

Article

Room-Temperature Catalytic and Photocatalytic Oxidation of CO over Pt/TiO₂ Catalysts in an LED-Based Flow Reactor: Effects of Pt Loading and Oxidation State

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Abstract

The build-up of carbon monoxide (CO) in confined spaces can pose serious risks to human health. One method of mitigating CO in air is catalytic oxidation. Low-temperature catalytic oxidation of CO is desirable to enhance energy efficiency and sustainability. Noble metal catalysts have been shown to be active catalysts for CO oxidation at low temperatures. In addition, photocatalysis has been demonstrated as a method to degrade harmful gas-phase compounds into less toxic ones at room temperature. In this study, the effects of platinum (Pt) loading and oxidation state on a TiO₂ (P25) support were investigated in a light-emitting diode (LED)-based flow reactor for the room-temperature catalytic and photocatalytic oxidation of CO in air. It was found that the catalytic activity increased with Pt loading up to 5 wt%, and catalytic activity was significantly higher when the Pt was reduced (Pt⁰) on the catalyst compared to oxidized platinum (Pt²⁺, Pt⁴⁺). Visible light absorption increased with increasing platinum loading on TiO₂, and the catalyst activity for room-temperature CO oxidation increased with exposure to both ultraviolet (UVA) and visible light. Platinum is responsible for the adsorption of CO and the activation of adsorbed oxygen and CO, and it interacts with the photogenerated electrons upon exposure to light to enhance the photocatalytic activity of Pt/TiO₂ catalysts for the room-temperature oxidation of CO.

Keywords: Pt/TiO₂; room-temperature carbon monoxide (CO) oxidation; LED photocatalytic reactor; Pt loading; Pt oxidation state; photocatalysis; visible light photocatalysis



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1. Introduction

Carbon monoxide (CO) oxidation to carbon dioxide (CO₂) is a well-studied reaction due to its importance in commercial applications, such as the prevention and remediation of CO air emissions from the incomplete combustion of fossil fuels [1–10]. This reaction also has important applications for the safety of people who may be exposed to indoor air pollution, who work in confined spaces, or who are passengers in vehicles that have limited exchange with fresh air (e.g., submarines and other military vehicles, spacecraft) [7,11–14]. CO is a highly toxic gas that binds with hemoglobin in blood, and so it inhibits the transport of oxygen to various parts of the body [15,16]. Therefore, in confined spaces, processes

must be in place to oxidize CO such that the continuous human exposure is less than 9 ppm [13,17].

The oxidation of CO is accomplished on commercial-scale using heterogenous catalysis, where the catalysts are often noble metals supported on metal oxides [1–10]. For example, a common three-way catalytic converter on an automobile contains platinum (Pt), palladium (Pd), and rhodium (Rh) supported on aluminum oxide, which simultaneously removes CO, volatile organic compounds (VOCs), and nitrogen oxides (NO_x) from automobile exhaust [18,19]. However, there are challenges with noble metal catalysts, including their relatively high cost [3], their tendency to lose activity upon exposure to CO and sulfur compounds [1,20], and their tendency to sinter [3]. Therefore, there is a desire to develop low-temperature and sustainable methods for removing CO from gas streams.

Pt/ TiO_2 catalysts are very attractive as catalysts for low-temperature CO oxidation [6,7,15,20–25]. The mechanism of low-temperature CO oxidation over Pt-based catalysts have been investigated, but consensus among researchers has not been achieved [1]. The mechanism of CO oxidation appears to depend on whether the reactants are dry or humid [1,2], as well as on the distribution, size, and oxidation state of Pt particles [1,2]. For single-atom catalysts, Beniya and Higashi (2019) state that CO oxidation can be roughly described by Mars van Krevelen or Langmuir–Hinshelwood reaction models [2]. Briefly, in the Mars van Krevelen model, lattice oxygen is used in the reaction with CO to form CO_2 , creating oxygen vacancies. At these vacancies, gas-phase oxygen is adsorbed and activated, promoting a reaction with adsorbed CO molecules at the Pt site to form CO_2 . The Langmuir–Hinshelwood model surmises that both CO and oxygen are adsorbed at the active sites. Oxygen dissociates and reacts with adsorbed CO to form CO_2 . Most researchers agree that the role of Pt on catalysts is to enhance the adsorption and activation of CO and oxygen species on the catalyst [3].

The photocatalytic oxidation of CO over Pt/ TiO_2 catalysts has also been studied [5,12,26–36]. In photocatalysis, light energy activates the catalyst, so the process can occur at room temperature. Therefore, photocatalysis has potential to be a sustainable and efficient method of oxidizing CO at low temperatures and in confined spaces. The mechanism for photocatalysis of CO on Pt/ TiO_2 catalysts has been researched [5,12,35,37,38] and can be described pictorially in Figure 1.

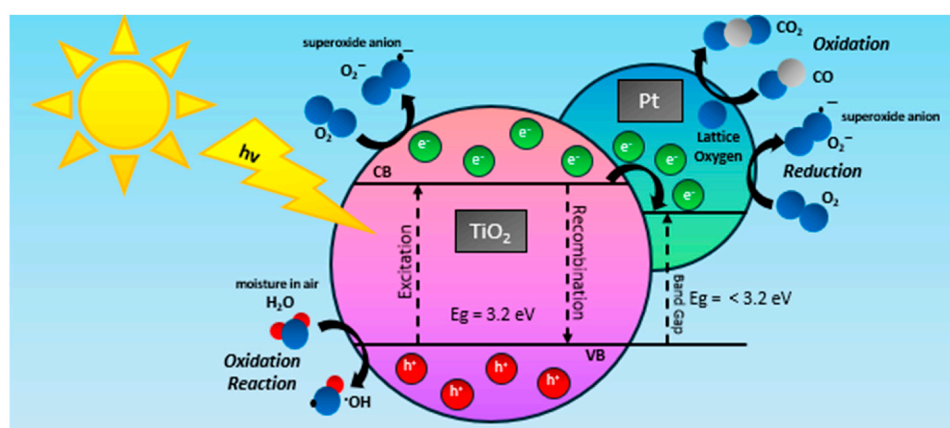


Figure 1. Schematic of Pt/ TiO_2 mechanism in the photocatalytic oxidation of CO.

Briefly, the absorption of light ($h\nu$), at sufficient energy to overcome the bandgap of the photocatalyst, will generate an electron–hole pair. The electron, e^- , jumps to the conduction band (CB), whereas the hole, h^+ , is left in the valence band (VB) of the electronic structure of the photocatalyst. These charges migrate to the surface of the photocatalyst where they undergo redox reactions. The hole, h^+ , will oxidize adsorbed water or a hydroxyl

group at the surface, forming a hydroxyl radical, as shown in Figure 1. The electron (e^-) will interact with oxygen at the surface of the photocatalyst, forming reactive oxygen species, such as a superoxide anion (O_2^-). The recombination of electrons and holes accounts for the inefficiency in the photocatalysis process [5,12].

The photocatalysis mechanism can be improved by incorporating noble metals (e.g., Pt) onto the catalyst surface. Following the reaction described above, the role of Pt on the catalyst is to accept photogenerated electrons and delay electron–hole recombination. Pt also facilitates the adsorption of oxygen and CO and the formation of reactive oxygen species [12,35], thus lowering the energy barrier for CO oxidation.

While the effects of Pt loading and Pt oxidation state have been investigated for CO oxidation previously, there are few studies in which Pt loading and oxidation state are systematically characterized and investigated for both catalytic and photocatalytic activity for CO oxidation at ambient temperatures. This study investigates the role of Pt loading (up to 5 wt%) and oxidation state for the room-temperature catalytic and photocatalytic oxidation of CO in air under visible and ultraviolet (UV) light-emitting diode (LED) light sources in a flow reactor. In addition, the effects of residence time, CO concentration, and humidity were investigated.

2. Results

2.1. Photocatalyst Characterization

The catalysts were synthesized with Pt loadings up to 5 wt% on TiO_2 and were calcined in air. These catalysts are designated as X.XPt/ TiO_2 , where X.X is the nominal wt% Pt. Aliquots of these catalysts were reduced with hydrogen at elevated temperature, resulting in metallic Pt (Pt^0) nanoparticles on the catalysts. The reduced catalysts are designated as X.XPt(R)/ TiO_2 .

The catalysts were characterized for Pt loading and oxidation state, morphology, surface area, and crystallinity. Their performances for room-temperature catalytic and photocatalytic oxidation of CO were investigated. The results are provided below.

2.1.1. Catalyst Morphology and Pt Loading

The morphology of the catalysts was investigated using scanning electron microscopy (SEM). Figure 2a,b compare the SEM micrographs of pure TiO_2 and 5.0Pt(R)/ TiO_2 . The morphologies of the catalysts are similar and appear to consist of small crystalline spheres of TiO_2 . The SEM results did not show noticeable differences in morphology with Pt loading or reduction at magnifications up to 50,000 $\times$.

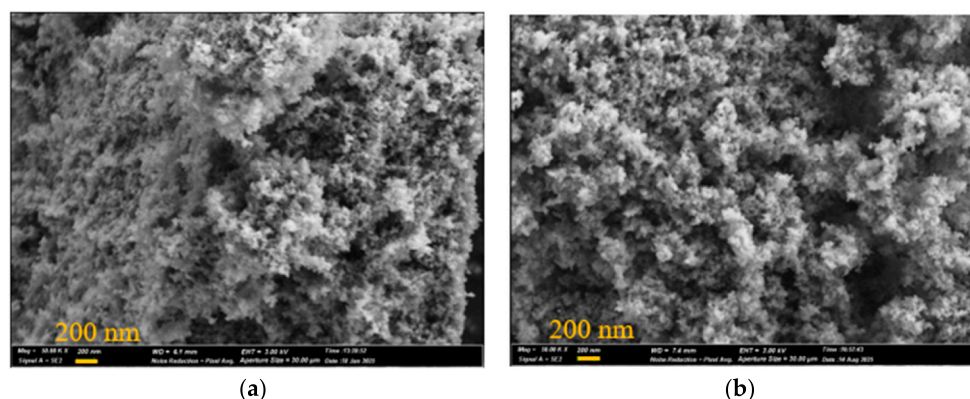


Figure 2. SEM micrographs of (a) TiO_2 , and (b) 5.0Pt(R)/ TiO_2 at 50,000 $\times$, 3 kV.

