

NO_x and CO₂ conversion efficiency of nitrogen-modified strontium titanate

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ABSTRACT

Nitrogen doping and surface modification are widely used strategies to enhance the photocatalytic performance of perovskite oxides such as SrTiO₃. These approaches can increase the lifetime of charge carriers and locally modify the electron density through the incorporation of elements with different electronegativity or surface modification, thus promoting NO_x and CO₂ conversion. In this work, SrTiO₃ was modified via a simple synthesis method, resulting in a nominal nitrogen content of 0.42–11.14 at.%. The sample containing 7.71 at.% nitrogen exhibited the highest photocatalytic activity, achieving a 67% improvement in NO_x conversion and a 2.2-fold enhancement in CO₂ conversion compared to unmodified SrTiO₃. Characterization results showed that low nitrogen precursor concentrations led to only limited nitrogen incorporation, which did not significantly affect the photocatalytic activity. However, surface amine groups were already detectable at low precursor amounts, while higher precursor amounts promoted increased nitrogen incorporation into the SrTiO₃ photocatalysts.

1. Introduction

Photocatalysts remain a frequently researched topic due to their wide range of applications, including air purification, water purification, and disinfection [1–3]. Photocatalysis is a subclass of advanced oxidation processes (AOPs), where generated radicals are used to degrade organic pollutants [4,5]. The conversion of pollutants takes place on the surface of photocatalysts or in their immediate environment. Photocatalysis proceeds efficiently when the photogenerated charge carriers are consumed by suitable donor and acceptor species, such as harmful pollutants (NO_x, HCOOH, phenol) [6]. The photocatalytic activity can be sustained as long as light irradiation and these active species are present.

Perovskites, including strontium titanate, are among the most popular photocatalysts, offering numerous advantages such as low cost, stability, inertness, and relatively high photocatalytic efficiency [7–10]. The aim is generally to increase their photocatalytic activity by

controlling the material properties. One such method is doping, in which a foreign element is incorporated into the crystal structure (e.g., Au, Pt, Rh, C, N), causing changes in the lattice and band structure [11,12]. Surface modification with functional groups represents another strategy, as it alters the surface properties and thus influences the interaction between the photocatalyst and the target molecules [13]. Moreover, surface modification through metal deposition can enhance photocatalytic performance via plasmonic effects or by providing active sites for adsorption, bond activation, or bond breaking. Alternatively, metal deposition or coupling with a second semiconductor can create heterojunctions (e.g., type-I, type-II, Z-scheme), which increases the recombination time [14,15].

Among non-metallic elements, nitrogen is one of the most commonly used dopants [16–18]. Doping causes changes in crystallographic properties such as lattice parameters, degree of crystallinity, and primary crystallite size. Doping introduces defect states within the band gap, which can serve as trapping sites for photogenerated electrons [19,

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20]. With a suitable nitrogen source, nitrogen-containing groups (e.g., amine groups, nitro groups) may also form on the surface in addition to doping. These have an electron-attracting effect, improving the reducing ability of the photocatalyst, in addition to increasing the recombination time of charge carriers. Many papers have investigated modification possibilities with nitrogen, but they mainly deal with doping. Most of this work has focused on widely studied materials such as TiO_2 and ZnO , but the modification of perovskites with nitrogen has also been reported [21]. In contrast, studies addressing the nitrogen modification of SrTiO_3 remain limited, and the few available reports [22,23] largely involve applications other than photocatalysis. This is noteworthy given that surface amination has proven effective for other photocatalysts, such as CuO , where it enhanced photocatalytic activity while simultaneously reducing ecotoxicity [24].

In this work, we present a simple synthesis method to produce SrTiO_3 containing nitrogen in various forms and concentrations. Beyond nitrogen doping, we also detected surface-bound, nitrogen-containing functional groups ($-\text{NH}_2$, $-\text{NO}_2^-/\text{NO}_3^-$). We determined the optimal nominal nitrogen content required to maximize both NO_x and CO_2 conversion. To our knowledge, no other study has investigated such dual applicability (i.e., NO_x , CO_2 conversion) on SrTiO_3 that is both doped and surface modified.

2. Materials and methods

2.1. Materials

Titanium tetraisopropoxide ($\text{Ti}(\text{OC}_3\text{H}_7)_4$, 97%, Aldrich), strontium nitrate ($\text{Sr}(\text{NO}_3)_2$, 98.5%, Molar), 2-propanol ($\text{C}_3\text{H}_8\text{O}$, technical, VWR), hexamethylenetetramine ($\text{C}_6\text{H}_{12}\text{N}_4$, >99%, Sigma-Aldrich), potassium hydroxide (KOH , 85.7%, VWR), and ultrapure (Milli-Q) water were used for the syntheses. All chemicals were used without further purification.

2.2. Synthesis

The samples were prepared via solvothermal synthesis, based on a synthesis method described in the literature [25]. First, titanium tetraisopropoxide was added to 2-propanol, setting a titanium content of 0.2 mol L^{-1} . Then, an aqueous solution containing 0.2 mol L^{-1} strontium and varying concentrations of hexamethylenetetramine (corresponding to the desired nitrogen content) was prepared. In the next step, the aqueous solution was added dropwise to the titanium-containing solution. After this step, 0.2 mol L^{-1} potassium hydroxide solution was added to the system ($\text{Sr}/\text{K} = 1/2$). The solution was heated at 95°C for 2 h to obtain an amorphous strontium titanate precipitate. Finally, this nitrogen-containing precipitate was placed in a Teflon-lined stainless-steel autoclave at 200°C for 5 h for crystallization. After cooling, the product was centrifuged three times in water and ethanol and dried at 50°C for 24 h. The samples are named “NS_X”, where “N” is nitrogen, “S” is SrTiO_3 , and “X” stands for the nominal nitrogen content in at.% (Table 1.). A reference sample was also prepared in the same way, without adding hexamethylene-tetramine.

Table 1

Primary crystallite size, lattice strain, and specific surface area of the prepared N-modified SrTiO_3 samples.

Sample name	Primary crystallite size (nm)	Lattice strain	Specific surface area (m^2g^{-1})
NS_0	30.4	$1.997\cdot 10^{-3}$	63.9
NS_0.42	32.9	$1.756\cdot 10^{-3}$	54.5
NS_0.83	30.3	$1.480\cdot 10^{-3}$	48.2
NS_1.24	32.3	$1.512\cdot 10^{-3}$	41.3
NS_1.64	29.2	$1.642\cdot 10^{-3}$	66.0
NS_4.01	29.2	$1.563\cdot 10^{-3}$	49.8
NS_7.71	29.0	$2.117\cdot 10^{-3}$	52.3
NS_11.14	29.7	$1.828\cdot 10^{-3}$	48.2

2.3. Characterisation

A Bruker D8 Advance X-ray diffractometer (Bruker, Billerica, Massachusetts, USA) was used to determine the crystalline composition of the samples in the range of $20\text{--}80^\circ 2\theta$ at 20°C . During the measurements, the following parameters were used: $\lambda_{\text{CuK}\alpha} = 0.15406 \text{ nm}$, 40 kV, and 30 mA. The Williamson-Hall plot was used for the calculation of crystal parameters and crystal strain [26].

