EXPERIMENTAL STUDY ON NO HETEROGENEOUS REDUCTION BY CHAR

by

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Original scientific paper
https://doi.org/10.2298/TSCI2205147Y

The NO heterogeneous reduction by char is one of the most prominent options to control NO_x emissions from coal-fired power plants. Experiments on the char-NO heterogeneous reaction were carried out in an electrically heated fixed-bed reactor to investigate the effect of temperature, type of char, pretreatment method, additives and reaction atmosphere on the NO reduction capacity of char. The results showed that temperature plays a crucial role in the NO reduction by different char. The kinetic analysis showed that char-NO heterogeneous reaction is controlled by chemical kinetic below T_t and by diffusion kinetic above T_t. The value of transition temperature T_t depends on the types of char and ranges from 600 °C to 800 °C. The synergistic effect of specific surface area, mineral abundance and reactivity combine to result in the reduction efficiencies of different char. Oxygen has a promoting effect on the char-NO heterogeneous reaction, and the oxygen content of the promoting peak moves to low oxygen content with increasing temperature. At 1050 °C, the denitrification efficiency at 0.25% O_2 content is 12.7% higher than that under oxygen-free conditions. At high temperatures, the promotion of the char-NO heterogeneous reaction by CO and the inhibition of the reaction by CH_4 were more obvious.

Key words: *coal char, heterogeneous reduction, NO reduction, synergistic effect, alkali metal*

Introduction

The NO_x emissions contribute to many environmental problems, including acid rain and the formation of fine particulate matter [1]. Many countries and regions have also enacted increasingly stringent pollutant emission standards. It is imperative to control the NO_x emissions at sites with large NO_x emissions, such as coal-fired power plants. Among several NO_x control technologies, CARBONO_x is considered an effective technology because of its high efficiency and low cost [2]. In the CARBONO_x process, NO is reduced to N_2 by homogeneous reduction with reducing gases and by heterogeneous reaction with char. Related study [3] has demonstrated that about 56-79% of NO reduction is due to the heterogeneous reduction of char during the reburning process of coal and biomass. Moreover, the alkali metals and alkaline earth metals in coal and biomass also have a specific catalytic effect on the heterogeneous

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reduction of NO_x. Therefore, the research of char-NO heterogeneous reaction mechanisms and factors is essential for the application of low NO_x emission technologies.

As an important by-product of the coal chemical industry, coal char is widely available and inexpensive. In addition, the large specific surface area and stable chemical properties of char make it an advantage in NO_x control. Therefore, the char-NO heterogeneous reduction has received more and more attention from researchers. Among numerous studies, quantum chemical calculations have been widely used as a helpful tool for studying molecular principles to investigate the reduction mechanisms of NO_x. Liu *et al.* [4] studied the mechanism of metal calcium catalyzed char-NO heterogeneous reduction by quantum chemical method. Zhang *et al.* [5] studied the microscopic mechanism of the effect of calcium and sodium on the char-NO heterogeneous reduction based on density functional theory. Zhang *et al.* [6] conducted the detailed kinetics of the char-NO heterogeneous reaction in the range of 973~1573 K by isothermal thermalgravimetric experiments. Zhu *et al.* [7] investigated the adsorption and reduction of NO_2 by N-doped activated carbon through density functional theory, and the simulation results were in good agreement with the experimental results.

Several experimental studies [8-10] have shown that coal char has a reduction effect on NO_x during coal combustion. Zhang *et al.* [11] illustrated the char-NO reaction in the range of 1073~1573 K by isothermal thermogravimetry using coal char obtained from an entrained-flow reactor, and found that chemical kinetics controlled the NO-char reaction at 1073~1273 K. Sun *et al.* [12] thermodynamically characterized the char-NO reaction in the range of 973~1573 K with isothermal thermogravimetry, and found a similar phenomenon: chemical kinetics controlled the char-NO reaction at low temperature, and diffusion kinetics controlled the reaction at high temperature.

