Effect of Nb doping on the structural, optical, and photocatalytic properties of SrTiO$_3$ nanopowder synthesized by sol-gel auto combustion technique

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ABSTRACT
Metal oxide photocatalyst is a promising wastewater treatment process due to its simplicity, high efficiency, and low cost, as well as being environmentally friendly and non-energy consuming. The perovskite structure material, SrTi$_{1-x}$Nb$_x$O$_3$ (STNO) with $x = 0, 0.01, 0.03,$ and $0.05,$ was successfully synthesized by the sol-gel auto-combustion method. Samples with a pure phase and cubic perovskite structure were obtained. The Rietveld refinement results showed that the lattice parameter and unit cell density increased as the Nb-doping level increased. The particle size of the STNO decreases with increasing Nb doping content. Photoluminescence was excited at 327 nm, producing violet, green, and blue emissions, while the Nb content asserts an insignificant effect on the bandgap ($E_g$). The Nyquist plot and Mott-Schottky analysis were used to determine the photoelectrode performance of the STNO samples. The photocatalytic activity of STNO on the decolorization of methylene blue (MB) under UV irradiation increased with increasing Nb content and optimization at $x = 0.05$, corresponding to the results obtained from photoluminescence, $E_g$, Nyquist plot, and Mott-Schottky analyses.

1. Introduction
The increasing world population together with the associated increase in industrial and agricultural development has resulted in the growth in the amount of organic contamination in water resources. Dyes are a major contaminant that can come from a variety of sources, including textiles, paint, paper, and plastics manufacture [1]. Photocatalysis mechanisms have been widely performed to decolorize organic pollutants in water sources due to an environmentally friendly process [2]. Semiconductor photocatalysis is a promising wastewater treatment process that is easy to use, is highly efficient and low cost. Furthermore, it is environmentally friendly, non-energy consuming, nontoxic, and nonpolluting [3]. Various semiconductor-based photocatalysts, such as TiO$_2$ [4], CaTiO$_3$ [5], SrTiO$_3$ [6,7], and ZnO [8], have shown improved photocatalytic activity and chemical stability.

Perovskite structure materials (ABO$_3$) are more effective in the application of photocatalysts, sensors, dye-sensitized solar cells (DSCC), and supercapacitors [9] due to the availability of several elements that were replaced or doped in A and B-sites to fabricate the new materials. These demonstrated excellent properties of flexibility in their chemical composition, structure, band gap, oxidation states, and valence states [10]. However, to increase the photocatalytic performance of the ABO$_3$, the development of perovskite oxide-based materials for photocatalytic application has been focused on band gap tuning, suppressing the recombination of photogenerated charges, and inducing the defect in the material structure [11].

Strontium titanate (SrTiO$_3$, STO) is an oxide ceramic which has an oxide crystal with a cubic perovskite structure. STO is also a semiconductor material with a wide band gap ($E_g$) with intriguing electronic, optical, magnetic, and photocatalytic properties. The electrical properties of STO are indicated by the sizable dielectric permittivity and ferroelectric phase. Furthermore, the band structure of STO is in two band gaps: direct 3.75 eV and indirect 3.2 eV [12]. Therefore, the photocatalyst properties of STO, with these outstanding features, were fascinating to study. Nanostructure, doping, and heterojunction are modification techniques of the STO based on the photocatalyst. Nanostructures are attractive because of their high specific surface area and the migration of $e^− - h^+$ on the surface in chemical reactions that occur before recombination [13]. These properties of nanostructures improve photocatalysis efficiency. Several processes for synthesizing materials result in nanoscale structures. Using doping to tune the luminescent, electronic, optical, and other physical properties leads to improved semiconductors with a wide band gap.
Many publications have identified various methods for creating nanostructures and doping elements that result in greater photocatalytic efficiency. Enhanced photocatalytic activity is enabled by having a large number of pores and small particle sizes (60–80 nm) of STO produced in a two-step process; solvothermal and solid-state method [14]. The flux treatment method created the nanoparticle sizes (200–400 nm) in STO powders, leading to enhanced photocatalytic activity [15]. The photocatalyst of Cr-doped STO using a sol-gel hydrothermal method had a small grain size (18–32 nm) [16]. The band gap energy of Al-doped STO prepared by solid-state reaction (3.45 eV) was better than STO (3.30 eV) [17]. Besides, the 25–40 nm Au-doped STO nanoparticles synthesized via sol-gel synthesis gave enhanced photocatalytic efficiency [18]. The Cr-doped STO, prepared by the co-precipitation technique, enhanced the efficiency by ~4 times by degrading the Methylene Blue (MB) dye [19].

The Nb-doped STO, prepared by conventional solid-state reaction, demonstrated improved electrical and structural properties [20]. Au nanoparticles were decorated on 0.01 wt% Nb-doped STO and the impact on visible light utilization was assessed. This widened the band gap of the 0.01 wt% Nb-doped STO semiconductor, enhancing its usefulness for various energy conversion and environmental purposes [21]. SrTi$_3$Nb$_2$O$_9$ where x = 0, 1, 2, 3 mol%, synthesized via wet synthesis, resulted in a shift of the Fermi level [22]. Additionally, 0.1% of Nb-doped TiO$_2$ was found to be the optimum level for suppressing the surface recombination [23]. Also, Nb-doped TiO$_2$ improved the transport of electrons [24]. MB dye was also used to investigate the decolorization of STO samples under various light sources. STO, prepared by the combustion method, also degraded MB under 30 W UV light [7]. B-STO, synthesized by facile solvothermal, was used to decolorize MB in a visible light environment [25].

