

In situ DRIFTS study of the selective reduction of NO_x with alcohols over $\text{Ag}/\text{Al}_2\text{O}_3$ catalyst: Role of surface enolic species

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Abstract

In situ diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) was used to clarify the formation and reactivity of enolic species and its role in the formation of the isocyanate species ($-\text{NCO}$) during the selective catalytic reduction of NO_x (SCR of NO_x) by alcohols (CH_3OH , $\text{C}_2\text{H}_5\text{OH}$ and isopropyl alcohol (IPA)) over $\text{Ag}/\text{Al}_2\text{O}_3$ catalyst. The enolic species, which was derived from the partial oxidation of $\text{C}_2\text{H}_5\text{OH}$ and IPA in the presence of O_2 over $\text{Ag}/\text{Al}_2\text{O}_3$, had a high reactivity with $\text{NO} + \text{O}_2$, resulting in a high surface concentration of $-\text{NCO}$ species and high efficiency of NO_x reduction when using $\text{C}_2\text{H}_5\text{OH}$ or IPA as reductants. In contrast, CH_3OH was much less effective for the SCR of NO_x . The order of the facility for the formation of enolic species and $-\text{NCO}$ species was in good agreement with the order of the activities in the SCR of NO_x process ($\text{C}_2\text{H}_5\text{OH} > \text{IPA} > \text{CH}_3\text{OH}$) in the moderate temperature range of 523–623 K.

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1. Introduction

The SCR of NO_x in the presence of excess O_2 can be applied to the removal of gaseous pollutants from diesel and lean-burn engines. In recent studies [1–10], alumina-supported silver catalysts (denoted as $\text{Ag}/\text{Al}_2\text{O}_3$), which are relatively durable and inexpensive, have been considered to be a candidate for practical use. In particular, $\text{C}_2\text{H}_5\text{OH}$ is extremely effective for the SCR of NO_x over $\text{Ag}/\text{Al}_2\text{O}_3$ even in the presence of water and SO_2 [1,4,5].

So far, several reaction mechanisms for NO_x reduction including the intermediates (such as NO_3^- , CH_3COO^- , $\text{R}-\text{NO}_2$, $-\text{CN}$ and $-\text{NCO}$ species, etc.) have been proposed [4–7,11–18]. Among these mechanisms, $-\text{NCO}$ species has often been proposed as a crucial intermediate. Our previous studies as well as other research works have also proved that the formation of $-\text{NCO}$ species and its effective reaction with NO or $\text{NO} + \text{O}_2$ are key steps in the SCR reaction [4,5,11,15,17]. We recently have proposed a novel

mechanism of the SCR of NO_x by $\text{C}_2\text{H}_5\text{OH}$ [19], where the surface enolic species related with the high surface concentration of $-\text{NCO}$ and the high efficiency of NO_x reduction by $\text{C}_2\text{H}_5\text{OH}$. However, other alcohols (such as IPA and CH_3OH , etc.) have not been proved to follow the same mechanism during the SCR of NO_x .

In this paper, we use in situ DRIFTS method to study the behavior of surface species on $\text{Ag}/\text{Al}_2\text{O}_3$ during the SCR of NO_x using $\text{C}_2\text{H}_5\text{OH}$, IPA and CH_3OH as reductants. Particular attention is given to the surface enolic species, which formed from the partial oxidation of alcohols during the SCR of NO_x . The results could provide new insights into the mechanisms for the SCR of NO_x by alcohols.

2. Experimental

2.1. Catalyst preparation

$\text{Ag}/\text{Al}_2\text{O}_3$ (5 wt.%) catalyst was prepared by an impregnation method as following: Al_2O_3 powder (200 m^2/g) was

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dissolved in an appropriate amount of silver nitrate aqueous solution. The sample was dried at 393 K for 3 h and calcined at 873 K for 3 h in air.

2.2. Activity measurements

The catalytic activity was measured in a fixed-bed reactor by passing a gaseous mixture of NO (800 ppm), reductant (C_2H_5OH 1565 ppm or IPA 1200 ppm or CH_3OH 2250 ppm) O_2 (10%) in N_2 balance at a total flow rate of 2000 mL/min. $W/F = 0.018 \text{ g s cm}^{-3}$ (GHSV $\sim 50,000 \text{ h}^{-1}$). NO_x conversion was analyzed on-line by a chemiluminescence $NO/NO_2/NO_x$ analyzer (42C-hl, Thermo Environmental). An aqueous C_2H_5OH (or IPA or CH_3OH) solution was supplied with a micropump into the gas stream and vaporized by a coiled heater at the inlet of the reactor. Analysis of the gas products was carried out using a NEXUS 670-FTIR spectrophotometer fitted with a gas cell of volume 2 dm^3 (all spectra were measured with a resolution of 1 cm^{-1} and with an accumulation of 32 scans) and gas chromatography (Agilent 6890N GC) coupled to a mass spectrometer (Agilent 5973N MS) with a column-containing HP-PLOT Q (Agilent $30 \text{ m} \times 0.320 \text{ mm}$).

