Catalysis Today 144 (2009) 37–41

Contents lists available at ScienceDirect

Catalysis Today

journal homepage: www.elsevier.com/locate/cattod

Photo-oxidation of nitrogen oxide over titanium(IV) oxide modified with platinum or rhodium chlorides under irradiation of visible light or UV light

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ARTICLE INFO

Article history:
Available online 6 February 2009

Keywords:
Photocatalyst
Visible light
Metal chloride
Titanium oxide
Nitrogen oxides

ABSTRACT

Titanium(IV) oxide samples modified with platinum or rhodium chloride (H_2PtCl_6/TiO_2 or $RhCl_3/TiO_2$) were prepared by an impregnation method and post-calcination at various temperatures and were used for photo-oxidation of nitrogen oxide under irradiation of visible light or UV light. Turnover numbers of both the catalysts were maintained at temperatures up to 350 °C under 24-h irradiation of visible light, although the specific surface area of the catalysts decreased greatly with increase in post-calcination temperature. The turnover number of H_2PtCl_6/TiO_2 was about two-times larger than that of $RhCl_3/TiO_2$. Only a small amount of released NO_2 was observed in the $RhCl_3/TiO_2$ catalyst, whereas in the H_2PtCl_6/TiO_2 catalyst, the amount of NO_2 released to gas phase increased with an increase in oxidation products. The small amount of released NO_2 indicates that most of the NOx adsorbed on $RhCl_3/TiO_2$ as an adsorption form of nonvolatile NO_3^-, whereas the amount of adsorbed NO_2 on H_2PtCl_6/TiO_2 was about four-times larger than that on $RhCl_3/TiO_2$. The results indicate that the oxidation rate of NO_2 to NO_3^- over $RhCl_3/TiO_2$ was faster than that over H_2PtCl_6/TiO_2. These results strongly suggest that the Cl radical induced by visible light was not directly related to the photo-oxidation of NO to NO_2 and NO_3^- and that the complex species of $RhCl_3$ and H_2PtCl_6 contributed to the photo-oxidation.

1. Introduction

Oxidative removal of nitrogen oxides (NOx) in air using photocatalysts has been noticed as a highly practicable technology to resolve serious environmental problems. Therefore, there have been many studies [1–5] on photocatalytic oxidation of nitrogen monoxide (NO) in air. Hybrids of photocatalysts and porous adsorbents such as zeolites have also been proposed for improving the removal of NOx [6–8]. Furthermore, catalytic materials that respond to visible light have been required to make better use of solar energy, because most conventional photocatalysts of semiconductor oxides such as titanium(IV) oxide (TiO_2), niobium(V) oxide and zirconium(IV) oxide with a high band-gap are excited by UV light. Visible-light-responding TiO_2 photocatalysts doped with impurity metal ions, anion, cation and nitrogen have been developed by many researchers [9–18]. However, a decrease in specific surface area often occurs at a high temperature in the doping procedure, and recombination of excited electrons and holes by doped ions increases. These phenomena lead to deactivation of the photocatalysts. Recently, another type of visible-light-responding photocatalyst, TiO_2 modified with metal chloride complexes, has been reported to degrade organic compounds in aqueous solution [19–22]. We have also been studying oxidative removal of NO in air over visible-light-responding photocatalysts loaded with metal chlorides on TiO_2. We already reported that some TiO_2 samples modified with metal chlorides such as rhodium(III) chloride ($RhCl_3$), chloroplatinic acid (H_2PtCl_6) and iron(III) chloride were effective for NOx removal under irradiation from a white fluorescent lamp and a blue fluorescent lamp (BFL) with a UV cut filter and that $RhCl_3/TiO_2$ exhibited a higher level of removal of NOx [23]. In this paper, we report about (1) high activity of $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 under irradiation of visible light, (2) mechanism by which nitrogen dioxide (NO_2) is released, (3) effects of post-calcination temperature on turnover number, (4) characterization of $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2, and (5) photo-oxidation sites.

