

# Novel Sn–Ce/Al<sub>2</sub>O<sub>3</sub> catalyst for the selective catalytic reduction of NO<sub>x</sub> under lean conditions

Zhiming Liu, Kwang Seok Oh, and Seong Ihl Woo\*

*Department of Chemical and Biomolecular Engineering, Center for Ultramicrochemical Process Systems, Korea Advanced Institute of Science and Technology, 373-1, Guseong-dong, Yuseong-gu, Daejeon 305-701, Korea*

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Effect of additives, Ce and Mn, on the catalytic performance of Sn/Al<sub>2</sub>O<sub>3</sub> catalyst prepared by sol–gel method for the selective reduction of NO<sub>x</sub> with propene under lean conditions was studied. Sn–Ce/Al<sub>2</sub>O<sub>3</sub> catalysts exhibited higher activity than Sn/Al<sub>2</sub>O<sub>3</sub> catalyst and the optimum Ce loading is 0.5–1%. The promoting effect of Ce is to enhance the oxidation of NO to NO<sub>2</sub> and facilitate the activation of propene, both of which are important steps for the NO<sub>x</sub> reduction. The presence of oxygen contributes to the oxidation of NO and shows a promoting effect.

**KEY WORDS:** Sn–Ce/Al<sub>2</sub>O<sub>3</sub>; selective reduction of NO<sub>x</sub>; propene.

## 1. Introduction

Selective catalytic reduction of NO<sub>x</sub> with hydrocarbons as reducing agents (HC-SCR) has received much attention as one of potential technology for reducing NO<sub>x</sub> emissions in the presence of excess oxygen. Since the pioneer work of Held et al. [1] and Iwamoto et al. [2] quite a few different catalyst systems have been investigated for HC-SCR [3–10]. Among them, the metal oxide catalysts are more desirable because they exhibit high activities at high temperatures, besides they are more stable than zeolite-based catalysts in the hydrothermal conditions [11–13].

Since SnO<sub>2</sub> was firstly reported to be an active catalyst for the NO<sub>x</sub> reduction by hydrocarbons in the presence of oxygen [14], some research was focused on Sn/Al<sub>2</sub>O<sub>3</sub> catalyst [15–19]. For Sn/Al<sub>2</sub>O<sub>3</sub> catalyst, the major disadvantage is that the NO<sub>x</sub> conversion is low at low-temperature region (250–400 °C). However, there is little report about enhancing the low-temperature activity of Sn/Al<sub>2</sub>O<sub>3</sub> catalyst. In the previous research, the higher activity can be observed when feeding NO<sub>2</sub> instead of NO during the HC-SCR over Sn/Al<sub>2</sub>O<sub>3</sub> catalyst [20]. That means the NO oxidation might be a limiting step in NO reduction and strengthening the NO oxidation function of Sn/Al<sub>2</sub>O<sub>3</sub> catalyst should enhance its activity for the NO reduction. CeO<sub>2</sub> was chosen as one of the active components for favouring the NO oxidation because of its well-known function as oxygen storage/activator in three-way catalysts [21]. And recently, Krishna et al. [22] reported that mixed CeO<sub>2</sub>

with H-FER zeolite catalyst showed high activity for HC-SCR. Besides CeO<sub>2</sub>, manganese oxide also served as an oxidation catalyst to promote the performance of deNO<sub>x</sub> catalyst [23,24]. So the present work attempts to improve the activity of Sn/Al<sub>2</sub>O<sub>3</sub> catalyst by adding cerium oxide and manganese oxide.