Different processes can prepare STO. The synthesis method and the different doping elements can affect the structure, morphology, optical, and other properties of the STO. There are few previously published works regarding the synthesizing of STO with sol-gel auto-combustion. However, this process has the advantages of a short duration of synthesis, easy stoichiometric control, high purity, homogeneous materials, and particle size at the nanoscale [26]. In past research, Nb-doped STO has also been rarely discussed in photocatalyst applications, even though Nb-doped elements have been shown to increase photoelectrode performance and electrical properties.

Sol-gel auto-combustion can be achieved with different fuels such as glycine, citric acid, ethylene glycol, and urea. The different fuels lead to different properties, such as when using urea as a fuel, smaller particles can be achieved, or where using ethylene glycol as a fuel can improve the magnetic properties of materials [27]. Cetyltrimethylammonium bromide, mixed with citric acid fuels in solution, used for combustion synthesis, led to a higher specific surface area [28]. Most publications use only one fuel to prepare a sample. For example, the individual samples of CoFe$_2$O$_4$ were prepared by sol-gel auto-combustion using different fuels: ethylene glycol, glycine, and urea [27]. Elsewhere, BaFe$_{12}$O$_{19}$ was prepared by sol-gel auto-combustion using glycine [29], and STO was prepared by sol-gel auto-combustion using citric acid [30]. However, our previous research reported a pure phase, small size as nanosized of La-doped SrTiO$_3$ and improved photocatalytic efficiency, using a mixture of fuel: glycine and citric acid [31].

Therefore, in our research, a mixture of glycine and citric acid was used as fuel to reduce ignition and improve the properties of the samples. We attempted to synthesize SrTi$_{1-x}$Nb$_x$O$_3$ (STNO) nanopowder by a new approach using sol-gel auto-combustion. The effect of various Nb-doped concentrations on the phase, structure, morphology, and optical properties of the STNO was characterized. The flat band potential of STNO is unknown, so a Mott–Schottky equation was used to determine it by the electrochemical impedance spectroscopy (EIS) technique. Charge transfer resistance and photocatalytic decolorization efficiency of MB dye under UV irradiation were further investigated, with the expectation of good photocatalytic activity.

2. Experimental

The SrTi$_{1-x}$Nb$_x$O$_3$ (STNO) samples with x = 0, 0.01, 0.03, and 0.05 were synthesized by a new synthesis route of sol-gel auto-combustion. Strontium nitrate (Sr(NO$_3$)$_2$, Sigma-Aldrich), titanium (IV) butoxide (C$_{16}$H$_{30}$O$_7$Ti, Sigma-Aldrich), niobium (V) chloride (NbCl$_5$, Sigma-Aldrich), glycine (C$_2$H$_7$NO$_2$, Ajax FineChem), citric acid monohydrate (C$_6$H$_8$O$_7$H$_2$O, Loba Chemie), hydrochloric acid (HCl, RCI Labscan Limited), ammonium nitrate (NH$_4$NO$_3$, Commercial), and ethylene glycol (C$_2$H$_4$O$_2$, Loba Chemie) were used as starting materials. Organic fuel and extra nitrates for the auto-combustion reaction were provided from glycine and citric acid mixture with a 1:2 molar ratio and ammonium nitrate, respectively.

First, stoichiometric amounts of 1 mol of Sr(NO$_3$)$_2$, 1 mol of C$_{16}$H$_{30}$O$_7$Ti, 1 mol of C$_2$H$_7$NO$_2$, 2 mol of C$_6$H$_8$O$_7$H$_2$O and 2.5 mol of NH$_4$NO$_3$ were dissolved in 100 ml of deionized water. NB solution, prepared by dissolving solid NbCl$_5$ in concentrated HCl solution, together with selected Nb mol fractions in STNO samples with x = 0, 0.01, 0.03, and 0.05, were then added to the mixture solution which was heated and constantly stirred at 80°C for 2 hr to achieve a transparent solution of homogeneous nitrate precursor.
A solution with the ratio of ethylene glycol: citric acid of 4:1 was then added to enhance the formation of metal/organic gels. The mixture solution was continuously heated and stirred until it transformed into a highly viscous gel. Suddenly, the gel was put in a furnace preheated to 300°C for self-ignited combustion synthesis. A dark brown precursor product was obtained after 1 h, which was then ground to fine particles and calcined at 1000°C for 12 h in a furnace. A white STNO powder was successfully produced.