2. Experimental

2.1. Materials

Rhodium(III) chloride trihydrate ($RhCl_3{\cdot}3H_2O$) and chloroplatinic acid hexahydrate ($H_2PtCl_6{\cdot}6H_2O$) were commercial materials of reagent grade and used without further purification. TiO_2

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doi:10.1016/j.cattod.2008.12.025

powders (ST-01) were supplied by Ishihara Sangyo Kaisha Ltd. NO gas was a commercial material.

2.2. Powder X-ray diffraction

XRD patterns of the samples were recorded using a Rigaku Multi Flex (Carbon monochromator, Cu*Kα*, 40 V–30 mA). Each sample was mounted on a sample board and measurements were immediately performed. The crystalline size of TiO_2 was determined from the half-height width of the 1 0 1 diffraction peak of anatase using the Sherrer equation; the value of the shape factor, K, was arbitrarily taken to be 0.9

2.3. Surface area measurements

Specific surface areas of the photocatalysts were determined using the BET single-point method on the basis of nitrogen uptake measured at −196 °C.

2.4. Preparation of photocatalysts

Photocatalysts were prepared by an impregnation method according to procedures described previously [23].

2.5. Photo-oxidation of NO

Photo-induced oxidative NO removal reaction was carried out in a fixed bed continuous flow reactor at atmospheric pressure according to procedures described previously [23]; the reactor was made of Pyrex glass equipped with a window (100 mm in diameter). In a typical run, the photocatalyst (100 mg) was weighed and loaded on a glass filter. The glass filter was placed on the sample board in the reactor. 10 ppm of gaseous NO (air balance) were introduced at a flow rate of 110 $cm^3\ min^{-1}$ into the reactor to attain the saturated adsorption of NO prior to irradiation of visible light as described previously [23]. Concentrations of NO and NOx ($NO + NO_2$) in the outlet gas were continuously monitored by a chemiluminescence NOx analyzer (ELC-88AO, Yanako) directly connected to the reactor. The photocatalyst was irradiated with a blue fluorescent lamp (10 W, S-3410, Sudo, maximum energy at 450 nm, designated as BFL hereafter) doubly covered with a UV cut filter, after attainment of NO adsorption equilibrium. The brightness on the surface of the photocatalyst spread on the glass filter in the reactor was 1200 lx, and the UV intensity was confirmed to be under the detection limit (0.1 $\mu W\ cm^{-2}$) of a UV radiometer (UM-10 + UM-360, Minolta). In other cases, a black light (10 W, FL-10BL, Hitachi, maximum UV energy at 369 nm) was also used to investigate the photo-oxidation activities of NO over the photocatalysts. The UV intensity on the surfaces of photocatalysts under the black light was 600 $\mu W\ cm^{-2}$. The total amount of NO removed from the gas phase, NO(fixed), was calculated by the following equation:

$$\mathrm{NO(fixed)} = \mathrm{NO(in)} - \mathrm{NOx(out)}, \quad (1)$$

where NO(in) and NOx(out) are the total amounts of NO introduced into the reactor and NOx ($NO + NO_2$) coming out of the reactor over a period of 5 or 24 h, respectively. Similarly, the total amount of NO oxidized to higher oxidation states, NO(oxidized), was calculated by the following equation:

$$\mathrm{NO(oxidized)} = \mathrm{NO(in)} - \mathrm{NO(out)}, \quad (2)$$

where NO(out) is the total amount of NO coming out of the reactor over a period of 5 or 24 h. This value is a summation of the amounts of NO fixed and NO_2 released to the gas phase. The turnover number was calculated from the ratio of NO(oxidized) to loaded metal chloride.

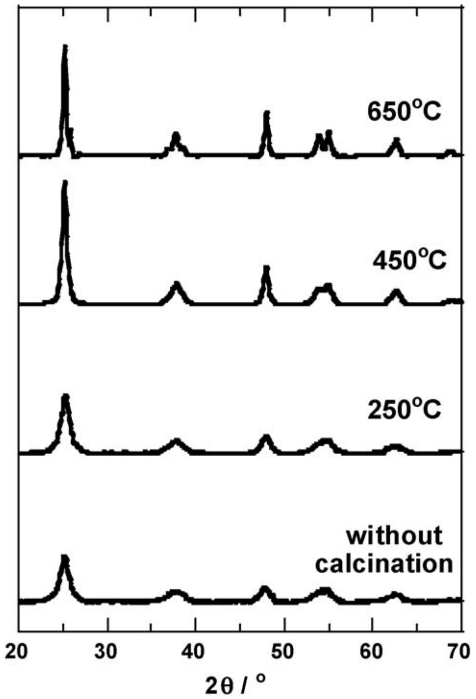

Fig. 1. Powder XRD patterns of $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts.

