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Low temperature selective catalytic reduction of NO by activated carbon fiber loading lanthanum oxide and ceria

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A B S T R A C T

Due to the contribution to air pollution, the controlling of NO discharge needs further studies. In this paper, loaded catalysts of 10–40% (w/w) CeO_2/ACF were prepared by impregnation method and used for the selective catalytic reduction of NO with ammonia in the presence of O_2. Such catalysts were characterized by surface area measurement (BET), thermo gravimetric (TG) analysis and Fourier transform infrared (FTIR) spectroscopy. The catalytic activity of 10–40% CeO_2/ACF at different temperatures, and the catalytic stability at 200 °C were studied. Moreover, the experimental results were compared with those of previous studied catalysts. The results show that the 10% CeO_2/ACF and 20% La_2O_3/ACF can yield higher NO conversion and maintain higher catalytic activity at higher temperature than others. 10% CeO_2/ACF yields about 70% NO conversion in the SCR of NO at 150 °C, and meanwhile 20% La_2O_3/ACF and 10% CeO_2/ACF yield over 90% NO conversion when the temperatures are higher than 350 °C. The catalytic activity and the catalyst stability of 10% CeO_2/ACF and 20% La_2O_3/ACF are both higher than those of many reported catalysts.

Based on the catalytic and characterizing results, such as BET, TG and FTIR, it can be found that the SCR of NO mechanism of loaded catalysts is different from those of ACF and HNO_3/ACF. ACF-C is the catalyst and reducing agent in the SCR of NO of ACF and HNO_3/ACF, while the metal oxides loaded by ACF are the catalytic centers, NH_3 is the main reducing agent in the SCR of loaded catalysts.

1. Introduction

Nitrogen oxides (NO, NO_2, and N_2O) contribute to photochemical smog, acid rain, ozone depletion and greenhouse effects [1,2], they remain to be a major source for air pollution. In recent years, many methods have been used to reduce the emission of nitrogen oxides. Catalytic technologies are attractive because of their low cost and high efficiency. Up to now, many kinds of catalysts including noble metals, such as Ag [3,4], Pt, Au [5,6]; metal oxides, such as CuO [7], V_2O_5, TiO_2, MnOx; and other catalysts, such as Fe-zeolites and Cu-ZSM-5 with zeolites or oxides, have been investigated for SCR [8–12].

For all of the researched NO catalysts, their catalytic abilities are greatly determined by the characteristics of prepared catalysts. So the finding of effective active component and suitable catalyst carrier plays a vital role in such researches. Activated carbon fiber (ACF) has been widely applied as a catalyst support for removing the pollution sources due to their special characteristics [13]. The catalytic conversion of NO into N_2 using ACF with a transition metal, such as Ni, Fe, Co, Mn, prepared by impregnation and precipitation with an aqueous solution of metal salts has been investigated for some time [14–16]. And furthermore, lanthanide, such as La and Ce, studied in this paper, has abundant storage in China, and they can be used in the SCR of NO. So it is desirable to use lanthanide in SCR of NO [17]. The authors have studied NO purification ability of ACF, ACF modified by HNO_3 (referred to HNO_3/ACF), ACF loaded La_2O_3 (referred to La_2O_3/ACF), and some interesting results, such as their relative high catalytic activity at low temperature and longer catalytic durability, have been found [17]. But the relationship among their catalytic ability, catalysts

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properties and the catalytic mechanism has not been further studied.

In this paper, a catalyst of ACF loaded CeO_2 (referred to CeO_2/ACF) was prepared by the same simple method as La_2O_3/ACF, and the properties of ACF, HNO_3/ACF, La_2O_3/ACF and CeO_2/ACF were studied, and their NO catalytic mechanism was also discussed.

2. Experimental

2.1. Catalysts preparation

The 10–40% (w/w) CeO_2/ACF was prepared as previous paper [17] reported, which is by dipping the ACF into certain concentration of $Ce(NO_3)_3$ solution for 2 h. Afterwards, the samples were dried at ambient temperature and pyrolyzed at 360 °C for 2 h in inert condition.

