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# Catalytic reduction of nitric oxide by carbon monoxide over coal gangue hollow ball

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ABSTRACT

The catalytic effect of coal gangue hollow ball with open pores on NO reduction by CO in fluidized bed was investigated, and the kinetics for the catalytic reduction of NO using CO was evaluated. In the absence of water vapor at 773–923 K, the reaction rate constant was

$$k = 5210\exp(-113600/(RT)).$$

NO reduction ratio rapidly increased with reaction residence time at initial stage (<1 s) and $T > 873$ K; high NO reduction ratio was achieved by increasing reaction temperature when the initial concentration ratio of CO to NO was less than 4. In the presence of water, the catalytic effect of coal gangue hollow ball on NO reduction was weakened, and the reaction rate constant was

$$k = 0.31\exp(-60750/(RT)).$$

NO reduction ratio is less than 0.2 when reaction residence time was 2.5 s at $T \leq 923$ K when the initial concentration ratio of CO to NO was 4, and the initial concentration ratio of CO to NO had negligible effect on NO reduction ratio at $T \leq 923$ K.

## 1. Introduction

Emissions of nitrogen oxides ($NO_x$) are of great concern because of their impacts on the environment, including acid rain and photochemical smog formation. $NO_x$ is the major combustion-generated pollutant from fossil fuel combustion [1]. The reduction of $NO_x$ emissions has become one of the greatest challenges in environment protection. The most practical and convenient method for removing nitric oxide (NO) is to use unburned compounds such as carbon monoxide (CO) and hydrocarbons as reducing agent in flame [2]. The use of CO as reducing agent has advantages for $NO_x$ reduction in practical application because of its presence in significant amounts in combustion process, CO oxidation is carried out by NO reduction, and NO removal is usually accomplished via reduction to form $N_2$ [3]:

$$NO + CO \longrightarrow CO_2 + 1/2N_2. \tag{R1}$$

The key steps in the reaction are the breaking of the NO molecule and a subsequent CO oxidation.

The catalytic reduction of $NO_x$ by CO has been one of the most important scientific challenges during the last few decades. For fluidized bed combustion of coal, several authors found that the rate of NO reduction increased significantly in the presence of CO, and solids presenting in a fluidized bed, such as calcined limestone (CaO), alumina ($Al_2O_3$) and sand ($SiO_2$), were found to catalyze NO reduction by CO [4–8]. It is well known that $SiO_2$, $Al_2O_3$ and $TiO_2$ have interesting capacities as catalyst support for $NO_x$ reduction, and base metal oxide catalysts are used to reduce NO by CO [9,10]. Tokio Iizuka found that the $Fe_2O_3$ supported on $SiO_2$ was the most active catalyst among a series of $Fe_2O_3$ catalysts for $NO_x$ reduction [11]. Acke et al. [12] found that CaO was capable of catalyzing the NO reduction by CO and the effectiveness depended on its catalytic activity and surface. Water vapor is inevitable in the flue gas of hydrocarbon fuel combustion, and it influences NO catalytic reduction by CO [13].

Coal gangue is a main industrial solid waste, and the chemical compositions are $SiO_2$, $Al_2O_3$, $Fe_2O_3$, CaO and some impurities, and these base metal oxides can be used as catalysts for NO reduction by CO. However, coal gangue has high sulfur low specific surface area, and the catalysis for NO reduction is not noticeable and results in high $SO_x$ emission. If coal gangue is prepared to hollow ball and sintered under oxidizing condition, the catalysis for NO reduction is enhanced and no $SO_x$ emissions.

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The goal of the paper is to investigate the catalytic effect of coal gangue hollow ball with open pores on NO reduction by CO in fluidized bed and to evaluate the kinetics for the catalytic reduction of NO using CO.

