Performance and mechanism study for low-temperature SCR of NO with propylene in excess oxygen over Pt/TiO$_2$ catalyst

Zhixiang Zhang, Mingxia Chen, Zhi Jiang, Wenfeng Shangguan*

*Research Center for Combustion and Environmental Technology, Shanghai Jiao Tong University,
Shanghai 200240, China. E-mail: zixon@139.com*

Abstract

A 0.5 wt.% Pt/TiO$_2$ catalyst was prepared and used for the low-temperature selective catalytic reduction (SCR) of NO with C$_3$H$_6$ in the presence of excess oxygen. The effects of Pt loading and O$_2$ concentration on Pt/TiO$_2$ catalytic performance for low-temperature SCR were investigated. It was found that optimal Pt loading was 0.5 wt.% and excess O$_2$ favored low-temperature SCR of NOx. The mechanism of low-temperature SCR of NO with C$_3$H$_6$ was investigated with respect to the behavior of adsorbed species over Pt/TiO$_2$ at 150°C using *in situ* DRIFTS. The results indicated that surface nitrosyl species (Pt$^{\delta+}$-NO and Ti^{3+}-NO) and Pt^{2+}-CO are main reaction intermediates during the interactions of NO, C$_3$H$_6$ and O$_2$. A simplified NO decomposition mechanism for the low-temperature SCR of NO with C$_3$H$_6$ was proposed.

Key words: NO; low-temperature SCR; Pt/TiO$_2$; *in situ* DRIFTS

DOI: 10.1016/S1001-0742(09)60273-4

Introduction

NOx (NO + NO$_2$) emitted from stationary and mobile sources results in serious air pollution, e.g., acid rain, photochemical smog, then possesses serious damages to human health and environmental safety (Wang et al., 2007). With the number of motor vehicles increasing instantly, exhaust gas pollution is being more and more serious. However, the traditional three-way catalysts (TWC) cannot be effective for low-concentration, oxygen-rich and low-temperature exhausts emitted from lean-burn engines and diesel vehicles. Therefore, it is needed to develop new catalysts with high performance for the removal of low-concentration exhausts at low temperature.

Selective catalytic reduction (SCR) technology is believed to be a promising method of deNOx and usually requires rather high reaction temperature (over 300°C) when hydrocarbons (HCs) or CO (Macleod and Lambert, 2002) are used as reducing agents. Although low-temperature removal of NOx by SCR can be achieved with the application of the toxic reducing agent NH$_3$, it is not suitable for mobile engines and immobile constructions (e.g., urban road tunnels and parking lots). If SCR of NOx with HC occurs over catalyst at low temperature (< 200°C) with high deNOx activity, the technology could compete with NH$_3$-SCR and be more practical for removal of NOx at stationary and mobile sources.

Low-temperature SCR of NOx by HCs has been extensively studied and a large number of catalysts have been evaluated. Noble metals (Pt, Pd, Rh, Ru) doped catalysts, e.g., Pt/Si-MCM-41 (Shen and Kawi, 2003), Rh/TiO$_2$ (Halkides et al., 2002), Pt/Al$_2$O$_3$ (Captain and Amiridis, 1999), are promising due to their high activity at low temperatures and good sulfur resistance and tolerance toward stream. Obuchi and co-workers (Obuchi et al., 1993) prepared a series of Pt, Pd, Rh and Ru supported γ-alumina and obtained high NO conversions in the presence of C$_3$H$_6$ at low temperature range (200–350°C). Kotsifa et al. (2007) developed 0.5 wt.% Pt/ZrO$_2$ catalyst which exhibited 40% NO conversation by C$_3$H$_6$ at 260°C. Moreover, quite different mechanisms have been proposed for those catalysts. Some suggested surface nitrate species as the main intermediates in the HC-SCR reactions (Flores-Moreno et al., 2005; Kantcheva, 2001), while others preferred the dissociation of NO over noble metal sites (Brown and King, 2000; Burch et al., 1994; Burch and Millington, 1996). Therefore, it is needed to investigate the specific mechanism of low-temperature HC-SCR.