2.3. In situ DRIFTS procedure

In situ DRIFTS spectra were recorded on a NEXUS 670-FTIR equipped with a smart collector and a MCT/A detector cooled by liquid N_2 . The catalyst was finely ground and placed in a ceramic crucible. Prior to each experiment, the catalyst was firstly heated in a flow of 10 vol% $O_2 + N_2$ for 60 min at 573 and 873 K, respectively, then cooled to the desired temperature and a spectrum of the catalyst in the flow of $N_2 + O_2$ served as the background was recorded. All gas mixtures were fed at a flow rate of 300 mL/min. All spectra were measured with a resolution of 4 cm^{-1} and with an accumulation of 100 scans.

3. Results and discussion

3.1. Reaction activity of various alcohols for the SCR of NO_x over Ag/Al_2O_3 catalyst

Fig. 1 shows the conversion of NO_x over Ag/Al_2O_3 catalyst for the various reaction systems ($NO-O_2$ -reductant). When using C_2H_5OH as a reductant, the maximal conversion of NO_x was up to 99.7% with a very broad active temperature window (597–747 K). In the case of IPA, it is worthwhile to note that the NO_x conversion was nearly the same as that of C_2H_5OH within the temperature range of 623–773 K. In contrast, there was a relatively lower NO_x conversion by CH_3OH in the temperature range of (547–691 K) and the highest conversion of NO_x was only 21.9%. These results indicate that the NO_x conversion is strongly influenced by the kinds of alcohols and the order of the

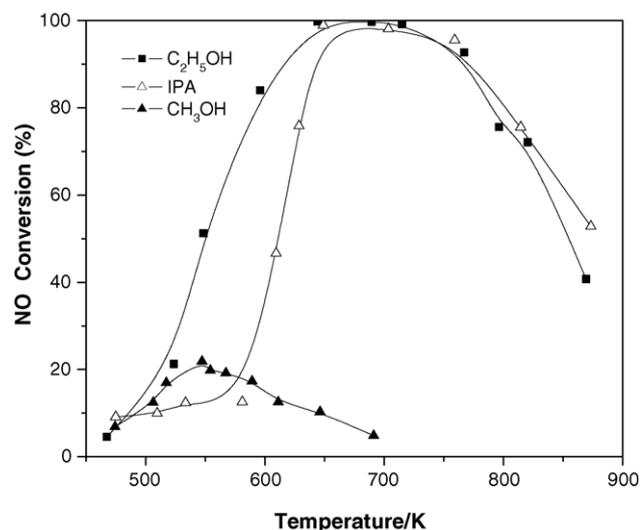


Fig. 1. Catalytic activity for NO_x reduction with different alcohols over Ag/Al_2O_3 catalyst at various temperatures. Conditions: NO 800 ppm; CH_3OH 2250 ppm or C_2H_5OH 1565 ppm or IPA 1200 ppm; O_2 10% in N_2 balance at total flow rate 2000 mL/min. $W/F = 0.018 \text{ g s cm}^{-3}$ (GHSV $\sim 50,000 \text{ h}^{-1}$).

effective reactivity is $C_2H_5OH > IPA > CH_3OH$ in the moderate temperature range of 523–623 K.

The main products of the SCR of NO_x over Ag/Al_2O_3 catalyst are N_2 and CO_2 . The diversity of by-products using different alcohols as reductants only could be seen at low temperatures, so we measure the by-product distributions at 473 K. GC-MS alone would not permit us to distinguish CO_2 from N_2O , N_2 from CO ; on the other hand, N_2 is not IR active, and hence could not be detected. As a result, both the two analysis systems are used in our experiments. From FTIR gas spectra, we detected small amount of N_2O ($<30 \text{ ppm}$) and traces of HCN and NH_3 . GC-MS analysis was used to detect other nitrogen-containing species, which were not detectable by FTIR. In the reduction of NO_x with C_2H_5OH (Fig. 2A), mainly N_2 and CO_2 were produced and the reduction was accompanied by side reactions leading to the formation of acetaldehyde (CH_3CHO , 200 ppm) along with 120 ppm unreacted C_2H_5OH and small amount of nitrogen-containing compounds such as N_2O and CH_3NO_2 . During the reduction of NO_x by IPA, acetone (CH_3COCH_3) was the main side product, together with other minor by-products such as C_2H_5OH , CH_3CHO , N_2O and CH_3NO_2 . Traces of HCN and C_3H_6 were also present (Fig. 2B). As for CH_3OH (Fig. 2C), the main products were CO_2 and N_2 , accompanied by small amount of unreacted CH_3OH . No by-products could be detected at 473 K and higher temperatures.

3.2. In situ DRIFTS study of the SCR of NO_x by different alcohols over Ag/Al_2O_3 catalyst

The difference of these three alcohols for the SCR of NO_x over the Ag/Al_2O_3 was investigated by DRIFTS method. Fig. 3 shows the in situ DRIFTS spectra of Ag/Al_2O_3