The mechanisms for synthesizing STNO by the sol-gel auto-combustion method are as follows:

\[
\text{NbCl}_5 + 2\text{HCl} \rightarrow \text{Nb} + 7\text{Cl} + 2\text{H} \quad (1)
\]

\[
8\text{Nb} + 9\text{NH}_4\text{NO}_3 + 7\text{H}_2\text{O} \rightarrow 8\text{Nb(NO}_3)_2 + 10\text{NH}_4\text{OH} \quad (2)
\]

\[
\text{TiC}_6\text{H}_6\text{O}_4 + 3\text{H}_2\text{O} \rightarrow \text{TiO(OH)}_2 + 4\text{C}_4\text{H}_4\text{OH} \quad (3)
\]

\[
\text{TiO(OH)}_2 + 2\text{NH}_4\text{NO}_3 \rightarrow \text{TiO(NO}_3)_2 + 2\text{NH}_4\text{OH} \quad (4)
\]

\[
8\text{Sr(NO}_3)_2 + (1 - x)\text{TiO(NO}_3)_2 + x\text{Nb(NO}_3)_3 + 8\text{C}_2\text{H}_6\text{O}_7 + 8\text{C}_2\text{H}_6\text{O}_2 + (26.25 + 2x)\text{O}_2 \rightarrow 8\text{SrTi}_{1-x}\text{Nb}_x\text{O}_3 + 30\text{CO}_2 + (2.5 - 0.5x)\text{N}_2 + 34.5\text{H}_2\text{O} \quad (5)
\]

The phase identification of the STNO powders was analyzed by X-ray diffraction (XRD) with Cu-K\text{α} radiation (\(\lambda = 1.5406 \text{ Å}\)) following Bragg’s angles (2θ = 10° – 80°) at a step size of 0.02 and time/step of 0.5 s. The morphologies of the particles were inspected employing a scanning electron microscope (FESEM, JEOL JSM-6335 F) with energy-dispersive X-ray spectroscopy (EDS). A UV-visible-near infrared spectrophotometer (UHS300, HITACHI) was used to measure the absorbance spectra of the samples that have been suspended in ethanol and sonicated for 30 min, at room temperature, in the range of 300–600 nm. Nyquist plot and Mott Schottky plot were carried out with Electrochemical Impedance Spectroscopy (EIS, PGSTAT302N, Metrohm Autolab B.V.), which was performed in 0.1 M Na_2SO_4 (pH 6) electrolyte solution under a typical three-electrode condition consisting a working electrode, a platinum wire counter electrode, and a saturated calomel reference electrode. The STNO thin film was coated on fluorine-doped tin oxide (FTO) glass for use as a working electrode. A spectrophotometer (FluoroMax – 4, HORIZA) was used to measure the photoluminescence spectra of the samples at room temperature with the excitation wavelength (\(\lambda_e\)) of 327 nm.

Additionally, the photocatalytic activity of the STNO powder was evaluated by the decolorization of MB in an aqueous solution under UV irradiation with a peak wavelength (\(\lambda_p\)) at 253.7 nm (100–280 nm) using UV lamps with a net output of 108 W (18 W x 6 tubes). The aqueous solution used here had been prepared using 100 mg of STNO catalyst dispersed into 200 ml of 10 ppm MB at room temperature, continuously stirred in the dark for 30 min, to achieve adsorption/desorption equilibrium on the surface of the STNO photocatalytic. During this process, the aqueous solution sample was 40 cm away from the UV lamps. The solution was then continuously stirred under UV lamps for 8 h, with a 5 ml sample taken every hour during this period. Using a UV-Vis spectrophotometer (UHS300, HITACHI), the absorption spectra of the aqueous solution sample were recorded at a whole wavelength of 400–800 nm. To determine the effect of the with/without STNO catalyst, the aqueous solution without STNO was used as a control sample and tested under the same conditions. The decolorization efficiency (%DE) [31,32] is expressed as the following equation:

\[
\%\text{DE} = \frac{C_a - C}{C_a} \times 100
\]

where \(C_a\) is the concentration of the initial MB for the solution to reach adsorption/desorption equilibrium condition and \(C\) is the concentration of the MB after UV irradiation of the solution sample.

### 3. Result and discussion

The XRD patterns of STNO (x = 0, 0.01, 0.03, and 0.05) nanopowder after calcination at 1000°C 12 h are presented in **Figure 1a**. All samples were compared with the standard data JCPDS no. 035–0734 [33] and each showed an agreement of the main peak at 2θ = 32.40°, 40.04°, 46.50°, 57.87°, 67.94°, and 77.27° belonging to (110), (111), (200), (211), (220), and (310) planes of a cubic perovskite STO phase (Pm-3m). This indicates that all samples were identified as pure STO without any detectable impure phase, which further confirms that a cubic perovskite STO nanocrystalline structure is retained after Nb\text{5+} doping. In addition, the main peak belonging to the (110) plane (**Figure 1b**) shows a slight shift to a lower diffraction angle with increasing niobium doping content. This is due to the substitution of a larger radius of Nb\text{5+} (0.64 Å) ions to the Ti\text{4+} (0.61 Å) sites, which corresponds to Bragg’s law equation: \(nλ = 2d\sinθ\) [34]. The crystallite size of all the STNO nanopowder was calculated using the Full Width at Half Maximum (FWHM) values of the XRD patterns from Scherer’s equation (Eq. 7) [31,35] as follows:

\[
D = \frac{kl}{\beta\cosθ}
\]