In our recent work, Pt/TiO$_2$ was found to be the most active catalyst among a series of noble metal (Pt, Pd, Rh and Ru) supported TiO$_2$ catalysts for low-temperature selective catalytic reduction (SCR) of NO with C$_3$H$_6$ in excess oxygen. Under an atmosphere of 150 ppm NO, 150 ppm C$_3$H$_6$ and 18 vol% O$_2$ (GHSV 72,000 hr^{-1}), the 0.5 wt.% Pt/TiO$_2$ achieved 84% C$_3$H$_6$ conversion and 63% NOx reduction simultaneously at 140°C. This indicates

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* Corresponding author. E-mail: shangguan@sjtu.edu.cn

that Pt supported TiO_2 is a promising catalyst for low-temperature C_3H_6-SCR. In the present work, we studied the effects of Pt loading and O_2 concentration on the Pt/TiO_2 activity for low-temperature SCR. The mechanism of low-temperature C_3H_6-SCR was also investigated with respect to the behavior of adsorbed species over Pt/TiO_2 at 150°C using *in situ* DRIFTS and a simplified NO decomposition mechanism was proposed.

1 Experimental

1.1 Catalyst preparation

The 0.5 wt.% Pt/TiO_2 catalyst was prepared by wetness impregnation method using P25 (Degussa, Germany) and $Pt(NH_3)_2(NO_2)_2$ (Sinopharm Chemical Reagent Co., China) as starting materials. The catalysts evaluated in the study were all crushed to 40–60 mesh.

1.2 Catalyst characterization

Measurements of the BET surface area were based on N_2 adsorption data from a NOVA1000a Sorptomatic apparatus (Quantachrome, USA). The XRD patterns of catalysts were recorded using a Rigaku D/max-2200/PC X-Ray Diffractometer (Rigaku, Japan) with Cu$K\alpha$ radiation.

1.3 Catalytic activity tests

The SCR activity of the prepared catalysts was determined in a continuous fixed-bed U-tube quartz reactor using a 200 mg catalyst. Experiments were conducted in the temperature range of 120–300°C with a gas mixture containing 150 ppm NO, 150 ppm C_3H_6, 18 vol% O_2 and Ar balanced at a total flow rate of 120 cm^3/min (GHSV = 72,000 hr^{-1}). The NOx (NO, NO_2) concentration was measured by a chemiluminescence NO/NO_2 analyzer (42i LS, Thermo Environmental Instruments, USA). CO, CO_2 and C_3H_6 were determined by a gas chromatograph (GC-9160, HUAAI, China) equipped with hydrogen flame ionization detector (FID), and other reactants and products were analyzed with a quardrupole mass spectrometer (Dycor DM100M, Ametek process instruments, USA). The activity data were collected after the reactions reached near steady-state for at least 30 min at each temperature.

1.4 *In situ* DRIFTS

In situ DRIFTS experiments were performed on a 6700 FT-IR spectrometer (Nicolet, USA) equipped with a liquid N_2 cooled MCT-A detector. All spectra were collected at a resolution of 4 cm^{-1} (64 scans) and background corrected. Approximately 70 mg sample was placed in a high-temperature reaction chamber, which was directly connected to a flow system regulated by mass flow controllers. Prior to each experiment, the sample was reduced in flowing 5 vol% H_2/Ar for 20 min at 400°C and subsequently purged in Ar for 30 min at the same temperature. The total flow rate of each experiment through the chamber was 60 cm^3/min.

2 Results and discussion

2.1 BET and XRD results

The BET surface areas of bare TiO_2 and Pt/TiO_2 are 55 and 50 m^2/g, respectively, indicating that noble metal impregnation brought a slight decrease of BET surface area, which is a normal phenomenon for the impregnation process due to the coverage of surface by low surface area clusters of active component.

The XRD patterns of the bare TiO_2 and 0.5 wt.% Pt/TiO_2 are given in Fig. 1. All showed a multiple structure of rutile and anatase. Moreover, no diffraction peak corresponding to Pt or PtO was observed for supported TiO_2, suggesting that noble metal was well dispersed over the support or the formed particles were too tiny to be detected by XRD.