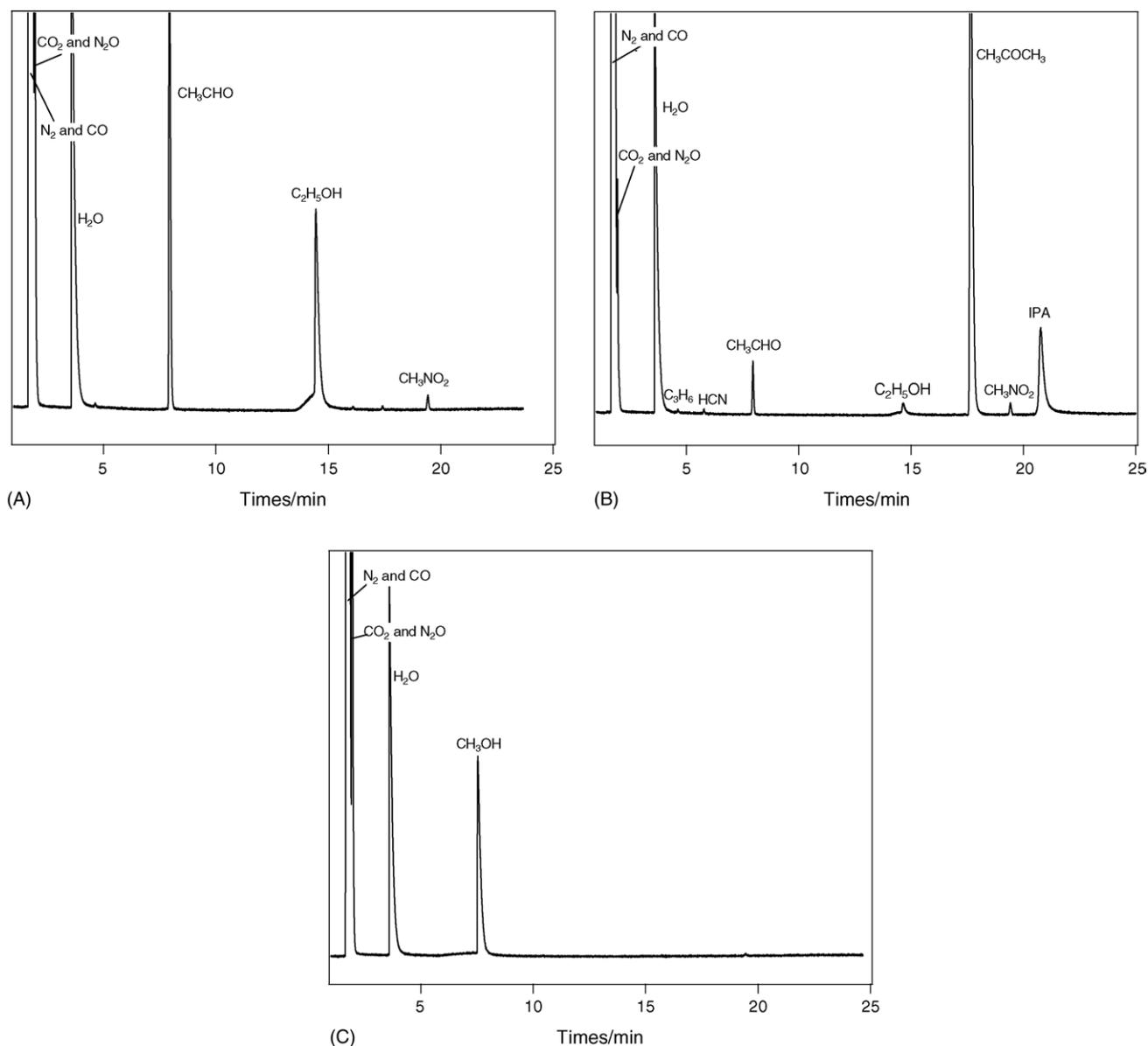


Fig. 2. (A) GC–MS chromatogram of gas products of $C_2H_5OH/NO/O_2$ reaction at 473 K on Ag/Al_2O_3 catalyst. Conditions: NO 800 ppm; C_2H_5OH 1565 ppm; O_2 10%. (B) GC–MS chromatogram of gas products of IPA/ NO/O_2 reaction at 473 K on Ag/Al_2O_3 catalyst. Conditions: NO 800 ppm; IPA 1200 ppm; O_2 10%. (C) GC–MS chromatogram of gas products of $CH_3OH/NO/O_2$ reaction at 473 K on Ag/Al_2O_3 catalyst. Conditions: NO 800 ppm; CH_3OH 2250 ppm; O_2 10%.

catalyst in the flow of $NO + C_2H_5OH + O_2$ in steady state at various temperatures. Based on the previous study [19], the bands at 1633, 1416 and 1336 cm^{-1} are assignable to the enolic species and the peaks at 1578 and 1464 cm^{-1} are associated with acetate species, whereas the bands at 1585 and 1304 cm^{-1} are due to adsorbed nitrates. In addition, a very strong peak appeared at 2233 cm^{-1} , which is assignable to $-NCO$ species. During $NO + IPA + O_2$ reaction over Ag/Al_2O_3 catalyst (Fig. 4), the characteristic peaks are similarly assigned to enolic species (1633 , 1412 and 1338 cm^{-1}), acetate species (1579 and 1468 cm^{-1}) and nitrates (1585 and 1302 cm^{-1}). The shoulder peak at 1612 cm^{-1} was also assigned to bridge adsorbed surface nitrate species [7,15,19]. The obvious difference is the