Specific surface areas were determined by N_2 adsorption at 77 K with a Micromeritics Gemini 2375 Surface Area Analyzer device (Micromeritics, Eindhoven, Netherlands).

FT-IR spectra were recorded with a JASCO 4700 Fourier-transform infrared spectroscope (ABL&E-JASCO, Budapest, Hungary). The measurement range was from 4000 to 500 cm^{-1} , with a resolution of 1 cm^{-1} . Before the measurements, we prepared KBr pellets containing 1 wt% of the given sample.

Pt reduction was used to verify the presence of surface amine groups. Pt ions were added to the photocatalyst suspension and then illuminated with UV light, without the addition of the reducing agent. The photo-deposited platinum on the surface was measured using an Olympus VMR X-ray fluorescence (XRF) spectrometer. The platinum $\text{K}\alpha_1$ electron transition was detected at a binding energy of 9.43 keV, with an excitation beam of 50 keV. Due to the electron-withdrawing effect of the surface-bound groups, electrons are trapped and can be utilized through the surface groups.

Diffuse reflectance spectra (DRS) were recorded between 200 and 800 nm using a Shimadzu UV-3600 Plus UV-VIS-NIR (Shimadzu Corporation, Kyoto, Japan) spectrophotometer with an integration sphere for optical characterization. The Kubelka-Munk method was used to determine the band gap values. By determining the first-order derivative of the spectra, we obtained the inflection point, which corresponds to the band gap value [27].

Time-resolved fluorescence lifetime and room-temperature steady-state photoluminescence (PL) were measured using a FluoroMax spectrofluorometer equipped with a DeltaHub TCSPC controller, with an excitation laser wavelength of $\lambda_{\text{ex}} = 375 \text{ nm}$.

The photocurrent density was determined in a classic three-electrode system in an aqueous medium. The instrument used was a Metrohm Autolab PGSTAT302n potentiostat/galvanostat (Metrohm AG, Herisau, Switzerland). Fluorine-doped tin oxide (FTO)/photocatalysts were prepared by cutting $1 \times 2.5 \text{ cm}$ FTO plates, and covering them with 10 mg cm^{-2} NS_X samples on a 1 cm^2 surface using the spray coating method. The FTO/photocatalyst was used as the working electrode, platinum wire was used as the counter electrode, and an Ag/AgCl electrode (in 3 mol L^{-1} NaCl) was used as the reference electrode. The experiments were carried out in a 0.5 mol L^{-1} Na_2SO_3 solution, which ensured conductivity and underwent oxidation as a hole acceptor on the working electrode, while H_2 evolution occurred on the counter electrode. The intensity of the incoming light was 100 mW cm^{-2} , and the current density was obtained in mA cm^{-2} . The photocurrent density was measured using linear sweep voltammetry (LSV) in the potential range of -1.2 V to 0.3 V at a sweeping rate of 2 mV s^{-1} . The spectrum of the light source used is shown in Fig. S1.

Electrochemical impedance spectroscopy (EIS) measurements were carried out at open circuit potential (OCP) under continuous UV illumination (100 mW cm^{-2}), using a 10 mV rms AC perturbation in the frequency range from 200 kHz to 0.1 Hz. For the Mott-Schottky analysis, EIS spectra were recorded at different potentials in the frequency range between 100 kHz and 1 Hz, and the flatband potential was determined at 1 kHz. Before each measurement, a pretreatment step was applied at the given potential for 30 s. The electrolyte was $0.5 \text{ M Na}_2\text{SO}_3$.

The morphology of the samples was studied using transmission electron microscopy (FEI TECNAI G2 20 X-Twin type transmission electron microscope), with a tungsten cathode operated at 200 kV.

2.4. Photocatalytic activity measurements

2.4.1. Photocatalytic NO_x conversion

The photocatalytic activity was examined through the conversion of NO_x gases (NO and NO₂). The measurements were largely performed in accordance with the ISO 22197-1:2016 standard, with only slight differences from the standard due to practical reasons or necessity [28]. The test gas composition contained ~1000 ppb NO in high-purity air, and 50% relative humidity. The test gas was fed into a stainless-steel reactor sealed at the top with a quartz window. The photocatalyst layer, which was formed on a 10 × 10 cm glass plate substrate (with 1 mg cm⁻² photocatalyst content) using a spray coating method, was placed in this reactor. The UV-A fluorescent tube light source (emission spectrum shown in Fig. S2) was placed 5 cm above the sample. Part of the test gas leaving the reactor at a flow rate of 1.5 L min⁻¹ was fed into an Environnement SA AC32SA NO_x analyzer (Envea, Poissy, France). Photocatalytic experiments were performed for 120 min, preceded by a 20-min equilibrium period to ensure the stability of the pollutant concentration, and followed by a 20-min period to ensure that the test gas concentration returned to its original value.

2.4.2. Photocatalytic CO₂ conversion

For the photocatalytic CO₂ hydrogenation, we used a flow micro-reactor consisting of two glass cylinders with an outer diameter of 10.2 cm, an inner diameter of 6.4 cm, and a length of 25 cm. The inner cylinder contained an 11 W UV-A light source (the spectrum is shown in Fig. S3). The photocatalyst (250 mg) was immobilized on the outer wall of the inner cylinder using the spray-coating method. During sample preparation, the sample was heated to 250 °C, then, various gases were introduced (Ar for 20 min, O₂ for 30 min, Ar for 10 min, and H₂ for 60 min). This was followed by the introduction of the test gas (CO₂ and H₂ in a ratio of 1:2). One photocatalytic test lasted for 250 min. The reactor was connected to a gas chromatograph (HP 5890 Series II GC), with a pump between the reactor and the GC to continuously circulate a pre-determined amount of gas mixture. A constant room temperature was maintained by circulating water in the reactor jacket. A 2-m-long (d = 0.635 cm) capillary column filled with a Porapak QS polymer was used to separate the products and reactants. A thermal conductivity detector (TCD) and a flame ionization detector (FID) were used to identify the reaction products.

3. Results and discussion

3.1. Synthesis

Over the course of the synthesis, the hydrolysis of titanium

isopropoxide occurs first, resulting in a cross-linked, amorphous titanium oxide-hydroxide precipitate. Subsequently, upon the addition of KOH, the precipitate partially dissolves, leading to the formation of dissociated K₂TiO₃ and Sr(OH)₂. Raising the temperature promotes the formation of amorphous SrTiO₃ in the presence of other intermediate products. Due to the alkaline medium, the dissolved hexamine is present in the form of NH₄⁺ ions (as NH₄OH or dissolved ammonia) since the pH is higher than the pK_b value of NH₄OH. During nucleation, ammonia adsorbs onto the surface of the nuclei and can be incorporated into the structure during growth, resulting in crystal defects.

