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A Study of the Optical Properties and Energy Gap Tuning Through Nickel Doping in Z-Scan and Diffuse Reflection Techniques for $La_{0.5}Sr_{1.5}Co_{2-x}Ni_xO_2$ Composite Particles

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ABSTRACT

Objective: *The primary focus of the study was to examine its linear and non-linear optical properties.* ***Method:*** *This research investigates the synthesis of the nanocomposite* $La_{0.5}Sr_{1.5}Co_{2-x}Ni_xO_2$*, which was prepared using the sol-gel method. Furthermore, the non-linear optical properties were studied using the Z-scan technique with both closed and open apertures in a Nd:YAG laser at a wavelength of 532 nm.* ***Results:*** *The results of the measurements revealed a gradual decrease in the optical bandgap, which ranges between 2.12–2.25 eV. This decrease was observed as the nickel doping fraction increased and may be attributed to orbital interaction between the oxygen, nickel and cobalt electrons, with the observation of localised states within the forbidden gap alongside the presence of a number of oxygen vacancies. The open-aperture results revealed saturated inverse non-linear absorption resulting from two-photon absorption. Furthermore, the closed-aperture results indicated the presence of negative non-linear refraction, with the sample having a ratio of* $x = 0.5$ *exhibiting a higher response in non-linear optical properties.* ***Novelty:*** *This result supports its use in promising applications such as laser safety devices, optical switches and photodetectors.*

INTRODUCTION

The fast development of nanocomposite research has greatly influenced contemporary materials science. One such group of materials that have gained attention in recent years due to their unique characteristics is TMO-based nanohybrids [1]. TMO nanohybrids can be seen as a combination of intrinsic properties of TMOs and the flexibility provided by hybrid systems. As a consequence, TMO nanohybrids have become a subject of extensive studies because of their numerous applications in various fields [2], [3]. The design of TMO nanohybrids with functional and biocompatible features marks an important milestone in the realms of materials science and biomaterials. They are not considered anymore as mere inorganic nanoparticles; rather, they are designed systems featuring adjustable interfaces and responses. Currently, the attention will be paid to their properties, possibilities for high performance and importance in the field of modern biomedical issues. Proper control of the synthesis parameters and interface chemistry is essential for improving the performance. It has been pointed out by Modak et al. [4], [5], [6], [7] that the diversity of metal alloys, oxides, and doped versions has expanded the applications significantly. TMOs have been highly sought after for their unique electronic, optical, and chemical characteristics. The partially filled d orbitals play a vital role in these attributes. According to Gupta et al. [8], by proper

material design, functional performance can be optimized for diverse applications. Likewise, Zhang et al. [9] highlighted that functionalization with tungsten oxide on the surface of materials can increase biocompatibility without compromising functional attributes. The existence of partly occupied d-orbitals sets the TMOs apart from several other classes of nanomaterials. The d-electrons take part in oxidation-reduction reactions and, therefore, are associated with catalytic and electrochemical characteristics [10]. Magnetic features also result from the existence of the unpaired d-electrons, allowing for such applications as MRI and magnetically directed drug delivery. In addition, the band gap of many TMOs (ranging between 2.4 and 2.8 eV) makes them suitable for photoactivity under visible light illumination that is important in physiological conditions [11]. The last important factor to be discussed is that of defects such as oxygen vacancies. Defects create local states in the band gap, thus increasing conductivity, catalytic properties, and biomolecular interactions [12]. Contrary to popular belief, defects are usually desirable for their effect on functionality. TMOs have some unique features that distinguish them from other types of nanomaterials. Nanomaterials based on carbon, for example, feature sp2 hybrid orbitals, giving them high conductivity and high strength. They also have zero bandgap in materials like graphene, while the bandgap in carbon nanotubes is tunable depending on various structural factors This makes them applicable in the fields of biosensing and electronics [13], [14], [15], 16], [17], [18].

RESEARCH METHOD

Materials: We obtained lithium nitrate, strontium, cobalt and pure nickel in ratios of (x = 0.5, 1). We mixed these with citric acid and ethylene glycol, which were added as chelating agents and polymerisation initiators, whilst distilled water was added as a solvent

The method adopted for the preparation was the sol-gel method: We dissolved an equimolar and equimass amount of nitrate in water, then added citric acid and ethylene glycol. We heated the mixture to a temperature of (80–90) °C to form a viscous gel. We then dried the mixture at a temperature of (110–150) °C to obtain a fine powder. We then ground it thoroughly and thermally calcined it in a furnace at a high temperature to induce a crystalline phase in the prepared particles. We subsequently examined it at a concentration of 0.01 mM to measure the optical energy gap.