2.2 Catalytic activity tests

2.2.1 Effect of Pt loading

A comparative study of NO catalytic reduction by C_3H_6 with 18 vol% O_2 was carried out over TiO_2 and Pt/TiO_2 catalysts varied from 0.02 wt.% to 2 wt.% Pt loadings (Fig. 2). Even the least 0.02 wt.% Pt loading enhanced the SCR activity of TiO_2 support significantly. The Pt sites over catalyst surface are regarded as vital active centre for catalytic reactions. With increase of Pt loading from 0.02 wt.% to 0.5 wt.%, the reduction of NOx was improved from 19.3% to 63.4%, while the temperature of maximum NOx reduction and 100% C_3H_6 conversion decreased from 240 to 140°C and from 260 to 160°C, respectively. Further increase of Pt loading from 0.5 wt.% to 2 wt.% did not affect the SCR activity of Pt/TiO_2. thus, the optimal Pt loading of Pt/TiO_2 catalyst was 0.5 wt.% for low-temperature C_3H_6-SCR.

Additionally, low-temperature SCR over bare TiO_2 catalyst was found to yield a few amount CO, while in that of Pt/TiO_2 catalysts only CO_2 was detected as the product of C_3H_6. The results indicated that Pt/TiO_2 catalysts have 100% CO_2 selectivity towards C_3H_6 oxidation during the SCR of NOx.
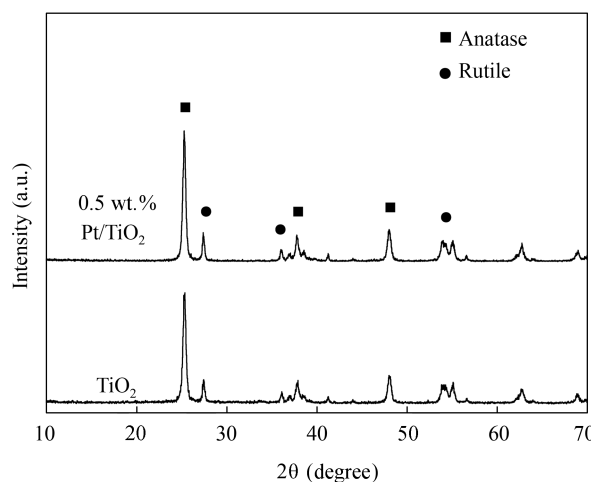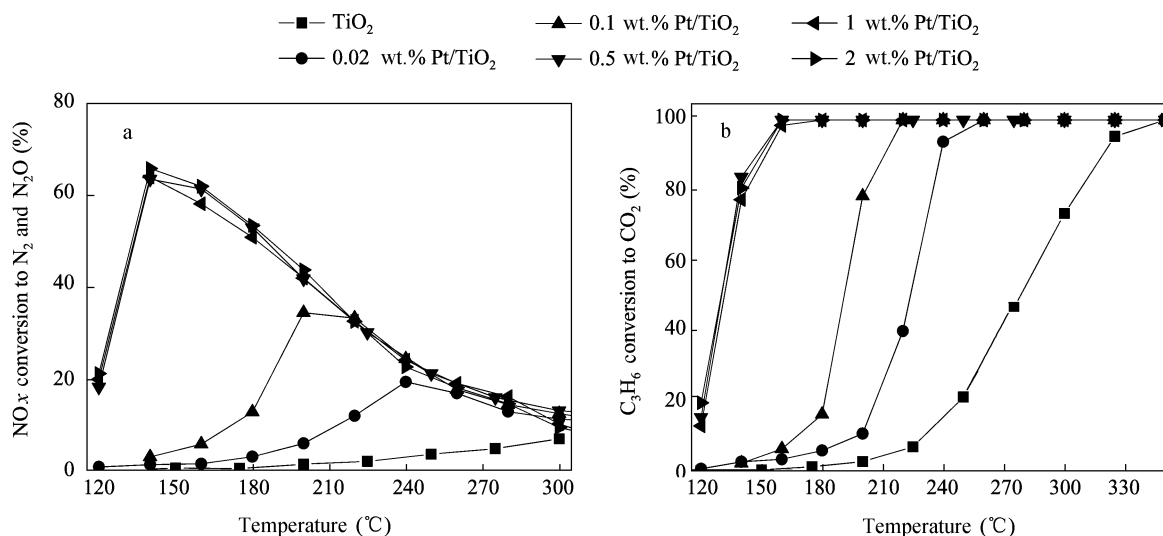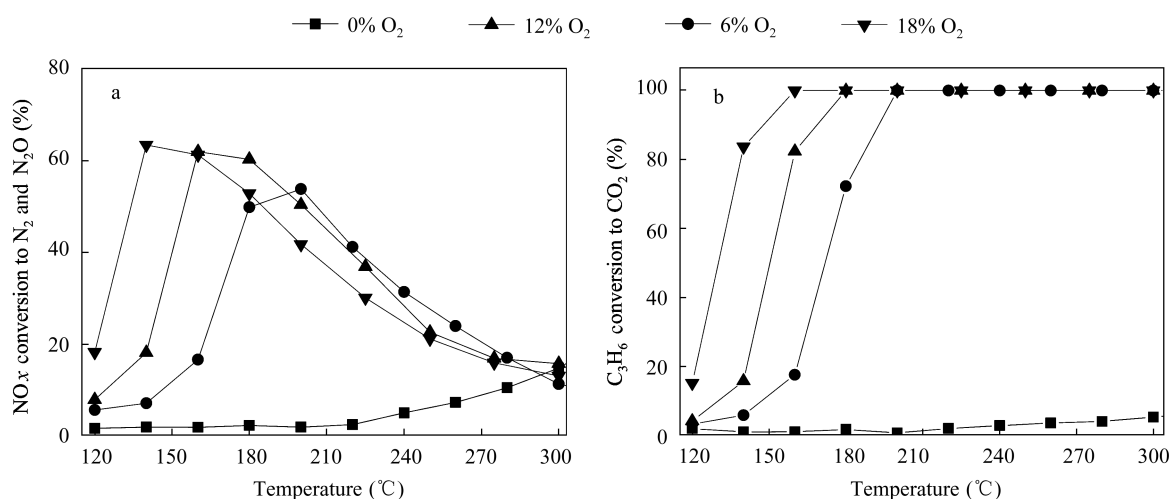