## 2. Experimental

### 2.1. Catalyst preparation

Metal oxide-doped Sn/Al<sub>2</sub>O<sub>3</sub> catalysts were prepared by single step sol–gel method. Ce and Mn were selected as an additive. Aluminium boehmite sol was first prepared by hydrolysis of aluminium(III) iso-propoxide(AIP) in hot water(85 °C) with a proper amount of nitric acid, and then mixed with a solution of tin chloride and metal nitrate of the doped metal oxide. After one day of stirring, the solvents were eliminated by heating under reduced pressure to form a gel. The obtained gel was dried at 120 °C for 24 h, followed by calcinations at 600 °C for 4 h in air. The loading of Sn was fixed at 5% since previous research showed that the optimum Sn loading is 5% for Sn/Al<sub>2</sub>O<sub>3</sub> catalyst [19]. While that of Ce additive varies from 0.5 to 5%. A 5%Sn/Al<sub>2</sub>O<sub>3</sub> was also prepared as a reference sample by the same sol–gel method.

The SBA15 support was synthesized according to a procedure described in the literature [25]. Pluronic P123 triblock copolymer surfactant (EO<sub>20</sub>PO<sub>70</sub>EO<sub>20</sub>) was used as a template, which was dissolved in hydrochloric acid solution. After stirring for a few hours, a suitable amount of TEOS (tetraethyl orthosilicate) was added.

\* To whom correspondence should be addressed.

E-mail: siwoo@kaist.ac.kr

The resulting mixture was stirred for 12 h at 30 °C and then at 100 °C for 24 h. The white product was filtered, washed and dried. Finally, the product was calcined at 550 °C for 4 h to remove the template. AISBA15 was obtained by impregnating SBA15 with a proper amount Al(NO<sub>3</sub>)<sub>3</sub> solution and the Al percentage is 1.3, dried at 120 °C for 12 h, followed by calcinations at 300 °C for 3 h in air. Then AISBA15 was impregnated with H<sub>2</sub>PtCl<sub>6</sub> solution, dried at 120 °C for 12 h, followed by calcinations at 500 °C for 3 h in air and yield the Pt/AISBA15 catalyst, which was compared with metal oxide-doped Sn1Al<sub>2</sub>O<sub>3</sub> catalyst.

## 2.2. Evaluation of catalyst activity

The activity measurements were carried out in a fixed-bed quartz reactor using a 0.2 g catalyst of 60–80 mesh. The feed gas mixture contained 2000 ppm NO<sub>x</sub>, 2700 ppm C<sub>3</sub>H<sub>6</sub>, 3% O<sub>2</sub> and helium as the balance gas. Unless noted, the total flow rate of the feed gas was 100 cm<sup>3</sup> min<sup>−1</sup>, which corresponded to a space velocity of 26,000 h<sup>−1</sup>. The reaction temperature was increased from 250 to 550 °C in steps of 50 °C. All the catalysts were pretreated in the reaction feed gas at 600 °C for 2 h before the activity test. In the previous research we compared the activities of two different 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalysts, one was only calcined in air and the other was pre-treated in the reaction gas after calcining in air. It was found that the 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst pre-treated in the reaction feed gas showed much higher activity than that without pre-treatment below 450 °C. And above this temperature there is almost no deactivation after feed gas treatment [19]. For Sn/Al<sub>2</sub>O<sub>3</sub> catalyst, besides SnO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> is an active site and contributes to the NO<sub>x</sub> reduction but could be prevented from doing so when its active centers are covered by dispersed SnO<sub>2</sub> [17]. From the TEM photographs it can be seen that pre-treatment leads to the crystallization and growth of the SnO<sub>2</sub> species [19]. Due to the agglomeration, Al<sub>2</sub>O<sub>3</sub> surface sites previously covered by dispersed SnO<sub>2</sub> species are re-exposed. So the activity of Sn/Al<sub>2</sub>O<sub>3</sub> catalyst was improved after pre-treatment in the reaction gas.

The composition of the product gas was analyzed by using gas chromatograph (equipped with Porapak Q and Molecular sieve 5A columns). A molecular-sieve 5A column was used for the analysis of N<sub>2</sub> and CO and Porapak Q column for that of N<sub>2</sub>O, CO<sub>2</sub> and C<sub>3</sub>H<sub>6</sub>. The activity data were collected when the catalytic reaction practically reached steady-state conditions at each temperature. The formation of N<sub>2</sub>O was found negligible in the present study. Thus, NO<sub>x</sub> conversion was determined by the N<sub>2</sub> produced.