Pt loading was visualized on TiO_2 using high-resolution transmission electron microscopy (HRTEM), as shown in Figure 3a–c. The particle size of TiO_2 is approximately

25 nm. The d-spacings for TiO₂ are 3.72 Å on TiO₂ without Pt, and 3.57 Å on 5.0Pt(R)/TiO₂. These d-spacings represent the (101) plane of anatase TiO₂. The d-spacing on the reduced Pt particle is 2.27 Å, which is representative of the (111) plane of Pt [39]. 5.0Pt/TiO₂ and 5.0Pt(R)/TiO₂ have Pt particle sizes ranging from 1.7 nm to 3.7 nm. These results are supported by Dou et al. (2026), who reported d-spacings of Pt/TiO₂ catalysts (2.5 wt% loading) of 3.57 Å and 2.27 Å for TiO₂ and Pt, respectively, and Pt particle sizes of 1 nm to 4 nm [39]. Figures S1 and S2 show TEM and bright field TEM images for TiO₂, 1.0Pt(R)/TiO₂, 5.0Pt/TiO₂, and 5.0Pt(R)/TiO₂, respectively, for comparison. The Pt loading changes the concentration of Pt nanoparticles on the surface, as expected, but the Pt oxidation state does not appear to impact the Pt particle size and distribution on the catalyst surface.

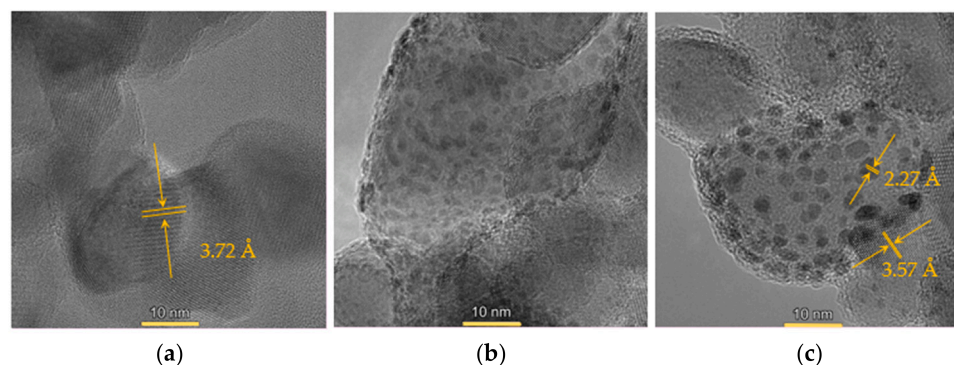


Figure 3. High-resolution TEM images of (a) TiO₂; (b) 5.0Pt/TiO₂; and (c) 5.0Pt(R)/TiO₂.

The Pt loadings on the catalysts were measured using energy-dispersive X-ray spectroscopy (EDS). Figure S3 and Table 1 compare the nominal and measured Pt/Ti atomic ratios for X.XPt(R)/TiO₂ catalysts. The correlation, shown in Figure S3, is linear with a slope of 0.998 and a coefficient of determination (R^2) equal to 0.997. These EDS analyses provide experimental evidence for the nominal Pt loadings. Figure S4 provides elemental mapping and an EDS spectrum for 5.0Pt(R)/TiO₂. The elemental mapping (Figure S4a) shows that Pt is well distributed on the catalyst.

Table 1. Summary of surface and crystallinity properties of catalysts.

Catalyst ID	Pt/Ti Atomic Ratio		Surface Area (m ² /g)	Average Pore Width ¹ (nm)	Pore Volume ¹ (cm ³ /g)	Anatase Crystal Size ² (nm)	Percent Anatase TiO ₂	Bandgap ³ (eV)
	Nominal	EDS						
TiO ₂	0	0	44.9	28.8	0.373	24.0	80.3%	3.21
0.5Pt/TiO ₂	0.0021	0.0014	46.2	28.9	0.383	22.5	79.6%	3.20
0.5Pt(R)/TiO ₂			45.7	29.4	0.377	23.1	79.7%	3.23
1.0Pt/TiO ₂	0.0041	0.0035	46.1	30.1	0.384	23.3	79.6%	3.19
1.0Pt(R)/TiO ₂			45.7	29.7	0.365	23.1	79.3%	3.21
2.5Pt/TiO ₂	0.011	0.011	45.7	28.6	0.349	23.5	79.1%	3.18
2.5Pt(R)/TiO ₂			43.7	30.4	0.355	24.0	80.9%	3.14
5.0Pt/TiO ₂	0.022	0.021	48.3	26.8	0.347	23.4	79.3%	3.21
5.0Pt(R)/TiO ₂			44.5	28.8	0.339	23.7	81.2%	3.13

¹ Based on the BJH model using N₂ desorption. ² Based on XRD patterns of the catalysts and the Scherrer equation. ³ Based on Tauc plots from diffuse reflectance UV-Vis spectroscopy data.

Additional efforts to confirm the nominal Pt loadings were conducted. Pt was extracted from measured masses of catalysts using aqua regia at 70 °C for 15 min. The extractant was used to make Pt-containing aqueous solutions that were analyzed using inductively coupled plasma (ICP). It was found that reduced Pt is extracted with much higher efficiency than oxidized Pt, and the extraction efficiencies increase with increasing Pt loadings. The Pt extraction efficiencies range from 67% to 92% for X.XPt(R)/TiO₂, whereas extraction

efficiencies range from 4% to 20% for X.XPt/TiO₂ catalysts. These results, shown graphically in Figure S5, indicate that a Pt reduction process should be conducted as a preliminary step before extracting Pt from TiO₂ catalysts with aqua regia. The extraction efficiencies from the reduced Pt samples are in agreement with Baghalha et al. (2009), who reported between 45% and 80% extraction efficiencies for Pt from spent reforming catalysts (0.23 wt% Pt) using aqua regia for 15 min at 80 °C [40].

Linear correlations between the Pt/Ti peak area ratios with nominal Pt loadings (wt%) were obtained from X-ray photoelectron spectroscopy (XPS) analyses. However, the correlations depended upon whether the Pt on the catalyst was oxidized or reduced. These results, provided in Figure S6, show that the relative Pt loadings follow expected catalyst compositions.

2.1.2. Platinum Oxidation State

The platinum oxidation states for all catalysts were investigated using XPS. A binding energy of 71.0 eV is representative of metallic Pt 4f_{7/2} (Pt⁰), whereas binding energies of 72.4 eV and 74.9 eV are representative of Pt²⁺ 4f_{7/2} and Pt⁴⁺ 4f_{7/2}, respectively [41]. All peaks have doublets (Pt 4f_{5/2}) due to spin–orbit coupling with binding energies that are approximately 3.35 eV greater than that of the 4f_{7/2} peak [41].

Figures 4a and 4b compare the XPS analyses for Pt in X.XPt/TiO₂ and in X.XPt(R)/TiO₂ catalysts, respectively. In Figure 4a, the X.XPt/TiO₂ catalysts have a mixture of Pt²⁺ and Pt⁴⁺, as indicated by the broad peaks with binding energies that range from ~71 eV to 80 eV. There are no metallic Pt species in the X.XPt/TiO₂ catalysts. The X.XPt(R)/TiO₂ catalysts (Figure 4b) have Pt4f_{7/2} and Pt4f_{5/2} peaks with maxima at binding energies ranging from 70.3 eV to 70.7 eV and from 73.5 eV to 74.3 eV, respectively. These peaks are attributed to metallic Pt (Pt⁰). The maximum heights of the Pt peaks generally trend toward lower binding energies with increasing Pt loading in X.XPt(R)/TiO₂ catalysts. Similar results were reported by Kim et al. (2022), and they attributed the shift to lower binding energies to increased electron density on the catalyst surface due to the interactions between the metallic Pt and the TiO₂ support [15]. While the X.XPt(R)/TiO₂ catalysts contain predominantly Pt⁰ species, the pronounced asymmetry of the doublets suggests that smaller concentrations of oxidized Pt species are also present on these catalysts.

Figure S7 compares the XPS binding energies for Ti in the X.XPt/TiO₂ and X.XPt(R)/TiO₂ catalysts. For TiO₂, the binding energy is expected to be 458.5 eV (Ti 2p_{3/2}) with the doublet being 5.7 eV higher at 464.2 eV (Ti 2p_{1/2}) [42]. The binding energies corresponding to the maximum height of the Ti 2p_{3/2} peak are provided in Table 2 for the catalysts in this study. The results show no apparent trend with Pt loading for X.XPt/TiO₂ catalysts, but they show a general shift toward lower binding energies as Pt loading increases in X.XPt(R)/TiO₂ catalysts. This shift is likely due to the presence of Ti³⁺ species following the reduction process and strong interactions between metallic Pt and TiO₂ [15].

Figure S8 compares the XPS binding energies for oxygen (O 1s) in the X.XPt/TiO₂ and X.XPt(R)/TiO₂ catalysts. The binding energies corresponding to the maximum height of the O 1s peak are provided in Table 2, and they are within the published range (529–530 eV) for O 1s in metal oxides [43]. The binding energies for O 1s are generally higher in X.XPt/TiO₂ catalysts than those in X.XPt(R)/TiO₂ catalysts. The shifts in O 1s binding energies can occur due to changes in the localized environments of lattice oxygen in TiO₂ [44], which can be affected by the oxidation state of the Pt species. For example, the general shift in O 1s to lower binding energies in X.XPt(R)/TiO₂ catalysts compared to those of X.XPt/TiO₂ catalysts is caused by the general decrease in the oxidation state of Pt on the surface as well as the reduction process during catalyst synthesis [45]. This is supported by Kim et al. (2022) who found that after loading and reducing Pt on TiO₂, a shift toward lower

binding energy of O 1s was observed [15]. They concluded that surface-adsorbed oxygen increased with Pt loading, which has better oxygen mobility than lattice oxygen and can contribute to the formation of oxygen vacancies and Ti^{3+} sites on the catalyst under reducing atmospheres [15].

Table 2. Summary of XPS binding energies for X.XPt/TiO₂ and X.XPt(R)/TiO₂ catalysts.

