deep-level emission band at 493 nm arises from point defects in SrTiO_3 , such as Sr vacancies/O interstitials [26]. The concentration of

oxygen vacancies and free electrons on SrTiO₃'s surface aid in the formation of recombination centers resulting to the green emission peaks [27]. A high recombination rate of photo-excited carriers correlates with strong PL intensity; whereas low recombination rate correlates with weak intensity [28].

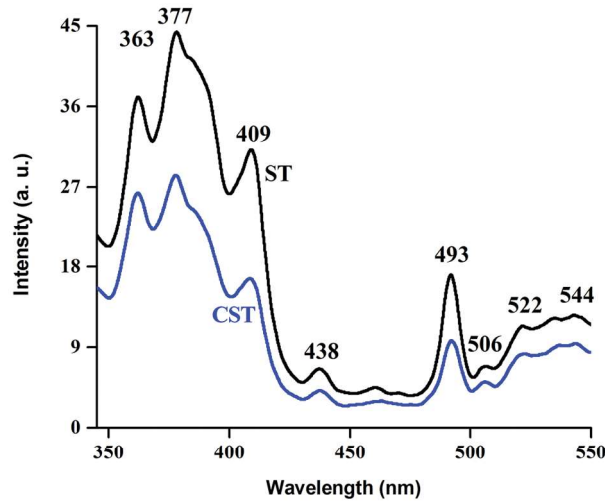


Figure 5: ST and CST's PL spectra

Hence, for ST with strong PL intensity, the recombination rate of photo-excited carriers is high compared to CST. The least PL intensity observed for CST indicates that it can effectively suppress the recombination process as electrons are transferred from the chitosan polymer to SrTiO₃.

3.5 Electrical and optoelectronic properties

The sheet resistance (R_{sh}), resistivity (ρ) values of ST and CST NPs calculated by the two-probe method were $8.6 \times 10^4 \Omega/\text{cm}$, $3.56 \times 10^1 \Omega/\text{cm}$ and, $2.68 \times 10^2 \Omega/\text{cm}$, $1.95 \times 10^{-1} \Omega/\text{cm}$, respectively. CST's reduced sheet resistance and resistivity values might be due to point defects such as oxygen vacancies created in CST's matrix through chitosan loading [29, 30]. Oxygen vacancies are known to rapidly increase ST's conductivity as a donor state is formed close to its conduction band minimum [31].

The figure of merit (ϕ), which helps to determine the optoelectronic quality of ST and CST NPs, is determined by the relation:

$$\phi = \frac{T_{avg}^{10}}{R_{sh}} \quad (1)$$

Where T_{avg} is the average transmittance, which was 65 and 78 % for ST and CST, respectively. The calculated ϕ values are $1.565 \times 10^{-6} \Omega^{-1}$ and $3.11 \times 10^{-4} \Omega^{-1}$ for ST and CST, respectively. Increased CST's ϕ value resulted from increased transparency, increased grain growth, which led to less scattering effects from grain boundaries [32]. With an increased figure of merit value, CST is well-suited for optoelectronic devices.

3.6 Z-scan studies

Z-scan studies confirmed that pure SrTiO_3 (ST) exhibits feeble nonlinear optical behaviour; whereas with chitosan loading, appreciable nonlinear optical characteristics were realized. With chitosan loading, free carrier absorption, two-photon absorption and free carrier refraction take place, improving CST's improved nonlinear optical behaviour. The development of a space-charge zone with a large electron base with chitosan loading increases the chance of photon transition, enhancing CST's nonlinear optical effect [33]. Figure 6 shows ST and CST's closed and open curves; whereas the closed-to-open aperture ratio curves are shown in Figure 7. The peak valley arrangement observed for both the samples indicates negative nonlinearity with a self-defocusing nature, which is a result of temperature-induced absorption of single photons or free carriers [34]. The nonlinear refractive index (n_2) values of ST and CST were 4.85 and $5.88 \times 10^{-8} \text{ cm}^2/\text{W}$, respectively. High n_2 value realized for CST is due to the introduction of more active N-sites by chitosan on SrTiO_3 's matrix which improves its stability, as chitosan is an N-containing biomass. Also, the amino and hydroxyl groups present in chitosan contain lone pair electrons, which chelate with Sr^{2+} and Ti^{4+} ions through coordination to form stable chelates, thereby increasing the absorption abilities of CST.