RESULTS AND DISCUSSION

Results

Optical Band Gap Studies and the Influence of Ni Doping

The optical behavior of $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ was studied using the technique of diffuse reflectance spectroscopy (DRS), and the optical band gap energies have been calculated using the Kubelka–Munk transformation of the spectra. As is seen from the Tauc plots in the inset, the optical band gap shows a decreasing trend with increasing Ni

doping level. From the diffuse reflectance spectroscopic measurements, the estimated band gaps are approximately 2.25, and 2.12 eV for x = 0.5, and 1.0, respectively. The decrease in the value of the band gap by nearly 0.30 eV proves that nickel doping does make substantial modifications in the electronic states close to the valence and conduction bands. This reduction in the band gap (Figure 1) can be justified by some reasons. First, the replacement of cobalt ions with nickel ions affects the d orbitals of transition metals that provide the electronic structure of the materials. As a result of overlap between the d orbitals of nickel (Ni 3d), cobalt (Co 3d), and oxygen (O 2p) atoms, the localized states appear in the forbidden energy range, which decreases the energy required to excite electrons. With the increase in nickel content, these localized states appear increasingly clearly and cause the band gap narrowing. One significant finding is the progressive red shift in the position of the absorption edge due to the increase in nickel concentration. This red shift means that there is increased absorption of visible light, which is an advantage in cases where such applications are needed. This is because a lower optical band gap makes the material more efficient in absorbing lower energy photons, which enhances the use of the material in various areas.

This decrease in the optical band gap could be attributed to the increase in oxygen vacancies. Oxygen vacancies arise due to the introduction of new cations through substitution into the compound structure to achieve charge neutrality. Such vacancies provide donor-type energy levels to the structure, thus lowering the optical band gap.

In conclusion, based on the optical properties, it can be seen that Ni is an effective band-gap engineering material in $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ As the continuous reduction from 2.40 to 2.10 eV increases the absorbance of the visible light without destroying the layered structure. With the tunability of the electronic properties along with their structural and magnetic changes, these Ni substituted La-Sr cobaltates have good potential as multi-functional materials in the fields of photocatalysis, magneto-optics, optoelectronics, and non-linear optics. Table 1. shows the effect of Ni substitution in $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$,

Table 1. Summary of the Ni substitution effect

Property	Trend with increasing Ni
Optical band gap (Eg)	Decreases (2.40 → 2.10 eV)
Absorption edge	Red shifts
Visible-light absorption	Increases
Lattice distortion	Slightly increases
Crystallite size	Decreases
Defect density	Increases
Ferromagnetic interaction	Weakens
Electronic localization	Increases
Photonic/optoelectronic potential	Enhanced

The gradual decline in the optical bandgap value with high correlation to the structural, morphological and magnetic behavior confirms the effectiveness of Ni doping on tuning the physical properties of $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ without destabilizing its layer structure.