Catalyst ID	Binding Energies				
	O 1s	Ti 2p _{3/2}	Pt ⁰ 4f _{7/2}	Pt ²⁺ 4f _{7/2}	Pt ⁴⁺ 4f _{7/2}
TiO ₂	529.2	458.6	N/A	N/A	N/A
0.5Pt/TiO ₂	530.0	458.6	N/A	72.15	74.7
1.0Pt/TiO ₂	529.6	458.8	N/A	72.15	74.75
2.5Pt/TiO ₂	529.9	458.8	N/A	72.2	74.3
5.0Pt/TiO ₂	529.7	458.8	N/A	72.55	74.4
0.5Pt(R)/TiO ₂	529.4	458.6	70.7	72.1	74.9
1.0Pt(R)/TiO ₂	529.3	458.7	70.7	72.2	74.9
2.5Pt(R)/TiO ₂	529.4	458.5	70.45	71.7	74.6
5.0Pt(R)/TiO ₂	529.8	458.3	70.3	71.7	74.2

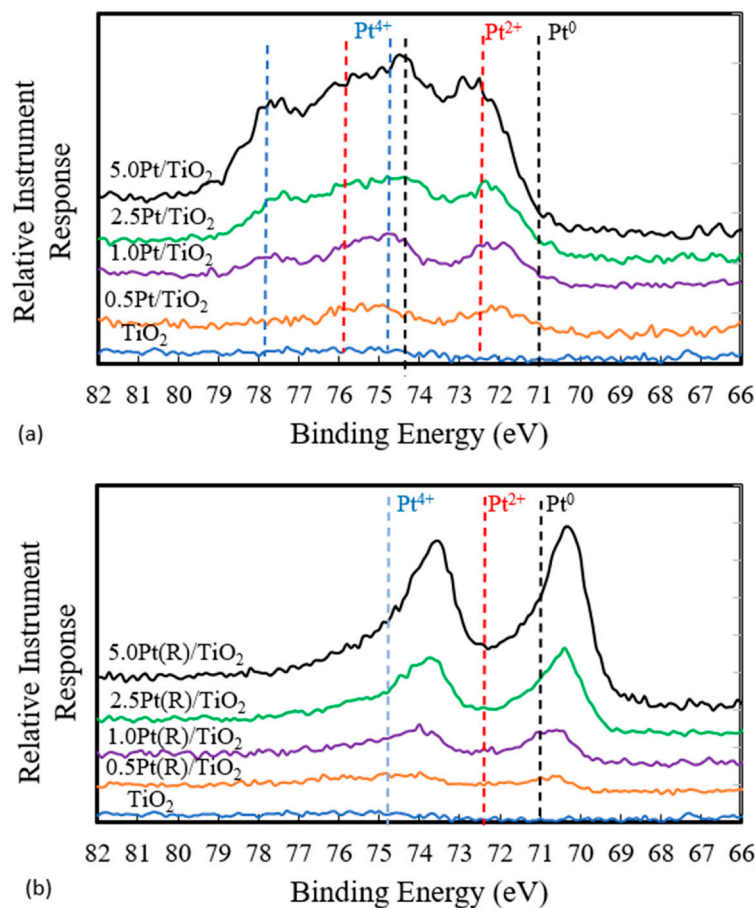


Figure 4. XPS analyses comparing (a) X.XPt/TiO₂ and (b) X.XPt(R)/TiO₂ oxidation states.

2.1.3. Thermogravimetric Analyses (TGAs)

TGAs were conducted to assess the thermal transitions that are accompanied by losses of mass. It is presumed that the reduction of Pt and Ti on the catalysts could be followed gravimetrically by the loss of oxygen from the catalysts with increasing temperatures. Figure 5a,b compare the TGAs of the oxidized and reduced catalysts. Using the TGA

data, mass losses in the ranges of <300 °C, between 300 °C and 700 °C, and >700 °C were calculated for each catalyst and are summarized in Table 3.

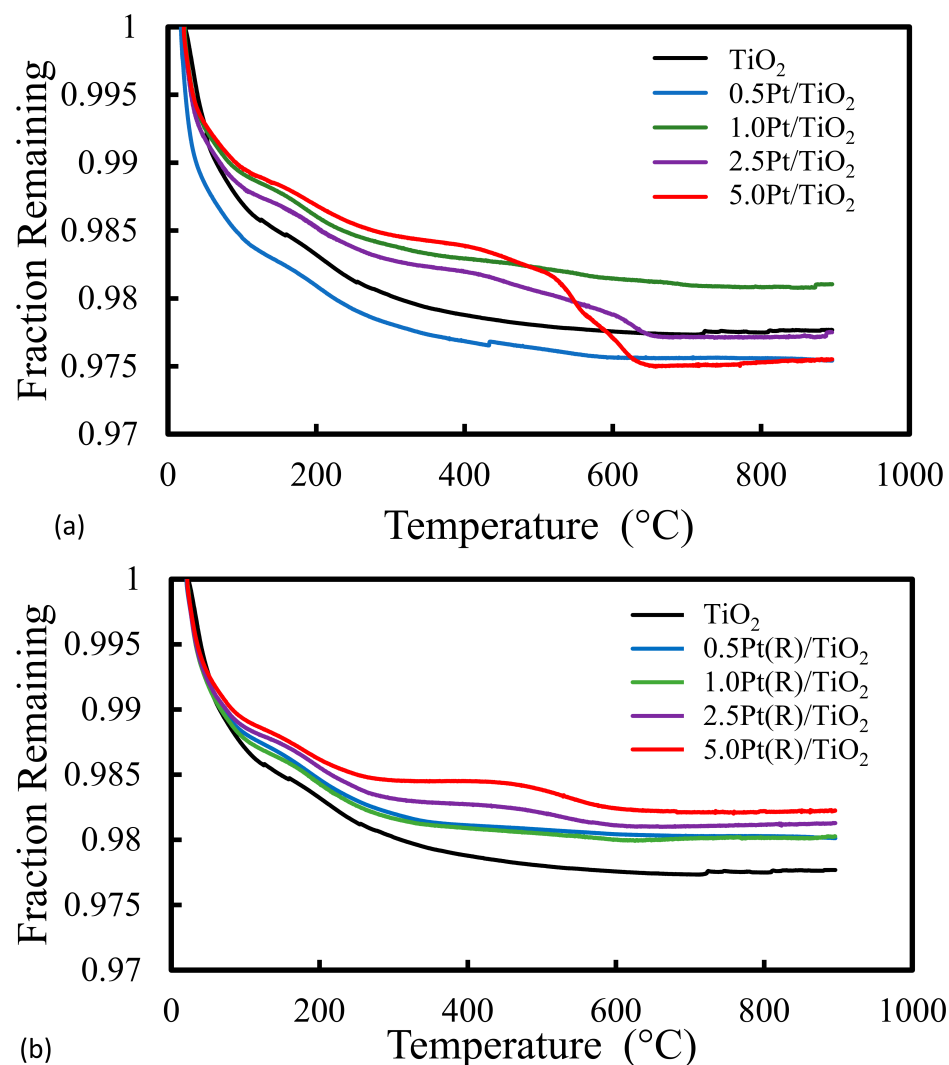


Figure 5. Thermogravimetric analyses of (a) X.XPt/TiO₂ and (b) X.XPt(R)/TiO₂ catalysts. The analyses were conducted in a nitrogen atmosphere with a temperature ramp of 10 °C/min.

Table 3. Mass losses (wt%)¹ from catalysts in a nitrogen atmosphere as a function of temperature.

Catalyst ID	<300 °C	300–700 °C	>700 °C
0Pt/TiO ₂	2.03%	0.29%	<0.05%
0.5Pt/TiO ₂	2.24%	0.27%	<0.05%
1.0Pt/TiO ₂	1.64%	0.30%	<0.05%
2.5Pt/TiO ₂	1.76%	0.58%	<0.05%
5.0Pt/TiO ₂	1.57%	0.99%	<0.05%
0.5Pt(R)/TiO ₂	1.84%	0.18%	<0.05%
1.0Pt(R)/TiO ₂	1.87%	0.16%	<0.05%
2.5Pt(R)/TiO ₂	1.72%	0.22%	<0.05%
5.0Pt(R)/TiO ₂	1.57%	0.25%	<0.05%

¹ % Losses were calculated with a basis mass equal to the mass of the catalyst after being heated to 900 °C.

The losses of mass from the catalysts at temperatures <300 °C are attributed to losses of adsorbed and bound water vapor and gases. These losses appear to generally decrease with

increasing Pt loadings. This suggests that Pt on TiO₂ may negatively affect the adsorption of water vapor on the catalyst.

Between 300 °C and 700 °C, the losses of mass increase with increasing Pt loadings for X.XPt/TiO₂ catalysts. In this temperature range, the losses of mass are attributed to the reduction of Pt, as PtO₂ is reduced to PtO and then to Pt⁰ as temperature increases. These results are supported by Nur et al. (2018), who found that PtO₂ decomposed to metallic Pt in air at 400 °C on anatase TiO₂ and at 650 °C on rutile TiO₂ [46]. Ono et al. (2010) observed that PtO₂ on TiO₂ decomposed with temperature according to a two-step thermal decomposition process (PtO₂ → PtO → Pt⁰) when heated in air [47]. In comparison, the reduced catalysts (X.XPt(R)/TiO₂) are more thermally stable, as indicated by lower losses of mass. The observed mass losses in X.XPt(R)/TiO₂ catalysts likely result from the reduction of the small concentrations of oxidized Pt species (supported by XPS analyses). A comparison of the mass losses from oxidized and reduced catalysts in the range of 300 °C and 700 °C are shown graphically in Figure S9. None of the catalysts lost a significant amount of mass at temperatures between 700 °C and 900 °C.

Figure S10 compares the fraction remaining and the derivative (d(Mass)/d(Temperature)) for TiO₂, 5.0Pt/TiO₂ and 5.0Pt(R)/TiO₂. At temperatures greater than 300 °C, 5.0Pt/TiO₂ loses mass predominantly at 550 °C and 615 °C, indicating the loss of oxygen from oxidized Pt species with different tendencies for reduction [27]. These results stem from the mixed valence states of Pt and their interaction with the TiO₂ surface. In comparison, 5.0Pt(R)/TiO₂ loses much less mass overall and predominantly at 540 °C.

2.1.4. Diffuse Reflectance UV-Vis Spectroscopy

Diffuse reflectance UV-Vis spectroscopy was used to assess light absorption as a function of wavelength. The results are shown in Figure 6a,b. As platinum loading increases, the light absorption increases in the visible light region ($\lambda > 380$ nm), and the absorption of visible light is greater with reduced Pt catalysts. However, the band edge is similar for all catalysts in this study. Tauc plots (shown in Figure S11) were used to calculate the bandgap of each catalyst, as summarized in Table 1. The bandgaps do not change significantly with Pt loading, as the observed differences were within 3% of the bandgap of P25 TiO₂. This indicates that Pt is deposited on the surface of the catalysts rather than incorporated into the crystal lattice [34,48,49]. Kubiak (2024) also concluded that Pt loading on TiO₂ does not expand the excitation absorption spectrum [48].

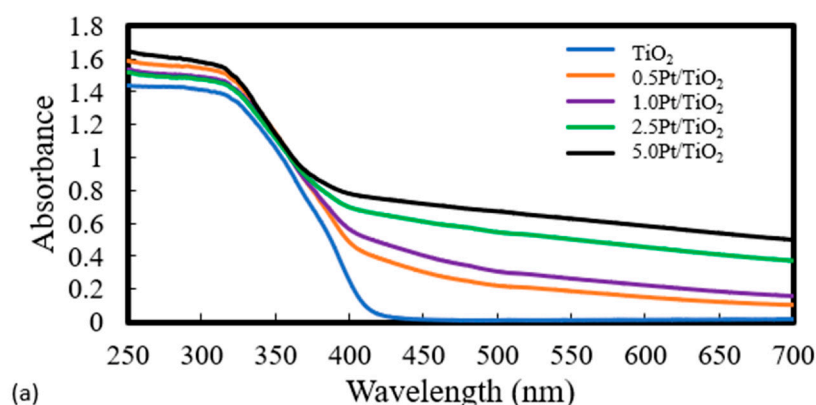


Figure 6. Cont.

