



A novel four-way combining catalysts for simultaneous removal of exhaust pollutants from diesel engine

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Abstract

A novel four-way combining catalysts containing double layers was applied to simultaneously remove four kinds of exhaust pollutants (NO_x, CO, HC and PM) emitted from diesel engine. The four-way catalysts were characterized using scanning electron microscope (SEM) and Ultraviolet visible diffuse reflectance spectroscopy (UV-Vis DRS). Their catalytic performances were evaluated by temperature-programmed reaction technology. The double layer catalysts could effectively remove the four main pollutants. The highest catalytic activity was given by the two-layered catalysts of La_{0.6}K_{0.4}CoO₃/Al₂O₃ and W/HZSM-5. Under the simulated exhaust gases conditions, the peak temperature of the soot combustion was 421°C, the maximal conversion of NO to N₂ was 74%, the temperature of the HC total conversion was 357°C, and the maximum conversion ratio of CO was 99%.

Key words: four-way combining catalyst; diesel engine exhaust; double layers; perovskite oxides; zeolite catalysts

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Introduction

With growing urbanization and increasing the numbers of automobiles, exhaust gas emissions started to cause a serious stress to air quality particularly in urban areas. The regulations include the limits on nitrogen oxides (NO_x), CO, particulate matters (PM) and hydrocarbon (HC) emissions. The catalytic removal is one of the most efficient methods (Yu et al., 2006). Most of automobiles in the developed countries such as United States, Europe and Japan are equipped with three-way catalysts (TWC). Through more than 30 years of research and development, significant reductions in CO, NO_x, and HC emissions have been realized. The TWC technology has been applied under the condition with oxygen free or around net oxygen-free, the technology was not suitable for the efficient removal of the pollutant emitted from diesel engines. Therefore, in order to reduce NO_x, CO, and HC as well as PM emissions, the effective four-way catalysts are needed developed.

The diesel-exhaust control technology is currently being developed. For PM emission, a filter in combination with oxidation catalysts, can be reduced up to 90%. The key of this technology is to develop the highly active catalysts which can catalyze soot oxidization at low temperature (Liu et al., 2008a). The perovskite-type oxides containing transition metal ions have been attracted interest for diesel soot oxidization elimination (Liu et al., 2007; Teraoka et al., 2000). Teraoka et al. (2001) found that La-K-Mn-O perovskite-type oxides were good candidate catalysts for

diesel soot combustion. Wang et al. (2005) investigated the catalytic performances of La_{1-x}K_xMnO₃ catalysts for soot particle combustion, and found that the nanometric La_{1-x}K_xMnO₃ oxides with the substitution quantity between $x = 0.20$ and $x = 0.25$ were good candidate catalysts for the soot particle removal reaction under loose contact conditions. In addition, several authors have reported that perovskite-type oxides were active for simultaneous NO_x-soot removal reaction (Peng et al., 2007; Misono, 2005). The incorporation of K into A-sites of perovskite-type (ABO₃) oxides was found to be quite effective for enhancing the activity and selectivity for the NO_x-O₂-soot reaction (Hong and Lee, 2000; Fino et al., 2003). However, due to the large ratio of air to fuel (A/F), excess O₂ exists in the exhaust, and thus, the conversion of nitrogen oxides to N₂ over perovskite catalysts was low (Wang et al., 2008a). To solve this problem, the selective catalytic reduction of NO with hydrocarbons (HC-SCR) has been extensively investigated. Wang et al. (2007) reported that the C₂H₂-SCR on metal oxide-exchanged zeolites (Ce, Zr) had high catalytic activity for NO conversion to N₂ in the presence of excess oxygen, and previous studies reported that Ag/Al₂O₃-C₂H₅OH combined system had very high catalytic activity for NO conversion and reduction (Li et al., 2009; Zhang et al., 2009).

For pollutants, such as NO_x, CO, HC and PM, which are harmful to the human being and atmosphere, HC-SCR technology can effectively reduce the pollution of NO_x and diesel particulate filter (DPF) can efficiently control the emissions of PM. However, as far as we know,

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there is little report about the available technologies to simultaneously reduce these four pollutants. Most of the control technologies addressed either the reduction of NO_x emissions such as HC-SCR or the oxidation of particulate matter. In this article, we introduced a new method, i.e., four-way combining catalysts composed of double layers. It can integrate the beneficial qualities of the two kinds of catalysts by the means of combining double layer catalysts and can simultaneously remove NO_x, CO, HC and PM pollutants in exhausts.