Most existing studies focused on the reaction mechanism and reaction kinetics. However, many factors affect the efficiency of char-NO heterogeneous reduction in the CARBONO_x process, such as temperature, pore structure, NO concentration and reaction atmosphere. Furthermore, while some studies have addressed the effects of these factors, most have focused on the high temperature stage. Experimental studies on a wider range of temperatures, char types and reaction atmospheres are scarce, which is a gap in the research on the use of char as a CARBONO_x resource to reduce nitrogen oxide emissions.

To sum up, this paper investigated the char-NO reactions of different types of char, pretreated methods and additives, reducing and oxidizing atmospheres over a wide temperature range (450~1050 °C). The effects of these factors on the NO reduction capacity of char are explored to provide an experimental basis for the progress of industrial applications of coal combustion.

Experimental section

Properties of samples

The experimental samples are carboniferous coal char, fruit tree char, particleboard char, activated carbon, and washed and pickled carboniferous coal char. Carboniferous coal char is prepared from carboniferous coal in a quartz fixed bed reactor at a heating rate of 10 K per minute and a constant temperature of 1000 °C for 30 minutes in the Ar atmosphere. Fruit tree char and particle board char are by-products obtained by pyrolysis at 800 °C in a biomass gasification plant of Xi'an. Activated carbon is a specific brand product made from bamboo. Deionized water and concentrated hydrochloric acid are used for washing and pickling carboniferous coal char to deash the coal char. The deashing process is carried out by

heating in a water bath at 60 °C for 12 hours. After being washed with plasma water to neutrality, it is dried at 105 °C for 2 hours before use. The proximate analysis is shown in tab. 1.

Table 1. Proximate analysis of coal samples

Sample	Proximate analysis [wt.%]			
	M_{ad}	A_{ad}	V_{ad}	FC_{ad}
CC	2.90	44.55	3.19	49.35
FT	14.82	72.24	6.14	6.79
PB	60.80	4.00	5.75	29.45
AC	5.35	19.85	13.40	61.41
WCC	2.63	42.23	1.81	49.33
PCC	2.91	40.95	3.94	48.20

ad – air-dried basis, CC – coal char, FT – fruit tree char, PB – particleboard char, AC – activated carbon, WCC – washed carboniferous coal char, PCC – pickled carboniferous coal char

Experimental system

The schematic diagram of the denitrification reaction device is shown in fig. 1, which composes a gas supply system, a temperature control system, a fixed bed reactor and a flue gas analysis system. The gas supply system comprises five gas mixtures, namely CH_4, CO, O_2, NO, and Ar. The gas-flow controlled by the mass-flow meter enters the reactor after premixing in the mixer.

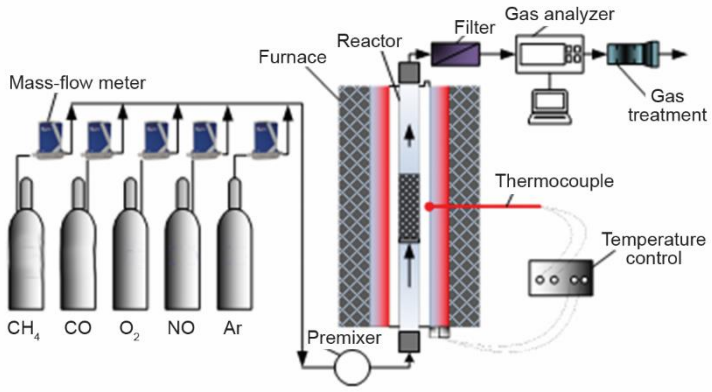

Figure 1. Schematic diagram of experimental device for reduction reaction

The heterogeneous reaction occurred in a vertical flow quartz reactor with an internal diameter of 60 mm. The length of the isothermal zone is about 100 mm. A quartz sieve plate is arranged in the middle of the isothermal zone to hold the char that participates in the heterogeneous reaction. The thermocouple is located in the middle of the furnace body, and the control accuracy is ±1 K. The air-flow participating in the reaction passes through the quartz sieve from bottom to top.