## 2.3. Catalyst characterization

BET surface areas were measured by N<sub>2</sub> adsorption using a NOVA4200 automated gas sorption system. X-

ray diffraction (XRD) measurements were carried out on a Bruker D8 Discover DADD S X-ray Diffractometer with CuKα radiation.

## 3. Results and discussion

### 3.1. Catalyst characterization

The BET surface areas of all the catalysts are summarized in table 1. No significant difference of surface area can be observed over Sn/Al<sub>2</sub>O<sub>3</sub>, Sn–Ce/Al<sub>2</sub>O<sub>3</sub> and Sn–Mn/Al<sub>2</sub>O<sub>3</sub> catalysts. Figure 1 shows the XRD patterns of 5%Sn/Al<sub>2</sub>O<sub>3</sub> and Sn–Ce/Al<sub>2</sub>O<sub>3</sub> catalysts. Except for 5%Sn–5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst, only the characteristic lines of γ-Al<sub>2</sub>O<sub>3</sub> can be observed. In addition to the γ-Al<sub>2</sub>O<sub>3</sub> phase, small peak assigned to CeO<sub>2</sub> is identified over 5%Sn–5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst. The peak ascribed to SnO<sub>2</sub> was not observed over all the catalysts. This fact suggests the presence of small SnO<sub>2</sub> species on Al<sub>2</sub>O<sub>3</sub>.

### 3.2. Activity test

#### 3.2.1. Effect of additives on the activity of 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst

The effect of metal additives on the activity of 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst is presented in figure 2. It can be seen

Table 1  
The specific surface area of the catalysts

| Catalyst                                   | Specific surface area (m <sup>2</sup> g <sup>−1</sup> ) |
|--------------------------------------------|---------------------------------------------------------|
| 5%Sn/Al <sub>2</sub> O <sub>3</sub>        | 170                                                     |
| 5%Sn–0.5%Ce/Al <sub>2</sub> O <sub>3</sub> | 185                                                     |
| 5%Sn–1%Ce/Al <sub>2</sub> O <sub>3</sub>   | 177                                                     |
| 5%Sn–2%Ce/Al <sub>2</sub> O <sub>3</sub>   | 175                                                     |
| 5%Sn–5%Ce/Al <sub>2</sub> O <sub>3</sub>   | 178                                                     |
| 5%Sn–1%Mn/Al <sub>2</sub> O <sub>3</sub>   | 176                                                     |

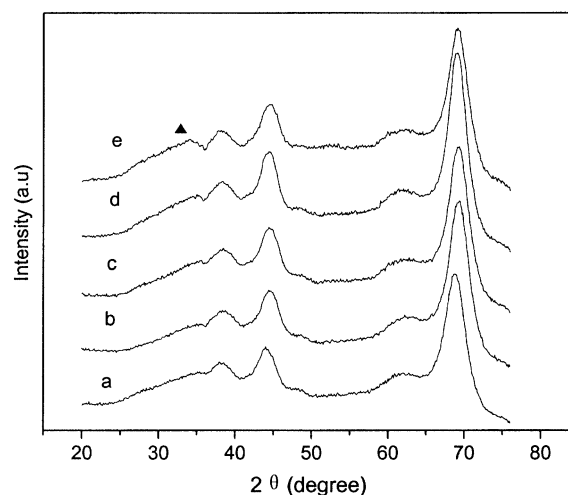


Figure 1. XRD patterns of catalysts: (a) 5%Sn/Al<sub>2</sub>O<sub>3</sub>; (b) 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub>; (c) 5%Sn–1%Ce/Al<sub>2</sub>O<sub>3</sub>; (d) 5%Sn–2%Ce/Al<sub>2</sub>O<sub>3</sub> and (e) 5%Sn–5%Ce/Al<sub>2</sub>O<sub>3</sub>. (▲)CeO<sub>2</sub>