1 Experimental

1.1 Catalyst preparation

The La_{1-x}K_xMnO₃ or La_{1-x}K_xCoO₃ perovskite-type oxides supported on the Al₂O₃ were prepared by impregnation method. The precursor was calcined at 800°C for 6 hr in static air, pressed into pellets, and sieved using 40–80 mesh for use.

HZSM-5-supported metal oxides were prepared by an incipient-wetness impregnation method and were denoted as M/HZSM-5 (M = Cr, Mo, W). Then, the samples were dried at 120°C overnight and calcined at 500°C in air for 5 hr. Finally, they were pressed into pellets, and sieved through 20–40 mesh for use.

1.2 Catalyst characterization

The UV-Vis DRS experiments of the catalysts were performed on UV-Vis spectrophotometer (U-4100, Hitachi Ltd., Japan) with the integration sphere diffuse reflectance attachment. The powder samples were loaded in a transparent quartz cell and were measured in the region of 200–800 nm at room temperature. The standard support reflectance was used as the baseline for the corresponding catalyst measurement. The powder morphology was observed by a scanning electron microscope (S-360, Cambridge Ltd., UK).

1.3 Catalytic activity measurement

The activities of the four-way catalysts were evaluated by the technique of the temperature-programmed reaction (TPR). The prepared samples were placed in a fixed-bed quartz reactor (*d*_i = 6 mm). HZSM-5-supported transitional oxides of 0.2 g were placed at bottom layer, and 0.1 g La_{0.6}K_{0.4}CoO₃ or La_{0.5}K_{0.5}MnO₃ perovskite-type oxides supported on the Al₂O₃ and 0.05 g soot (20:1, W/W) were loosely mixed and placed at top layer. The two-layered of the catalysts were separated by quartz wool. Then, their catalytic performances for the simultaneous removal of PM, CO, HC and NO_x were measured. The reaction temperature was controlled by a PID-regulation system based on the measurements of a K-type thermocouple and varied from 200 to 600°C at a temperature rising rate of 2°C/min during each TPR run. The soot used in this work was Printex-U supplied by Degussa Company (Germany) as model soot. Its primary particle size was 25 nm and specific surface was 100 m²/g. Reactant gases containing 5 Vol.% O₂, 1000 ppm C₂H₂, 2000 ppm CO and 2000 ppm NO balanced with He were passed through

the quartz micro reactor at a flow rate of 50 mL/min. The outlet gas mixture from the reactor passed through a 1-cm³ sampling loop of a six-point gas-sampling valve before it was being injected into an on-line gas chromatograph (GC, sp-3420, Beijing Analytical Instrument Factory, China). The GC was equipped with a thermal conductivity detector (TCD) and a flame ionization detector (FID) to analyze the gaseous mixture. The TCD was used to measure the concentration of O₂, N₂, C₂H₂, CO after separating these gases over molecular sieve 5A column. The FID was employed to determine CO, CO₂ and C₂H₂ concentrations after separating these gases over a Porapak N column and converting them to methane over a Ni catalyst at 380°C.

From the TPR results, the four parameters were derived to evaluate the performances of the catalysts. The first one is the peak temperature of the soot maximal combustion rate (*T*_m), and the second one is the maximal conversion of CO (*X*_m^{CO}), and the third one was the temperature of the HC total conversion (*T*_f) obtained by the temperature of corresponding the zero hydrocarbon concentration. The last one is the maximal conversion of nitrogen oxide to nitrogen molecule (*X*_m^{NO}). The *X*_m^{NO} can be defined as:

$$X_{\text{NO}}^{\text{m}} = \frac{2[\text{N}_2]_{\text{out}}}{[\text{NO}]_{\text{in}}} \times 100\% \quad (1)$$

where, [NO]_{in} (ppm) is NO concentration in the inlet gases, and [N₂]_{out} (ppm) is N₂ concentration in outlet gas.