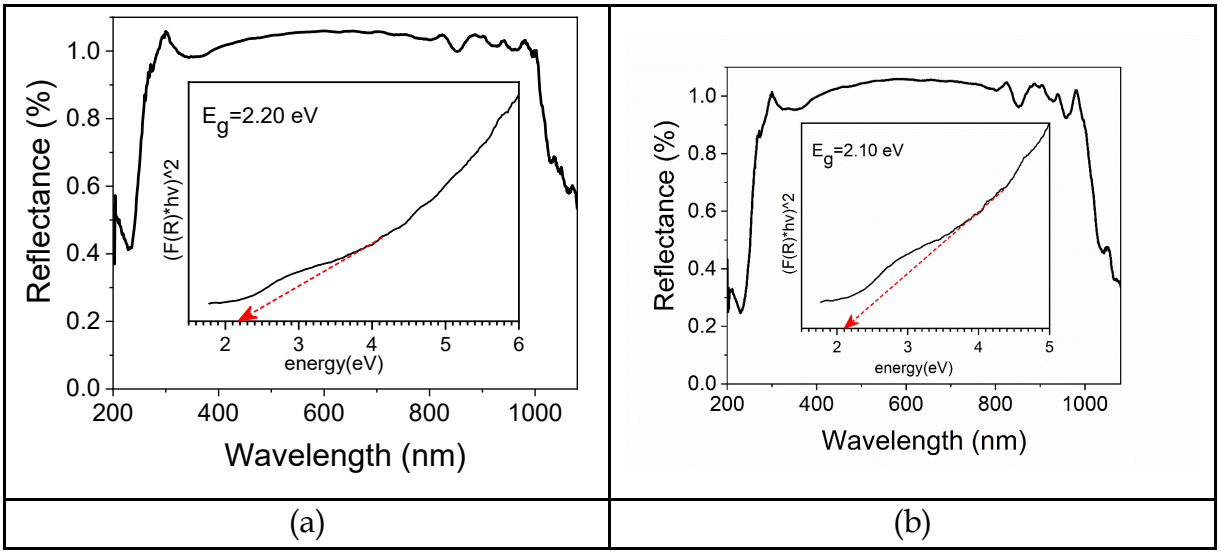


Figure 1. Reflectance spectra of $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ for (a) x = 0.5, and (b) x = 1.0 (inset: Band gap calculation of samples by Kubelka-Munk)

Properties related to nonlinear optics

We have investigated the non-linear optical properties of nanoparticles of the aforementioned compound $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ by employing a z-axis scanning technique using two modes—the closed mode and the open mode—via a Nd:YAG laser with a wavelength of approximately 532 nanometres, such that the beam spreads along the z-axis over the sample mounted on the stage, which is moved under computer control. Figure 2 shows the diagrams we used on the z-scanner, where measurements were taken using the open aperture (S = 1), set to a concentration of 0.012 mmol of the nanoparticle compound under study.

Open-Aperture Z-scan Study

By analysing the technical data from the in-axis scan, we can observe a marked increase in non-linear absorption in the nickel-doped material compared to the pyrrolysine viscose material. It is also evident that, at all concentrations, there is a significant decrease in permeability in the focal region; this in particular indicates that reverse absorption exerts a strong influence on both two-photon absorption and excited-state absorption. As is customary, non-linear absorption can be defined as a two-photon absorption phenomenon. Our study of the sample using the aforementioned technique has shown that the normalised transmittance decreases directionally as we approach the centre of the, which illustrates a sharp drop in the transmittance curve at the focal point. The symmetry of the curve at the focal centre indicates that the two-photon absorption

effect dominates the non-linear absorption phenomenon in the nanoparticles of the compound under study $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$, as shown in Figure (3).

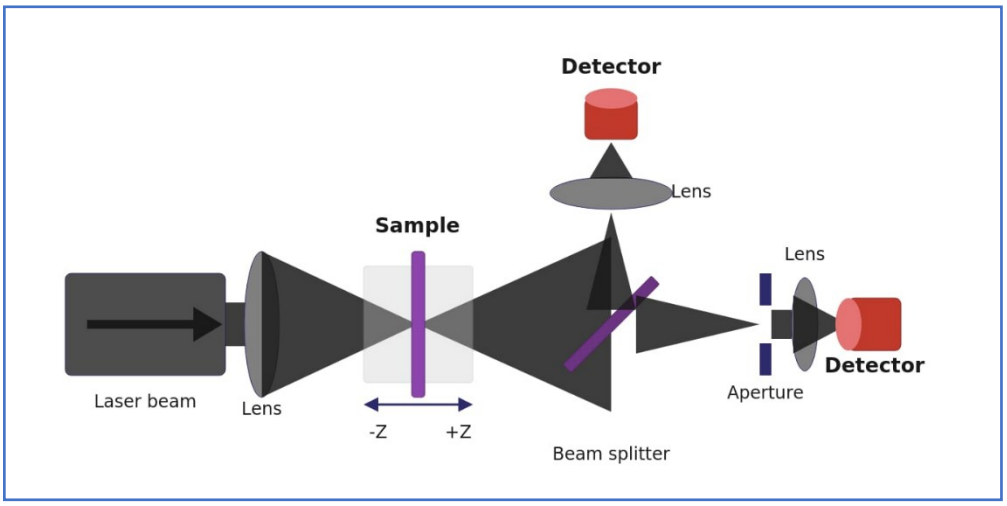


Figure 2. The experimental Z-scan setup.