where \(D\) is the average crystallite size, \(k\) is the constant 0.9, \(λ\) is the wavelength of Cu-K\text{α} radiation (1.5406 Å), \(β\) is the FWHM, and \(θ\) is the Bragg’s angle. The average crystallite size of the STNO samples calculated is shown in **Table 1**. The crystallite size of STO was around 23 nm and 21 nm for the STNO samples. The results indicated...
that there was an insignificant effect of the Nb doping content on the crystallite size of the STNO powder synthesized by the sol-gel auto-combustion method. Additionally, it was observed that the crystallite size of the STNO powders prepared in this way is relatively smaller than the crystallite size of the STO nanopowder synthesized by a modified aerosol procedure (25 nm) \cite{36}, the Sr_{0.94}Ti_{0.9}Nb_{0.1}O_{3} (80 nm) synthesized by a modified glycine-nitrate process (80 nm) \cite{37}, or the STNO (x = 0–0.03) nanopowders prepared by the wet synthesis-sol-gel method (160–240 nm) \cite{22}. Considering the effect of Nb doping on the STNO cubic perovskite structure, the specific structural parameters were calculated by Rietveld refinement analysis of the experimented XRD data using the Fullprof program. The refinement parameters for the process that were used included the scale factor, the background intensity, the lattice constant, profile half-width parameters (u, v, and w), functional position of the atoms, and occupancies, of the STNO sample. The background parameter was determined by the Chebyshev function. In addition, a pseudo-Voigt function was examined to fit the diffraction peak profiles of the samples.

Figure 2 presents the Rietveld refinement analysis results of all the STNO samples. The resultant parameters after Rietveld refinement analysis of the STNO sample and their densities are also summarized in Table 1. The peaks of the STNO samples show a good fitting with the low Chi-squared parameter (\( \chi^{2} < 4 \)) and the Rietveld discrepancy factors (\( R_{p}, R_{exp}, R_{exp} < 10\% \)). These results show that the refined XRD patterns were in good agreement with the experimental data and good consistency with the JCPDS#35-0734 database (\( a = b = c = 3.9050 \text{ Å}, d_{cal} = 5.12 \text{ g/cm}^{3} \)).

Figure 3(a) shows the lattice parameter and calculated cell density of STNO versus the Nb concentration compared with JCPDS#35-0734 of the STO database. The lattice parameter STNO with \( x = 0 \) is similar to the database and previous work: the calculation of the lattice parameter of STO (3.9050 Å) \cite{38}. In addition,
the lattice parameter value of the undoped STNO sample was lower than that of the STO value (3.920–3.925 Å), which had been prepared by the microwave-assisted hydrothermal method [35]. The result indicated that the sol-gel auto-combustion provides a lattice parameter that resembles the conventional technique. The STNO with \( x = 0.01 \)–0.05 were slightly expanded with an increase in Nb content in both volume and the lattice parameter. The lattice parameter, density, and volume of the STNO samples exhibited a slight increase when the Nb content increased due to the substitution of Nb\(^{5+}\) (0.64 Å) ions to Ti\(^{4+}\) (0.61 Å) sites. This is in agreement with previous research on the lattice parameter and volume of Nb-doped SrTiO\(_3\) [20,34]. It was also found that the lattice parameter of STNO with \( x = 0.01, 0.03, \) and 0.05 is similar to the lattice parameter and the same trends of Nb-doped STO (3.905–3.918 Å) prepared by the colloidal synthetic process [39] and the (Nb, Zn) co-doped STO ceramics (3.9064–3.9069 Å) prepared by the traditional solid-state technique [33]. Figure 3b shows the crystal structure of STNO with \( x = 0.05 \) calculated by Rietveld refinement analysis, which substituted the Nb\(^{5+}\) at the Ti\(^{4+}\) site, with occupancy numbers of Nb\(^{5+}\) and Ti\(^{4+}\) to be 0.0501 and 0.9570, respectively. The result indicated that the sol-gel auto-combustion provides a lattice parameter that resembles the conventional technique.

The SEM images of the STNO (\( x = 0, 0.01, 0.03, \) and 0.05) powders are shown in Figure 4. The morphology of both the undoped and the Nb-doped STNO samples presented as agglomerated particles and indeterminate shapes due to the adhesion of particles to each other by weak forces [40]. It was observed that all the samples exhibited smooth and dense surfaces with no porosity of the particles. The agglomeration of particles in the STNO sample showed more particles sticking together when the amount of Nb doping increased. The ImageJ program was used to determine the average particle size of all the samples, which measured 200 particles/sample. The average particle size of the undoped STNO was 235 nm, while the Nb-doped STNO samples with \( x = 0.01, 0.03, \) and 0.05 had an average particle size of 128 nm, 105 nm, and 84 nm, respectively. This indicates that the average particle size of STNO samples slightly decreased with the increase in the Nb content. These results showed that the STO nanopowders were closer and lower than others reported as being prepared by various techniques such as the flux treatment method (200–400 nm).
The STNO results are also closer and lower than others reported in the literature, prepared by various techniques, such as the Sr_{0.94}Ti_{0.9}Nb_{0.1}O_3 powder synthesized by a modified glycine-nitrate process (100 nm) [37] and the SrTiO_{3.95}Nb_{0.05}O_3 powders prepared by ball milling (1 µm) [41].