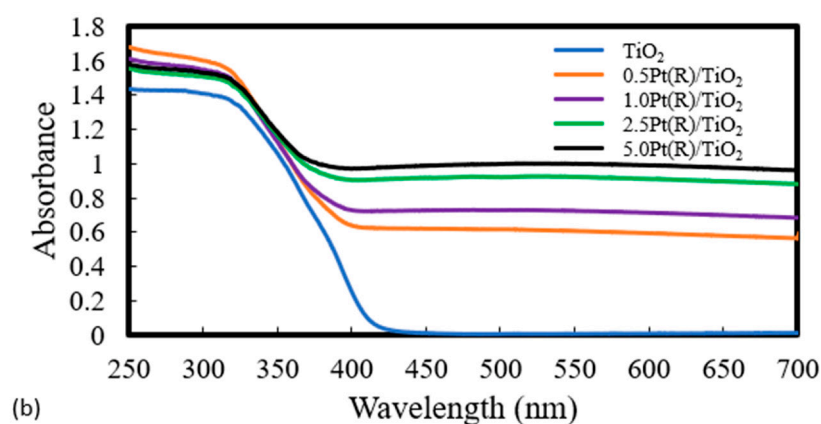


Figure 6. Diffuse reflectance UV-Vis analyses of (a) oxidized and (b) reduced Pt/TiO₂ catalysts.

2.1.5. BET Surface Area

The BET surface area of each catalyst was measured by nitrogen adsorption at 77 K, and the results are summarized in Table 1. Adsorption isotherms and pore size distributions for the catalysts are provided in Figures S12 and S13. All catalysts had surface areas in the range of 43.7 m²/g to 48.3 m²/g. Pt loadings up to 5 wt% did not appear to affect the surface area, average pore width, or pore volume significantly. Generally, the reduction process for synthesizing X.XPt(R)/TiO₂ catalysts appeared to decrease the surface area by less than 10% and increase the pore widths of the catalysts slightly. These results are supported by Kolobov et al. (2017), who observed less than a 6% change in surface area and less than a 10% change in pore volume for Pt/TiO₂ catalysts with Pt loadings up to 1 wt% [28].

2.1.6. Crystallinity

Figure 7 shows X-ray diffraction (XRD) patterns that represent the crystallinity of the X.XPt(R)/TiO₂ catalysts. The XRD patterns for X.XPt/TiO₂ catalysts are similar and are provided in Figure S14. The catalysts are a mixture of anatase and rutile TiO₂, similar to that of the starting support, P25 TiO₂. The anatase crystal sizes were calculated using the predominant anatase peak (101 reflection at $2\theta = 25.4^\circ$) and the Scherrer equation, shown in Equation (1). In Equation (1), d is the crystal size (nm), K is a dimensionless shape factor typically taken as 0.9, λ is X-ray wavelength (0.154 nm for copper K α radiation), B is the width of the diffraction peak at half maximum height (radians), and θ is the Bragg angle. The fraction anatase TiO₂ in the catalysts were calculated according to Equation (2) [50]. The results are summarized as percentages anatase TiO₂ in Table 1. There were no noticeable trends in the anatase crystal size or in the anatase fractions with Pt loading or oxidation state. This suggests that the Pt loading is on the surface of the TiO₂ rather than in the crystal lattice.

$$d = \frac{K \lambda}{B \cos(\theta)} \quad (1)$$

$$f = \frac{1}{1 + 1.26 * \frac{I_R}{I_A}} \quad (2)$$

There are no observable peaks that are unique to platinum in the XRD patterns. This suggests that the platinum is well distributed over the TiO₂ support and is either amorphous or below the detection limits of the XRD instrument. This is supported by Seo et al. (2010) [6] and Huang et al. (2015) [31] who also did not see peaks unique to platinum in XRD patterns of Pt-based catalysts with Pt loadings of up to 1.5 wt%.

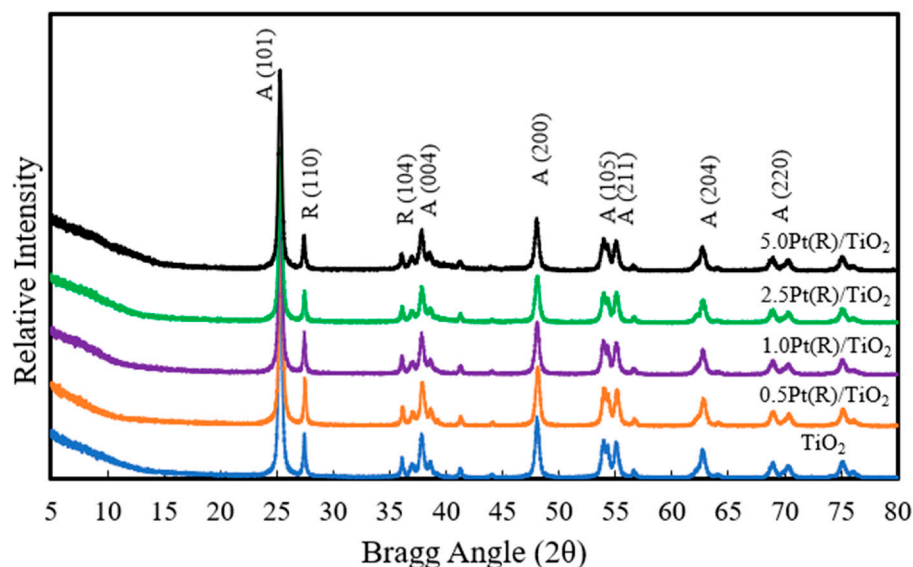


Figure 7. X-ray diffraction patterns for X.XPt(R)/TiO₂ catalysts. A = anatase TiO₂ crystal feature; R = rutile TiO₂ crystal feature.

Figure 8 shows the Raman spectra of the X.XPt/TiO₂ catalysts. The Raman peaks are representative of anatase TiO₂, with a small peak at $\sim 197\text{ cm}^{-1}$ that represents rutile TiO₂. Figure S15 compares 0.5Pt/TiO₂ and 0.5Pt(R)/TiO₂, with no noticeable differences between them. The Pt loading and oxidation state did not promote noticeable changes in the Raman spectra of the catalysts. This is supported by Melvin et al. (2015), who also reported no shift in TiO₂ peaks as a result of Pt loading [51].

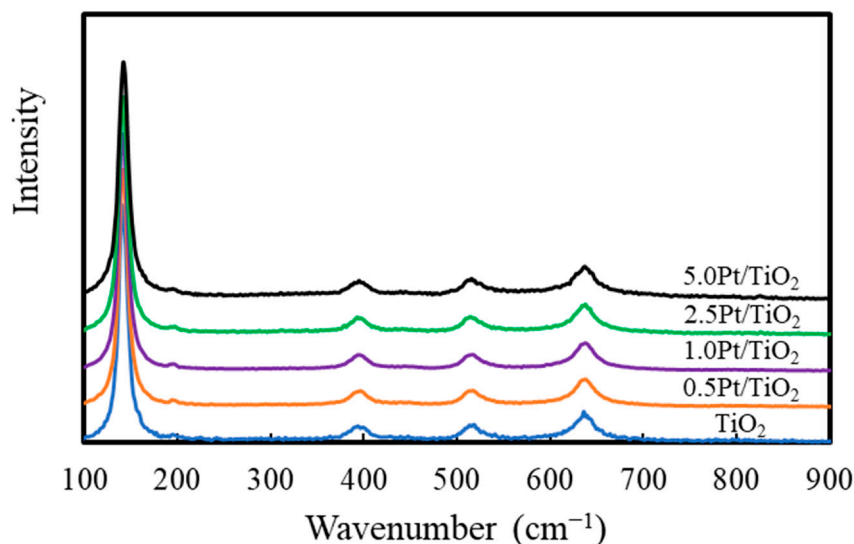


Figure 8. Raman spectra of X.XPt/TiO₂ catalysts.

Figure 9 shows FTIR spectra of the X.XPt/TiO₂ catalysts. Figure S16 shows similar FTIR spectra for X.XPt(R)/TiO₂ catalysts. Features at wavenumbers 3700 cm^{-1} and 1630 cm^{-1} (red arrows) are attributed to O-H stretching and vibration from adsorbed water [29,52]. The peak at 3700 cm^{-1} appears to decrease with increasing Pt loadings. This suggests that increasing Pt loading on TiO₂ may impact the absorption of water vapor on the catalyst.

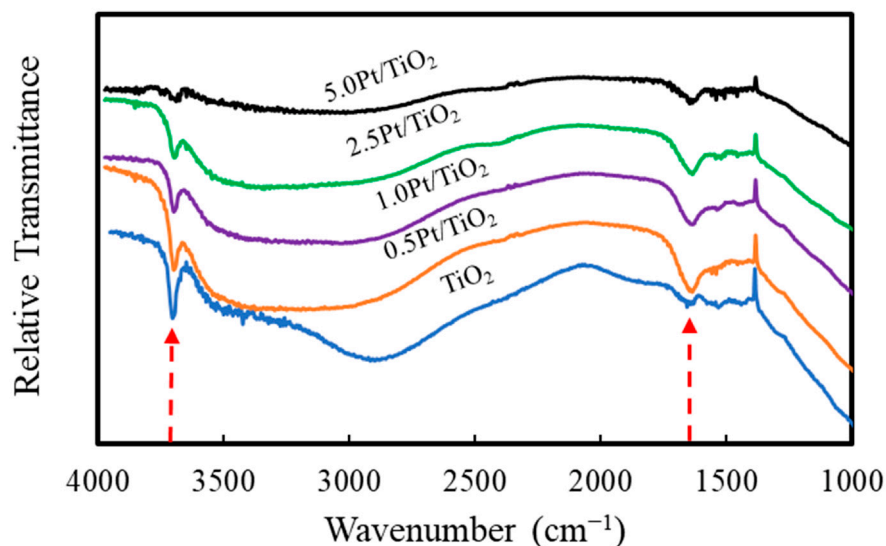


Figure 9. FTIR spectra of X.XPt/TiO₂ catalysts.

Many researchers have used FTIR to investigate the adsorption of CO on Pt [20,21,31,53–59]. Ivanova et al. (2007) state that Pt⁴⁺ cations in PtO₂ are not able to adsorb CO because it is fully coordinated with oxygen [57]. Lentz et al. (2017) have found that CO adsorption on Pt is both site and size-dependent, and the wavenumber for CO adsorption on metallic Pt particles increases with increasing particle size [58]. Figure 10 compares the adsorption of CO on 5.0Pt/TiO₂ and 5.0Pt(R)/TiO₂ at room temperature (21 °C). CO adsorption on 5.0Pt(R)/TiO₂ occurs at 2060 cm^{−1}, whereas CO adsorption on 5.0Pt/TiO₂ occurs at 2100 cm^{−1}. The adsorption of CO on TiO₂ is below the detection limits of the FTIR. CO adsorption on 5.0Pt(R)/TiO₂ occurs in the published range for metallic Pt (2028 cm^{−1} to 2079 cm^{−1}) whereas the CO adsorption on 5.0Pt/TiO₂ occurs at wavenumbers within and slightly lower than the published range for Pt²⁺ species (2105 cm^{−1} to 2117 cm^{−1}) [57]. The broadness of the CO adsorption peak for 5.0Pt(R)/TiO₂ provides evidence for Pt loading with both cationic and metallic Pt with a range of particle sizes. The lower intensity of the CO adsorption peak on 5.0Pt/TiO₂ compared to that of 5.0Pt(R)/TiO₂ demonstrates the relative tendencies for CO adsorption on Pt species on the catalysts [54].

