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Nature of the surface species on Ag/Al_2O_3 catalyst in SCR of NO by propene under lean-burn condition through temperature programmed technique

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Abstract

The surface species occurring on Ag/Al_2O_3 in SCR of NO by propene were characterized using temperature programmed techniques. An experimental set of three steps containing the step of production of the SCR species, the temperature programmed desorption (TPD) step and the temperature programmed oxidation (TPO) step shows that there are at least five groups of surface species on Ag/Al_2O_3. These species consist of two main types. The weak adsorbed species (the TPD species) decompose during TPD step while the strong adsorbed species (the TPO species) are necessarily oxidized with some oxidants in TPO step. The reactivity of all the surface species with NO + O_2 was also studied to identify which species is an intermediate. The reactivity results suggest that some of the TPO species is more important to the reduction of NO to produce N_2. Some of the TPD species is an inhibiter for C_3H_6-SCR of NO on low loading Ag/Al_2O_3 but become an assisting intermediate on high-loading catalyst.

Keywords: Ag/Al_2O_3; SCR of NO; TPD; TPO and surface species

1. Introduction

A silver-based formulation is an effective catalyst for NO_x removal from exhaust gases under lean-burn conditions. It shows a good activity and selectivity for the SCR of NO by various hydrocarbons, e.g., ethanol [1–3], octane [4] and propene [5-7]. Also, several studies investigated the water- and sulfur-resistance of the silver-based material. The catalyst activity is little diminished by water but this inhibition is fully reversible [8]. Most of researchers reported that the introduction of SO_2 into the feed stream reduces significantly and permanently the NO reduction activity [9,10]. On the contrary, Angelidis et al. [11] found a promotional effect on the catalyst activity when 50 ppm of SO_2 was present during the C_3H_6-SCR of NO. At the present time, although many authors have reported to advantages of the silver-based catalyst in the HC-SCR of NO under excess oxygen, the reaction mechanism and active intermediate species are rather complicated and have not been fully elucidated. Typically, application of in situ Fourier transform infrared spectroscopy has been only used to predict the reaction mechanism and identify the intermediates [12–20]: Meunier et al. [14] proposed the different mechanism dependent on types of Ag phase. Large Ag^0 particles likely observed on high-loading silver catalyze the decomposition-reduction of NO whereas Ag^+ species prevailing on low-loading sample favor the oxidation of NO to ad-NO_x species, which subsequently react through the intermediacy of organonitrogen compounds. The possible simultaneous occurrence of at least two reaction pathways results in complication of the analysis and therefore other procedures have to be necessarily made in order to obtain more information. In the previous work, our research group has

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employed temperature programmed technique to investigate nature of the surface species in the SCR of NO by C_3H_6 over zeolite- and platinum-based catalysts [21–23]. This method is very promising to indicate classification of the surface species via decomposition and oxidation characteristics. Hence, in this work we have applied temperature programmed techniques to study nature and reactivity of the surface species on Ag/Al_2O_3 in the SCR of NO by C_3H_6 under lean-burn condition.

2. Experimental

Ag/Al_2O_3 catalysts were prepared by incipient wetness impregnation of a γ-Al_2O_3 obtained from Sumitomo Aluminum Smelting (type NKH-3) with silver nitrate ($AgNO_3$) obtained from Aldrich. The solution used for the dry impregnation was obtained by mixing an appropriate amount of $AgNO_3$ in a volume of deionised water equal to the porous volume of the alumina. The silver loadings were fixed as 2 and 10 wt.% on a dry basis. After impregnation, the resulting samples were died at 110 °C overnight and calcined in air at 600 °C for 2 h.

All experiments were performed in a quartz tube with a 6 mm inside diameter. This tube was filled with 0.4 g of catalyst held in place with quartz wool. The tube was placed inside an electrically heated sleeve furnace equipped with a temperature controller. Temperature ramping for temperature programmed reaction (TPR), temperature programmed desorption (TPD) and temperature programmed oxidation (TPO) experiments employed a variac coil.

Composition of the reactor off gases was measured chromatographically using two 6 port sampling valves and two TCD GCs (SHIMADZU GC 8A), one equipped with a molecular sieve 5A column (for separating O_2, N_2 and CO) and the other with a Porapak QS column (for separating CH_4, CO_2, N_2O and C_3H_6).