## 2. Experimental

### 2.1. Structural properties of coal gangue hollow sphere with open pores

In the experiment, coal gangue hollow ball with open pores is used as catalyst for NO reduction by CO in fluidized bed reactor. The preparation process of coal gangue hollow ball with open pores includes the following steps: coal gangue is smashed into 0.1–10 μm; the powders are mixed with water, $Y_2O_3$ sintering additive and carboxymethyl cellulose dispersing agent (CMC) to make slurry by mechanical stirring for 12 h, followed by the gradual addition of dissolved propyl gallate/TritonX-114 in the surry, and the suspension foam surry is prepared [14]; the stable foam slurry is rapidly atomized and dried, and then dry coal gangue hollow balls are prepared; the dry sphere is sintered at 1223 K under oxidizing condition for 2 h, and the microstructures are shown in Fig. 1. Field emission-scanning electron microscope (FE-SEM) images were recorded using a JSM-6701F instrument. Known from FE-SEM image of inner structure of hollow ball, there are many smaller hollow balls in the coal gangue hollow ball with open pores, thus the ball has high specific surface area (SSA). SSA ($m^2$/g) was calculated by the BET method from the $N_2$ adsorption/desorption isotherms obtained at 77 K [15]. The properties of hollow balls are shown in Table 1, and there are no C and S residuals due to the sintering oxidization of hollow balls at 1223 K for 2 h before experiments.

The chemical compositions of sintered coal gangue hollow ball were measured by Model 1800 X-ray fluorescence (XRF) analyzer, and the result is shown in Table 2. The coal gangue hollow ball consists of $SiO_2$, $Al_2O_3$, $Fe_2O_3$, CaO, $TiO_2$ and $K_2O$.

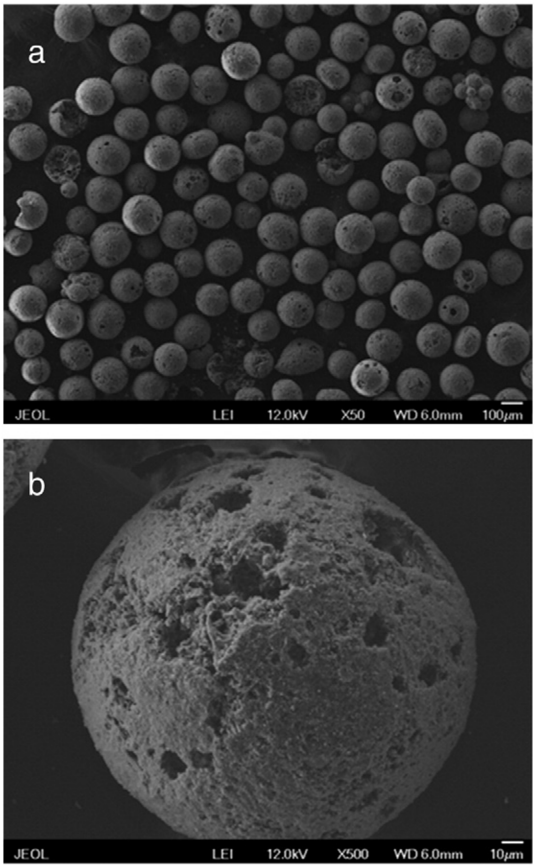

**Fig. 1.** FE-SEM images showing the microstructures of coal gangue hollow ball (a) and hollow ball with open pores (b).

**Table 1**
BET surface, average ball size and density of coal gangue hollow ball.

| Specific surface area ($cm^2$/g) | Average ball size (μm) | Real density ($kg/m^3$) |
|---|---|---|
| 253.9 | 210 | 2320 |

### 2.2. Apparatus

Fig. 2 shows a schematic diagram of the experimental apparatus, and it consists of a gas assembly, reactor, gas analyzer and a temperature controller. The quartz reactor is placed inside of the electrically heating furnace, its inner diameter and height are respectively 25 mm and 600 mm, and the height of valid reaction zone is 300 mm. A gas distributor made of sintered quartz was mounted at the lower part of the reactor, which was located at 200 mm from the reactor inlet. The gas components were provided by high pressure cylinders of argon ($Ar \geq 99.99\%$), carbon monoxide ($CO \geq 99.99\%$), and standard NO prepared in advance (5% NO, Ar balance). The gas flow of every component gas was controlled precisely by mass flow meters, gas concentrations were adjusted by mass-flow controllers, and gases were mixed in a gas mixer. In order to investigate the effect of $H_2O$ on catalytic reduction of NO, the gas mixture went through liquid $H_2O$ contained in a bottle, and the relative humidity and temperature of gas mixture at the outlet were 84% and 305 K, respectively. The gas was analyzed with Flue Gas Analyzer (Ecom-J2KN) manufactured by Prism Gas Detection Pvt. Ltd. The maximum relative error for the measured species was 5%.

In the experiment, the initial concentration ratio of CO to NO is important for NO reduction, and the ratio ($\varepsilon$) is defined as

$$\varepsilon = \frac{[CO]_{in}}{[NO]_{in}} \tag{1}$$

where $[CO]_{in}$ and $[NO]_{in}$ are the CO and NO concentrations at the reactor inlet (ppm), respectively. $[NO]_{in}$ was kept constant (315 ppm), and $\varepsilon$ ranged from 1 to 15.