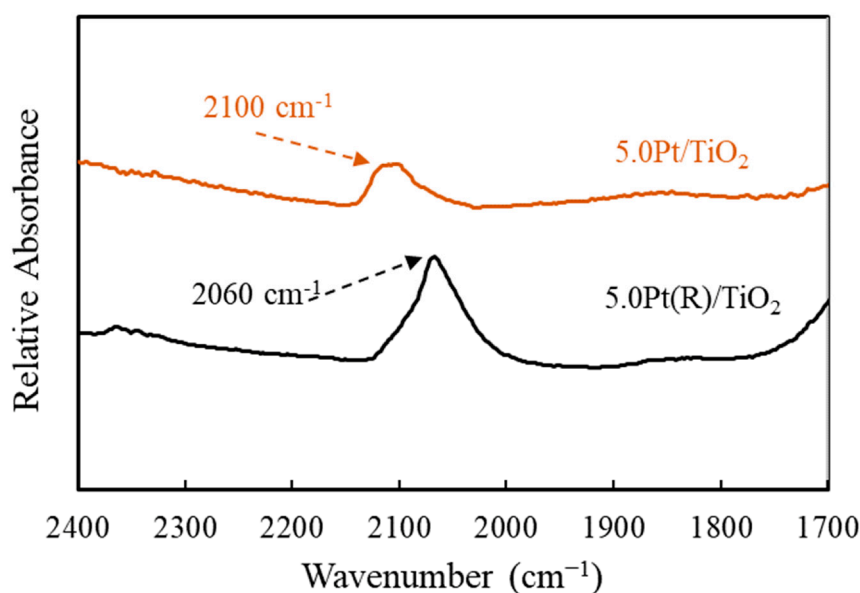


Figure 10. Comparison of FTIR spectra on 5.0Pt(R)TiO₂ with and without CO adsorption.

2.2. Photocatalyst Performance

The catalytic oxidation and photocatalytic oxidation of CO was investigated at room temperature (21 °C). Typical experimental trials are shown graphically in Figures 11 and 12. In Figures 11 and 12, “by-pass” represents the inlet concentrations of CO (blue) and CO₂ (orange) and the sum of CO and CO₂ (black); “LEDs Off” represents the reactor effluent without illumination (catalytic oxidation), and “LEDs On” represents the reactor effluent with illumination of the catalyst (photocatalytic oxidation). Since house air was used, the CO₂ concentration in the by-pass is representative of that in ambient air.

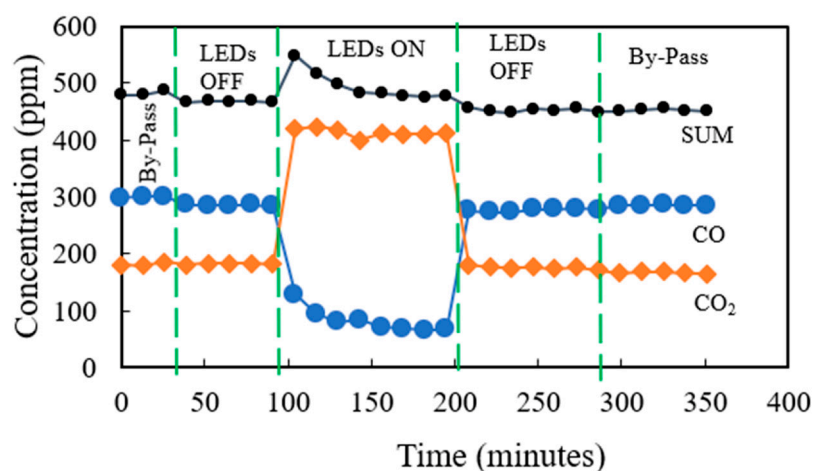


Figure 11. Experimental trials of room-temperature CO oxidation over 1.0Pt/TiO₂. Residence time = 60 ± 2 s; [CO] = 300 ppm; λ = 365 nm.

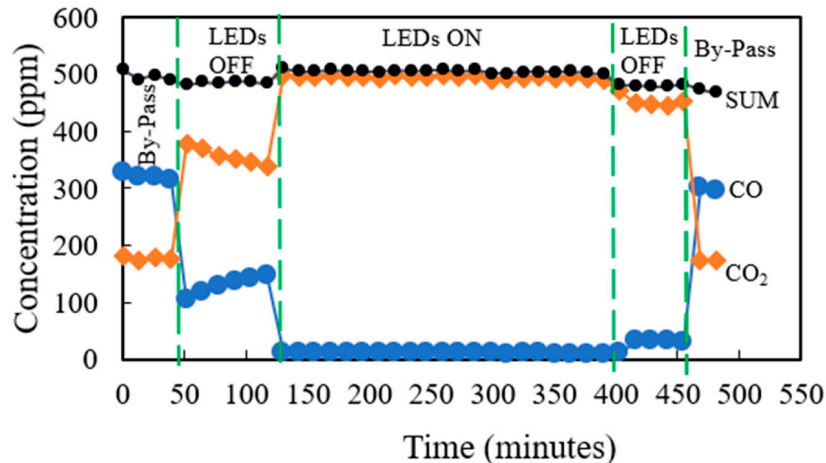


Figure 12. Experimental trials of room-temperature CO oxidation over 1.0Pt(R)/TiO₂. Residence time = 60 ± 2 s; [CO] = 300 ppm; λ = 365 nm.

Figure 11 shows the performance of 1.0Pt/TiO₂, on which the Pt species are a mixture of Pt²⁺ and Pt⁴⁺. The CO concentration decreased only slightly with LEDs off compared to the by-pass CO concentration. However, when the LEDs (λ = 365 nm) were turned on, the CO concentration decreased by approximately 70% while the CO₂ concentration increased stoichiometrically. The sum of CO and CO₂ initially spiked after the LEDs were turned on before settling to a steady-state value. The spike in the sum of CO and CO₂ is attributed to the adsorbed CO being photocatalytically oxidized quickly to CO₂ after the LEDs are turned on. When the LEDs were turned off, the concentrations of CO increased and CO₂ decreased to their pre-illumination values. These results indicate that 1.0Pt/TiO₂ has low catalytic activity but is still photocatalytically active for CO oxidation at room temperature.

Figure 12 shows the performance of $1.0\text{Pt(R)}/\text{TiO}_2$, on which Pt is predominantly Pt^0 and much smaller concentrations of Pt^{2+} and Pt^{4+} species. In contrast to the results with $1.0\text{Pt}/\text{TiO}_2$, the reduced catalyst has significant catalytic activity at room temperature for CO oxidation, as demonstrated by the decrease in CO concentration and corresponding stoichiometric increase in CO_2 concentration without illumination. However, the CO concentration appeared to continuously increase in the reactor effluent from approximately 100 ppm at 50 min to 150 ppm at 150 min, suggesting that the continuous adsorption of CO may inhibit catalytic activity. When the LEDs are turned on, the CO concentration decreases to below the detection limits of our analytical methods, while the CO_2 concentration stoichiometrically increases. These results represent the contribution of photocatalytic activity to room-temperature CO oxidation. When the LEDs are turned off again, the CO concentration increases only slightly and stays significantly below the CO concentration before illumination. This suggests that the Pt adsorption sites for CO were re-activated by the photocatalytic action and that the catalytic activity of $1.0\text{Pt(R)}/\text{TiO}_2$ for CO oxidation was improved. This is supported by Weon (2022), who concluded that the Pt active sites on Pt/TiO_2 are continuously cleaned during CO photocatalytic oxidation [12].

A comparison of Figures 11 and 12 elucidate that the reduced Pt (Pt^0) on TiO_2 is significantly more active for room-temperature CO oxidation than oxidized Pt (Pt^{2+} and Pt^{4+}). Figure S17 compares the catalytic and photocatalytic conversions of CO over $\text{X.XPt}/\text{TiO}_2$ and $\text{X.XPt(R)}/\text{TiO}_2$ catalysts, where $\text{X.X} = 0.5, 1.0$, and 5.0 wt% Pt. In each case, the reduced Pt catalysts had higher apparent catalytic and photocatalytic activities for room-temperature CO oxidation compared to the catalysts containing only oxidized Pt species.

The catalytic performances of $0.5\text{Pt}/\text{TiO}_2$ and $0.5\text{Pt(R)}/\text{TiO}_2$ were further compared as a function of residence time, as shown in Figure 13. Figures S18 and S19 provide the experimental trials used for Figure 13. $0.5\text{Pt(R)}/\text{TiO}_2$ exhibited higher catalytic activity with CO conversions between 30% and 40% compared to $0.5\text{Pt}/\text{TiO}_2$, which had CO conversions $< 10\%$ over residence times in the range of 17 s to ~ 100 s. These results are supported by Kim et al. (2016), who concluded that the active site for CO oxidation at room-temperature is metallic Pt [21]. Seo et al. (2010) also found that reduced Pt/TiO_2 catalysts had superior activity compared to calcined Pt/TiO_2 catalysts for room-temperature CO oxidation [6].

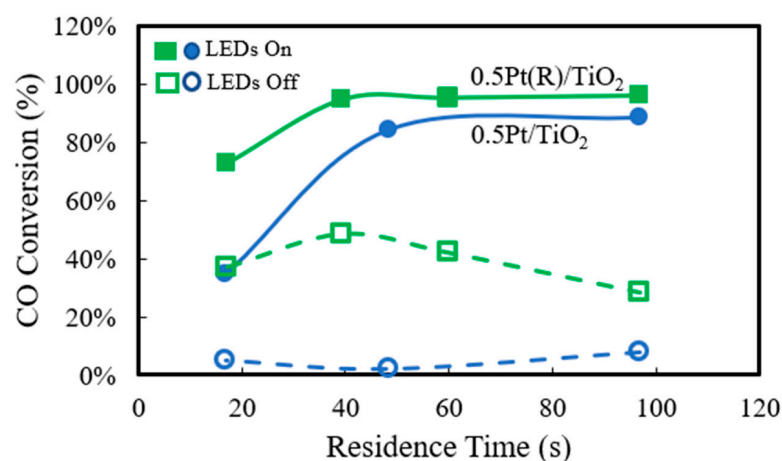


Figure 13. The effect of residence time over $0.5\text{Pt}/\text{TiO}_2$ and $0.5\text{Pt(R)}/\text{TiO}_2$ for both catalytic and photocatalytic oxidation of CO at room temperature. $[\text{CO}] = 300$ ppm; $\lambda = 365$ nm. Green square symbols = $0.5\text{Pt(R)}/\text{TiO}_2$, blue circle symbols = $0.5\text{Pt}/\text{TiO}_2$. Open symbols represent results without illumination; closed symbols represent results with illumination.