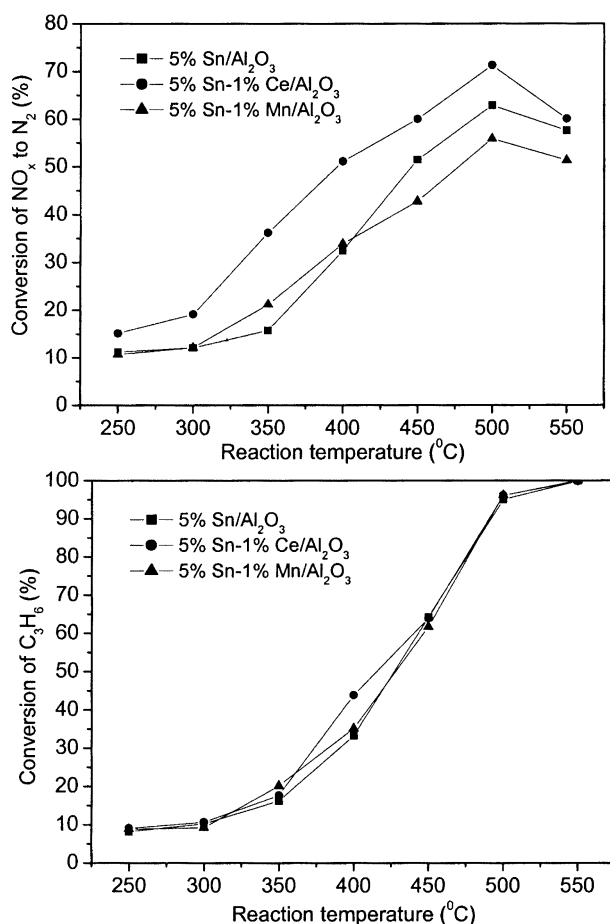


Figure 2. Activities of different metal modified 5% Sn/Al<sub>2</sub>O<sub>3</sub> catalysts for NO reduction at different reaction temperatures (reaction conditions: NO = 2000 ppm, C<sub>3</sub>H<sub>6</sub> = 2700 ppm, O<sub>2</sub> = 3%, GHSV = 26,000 h<sup>-1</sup>).

that the addition of Mn causes a pronounced decrease in the catalytic activity of 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst above 400 °C, although the activity of added Mn catalyst at 350 °C was slightly higher than that of 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst. On the other hand, the addition of Ce showed a remarkable promoting effect. The added Ce catalyst exhibited higher activity than 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst over the whole temperature range. Over the three catalysts, the maximum NO<sub>x</sub> conversion were all obtained at 500 °C, at which the propene was almost completely converted. The facts confirm that the temperature of maximum NO<sub>x</sub> conversion temperature was closely related to that of almost full combustion of reducing agent.

### 3.2.2. Effect of Ce loading on the activity of 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst

Since the additive of Ce showed a beneficial effect on the activity of 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst, the effect of varying the Ce content on the performance of 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst was further investigated and the results are shown in figure 3. The activity of 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst hardly changed with increase in the Ce loading from 0.5