The SEM image, EDS spectrum, and mapping images of the STNO nanopowder with x = 0.05 are shown in Figure 5. In the EDS spectrum Figure 5(b), the signals corresponding to Sr, Ti, Nb, and O are exhibited in Figure 5(c-f). It is observed that elements Sr, Ti, Nb, and O are uniformly distributed in the STNO nanopowder. The quantitative EDS analysis confirmed that the atomic ratio of Sr, Ti, Nb, and O in STNO nanopowder with x = 0.05 is 1:0.85:0.31:3.43. Although the actual chemical composition was examined by EDS analysis, it was inconsistent with the nominal composition due to there being low concentrations of Nb doping, no flat surface in the selected area allowing accurate mapping analysis and measurement (different working distance

![Figure 3](image-url) Figure 3. (a) The lattice parameter and calculated cell density (d_{cal}) of STNO nanopowder and (b) the crystal structure of STNO with x = 0.05 from Rietveld refinement analysis compared with SrTiO_3 database.

![Figure 4](image-url) Figure 4. SEM image of STNO nanopowder: (a) x = 0, (b) x = 0.01, (c) x = 0.03, and (d) x = 0.05.
of the electron beam) and the accuracy of the EDS spectrum was within ±5% relative to the actual value [42].

The optical properties of the STNO samples were investigated by UV-Visible spectra absorbance with $A = -\log(I/I_0)$, with $I_0$ (intensity of incidence) and $I$ (transmitted radiation) being recorded at wavelengths between 300–600 nm at room temperature (Figure 6). In a general sense, the spectra absorbance was used to observe the absorption edge and energy hole by following the movement of the electrons from the upper bound of the valence band to the lower bound of the conduction band [43]. The absorption capacity of all the samples showed a comparable trend at the absorption edge at 311 nm wavelength. In the high energy region of the absorption spectrum, the ability of light absorption linearly increased when the photon energy increased. The relationship of the absorption and photon energy ($h\nu$) is represented by Tauc’s relation [31,44];

$$\alpha h\nu = B(h\nu - E_g)^n$$  \hspace{1cm} (8)

where $\alpha$ denotes the absorption coefficient, $B$ is a constant, $E_g$ is the band gap, and $n$ depends on the type of transition. For the directly allowed transitions, $n= 1/2$, for the indirectly allowed transitions, $n = 2$, for direct forbidden transitions, $n = 3/2$, and indirect forbidden transitions, $n = 3$ [43,45]. The $E_g$ of these samples was investigated by extrapolating the linear portion of the curve to zero absorbance, shown in Figure 6 as the inset plot of $(\alpha h\nu)^2$ versus $h\nu$ for a direct allowed transition. It was found that the $E_g$ of all the STNO samples is 4.11 eV.

The optical properties of nanopowder depend on their crystallinity, particle size, and synthesis method, such as when the $E_g$ increases with the decreasing particle size. For the undoped STNO samples, the $E_g$ in this study was higher than reported in another research. For example, the $E_g$ of the STO samples prepared by the microwave-assisted hydrothermal method were 3.5 ±0.1 eV [35], and for the STO powders prepared by the liquid-solid reaction method, the $E_g$ was 3.4–3.5 eV [46], and when the STO were prepared by high-temperature solid-state reaction, the direct $E_g$ was 3.55–3.67 eV [47]. In addition, the STO samples that had a cubic structure showed a higher $E_g$ than STO prepared by the topotactic, with rodlike and cubic samples exhibiting $E_g$ of 3.0 and 3.04 eV [48]. On the other hand, for Nb-doped STNO samples, the doping usually affects the $E_g$ value, such as the $E_g$ of Al-doped STO (3.45 eV) being better than the undoped STO (3.30 eV), where both of them were prepared by solid-state reaction [17]. Unfortunately, in our research, the

Figure 5. SEM-EDS images and spectrum of the STNO nanopowder with $x = 0.05$.

Figure 6. UV-vis spectra of the STNO samples ($x = 0, 0.01, 0.03$ and $0.05$) and inset plots of $(\alpha h\nu)^2$ as a function of photon energy ($h\nu$).
$E_g$ of the STNO was not changed at varying concentrations of Nb-doped. This may have been due to the low concentration of Nb-doped that we used.

Generally, the decolorization of dye is used to determine the effectiveness of a photocatalytic reaction. The decolorization efficiency (%DE) follows Eq. 6. Figure 7 (a) presents the %DE of MB by STNO with $x = 0, 0.01, 0.03$, and $0.05$ as the photocatalyst under a UV light. The photolysis tests showed a slight change in the concentration of MB of not more than 10%, which is within the acceptable value range. The photocatalytic activities of STNO with $x = 0, 0.01, 0.03$, and $0.05$ increased with higher Nb content. The %DE of MB for all STNO increased hourly until 8 h; the %DE using STNO with $x = 0$ and $0.01$ was similar at around 20%, while $x = 0.03$ and $0.05$ were similar at around 50%. The factors affecting photocatalytic efficiency are crystallinity, crystal structure, and particle size of the material [49]. The results indicated that the concentration of Nb dopant leads to increased photocatalytic activity of the Nb-doped STNO due to the smallest particle size providing a larger active reaction site and empowering the adsorption of more MB on the STNO photocatalyst surface, indicating that photocatalytic efficiency reached a maximum at the highest level of Nb, $x = 0.05$.