Both $0.5\text{Pt}/\text{TiO}_2$ and $0.5\text{Pt(R)}/\text{TiO}_2$ showed photocatalytic activity with 365 nm LEDs, with CO conversions over 80% for $0.5\text{Pt}/\text{TiO}_2$ and over 90% for $0.5\text{Pt(R)}/\text{TiO}_2$ at residence

times greater than 40 s. The promotion of catalytic activity with illumination was reported by Huang et al. (2015), who concluded that UV illumination can cause an increase in surface electron density of Pt nanoparticles by the photo-induced electron transfer from TiO_2 to Pt [31]. This results in enhanced adsorption, activation, and oxidation of CO on the catalyst [31]. Kubiak (2024) also concluded that the photocatalytic enhancement with Pt loading is due to more efficient charge carrier separation [48].

At low residence times (17 s), the conversion of CO decreases. The lower CO conversions may be the result of active site availability or mass transfer limitations for gas-phase reactions over a catalyst film. The relative dynamics of adsorption and desorption of CO and oxygen species on the catalysts and the intrinsic rates of photogenerated charges and recombination may limit the availability of active sites and apparent rates of CO oxidation. While the kinetics of room-temperature CO oxidation are beyond the scope of this study, Yu et al. (2017) conclude that different reaction pathways may occur, depending upon the reaction conditions, the oxidation state of Pt on the catalysts, the interaction of Pt with the support, and the availability and mobility of oxygen species at or near the Pt active sites on the catalysts [60].

The effect of Pt loading in X.XPt(R)/ TiO_2 catalysts is shown in Figure 14. The experimental trials for the effect of Pt loading are provided in Figure S20. Generally, the catalytic activity increased with increasing Pt loadings up to 5 wt%. With illumination ($\lambda = 365 \text{ nm}$), the activity of the catalysts was enhanced. Wang et al. (2005) found that Pt on TiO_2 promoted the charge carrier separation on UV-excited TiO_2 [22]. Otherwise, no trend with Pt loading could be assessed for the photocatalytic oxidation of CO, since the conversions of CO were greater than 95% for Pt loadings from 0.5 wt% to 5 wt%. TiO_2 without Pt was relatively inactive for both catalytic and photocatalytic oxidation of CO. Kolobov et al. (2017) reported similar results for up to 4 wt% Pt loadings on TiO_2 [28]. They further assessed the relative contributions of catalytic and photocatalytic oxidation of CO and found the highest contribution of photocatalysis occurred on a catalyst containing 1 wt% Pt on TiO_2 . Hu et al. (2019) also found an optimal Pt content of 1 wt% on $\text{Pt}/\text{WO}_3/\text{TiO}_2$ catalysts for the decomposition of exhaust species (CO , NO_x , and hydrocarbons (HC)) [29]. They conclude that metal loadings above the optimum will decrease charge carrier distances and hence facilitate charge recombination [29]. While an optimum Pt loading may be present, the optimum Pt loading for photocatalytic activity for catalysts with reduced Pt loadings could not be assessed in this study. However, Figure S17 supports the possibility of an optimum Pt loading on X.XPt/ TiO_2 catalysts for the photocatalytic oxidation of CO.

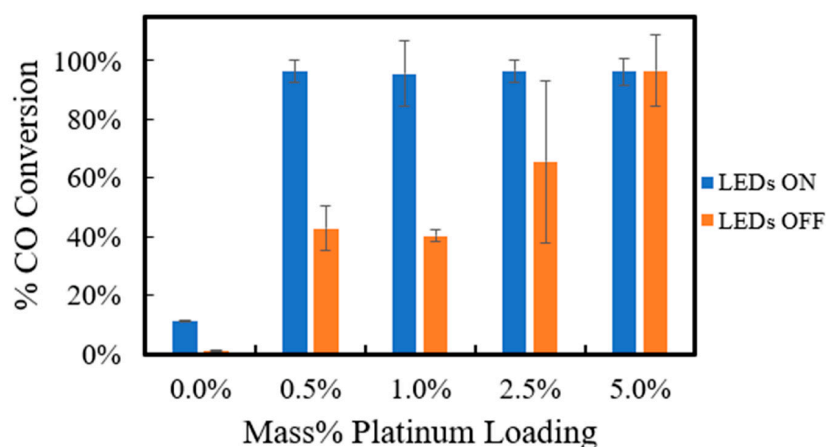


Figure 14. The effect of Pt loading in X.XPt(R)/ TiO_2 catalysts. Residence time = $60 \pm 2 \text{ s}$; $[\text{CO}] = 300 \text{ ppm}$; $\lambda = 365 \text{ nm}$.

The effect of inlet CO concentration on the apparent photocatalytic reaction rate for 0.5Pt(R)/TiO₂ was investigated, and the experimental data was fit to the Langmuir–Hinshelwood model (Equation (3), Figures S21 and S22). In this model, $-r_{CO}$ is the reaction rate ($\mu\text{moles}/\text{min}/\text{m}^2$ illuminated catalyst surface), k is the surface reaction rate constant (min^{-1}), K is an equilibrium constant ($\mu\text{moles m}^{-2} \text{ppm}^{-1}$), and C_{CO} is the concentration of CO (ppm) in the influent to the reactor. This model is supported for CO oxidation by Zhang et al. (2024), who concludes that in a dry environment, catalytic oxidation of CO on most noble metal catalysts follows the Langmuir–Hinshelwood mechanism [1]. Einaga et al. (2003) explained the dependencies of reaction rate on the concentrations of CO and water vapor in terms of the Langmuir–Hinshelwood type mechanism [30]. In this study, the catalyst, 0.5Pt(R)/TiO₂, was investigated in dry air (<10% relative humidity), 60 s residence time, and 365 nm LEDs. All conditions were held constant with the exception of the inlet CO concentration to the reactor, which was varied from 27 ppm to 500 ppm (Figure S21). Table S1 summarizes the model parameters and regression analysis used to fit the experimental data.

$$-r_{CO} = \frac{k K C_{CO}}{1 + K C_{CO}} \quad (3)$$

Visible light photocatalytic activity is demonstrated in Figure 15 and Figure S23 using 1.0Pt(R)/TiO₂ and LEDs varying in wavelengths from 365 nm to 525 nm. Notably, illumination of the catalyst with all wavelengths studied enhanced the activity of the catalysts for CO oxidation. Visible light photocatalytic activity of Pt/TiO₂ catalysts was also demonstrated by Abdelsalam et al. (2020), who used Pt/TiO₂ catalysts illuminated by visible light to photodegrade NO_x compounds [61].

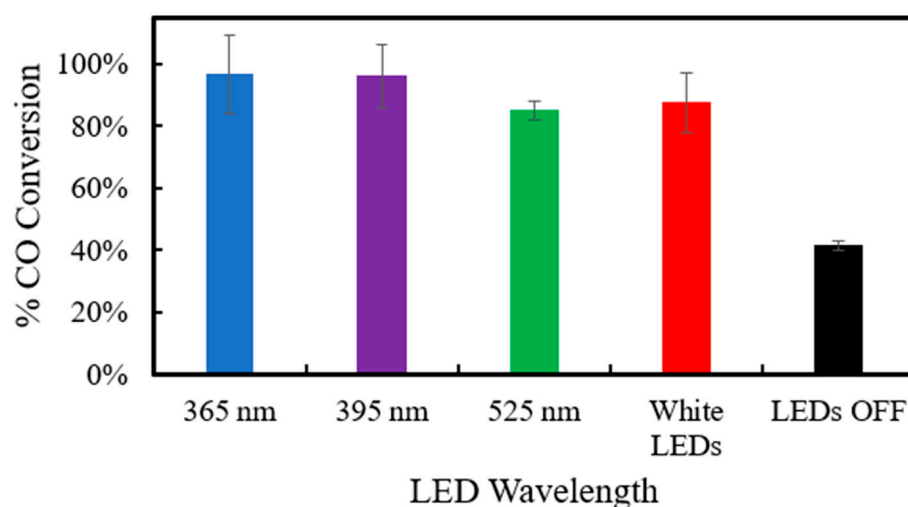


Figure 15. Effect of wavelength of illumination. Catalyst = 1.0Pt(R)/TiO₂; [CO] = 300 ppm; residence time = 60 ± 2 s, and % relative humidity < 10%.

Given that there was very little change in bandgap energies with Pt loading, the visible light photocatalytic activity of 1.0Pt(R)/TiO₂ for CO oxidation can be attributed to visible light photosensitization by Pt. According to Li and Li (2002), visible light photosensitization of Pt/TiO₂ may be due to the production of defect states as a result of Pt loading, which have lower energy levels within the TiO₂ bandgap [62]. Electrons can be excited from defect states to the TiO₂ conduction band, where they can undergo redox reactions. El Mchaouri et al. (2025) state that the presence of noble metals on TiO₂ allows for the capture of visible light due to the localized surface plasmon resonance effect, which enhances the excitation of electrons at the surface of the photocatalysts [63]. This effect is also described by Zerjav et al. (2023), where the surface plasmon resonance effect in Pt particles generates “hot”

electrons [64], some of which transfer from Pt to the conduction band of TiO_2 where they can react with adsorbed oxygen and CO.

The effect of humidity on the conversion of CO was investigated using $1.0\text{Pt(R)}/\text{TiO}_2$, and the results are provided in Figure 16 and in Figure S24. Humidity was detrimental to the room-temperature catalytic oxidation of CO, but there was no apparent effect on the photocatalytic oxidation of CO. In catalytic oxidation of CO, the adsorption of water vapor may compete with the adsorption of oxygen and CO on the surface of the catalyst. However, in photocatalysis, no apparent competitive adsorption was observed. This is supported by Einaga et al. (2003) who concluded that the adsorption of CO on Pt active sites is not significantly inhibited by water vapor, while the adsorption of CO on the surface of TiO_2 is significantly inhibited by water vapor [30].