Temperature programmed reaction was performed to observe catalyst activity as a function of temperature. Feed gas contained 1000 ppm NO, 1000 ppm C_3H_6 and 5 vol.% O_2 at GSHV of 16000 h^{-1}. Carrier gas was helium. These compositions are typical of diesel or lean-burn gasoline exhausts, except that the oxygen concentration is at the lower limit of the range observed. The reaction gases were introduced to the reactor whose temperature was raised from ambient temperature to 600 °C stepwise. At each step, the catalyst bed was held at constant temperature until steady state was reached. This was achieved within 20 min.

The nature of surface species on the catalyst exposed to the reactants was investigated by an experiment consisting of three steps: (1) SCR of NO by C_3H_6 to produce the surface species, (2) temperature programmed desorption to remove as much of the adsorbed surface species as possible and (3) temperature programmed oxidation to remove any remaining deposits. Dosing step was carried out at operating temperature above the onset temperature for C_3H_6 oxidation in the simulated exhaust gases for our system, but below the temperature at which combustion goes to completion. Feed gas was similar to that of the TPR experiment. After dosing the catalyst in the first step for 2 h, the catalyst was immediately flushed with He at the dosing temperature and then cooled to room temperature. During TPD step, temperature was ramped at a constant 5 °C/min to 800 °C under 50 ml/min He flow. On completion of the TPD experiment, a TPO run using 1 vol.% O_2 in He was carried out to determine if there were any residual carbonaceous materials on the catalyst.

Reactivity of the surface species with an oxidizing gas was also examined in order to identify that the hypothesized surface species are whether intermediates or spectators. It was evaluated using a TPO technique after production of the surface species through dosing with a gas mixture of C_3H_6 + NO + O_2. Typically, a gas mixture of 1000 ppm NO and 1% O_2 was used in the TPO experiment.

Gases used for all experiments were ultra high purity. Mixtures for the dosing and TPD–TPO experiments were prepared from ultrapure sources by Thai Industrial Gases. Three mixtures were used: (1) 10% O_2, (2) 1% NO and (3) 3% C_3H_6, all in He. These gases were monitored and mixed prior to entering the bed of catalyst.

Reproducibility of the experimental results was tested by repeating all the TPR and the 3-steps experiments. Results for the former experiment were identical and just minor difference in TPD–TPO peak heights was observed in the latter.

3. Results and discussion

Before considering nature of the surface species, TPR of C_3H_6 and NO conversions was made. TPR results are shown in Fig. 1 for the 2 wt.% Ag/Al_2O_3 and in Fig. 2 for the 10 wt.% Ag/Al_2O_3. The 2 wt.% Ag/Al_2O_3 is a more effective catalyst but is active at a higher temperature than the 10 wt.% Ag/Al_2O_3. The overall NO reduction conversion reaches a sharp maximum of approximately 90% at

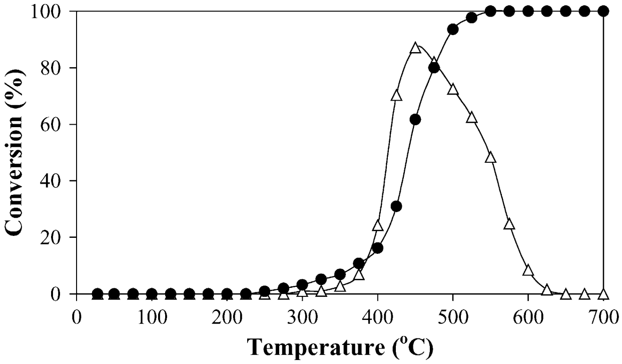

Fig. 1. The temperature programmed reaction profiles of 2 wt.% Ag/Al_2O_3 for the selective catalytic reduction of NO by C_3H_6 under excess oxygen: (●) C_3H_6 conversion and (△) NO conversion.