Fig. 1 XRD patterns of TiO_2 and 0.5 wt.% Pt/TiO_2.

Fig. 2 Effect of Pt content on the SCR reactions over Pt/TiO$_2$ catalysts. (a) NO conversion to N$_2$+N$_2$O; (b) C$_3$H$_6$ conversion to CO$_2$. Reaction conditions: 150 ppm NO; 150 ppm C$_3$H$_6$; 18 vol% O$_2$; Ar balance; 200 mg catalyst; total flow rate 120 cm^3/min; GHSV 72,000 hr^{-1}.

Fig. 3 Effect of O$_2$ concentration on the SCR reactions over 0.5 wt.% Pt/TiO$_2$ catalyst. (a) NO conversion to N$_2$+N$_2$O; (b) C$_3$H$_6$ conversion to CO$_2$. Reaction conditions: 150 ppm NO; 150 ppm C$_3$H$_6$; O$_2$ concentration at 0/6/12/18 vol% respectively; Ar balance; 200 mg catalyst; total flow rate 120 cm^3/min, GHSV 72,000 hr^{-1}.

2.2.2 Effect of O$_2$ concentration

The effect of O$_2$ concentration on the SCR reactions over 0.5 wt.% Pt/TiO$_2$ was studied and the results are shown in Fig. 3. In the absence of O$_2$, the reduction of NOx by C$_3$H$_6$ displayed rather low activity. Under the oxygen-rich conditions, increasing O$_2$ concentration from 6 vol% to 18 vol%, the activity of SCR was improved significantly and the active temperature window was shifted to lower temperatures. The positive effect of excess O$_2$ is also reported in other literature (Coónsul et al., 2004; Yentekakis et al., 2005). These results indicate that oxygen plays a key role in C$_3$H$_6$-SCR. Additionally, oxygen may also have a role to play in preventing coking of the catalyst surface from combustion of C$_3$H$_6$ (Burch et al., 2002).

During the process of SCR, C$_3$H$_6$ is easily to be partially oxidized to C$_x$H$_y$O$_z$ by surface oxygen atoms (e.g., Pt-O), which is suggested as crucial active intermediate species in the NO reduction process (Kotsifa et al., 2007; Zhang et al., 2009). The as-formed C$_x$H$_y$O$_z$ would selectively react with surface ad-NOx (e.g., Pt$^{\delta+}$-NO, Ti^{3+}-NO) species. Oxygen contributes to important reaction steps such as partially oxidation of C$_3$H$_6$ and red-ox of Pt sites. Therefore, increasing O$_2$ concentration promotes the formation of C$_x$H$_y$O$_z$ by providing more surface Pt-O species rather than unselective combustion of reductant with gaseous O$_2$.