The rate of reaction can be specified by chemical kinetics. The first-order reaction kinetics constant ($k$) is determined using the equation: $(kt = \ln(C_0/C))$ [44], where $C$ and $C_0$ are the real-time and initial concentrations of MB. The $k$ value for STNO also increased with Nb doping content increases, as shown in Figure 7(b). Obviously, for STNO with $x = 0$ and $0.01$, the $k$ value is similar at around $0.033-0.034$. With $x = 0.03$ and $0.05$ the $k$ values are similar at $0.076-0.079$, according to the %DE shown in Figure 7(a). The great value for Nb-doped STNO as a photocatalysis reaction was in the STNO sample with $x = 0.05$, as shown in Figure 7(c). The absorption peak ($\lambda_{\text{max}}$) of MB was at 663 nm. After 8 h, the $\lambda_{\text{max}}$ of MB continuously decreased and shifted to the lower wavelength from 663 nm to 656 nm. The blue shift is due to the stepwise removal of methyl or methyamine, and the decolorization of the MB, which results in

![Image of Figure 7](image-url)

**Figure 7.** (a) The photocatalytic decolorization efficiency (%DE) of MB irradiated beneath the UV lamp, (b) The Pseudo-first order kinetics of photocatalytic decolorization of MB, (c) Time-dependent absorption spectra of MB solution using STNO photocatalyst with $x = 0.05$ and (d) photoluminescence spectra of the STNO sample with $x = 0$ and $0.05$. 
the $\lambda_{\text{max}}$ of the MB decreasing [32]. For all STNO samples, the spectra absorbance follows the same trends.

Photoluminescence is widely used to characterize the optical properties of semiconductors. The electrons of the semiconductor substance are stimulated by the photon absorbance, where its electrons move to a higher energy state from a lower energy state, after which there is a relaxation phase during which photons are again emitted or released. The interval between the absorption and emission phases of the photons may vary depending on the substance. Most UV light absorbers emit light in the visible range so that colors can be seen in these substances. The photoluminescence of all our STNO samples, after excitation at 327 nm in the range of 350–600 nm at room temperature, is shown in Figure 7(d). The emission peaks of all the samples are similar trends, 397, 438, 450, 466, 479, 491, and 562 nm, indicated in violet, blue, and green. The high-intensity emission band at 397 nm indicates high electron-hole recombination [50]. The peak emission at 395 nm indicates that the violet colors are due to shallow surface defects [35]. Also, peak emissions at 438, 450, 466, 479, and 491 nm are blue due to Sr deficiency in the crystal structure, and the peaks emitted at 562 nm, showing as green emissions, are caused by oxygen vacancy in the STO lattice, according to the previous study [51]. Moreover, the photoluminescence emission intensity related to the recombination of charge carriers in a semiconductor can be used to explain the photocatalytic activity of the photocatalyst. From Figure 7(d), the photoluminescence emission intensity of Nb-doped STNO nanopowder is lower than the undoped STNO sample ($x = 0$), indicating lower electron-hole pair recombination and photocatalytic reaction enhancement. Remarkably, the result of the photoluminescence emission intensity of the Nb-doped STNO with $x = 0$ and 0.01 are nearly closed, while $x = 0.03$ and 0.05 are closed together with lower than those of $x = 0$ and 0.01. It is indicated that the photoluminescence emission intensity of the Nb-doped STNO corresponds to the photocatalytic efficiency of the sample, as shown in Figure 7(a).

The Nyquist plot from the Electrochemical Impedance Spectroscopy (EIS) is used to analyze the charge carrier migration of photoelectrode in a three-electrode system. The semicircle accords with the charge transfer resistance at the photoelectrode interface $R_\Omega$, which is the solution resistance. This is estimated by the $x$–intercept of the Nyquist plot and describes the overall resistance between the photoelectrode and the electrolyte. Figure 8 shows a Nyquist plot of all the STNO samples, using an Autolab PGSTAT302N at an applied potential of 1 $V_{\text{ref}}$ at pH 6 (Na$_2$SO$_4$ electrolyte 0.1 M) and the frequency range from 1–1000 Hz and amplitude of 0.01. The experimental data are compared with the simulation data by fitting the experimental data by the equivalent circuit, as shown in the inset of Figure 8. The simulations were performed, with the impedance values being 5260.82 $\Omega$ for $x = 0$, 4827.40 $\Omega$ for $x = 0.01$, 4320.14 $\Omega$ for $x = 0.03$, and 1712.73 $\Omega$ for $x = 0.05$. The equivalent circuit of all STNO samples is shown in the inset of Figure 8. The smaller semicircle in the Nyquist plots is for increases in the Nb content, suggesting that the highest level of effective charge transfer efficiency occurs at higher Nb content but not at low Nb content or without doping [52].