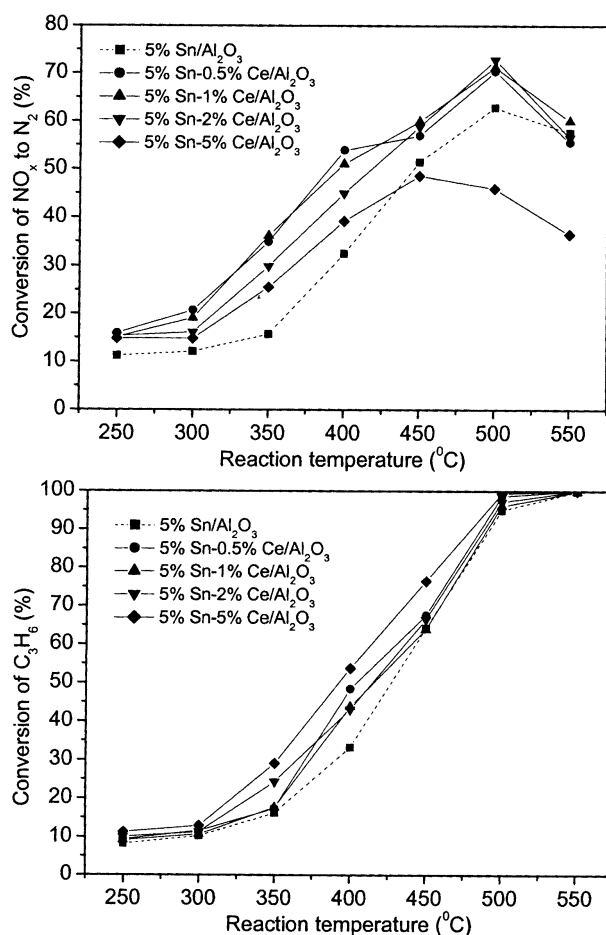


Figure 3. Effect of Ce loading on NO<sub>x</sub> conversion over 5%Sn-x%Ce/Al<sub>2</sub>O<sub>3</sub> catalysts for NO reduction by propene: (Reaction conditions: NO = 2000 ppm, C<sub>3</sub>H<sub>6</sub> = 2700 ppm, O<sub>2</sub> = 3%, GHSV = 26,000 h<sup>-1</sup>).

to 1%. Further increasing the Ce loading leads to the decreased activity and when the Ce loading reached 5%, the NO conversion was even lower than that over 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst above 450 °C. However, the low temperature activity of 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst was still improved. Thus, a small amount of Ce (0.5–1%) provide the maximum promotion of the SCR reaction. When the Ce loading was low, no peak ascribed to CeO<sub>2</sub> was observed from the XRD patterns. So high-dispersed Ce was present on the catalyst. At higher Ce loading, large CeO<sub>2</sub> particles were formed, which were active for the combustion of propene as a side reaction. Above 450 °C, the side reaction predominated and there was not enough reductant to reduce NO<sub>x</sub>, so the conversion of NO<sub>x</sub> over 5%Sn-5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst was even lower than that over 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst.

### 3.2.3. Comparison of activities of 5%Sn-0.5%Ce/Al<sub>2</sub>O<sub>3</sub> and 2.3%Pt/AlSBA15 catalysts

Among the catalysts tested for the selective reduction of NO by propene, Pt supported catalysts were extensively studied due to its high low-temperature activity [26–28]. So in this work the activities of 5%Sn-0.5%Ce/

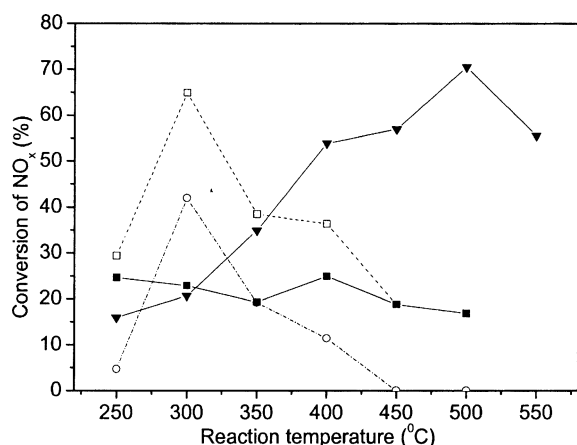


Figure 4. Comparison of activities of 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub>(▼) and 2.3% Pt/AlSBA15(□ total conversion of NO<sub>x</sub>; ○: conversion to N<sub>2</sub>O; ■: conversion to N<sub>2</sub>) catalysts (Reaction conditions: NO = 2000 ppm, C<sub>3</sub>H<sub>6</sub> = 2700 ppm, O<sub>2</sub> = 3%, GHSV = 26,000 h<sup>-1</sup>).