2.3 *In situ* DRIFTS

2.3.1 NO + O$_2$ co-adsorption on the Pt/TiO$_2$ and TiO$_2$

The infrared spectra were recorded over Pt/TiO$_2$ and TiO$_2$ after exposure to a mixture of 150 ppm NO + 18 vol% O$_2$ at 150°C for 10 min, as shown in Fig. 4. Weak bands in 1734–1700 cm^{-1} region are attributed to Ti^{3+}-NO and bent NO molecules adsorbed on TiO$_2$ (Kantcheva, 2001) (Fig. 4, line b). In Fig. 4, line a, the bands at 1772
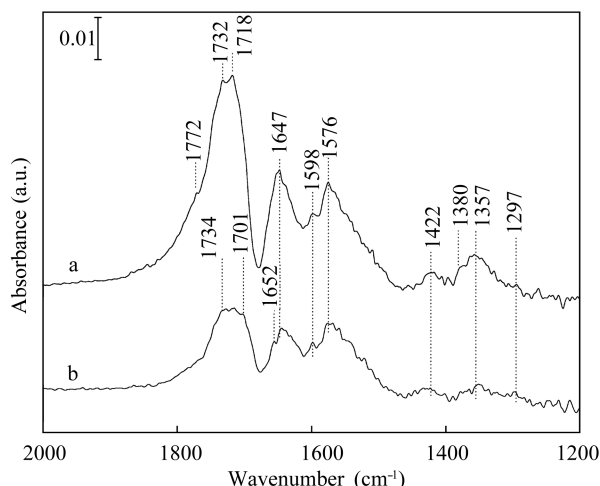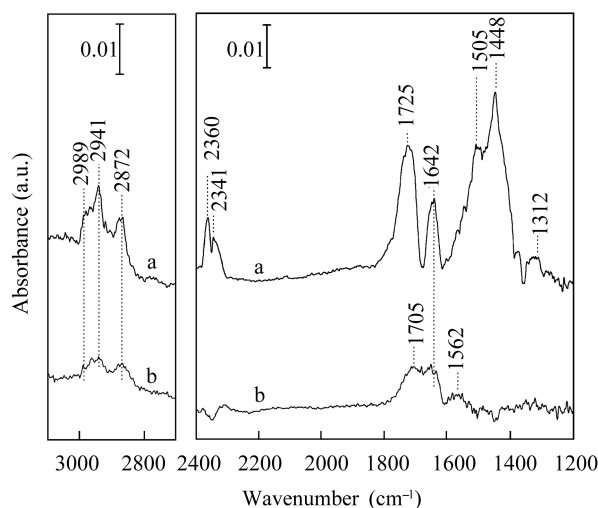
Fig. 4 *In situ* FT-IR spectra of Pt/TiO_2 (line a) and TiO_2 (line b) after exposure to 150 ppm NO + 18 vol% O_2 at 150°C for 10 min in an Ar flow. The spectra are background corrected.

and 1718 cm^{-1} are attributed to Pt^0–NO (Ivanova et al., 2007; Kotsifa et al., 2007) adsorbed on Pt sites, on which spillover of –NO is readily to occur and migrates from Pt to TiO_2 surface. Most –NO spillover are well accepted by Lewis acid Ti^{4+} sites, then leading to the formation of Ti^{3+}–NO species (Flores-Moreno et al., 2005). Therefore, band of Ti^{3+}–NO (1732 cm^{-1}) (Captain and Amiridis, 1999) in Fig. 4, line a was dramatically higher than that in Fig. 4, line b. Additionally, due to small amount of nitrates formed by NO_2 (came from NO oxidation) over Pt/TiO_2, bands located at 1647, 1576 and 1357 cm^{-1}, corresponding to bridging bidentate, chelating bidentate and mono-dentate nitrates (Flores-Moreno et al., 2005; Hadjiivanov and Knoözinger, 2000; Ivanova et al., 2007) associated with TiO_2 support, were slightly strengthened by Pt loading.