The experiment process is: Firstly, the sample placed on the quartz sieve was heated to the target temperature at a rate of 10 K per minute in an argon atmosphere. Finally, the mixed air-flow was introduced to participate in the heterogeneous reaction. After filtering, the

flue gas was analyzed by Testo 350 flue gas analyzer to determine the concentrations of NO and other residual gas.

Data processing method

In order to directly compare the differences in the ability of char samples to reduce NO under different reaction conditions. The NO heterogeneous reduction efficiency by char samples is calculated according to:

$$\eta = \frac{c_{NO}^{in} - c_{NO}^{out}}{c_{NO}^{in}} \tag{1}$$

where c_{NO}^{in} is the inlet concentration of NO and c_{NO}^{out} is the outlet concentration of NO.

Results and discussion

The effect of char type on the NO heterogeneous reduction efficiency

The comparison of the NO heterogeneous reduction efficiency of four different types of char is shown in fig. 2. It can be obtained from the figure that there are obvious differences in the reduction efficiency of different types of char samples. In general, the order of reduction efficiency is PB > AC > FT > CC. The reduction efficiency of different types of char samples varies significantly with temperature. At the low temperature section (T < 450 °C), the reduction efficiency of different char samples is not much obvious. The difference in reduction efficiency between char samples gradually increases as the temperature rises in the transition section (450 °C < T < 800 °C). At the high temperature section (T > 800 °C), the difference in reduction efficiency between char samples remains unchanged. Temperature plays an important role in the reaction rate of the char-NO heterogeneous reaction. There is a transition temperature T_t, and the reaction is under chemical kinetic control below T_t and diffusion control above T_t [12]. However, the transition temperature, T_t, is related to the types of char samples. Sun *et al.* [12] performed kinetic characterization of NO-char reaction by isothermal thermogravimetry, and the result showed the transition temperatures for raw char is around 800 °C, which is similar to the results obtained in this study.

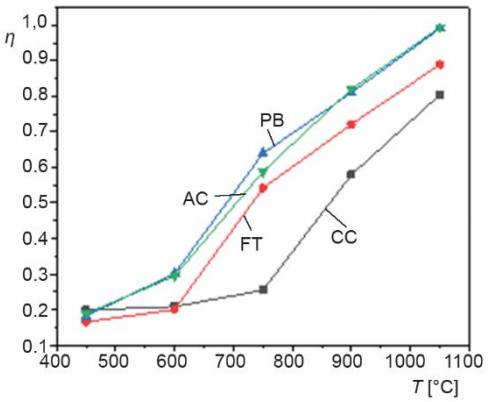

Figure 2. Comparison of reduction efficiency of different char samples

Figure 3 shows the Arrhenius plots of four chars at different given temperatures. The NO reduction conversion rate can define the specific reaction rate of a NO-char heterogeneous reaction. An excellent linear relationship can be obtained for all cases. Although the difference is not particularly obvious due to the small amount of data, there are two temperature regimes of activation energy (*i.e.*, the slopes of lines). The T_t for different chars is around 600-800 °C. In fact, the exact value of T_t is not our concern. Furthermore, the variations of activation energies indicate that the reactions were under chemical kinetic control in the low-temperature (below T_t) and under diffusion control in the high-temperature (above T_t).

The specific surface area of the char sample was tested with the automatic physical and chemical adsorption instrument, and the results are shown in fig. 4. The specific surface area of PB, AC, and FT is much larger than that of CC. This should explain why the denitrification efficiency of PB, AC, and FT is higher than that of CC at high temperatures. The specific surface area of PB is much larger than that of AC, FT, and CC, while the reduction efficiency of PB is not much higher than that of other types of char. That is, the specific surface area is not the main factor affecting the reduction efficiency.

Combined with the thermogravimetric analysis experiment of different char samples, the thermogravimetric analysis experiment was heated to 1100 °C at a heating rate of 10 K per minute under a 79% N_2, 21% O_2 atmosphere. Figure 5 shows the results of thermogravimetric analysis of four char samples. By comparing the weight loss curves and DTG curves of different char samples, the chemical reaction activities of different char samples are obtained. It can be seen from fig. 5, the chemical reaction activities of PB are slightly more significant than that of AC and FT. Therefore, the reduction efficiency of PB is higher than that of AC and PB, CC at high temperatures. The chemical reaction activity has a more pronounced influence on the char-NO heterogeneous reaction efficiency than the specific surface area.