2 Results and discussion

2.1 SEM results of oxidative catalysts

The SEM photographs of La_{0.6}K_{0.4}CoO₃/Al₂O₃, La_{0.5}K_{0.5}MnO₃/Al₂O₃ and Al₂O₃ are exhibited in Fig. 1. The SEM images of La_{0.6}K_{0.4}CoO₃/Al₂O₃ and La_{0.5}K_{0.5}MnO₃/Al₂O₃ show that the catalyst particles have dispersed on the surface of the Al₂O₃ support. According to the results of SEM characterization, it was proposed that the formation of the perovskite-oxides was located at the external surface of Al₂O₃.

2.2 UV-Vis DRS results of reductive catalysts

Figure 2 shows the UV-Vis DRS spectra of M/HZSM-5 (M = Cr, Mo, W). There was one band at about 200–400 nm for all of the catalysts, which was due to the charge transfer from O₂⁻ to transitional metal ions with oxidation state M⁶⁺. At the same time, weak d–d electron transition bands of Cr(III) which are due to 4A^{2g}→4T^{2g} and 4A^{2g}→4T^{1g} located at 450–600 nm. The absorption spectra of Mo/HZSM-5 and W/HZSM-5 are very similar. The overlapped band at about 210–300 nm appeared in these two samples. At this absorption region, the peak of Mo/HZSM-5 is broader than that of W/HZSM-5. No absorbance band could be observed in the range of 500–800 nm. Thus, it can be concluded that the status of molybdenum and tungsten are Mo(VI) and W(VI) (Liu et al., 1997). It is beneficial for the C₂H₂-SCR of NO to N₂. Isolated Mo (T_d) species formed by molybdenum incorporation to HZSM-5 favor the target reaction by accelerating NO oxidation with the acidic sites' cooperation