The Mott–Schottky plots of STNO samples were carried out with the EIS that measures an electrochemical interface as its DC voltage changes and with a fixed frequency. The graph can be plotted by the voltage and capacitance and are used to characterize the flat band potential and understand the charge transport in STNO. So, to consider the donor density ($N_D$) and the flat band potential ($V_{\text{fb}}$) of STNO photoelectrode, a Mott–Schottky analysis was performed. Depending on the depletion layer model, the capacitance of the semiconductor space charge layer ($C_{SC}$) is based on the applied potential ($V_{\text{appl}}$). The Mott–Schottky equation is expressed in equation 9 [39]:

$$\frac{1}{C^2} = \frac{2}{\varepsilon_0 \varepsilon_r N_D} (V_{\text{appl}} - V_{\text{fb}} - k_0 T / \varepsilon)$$

where $\varepsilon$ is the charge of the electron, $\varepsilon_r$ is the semiconductor dielectric constant, $\varepsilon_0$ is the vacuum permittivity, $T$ is the absolute temperature, and $k_0$ is the Boltzmann constant $(1.38 \times 10^{-23} \text{J/K})$.

Figure 9 shows the Mott–Schottky plot of the STNO samples from the EIS spectroscopy analysis (Autolab PGSTAT302N) by applying the potential range of 1.2–2.0 V at pH 6 (Na$_2$SO$_4$ electrolyte 0.1 M) at a 1000 Hz frequency. The Mott–Schottky curves for all samples demonstrated a positive slope, indicating their n-type character similar to that reported in the literature [39,53]. The flat band potentials ($V_{\text{fb}}$) determined
from the x-intercepts of the curves are −0.10, −0.18, -0.25, and –0.30 V for STNO samples with x = 0, 0.01, 0.03, and 0.05, respectively. The applied potential in NHE scale (E_{NHE}) was calculated by the following equation:

\[ E_{NHE} = E_{Ag/AgCl} + 0.21 \]

(10)

Accordingly, the V_{fb} values of STNO samples with x = 0, 0.01, 0.03, and 0.05 are 0.11, 0.03, −0.04 and −0.11 (vs. NHE), respectively. It was found that the V_{fb} values for STO and STNO samples were shifted toward negative value when Nb content increased, favored its better photocatalytic activity. As a rule of thumb, a conduction band potential (E_{cb}) of n-type semiconductor is more negative by −0.1 eV than E_{fb} level [54]. Therefore, E_{cb} of STNO samples with x = 0, 0.01, 0.03, and 0.05 could be determined as 0.01, −0.07, −0.14, and −0.21 (vs.NHE), respectively. For the valence band potentials (E_{vb}) of the STNO samples are a result from combining the E_{cb} with the E_{y} values. Thus, the E_{vb} of the STNO samples are 4.12 V (vs.NHE) for x = 0, 4.04 V (vs.NHE) for x = 0.01, 3.97 V (vs.NHE) for x = 0.03, and 3.90 V (vs.NHE) for x = 0.05. This result indicates in Figure 10 cooperate with the mechanism of photocatalysis process.

Thereby, the E_{vb} of the STNO samples are 0.01 V (vs. NHE) for x = 0, −0.07 V (vs.NHE) for x = 0.01, −0.14 V (vs. NHE) for x = 0.03, and −0.21 V (vs.NHE) for x = 0.05. The mechanism of photocatalytic decolorization of MB dye on STNO photocatalyst under UV irradiation is shown in Figure 10. The process of photocatalysis reaction can be partitioned into three steps. First, the photocatalysis of STNO is energized by UV light with photo-energy higher than the E_{g} of the photocatalysis to create photoinduced electrons (e^-) and gaps (h^+). The electron is produced when electrons from the valence band (VB) move to the conduction band (CB), during which process gaps within the valence band are produced (Eq. 11). Next, the e^- and h^+ respond with O_2 and H_2O or (OH^-) to produce highly receptive hydroxyl radicals (OH) and superoxide radicals (O_2^-). The H_2O or OH^- that are adsorbed on the surface of the photocatalysis are oxidized by the h^+ to create OH^- (Eq.12). The O_2 accompanying the adsorption on the surface by the photocatalysis reacts with e^- to generate the O_2^- (Eq.13). In a further step, O_2^- reacts with H_2O to

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**Figure 9.** A Mott-Schottky plot of STNO nanopowder.

**Figure 10.** The charge transfer and photocatalytic mechanism of STNO nanopowder.
produce hydroperoxyl radical (HO$_2^-$) (Eq.14). Finally, these hydroxyl radicals, superoxide radicals, and hydroperoxyl radicals decompose the MB dye into CO$_2$, H$_2$O and degraded products of MB, which could be 2-amino-5-(NN-methylformamide) benzene sulfonic acid, 2-amino-5-(methylamino)-hydroxybenzene sulfonic acid, and benzenesulfonic acid [55] (Eq.15). This entire process can be summarized in the following equations:

\[
\text{STNO} + \text{hv} \rightarrow e^- + h^+ \quad (11)
\]

\[
\text{H}_2\text{O}/\text{OH}^- + h^+ \rightarrow \text{OH}^+ \quad (12)
\]

\[
\text{O}_2 + e^- \rightarrow \text{O}_2^- \quad (13)
\]

\[
\text{O}_2^- + \text{H}_2\text{O} \rightarrow \text{HO}_2^- \quad (14)
\]

\[
\text{OH}^- /\text{HO}_2^- /\text{O}_2^- + \text{MB} \rightarrow \text{degraded products} + \text{CO}_2 + \text{H}_2\text{O} \quad (15)
\]