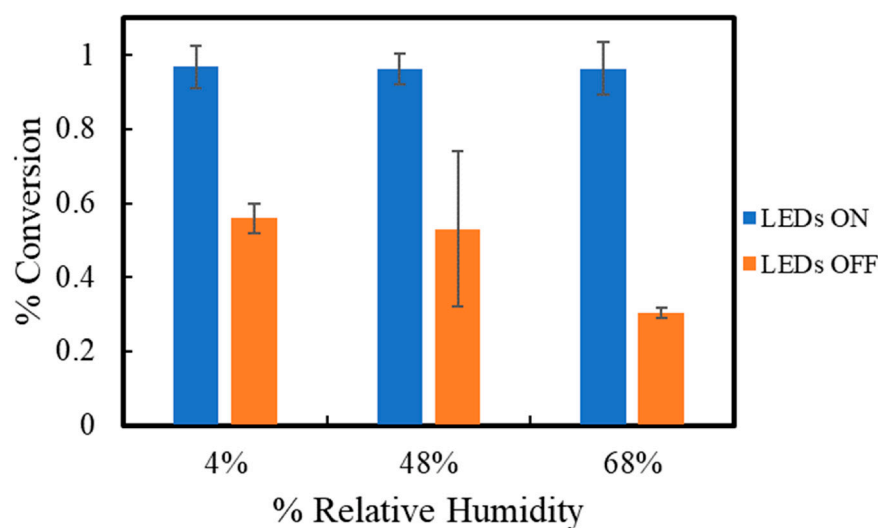


Figure 16. Effect of relative humidity on the room-temperature oxidation of CO. Catalyst = $1.0\text{Pt(R)}/\text{TiO}_2$; $[\text{CO}] = 300 \text{ ppm}$; residence time = $60 \pm 2 \text{ s}$; $\lambda = 365 \text{ nm}$.

According to Uner (2010), CO oxidation, both with and without illumination, takes place at the Pt sites [38]. The oxidation of CO proceeds with oxygen species generated from gas-phase oxygen. Bingam and Mills (2021) state that the oxidation of CO and reduction of water occur at two different sites, thus exhibiting non-competitive adsorption behavior of CO and water vapor [65]. Kim et al. (2026) state that metallic Pt surfaces were preserved during CO oxidation in the presence of both H_2O and SO_2 [25]. Kale et al. (2017) also showed that Pt catalysts did not lose activity with exposure to 2% water vapor during experimental trials [11].

3. Discussion

In this study, the effects of Pt loading (up to 5 wt%) and Pt oxidation state on a TiO_2 (P25) support were investigated using an LED-based flow reactor for the room-temperature catalytic and photocatalytic oxidation of CO in air. Catalytic activity increased with Pt loading up to 5 wt% and was significantly higher for catalysts containing reduced platinum (Pt^0) than for those containing only oxidized platinum species (Pt^{2+} , Pt^{4+}). In contrast, TiO_2 without Pt loading exhibited negligible catalytic and photocatalytic activity for CO oxidation, indicating that both the catalytic and photocatalytic oxidation of CO occur at or near the Pt active sites.

Catalyst characterizations showed that Pt loading formed nanoparticles in the range of 1 nm to 4 nm at the catalyst surface. However, Pt loading did not appear to affect the crystal lattice of TiO_2 . This is supported by the lack of observable changes in TiO_2 crys-

tallinity, crystal phase, surface area, and pore structure (Table 1). XPS analyses showed that X.XPt/TiO₂ catalysts contained both Pt²⁺ and Pt⁴⁺ species, whereas the oxidation state of X.XPt(R)/TiO₂ catalysts were predominantly metallic Pt (Pt⁰), with smaller concentrations of Pt²⁺ and Pt⁴⁺ (Figure 4a,b). Both TGAs and FTIR analyses of CO adsorption on the catalysts provided further evidence regarding the oxidation states of Pt on the catalysts.

The catalytic performance, measured in the absence of illumination, demonstrated that room-temperature CO oxidation is strongly influenced by both Pt loading and Pt oxidation state. The X.XPt(R)/TiO₂ catalysts consistently exhibited higher catalytic activity than the corresponding X.XPt/TiO₂ catalysts, and activity increased with increasing Pt loadings up to 5 wt%. Higher Pt loadings provide a greater concentration of active sites and increase the amount of surface-adsorbed oxygen and CO available for reaction [15], thereby promoting the adsorption and oxidation of CO. Reduced Pt (Pt⁰) is more effective at adsorbing and activating molecular oxygen [6,38], which is essential for efficient room-temperature CO oxidation. Strong metal–support interactions between Pt⁰ and TiO₂ may further modify the local lattice oxygen environment [44], enhancing oxygen mobility and promoting the formation of reactive oxygen species that participate in the oxidation reaction [15]. The results in this study clearly demonstrate the superior catalytic activity of X.XPt(R)/TiO₂ compared to X.XPt/TiO₂, which arises from the combined effects of enhanced CO adsorption, improved oxygen adsorption, mobility, and activation, and favorable metal–support interactions [66].

Both 1.0Pt/TiO₂ and 1.0Pt(R)/TiO₂ catalysts showed photocatalytic activity when illuminated with UV light energy (365 nm). When illuminated with ultraviolet (UV) light ($\lambda = 365$ nm), the photogenerated electrons in the conduction band of TiO₂ can be transferred to Pt, where they activate adsorbed oxygen and CO. The most widely accepted effect of Pt in the photocatalytic oxidation of CO is enhanced charge separation due to the formation of a Schottky barrier [38]. A Schottky barrier is a potential energy barrier for electrons formed at a metal–semiconductor interface [67]. This barrier increases the lifetimes of photogenerated electrons and holes [38], thus increasing the efficiency of the photocatalyst. Under the experimental conditions in this study, the photocatalytic oxidation of CO resulted in CO conversions > 95% for all X.XPt(R)/TiO₂ catalysts. Supplementary data in this study suggest that there may be an optimum Pt loading for the photocatalytic oxidation of CO over X.XPt/TiO₂ catalysts (Figure S17), which is supported by other published works [28,29]. An optimum would exist due to competing benefits of Pt active sites that can adsorb and activate CO and oxygen species and the detriments of possible formation of electron–hole recombination centers at higher Pt loadings.

Pt loading on TiO₂ enhanced visible light absorption for both oxidized and reduced catalysts (Figure 6). However, the bandgap of the Pt-loaded catalysts decreased by less than 2.5% relative to TiO₂ (Table 1). While the bandgap of Pt/TiO₂ catalysts were comparable to that of TiO₂, the visible light photocatalytic activity of 1.0Pt(R)/TiO₂ for CO oxidation was demonstrated in this study. Visible light photosensitization of Pt/TiO₂ may be attributed to the interaction of Pt and TiO₂ that produce defect energy states within the Pt/TiO₂ catalysts, thus giving the catalysts visible light activity [62]. Others attribute visible light photocatalytic activity to the localized surface plasmon resonance effect [63,64,68,69]. In this effect, the Pt/TiO₂ catalysts absorb visible light energy, which causes the excitation and generation of “hot” electrons [64,68,69]. Some of the “hot” electrons can overcome the Schottky barrier and transfer from Pt to the conduction band of TiO₂, which results in the activation of adsorbed oxygen and the apparent visible light photocatalytic activity for CO oxidation [64].

Humidity was detrimental to the room-temperature catalytic oxidation of CO, but there was no apparent effect on the photocatalytic oxidation of CO over 1.0Pt(R)/TiO₂. In

catalytic oxidation of CO, the adsorption of water vapor competes with the adsorption of CO and oxygen on the surface of TiO₂. However, in the photocatalytic oxidation of CO, the adsorbed water vapor did not appear to hinder the activity of the catalysts. The photocatalytic oxidation of CO and photoreduction of water occur at two different sites, thus exhibiting non-competitive adsorption behavior of CO and water vapor [65].

4. Materials and Methods

4.1. Materials

The source of platinum in the catalysts used in this study was chloroplatinic acid hexahydrate (Thermo Fisher Scientific, Waltham, MA, USA). The catalyst support for all catalysts is P25 TiO₂ (Evonik, Piscataway, NJ, USA). P25 TiO₂ was selected because it is a highly researched and commercially available photocatalyst [70]. Concentrated hydrochloric acid and nitric acid were purchased from Fisher Scientific (Carlsbad, CA, USA) and were used to make aqua regia in a HCl:HNO₃ volume ratio = 3:1.

4.2. Preparation of Catalysts

The synthesis of Pt-loaded TiO₂ was achieved using an incipient wetness impregnation approach. A stock solution Pt was prepared by adding 0.88 g chloroplatinic acid to 100 mL deionized water. This stock solution was used to add measured amounts of Pt to a slurry of 5 g of P25 TiO₂ and 100 mL of deionized water to obtain catalysts with desired Pt loadings. For each catalyst, the slurry was continuously heated and stirred to evaporate water until the slurry had the consistency of a paste. The catalyst was then dried in an oven at 105 °C for 20 h, crushed using a mortar and pestle, transferred to a crucible, and calcined in air at 400 °C for 20 h. The calcined catalyst was then crushed again with mortar and pestle and was stored and labeled as X.XPt/TiO₂, where X.X is the nominal wt% Pt loading (0.5 wt%, 1.0 wt%, 2.5 wt%, and 5.0 wt% Pt). Aliquots of the calcined catalysts were transferred to ceramic boats and were reduced for 1.5 h in a tube furnace at 400 °C in an atmosphere of 10 vol% hydrogen (H₂) in argon. The reduced catalysts are labeled as X.XPt(R)/TiO₂. Evidence of the nominal Pt loadings in the catalysts is provided in Figures S3, S5 and S6.

4.3. Catalyst Characterization

The catalysts were characterized to assess their chemical and physical properties. In an effort to confirm the Pt loading on the catalysts, Pt was extracted from the catalysts using aqua regia (HCl:HNO₃ volume ratio = 3:1, prepare on-site) at 70 °C. The extracted solutions were diluted in DI water and were analyzed using an Agilent 720 Inductively Coupled Plasma-Optical Emission Spectrometer (Santa Clara, CA, USA). The results of this effort are shown in Figure S5.

The BET surface area of each catalyst was measured using a Micromeritics TriStar II surface area analyzer (Norcross, GA, USA). The surface area was obtained at 77 K and at relative pressures < 0.25 using nitrogen as the adsorbate. The adsorption-desorption data was used to calculate the pore volume and pore size distribution using the BJH model.

The crystallinity and crystal structure of each catalyst was measured on a Bruker D8 Advance powder X-ray diffractometer (XRD) (Billerica, MA USA) at 2θ values ranging from 5° to 80°. The morphology of each catalyst was assessed using a Zeiss Supra 35 variable pressure FEG SEM (Oberkochen, Germany). The images were taken at 50,000× magnification using 3 KeV EHT, a working distance of 6 mm to 8 mm, and a 30 μm standard aperture. Energy-dispersive X-ray spectrometry (EDS, MahWah, NJ, USA) was used to confirm the nominal Pt loadings (Table 1, Figure S3) and to visualize the Pt distribution on the catalyst (Figure S4). The EDS analyses were conducted at 800× magnification, 25 KeV

and a 10 mm working distance with an aperture size of 60 μm . Bruker Esprit v.2.1 software was used for the EDS analyses.

Thermogravimetric analyses (TGAs) were conducted on all catalysts using a TA Instruments Q500 TGA (New Castle, DE, USA). The sample temperature was ramped to 900 $^{\circ}\text{C}$ at 10 $^{\circ}\text{C}/\text{min}$ in atmospheres of either nitrogen or air.