3.2. Characterization

Fig. 1a shows the diffraction patterns of the samples. The following crystal planes can be identified: (100), (110), (111), (200), (210), (211), (220), (300), and (310), all belonging to SrTiO₃ (JCPDS No. 41-1426). The calculated primary crystallite size (based on the Scherrer equation) and lattice stress values are shown in Table 1. The lattice strain is also shown graphically in Fig. 1b. The change is relatively small, so only a few additional defects were created during doping [29]. Initially, the incorporation of nitrogen stabilizes the lattice; however, further nitrogen content induces strains, as illustrated in the figure. The largest lattice strain is observed in the NS_7.71 sample, which is the only sample exhibiting a lattice strain greater than that of the unmodified SrTiO₃. This can be explained by the fact that different types of lattice defects form in the presence of nitrogen.

The specific surface areas are presented in Table 1. In almost all cases, modification with nitrogen decreased the specific surface areas, even in cases where the primary crystallite size is smaller. This is likely attributable to the lower degree of crystallinity.

Fig. 2 shows the FT-IR spectrum of NS_7.71, including the regions characteristic of amine and nitrate groups marked. The signal appearing between 1330 cm⁻¹ and 1450 cm⁻¹ is the symmetric stretching vibration of the N–O bonds in NO₂ groups (ν_s) [30]. The signal at 1410–1570 cm⁻¹ corresponds to the stretching vibration of N=O bonds (ν) [30]. The vibration between 1560 and 1695 cm⁻¹ corresponds to N–H scissoring vibrations (δ), proving the presence of surface amine groups [31]. The large number of NO₃ and NO₂ groups on the surface is due to the Sr (NO₃)₂ used during synthesis. The NH₂ groups on the surface originate from NH₃ produced during synthesis.

To determine the band gap value of the samples, DRS spectra were recorded (Fig. 3). The figure shows that the samples exhibit highly similar optical characteristics. The indirect band gap value of SrTiO₃ is 372 nm, which decreases continuously to 366 nm with increasing nominal nitrogen content. This slight blue shift indicates the slight modification of the structure. This change was previously confirmed via

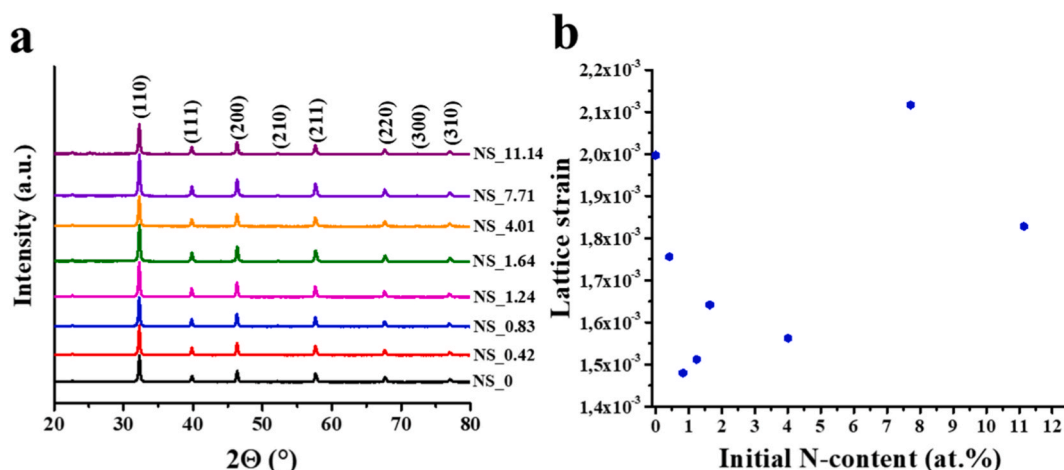


Fig. 1. XRD patterns of the NS_X samples (a) and lattice strains obtained via the Williamson–Hall plot (b).

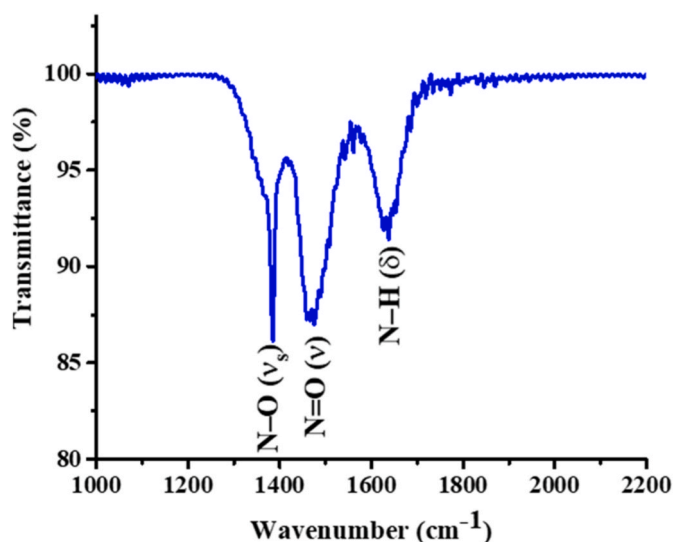


Fig. 2. FT-IR spectrum of nitrogen-containing compounds in NS_7.71.

the lattice strains. The second detectable inflection point (at ~ 328 nm) corresponds to the direct bandgap value of SrTiO_3 [32].

Photoluminescence results provide information on the recombination of the sample excited via photon emission [33]. The number of emission maxima corresponds to the number of electron transitions that recombine with photon emission. The intensity enables a relative comparison of the probability of radiative recombination at a specific energy transition. A new energy level can form not only due to the incorporation of a foreign element, but also due to other lattice defects. The existence and number of new energy levels depend on the synthesis conditions. The emission spectrum of the samples is shown in Fig. 4. An emission maximum can be observed at around 470 nm, which is due to direct recombination. This recombination is usually close to the wavelength of the exciting photons [34]. Another much weaker recombination within the band gap can also be observed around 522 nm. Based on literature data, this is a defect site, an oxygen vacancy [35]. The emission intensity of the samples decreases with increasing nominal nitrogen concentration, which is beneficial for enhancing photocatalytic activity. Most importantly, assuming that the same number of electrons are excited in all samples, the fewer electrons recombine with photon emission, the greater the statistical chance that the excited electrons recombine or get trapped in another process.

Since the spectra are identical and no additional emission maximum

can be detected, this method could not detect the new energy level associated with nitrogen adsorbed on the surface. This is because the structure of SrTiO_3 does not change with the appearance of surface-bound groups. These groups mainly act as electron traps, which can lead to delayed recombination. Time-resolved photoluminescence measurements were used to measure this.