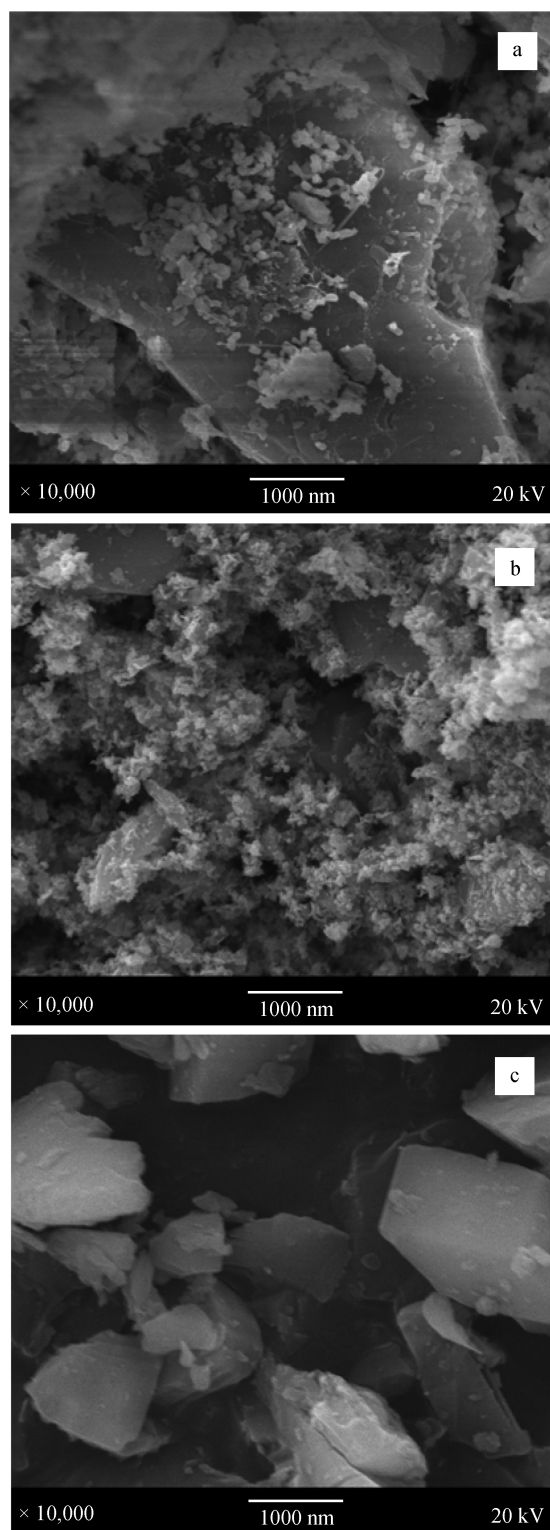


Fig. 1 SEM photographs of catalysts and Al_2O_3 . (a) $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$; (b) $\text{La}_{0.5}\text{K}_{0.5}\text{MnO}_3/\text{Al}_2\text{O}_3$; (c) Al_2O_3 .

or by strengthening the adsorption of NO_x on the catalyst surface (Wang et al., 2007).

In addition, the d electron transition of tungsten (d^4) is easier than that of molybdenum (d^5) (Wang et al., 2008b). Therefore, the activity for C_2H_2 -SCR of NO reaction over W/HZSM-5 would be better than that over Mo/HZSM-5.

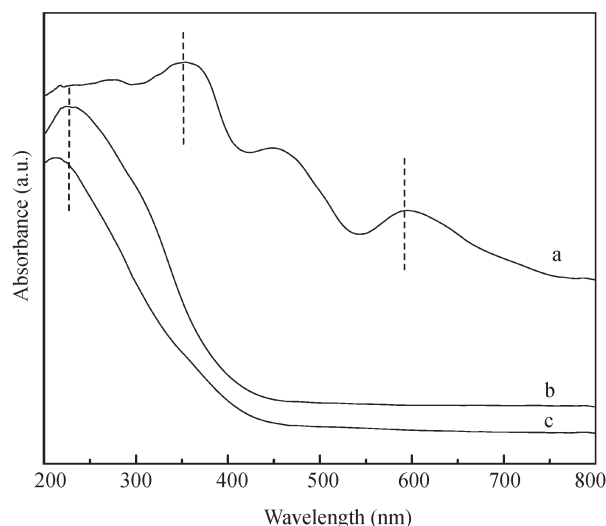


Fig. 2 UV-Vis spectra of Cr/HZSM-5 (line a), Mo/HZSM-5 (line b), and W/HZSM-5 (line c).

2.3 Evaluation results of catalytic performances

As far as we know, the catalysts reported in public literature could not efficiently simultaneously remove the four kinds of exhaust pollutants emitted from diesel engine. To solve this problem, two catalysts prepared in this study were filled in the reactor, the mixing gases of CO, C_2H_2 and NO firstly went through the NO_x reduction catalyst, then through the mixture of oxidative catalyst and soot.

The four-way catalytic activities of double-layer catalysts are shown in Table 1. In this work, the perovskite-type catalysts $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$ or $\text{La}_{0.5}\text{K}_{0.5}\text{MnO}_3/\text{Al}_2\text{O}_3$ separately assembled with the Cr/HZSM-5, Mo/HZSM-5 or W/HZSM-5.

In Table 1, it can be seen that the double-layer catalysts could effectively oxidize the PM, CO, HC and reduce NO to N_2 . The conversion of NO to N_2 could exceed 70% over the $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM-5}$. Especially, the X_m^{NO} over the $\text{La}_{0.5}\text{K}_{0.5}\text{MnO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM-5}$ was 79% that can not be achieved only on the perovskite-type catalyst. And the temperature of the total conversion (T_f) of HC over the $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM-5}$ was 357°C. It seems to directly correlate with the content of oxygen vacancy (ν_0) in perovskite-type oxides of $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$ and $\text{La}_{0.5}\text{K}_{0.5}\text{MnO}_3/\text{Al}_2\text{O}_3$. The oxygen vacancy enhanced the adsorption and activation oxygen at catalyst surface and thus improved the oxidation activity (Wang et al., 2005). On the other hand, the result showed that the conversion of NO was correlative with T_m

Table 1 Catalytic performance of double layer catalysts for the simultaneous removal of NO_x , CO, C_2H_2 and soot