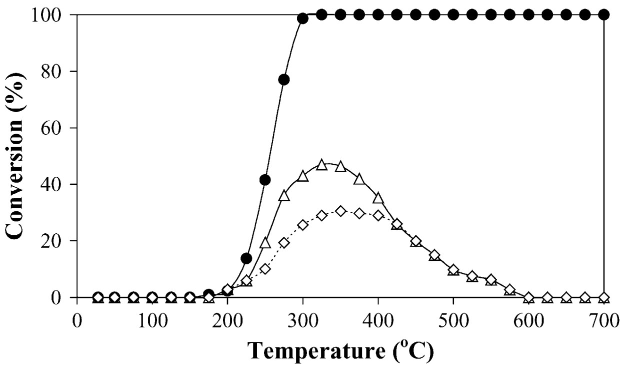

Fig. 2. The temperature programmed reaction profiles of 10 wt.% Ag/Al_2O_3 for the selective catalytic reduction of NO by C_3H_6 under excess oxygen: (●) C_3H_6 conversion, (△) NO conversion and (◇) NO conversion to N_2.

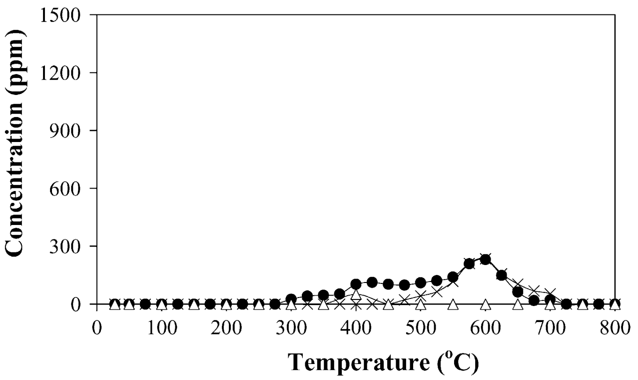

Fig. 3. The temperature programmed desorption profiles of 2 wt.% Ag/Al_2O_3 after dosing 1000 ppm C_3H_6 + 1000 ppm NO + 5% O_2 at 370 °C for 2 h: (△) N_2, (●) CO_2 and (×) CH_4.

450 °C and 50% at 350 °C for the 2 and 10 wt.% Ag catalysts, respectively. The main products are CO_2 and N_2. No CO is observed while N_2O product occurs on only high-loading silver material. There is some variation of selectivity to N_2O with changing temperature. It is approximately in the range of 15–50% between 250 and 400 °C. Both catalysts show coincidence of the onset of NO reduction and C_3H_6 oxidation. Nevertheless, there is also some difference in the behavior patterns of both TPR profiles. Maximum NO_x conversion is coincident with 100% C_3H_6 conversion for the 10 wt.% Ag/Al_2O_3 but not for the 2 wt.% Ag/Al_2O_3. The low-loading silver shows occurrence of the maxima for total NO conversion at approximately 100 °C before combustion approaches to completion. This phenomenon is in agreement with the results reported by several authors [7,8,19].

3.1. Characteristics of the surface species

Series of the 3-steps experiments were undertaken to identify the active intermediate species and proposed the possible reaction pathways in the C_3H_6-SCR of NO over Ag-based catalysts. The composition of the effluent gases in these experiments would be expected to provide some insight into the nature of the adsorbed species on the catalyst surface. However, to prevent elimination of all the intermediates by O_2 during the production step of the surface species, in the 3-steps experiments, the surface species have to be introduced at a temperature above an onset temperature for C_3H_6 oxidation, but below the temperature at which combustion goes to completion, that is, between 325 and 550 °C for the 2 wt.% Ag/Al_2O_3 and between 200 and 300 °C for the 10 wt.% Ag/Al_2O_3 as seen in Figs. 1 and 2. Because of this, the dosing temperatures of 370 and 210 °C in the first step of the 3-steps experiments are used for the 2 wt.% and the 10 wt.% Ag/Al_2O_3, respectively.