2.3.2 C_3H_6 + O_2 co-adsorption on the Pt/TiO_2 and TiO_2

The infrared spectra of 150 ppm C_3H_6 + 18 vol% O_2 co-adsorption on the Pt/TiO_2 and TiO_2 at 150°C for 10 min are shown in Fig. 5. The weak bands (1705, 1642 and 1562 cm^{-1}) assigned to acetone (Sobczak et al., 2008), H_2O and acetate on TiO_2 (Zhang et al., 2006) were detected in Fig. 5, line b; meanwhile, weak ν (C–H) bands (2989, 2941 and 2872 cm^{-1}) (Beutel et al., 1996; Halkides et al., 2002; Zhang et al., 2006) were also observed. It could be explained that a small amount of C_3H_6 was oxidized over TiO_2 surface. By contrast, more complicated and abundant IR bands assigned to the oxidation products of C_3H_6 appeared in the spectrum of Pt/TiO_2 (Fig. 5, line a), including CO_2 (2360, 2341 cm^{-1}) (Halkides et al., 2002; Zhang et al., 2006), ν (C=O) (1725 cm^{-1}) (Halkides et al., 2002; Sobczak et al., 2008), ν_s (COO$^-$) (1448 cm^{-1}) (Halkides et al., 2002; Kotsifa et al., 2007) and carbonate (1312 cm^{-1}) (Kumar et al., 2008). This can be attributed to the outstanding catalytic performance of Pt/TiO_2 for the oxidation of C_3H_6 (Zhang et al., 2009), which is believed to be an important step in the reaction of SCR (Kotsifa et al., 2007; Yentekakis et al., 2005; Zhang et al., 2009).


Fig. 5 *In situ* FT-IR spectra of Pt/TiO_2 (line a) and pure TiO_2 (line b) after exposure to 150 ppm C_3H_6 + 18 vol% O_2 at 150°C for 5 min in an Ar flow. The spectra are background corrected.

2.3.3 Interaction of NO-pretreated Pt/TiO_2 with C_3H_6 and O_2

To further understand the detailed mechanism of low-temperature SCR of NO with C_3H_6 in excess O_2, the time evolution of the FT-IR spectra has been collected during the interaction of Pt/TiO_2 with NO, NO + C_3H_6 and NO + C_3H_6 + O_2 in sequence (Fig. 6).

As shown in Fig. 6, line a (1–10 min), weak bands of $(O_{ads})Pt$–NO (1788 cm^{-1}), i.e., nitrosyl species surrounded by Pt–O species (Captain and Amiridis, 1999; Kotsifa et al., 2007), Pt^0–NO (1717 cm^{-1}) (Ivanova et al., 2007; Kotsifa et al., 2007) and nitrates (1637, 1588 and 1558 cm^{-1}) (Halkides et al., 2002) associated with the adsorption of NO on Pt/TiO_2 surface, appeared immediately after the introduction of NO into the chamber, but the peak intensity remained little change in the following 10 min. The differences between the spectra in Fig. 6, line a (1–
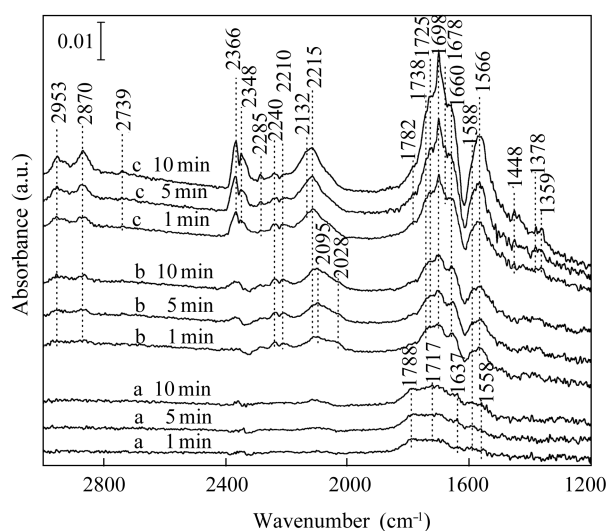
Fig. 6 FT-IR spectra obtained after interaction of the Pt/TiO_2 with 150 ppm NO for 10 min at 150°C (line a), following by introduction of 150 ppm C_3H_6 (line b) and subsequent 18 vol% O_2 (line c).
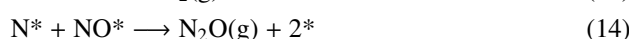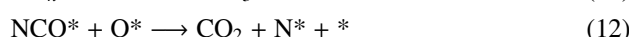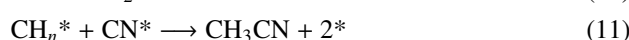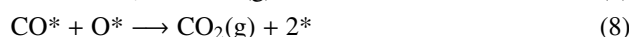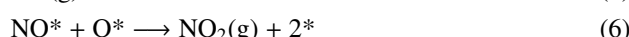
10 min) and Fig. 4, line a could be caused by the scarcity of oxidative phase of Pt sites ($Pt^{\delta+}$, Pt^{2+}) over the H_2-prereduced Pt/TiO_2, which were readily to associate with NO in the presence of O_2 (Kotsifa et al., 2007).