2.2. Activity tests

The experiments were carried out as previous paper [17] reported. In the catalytic experiments, the powder catalyst was loaded, but not closely packed, into a glass tube of 20 mm diameter. The space velocity was 5000 $m^3\ h^{-1}\ m^{-3}$. In each catalytic activity experiment, 0.5000 g of catalyst was loaded; and under those conditions, its pressure drop was about 4 Pa. Each experiment ended when a steady-state conversion of NO was achieved. The catalyst stability experiments of 5.0000 g 10% CeO_2/ACF and 20% La_2O_3/ACF were carried out at 200 °C.

2.3. Catalysts characterization

Specific surface area was calculated from the N_2 adsorption isotherms by applying the Brunauer–Emmett–Teller (BET) equation which yielded important information about structural features. The total micropore, narrower micropore ($D_p < 6$ nm) and medium size micropore ($D_p = 6–12$ nm) volumes were calculated using the de Bore equation.

The processes of decomposition and oxidation of ACF, HNO_3/ACF and the loaded catalysts were followed by the experiments of thermal gravimetric (TG) analyzer (STA-409PC/PG).

The powder X-ray diffraction (XRD) measurements were carried out with a Rigaku Rotaflex D/Max-C system with Cu KR ($\lambda = 0.1543$ nm) radiation. The samples were loaded on a sample holder with a depth of 1 mm.

Infrared spectra were recorded on a WQF-410 FTIR spectrometer with a DTGS detector. Self-supported wafer of 1.3 cm diameter was prepared by pressing 15 mg sample and then loaded into an IR cell with KBr coated germanium windows. In the experiment, the IR spectra were recorded by accumulating 32 scans at a spectra resolution of 4 cm^{-1}.

3. Results and discussion

3.1. Effect of load mass of CeO_2 and catalyst species on NO conversion

In previous paper [17], when we tried to reach considerable NO removals, the key role of the characteristics of the ACF support was identified. The use of a non-activated ACF, ACF modified by HNO_3 and La_2O_3 in SCR of NO, ACF modified by HNO_3 and La_2O_3 could significantly improve its NO conversion. The metal oxides, loaded by ACF, were the catalytic center in the SCR of NO. 20% La_2O_3/ACF was the best one among 10–40% (w/w) La_2O_3/ACF. In the present work, we reported catalysts prepared with the ACF loaded CeO_2.

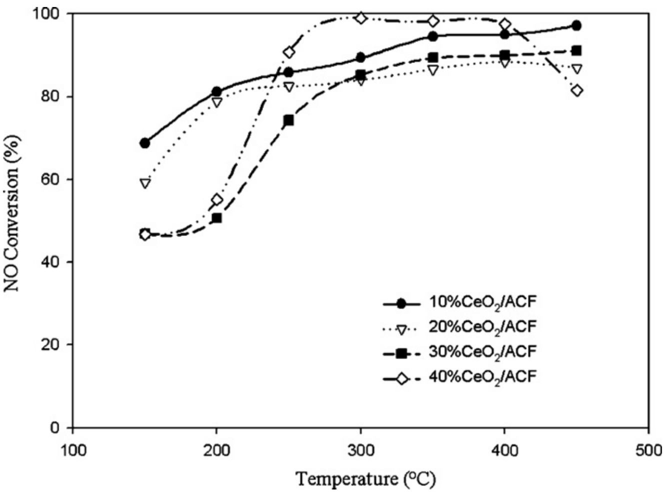

Fig. 1. Effect of load mass of CeO_2 on NO conversion.

The effect of load mass of CeO_2 on NO conversion is shown in Fig. 1. In these experiments, NO conversion of CeO_2/ACF catalysts changed with increasing of temperature and CeO_2 loaded mass. 10% CeO_2/ACF yielded the highest NO conversion at 150 °C of all catalysts, and it also yielded a higher and more stable NO conversion from 250 °C to 450 °C. As for CeO_2/ACF, the suitable loaded mass was 10% with the temperature ranging from 150 °C to 450 °C.

Fig. 2 shows the variation of NO conversion for different ACF catalyst. Obviously, ACF, modified by HNO_3 and metal oxides, greatly affected NO conversion. 10% CeO_2/ACF yielded about 70% NO conversion at 150 °C, which is slightly lower than that of HNO_3/ACF. Its NO conversion efficiency was significantly higher than that of other catalysts with exception that its NO conversion was partly lower than that of 40% CeO_2/ACF from 230 °C to 410 °C. So catalytic activity of 10% CeO_2/ACF not only had a wide temperature window, but also could yield a higher NO conversion.