In order to describe the extent of NO reduction, the conversion ($x$) is defined as

$$x = \frac{[NO]_{in} - [NO]_{out}}{[NO]_{in}} \tag{2}$$

where $[NO]_{out}$ is the NO concentration at the reactor outlet, ppm.

Gas reaction residence time ($t$) is defined as

$$t = \frac{V}{F} \times \frac{T_f}{273.15} \tag{3}$$

where $F$ is the gas flow rate at 101.325 kPa and 273.15 K, and it is kept constant, $2 \times 10^{-5}$ $m^3$/s in the experiment; $V$ is the reactor volume of the valid reaction zone, $m^3$; $T_f$ is the reaction temperature, K. Gas reaction residence time ranges from 1.5 to 2.6 s.

## 3. Results and discussion

### 3.1. Reaction scheme and rate measurement

Fig. 3 shows NO and CO concentration variations at reactor inlet and outlet, and the results indicate that the consumption of carbon monoxide coincides approximately with that of the nitric oxide:

$$[NO]_{in} - [NO]_{out} = [CO]_{in} - [CO]_{out}. \tag{4}$$

The initial and final chemical compositions of sintered coal gangue hollow balls were not changed in the experiment of catalytic reduction

**Table 2**
Compositions of coal gangue hollow balls before/after sintering by XRF analysis.

| Sintering | Content (wt%) | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | $TiO_2$ | $K_2O$ | $Na_2O$ | MgO | MnO | $SO_3$ | $ZrO_2$ | $Cr_2O_3$ | $P_2O_5$ | SrO | NiO | ZnO | $Rb_2O$ | $Y_2O_3$ |
| Before | 53.389 | 27.703 | 5.638 | 3.739 | 1.147 | 1.131 | 0.098 | 0.354 | 0.034 | 6.533 | 0.039 | 0.049 | 0.083 | 0.035 | 0.011 | 0.008 | 0.007 | 0.006 |
| After | 51.358 | 33.832 | 7.211 | 4.163 | 1.332 | 1.255 | 0.129 | 0.426 | 0.039 | 0.00 | 0.047 | 0.046 | 0.085 | 0.041 | 0.013 | 0.009 | 0.008 | 0.006 |

NO, and then it appears that reaction scheme is the reaction (R1). The similar reaction scheme was reported for NO reduction which was catalyzed over the surface of char or calcined limestone [2,4–7,16].

The reaction rate of R1 is evaluated by assuming that the surface-catalyzed reaction is first-order with respect to both nitric oxide and carbon monoxide, and it is expressed as [7,16]

$$r = k[\mathrm{NO}][\mathrm{CO}] \tag{5}$$

where [CO] and [NO] are the concentrations of CO and NO at time $t$, and $k$ is the second-order rate constant. Introducing $\varepsilon$ and $x$ (see Eqs. (1) and (2)), Eq. (6) results in

$$r = k[\mathrm{NO}]_{\mathrm{in}}(1-x)[\mathrm{NO}]_{\mathrm{in}}(\varepsilon-x). \tag{6}$$

It is assumed that coal gangue hollow balls and temperature uniformly distribute in the reactor space, the reaction only occurs on the surfaces of gangue hollow balls, and the conversion of NO reduction is expressed as

$$\mathrm{d}[\mathrm{NO}] = (-r)\mathrm{d}t. \tag{7}$$

Eq. (7) integrated by using Eq. (6) results in

$$\ln\frac{\varepsilon-x}{\varepsilon(1-x)} = k(\varepsilon-1)[\mathrm{NO}]_{\mathrm{in}}t. \tag{8}$$

The experiments were designed to measure the left side of Eq. (8) at various $t$ and $\varepsilon$, and the rate of NO reduction is calculated.