Two-photon absorption (TPA) occurs when two photons absorb simultaneously, having either same or different energies in such a way that electrons are excited from ground state to excited states (S0→S1). The transition energy in this case is the sum of energy of two photons. The nonlinear absorption coefficient (β) can be calculated using open aperture Z-scan data by the following equation :

$$\beta(cm/W) = \frac{2\sqrt{2}\Delta T}{I_0 L_{eff}} \quad (1)$$

where $\Delta T = 1 - T(z)$ is the normalized transmittance at z = 0, and $L_{eff} = [(1 - \exp(-\alpha L))/\alpha]$ is the effective length of the sample. Based on the fitted curves solid line in Figure 3, the It would be possible to fit the experimental data using the NLA models. The empirical results for NLA (scatter plots) were confirmed by the theoretical results (continuous curves) using the following equations :

$$T_{norm}(z) = \sum_{m=0}^{\infty} \frac{[-q_0(z,0)]^m}{(m+1)^{3/2}} \quad (2)$$

$$q_0(z,t) = (\beta I_0 L_{eff}) / (1 + z^2 / z^2_0) \quad (3)$$

Whereas is associated with multi-photon effect. The value of the NLA coefficient, which has been measured using open aperture Z-scan approach. The nonlinear optical properties of the $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ nanoparticles are listed in Table 2, through Z-scan

measurement. It has been seen that the $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ nanoparticles possess TPA response in CW mode and also their values vary for different Ni dopants.

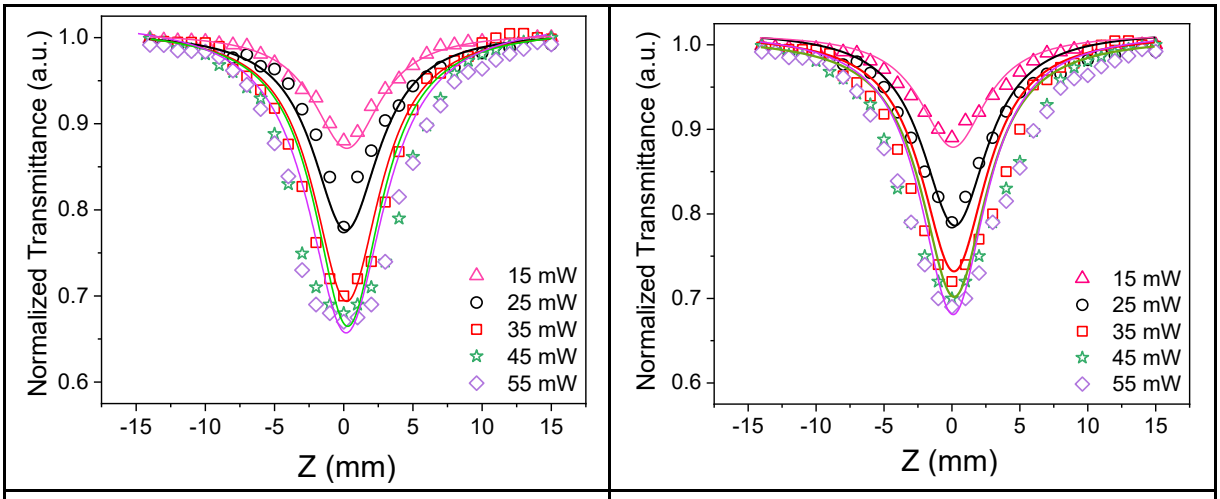


Figure 3. Open aperture experimental data (spot line), fitted data (straight line) of $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ of (a) x = 0.5, and (b) x = 1.0