Catalyst	X_m^{NO} (%)	T_m (°C)	T_f (°C)	X_m^{CO} (%)
$\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{Cr}/\text{HZSM-5}$	48	431	296	99
$\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{Mo}/\text{HZSM-5}$	41	402	349	98
$\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM-5}$	74	421	357	99
$\text{La}_{0.5}\text{K}_{0.5}\text{MnO}_3/\text{Al}_2\text{O}_3 + \text{Cr}/\text{HZSM-5}$	64	392	299	97
$\text{La}_{0.5}\text{K}_{0.5}\text{MnO}_3/\text{Al}_2\text{O}_3 + \text{Mo}/\text{HZSM-5}$	51	395	373	94
$\text{La}_{0.5}\text{K}_{0.5}\text{MnO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM-5}$	79	448	364	98

of soot combustion, for example, X_m^{NO} was increased from 64% to 79% and the T_m also increased by 56°C, but the maximal conversion of CO (X_m^{CO}) was not influenced by them and was more than 90%.

In order to compare the catalytic activity of the double-layer catalysts with that of the single layer catalyst for simultaneous removal of four pollutants, the TPR curves are shown in Fig. 3.

Figure 3a exhibits the result of NO conversion to N_2 over single layer catalyst ($\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$) and the double layers catalysts ($\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM}-5$). It can be seen that the reductive catalytic activity of the double layer catalysts were much higher than that of single layer catalyst. The maximum of NO conversion to N_2 over the double layer catalysts was increased by 34%. Figure 3b shows the result of single layer catalyst ($\text{W}/\text{HZSM}-5$) compared with the double-layer catalysts ($\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM}-5$). The temperature for soot combustion was about 638°C over the single layer catalyst, but it was lowered by 200°C on the double layer

catalysts comparing with that on the $\text{W}/\text{HZSM}-5$ catalyst. Thereby, it is apparent that perovskite-type oxides and $\text{M}/\text{HZSM}-5$ ($\text{M} = \text{Cr}, \text{Mo}, \text{W}$) double-layer catalysts are excellent candidate catalysts for simultaneous purification of the four pollutants emitted from diesel engine.

2.4 Effect of different assemble sequence for the two-layered catalysts of $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$ and $\text{W}/\text{HZSM}-5$

In order to investigate the effect of the assembling sequence, the $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM}-5$ and $\text{W}/\text{HZSM}-5 + \text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$ were investigated under the same reaction conditions (the reactant gases containing 5 Vol.% O_2 , 1000 ppm C_2H_2 , 2000 ppm CO and 2000 ppm NO balanced with He were passed through the quartz micro reactor at a flow rate of 50 mL/min).

Table 2 lists the activity measurement results of the different assembling sequence of catalysts. When $\text{W}/\text{HZSM}-5$ was assembled at the bottom of reactor, the maximum of NO conversion to N_2 is much higher than that $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$ was assembled at the bottom of reactor, while the oxidization activity was not lowered. It is concluded that the assembling sequence of catalysts affected the result of the reaction. In this study, the reactant gas mixture flow upwards, i.e., from the bottom to the top of the reactor. It is much better that reductive catalyst at bottom of reactor and the NO could be selectively converted by the C_2H_2 . If the oxidative catalyst was assembled at the bottom of reactor, the C_2H_2 was firstly oxidized by the catalyst and NO could not be selectively reduced to N_2 .

Table 2 Effect of different combining method of the $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$ and $\text{W}/\text{HZSM}-5$

Catalyst	X_m^{NO} (%)	T_m (°C)	T_f (°C)	X_m^{CO} (%)
$\text{W}/\text{HZSM}-5 + \text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$	15	424	322	99
$\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM}-5$	74	421	357	99

2.5 Relationship between the physic-chemical properties of the double layer catalysts and their catalytic performances

It is a complicated multi-phase reaction process for catalytic conversion reaction of four exhausts emitted from diesel engine. There are several main controlling factors which govern the catalytic properties of soot, CO, HC oxidation and NO reduction over the mixed oxide catalysts.