In addition, it can be obtained by XRD image analysis of four char samples in fig. 6 that there are many minerals in fruit tree char and particle board char, and the mineral richness of AC, PB, and FT is higher than that of CC. The minerals in the coal char can promote the heterogeneous reduction of NO in the coal char to a certain extent [13]. The mineral has a strong catalytic effect on the char and NO reaction process. The catalytic effect of the mineral is mainly derived from alkali metals and alkaline earth metals.

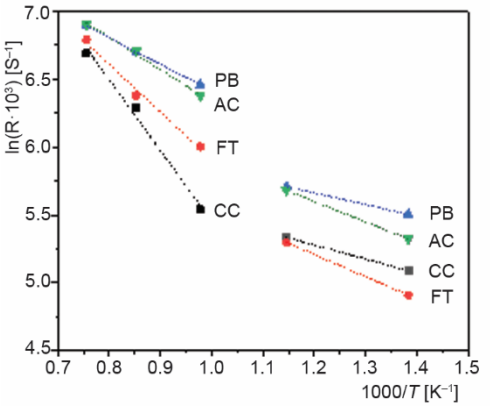

Figure 3. Arrhenius plots of NO-char heterogenous reaction

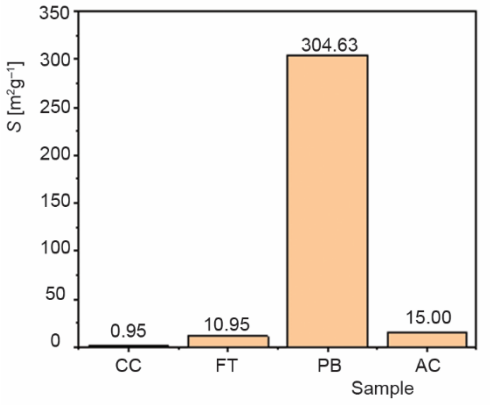

Figure 4. The specific surface area, *S*, of the samples

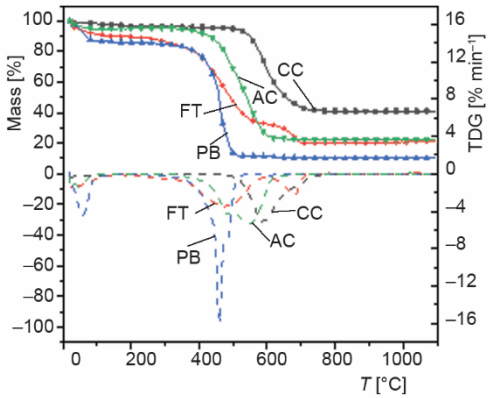

Figure 5. Results of thermogravimetric experiment with different char samples

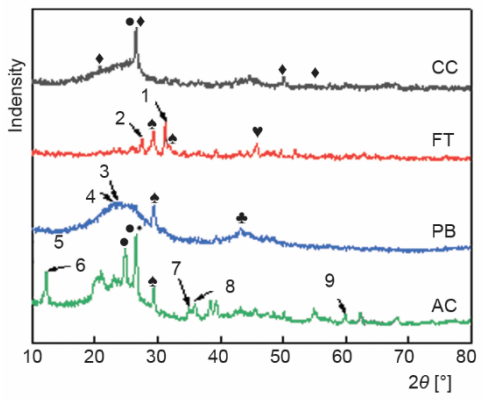

Figure 6. The XRD images of different focal samples; ● C, ♦ SiO_2, ♥ Al_2O_3, ♠ $CaCO_3$, and ♣ Fe_3C; *1 – $Ca_2MgSi^2O^7$, 2 – $NaAlSiO_4/NaCO_3$, 3 – $AlSi_{0.5}O_{2.5}$, 4 – $Al_{0.5}Si_{0.75}O_{2.25}$, 5 – $Al_2Si_2O_5(OH)_4$, 6 – $CaMnSi/K_{0.27}MnO_2$, 7 – $Fe_3+2Al_2(SiO_4)_3$, 8 – Cu_2MgO_3, 9 – $Fe_3+2Al_2(SiO_4)_3$*