Al<sub>2</sub>O<sub>3</sub> and 2.3%Pt/AlSBA15 catalysts were compared under identical reaction conditions. As shown in figure 4, the activity of Pt/AlSBA15 catalyst was high at low temperature region and the maximum NO conversion was obtained at 300 °C. However, the high selectivity to N<sub>2</sub>O and the narrow temperature window are the obstacle for practical application. In contrast, the selectivity towards N<sub>2</sub> over 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst is almost 100%. In view of the conversion to N<sub>2</sub>, 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst was more active at temperatures above 300 °C and the temperature window was wider, though Pt/AlSBA15 catalyst was slightly more active at 250 °C.

### 3.2.4. Effect of O<sub>2</sub>

Figure 5 illustrates the changes in NO and C<sub>3</sub>H<sub>6</sub> conversions with respect to O<sub>2</sub> concentration over 5%Sn/Al<sub>2</sub>O<sub>3</sub> and 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalysts, respectively. At 350 °C, the increase of O<sub>2</sub> concentration was found to be favorable for NO<sub>x</sub> reduction over the two catalysts. Especially for 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst, the conversion of NO<sub>x</sub> significantly improved with the increase of oxygen concentration from 1 to 3%. With further increase in oxygen concentration the NO<sub>x</sub> conversion remained almost unchanged, although the conversion of propene was further increased. At 450 °C, with the O<sub>2</sub> concentration further increasing to 10% from 6%, the NO<sub>x</sub> conversion over 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst was slightly decreased, which was a little lower than that over 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst. The possible reason is that at higher temperatures more reducing agent was combusted and there was not enough reductant to reduce NO<sub>x</sub> over 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst. The fact indicates that the oxygen plays an important role in the selective reduction of NO<sub>x</sub>. TPD-NO<sub>x</sub> results over Sn/Al<sub>2</sub>O<sub>3</sub> showed that the amount of desorbed species after adsorption of NO + O<sub>2</sub> were much larger than that of adsorption of NO [20]. This means the NO<sub>x</sub>