The photoluminescence lifetime of the samples was also measured after recording the instrument response function (IRF) signal, which was then used to correct the corresponding sample data. Fig. 5 shows the photoluminescence emission spectrum of the initial, unmodified SrTiO_3 and that of the NS_7.71 sample. The measured values are summarized in Fig. 6. The photoluminescence lifetime was calculated from the fitted function, which was higher for all modified SrTiO_3 samples than for SrTiO_3 , with the highest value being 2.31 ns for the NS_7.71 sample. The accuracy of the fit is shown in the inserted smaller spectrum, which shows the difference between the fitted and measured functions. The two curves fit well, as evidenced by residual values that are four orders of magnitude smaller than the signal intensity for SrTiO_3 and three orders of magnitude smaller for the NS_7.71 sample than for the measured and fitted curves. The results show that modifying SrTiO_3 increases the lifetime of excited electrons, which is beneficial for photocatalytic applications. These results are consistent with the photocatalytic results presented later and with those obtained from photocurrent density measurements.

The photocurrent density results, measured at -0.1 V (vs Ag/AgCl), are shown in Table 2. The new energy levels created by defect sites can trap excited electrons, which therefore do not contribute to the photocurrent [36], while the nitrogen-containing groups on the surface increase the lifetime of excited electrons due to their electron-withdrawing effect. This allows the remaining positive holes to participate in oxidation reactions, thereby increasing the photocurrent. Since the measured values increase with nominal nitrogen content, the effect of the surface groups appears to dominate. In NS_11.14, which has the highest nominal N content, the photocurrent decreased. This is plausible given the maximum nitrogen doping level in this sample, which leads to significant electron trapping by the newly formed energy level. Fig. 7a shows the LSV voltammograms of the pristine SrTiO_3 and NS_7.71 samples compared to that of the dark current. The curves are similar in shape, but the modified sample clearly shows much higher values at the same voltage.

The Mott-Schottky measurements were carried out to identify the flatband potential of pristine strontium titanate and the best-performing nitrogen-modified sample (Fig. 7b). The flat band potential determines the theoretical photocurrent onset of a given photoelectrode. The flat-band potential was similar for the two electrodes, specifically it was

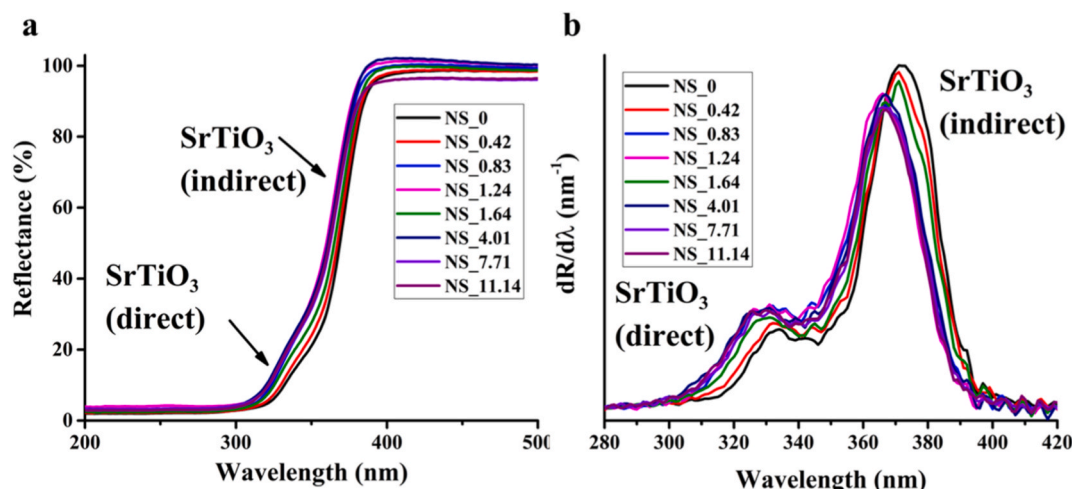


Fig. 3. Absorbance spectrum of the N-modified SrTiO_3 (a) and the first-order derivative spectrum of the samples.

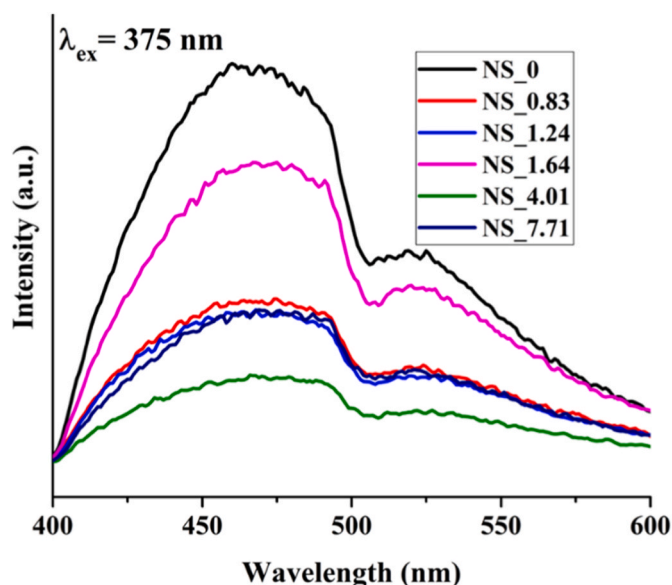


Fig. 4. PL spectrum of selected N-modified samples.

–1.17 V for the pristine and –1.15 V for the N-modified sample. This is not surprising, since the semiconductor material is the same in both cases. The slight shift in the flat-band potential of NS_7.71 suggests that the quality and/or quantity of defects in this sample differs from those in pristine SrTiO₃ [37,38]. This supports previous reports indicating that the defect structure of SrTiO₃ is modified—either directly through nitrogen doping or indirectly via nitrogen-induced changes in other defect types. The determined flatband potential is consistent with the observed photocurrent onsets in the photovoltammograms (Fig. 7a). However, the modified electrode exhibits much higher photoelectrochemical activity, which can be attributed to the reduced R_{ct} (as detailed in our previous response) and consequently to the enhanced charge extraction efficiency.

The EIS measurements were performed at open circuit potential (OCP) under 100 mW cm^{–2} UV illumination for both pristine strontium titanate and the best-performing nitrogen-modified sample (NS7.71). The Nyquist plots exhibit a single semicircle in both cases (Fig. 8a) and were fitted using an Rs–(R_{ct}||Q) equivalent circuit (Fig. 8b), where Rs represents the series resistance, R_{ct} the charge transfer resistance, and Q the constant phase element. While the Rs values were identical for both electrodes (20.1 Ω for the pristine and 20.7 Ω for the N-modified sample), a substantial difference was observed in the R_{ct} values (70.9 kΩ

and 42.0 kΩ, respectively). This approximately two fold decrease in R_{ct} accounts for the markedly higher photocurrents observed for the N-modified strontium titanate, as shown in the manuscript (and in Fig. 8b). This provides further confirmation of the findings described in the Mott-Schottky analysis and the photocurrent density measurement.

The TEM image of NS_7.71 (Fig. 9) shows that the particles are nearly cubic in shape, which is consistent with the cubic structure determined by XRD. It can also be seen that the particles are approximately the same size, i.e., the sample has a homogeneous size distribution.