As shown in Fig. 6, line b (1–10 min), after subsequent introduction of C_3H_6, it is observed that the bands of $(O_{ads})Pt$–NO (1788 cm^{-1}) disappeared, while bands of Ti^{3+}–NO (1738 cm^{-1}) and v (C=O) (1725 cm^{-1}) (Halkides et al., 2002; Sobczak et al., 2008) increased and new bands assigned to Pt^0–CO (2095, 2028 cm^{-1}) (Halkides et al., 2002; Ivanova et al., 2007; Kotsifa et al., 2007; Zhang et al., 2006) formed. This suggested that the introduction of C_3H_6 led to the reduction of Pt–O to Pt^0 by C_3H_6 and the oxidation of the primarily oxidized $C_xH_yO_z$ to adsorbed CO by lattice oxygen from TiO_2; moreover, the as-formed CO species was more easily adhered to metallic Pt sites than NO at low temperature (Ivanova et al., 2007). At the same time, Ti^{3+}–NO (1738 cm^{-1}) (Kantcheva, 2001) was formed as a result of –NO spillover from Pt to the support surface (Flores-Moreno et al., 2005). Additionally, ascertainable bands attributed to –NCO (2240, 2210 cm^{-1}) (Captain and Amiridis, 1999; Sazama et al., 2005), acetonitrile (CH_3CN) (2115 cm^{-1}) (Flores-Moreno et al., 2005), acrolein (1698 cm^{-1}) (Halkides et al., 2002; Sobczak et al., 2008), v(C=C) (1660 cm^{-1}) (Halkides et al., 2002) and v_{as} (COO$^-$) (1566 cm^{-1}) (Zhang et al., 2006) adsorbed on TiO_2 support developed with time increasing.

Finally, the addition of O_2 resulted in stronger bands of v(C=O) (1725 cm^{-1}), acrolein (1698 cm^{-1}), v (C–H) (2953, 2870 cm^{-1}) (Halkides et al., 2002; Sobczak et al., 2008) and CO_2 (2366, 2348 cm^{-1}) (Halkides et al., 2002; Zhang et al., 2006). This increase can be ascribed to the catalytic activity of Pt for oxidation of C_3H_6, which is in agreement with reports elsewhere (Captain and Amiridis, 1999; Zhang et al., 2009). Bands at 1698 and 1566 cm^{-1} were assigned to the main surface $C_xH_yO_z$ species involved, i.e., acrolein and v_{as} (COO$^-$) (Captain and Amiridis, 1999; Halkides et al., 2002). It is also observed that intensity of chemisorbed Pt^{2+}–CO (2132 cm^{-1}) (Ivanova et al., 2007) increased, while $(O_{ads})Pt$–NO (1782 cm^{-1}) appeared but Pt^0–CO species vanished as a result of recovering of Pt–O sites with the admission of O_2. The formed –CO and –NO species could not only react with other intermediates separately, but also interact mutually. Based on the *in situ* DRIFTS results above and report elsewhere (Halkides et al., 2002), it is suggested that Pt^{2+}–CO could directly react with $(O_{ads})Pt$–NO (1782 cm^{-1}) and Ti^{3+}–NO species, as important steps in the SCR reaction. In our work, no significant evidence suggested that nitrates species participated directly in the SCR reaction, which was described to be an important intermediate that react with $C_xH_yO_z$ species elsewhere (Flores-Moreno et al., 2005; Kantcheva, 2001). In addition, prolonged exposure to reaction mixture leads to a modest increase of new bands 2739, 2285, 1448 and 1359 cm^{-1}, which were assigned to formate ion (Kantcheva, 2001), cyanide (Ti^{4+}–CN) (Flores-Moreno et al., 2005), v_s (COO$^-$) (Halkides et al., 2002; Kotsifa et al., 2007) and monodentate nitrate (Hadjiivanov, 2000) as reaction