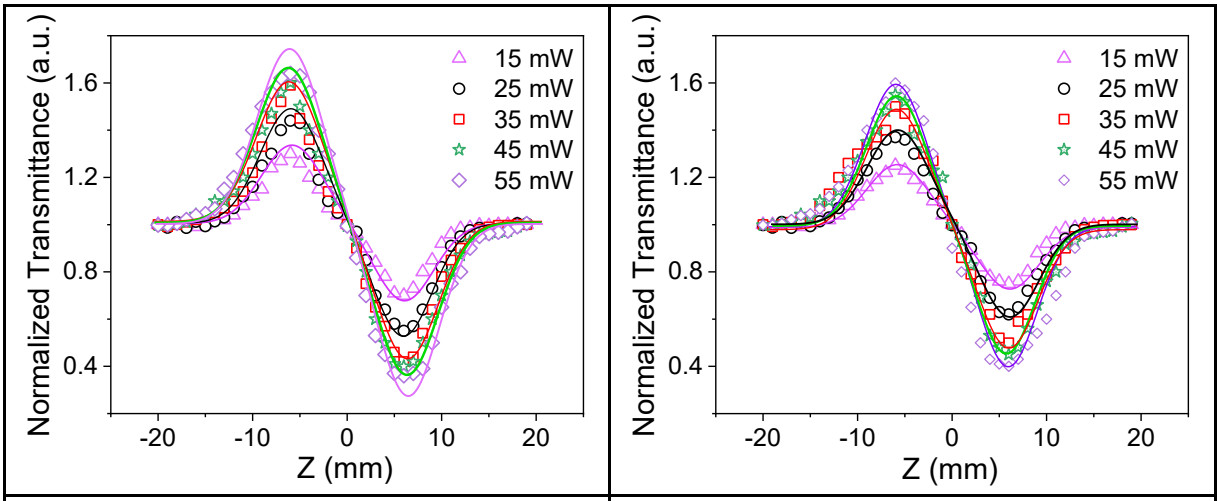
Nonlinear absorption being high for x = 0.5 implies that this is the best ratio with respect to balancing of defect assisted excitation and carrier mobility. Even though the x = 1.0 composition has the smallest crystallite size and low band gap, structural disorder and carrier scattering could limit the efficiency of nonlinear absorption due to increased nonradiative relaxation paths. That is why there is a decrease in β at the highest concentration of Ni. The dependence of β on laser power also confirms this explanation. Raising laser power from 15 to 55 mW will increase the number of photons at the focus region. This raises the probability of simultaneous absorption of photons and population of the excited state. Therefore, there is high nonlinear absorption for all the compositions.
Analysis of Closed-Aperture Z-scan Results

In all the solutions investigated, the results of the closed aperture Z-scan measurements exhibit the peak-valley structure, suggesting negative nonlinear refraction and self-defocusing. The value of the peak-to-valley difference becomes larger as the values of laser power and nickel concentration increase, showing that there has been a remarkable increase in the nonlinear refractive response. Closed aperture Z-scan technique has been utilized to determine the sign and value of the nonlinear refraction index, where an appropriate aperture has been placed in front of the detector. This set of data includes not only the NLR but also NLA. It is necessary to separate the NLA from NLR to obtain pure nonlinear refraction. To this end, a facile and accurate approach has been adopted. The pure nonlinear refraction graph of La0.7Sr1.3Co1-xNixO4 aqueous solution (0.01mM) is shown in Figure 4. The negative value of the nonlinear refraction index (n_2) and self-defocusing phenomenon have been determined based on the peak-valley structure of the transmittance graph. This kind of self-defocusing phenomenon can

be attributed to the thermal nonlinearity due to the absorption of the laser at 532 nm. Temperature distribution in the solution arises due to the local absorption of a focused beam that is transmitted through the samples. This finally results in spatially varying refractive index behaving like a thermal lens causing beam phase distortion. Here, ΔT_{P-V} represents the difference between the normalized transmittance peaks and valleys (T_P-T_V). The above quantity can be expressed using the relation :

$$\Delta T_{P-V} = 0.406(1-S)^{0.25}\left|\Delta\phi_0\right| \quad (4)$$

Where $|\Delta\phi_0|$ indicates the phase shift on the axis at the focal point, $S = 1-\exp(-2r_0^2/\omega_0^2)$ indicates the linear transmittance of the aperture, ω_0 indicates the aperture radius, while indicates the radius of the beam at the aperture position in the linear regime.



(c) (d)

Figure 4. Closed aperture experimental data (spot line), fitted data (straight line) of $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ of (a) x = 0.5, and (b) x = 1.0

In contrast to typical semiconducting nanostructures where quantum confinement determines optical properties, the current crystallites are quite large as compared to the confinement regime. Thus, the increase in non-linear refractive index is not determined by the size quantization effect. Rather, this phenomenon is caused by the band structure modification due to the substitution of Co by Ni. The substitution of cobalt by nickel leads to an appearance of additional 3d bands and the change of the hybridization of transition-metal d bands and oxygen 2p bands. This causes an increase in the concentration of electronic states near the edges of the band and thus makes possible electron transfer during optical excitation. With the decrease in optical band gap, the polarization of electrons by the incident electromagnetic field requires less energy; therefore, the third-order susceptibility increases and non-linear refractive index becomes larger ,

Furthermore, the decrease in the crystallite size is an indirect factor responsible for the increase in nonlinear refraction. The crystallites with smaller sizes have higher densities of grain boundaries and structural defects which result in the formation of localized electronic states. These states increase the electronic polarizability and the interaction of the optical field with the electronic structure, leading to increased refractive index modulation during laser illumination. The highest value of the nonlinear refraction coefficient reached when x = 0.5 suggests that this ratio provides the optimal compromise between the density of the electronic states and crystal structure. Moderate amounts of nickel allow increasing the number of the localized states and decreasing the band gap energy while not damaging the crystal structure. On the other hand, x = 1.0 might result in higher defect concentrations and crystal lattice deformation that can lead to increased carrier trapping and scattering processes thus reducing the effectiveness of coherent electronic polarization.