The effect of pretreatment on the NO heterogeneous reduction efficiency

After pretreatment, the char-NO heterogeneous reduction shows differences at different temperatures. It can be seen from fig. 7 that there is no significant difference in the NO reduction efficiency between WCC, PCC, and the unpretreated CC in the middle and low-temperature section (t < 800 °C). As the temperature rises, compared with untreated CC, the heterogeneous reduction ability of pretreated CC on NO decreases significantly. When the reaction temperature is 900 °C, the NO reduction efficiency of PCC and WCC is 11.86% and 10.27% lower than that of untreated CC. When the reaction temperature was 1050 °C, the reduction efficiency decreased by 19.3% and 18.36%, respectively. In general, the ability of pretreated carbon char to reduce NO is lower than that of untreated carbon char, and the downward trend increases with the increase of reaction temperature.

Pickling can remove minerals in the sample and change the organic functional groups, thereby changing the structure. For Zhundong coal, water washing can remove 58.8% ash and 73.2% sodium, acid washing can remove 95% of ash and almost all alkali metals [14]. The removal of catalytic alkali metals reduces the ability of char to reduce NO.

Figure 8 shows the XRD spectra of PCC, WCC, and untreated carboniferous coal char. The 002 peak at 25°~27° is more apparent, comparable to natural graphite 26.6° 002

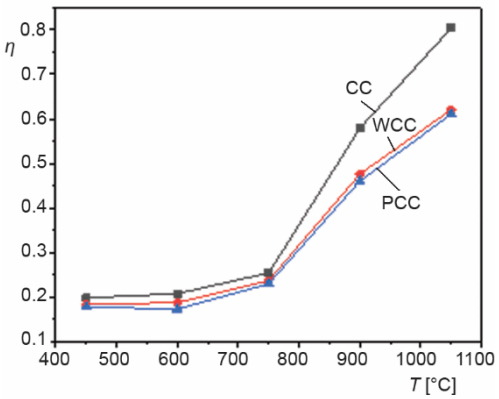

Figure 7. Comparison of reduction efficiency between pretreated and untreated char

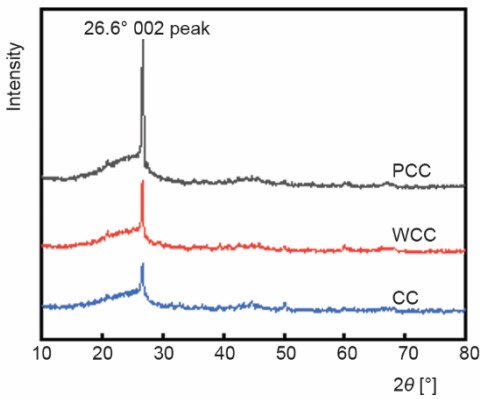

Figure 8. The XRD images of pre-processed and unprocessed carboniferous coal char

peak. The 002 peak represents the stacking height of the microcrystalline carbon mesh. That is the degree of orientation of the aromatic carbon network, which shows that the main crystal phase in the char is carbon. It should be noted that a diffraction peak of quartz was found near the 002 peak by phase detection. The quartz peak on the spectrum after pretreatment is enhanced. Compared with CC, the exposed ash was removed from the surface structure of PCC and WCC, the char layer was more compact, and the stacking thickness of the char layer and the number of carbon rings on the char layer were reduced.