3.3. Photocatalytic activity measurements

3.3.1. Photocatalytic NO_x conversion

The photocatalytic activity was examined through the conversion of NO_x gases. SrTiO₃ without nitrogen was examined as a reference sample (NS_0). The results are summarized in Table 3 and in Fig. 10. NO_x conversion increases with increasing nominal N content, and the highest conversion (10.45%) can be observed for the NS_7.71 sample. This result correlates well with the previously presented ones: this sample has the highest lattice strain, the highest photocurrent density, and the best parameters based on the steady-state and time-resolved PL measurements. As the characterization results showed before, this sample exhibits the highest degree of modification. The material properties of NS_11.14 are less strongly modified than those of NS_7.71 under the specific synthesis conditions used. Table 3 also shows the NO conversion and NO₂ generation data. High NO conversion is observed to result in high NO₂ generation, which is consistent with the NO→NO₂ conversion being the dominant reaction pathway in the reactor [39]. Therefore, in the context of overall NO_x conversion, the rate and extent of NO consumption are not the most critical factors. Due to the presence of water vapor, an HNO₂/HNO₃ layer can also be formed on the surface, which passivates the surface, but the excited photocatalyst can reduce the nitric acid on the surface to NO₂, thus creating an equilibrium, in which the NO→NO₂ conversion dominates overall, resulting in a very slight decrease in NO_x concentration [40].

The decrease is due to reductive processes: SrTiO₃ has a relatively negative conduction band, so the excited electron has relatively high energy, making it potentially capable of reducing NO_x gases. Due to the rapid gas flow through the reactor, complete reduction to N₂ or NH₃ is unlikely. Instead, the formation of N₂O or N₂O₅ is expected. Since the instrument cannot measure these specific products, their formation is reflected in the data as a decrease in the total NO_x concentration [41]. This phenomenon is not observed in the case of TiO₂; after a short time, the NO_x concentration returns to its initial value and does not change, with only NO→NO₂ conversion occurring. Therefore, even though the NO concentration drops significantly at the beginning due to the

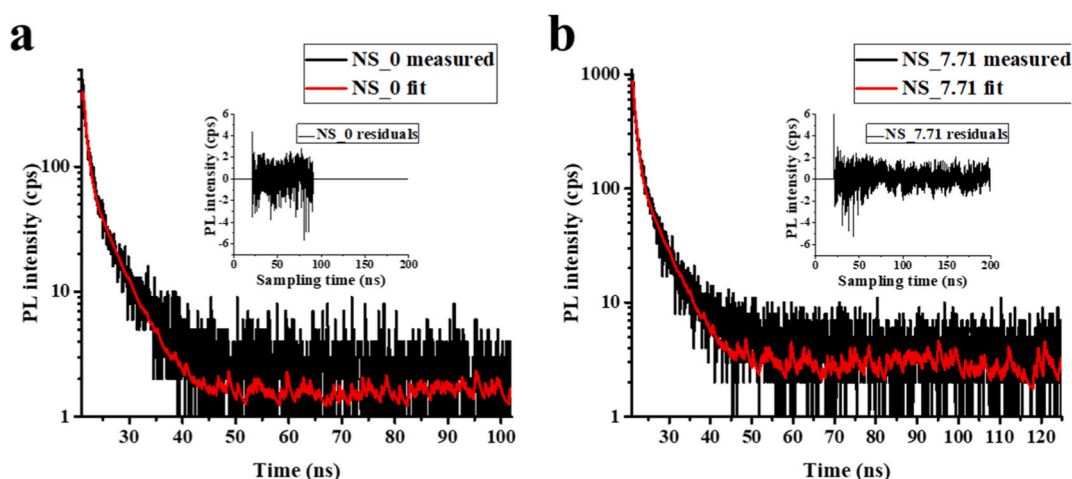


Fig. 5. Time-resolved fluorescence photoluminescence decay of NS_0 and NS_7.71 samples.

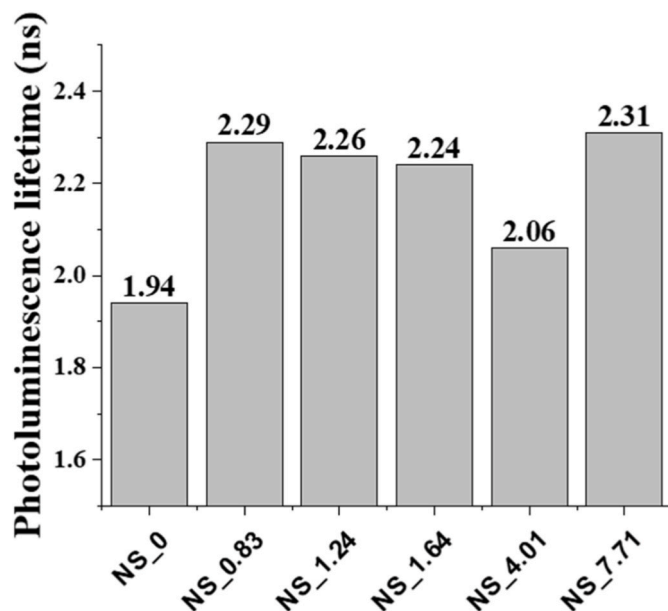


Fig. 6. The average photoluminescence lifetime of excitons in N-modified SrTiO₃ photocatalysts.

Table 2

Photocurrent density values of the N-modified SrTiO₃ samples.

Sample name	Photocurrent density (mA·cm ⁻²)
NS_0	9.42
NS_0.42	15.46
NS_0.83	23.86
NS_1.24	16.91
NS_1.64	45.67
NS_4.01	65.65
NS_7.71	72.42
NS_11.14	38.14

outstanding photocatalytic properties of TiO₂, it is not suitable for NO_x removal over intermediate and long timescales due to the aforementioned problem [40,42]. SrTiO₃-based photocatalysts are more suitable due to the higher reduction potential of the excited electron, and this effect is enhanced by the surface electron-attracting groups. Fig. 11 shows the NO_x conversion curves. In several cases, when the lamp is

switched on, the NO_x concentration increases significantly for a very short time due to a sudden jump in the NO₂ concentration. This can be explained by two factors: one is the desorption of NO_x gases adsorbed on the surface prior to measurement, and the other is the transition of some of the surface nitrogen-containing groups formed during synthesis into the gas phase.

In terms of real-world applicability, it is worth noting that the environmental NO_x concentration is two orders of magnitude lower than the standard starting NO_x concentration, so nitric acidification of the surface is not a significant factor. Behavior in the real environment is also influenced by other components in the air. For these reasons, it can be said that the current ISO standard is not perfectly suited in all respects for comparing photocatalysts in terms of NO_x conversion efficiency.