Table 2. The linear and nonlinear optical properties of $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ (x = 0.5, 1.0) at incident powers P_{in} = 45, and 55 mW.

Sample (x)	P_{in} (mW)	α (cm^{-1})	L_eff (mm)	$n_2 \times 10^{-8}$ (cm^2/W)	$\beta \times 10^{-4}$ (cm/W)
0.5	35	0.46	0.97	−11.57	37.43
	45	0.20	0.99	−12.19	41.33
	55	0.17	0.99	−14.46	47.42
	15	1.02	0.95	−9.61	26.85
	25	0.82	0.96	−9.83	34.96
1.0	35	0.56	0.97	−9.87	35.11
	45	0.33	0.98	−11.26	39.01
	55	0.11	0.99	−13.53	44.39

Power of the laser is equally significant in defining the nonlinear refraction phenomenon. As the power increases from 15 mW to 55 mW, there is an increase in the optical intensity in the vicinity of the focal point, resulting in more carrier excitation and more induced polarization, causing the phase deformation to become significant and producing greater peak-to-valley separation. In summary, the nonlinear refractivity in $La_{0.5}Sr_{1.5}Co_{2-x}Ni^xO_2$ is due to the result of the synergistic action of Ni-induced band gap narrowing, increase in defect state concentration, increased electronic polarizability, and laser intensity-dependent electron excitation. The close relationship between decreased crystallite size, optical band gap narrowing, and nonlinear refractive effect is indicative of the effectiveness of Ni-substitution in tuning the third-order nonlinear optical behavior of layered oxides. The best performance obtained at x = 0.5 demonstrates its potential use in optical switches, optical modulators, laser protection, and novel photonic devices. In recent studies by Z-scan technique on oxides and oxide-based nanomaterials, the compositional and excitation dependent nonlinear absorption/refractivity, including

reverse saturable absorption and negative nonlinear refractivity/ self-defocusing have been reported.

Discussion

The decrease in the optical bandgap from 2.25 eV at $x = 0.5$to 2.12 eV at $x = 1.0$indicates that Ni substitution modifies the electronic structure of $La_{0.5}Sr_{1.5}Co_{2-x}Ni_xO_2$. The optical bandgap values were determined from diffuse-reflectance data using the Kubelka–Munk transformation, which provides a relationship between diffuse reflectance and the effective absorption behavior of powdered materials [19]. The observed bandgap narrowing can be associated with changes in the hybridization among Ni 3d, Co 3d, and O 2p orbitals, together with the formation of localized states and oxygen vacancies within the forbidden energy region. In transition-metal systems with octahedral coordination, the intensity of nominally forbidden electronic transitions may be influenced by local symmetry, orbital interactions, and lattice distortions [20]. Therefore, the structural and electronic modifications induced by Ni substitution may facilitate optical transitions and shift the absorption edge toward lower photon energies. Although $x = 1.0$produced the lowest optical bandgap, the stronger non-linear optical response at $x = 0.5$suggests that excessive Ni incorporation increases structural disorder and carrier scattering. Thus, the $x = 0.5$composition provides a more favorable balance between defect-assisted optical excitation, electronic polarizability, and preservation of the crystal structure.

CONCLUSION

Fundamental Finding: Nickel doping in $La_{0.5}Sr_{1.5}Co_{2-x}Ni_xO_2$ narrows the optical bandgap by creating electronic states and increasing oxygen vacancies, thereby enhancing the non-linear optical response, with the best performance achieved at x = 0.5. **Implication:** This optimal doping level balances the material properties and supports promising applications in optical switches and laser protection. **Limitation:** A higher doping ratio of x = 1 may cause excessive structural defects that reduce material performance. **Future Research:** Future studies should investigate intermediate doping ratios and optimize the synthesis conditions to minimize structural defects and further improve the material's non-linear optical properties.

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