On one hand, the good redox properties of the oxidative catalysts are very important, especially the oxidizing ability of B-site cation in ABO_3 perovskite-type oxides. This is the most important one for the activation and oxidation of the soot, CO, and HC, which is due to the intrinsic property of the catalysts. For perovskite-type oxide catalyst, B-site cation plays a key role in the oxidation of soot, CO, HC. The cation at A-site (La^{3+}) is generally trivalent, as La^{3+} at A-site is replaced by lower valency cation K^+ , according to the principle of electron neutrality, the positive charge reduced could be balanced either by the

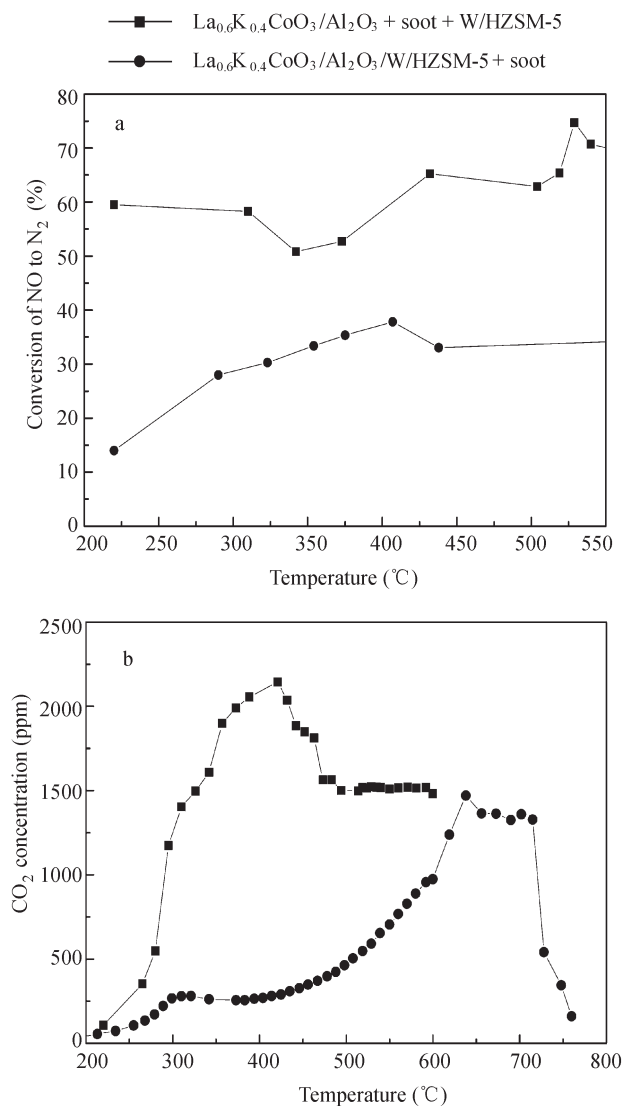
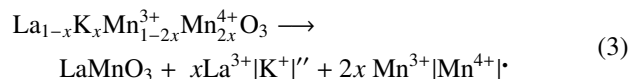
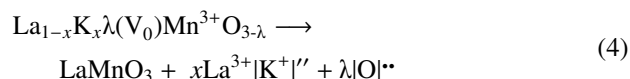


Fig. 3 TPR profiles for NO conversion to N_2 (a) and for the oxidation of C_2H_2 , CO and soot (b) over the double layers catalysts of $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM}-5$ and the single layer catalyst of $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3$ or $\text{W}/\text{HZSM}-5$.

formation of higher oxidation state ion (Co^{4+} or Mn^{4+} ions in the complex oxide.) at B-site (Wang et al., 2005; Liu et al., 2008b; Liu et al., 2008c), or by the formation of oxygen vacancy in perovskite-type catalysts, i.e.,



or by the formation of oxygen vacancy (V_O) in $\text{La}_{1-x}\text{K}_x\text{MnO}_3$,



The oxygen vacancy is favorable for the adsorption and activation of oxygen. On the other hand, the presence of oxygen vacancy is favorable for the mobility of lattice oxygen, thus enhance the catalytic activity for oxidation. Moreover, the SEM results reveal that, after part of La^{3+} is replaced by K^+ , the composite oxides have a uniform and fine particle size, and thus they are easier to be reduced and exhibit a lower reduction peak temperature. Therefore, the catalytic activity of the catalysts for soot, CO, HC oxidation could be enhanced.