In this paper, we discussed the reason why 10% CeO_2/ACF and 20% La_2O_3/ACF get high activity in the SCR experiments and why 10% CeO_2/ACF yield higher NO conversion than 20% La_2O_3/ACF at low temperature. When the catalysts composition was taken into consideration, those might result from the metal oxides of the loaded catalyst and the metal special electronic structure. The electronic structures of rare earth elements were characterized with containing the 4f electron. The electron structure of Ce is $4f^1 5d^1 6s^2$, and that of lanthanum is $4f^0 5d^1 6s^2$. For Ce and La electronic struc-

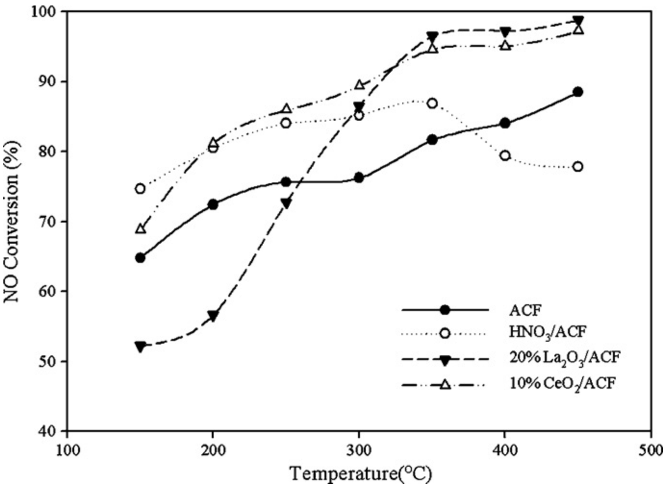

Fig. 2. Effect of catalyst species on NO conversion.

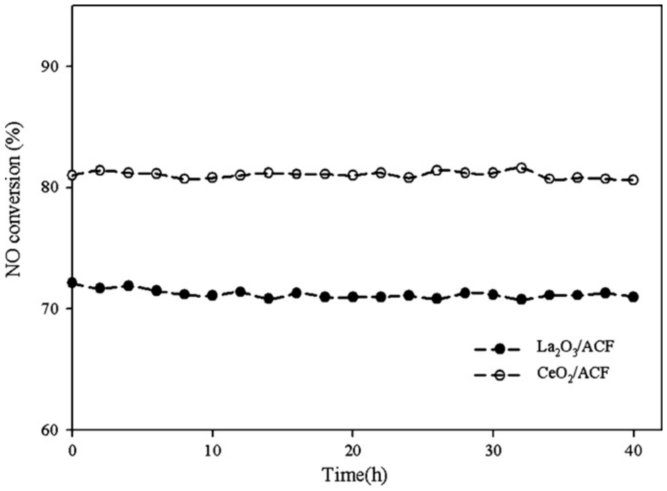

Fig. 3. Activity of the catalysts at 200 °C, over 40 h reaction.

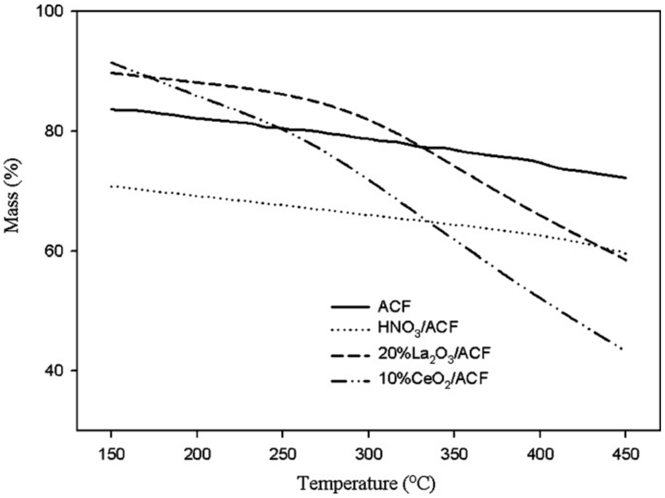

Fig. 4. TG analysis of the catalysts.

ture, their 5d orbit has only one electron, which provides a good electron transfer orbit. So such two rare earth elements could serve as catalysts for the "electron transfer station", and their compounds had high catalytic activity.