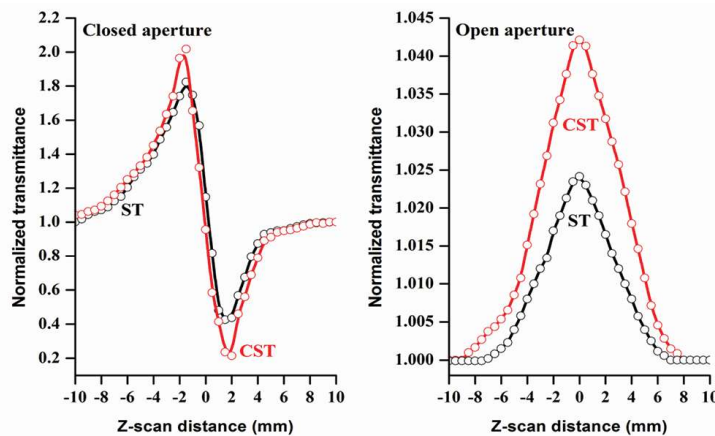


Figure 6: ST and CST's closed and open aperture curves

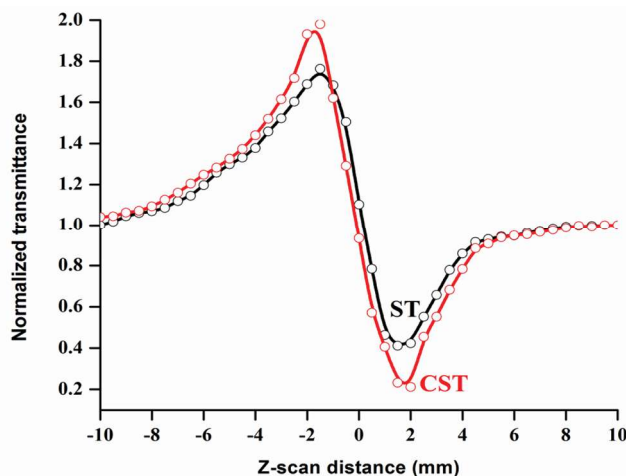


Figure 7: ST and CST's closed-to-open ratio curves

From the open aperture curves, both ST and CST show reverse saturable behaviour with two-photon absorption [35]. The transmittance dip is found to be deeper for CST, indicating its stronger nonlinear absorption performance. The absorption coefficient (β), susceptibility (χ^3) values of ST and CST calculated from Figure 7 were 2.02×10^{-6} cm/W, 2.14×10^{-6} esu and 2.97×10^{-6} cm/W, 3.99×10^{-6} esu, respectively. Increased β value observed for CST might be due to the following possible reasons: i) oxygen deficiency existing on SrTiO_3 's surface with chitosan loading, ii) change in energy levels and shifting in the optical band gap, iii) decreased crystallite size as NLO properties are size dependent and iv) outside electron donor and acceptor, which originates when SrTiO_3 is loaded with chitosan. Higher susceptibility value realized for CST might be due to increased polarization effect and non-homogeneous lattice strain induced into SrTiO_3 's matrix with chitosan loading. Thus, due to improved NLO qualities, CST is well suited for optical switching devices. The results obtained in this work for chitosan, a cationic polymer loaded SrTiO_3 , with the other polymer-based SrTiO_3 nanocomposites have been compiled in Table 3.

Table 3: Obtained results compared with other polymer-based SrTiO_3 nanocomposites

Composite	Form	Synthesis procedure	Results obtained			Reference
			Optical band gap (eV)	Nonlinear refractive index, n_2	Nonlinear susceptibility (χ^3)	
PVC/PVP/ SrTiO_3	Film	Thermal decomposition	5.96	580×10^{-12} esu	491.75×10^{-13} esu	[36]

PVA/ SrTiO ₃	Film	Solution casting	4.5	---	---	[37]
Polyaniline /SrTiO ₃	Powder	In-situ oxidative polymerization	3.2	---	---	[38]
PVDF/ SrTiO ₃	Film	Solution casting	5.53	5.28×10^{-11} esu	5.98×10^{-12} esu	[39]
Chitosan/ SrTiO ₃	Powder	Chemical precipitation	3.07	5.88×10^{-8} cm ² /W	3.99×10^{-6} esu	Present work

4. Conclusion

Pure SrTiO₃ and chitosan-loaded SrTiO₃ NPs were successfully synthesized by the chemical precipitation method. With chitosan loading, significant enhancement has been observed in the third-order nonlinear optical properties of SrTiO₃ due to decreased crystallite size and alteration in the Fermi energy levels and band gap. Compared to pure SrTiO₃, chitosan-loaded SrTiO₃ exhibited increased figure of merit ($3.11 \times 10^{-4} \Omega^{-1}$), nonlinear absorption coefficient ($2.97 \times 10^{-6} \text{ cm/W}$) and susceptibility ($3.99 \times 10^{-6} \text{ esu}$) values, which confirmed its utility in optoelectronic and optical switching devices.

Acknowledgements

For NLO studies, we thank Dr. G. Vinitha of VIT, Chennai.

Statements and Declarations

Funding

The authors declare that no funds, grants, or other support were received during the preparation of this manuscript.

Competing Interests

The authors have no relevant financial or non-financial interests to disclose.

Authors' contributions

All authors contributed to the study conception and design. Conceptualization: R. Sridevi; Methodology: M. Suganya; Formal analysis and investigation: A. Vinith; Writing - original draft preparation: A.R. Balu; Writing - Review and editing: A.R. Balu; Funding acquisition: V. Rajamani; Data curation: N. Arunkumar; Interpretation of data and software resources: M. Sriramraj.

All the authors read and approved the final manuscript.

Availability of Data and Materials

Data cannot be shared openly, but is available on request from authors.

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