3. Results and discussion

3.1. Characterization of the catalysts

3.1.1. XRD

Typical powder XRD patterns of $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 samples calcined at various temperatures are shown in Fig. 1. Peaks assignable to anatase-type TiO_2 were observed, whereas those assignable to rhodium chloride and platinum chlorides were not detected, indicating fine dispersion of those chlorides on the surface of TiO_2. Crystalline sizes calculated from the 1 0 1 diffraction peak of the anatase using the Sherrer equation are summarized in Table 1. The crystalline sizes of anatase in the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 samples increased with an increase in post-calcination temperature.

Table 1
Surface area and crystalline size of TiO_2 samples modified with metal chloride after calcination at various temperatures.

Metal chlorides[a]	T_{cal} (°C)	S_{BET} ($m^2\ g^{-1}$)	$d_{1\,0\,1}$[b] (nm)
$RhCl_3$	60	320	6
$RhCl_3$	150	330	6
$RhCl_3$	250	290	7
$RhCl_3$	350	210	7
$RhCl_3$	450	160	12
$RhCl_3$	550	120	18
$RhCl_3$	650	80	18
H_2PtCl_6	60	340	6
H_2PtCl_6	150	350	7
H_2PtCl_6	250	280	7
H_2PtCl_6	350	180	9
H_2PtCl_6	450	110	13
H_2PtCl_6	650	50	22
TiO_2	–	340	6

[a] Rh loading: 97.2 $\mu mol\ g\text{-cat}^{-1}$, Pt loading: 51.3 $\mu mol\ g\text{-cat}^{-1}$.
[b] Calculated from the 1 0 1 diffraction peak of anatase.

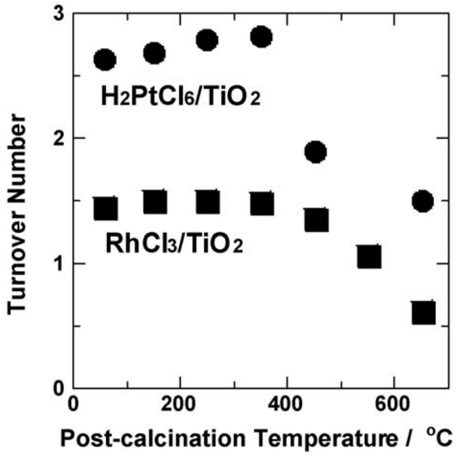

Fig. 2. Effects of post-calcination temperature on turnover number.

3.1.2. BET surface area

Specific surface areas of the photocatalysts were determined on the basis of nitrogen adsorption at −196 °C using the BET method to elucidate the relations between their photo-oxidation activity and surface area. The results are also summarized in Table 1. The specific surface area decreased greatly with an increase in post-calcination temperature. The decrease corresponded to the increase in crystalline size of anatase shown in Table 1. Loading of H_2PtCl_6 accelerated crystal growth of anatase TiO_2 at temperatures above 250 °C and significantly enhanced the decrease in surface area, compared with $RhCl_3$.

3.2. Photo-oxidation of NO

Photocatalytic oxidative removal of NOx in air involves two main processes: oxidation of NO to NO_2 and subsequent oxidation of NO_2 to NO_3^- [1,3,24–26]. Totally, NO was fixed as NO_3^- on the surface of the photocatalyst. Since accumulation of NO_3^- gradually decreases adsorption of NOx, higher holding capacity for NO_3^- and NO_2 is required for a photocatalytic material. The intermediate compound, NO_2, is more toxic than original NO. Therefore, release of NO_2 to gas phase should be suppressed, i.e., a low level of NO_2 release in addition to a high level of NOx removal is required for photocatalytic materials in the removal of NOx.