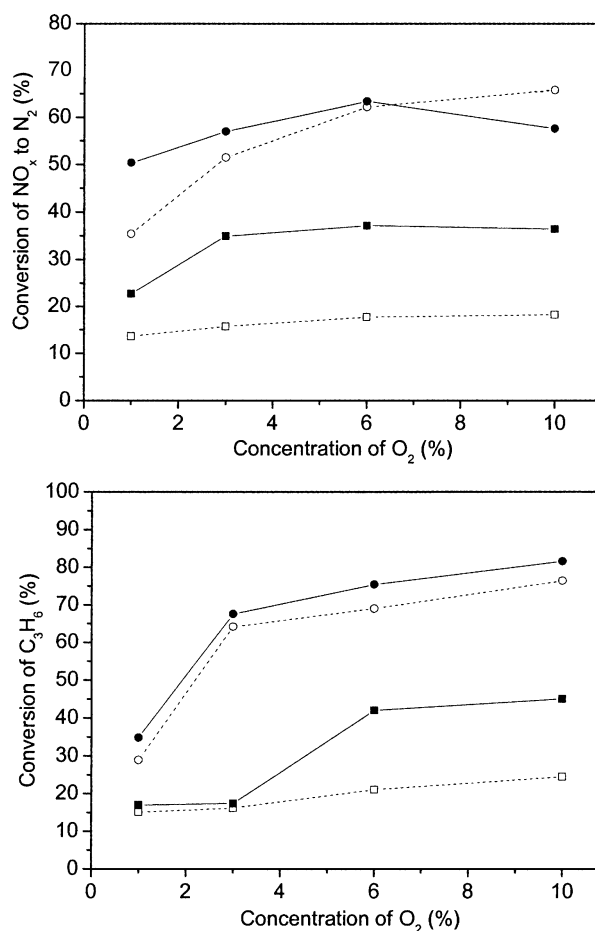


Figure 5. Effect of O<sub>2</sub> concentration on the activities of 5%Sn/Al<sub>2</sub>O<sub>3</sub> (dashed line) and 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> (Solid line) catalysts at 350 °C(□, ■) and 450 °C(○, ●) (Reaction conditions: NO = 2000 ppm, C<sub>3</sub>H<sub>6</sub> = 2700 ppm, O<sub>2</sub> = 1–10%, GHSV = 26,000 h<sup>-1</sup>).

species are more easily adsorbed on the surface of the catalyst in the presence of oxygen and further converted to surface nitrates, which is an important reaction intermediate for NO<sub>x</sub> reduction [9]. The presence of oxygen also contributes to the oxidation of NO to NO<sub>2</sub>, which is more reactive than NO and as a result is more likely to react quickly with some other key reaction intermediates to form N<sub>2</sub>.

### 3.2.5. Effect of C<sub>3</sub>H<sub>6</sub>

Figure 6 presents the effect of C<sub>3</sub>H<sub>6</sub> on the activities of 5%Sn/Al<sub>2</sub>O<sub>3</sub> and 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalysts. Over the latter catalyst, the NO<sub>x</sub> conversion was increased when the concentration of propene was increased from 1000 to 4000 ppm. This result indicates that high concentration of reductant is beneficial to the reduction of NO<sub>x</sub>. The effect is in accordance with the previous research [29,30] and is attributed to the fact that the larger amount of reducing agent makes more reaction intermediates species formed to facilitate the NO reduction. On the other hand, the NO<sub>x</sub> conversion appeared to decrease over 5%Sn/Al<sub>2</sub>O<sub>3</sub> catalyst as the

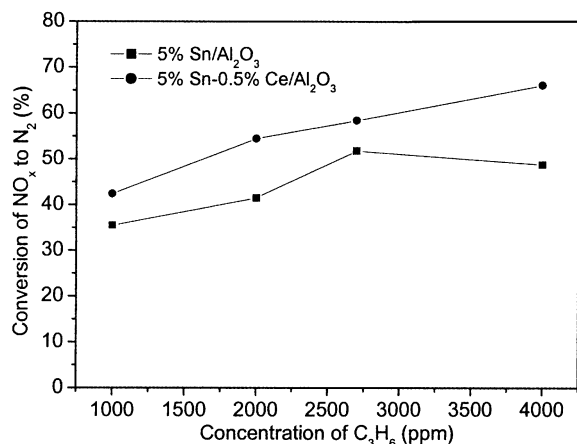


Figure 6. Effect of C<sub>3</sub>H<sub>6</sub> on the activities of 5%Sn/Al<sub>2</sub>O<sub>3</sub> and 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalysts at 450 °C. (Reaction conditions: NO = 2000 ppm, C<sub>3</sub>H<sub>6</sub> = 1000–4000 ppm, O<sub>2</sub> = 3%, GHSV = 26,000 h<sup>–1</sup>).

propene concentration was increased from 2700 to 4000 ppm. This phenomenon is similar to the report by L.F.Córdoba et al. [31]. It was speculated that the carbonaceous deposits were more easily formed on the catalyst surface when the propene concentration was high and inhibited the NO<sub>x</sub> reduction [12].

### 3.2.6. Effect of space velocity

Figure 7 shows the effect of space velocity of feed gas on the activity of 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst. When the GHSV was 13,000 h<sup>–1</sup>, it showed very high activity, 53% NO<sub>x</sub> conversion can be obtained at temperature as low as 350 °C. As expected, the conversion of NO<sub>x</sub> to N<sub>2</sub> decreased with increasing space velocity. However, the maximum NO<sub>x</sub> conversion was only slightly decreased at a space velocity of 39,000 h<sup>–1</sup>.