FTIR analyses were conducted on a Thermo Nicolet NEXUS 670 FTIR spectrometer (Thermo Fisher Scientific, Waltham, MA, USA). The FTIR was used to qualitatively assess the adsorption of CO on Pt. Potassium bromide (KBr) was used to secure powder catalyst samples in the instrument.

HRTEM of selected catalysts were conducted on a Thermo Scientific Talos F200i S/TEM (Waltham, MA, USA) at the University of Cincinnati's Advanced Materials Characterization Center. These analyses were used to assess the deposition and particle sizes of Pt on the catalysts. XPS analyses were conducted at the Ohio State University Department of Chemistry's Surface Analysis Laboratory to assess the oxidation state of Pt using a Thermo Scientific Nexsa G2 XPS (Waltham, MA, USA). All binding energies were calibrated to the carbon C 1s peak at 284.8 eV. Powders were pressed onto Cu tape that was placed on double sided insulating tape to isolate samples from instruments ground, and a charge neutralizer was utilized. No Cu was observed in the survey spectra so all analyses were devoid of substrate effects. Survey spectra were collected at 100 eV pass energy while high-resolution spectra were collected at 20 eV.

4.4. Catalyst Performance Tests

The photocatalytic test system has been described in detail previously [71] and is shown schematically in Figure 17. In brief, the test system consisted of a UV LED reactor, a DC power supply, and mass flow controllers to control the flow rates of air and CO through the system. The UV LED reactor was made of aluminum (internal dimensions of 11.7 cm $\times$ 11.7 cm $\times$ 1.91 cm) and was coated with 0.5 g catalyst (36.5 g catalyst/ m^2 illuminated area). To coat the reactor, a slurry of 0.5 g catalyst was added to 10 mL of ethanol. The slurry was vigorously agitated manually and then poured into the aluminum reactor base. The aluminum base was then heated over a hot plate and simultaneously swirled until the ethanol evaporated, leaving a dried catalyst film on the reactor base. The reactor base was then cooled to room temperature before use. The top of the reactor contained five equally spaced strips of UV-LEDs (Waveform Lighting [72]), and each strip consisted of eleven LEDs. The UV-LEDs were powered with a 12 V DC power supply, and the total power to the reactor was approximately 7 W. The light intensity at the surface of the photocatalyst was approximately 12 mW/cm^2 (see Figure S25). CO and CO₂ were analyzed at 13 min intervals using an SRI Model 8610C gas chromatograph (Torrance, CA, USA), equipped with a thermal conductivity detector (TCD) and a flame ionization detector (FID) with a methanator. Peaksimple software (Peak500 for Windows 10/11; SRI Instruments, Torrance, CA, USA) was used to obtain chromatographs and peak areas from the gas chromatograph. Calibration curves were used to correlate the peak areas for CO and CO₂ to their respective concentrations. The humidity was tracked using a CO₂ and humidity sensor (AZ Instrument Corp., Taichung, Taiwan) at the effluent of the reactor. Humidity was controlled by bubbling a fraction of the inlet air stream through a midjet impinger filled with water. The air humidification was done prior to mixing the air stream with CO. The source of air used was relatively dry, with a relative humidity = 4%. The relative humidity in the air was increased to 48% and then to 68% by changing the fraction of the inlet air stream that was bubbled through water.

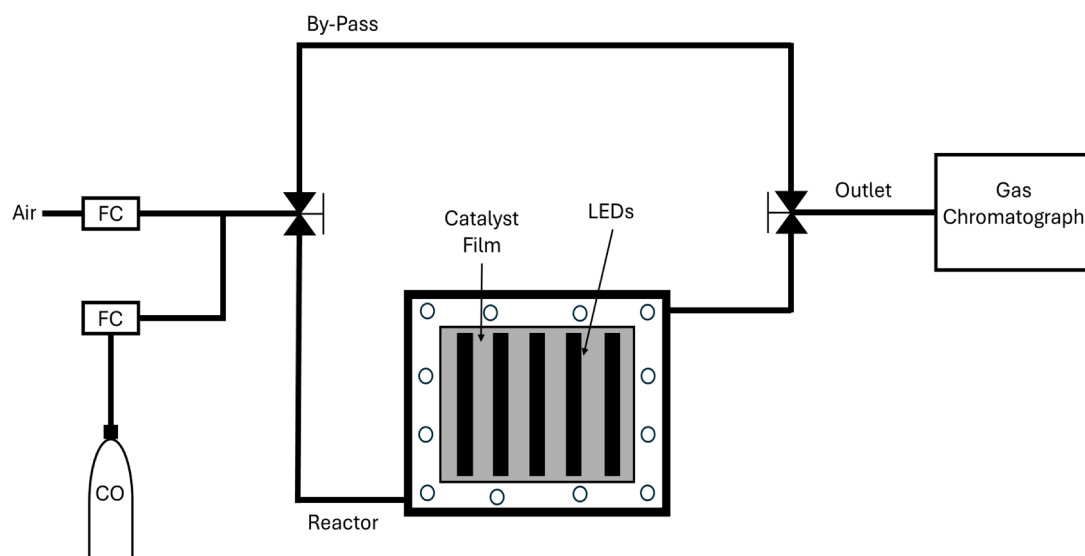


Figure 17. Schematic of the test system used for catalyst performance analysis.

5. Conclusions

This study has systematically investigated the effects of Pt loadings and oxidation states for both catalytic and photocatalytic oxidation of CO at room temperature in a flow reactor. Catalysts loaded with metallic Pt have significantly greater room-temperature catalytic activities than those loaded with only oxidized Pt species. Illuminating the catalysts with UVA ($\lambda = 365$ nm) LEDs enhanced the conversion of CO for both X.XPt/TiO₂ and X.XPt(R)/TiO₂ catalysts, but the photocatalytic effect was more pronounced with X.XPt(R)/TiO₂ catalysts. The photocatalytic activity of 1.0Pt(R)/TiO₂ was apparent even in visible light ($\lambda = 525$ nm), despite the catalyst having a bandgap similar to that of TiO₂. This suggests that mechanisms other than bandgap narrowing may contribute to the observed visible light photocatalytic activity.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/catal16090764/s1>, Figure S1: TEM images of (a) TiO₂, (b) 1.0Pt(R)/TiO₂, (c) 5.0Pt/TiO₂, (d) 5.0Pt(R)/TiO₂; Figure S2: Bright-field TEM images of (a) TiO₂, (b) 1.0Pt(R)/TiO₂, (c) 5.0Pt/TiO₂; (d) 5.0Pt(R)/TiO₂; Figure S3: Comparison of nominal and measured Pt/Ti atomic ratios from EDS analyses in X.XPt(R)/TiO₂ catalysts; Figure S4: EDS analyses (a) elemental mapping, and (b) EDS spectrum for 5.0Pt(R)/TiO₂; Figure S5: Pt extraction efficiencies as a function of nominal Pt loading on X.XPt/TiO₂ catalysts (blue circles) and X.XPt(R)/TiO₂ catalysts (orange diamonds); Figure S6: Pt/Ti peak area ratios from XPS analyses as a function of nominal wt% Pt on TiO₂ (Blue markers and trendline = X.XPt/TiO₂, Orange markers and trendline = X.XPt(R)/TiO₂); Figure S7: Comparison of binding energies for Ti in (a) X.XPt/TiO₂ catalysts and (b) X.XPt(R)/TiO₂ catalysts; Figure S8: Comparison of binding energies for O 1s in (a) X.XPt/TiO₂ catalysts and (b) X.XPt(R)/TiO₂ catalysts; Figure S9: Comparison of mass losses of X.XPt/TiO₂ and X.XPt(R)/TiO₂ catalysts between 300 °C and 700 °C; Figure S10: Comparison of mass losses of TiO₂, 5.0Pt/TiO₂ and 5.0Pt(R)/TiO₂ catalysts (a) fraction remaining vs. temperature; (b) derivative d(Mass)/d(Temp) vs. temperature; Figure S11: Tauc plots for (a) X.XPt/TiO₂ and (b) X.XPt(R)/TiO₂ catalysts; Figure S12: (a) Adsorption isotherms and (b) BJH desorption pore width plots for X.XPt/TiO₂ catalysts; Figure S13: (a) Adsorption isotherms and (b) BJH desorption pore width plots for X.XPt(R)/TiO₂ catalysts; Figure S14: XRD patterns for X.XPt/TiO₂ catalysts; Figure S15: Comparison of Raman analyses for TiO₂, 0.5Pt/TiO₂ and 0.5Pt(R)/TiO₂ catalysts; Figure S16: Comparison of FTIR analyses for X.XPt(R)/TiO₂ catalysts; Figure S17: Comparisons of X.XPt/TiO₂ and X.XPt(R)/TiO₂ catalysts for the catalytic (LEDs OFF) and photocatalytic (LEDs ON, $\lambda = 365$ nm) oxidation of CO (a) X.X = 0.5, residence time = 17 s, [CO] = 100 ppm, (b) X.X = 1.0, resi-

dence time = 62 s, [CO] = 300 ppm, (c) X.X = 5.0, residence time = 135 s, [CO] = 300 ppm; Figure S18: Effect of residence time for 0.5Pt(R)/TiO₂ at residence times of (a) 15 s, (b) 17 s, (c) 48 s, (d) 60 s, (e) 97 s, [CO] = 100 ± 20 ppm, λ = 365 nm; Figure S19: Effect of residence time for 0.5Pt/TiO₂ at residence times of (a) 17 s, (b) 48 s, (c) 97 s, [CO] = 100 ± 20 ppm, λ = 365 nm; Figure S20: Effect of Pt loading (a) TiO₂, (b) 0.5Pt(R)/TiO₂, (c) 1.0Pt(R)/TiO₂, (d) 2.5Pt(R)/TiO₂, (e) 5.0Pt(R)/TiO₂, Residence time = 60 s, λ = 365 nm; Figure S21: Effect of inlet CO concentration on the apparent reaction rate of CO over 0.5Pt(R)/TiO₂, Residence time = 60 s, λ = 365 nm; Figure S22: Effect of CO concentration experimental trials, [CO] = (a) 30 ppm, (b) 180 ppm, (c) 300 ppm, (d) 450 ppm, Residence time = 60 s, λ = 365 nm; Figure S23: Experimental trials to assess the effect of wavelength (a) 365 nm, (b) 395 nm, (c) 525 nm, (d) white LEDs; Figure S24: Experimental trials to assess the effect of relative humidity, (a) 4%, (b) 48%, (c) 68%, [CO] = 300 ± 20 ppm, λ = 365 nm, residence time = 60 s; Figure S25: Light intensity measurements (a) 365 nm measured with a UV light meter with 365 nm sensor, (b) 525 nm measured with a digital LUX meter (model LX1330B). Table S1: Summary of Sum-of-Least Squares Fit of experimental data with Langmuir-Hinshelwood kinetic model.

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