At the same time, as the 4f orbital is effectively shielded by the outer electron and $E_{4f} < E_{5d}$, so in the structure of $4f^n5d^{0-1}6s^2$, the electron of 4f orbit must be transferred to the 5d orbit in the reaction. On the other hand, there will be a formation of new bonding electron and the bond energy will be released when the electron of 4f is excited. For most of lanthanide atoms, the released bond energy is greater than the excitation energy. The 4f orbit has only one electron in Ce atom which can easily be excited. Therefore, the CeO_2/ACF could yield higher NO conversion at low temperature than La_2O_3/ACF.

3.2. *Catalyst stability experiment*

Fig. 3 shows catalytic activities of 5.0000 g 10% CeO_2/ACF and 20% La_2O_3/ACF during 42 h at 200 °C. It can be seen that 5.0000 g 10% CeO_2/ACF and 20% La_2O_3/ACF can last more than 42 h with about 81% and 70% NO conversion at 200 °C, respectively. They are higher than catalytic activity of Cu/AC prepared by electroless plating [18] and no less than that of the results reported by Lázaro et al. [19]. When the stability of catalysts is taken into consideration, the result is desirable.

3.3. *BET characterization of catalysts*

Table 1 shows the values of S_{BET}, total micropore volume (V_m), narrow micropore volume (V_{m6}) and medium size micropore volume (V_{mm}) of the catalysts. It can be observed that, as expected, upon modification there is a significant change of porosity and that of S_{BET} and micropore volume with the ACF modified by HNO_3, cerium and lanthanum oxide. The modification resulted in an obvious change in porosity due to the development of new interconnecting pores and metal oxides loaded [20]. A considerable surface area and a well-developed porosity are thought to be essential for achieving large metal oxide dispersions, which usually results in a high catalytic activity [21]. It might be said, except 30% CeO_2/ACF and 40% CeO_2/ACF, microporous structure accounted for the catalysts mainly.

Table 1
Values of S_{BET}, total micropore volume (V_m), narrow micropore volume (V_{m6}) and medium size micropore volume (V_{mm}) of the catalysts.

Sample	S_{BET} (m^2/g)	V_m (cm^3/g)	V_{m6} (cm^3/g)	V_{mm} (cm^3/g)
ACF	748.38	0.38	0.22	0.078
HNO_3/ACF	809.74	0.41	0.26	0.068
20% La_2O_3/ACF	609.50	0.29	0.15	0.052
10% CeO_2/ACF	709.54	0.36	0.21	0.065
20% CeO_2/ACF	575.82	0.29	0.14	0.046
30% CeO_2/ACF	456.21	0.23	0.08	0.032
40% CeO_2/ACF	307.32	0.16	0.04	0.020

The values of S_{BET} decreased with the increasing loaded mass of metal oxides. Pore blockages were even more noticeable when excessive amount of metal oxides were loaded, probably due to the way they were loaded since the impregnating solution was more a suspension than a solution, in agreement with what was observed in previous studies [17]. So the lowest values of S_{BET} and V_m were measured for the 40% CeO_2/ACF. The characterization of the porous structure of ACF, gave out N_2 adsorption isotherms which shape suggested that an amount destruction of the porosity occurred as a result of HNO_3 treatment.

3.4. *TG characterization of catalysts*

For the loaded catalysts, the stability of ACF, used as carrier, is the key factor to maintain the activity of the catalyst. In order to study the thermal stability of ACF, HNO_3/ACF, 20% La_2O_3/ACF and 10% CeO_2/ACF, the TG experiments were carried out in air stream, and the heating rate was 20 °C/min. The experimental data were recorded from 150 °C to 450 °C.