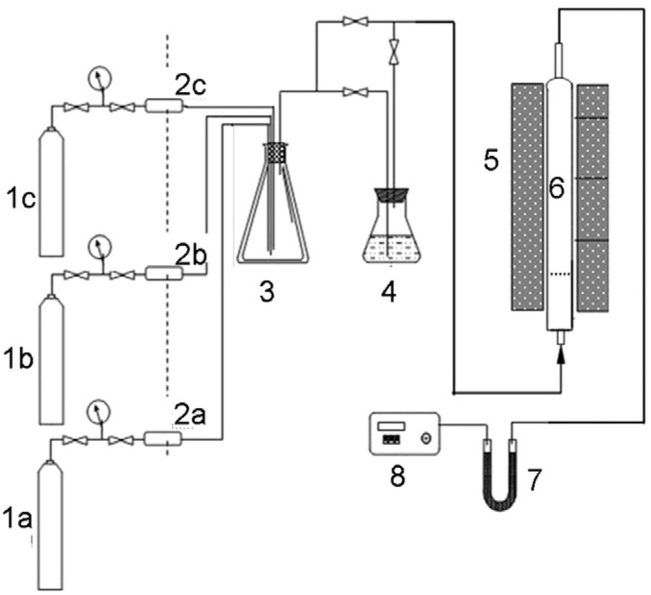

**Fig. 2.** The experimental setup of the test unit.

### *3.2. The reaction rate of NO reduction by CO over the surface of coal gangue hollow ball in the absence of water vapor*

In the experiment, the effect of CO on NO reduction without the addition of coal gangue hollow ball is first investigated at T < 923 K, and the results indicate that CO has no effect on NO reduction. CO molecule has a strong binding energy of 11.23 eV, NO is bound by 6.51 eV, N2 is bound by 9.76 eV [17] and thus NO reduction by CO is difficult to accomplish at T < 923 K without catalysis.

In the absence of water vapor, the experiment on the catalytic effect of coal gangue hollow ball on NO reduction was performed, and the relationship between $\ln((\varepsilon - x) / (1 - x))$ and $(\varepsilon - 1)[\mathrm{NO}]_{\mathrm{in}} \cdot t$ of Eq. (8) is shown in Fig. 4. The results indicate that the left side of Eq. (8) is approximately linearly proportional to the term $(\varepsilon - 1)[\mathrm{NO}]_{\mathrm{in}} \cdot t$ on the right-hand side, and this validates the assumption concerning the kinetics expressed by Eq. (5). The rate constant is evaluated from the slope of the straight lines in Fig. 4 by use of the least squares method, the linear correction coefficients are larger than 0.976, standard errors are less than $1.313 \times 10^{-4}$, and residual sum of squares is 0.322. The relationship between reaction rate constant and temperature is indicated in Fig. 5, and the activation energy and frequency factor obtained from Fig. 5 are shown in Table 3, where R is the gas constant ($8.314\ \mathrm{J{\cdot}K^{-1}{\cdot}mol^{-1}}$).

According to Eqs. (6) and (7), the influences of reaction temperature, residence time, the initial concentration ratio of $[\mathrm{CO}]_{\mathrm{in}}$ to $[\mathrm{NO}]_{\mathrm{in}}$ and the initial NO concentration on NO reduction ratio are modeled, and the simulation results are shown in Fig. 6. The results indicate that NO