For the C<sub>3</sub>H<sub>6</sub>-SCR of NO<sub>x</sub> over Sn/Al<sub>2</sub>O<sub>3</sub> catalyst, SnO<sub>2</sub> was found to promote the formation of acetaldehyde from propene and subsequently the acetaldehyde was oxidized to form acetates[33]. The acetate species are then believed to react with the surface adsorbed nitrates to yield organo-nitrogen species. The formation of the organo-nitrogen species is likely to be the rate-determining step of the reaction. The organo-nitrogen species will be decomposed to CN and/or NH<sub>3</sub> species, which then react with NO and/or NO<sub>2</sub> to form N<sub>2</sub> [9,33]. Kung et al. [33] proposed that acetate species react with the surface adsorbed nitrates to yield organo-nitrogen species and the formation of N<sub>2</sub> occurred over Al<sub>2</sub>O<sub>3</sub> sites.

For bifunctional catalyst, a key issue related to the development of the NO oxidation function is to avoid the oxidation of hydrocarbon with oxygen. Sn/Al<sub>2</sub>O<sub>3</sub> catalyst was regarded as a unique kind of lean NO<sub>x</sub> catalysts [17,32] due to its poor ability to activate hydrocarbons. So a small amount of CeO<sub>2</sub> addition to Sn/Al<sub>2</sub>O<sub>3</sub> catalyst did not lead to the combustion of propene but contributed to the activation of the reductant to form hydrocarbon intermediates such as acetal-

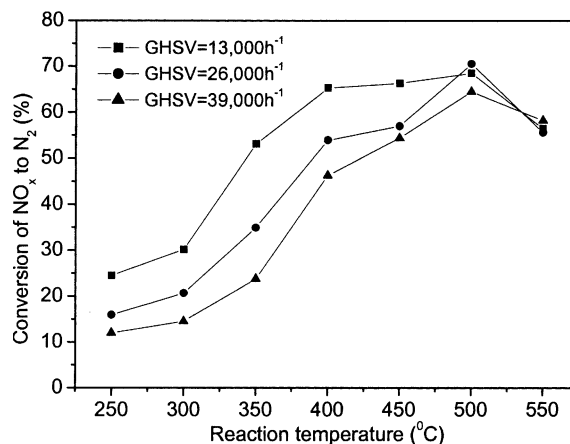


Figure 7. Effect of space velocity of feed gas on the activities of 5%Sn–0.5%Ce/Al<sub>2</sub>O<sub>3</sub> catalyst. (Reaction conditions: NO = 2000 ppm, C<sub>3</sub>H<sub>6</sub> = 2700 ppm, O<sub>2</sub> = 3%, GHSV = 13,000–39,000 h<sup>–1</sup>).

dehyde, which is an important step for the NO<sub>x</sub> reduction to proceed [6,33]. At the same time, CeO<sub>2</sub> catalyze the oxidation of NO to NO<sub>2</sub> [34,35], which is more active species. Both aspects facilitate the formation of N<sub>2</sub>. So Sn–Ce/Al<sub>2</sub>O<sub>3</sub> catalyst exhibited higher activity than Sn/Al<sub>2</sub>O<sub>3</sub> catalyst.

## 4. Conclusions

Sn–Ce/Al<sub>2</sub>O<sub>3</sub> catalysts were more active than Sn/Al<sub>2</sub>O<sub>3</sub> catalyst for the selective reduction of NO<sub>x</sub> and the optimum Ce loading is 0.5–1%. The oxygen dependency of these catalysts is desirable for NO<sub>x</sub> removal of a lean burn engine. The addition of Ce to Sn/Al<sub>2</sub>O<sub>3</sub> catalyst contributes to the oxidation of NO to NO<sub>2</sub> and the activation of propene, thus improving the yield of the whole NO reduction reaction.

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