In Fig. 4, the chemical stability of ACF and HNO_3/ACF are higher than those of other catalysts. With the temperature increasing, their curves are almost two straight lines and parallel to each other from 150 °C to 450 °C, both of their weight loss ratios were about 0.7% of sample mass per minute. For 10% CeO_2/ACF and 20% La_2O_3/ACF, their weight loss rates are significantly higher than those of ACF and HNO_3/ACF. Especially when the temperature was over 280 °C, their weight lost ratios were both about 3.5% of sample mass per minute, which were greatly higher than those of ACF and HNO_3/ACF. At 250 °C, the mass percent of 10% CeO_2/ACF was equal to that of ACF, and while the temperature rose to 330 °C, the same phenomenon occurred between 10% CeO_2/ACF and HNO_3/ACF. For 20% La_2O_3/ACF, the similar phenomenon occurred at 330 °C and 430 °C, respectively. In Fig. 5, there are obvious sorption heat peaks in the curves of 10% CeO_2/ACF and 20% La_2O_3/ACF, while the sorption heat peak is more obvious in the curves of 10% CeO_2/ACF. However, the DTA curves of ACF and HNO_3/ACF have no obvious sorption

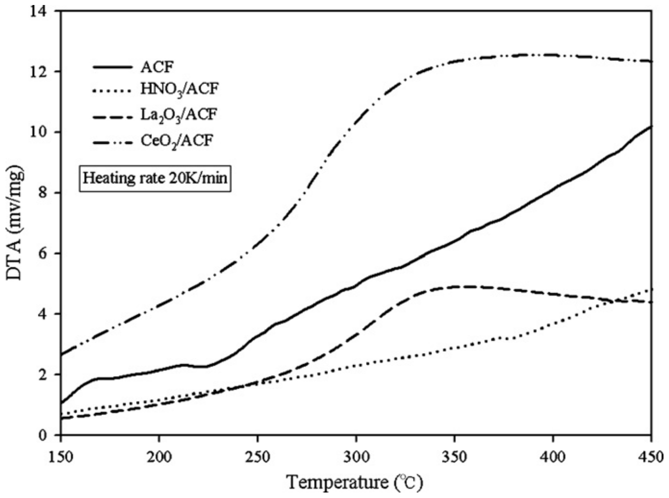

Fig. 5. DTA analysis of the catalysts.

heat area. These phenomena discussed above might result from that ACF and HNO_3/ACF were continuously oxidized in oxidizing condition, and meanwhile 10% CeO_2/ACF and 20% La_2O_3/ACF were continuously oxidized and decomposed. Therefore, the physical and chemical properties of ACF, used as carrier, had changed when ACF was modified by La_2O_3 and CeO_2, which might have relationship with the change of S_{BET}, functional groups and metal oxides loaded. Serrano-Ruiz et al. [22] also found similar phenomenon when ACF was modified.

3.5. XRD studies

Fig. 6 shows the XRD experimental results of ACF, HNO_3/ACF, 20% La_2O_3/ACF and 20% La_2O_3/ACF SCR of NO for 1.5 h. For ACF, HNO_3/ACF, the XRD patterns showed graphite-like crystallite, and when ACF was modified by HNO_3, the crystallite became smaller, which resulted in its diffraction angle becoming larger. For 20% La_2O_3/ACF and 20% La_2O_3/ACF of SCR of NO for 1.5 h, their XRD patterns do not show intense sharp peaks for La_2O_3. However, after 20% La_2O_3/ACF SCR of NO for 1.5 h, its XRD patterns became sharper and wider than that of its original state, because its surface areas became higher when the SCR of NO involved. This result is consistent with the report of Qi et al. [23] and Daley et al. [24].

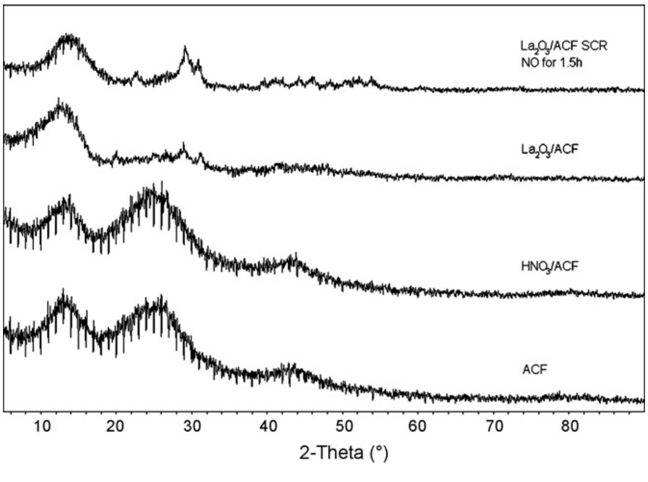

Fig. 6. XRD analysis of the catalysts.