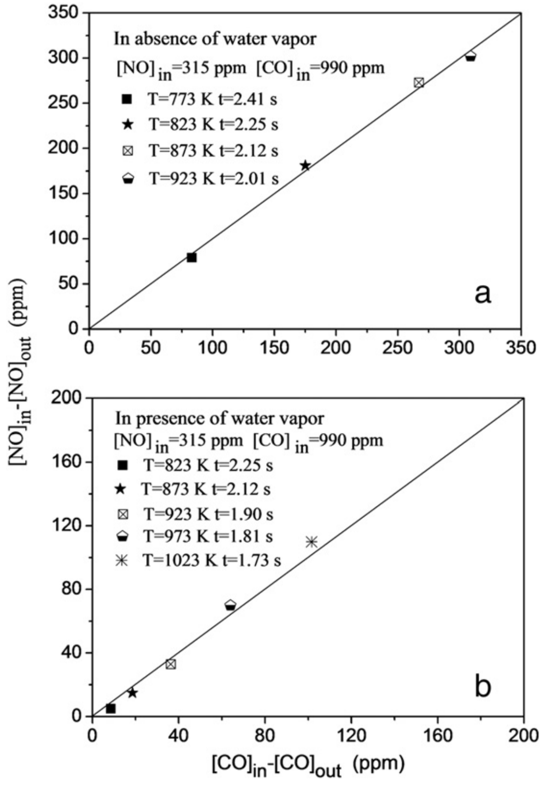

**Fig. 3.** The comparison of NO and CO concentration variations at reactor inlet and outlet.

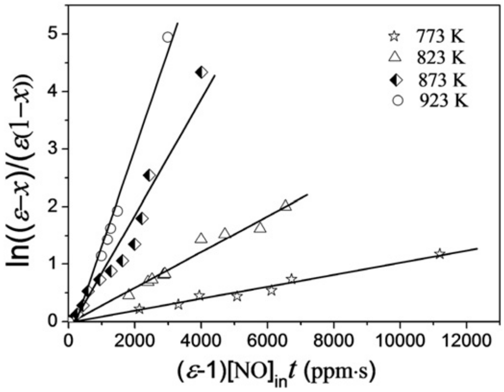

**Fig. 4.** The relationship between $\ln((\varepsilon - x) / (1 - x))$ and $(\varepsilon - 1)[NO]_{in} \cdot t$ in the absence of water vapor.

reduction ratio rapidly increases with reaction residence time at initial stage (<1 s) when reaction temperature is greater than 873 K, and increasing reaction temperature makes NO reduction ratio rapidly increase to about 0.9 when reaction residence time increase to 1 s at $T = 923$ K (see Fig. 6a); NO reduction ratio increases with the increase of the initial concentration ratio of $[CO]_{in}$ to $[NO]_{in}$, high NO reduction ratio can be achieved by increasing reaction temperature at $\varepsilon < 4$, and NO reduction ratio can reach above 0.8 at $\varepsilon = 4$ and $T \geq 873$ K (see Fig. 6b); with the increase of initial NO concentration, NO reduction ratio increases (see Fig. 6c). Compared to NO reduction by CO without catalysis, coal gangue hollow ball has a greater catalytic effect on NO reduction.

Above experimental results indicate that coal gangue hollow ball has noticeable catalytic effect on NO reduction by CO, the initial and final chemical compositions of coal gangue hollow ball were analyzed by XRF, the compositions were not changed in the experiment, and thus this indicates that coal gangue hollow ball only plays catalytic role for NO reduction. Known from Table 2, coal gangue hollow ball consists of $SiO_2$, $Al_2O_3$, $Fe_2O_3$, CaO, $TiO_2$ and $K_2O$, and these complex metal oxides play catalysis for NO reduction by CO in the experiment.

The mechanism of NO reduction by CO over metal oxide catalysts is very complex, and a number of studies have been reported in the literature. The mechanism for catalytic reduction of NO by CO is generally believed to proceed through NO adsorption and dissociation followed by nitrogen desorption and subsequent oxidation of adsorbed CO to $CO_2$. Gandhi et al. [18–21] investigated the chemisorption of NO on iron oxides, manganese oxides and cobalt oxides, it was found that NO was adsorbed more strongly than CO, and the adsorption of NO depended on the reduction state of the metal oxides.

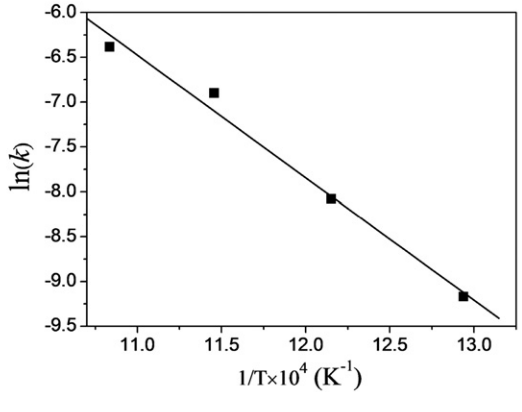

**Fig. 5.** Arrhenius plot based on second-order kinetics in the absence of water vapor.

**Table 3**
Reaction rate constant of NO reduction by CO over coal gangue hollow ball.

| Temperature range (K) | $k$ (ppm$^{-1}$·s$^{-1}$) |
|---|---|
| 773–923 (without water vapor) | $5210\exp(-113600/(RT))$ |
| 823–1073 (with water vapor) | $0.31\exp(-60750/(RT))$ |