The effect of additives on the NO heterogeneous reduction efficiency

The CaO and Fe_3O_4 were mixed as additives in CC to explore their influence on the NO heterogeneous reduction. As shown in fig. 9, the impact of additives on the heterogeneous reduction of NO did not appear to have prominent temperature-dependent zones. Under various working conditions, the blending of additives improves the efficiency of heterogeneous reduction of NO by carbon char by about 5%, and it tends to be evident with the increase of temperature. On the one hand, the use of additives can adsorb more NO gas molecules and play a specific catalytic effect on the NO reduction reaction. On the other hand, CaO is easily reduced to Ca by coal char at high temperatures, and the activation energy for NO reduction by Ca is much lower than for NO reduction by coal char. The reduced ability of Ca on NO is significantly higher than that of coal char [5]. The Fe_xO_y or Fe with reducing properties participates in the dissociation reaction of NO and is converted to Fe_xO_{y+1} or FeO, and then O is transferred to the coal char, and the Fe_xO_{y+1} or FeO is restored to the reduced state to realize the catalytic cycle [15]. Iron oxide as a catalyst can participate in the char-NO heterogeneous reduction and improve the ability to reduce NO.

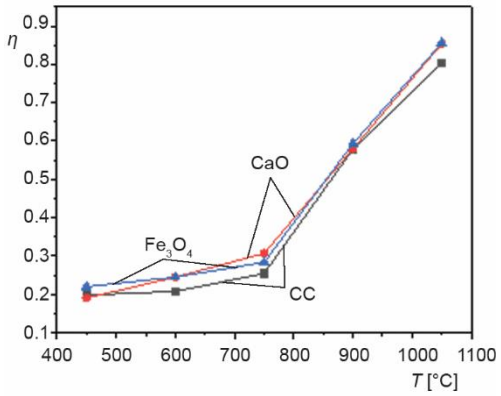

Figure 9. Comparison of denitrification efficiency of CC blended with additives

The effect of oxygen on the NO heterogeneous reduction efficiency

The participation of O_2 generally accompanies the actual process of reducing NO with CC. The presence of O_2 will cause the oxidation of coal char, which strongly impacts coal char's physical properties and reducibility. Therefore, it is imperative to study the effect of O_2 on the process of NO heterogeneous reduction.

The effect of oxygen on the reduction efficiency of CC is shown in fig. 10. The oxygen atmosphere can promote the reaction of CC to reduce NO and improve the efficiency of CC for the NO heterogeneous reduction. When the reaction temperature is 1050 °C, the promotion effect of oxygen to focus heterogeneous reduction of NO first increases and then decreases. At 1050 °C, the denitrification efficiency under 0.25% oxygen content is 12.7% higher than that under oxygen-free condition. There is a peak of oxygen char-NO reaction promotion effect, and the peak gradually shifts to a higher oxygen content as the reaction

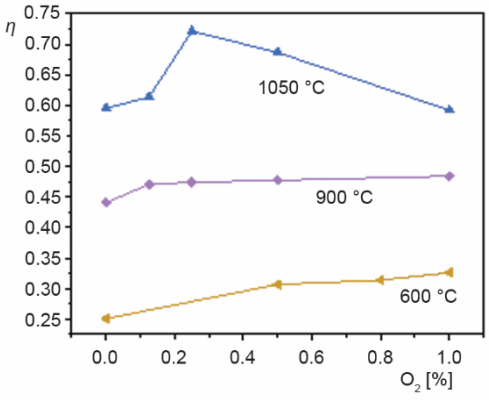

Figure 10. Effect of oxygen on the denitrification efficiency of CC

temperature decreases. The higher the reaction temperature, the lower the oxygen content required to achieve the maximum promotion of the oxygen char-NO reaction.

There is a critical O_2 concentration, the most considerable reduction of NO by coal char [16]. The O_2 concentration of less than 1% can promote NO reduction by coal char, but this promotion effect is reduced if it exceeds 1% [17]. The promoting effect of O_2 on the char-NO heterogeneous reaction is that abundant carbon and oxygen complexes, C(O), are formed on the surface of coal char under the O_2 atmosphere, and C(O) on the surface of coal char is further desorbed that participates in the reduction of NO. The oxidation of the coal char by oxygen increases the specific surface area of char as the reaction progresses, which is also the reason for the increase in the reduction rate of NO by the coal char. In addition, Gupta *et al.* [10] used the temperature-programmed method to demonstrate that the O_2 atmosphere can reduce the initial temperature of coal char to reduce NO. However, the reaction rate of char-O_2 is more than two orders of magnitude higher than that of char-NO. Therefore, as the oxygen concentration increases, the oxidation of O_2 quickly consumes the char. Thus, the effect of coal char on NO with the increase of oxygen concentration, the reduction efficiency shows a trend of first increasing and then decreasing.