appearance of the formate bands at 1591 and 1382 cm^{-1} , while the peak at 1392 cm^{-1} could be associated with $\delta(C-H)$ [13,17,19,22]. In the case of $NO + CH_3OH + O_2$ reaction (Fig. 5), very weak $-NCO$ peak at 2233 cm^{-1} and strong formate peaks at 1595 and 1379 cm^{-1} were observed. Whereas the bands at 1300 , 1589 and 1612 cm^{-1} are similarly assigned to adsorbed nitrates and the band at 1390 cm^{-1} is associated with $\delta(C-H)$, as shown in Fig. 4. The bands at 1639 , 3732 , 3697 and 3589 cm^{-1} are considered to relate with the formation of H_2O by dehydration of CH_3OH and it disappeared by increasing temperature to 623 K. According to the literature [20,21], CH_3OH was selectively dehydrated to dimethyl ether (DME) over $\gamma-Al_2O_3$ and promoted $\gamma-Al_2O_3$ in the

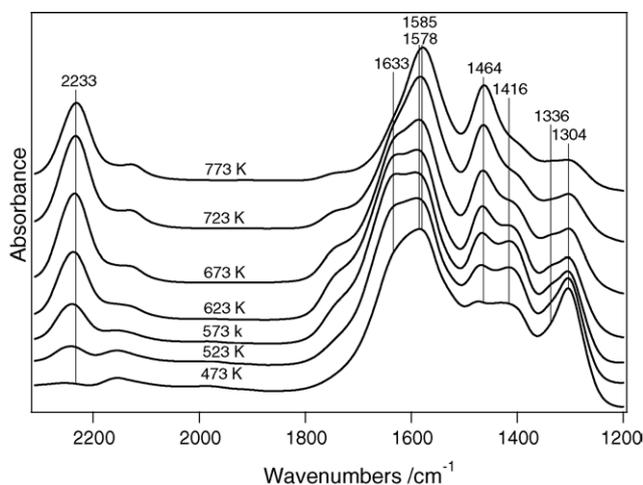


Fig. 3. In situ DRIFTS spectra of adsorbed species in steady state on Ag/Al₂O₃ in a flow of C₂H₅OH + O₂ + NO at various temperatures. Conditions: NO 800 ppm; C₂H₅OH 1565 ppm; O₂ 10%.

temperature of 423–573 K, accompanied by the formation of H₂O.

As shown in Figs. 3–5, the formation of –NCO species in the NO–O₂–C₂H₅OH is similar to that in the NO–O₂–IPA system, which is different from that in the NO–O₂–CH₃OH system. When the Ag/Al₂O₃ catalyst was exposed to a NO–O₂–CH₃OH mixture under the same experimental conditions, very weak band of –NCO species was observed compared with the cases using C₂H₅OH or IPA as reductants. We have proposed the mechanism of the SCR of NO_x by C₂H₅OH over Ag/Al₂O₃, as shown in Scheme 1 [19]. It has been widely accepted that –NCO species is a crucial intermediate in the SCR of NO_x over Ag/Al₂O₃ and its high productivity results in a high efficiency of NO_x reduction. The linkage between the formation tendency of –NCO species and enolic species was clearly observed in Figs. 3–5. As for Ag/Al₂O₃, the order of the facility for the formation of –NCO species was in good agreement with the

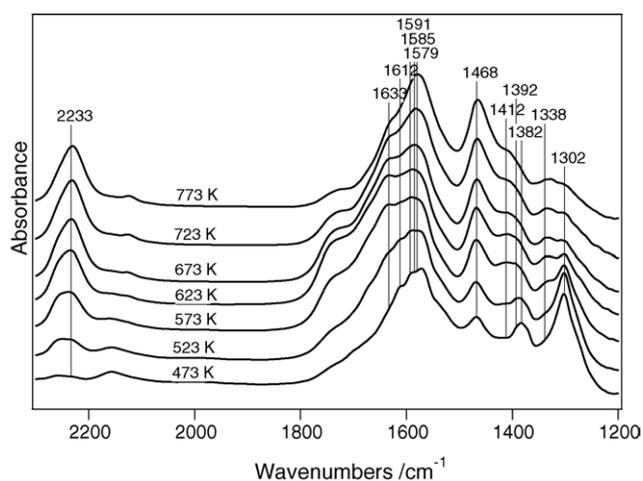


Fig. 4. In situ DRIFTS spectra of adsorbed species in steady state on Ag/Al₂O₃ in a flow of IPA + O₂ + NO at various temperatures. Conditions: NO 800 ppm; IPA 1200 ppm; O₂ 10%.

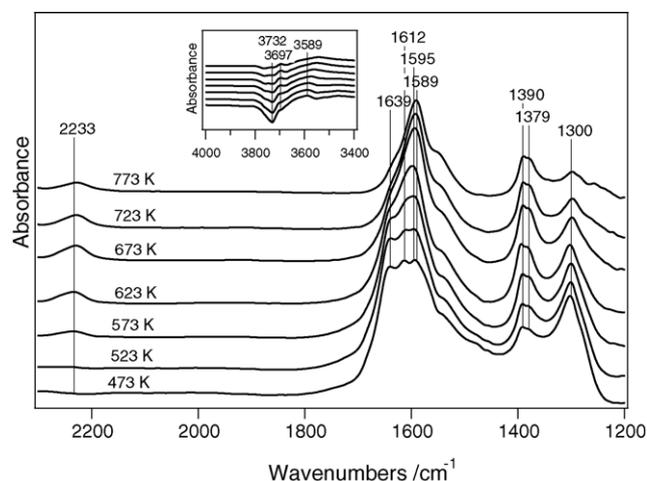


Fig. 5. In situ DRIFTS spectra of adsorbed species in steady state on Ag/Al₂O₃ in a flow of CH₃OH + O₂ + NO at various temperatures. Conditions: NO 800 ppm; CH₃OH 2250 ppm; O₂ 10%.