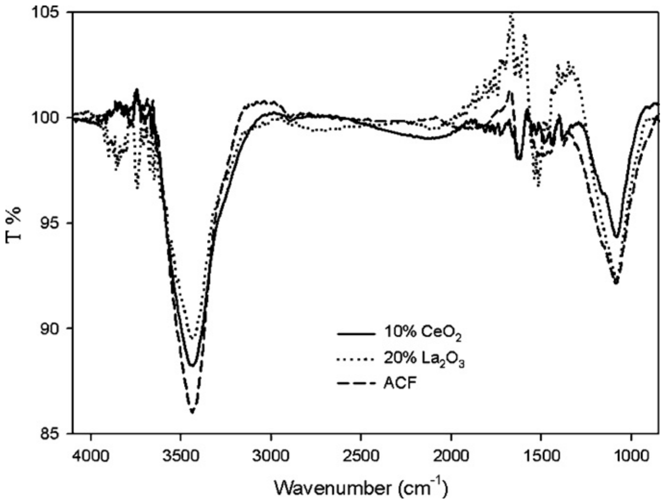

Fig. 7. FTIR analysis of the catalysts.

3.6. FTIR studies

The FTIR experimental results of ACF, 20% La_2O_3/ACF and 10% CeO_2/ACF is shown in Fig. 7. It could be easily seen that there were certain differences among their wave number bands, due to the modification of ACF by metal oxides. The heating in inert condition and nitrate decomposition might partly contribute to the surface structure change of ACF. Bands at 3440 cm^{-1} and 1080 cm^{-1} were detected and some weak bands in the range of 3860–3740 cm^{-1} and 1500–1630 cm^{-1} were also detected. The band at 3440 cm^{-1} was phenolic or hydroxyl group [25]. The weak bands from 1500 cm^{-1} to 1630 cm^{-1} could be assigned to bidentate nitrate and monodentate nitrite vibrations, respectively [23].

3.7. Mechanism of SCR of NO

Some researchers have studied the mechanism of SCR of NO and suggested some mechanism schemes. Muñiz et al. [26] suggested that NO could be oxidized on the ACF and reduced by ACF-C, and the oxidation active center was ACF-C. Adapa et al. [27] and Mochida et al. [28] proposed similar NO oxidation mechanism on ACF. Therefore, for ACF and HNO_3/ACF, when NO was reduced by NH_3 in this study, the ACF-C was also involved in the redox of NO, which resulted in that little amount of CO_2 that could be measured as described in previous paper [17]. The ACF was consumed when NO was reduced by NH_3 on the surface of ACF and HNO_3/ACF. ACF and HNO_3/ACF were catalytic center and reducing agent in this study. Therefore, the bigger BET surface area of HNO_3/ACF resulted in higher NO conversion than that of ACF at low temperature, as showed in Table 1 and Fig. 2. The following reactions were suggested to take place on surfaces of ACF and HNO_3/ACF:

$$O_2 + 2NO \rightleftharpoons 2NO_2, \quad \Delta G_1 = -70.48\,\text{kJ/mol} \tag{1}$$

$$2NO + C \rightarrow N_2 + CO_2, \quad \Delta G_2 = -567.458\,\text{kJ/mol} \tag{2}$$

$$4NO + 4NH_3 + O_2 \rightarrow 4N_2 + 6H_2O(g), \quad \Delta G_3 = -1651.832\,\text{kJ/mol} \tag{3}$$

$$2NO_2 + 4NH_3 + O_2 \rightarrow 3N_2 + 6H_2O(g), \quad \Delta G_4 = -1408.252\,\text{kJ/mol} \tag{4}$$

Judged by the Gibbs free energy of formations (ΔG, 25 °C, 100 kPa), all of the reactions could take place on the surface of ACF and HNO_3/ACF. NO_2, generated in reaction (1), could be reduced fast in reaction (4) because ΔG_4 was −1408.252 kJ/mol. However, because ΔG_1, −70.48 kJ/mol, was the highest one in ΔG_{1-4}, reaction (4) was limited by reaction (1). Therefore, the main reactions on the surface of ACF and HNO_3/ACF were reactions (2) and (3).