3.3.2. Photocatalytic CO₂ conversion

The photoreduction ability of the photocatalysts produced was examined through CO₂ hydrogenation. The values increased continuously with the increase in nominal N content (Fig. 12). The CO₂ conversion reached a maximum value (~30.5%) for sample NS_7.71 after 250 min. This sample stands out for the same reasons as described in the case of NO_x conversion. All modified samples showed increased CO₂ conversion compared to unmodified SrTiO₃. In addition to CO₂ conversion, we also monitored the quality of the products formed, namely CO and CH₄. Increasing the nominal N content increased the selectivity for CH₄ (from 3.9% to 6.1%). More electrons are required for CH₄ formation than for CO formation [43], and surface amine groups favor this due to their high electron density.

The presence of surface electron-rich groups was investigated using hydrochloric acid. The best-performing sample, NS_7.71, was immersed in a 0.01 mol L⁻¹ HCl solution, then washed, dried, and subjected to CO₂ conversion measurement again. After this acidic treatment, the efficiency dropped from 30.50% to 20.80%, and the CH₄ selectivity dropped from 5.81 to 3.54. Since the CH₄ selectivity was reduced to that of unmodified SrTiO₃, this indicates that efficient methanation requires many electrons, which in our case were donated to CO₂ by surface groups (mainly -NH₂). The reason CO₂ conversion was not reduced to that of unmodified SrTiO₃ is that hydrochloric acid immersion only changed the surface, not the bulk phase, so photoactivity enhancement via nitrogen doping was not compromised.

To prove the enhanced photoreductive properties of the surface, we performed a platinum detachment experiment on the modified SrTiO₃ and NS_7.71 samples. We added Pt salt to the photocatalyst suspensions and then irradiated the samples with UV-A light. In the case of the unmodified SrTiO₃ sample, no changes can be observed (either visually or

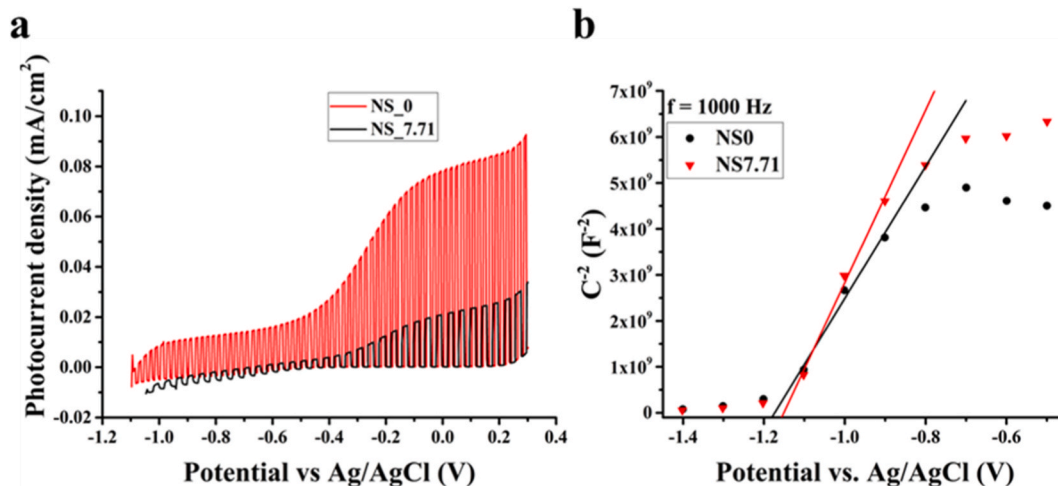


Fig. 7. Photovoltammograms for pristine and N-modified strontium titanate (NS_7.71) (a) and Mott-Schottky plots for pristine and N-modified strontium titanate at a frequency of 1 kHz (b).

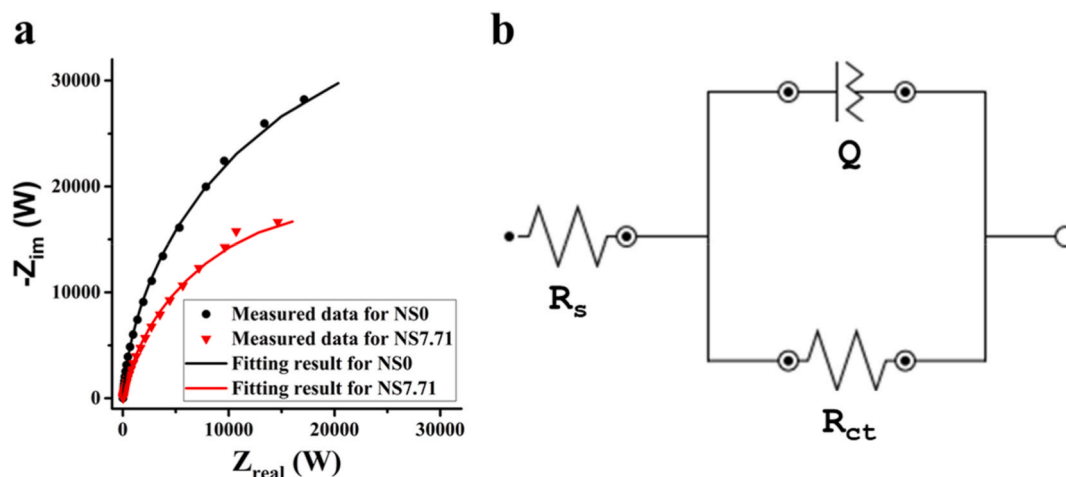


Fig. 8. Fitted Nyquist plots for pristine and N-modified strontium titanate samples recorded at OCP (a) and Equivalent circuit used to fit the electrochemical impedance spectroscopy data, where R_s represents the series resistance, R_{ct} denotes the charge transfer resistance, and Q corresponds to the constant phase element (b).

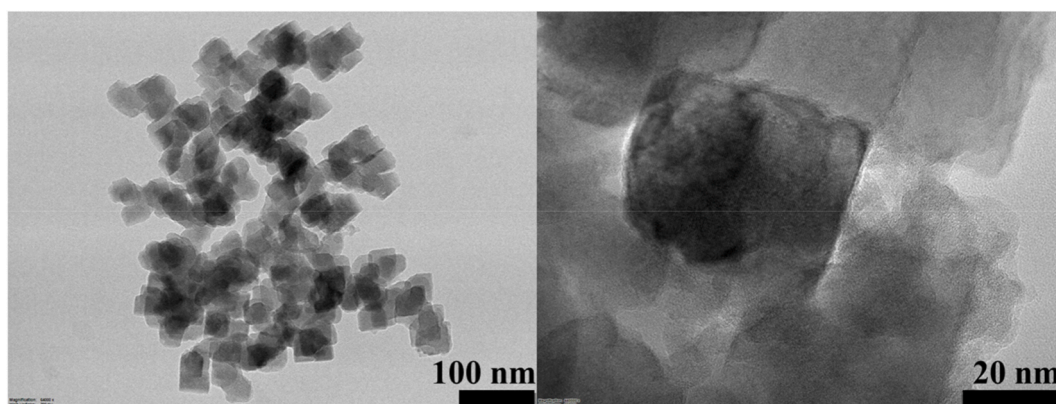


Fig. 9. Morphology of the NS_7.71 sample.