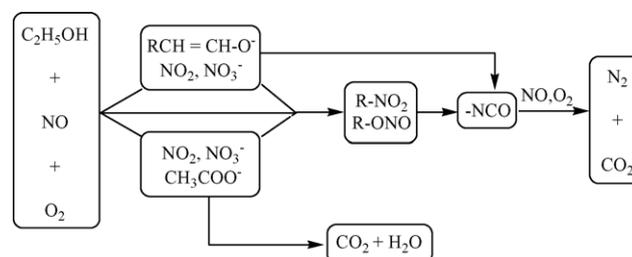
order of the activities in the SCR of NO_x process (Fig. 1: C₂H₅OH > IPA > CH₃OH) in the moderate temperature range of 523–623 K.

GC–MS and FTIR experiments indicated the formation of nitrogen-containing molecule (such as CH₃NO₂) and oxygen-containing molecules (such as CH₃CHO and CH₃COCH₃) during the SCR of NO_x by C₂H₅OH or IPA over Ag/Al₂O₃. Nitrogen-containing molecules and oxygen-containing molecules have been proposed to be the key intermediates in the reduction of NO_x [3,4,15,19]. The analysis of gas products together with in situ DRIFTS spectra proved the similar mechanisms of the SCR of NO_x by C₂H₅OH and IPA over Ag/Al₂O₃, whereas CH₃OH was not found to follow the similar mechanism. More evidences for the different reaction mechanisms will be discussed below.

3.3. Formation of adsorbed enolic species in the partial oxidation reaction over Ag/Al₂O₃

To elucidate the differences among the reductants for the SCR of NO_x over Ag/Al₂O₃, we investigated the surface species formed on the Ag/Al₂O₃ catalyst during the oxidation reaction of different alcohols.

Fig. 6 shows the in situ DRIFTS spectra of Ag/Al₂O₃ at various temperatures in steady state after the exposure to



Scheme 1. The mechanism of the SCR of NO_x by C₂H₅OH over Ag/Al₂O₃.

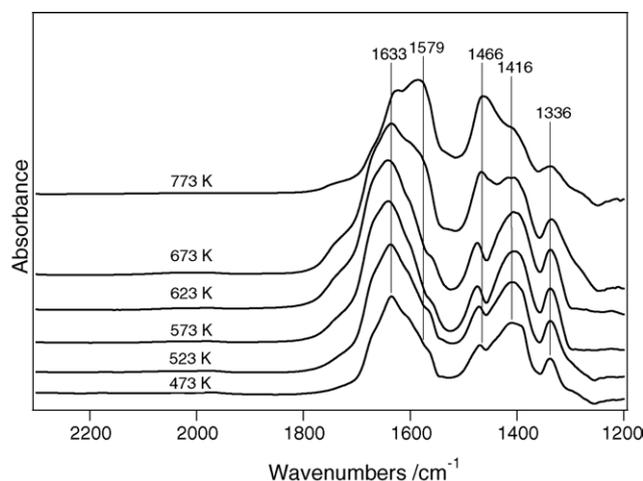


Fig. 6. In situ DRIFTS spectra of adsorbed species in steady state on Ag/Al₂O₃ in a flow of C₂H₅OH + O₂ at various temperatures. Conditions: C₂H₅OH 1565 ppm; O₂ 10%.

C₂H₅OH + O₂ mixture gas, where the five peaks appeared at 1633, 1416, 1336, 1579 and 1466 cm⁻¹. The peaks at 1633, 1416 and 1336 cm⁻¹ are attributed to the enolic species and the peaks at 1579 and 1466 cm⁻¹ are assigned to acetate species. As shown in Fig. 6, the enolic species is dominantly formed from the partial oxidation of C₂H₅OH in the temperature range of 473–673 K, whereas at 773 K, the enolic species was further oxidized into the acetate species.

The same set of experiment was conducted after exposing the catalyst to a flow of IPA + O₂. As shown in Fig. 7, within the temperature range of 573–723 K, a very strong peak at 1633 cm⁻¹ due to the enolic species was also observed, which accompanied by the appearance of the peaks at 1408 and 1335 cm⁻¹. The bands at 1595 and 1379 cm⁻¹ are assigned to formate species, the bands at 1581 and 1468 cm⁻¹ are attributed to acetate species and the peak at 1392 cm⁻¹ can be associated with δ(C–H), as the same as in Fig. 4. At low temperatures, the formate species was