Effects of post-calcination temperature on the turnover number in the photo-oxidation of NO over $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts were studied under 5-h irradiation of visible light, and the results are shown in Fig. 2. The turnover number of the H_2PtCl_6/TiO_2 catalyst was about two-times larger than that of the $RhCl_3/TiO_2$ catalyst. The turnover numbers of the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts were maintained at post-calcination temperatures up to 350 °C, though the turnover number of the H_2PtCl_6/TiO_2 catalyst increased slightly with an increase in post-calcination temperature, that is, as shown in Table 1 and Fig. 2, there was little change in the turnover number up to 350 °C, though the specific surface area decreased greatly as a result of the post-calcination. The constant turnover numbers indicate that surface area had little effect on photo-oxidation activity and that the physical properties and number of active sites in the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts did not vary over the temperature range of 60–350 °C. Therefore, it is concluded that the active species were finely dispersed on the catalytic surface without aggregation and/or solid solution of catalytic species into TiO_2 bulk. At temperatures above 450 °C, the turnover numbers as well as specific surface areas of both catalysts greatly decreased, as shown in Table 1 and Fig. 2. Moreover, it is known that $RhCl_3$ and H_2PtCl_6 decompose at 450–500 °C [27] and at 370 °C [27], respectively. It is reasonable to conclude that the thermal decomposition of $RhCl_3$ and H_2PtCl_6 as well as decrease in surface area reflects the deactivation of photocatalysts.

The photo-oxidation of NO over the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts was studied under 24-h irradiation of visible light; the former was pretreated at 250 °C and the latter was pretreated at 350 °C. The results are summarized in Table 2. The turnover numbers per 1 h were determined to be 0.30 and 0.54 h^{-1} for the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts, respectively. The values were nearly equal to the values of 0.30 and 0.56 h^{-1} under 5-h irradiation of visible light for the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts, respectively (in Fig. 2). The results indicate that accumulation of NO_3^- on the surfaces of the photocatalysts scarcely suppressed the photo-oxidation of NO. Moreover, the turnover numbers of 7.2 and 12.9 indicate that the photo-oxidation of NO over $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts occurred catalytically under irradiation of visible light. In addition, the amount of NO_3^- accumulated in the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts increased with an increase in the reaction time and the amounts after 24-h reaction were much larger than the amounts of metal chlorides loaded. These results indicate that the active sites in the photocatalysts were different from adsorption sites. If it is assumed that NO_2 formed by photo-oxidation of NO is adsorbed on the surface of TiO_2, the equilibrium of NO_2 adsorption can be calculated as:

$$[NO_2]_{ad} = K \cdot P_{NO_2}, \tag{3}$$

where $[NO_2]_{ad}$, K, and P_{NO_2} are the amount of NO_2 adsorbed, equilibrium constant of NO_2 adsorption on the anatase surface, and partial pressure of NO_2, respectively. In this study, the concentration of NO_2 in the outlet gas at 24 h was used as P_{NO_2}. It was assumed that *K* for each photocatalyst was almost the same as that

Table 2
Photoinduced oxidation of NO over $RhCl_3/TiO_2$, H_2PtCl_6/TiO_2 and TiO_2 catalysts[a].

Sample	Metal loaded (μmol g^{-1})	Light irradiated[b]	NO fixed[c] (μmol g^{-1})	$C(NO_2)_{24}$[d] (ppm)	NO oxidized[e] (μmol $g^{-1)}$	Relative ratio of adsorbed NO_2
$RhCl_3/TiO_2$	97.2	Vis	677	0.31	699	0.55
$RhCl_3/TiO_2$	97.2	UV	698	0.11	705	0.20
H_2PtCl_6/TiO_2	51.3	Vis	571	1.3	663	2.3
H_2PtCl_6/TiO_2	51.3	UV	599	1.1	677	2.0
TiO_2	–	Vis	155	0	155	0
TiO_2	–	UV	592	0.56	632	1.0