After exposure the Ag catalysts to a gas mixture of C_3H_6, NO and O_2 at a given temperature, there appears gas evolution during TPD and TPO steps, unlike zeolite- and platinum-based catalysts as reported previously elsewhere [21–23]. The surface species on Pt formulation are almost totally released during TPD step whereas those on metal ion exchange ZSM-5 are necessarily removed by the oxidant on TPO step. Figs. 3 and 4 show gas evolution from surface of the 2 wt.% Ag/Al_2O_3 dosed with reactants (C_3H_6 + NO + O_2) at 370 °C for 2 h during TPD and TPO steps, respectively. These effluent gases contained CO_2, CH_4 and N_2 in the TPD step and only CO_2 and N_2 in the TPO step. Other species expected to be present such as NO, NO_2 and NH_3 were not observed in this experiment due to limitations of our analysis. Zuzaniuk et al. [17] reported production of NO_2 and NH_3 in oxidation of nitromethane and *tert*-butyl nitrite, which were assigned as models of possible intermediates. Considering the TPD profile, the effluent gases are obtained by decomposition process of some surface species, perhaps intermediates. A 600 °C CO_2 peak occurs at about the same temperature as a CH_4 peak. There is in addition a broad CO_2 peak appearing at approximately 400 °C coincident with a very little N_2 peak. The TPO profile shows oxidation characteristic of the remaining deposits. We also remind that these deposits cannot decompose under He atmosphere at below 800 °C. Three large CO_2 peaks are centered at 625, 700 and 775 °C. There

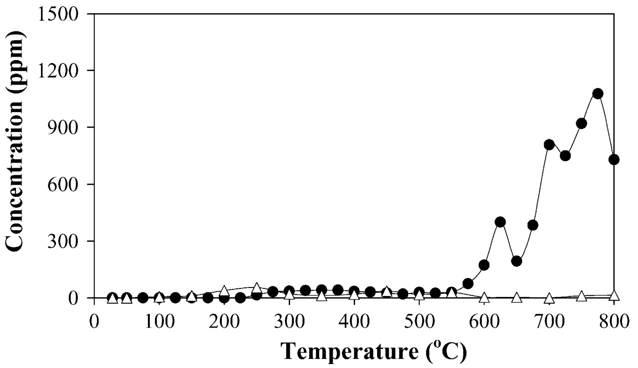

Fig. 4. The temperature programmed oxidation profiles of 2 wt.% Ag/Al_2O_3 by using 1% O_2 as the oxidizing gas after the two steps, reaction and TPD steps: (△) N_2 and (●) CO_2.

is in addition very small signal of CO_2 and N_2 at below 500 °C, but probably significant.

We speculate that each peak should be assigned to decomposition or oxidation of at least one surface species, but possibly two or more may be involved. However, when there is a coincidence of peaks both probably arise from the same single surface species. Deconvolution of TPD profile suggests that at least two surfaces species on catalyst surface can decompose even in an inert gas. Since their structure and composition are as yet unknown, we designate these species as S_{TPDL} and S_{TPDH}. The former species is associated with the CO_2 and N_2 peaks at about 400 °C, while the latter species appears related to the CO_2 and CH_4 peaks at 600 °C. Nitrogen is present only in the structure of S_{TPDL}. Chafik et al. [24] found that cyanide (CN) and isocyanate (NCO) on Ag/Al_2O_3 disappeared distinctly when flushed with He flow at about 350 and 500 °C, respectively, and also that formate species was easily decomposing above 500 °C. These are consistent with our discovery that S_{TPDL} and S_{TPDH} can decompose under He atmosphere in the temperature range of 300–500 °C and above 500 °C, respectively. Importantly, observation of cyanide and isocyanate as reported by Chafik et al. [24] relative with behavior of S_{TPDL} in our result is evident that only one peak observed in temperature programmed experiment can represent to more than one functional group mode of surface species appearing in IR spectra. For the TPO profile, there are at least three surface species being oxidized by O_2 at high temperature. To accommodate for discussion, we combine the three species to be only one group and designate a group of these species as S_{TPOH}.