A reaction mechanism is proposed for the catalytic reduction of NO by CO over CaO [13]:

$$NO + * \rightleftharpoons NO_{ads} \tag{R2}$$

$$NO_{ads} + CO \longrightarrow NO \cdot CO_{ads} \tag{R3}$$

$$NO \cdot CO_{ads} \rightleftharpoons CO_2 + N_{ads} \tag{R4}$$

$$2N_{ads} \longrightarrow N_2 + 2* \tag{R5}$$

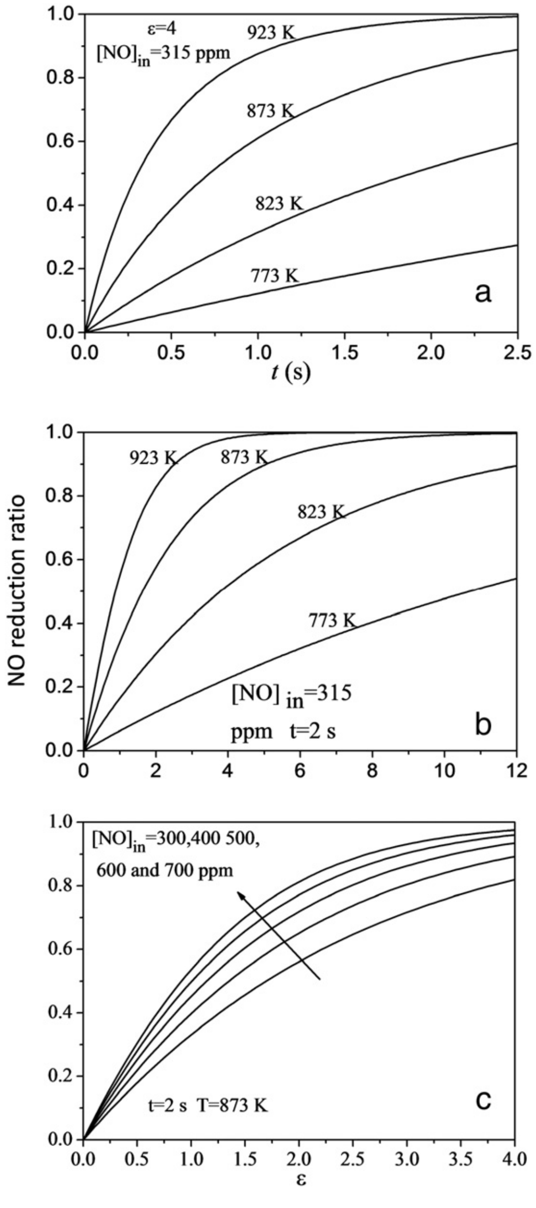

**Fig. 6.** Simulation results of the effect of reaction variables on NO reduction ratio in the absence of water vapor.

where * represents active sites on the CaO surface. It is generally accepted that nitric oxide is initially adsorbed on the catalytic surface. Dam-Johansen et al. [13,22,23] suggested that the dissociation of NO ($NO_{ads} + {}^{*} \leftrightarrow N_{ads} + O_{ads}$) was the next step in the nitric oxide reduction mechanism:

$$NO_{ads} + * \rightleftharpoons N_{ads} + O_{ads}. \tag{R6}$$

This step was then followed by $N_{ads}$ reacting with $N_{ads}$ to form $N_2(g)$ and * (see R5), or followed by $N_{ads}$ reacting with $NO_{ads}$:

$$N_{ads} + NO_{ads} \longrightarrow N_2 + 2O_{ads} \tag{R7}$$

to form $N_2$ and $O_{ads}$. Finally, $O_{ads}$ reacts with adsorbed carbon monoxide ($CO_{ads}$), forming $CO_2(g)$ and two free surface sites:

$$O_{ads} + CO_{ads} \longrightarrow CO_2 + 2*. \tag{R8}$$

This mechanism may satisfactorily describe the catalytic mechanism of metal oxides of coal gangue hollow ball in the experiment.

It was found that CaO was capable of catalyzing the NO reduction by CO and the effectiveness depended on its catalytic activity and surface [12], NO reduction by CO catalyzed by two Japanese limestones was studied, and the heterogeneous reaction is

$$2NO + 2CO \xrightleftharpoons{C_aO} N_2 + 2CO_2. \tag{R9}$$

Tokio Iizuka found that the $Fe_2O_3$ supported on $SiO_2$ was high active catalyst for NO reduction [11]. Hayhurst et al. [24–26] reported that, in a fluidized-bed combustor, NO can be catalytically reduced by CO over an $Fe_2O_3$ surface, and the heterogeneous reactions are

$$2CO + Fe_2O_3 \longrightarrow 3CO_2 + 2Fe \tag{R10}$$

$$2Fe + 3NO \longrightarrow 1.5N_2 + Fe_2O_3. \tag{R11}$$

Giving an overall conversion reaction is

$$CO + NO \longrightarrow CO_2 + 0.5N_2. \tag{R12}$$

$Al_2O_3$ is active for CO oxidation at temperature higher than 873 K. It is well known that $SiO_2$, $Al_2O_3$ and $TiO_2$ have interesting capacities as catalyst support for $NO_x$ reduction [9,10], and a series of unitary compounds including $TiO_2$, $Al_2O_3$, $SiO_2$ have been studied as the support of the low temperature SCR catalyst [27–31].