Based on the above mechanisms, there are three reasons for the occurrence of the outstanding photocatalytic activity of the STNO samples. Firstly, the particle size of the Nb-doped STNO samples prepared by the sol-gel auto-combustion method provides a nanosized particle, and the particle size decreases with increasing Nb doping content. Secondly, the smaller semicircle of the Nyquist plots Nb concentration increases, suggesting a higher electron transfer conductivity. Finally, the $E_{cb}$ calculated from the Mott-Schottky equation of the STNO samples is more negative than the reduction potential of O$_2$/$\text{O}_2^- (-0.046 \text{ V vs. NHE})$ [54], and the $E_{vb}$ of the STNO samples are more positive than the reduction potential of OH$^-$/OH$^-$ (+2.40 V vs. NHE) [54], so has a good oxidation ability to degrade the organic pollutants [56]. Therefore, the Nb-doped STNO sample with $x = 0.05$ has the smallest particle size, the lowest semicircle of the Nyquist plots, and the lowest $E_{cb}$ and $E_{vb}$ calculated from the Mott-Schottky equation. These results for the STNO sample with $x = 0.05$ indicate the highest %DE of photocatalytic activity.

Table 2 illustrates the decolorization of MB in an aqueous solution under a UV source as identified in previous research [6,7]. When comparing our results with that previous research, it was found that the photocatalytic activity of undoped STNO was lower than STO. This was due to the larger particle sizes and the undoped STNO having no porous structures. Comparing the photodecolorization efficiency of MB under UV-visible irradiation with several other methods [25,32,49], the photocatalytic activity of undoped STNO was less than STO synthesized by those methods (also illustrated in Table 2).

The photocatalytic activity of metal-doped STNO (Table 2) could not be directly compared to our research due to different conditions of the photocatalytic system such as light source intensity, amount of photocatalytic loading, and the concentration of dye. However, no report on the photodecolorization efficiency of MB using Nb-doped STNO as photocatalysts under UV irradiation has been found. In our study, the Nb content was significantly enhanced the photocatalytic activities of STNO nanopowder.

### Table 2. The summary of the photodecolorization efficiency (%DE) of several dyes using undoped-STO and metal-doped STO photocatalysts with different preparation methods, light sources, and photocatalytic conditions.

| Photocatalyst | Preparation method | Light sources | Types of dye | Photocatalyst Loading | Initial Concentration | Reaction Time | %DE | Ref. |
|---------------|--------------------|---------------|--------------|----------------------|----------------------|--------------|------|------|
| SrTiO$_3$     | Polyacrylamide gel route | 20 W Mercury lamp (UV) | CR | 1000 mg/L | 10 ppm | 10 h | 90% | [6]  |
| SrTiO$_3$     | Combustion | 30 W UV | CR | 200 mg/L | 31.98 ppm | 70 min | 65% | [7]  |
| SrTiO$_3$     | Facile solvothermal | Visible light | MO | 500 mg/L | 10 ppm | 120 min | 62.7% | [25] |
| SrTiO$_3$     | Amino acids as dopants and surface area promoters | UV-Visible irradiation | MB | 200 mg/L | 5 ppm | 180 min | 100% | [32] |
| STO-P STO-20  | Ball milled for different time | 125 W UV-Visible irradiation | MB | 1000 mg/L | 3.19 ppm | 210 min | 100% | [49] |
| La-doped SrTiO$_3$ (La = 0.5) | Sol-gel | 500 W Xe arc lamp | K$_2$Cr$_2$O$_7$ (Cr) | 1000 mg/L | 20 ppm | 100 min | 84% | [57] |
| La-doped SrTiO$_3$ (La,Cr)-6 | Sol-gel hydrothermal | 300 W Xe lamp | Tetracycline (TC) | 500 mg/L | 20 ppm | 90 min | 80% | [58] |
| V-doped SrTiO$_3$ | Electrospinning and thermal diffusion | 175 W mercury lamp | MO | 300 mg/L | 10 ppm | 75 min | 90% | [59] |
| La-Fe co-doped SrTiO$_3$ | Solid state reaction | Visible light | MO | 600 mg/L | 5 ppm | 150 min | 90% | [60] |
| Nb-doped SrTiO$_3$ (Nb = 0.05) | Solid-gel auto combustion | 108 W UV | MO | 500 mg/L | 10 ppm | 8 h | 39% | [This study] |
4. Conclusion
SrTi$_2$NbO$_9$ (STNO) nanopowder with $x=0, 0.01, 0.03$, and 0.05 was successfully synthesized by the sol-gel auto combustion technique with two fuel mixtures. The STNO nanopowder exhibited a pure phase with cubic perovskite structures. The increased Nb doping content increased the lattice parameter. Also, the unit cell density increased while the particle size decreased with a non-porous structure. The electron-hole pair recombination and charge transfer resistance on the surface of the STNO photoelectrode materials decreased with increased Nb doping content. These factors promoted the transportation of charge carriers, leading to improved photocolorization efficiency. Therefore, STNO nanopowder with $x = 0.05$ is the optimal level for photocatalytic decolorization of MB under UV irradiation. These findings demonstrate that the structural, optical, and electrochemical properties of STNO nanopowder synthesized by the sol-gel auto-combustion technique enhance photocatalytic activity.

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No potential conflict of interest was reported by the author(s).

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