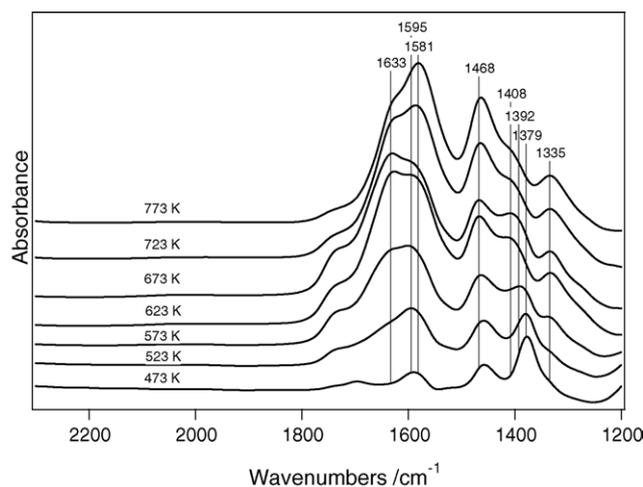


Fig. 7. In situ DRIFTS spectra of adsorbed species in steady state on Ag/Al₂O₃ in a flow of IPA + O₂ at various temperatures. Conditions: IPA 1200 ppm; O₂ 10%.

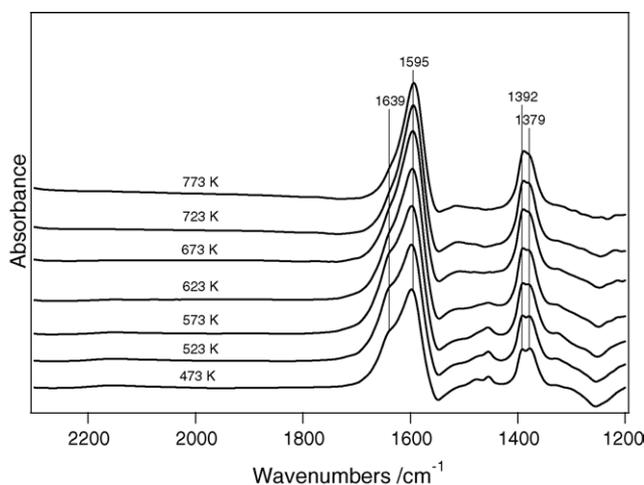


Fig. 8. In situ DRIFTS spectra of adsorbed species in steady state on Ag/Al₂O₃ in a flow of CH₃OH + O₂ at various temperatures. Conditions: CH₃OH 2250 ppm; O₂ 10%.

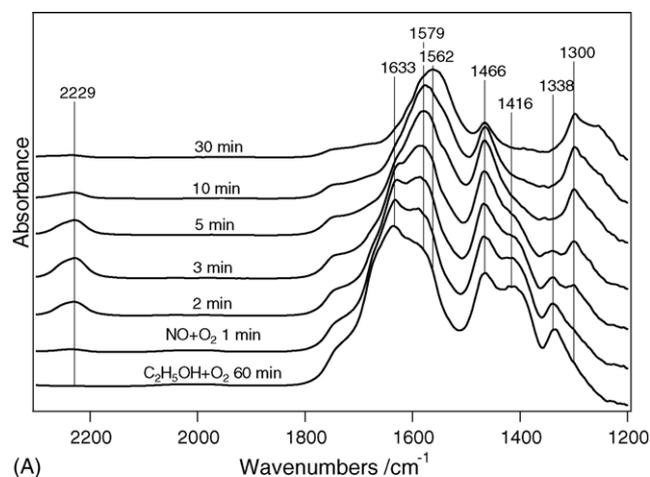
predominant, while within the temperature range of 623–673 K, the enolic species became predominant, indicating that the partial oxidation occurs preferentially on Ag/Al₂O₃. However, the acetate species resulted from further oxidation of the enolic species is dominant at temperatures above 723 K.

Exposing Ag/Al₂O₃ catalyst to a flow of CH₃OH + O₂ resulted in the appearance of four peaks (1639, 1595, 1392 and 1379 cm⁻¹). As can be seen from Fig. 8, the band at 1595 and 1379 cm⁻¹ are assigned to the formate species and the very weak band at 1639 cm⁻¹ is due to the formation of H₂O by dehydration of CH₃OH, as discussed in Fig. 5. While the peak at 1392 cm⁻¹ is associated with δ(C–H). Apparently, the deep oxidation of CH₃OH occurred preferentially above 573 K on Ag/Al₂O₃, because the formate species is predominant during the oxidation reaction and the characteristic bands of the enolic species were not detected in this study. Comparative studies show that partial oxidation of CH₃OH is different from that of other alcohols. As a fact, while the enolic species was the dominant surface species on Ag/Al₂O₃ during the partial oxidation of C₂H₅OH and IPA, it is not the case for CH₃OH, which may account for the relatively low efficiency in the SCR of NO_x.