intermediates. The –NCO and –CN species have also been considered as intermediates for HC–SCR in many studies (Captain and Amiridis, 1999; Halkides et al., 2002; Kotsifa et al., 2007).

Based on the discussion above, we propose the following mechanism for the low-temperature SCR:

$$O_2(g) + 2* \rightarrow 2O* \tag{1}$$
$$C_3H_6(g) + * \longrightarrow CH_n* \tag{2}$$
$$CH_n* + O* \rightarrow C_xH_yO_z* + CO* + CO_2(g) + H_2O(g) \tag{3}$$
$$NO(g) + * \longrightarrow NO* \tag{4}$$
$$NO(g) + 2* \longrightarrow N* + O* \tag{5}$$
$$NO* + O* \longrightarrow NO_2(g) + 2* \tag{6}$$
$$NO* + CH_n* \rightarrow H_2O(g) + CN* + * \tag{7}$$
$$CO* + O* \longrightarrow CO_2(g) + 2* \tag{8}$$
$$N* + CO* \longrightarrow NCO* + * \tag{9}$$
$$2CN* + O_2 \longrightarrow 2NCO* \tag{10}$$
$$CH_n* + CN* \longrightarrow CH_3CN + 2* \tag{11}$$
$$NCO* + O* \longrightarrow CO_2 + N* + * \tag{12}$$
$$N* + N* \longrightarrow N_2(g) + 2* \tag{13}$$
$$N* + NO* \longrightarrow N_2O(g) + 2* \tag{14}$$

The reactants (C_3H_6, NO and O_2) are supposed to be first adsorbed on the active Pt sites (denoted as *) over Pt/TiO_2 surface. As expressed in equations above, C_3H_6 is split to CH_n* species followed by oxidation to species of CO* and $C_xH_yO_z*$ (e.g., acrolein, ketene, formate and acetate). Meanwhile, the partially charged NO* could be further oxidized to NO_2 by O* or reduced to CN* by CH_n*. The observation of CH_3CN (2115 cm^{-1}) provides an evidence of interaction of CH_n* and CN* in the SCR reaction. Between the Pt and Ti^{4+} over Pt/TiO_2 surface, NO* spillover occurs and then Ti^{3+}-NO species is formed. The interactions of NO*, CO* and other $C_xH_yO_z$ species are key steps during the C_3H_6-SCR reaction. Accordingly, the CN* species is oxidized to surface NCO* species by O_2 from gas phase or TiO_2 lattice, which would decompose to radical N* and CO*. Finally, N_2 was formed from the combination of N*, while N_2O from N* and NO*.

3 Conclusions

The following conclusions may be drawn from the results of the present study. (1) The optimal Pt loading for low-temperature C_3H_6-SCR of NOx with Pt/TiO_2 catalyst was 0.5 wt.% and further increase of Pt loading did not affect the SCR activity. (2) Excess O_2 has positive effect on the C_3H_6-SCR reaction over Pt/TiO_2. Increasing O_2 concentration promotes the SCR of NOx rather than unselective combustion of reductant with gaseous O_2. (3) Based on the present study, NO dissociation mechanism is proposed for the low-temperature C_3H_6-SCR with Pt/TiO_2 catalyst. The interaction of Pt^{2+}–CO and adsorbed NO* species, e.g., $(O_{ads})Pt$–NO, Ti^{3+}–NO, were supposed to be one of the most important steps in the low-temperature SCR of NO by C_3H_6 in excess O_2. N_2 and N_2O might not come from the direct reduction of nitrates, but direct

interactions of NO* species with N* radicals derived from NO dissociation and –NCO decomposition.

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