Because of the results of Meunier et al. [14] mentioned above, nature of the surface species on the high-loading Ag catalyst was additionally studied in order to fulfill our information. Gas evolutions during TPD and TPO experiments of the 10 wt.% Ag/Al_2O_3 exposed to a gas mixture of C_3H_6, NO and O_2 at 210 °C for 2 h are shown in Figs. 5 and 6, respectively. Clearly, there are some differences in TPD–TPO patterns compared to the low-loading Ag catalyst. Considering gas evolution in the TPD profiles (see Fig. 5), there are two CO_2 peaks at 325 and 575 °C as well as a shoulder at ca. 450 °C while N_2 and CH_4 show only one peak at 275 and 575 °C, respectively. The 575 °C carbon-containing species (coincidence of CO_2 and CH_4 peaks) seems to show the same characteristic of S_{TPDH} on low-loading silver but much more production. The N_2 peak is a 125 °C shift from the experiment of the low-loading silver. This is relative with a shift of the 325 °C CO_2 peak although just 75 °C of the maximum CO_2 position is shifted. However, we believe that these N_2 and CO_2 decompose from the same sort of surface species. Two possible reasons are pronounced to explain the shift of the N_2 and CO_2 peaks. First, these species are actually S_{TPDL} but either their decomposition behavior or structural composition is changed. An example is change of the decomposition temperature of isocyanate species if this species occurs on the different catalyst surface. Over 2 wt.% Ag/Al_2O_3 the isocyanate species disappeared completely at about 500 °C [24] while it vanished definitely at just 350 °C on 0.8 wt.% Pt/Al_2O_3 [25]. The second reason is occurrence of new surface species definitely independent on S_{TPDL}. Iglesias et al. [18] reported about the different of surface species for high and low silver loading. That significant difference was identification of cyanide species appearing initially at 300 °C on high silver loading. However, in this work we have identified the TPD N-containing species as S_{TPDL} and speculate that the decomposition temperature of this species can nearly follow the temperature where the maximum NO conversion of TPR appears. In addition to encounter the S_{TPDL} and the S_{TPDH} on the 10 wt.% Ag/Al_2O_3, we also found a shoulder of CO_2 at 400 °C and designated this new species to be the S_{TPDM}. The characteristics of S_{TPDM} are close to those of the aldehyde species observed by Chafik et al. [24]. They reported that the aldehyde group disappeared distinctly at below 500 °C under He atmosphere. Interestingly, some behaviors of all the TPD surface species covered on the high-loading Ag/Al_2O_3 are coincident with the three surface species prevailing on the Pt/Al_2O_3 as reported in the previous work [21].

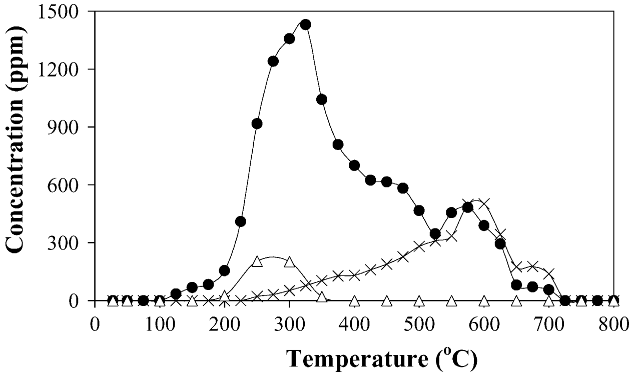

Fig. 5. The temperature programmed desorption profiles of 10 wt.% Ag/Al_2O_3 after dosing 1000 ppm C_3H_6 + 1000 ppm NO + 5% O_2 at 210 °C for 2 h: (△) N_2, (●) CO_2 and (×) CH_4.

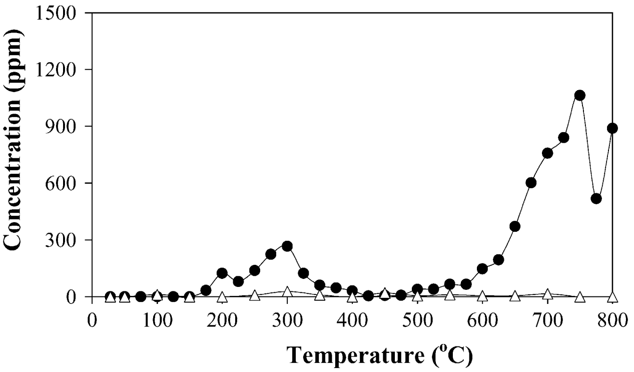

Fig. 6. The temperature programmed oxidation profiles of 10 wt.% Ag/Al_2O_3 by using 1% O_2 as the oxidizing gas after the two steps, reaction and TPD steps: (△) N_2 and (●) CO_2.