Kang et al. [10,30,31] reported that metal oxides, such as CeO_2, CuO and MnO_x, could catalyze the reduction of NO with NH_3 by transferring the electron. Miura et al. [29] and Park and Kim [18] reported metal (Ni, Cu), loaded porous carbon, was electron transfer station during NO reduction, and both of them also suggested that C was a reducing agent. Marbán et al. [32] reported the mechanism of NO reduction with NH_3 over carbon-supported Mn_3O_4, Mn_3O_4 was also the electron transfer station among O_2, NO and NH_3, and the function of carbon was a reducing agent. In this study, there were metal oxides and ACF-C in the loaded catalysts. Therefore, NO conversion mechanisms on loaded catalysts were different from that of ACF and HNO_3/ACF. During the process of NO reduction of the loaded catalysts, the catalytic centers were metal oxides [17,26,29,30]. Therefore, reaction (1) took place mainly on the metal oxides, rather than the surface of ACF and HNO_3/ACF. The NO reduction processes on loaded catalysis could be showed as follows:

$$2MO + O_2 \leftrightharpoons 2MO_2^{\bullet} \tag{5}$$

$$MO_2^{\bullet} + NO \leftrightharpoons NO_2 + MO \tag{6}$$

$$MO_2^{\bullet} + 2NO + 2NH_3 \rightarrow 2N_2 + 3H_2O + MO \tag{7}$$

$$2MO_2^{\bullet} + 2NO_2 + 4NH_3 \rightarrow 3N_2 + 6H_2O + 2MO \tag{8}$$

$$MO_2^{\bullet} + \text{ACF-C} \rightarrow MO + CO \tag{9}$$

$$MO_2^{\bullet} + CO \rightarrow MO + CO_2 \tag{10}$$

$$2CO + 2NO + O_2 \rightarrow N_2 + 2CO_2, \quad \Delta G_5 = -685.48\,\text{kJ/mol} \tag{11}$$

Reactions (5) and (7) could be summarized as reaction (3), and reactions (5) and (8) could be summarized as reaction (4). However, though Kang et al. [30] and Shen et al. [31] suggested the overall process of NO reduction was reaction (5), and the reaction (4) should be the main overall process during NO reduction in this study, which was not only because of the research results of Marbán et al. [32], but also due to the difference of ΔG_3 and ΔG_4. At the same time, because of the existence of little amount CO_2 in the outlet gases, the ACF-C, the carrier of metal oxides, also took place, as reactions (5) and (9)–(11) showed. Therefore, compared with reactions (5) and (7), reactions (9)–(11) were very minor.

4. Conclusions

The SCR activity of ACF could be greatly improved by HNO_3 and loaded metal oxides (CeO_2 and La_2O_3), especially by CeO_2 and La_2O_3, and the best suitable loaded mass percentage of CeO_2 is 10%. Among the catalysts studied, the catalytic activity of 10% CeO_2/ACF and HNO_3/ACF is higher than that of other two catalysts at low temperature, and meanwhile, the 20% La_2O_3/ACF and 10% CeO_2/ACF can maintain higher activity at higher temperature. When the temperature rises above 350 °C, 20% La_2O_3/ACF yields over 90% NO conversion, and the 10% CeO_2/ACF almost yields the same NO conversion. However, the stability of HNO_3/ACF and the catalytic activity of 20% La_2O_3/ACF at low temperature should be improved in the future researches.

Furthermore, the SCR of NO mechanism of ACF and HNO_3/ACF is different from that of 10% CeO_2/ACF and 20% La_2O_3/ACF. For ACF and HNO_3/ACF, the ACF-C is reducing agent and catalytic center. While for the loaded catalysts, the catalytic centers are metal oxides, and the reducing agents are NH_3 and CO.

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