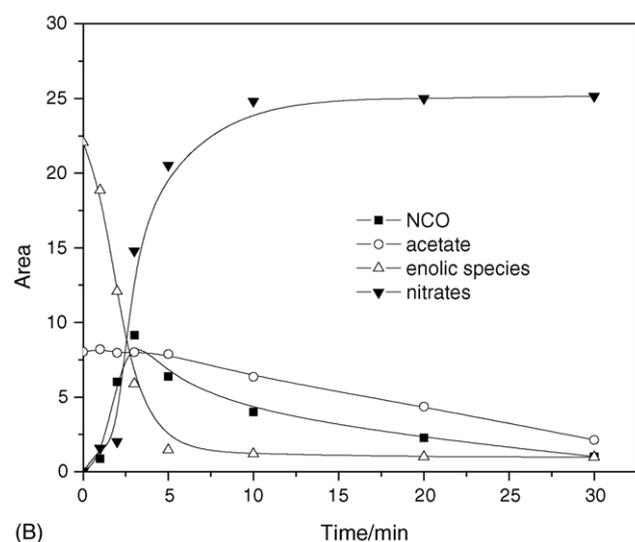
3.4. Reactivity of the surface enolic species over Ag/Al₂O₃

To obtain further information on the reactivity of the surface enolic species, the reactivity of the enolic species toward NO + O₂ was studied by the transient response of DRIFTS method.

Fig. 9A shows the dynamic changes of in situ DRIFTS spectra of the adsorbed species in a flow of NO + O₂ on Ag/Al₂O₃ at 673 K. The integrated areas of these peaks in Fig. 9A are displayed as a function of time in Fig. 9B. When the catalyst was exposed to C₂H₅OH + O₂ for 60 min, very strong enolic species peaks appeared at 1633, 1416 and



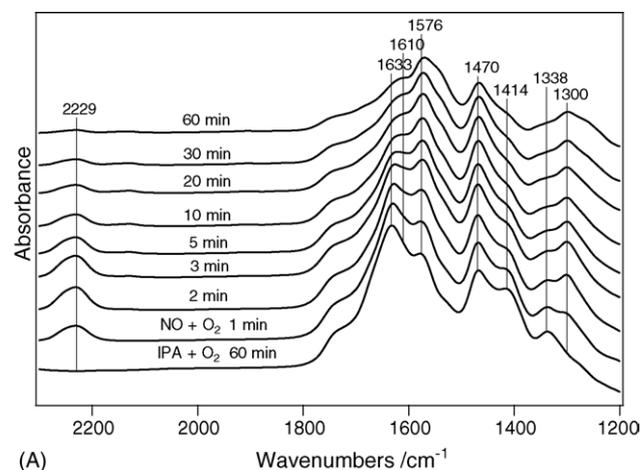
(A)



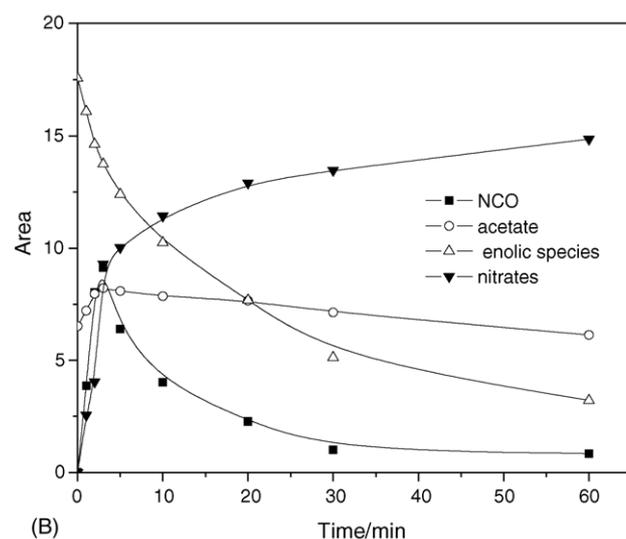
(B)

Fig. 9. (A) Dynamic changes of in situ DRIFTS spectra of adsorbed species on Ag/Al₂O₃ as a function of time in a flow of NO + O₂ at 673 K. Before measurement, the catalyst was pre-exposed to a flow of C₂H₅OH + O₂ for 60 min at 673 K. Conditions: NO 800 ppm; C₂H₅OH 1565 ppm; O₂ 10%. (B) Time dependence of the integrated areas of the peaks for different adsorbed species in (A): (■) –NCO, 2160–2308 cm⁻¹; (○) acetate, 1440–1505 cm⁻¹; (▼) nitrates, 1210–1322 cm⁻¹; (△) enolic species 1612–1710 cm⁻¹.

1338 cm⁻¹ and acetate peaks were also observed at 1579 and 1466 cm⁻¹. Switching the feed gas to a flow of NO + O₂ resulted in a sharp decrease of the enolic species. Meanwhile, a new –NCO peak appeared at 2229 cm⁻¹, with its intensity increased initially and then decreased after it reached a maximum. The linkage between the formation of the –NCO species and the consumption of the enolic species strongly suggests the participation of the enolic species in the formation of –NCO species. When the enolic species was completely consumed, a large amount of nitrates and acetate became dominant surface species on Ag/Al₂O₃, while the surface concentration of –NCO was very low. This result indicates that the enolic species effectively reacts toward NO + O₂ to form –NCO species than acetate species does. As a result, it is the enolic species rather than the acetate



(A)



(B)

Fig. 10. (A) Dynamic changes of in situ DRIFTS spectra of adsorbed species on Ag/Al₂O₃ as a function of time in a flow of NO + O₂ at 673 K. Before measurement, the catalyst was pre-exposed to a flow of IPA + O₂ for 60 min at 673 K. Conditions: NO 800 ppm; IPA 1200 ppm; O₂ 10%. (B) Time dependence of the integrated areas of the peaks for different adsorbed species in (A): (■) –NCO, 2160–2308 cm⁻¹; (○) acetate, 1440–1505 cm⁻¹; (▼) nitrates, 1210–1322 cm⁻¹; (△) enolic species, 1612–1710 cm⁻¹.

species, that plays a crucial role in the –NCO formation by reaction with NO + O₂.