The TPO profile in Fig. 6 shows the presence of a group of S_{TPOH} on the high-loading silver similar to the low-loading one. However, there are in addition to some of CO_2 and N_2 signals at low temperature (300 °C) as well. This species is assigned as S_{TPOL}.

Certainly, some of surface species may be the intermediates but some becomes the spectator. Therefore, all the surface species need to be tested the reactivity with a gas mixture of NO + O_2. The species, which can be decomposing or oxidized at a relative reaction temperature, is possible to be an intermediate.

3.2. The reactivity of surface species

The reactivity of surface species with oxidizing reactant gases was studied to identify their characteristics. The reactivity was evaluated using a TPO technique. Before the reactivity tests, all the surface species were produced by dosing a gas mixture of C_3H_6 + NO + O_2 at 370 and 210 °C for 2 and 10 wt.% Ag/Al_2O_3, respectively. Some of these experiments were conducted after a TPD step in order to obtain the specific reactivity of only TPO surface species.

To obtain the reactivity of the TPD surface species, the TPO experiment by O_2 without a TPD step was first made. Fig. 7 shows the traces of outlet gases (CO_2 and N_2) obtained from oxidation by O_2 of the surface species on 2 wt.% Ag/Al_2O_3 catalyst after exposed to C_3H_6 + NO + O_2. It is found that the position of CO_2 peak addressed as S_{TPDL} was unaffected with introducing O_2 while the CO_2 and CH_4 peaks addressed as S_{TPDH} disappeared. We speculate that S_{TPDH} should be oxidized by O_2 and transformed to be CO_2 centered at 425 °C. It is notable that appearance of the N_2 signal centered at 400 °C is doubtable. However, we propose that it may be transfigured from the undetectable N-containing compounds (NO, NO_2 and NH_3) as pronounced above. This result reveals that S_{TPDL} still decomposes even in a large excess of oxygen at the same temperature where it decomposes under He flow. On the other hand, S_{TPDH} decomposes at high temperature but it is easily oxidized by O_2 at lower temperature.

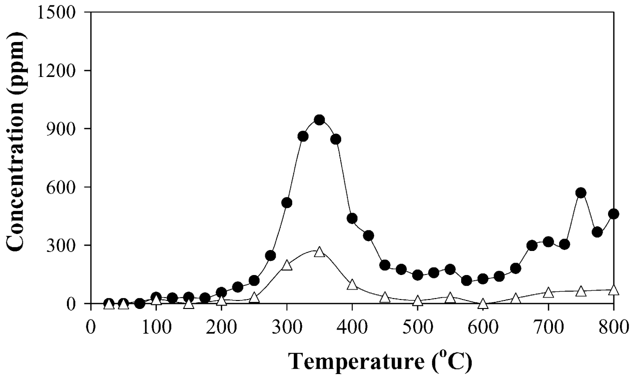

Fig. 8. The temperature programmed oxidation profiles of 2 wt.% Ag/Al_2O_3 by using NO+O_2 as the oxidizing gas after dosing 1000 ppm C_3H_6 + 1000 ppm NO + 5% O_2 at 370 °C for 2 h without TPD: (△) N_2 and (●) CO_2.

The reactivities of surface species with NO + O_2 after TPD step and without TPD step are shown in Figs. 8 and 9, respectively. A large amount of CO_2 released at low temperature relative with disappearance of a group of the CO_2 peaks assigned as S_{TPOH} implies that the S_{TPOH} is reactive with NO + O_2. However, the remainder of CO_2 peak at high temperature reveals that some of S_{TPOH} should be inactive with NO+O_2. The reactivity of S_{TPOH} is in agreement with the results of Shimizu et al. [26] and Meunier et al. [14]. Shimizu et al. [26] reported that acetate, which was rather stable and hardly oxidized in O_2 atmosphere, was reactive in NO + O_2. In addition, Meunier et al. [14] suggested that nitrate species could still be observed over 600 °C in the SCR condition. In addition to the partial reactivity of S_{TPOH}, this reactive species is easily removed by NO + O_2 at the lower temperature when the TPD step is ahead employed (compared Figs. 8 and 9). This indicates that coverage of the TPD species on catalyst surface may inhibit strongly the reaction of the TPO species.