Table 3
NO_x conversion of the N-modified SrTiO₃ samples.

Sample name	NO conversion (%)	NO ₂ generation (%)	NO _x conversion (%)
NS_0	17.45	10.46	7.00
NS_0.42	19.68	11.81	7.88
NS_0.83	22.44	14.50	7.93
NS_1.24	18.30	9.43	8.88
NS_1.64	16.22	9.07	7.59
NS_4.01	22.90	12.97	9.93
NS_7.71	19.43	8.99	10.45
NS_11.14	25.24	16.12	9.12

by XRF measurements). However, the NS_7.71 suspension turned gray, and after washing and drying the sample, photoreduced platinum could be detected on the surface during the XRF measurements. The difference between the two samples is the absence/presence of electron-attracting groups on the surface (that the Pt can utilize for photoreduction). The results of the XRF measurement are shown in Fig. 13.

Our results were also compared with the photocatalytic performance of similar materials reported in the literature. Table 4 summarizes the photocatalytic efficiencies of various strontium titanates in NO conversion, along with the corresponding measurement parameters. This comparison should be considered approximate, as previous studies did not follow the ISO 22197-1:2016 standard, whereas the present work is based on it. Table 5 provides additional comparative data on CO₂

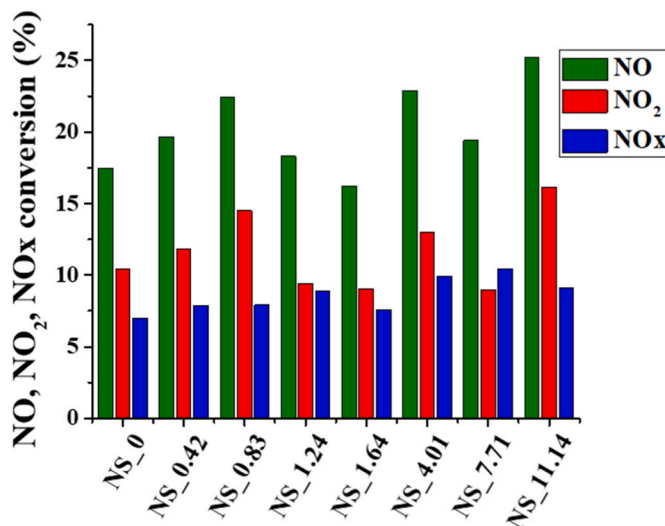


Fig. 10. NO_x conversion values on the N-modified SrTiO₃ samples.

conversion. It is important to note that all referenced studies assessed photocatalytic activity by quantifying CO formation in a flow-through reactor without recirculation, while our study evaluated the relative

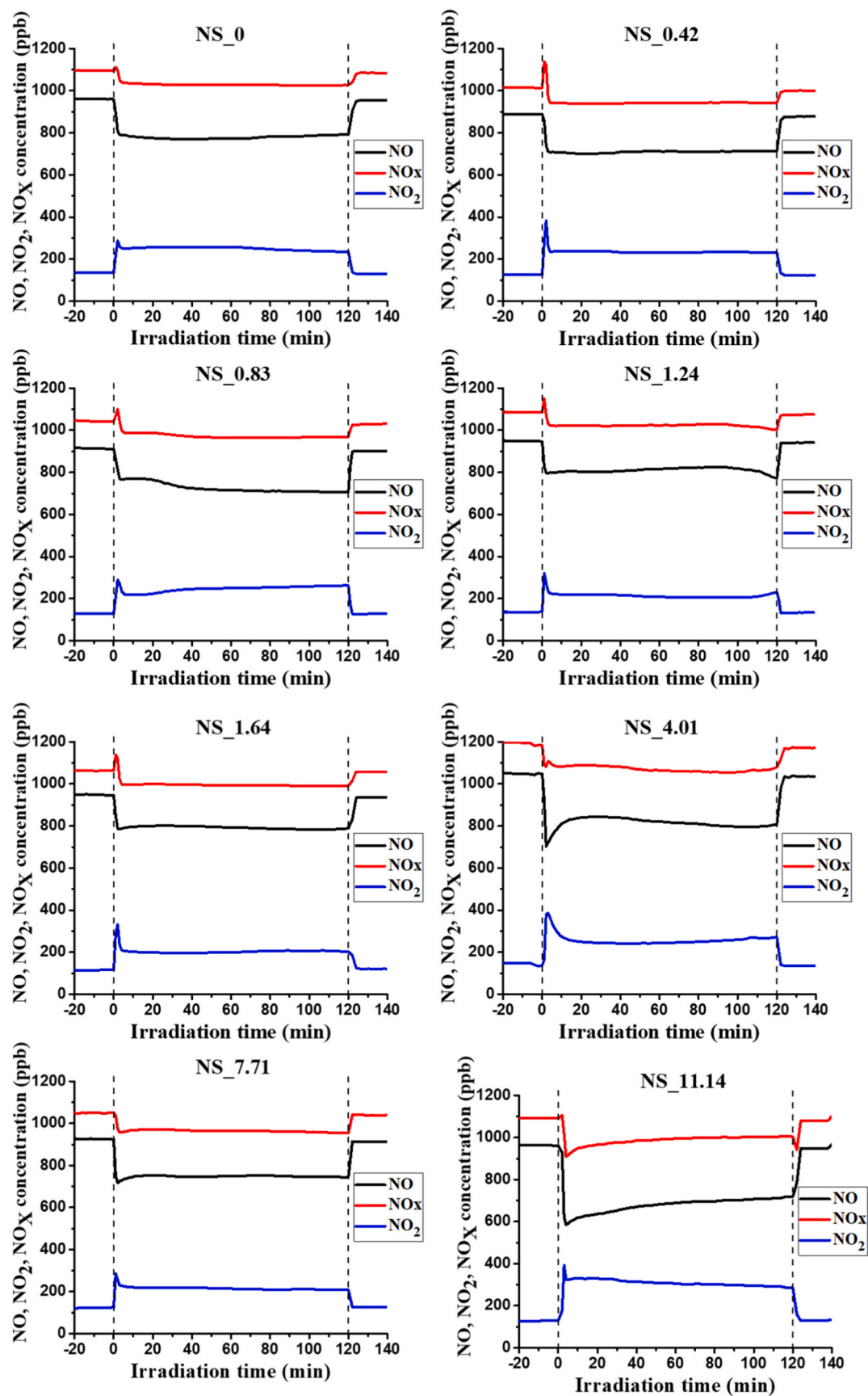


Fig. 11. The evolution of NO, NO₂, and NO_x concentrations during irradiation over the N-modified SrTiO₃ samples.