[a] Catalyst: 100 mg, NO concentration: 10 ppm, flow rate: 110 cm^3 min^{-1}, reaction time: 24 h.
[b] Vis: visible light from a blue fluorescent lamp with a UV cut film, UV: UV light from black light.
[c] Total amount of NO removed from gas phase.
[d] Concentration of NO_2 coming out of the reactor at the end of 24-h reaction.
[e] Total amount of NO oxidized to NO_2 and NO_3^-.

for bare TiO_2 because most of the components of these photocatalysts were anatase-type TiO_2 as described below, and the amount of NO_2 adsorbed on the surface of bare anatase-type TiO_2 under 24-h irradiation of UV light was defined as the standard value. The relative amount of NO_2 adsorbed on each photocatalyst was hence estimated from the concentration of NO_2 in the outlet gas at 24 h using Eq. (3), and the values are also summarized in Table 2. The relative ratios of the amounts of NO_2 adsorbed on the photocatalysts were determined to be 0.55 for $RhCl_3/TiO_2$ and 2.3 for H_2PtCl_6/TiO_2 under 24-h irradiation of visible light and 0.20 for $RhCl_3/TiO_2$ and 2.0 for H_2PtCl_6/TiO_2 under 24-h irradiation of UV light. In the H_2PtCl_6/TiO_2 catalyst, the total amount of NO removed was 571 μmol g-cat^{-1} (Table 2) and the maximum amount of adsorbed NO_2 ($[NO_2]_{ad}$) should be less than 571 μmol g-cat^{-1}. Therefore, the maximum amount of $[NO_2]_{ad}$ on the surface of $RhCl_3/TiO_2$ was calculated to be less than 137 (=571 × 0.55/2.3) μmol g-cat^{-1} on the basis of comparison with the results for H_2PtCl_6/TiO_2 shown in Table 2. This indicates that maximum $[NO_2]_{ad}$ was 20% (=(137/677) × 100) of NO(fixed) for the $RhCl_3/TiO_2$ catalyst (677 μmol g-cat^{-1}). The maximum value of $[NO_2]_{ad}$ under 24-h irradiation of UV light was also determined to be less than 57 (=571 × 0.20/2.0) μmol g-cat^{-1} in the $RhCl_3/TiO_2$ catalyst, and the maximum amount of $[NO_2]_{ad}$ was therefore only 8.1% (=(57/698) × 100) of NO(fixed) (698 μmol g-cat^{-1}). These results indicate that most of the fixed species on the surface of the $RhCl_3/TiO_2$ catalyst was nonvolatile NO_3^-. In fact, the amount of NO_3^- recovered from the used $RhCl_3/TiO_2$ catalyst was 98% of the total amount of adsorbed NOx; the amount of NO_3^- recovered from the used catalyst with water elution was determined using absorption spectrophotometry. It is hence concluded that photo-oxidation of NO to NO_3^- occurred predominantly and that the small amount of NO_2 released was due to a small amount of NO_2 formed in the $RhCl_3/TiO_2$ catalyst.

The amounts of NO_2 released in the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts were studied under irradiation of visible light to elucidate the relation between the amounts of released NO_2 and NOx coverage: NOx coverage was calculated on the basis of the difference between NO(in) and NOx(out) as follows:

$$\text{NOx coverage} = \frac{N \cdot \text{MCS} \cdot \text{NO(fixed)}}{W \cdot S_{\text{BET}}}, \qquad (4)$$