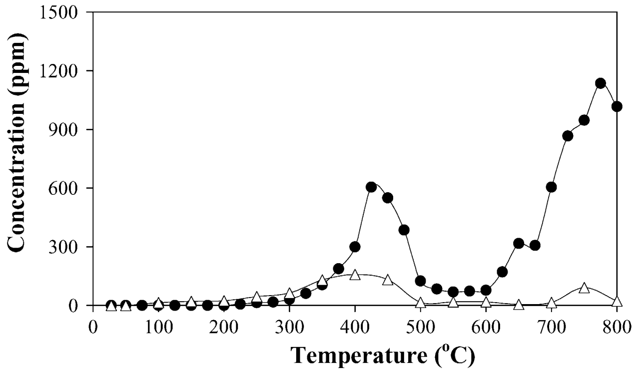

Fig. 7. The temperature programmed oxidation profiles of 2 wt.% Ag/Al_2O_3 by using 1% O_2 as the oxidizing gas after dosing 1000 ppm C_3H_6 + 1000 ppm NO + 5% O_2 at 370 °C for 2 h without TPD: (△) N_2 and (●) CO_2.

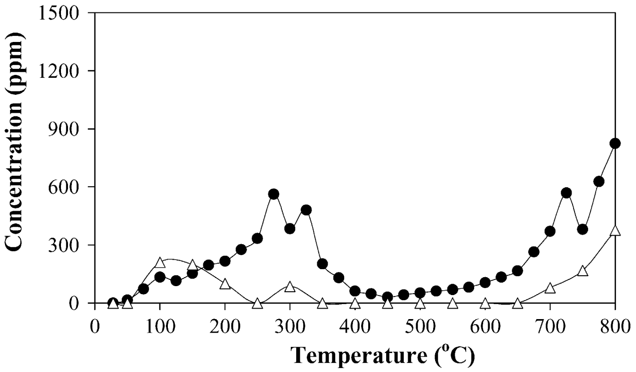

Fig. 9. The temperature programmed oxidation profiles of 2 wt.% Ag/Al_2O_3 by using NO+O_2 as the oxidizing gas after dosing 1000 ppm C_3H_6 + 1000 ppm NO + 5% O_2 at 370 °C for 2 h and subsequently TPD: (△) N_2 and (●) CO_2.

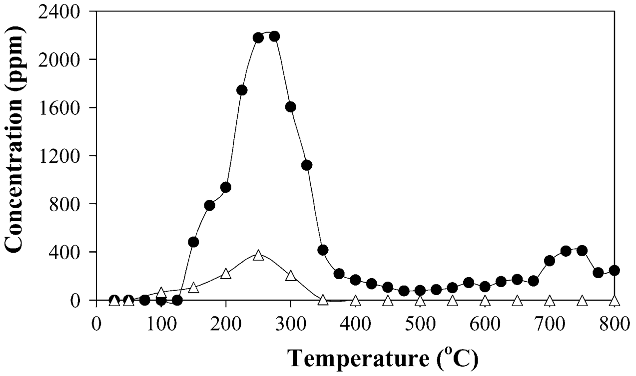

Fig. 10. The temperature programmed oxidation profiles of 10 wt.% Ag/ Al_2O_3 by using NO+O_2 as the oxidizing gas after dosing 1000 ppm C_3H_6 + 1000 ppm NO + 5% O_2 at 210 °C for 2 h without TPD: (△) N_2 and (●) CO_2.

Unfortunately, for the low-loading silver catalyst we cannot determine that weather S_{TPOL} is reactive with NO + O_2 or not because a little amount of this species is released in TPO step at low temperature. Also, it is hard to clarify the reactivity behavior of the TPD surface species.