Known from the above catalytic mechanism analysis, these complex metal oxides in coal gangue hollow ball can effectively catalyze NO reduction by CO in the experiment.

### 3.3. The reaction rate of NO reduction by CO over the surface of coal gangue hollow ball in the presence of water vapor

In the presence of water vapor, the experiment on the catalytic effect of coal gangue hollow ball on NO reduction was performed at T = 823–1073 K. In the experiment, the precise and stable adjustment of $H_2O$ vapor is very difficult when the concentration is less than 0.5%. In the study, in order to assure the accuracy of the experimental result, the concentration of $H_2O$ vapor in gas mixture was kept constant (4.0%) in the experiment.

The relationship between $\ln((\varepsilon - x) / (1 - x))$ and $(\varepsilon - 1)[NO]_{in} \cdot t$ of Eq. (8) is shown in Fig. 7, the linear correction coefficients are larger than 0.970, standard errors are less than $1.468 \times 10^{-5}$, and residual sum of squares is 0.217. The results indicate that the left side of Eq. (8) is also approximately linearly proportional to the term $(\varepsilon - 1)[NO]_{in} \cdot t$ on the right-hand side, and the results also confirm with the assumption concerning the kinetics expressed by Eq. (5), which indicates that the occurrence of water vapor in NO reduction by CO does not change the scheme of reaction (R1) in the experiment.

The rate constant is evaluated from the slope of the straight lines in Fig. 7 by use of the least squares method. The relationship between reaction rate constant and temperature is indicated in Fig. 8, and the activation energy and frequency factor obtained from Fig. 8 are shown in Table 3.

The influences of reaction temperature, residence time, the initial concentration ratio of $[CO]_{in}$ to $[NO]_{in}$ and the initial NO concentration on NO reduction ratio are also modeled, and the simulation results are shown in Fig. 9. Compared to Fig. 6 in the absence of water vapor, the catalytic effect of coal gangue hollow ball on NO reduction is weakened by water vapor. NO reduction ratio increases slowly with the increase of reaction residence time, it is less than 0.2 when reaction residence time is 2.5 s at $\varepsilon = 4$ and $T \leq 923$ K, and the initial concentration ratio of $[CO]_{in}$ to $[NO]_{in}$ has negligible effect on NO reduction ratio at $T \leq 923$ K. In order to achieve high NO reduction ratio in the presence of water vapor, the initial concentration ratio of $[CO]_{in}$ to $[NO]_{in}$, the reaction residence time and temperature should be greater than those in the absence of water vapor.

Experimental results indicate that the presence of water vapor suppresses NO reduction by CO, and NO reduction ratio decreases to some degree compared to the reaction results without water vapor. Since water vapor is always present in exhaust gases, the stability of catalysts under these conditions is a very important factor. Kobylinski reported a poisoning of the supported oxides in the presence of water vapor [32]. This might be correlated with the fact that water vapor competes with reaction gas in adsorption on the surface of active catalytic sites [33]. $H_2O$ and NO compete for the active sites and allow for the possibility of a dissociative adsorption of water as $H^+$ and $OH^-$, the proton would get associated with the coordinatively unsaturated $O^{2-}$ site

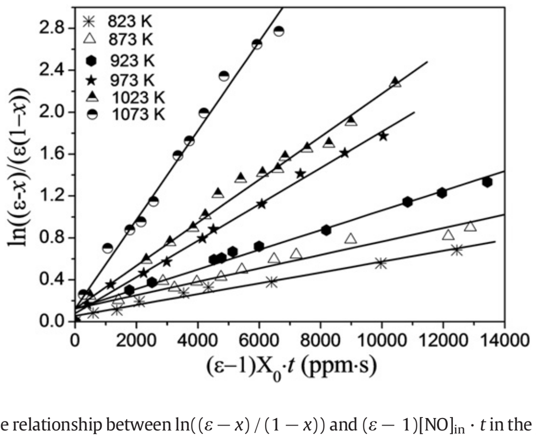

**Fig. 7.** The relationship between $\ln((\varepsilon - x) / (1 - x))$ and $(\varepsilon - 1)[NO]_{in} \cdot t$ in the presence of water vapor.