where N, MCS, W and S_{BET} are Avogadro constant, molecular cross-section, catalyst weight and specific surface area of the catalyst, respectively. The surface area occupied with adsorbed NO_2 was determined from the product of number of adsorbed NO_2 molecular by a cross-section of the molecular. For NO_2 molecular, MCS was arbitrarily taken to be 0.171×10^{-18} m^2. The results are shown in Fig. 3. The amount of NO_2 released in the H_2PtCl_6/TiO_2 catalysts increased with an increase in NOx coverage, whereas the $RhCl_3/TiO_2$ catalysts began to release NO_2 to gas phase at around 4% of coverage. These results also indicate that the photo-oxidation of NO over $RhCl_3/TiO_2$ catalysts proceeded predominantly to form nonvolatile NO_3^- and that the photo-oxidation rate of NO_2 to NO_3^- over H_2PtCl_6/TiO_2 catalysts was slow compared with that of NO to NO_2. In both the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts with various surface areas, the decrease in specific surface area resulting from the post-calcination accompanied an increase of NOx coverage, because the coverage increases when the amounts of adsorbed NOx are nearly equal to those of the post-calcination, and so enhanced the release of NO_2 to gas phase. Therefore, the photocatalysts must be pretreated at <150 °C to suppress NO_2 release. The features of $RhCl_3$ and $PtCl_6$ should be reflected in the distribution of photo-oxidation products in the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts. Kisch and co-workers reported the following photo-oxidation mechanism for the degradation of organic compounds in aqueous solution using metal chloride complex-modified TiO_2: metal chloride complex (MCl_n) loaded on TiO_2 absorbs visible light, yielding a reduced species (MCl_{n-1}) and Cl radical; the Cl radical oxidizes an organic compound [19]. However, the difference in the product distribution between the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts strongly suggests that the Cl radical induced by visible light is not directly related to the photo-oxidation of NO to NO_2 and NO_3^- and that the complex species of $RhCl_3$ and H_2PtCl_6 contribute to the photo-oxidation.

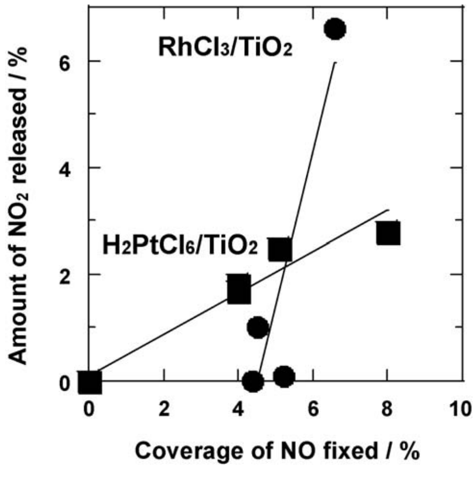

Fig. 3. Relation between amount of NO_2 released and coverage of NO fixed.

4. Conclusion

Photo-oxidation of NO over $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts was studied under irradiation of visible light. Turnover numbers of H_2PtCl_6/TiO_2 catalysts were about two-times larger than those of $RhCl_3/TiO_2$ catalysts. The turnover numbers of the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts were constant at post-calcination temperatures up to 350 °C, though the specific surface area decreased greatly, whereas at temperatures above 450 °C, the turnover numbers decreased with an increase in post-calcination temperature. Therefore, it is concluded that the active species were finely dispersed on the catalytic surface without aggregation and/or solid solution of catalytic species into TiO_2 bulk. The results indicate that the number of active sites did not change up to 350 °C. In addition, the rich distribution of photo-oxidation products, NO_3^-, indicates that the photo-oxidation of NO to NO_3^- occurred predominantly and that the small amount of NO_2 released was due to a small amount of NO_2 formed in the $RhCl_3/TiO_2$ catalyst. The characteristics of $RhCl_3$ and H_2PtCl_6 should be reflected in the distribution of the photo-oxidation products in $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts. The difference in photo-oxidation products strongly suggests that Cl radical induced by visible light was not directly related to the photo-oxidation of NO to NO_2 and NO_3^- and that the complex species of $RhCl_3$ and H_2PtCl_6 contributed to the photo-oxidation. The amounts of NOx adsorbed in the $RhCl_3/TiO_2$ and H_2PtCl_6/TiO_2 catalysts scarcely suppressed the photo-oxidation of NO and that the amounts adsorbed for 24-h irradiation were much larger than the amounts of loaded metal chlorides. These results indicate that the active sites in the photo-catalysts were different from the adsorption sites.

Acknowledgements

This work was partly supported by a Grant-in-Aid for Scientific Research on Priority Areas (417) and for Scientific Research (Nos. 19560773 and 20560723) from the Ministry of Education, Culture, Science, and Technology (MEXT) of Japan. This work was partly supported by a fund (Project No. P07020) from the Environmental Technology Development Department of New Energy and Industrial Technology Development Organization (NEDO). One of the authors (H. K.) is also grateful for the financial support from the Faculty of Science and Engineering, Kinki University.

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