The reactivity tests of surface species over 10 wt.% Ag/ Al_2O_3 were made to give the more information. Figs. 10 and 11 show the reactivities of surface species with NO + O_2 without TPD step and after TPD step. Considering the reactivity of TPD species, the results exhibit clearly that S_{TPDM} and S_{TPDH} disappeared when exposure to NO + O_2. For S_{TPDL} species, we found that position of the CO_2 peak is slightly affected with introducing NO+O_2 as the oxidizing gas. In addition, the deconvolution of CO_2 profile obtained from the reactivities of surface species without TPD step shows peaks at 200 and 250 °C. The coincidence of CO_2 and N_2 peaks at 250 °C is similar to the characteristic of S_{TPDL} species. Therefore, it is speculated that a group of S_{TPDL} species was hardly oxidized with NO + O_2. In case of TPO species, the same result with low loading was observed. The disappearance of S_{TPOH} species when exposure to NO + O_2 indicates that this surface species is easily removed in NO + O_2 atmospheres. However, the reactivity test of S_{TPOL} species can not explain the behavior of this species because the reactivity of S_{TPOH} appears at the same temperature of occurring S_{TPOL} species.

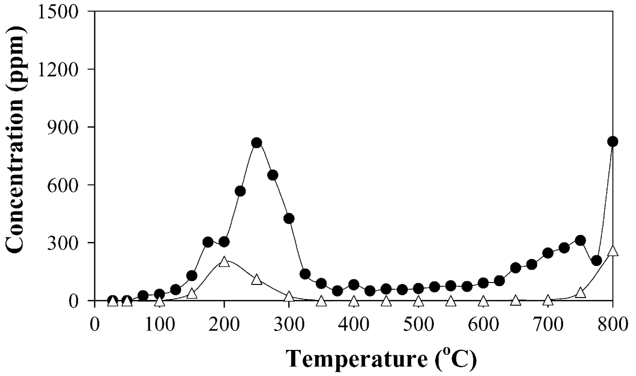

Fig. 11. The temperature programmed oxidation profiles of 10 wt.% Ag/ Al_2O_3 by using NO+O_2 as the oxidizing gas after dosing 1000 ppm C_3H_6 + 1000 ppm NO + 5% O_2 at 210 °C for 2 h and subsequently TPD: (△) N_2 and (●) CO_2.

Importantly, it should be noted that the different behavior of surface species between low and high-loading silver was observed. For 2% Ag/Al_2O_3, we found that carbon dioxide and nitrogen released during TPO step by NO + O_2 after pretreatment in TPD step (Fig. 7) were shifted to lower temperature when compared with the temperature of outlet gases released during TPO step by NO + O_2 without TPD step (Fig. 8). It may be possible that the reaction of TPO surface species was inhibited by TPD surface species, because TPD surface species are more active with oxidizing gas than TPO surface species. On the other hand, in case of high-loading, the surface species was reactivated to the same temperature (250 °C) both experiments.

Certainly, we can speculate that all surface species involve the formation of N_2 and CO_2 in SCR on Ag/Al_2O_3 because these species were reacted with reactant gases at reaction temperature. However, there are only two surface species, S_{TPDL} and S_{TPOH}, play an important role to produce N_2. From the above results, there are at least two reaction pathways dependent on characteristic of surface species and temperature of reaction [14,17]. The parallel reaction mechanisms are decomposition and NO reduction of surface species. First, S_{TPDL} species decompose and transform to nitrogen and carbon dioxide. Second, the S_{TPOH} species is oxidized by NO + O_2 and O_2.

4. Conclusions

The use of temperature programmed techniques in order to investigate the decomposition and oxidation characteristics of the SCR surface species on Ag-containing catalyst is a promising procedure. The results show coverage of at least five types of surface species on catalyst surface. These consist of three TPD species (S_{TPDL}, S_{TPDM} and S_{TPDH}) and two TPO species (S_{TPOL} and S_{TPOH}). All of them appear on the high-loading Ag/Al_2O_3 whereas only S_{TPDL} S_{TPDH} and S_{TPOH} are distinctly observed on the low-loading one. Some of S_{TPOH} seems to be only intermediate in C_3H_6-SCR of NO on low-loading Ag/Al_2O_3. The TPD species (S_{TPDL} and S_{TPDH}) show the inhibiting behavior to the reaction of S_{TPOH}. On the other hand, either S_{TPDL} or some of S_{TPOH} is cooperative intermediates in the reaction pathway on high-loading Ag/Al_2O_3: decomposition of S_{TPDL} and NO reduction by S_{TPOH}.

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