The same experiment was performed after exposing the catalyst to a flow of IPA + O₂ and we derived the same conclusion. As shown in Fig. 10A and B, after switching the feed gas to NO + O₂ within 30 min, the intensity of the enolic species decreased, accompanied by a clear increase in the intensity of –NCO species. This result strongly confirms that the enolic species is the key intermediate in the formation of –NCO.

Fig. 11A shows the dynamic changes of in situ DRIFTS spectra of the Ag/Al₂O₃ catalyst firstly exposed to CH₃OH + O₂ for 60 min at 673 K and then switched to a flow of NO + O₂. Compared with C₂H₅OH and IPA, it should be noted that the peak due to the enolic species

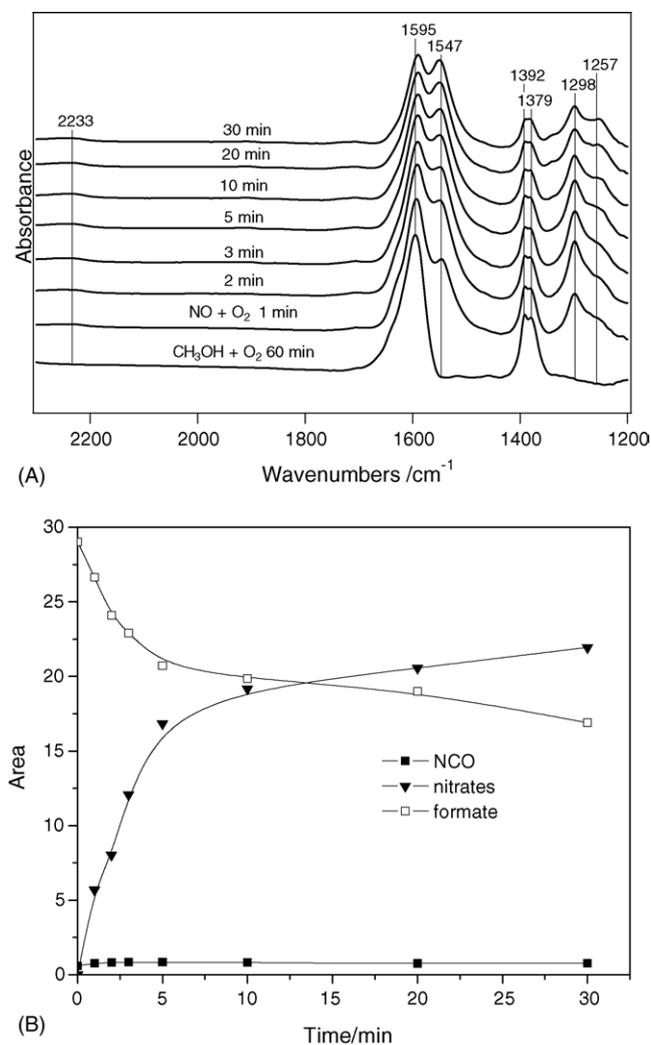


Fig. 11. (A) Dynamic changes of in situ DRIFTS spectra of adsorbed species on Ag/Al₂O₃ as a function of time in a flow of NO + O₂ at 673 K. Before measurement, the catalyst was pre-exposed to a flow of CH₃OH + O₂ for 60 min at 673 K. Conditions: NO 800 ppm; CH₃OH 2250 ppm; O₂ 10%. (B) Time dependence of the integrated areas of the peaks for different adsorbed species in (A): (■) –NCO, 2160–2308 cm⁻¹; (▼) nitrates, 1210–1322 cm⁻¹; (□) formate, 1533–1720 cm⁻¹.

(around 1633 cm⁻¹) was not observed, whereas the peaks of the formate species (1595 and 1379 cm⁻¹) were very strong. On the other hand, very weak band due to the –NCO species (2233 cm⁻¹) was observed, as can be seen from Fig. 11A and B. This result strongly suggests again that the enolic species plays a crucial role in the –NCO formation during the SCR of NO_x by alcohols.

4. Conclusions

Based on the DRIFTS studies, the enolic species has been proved to be the main surface species during the partial oxidation of C₂H₅OH or IPA over Ag/Al₂O₃ catalyst. The

high reactivity of the enolic species with NO + O₂ results in high surface concentration of –NCO species and high efficiency of NO_x reduction when using C₂H₅OH or IPA as reductants in the SCR of NO_x. The linkage between the formation of the –NCO species and the consumption of the enolic species has been found. We have confirmed the similar mechanisms of the SCR of NO_x by C₂H₅OH and IPA over Ag/Al₂O₃. However, CH₃OH has not been found to follow the similar mechanism. CH₃OH is much less reactive for the formation of –NCO species. The order of the facility for the formation of enolic species and –NCO species is in good agreement with the order of the activities (C₂H₅OH > IPA > CH₃OH) in the moderate temperature range of 523–623 K during the SCR of NO_x.

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