For the reduction of NO, the structure of Mo/HZSM-5 and W/HZSM-5 affected the catalytic performances on the reaction of C_2H_2 -SCR of NO. One of the reasons is maybe that the pore size of zeolite exactly fitted for the diameter of C_2H_2 molecule. Another reason is the optimal loading amount of molybdenum and tungsten on HZSM-5 which changes in direct proportion with the population of acid sites on the zeolite (Falcon et al., 2003). Moreover, in the HC-SCR of NO, the reductant also plays a very important role in determining the activity of the catalyst system. Wang et al. (2007) reported that the NO elimination level using C_2H_2 was ca. 3–4 times as high as that using methane or propene as reductant under the same reaction conditions over the same catalyst. Acetylene has rather lower adsorption tendency on the catalysts compared with propene, which causes the catalyst's rapid deactivation in the C_3H_6 -SCR of NO. HZSM-5 is a better catalyst support for the C_2H_2 -SCR of NO. Acidic sites in the zeolite are essential to the C_2H_2 -SCR reaction. Therefore, the incorporation of aluminum considerably improved the catalytic performance of HZSM-5 zeolite, while the Si/Al ratio increase in the zeolite framework resulted in the activity decrease. Isolated W (or Mo) species formed by W (Mo) incorporation to HZSM-5 favors the C_2H_2 -SCR reaction both by accelerating NO oxidation with the acidic sites' cooperation, and by strengthening the adsorption of NOx on the catalyst surface. In summary, the good results of the catalytic purification for the four pollutants (NOx, CO, HC and PM) were obtained over the four-way combining catalyst in this work.

3 Conclusions

The four-way combining catalyst technology is an efficient method to purify the four main kinds of pollutants

emitted from diesel engines.

The combination of oxidative catalysts and reductive catalysts could purify the exhaust gases emitted from diesel engine without disturbing each other. It is clear that the perovskite-type catalysts could effectively oxidize the PM, CO, HC and the W/HZSM-5 catalyst could efficiently reduce the NO to N_2 . Over the $\text{La}_{0.6}\text{K}_{0.4}\text{CoO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM-5}$ and $\text{La}_{0.5}\text{K}_{0.5}\text{MnO}_3/\text{Al}_2\text{O}_3 + \text{W}/\text{HZSM-5}$, the peak temperature of soot combustion was 421 and 448°C, respectively. It was decreased by more than 200°C compared with the peak temperature of the bare soot combustion (without catalyst). The conversion of NO to N_2 was above 70%; the temperature of the HC total combustion was below 364°C, and the maximum selectivity of CO_2 production was above 97%.

Acknowledgments

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Erratum

- (1) In “Ralf Kunkel, Peter Kreins, Björn Tetzlaff, Frank Wendland, 2010. Forecasting the effects of EU policy measures on the nitrate pollution of groundwater and surface waters. *Journal of Environmental Sciences*, 22(6): 871–877”, the word “REGFLUD” should be “AGRUM”.
- (2) In “Yoshiaki Tsuzuki, Masato Fujii, Yasuo Mochihara, Kouki Matsuda, Minoru Yoneda, 2010. Natural purification effects in the river in consideration with domestic wastewater pollutant discharge reduction effects. *Journal of Environmental Sciences*, 22(6): 892–897”, the correct form of Fig. 4 is as the following:

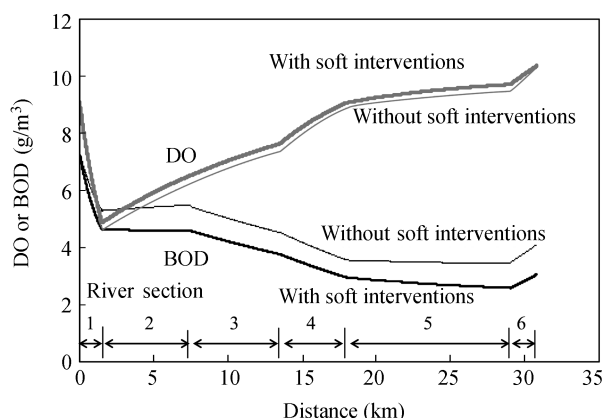


Fig. 4 BOD and DO estimation results of one dimensional water quality model with and without “soft interventions” in households to reduce pollutant discharge.