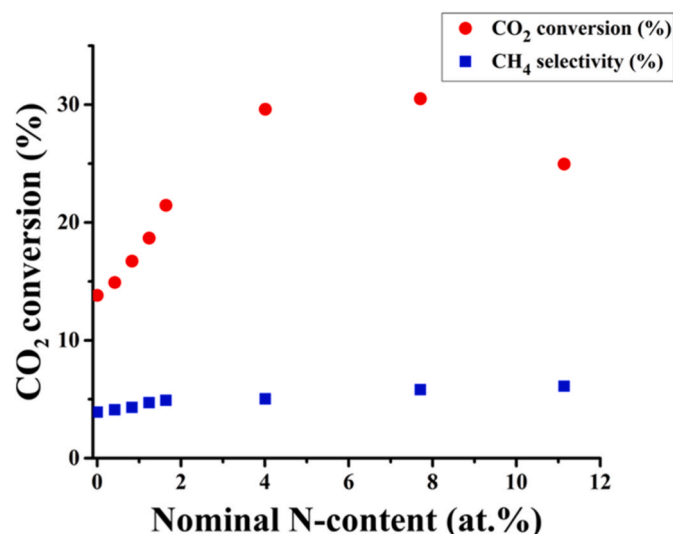


Fig. 12. CH₄ selectivity and photocatalytic conversion of CO₂ using N-modified SrTiO₃ photocatalysts.

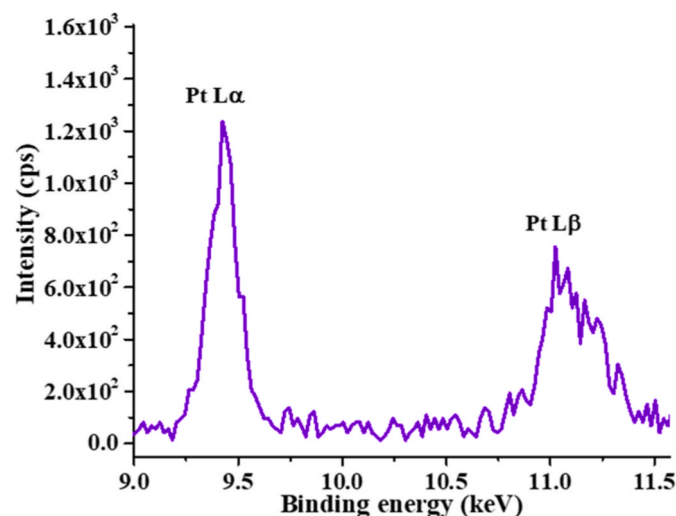


Fig. 13. Pt region of the XRF spectrum of the NS_7.71 sample after the platinum detachment experiment.

decrease in CO₂ concentration in a recirculating reactor system.

4. Conclusions

A simple synthesis method was used to modify SrTiO₃ with nitrogen in two ways in a single step: doping and forming N-containing groups on the surface. XRD and DRS measurements confirmed lattice changes (doping), while PL and photocurrent density measurements confirmed the increased lifetime of excitons. The presence of surface groups, identified by FT-IR as primarily –NO₂ and –NH₂ groups, was confirmed by measurements conducted after hydrochloric acid treatment (CO₂ conversion and XRF-Pt photoreduction). The SrTiO₃ containing nitrogen with a nominal concentration of 7.71 at.% (NS_7.71) was the sample that underwent the most extensive modification. Based on the parameters determined by various material testing methods, a local maximum was consistently observed in this sample as a function of nominal nitrogen content. These parameters include lattice strain, steady-state PL, photoluminescence lifetime, and photocurrent density. The EIS and Mott–Schottky measurements reveal clear differences between pristine SrTiO₃ and the most photocatalytically active nitrogen-modified SrTiO₃ (NS_7.71). These differences indicate that the two materials contain defect sites in distinct quantities and/or with different qualities. Such variations arise from the nitrogen modification and, in line with the photocatalytic results, contribute positively to the enhanced photocatalytic performance. These parameters include lattice strain, steady-state PL, photoluminescence lifetime, and photocurrent density. The modified samples showed increased photocatalytic activity for NO_x and CO₂ conversion. In the former case, conversion increased continuously with increasing nominal nitrogen content, reaching a maximum of 10.45% for sample NS_7.71, which is a 67% increase compared to the conversion efficiency of unmodified SrTiO₃. In the latter case, the NS_7.71 sample also exhibited the highest performance, achieving an efficiency of 30%, which is more than double the performance observed for the unmodified SrTiO₃. This increased performance is due to the electron-rich and electron-attracting groups on the surface. Regarding product selectivity, the CH₄ yield shows a steadily increasing trend with nominal nitrogen content. This result is attributed to the electron-rich surface groups, which are necessary because CH₄ formation requires more electrons than the formation of CO. Therefore, under the applied experimental conditions, our results demonstrate that the optimal nominal nitrogen content is 7.71 at.% for both NO_x and CO₂ conversion, indicating that this composition provides the most efficient photocatalytic activity observed in this study.

CRediT authorship contribution statement

Áron Ágoston: Writing – original draft, Methodology, Investigation, Conceptualization. Lilla Balassa: Writing – review & editing. Mohit Yadav: Investigation. Cintia Hajdu: Methodology. Gergő Ballai: Investigation. Tamás Gyulavári: Writing – review & editing. Karolina Solymos: Investigation. Ákos Kukovecz: Resources. Zoltán Kónya:

Table 4

Comparison of photocatalytic efficiencies of different SrTiO₃-based photocatalysts in the literature in the case of NO conversion.

Material	Light source	C _{catalyst} (mg)	Model pollutant	C _{pollutant} (ppb)	Efficiency (%)	Ref.
Ag-SrTiO ₃	LED	50 (3.3 × 11.5 cm)	NO	500 (3L·min ⁻¹)	74% (3 h)	[44]
SrTiO ₃ /SrCO ₃	Vis (Xe)	100 (φ = 80 mm)	NO	49600 (1L·min ⁻¹)	45% (0.5 h)	[2]
SrTiO ₃ /SrCO ₃	Vis (Xe)	50	NO	500	47% (0.2 h)	[45]
N-SrTiO ₃	UV-A	100 (10 × 10 cm)	NO	1000 (1.5 L min ⁻¹)	10.45% (2 h)	this work

Table 5

Comparison of photocatalytic efficiencies of different SrTiO₃-based photocatalysts in the literature in the case of CO₂ conversion.

Material	Light source	C _{catalyst} (mg)	Model pollutant	CO ₂ flow (mL·min ⁻¹)	CO yield (μmol)	Ref.
SrTiO ₃	solar simulator	10	CO ₂	1	0.02 (43 h)	[46]
Cu-SrTiO ₃	solar simulator	10	CO ₂	1	0.06 (0.4 h)	[46]
SiO ₂ /SrTiO ₃	UV-A	210	CO ₂	10	0.63 (7 h)	[47]

Resources. **Péter Sándor Lehoczky:** Writing – review & editing. **László Janovák:** Writing – review & editing, Funding acquisition. **Zsolt Pap:** Writing – review & editing, Supervision, Resources, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.ceramint.2026.03.337>.

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