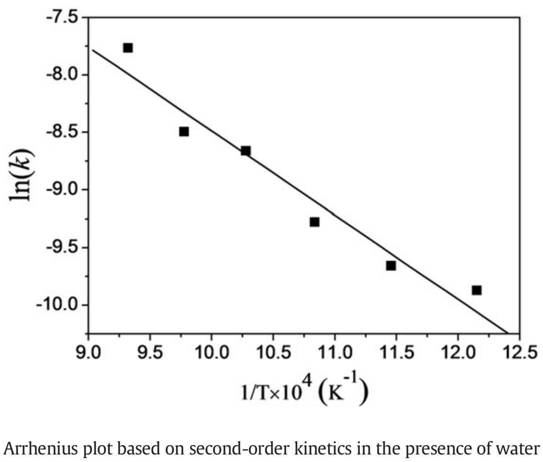

**Fig. 8.** Arrhenius plot based on second-order kinetics in the presence of water vapor.

and $OH^-$ with the cation site, and the activity could be restored only after regeneration at high temperatures.

### 3.4. Conclusion

Complex metal oxides ($SiO_2$, $Al_2O_3$, $Fe_2O_3$, CaO, $TiO_2$, $K_2O$, etc.) in coal gangue hollow ball can effectively catalyze NO reduction by CO in the experiment. In the absence of water vapor at 773–923 K, the reaction rate constant is

$$k = 5210\exp(-113600/(RT)).$$

NO reduction ratio rapidly increases with reaction residence time at initial stage (<1 s) and $T > 873$ K; high NO reduction ratio can be achieved by increasing reaction temperature at $\varepsilon < 4$.

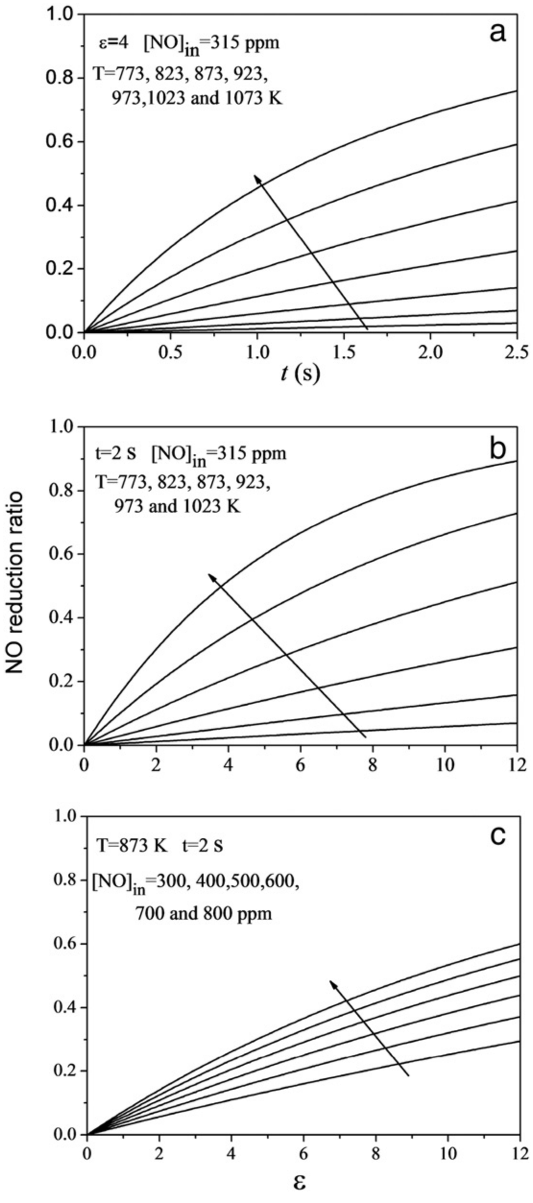

**Fig. 9.** Simulation results of the effect of reaction variables on NO reduction ratio in the presence of water vapor.

In the presence of water, water vapor suppresses NO reduction by CO, and the reaction rate constant is

$$k = 0.31\exp(-60750/(RT)).$$

In order to achieve high NO reduction ratio in the presence of water vapor, the initial concentration ratio of $[CO]_{in}$ to $[NO]_{in}$, reaction residence time and temperature should be greater than those in the absence of water vapor.

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