The effect of reducing atmosphere on the NO heterogeneous reduction efficiency

As shown in fig. 11, CO has a noticeable promotion effect on the char-NO heterogeneous reaction process in the high temperature section, but the promotion effect in the middle and low temperature sections is relatively weak. Chen *et al.* [18] pointed out that CO can remove the C(O) complex on the surface of the char, which has an inhibitory effect on the reduction of NO. The C(O) complex reacts with CO and desorbs on the surface of coal char to form free carbon active sites. However, related [19] shows that the stability of C(O) complexes formed on the surface of coal char is different, and complexes with weak binding force can be reversibly desorbed in the form of CO or CO_2 at the formation temperature. However, complexes with strong binding forces require a higher temperature to complete desorption. The unevenness of the energy distribution of carbon atoms on the surface of coal char leads to the difference in the reactivity of C(O) on the surface of coal char [20]. The C(O) with low desorption activation energy and higher reactivity is an active intermediate for reducing NO, and C(O) with high desorption activation energy and low reactivity has an inhibitory effect on the focus-NO reaction. Therefore, when the reaction temperature is low, C(O) occupies the

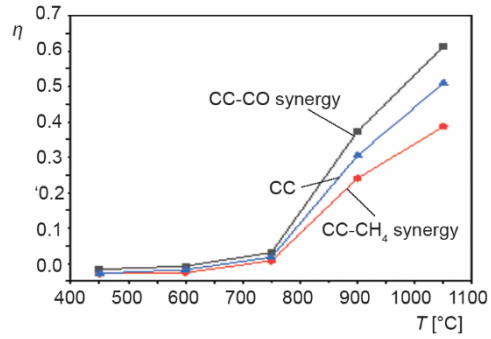

Figure 11. The influence of CO and CH_4 atmosphere on the denitrification efficiency of CC

active sites on the char surface, or CO can only desorb part of C(O), and it is difficult to desorb C(O), which has a strong binding force and inhibits the reaction. And as the temperature rises, the ability of CO to desorb C(O) increases, providing a large number of carbon active sites for the reduction of NO, and the reduction rate of NO increases.

In addition, the promotion of CO-NO reaction may also be through direct participation in the heterogeneous catalytic reduction reaction of NO. The metal oxides or carbon components in coal char minerals catalyze the CO-NO reaction. The CH_4 has a significant inhibitory effect on the char-NO heterogeneous reaction process in the high temperature section, while the inhibitory effect in the middle and low temperature sections is relatively weak. Under the catalysis of char, the cleavage reaction of CH_4 starts at a high temperature near 1000 °C, and as the temperature increases, the CH_4 cracking conversion rate increases [21]. Under the conditions of using carbon-based catalysts, the conversion rate of CH_4 at 800 °C can reach 50%. At higher temperatures, methane cracks: $CH_4 \rightarrow C+2H_2$, methane cracks into elemental carbon and hydrogen [22]. The elemental carbon is dissolved in the char and forms carbon filaments. The accumulation and collapse of carbon filaments affect the internal structure of the char. As the temperature rises, the char deposition caused by methane cracking becomes more apparent, limiting the adsorption and further reaction of NO by the char, resulting in a decrease in the denitrification efficiency.

Conclusions

In this paper, the effects of temperatures, different char types, pretreatment, additives, and O_2, CO, and CH_4 on the char-NO heterogeneous reduction were investigated. The main conclusions are as follows.

- The char-NO heterogeneous reduction efficiency is most affected by temperatures. There is a transition temperature T_t in the char-NO reaction process. The T_t may vary depending on the type of charcoal sample and ranges from 600-800 °C. The reaction is under chemical dynamic control below T_t, and diffusion control above T_t.
- The reduction efficiency order of different types of char is PB > AC > FT > CC. The efficiency of the char-NO heterogeneous reduction is affected by multiple factors. In addition to temperature, it is mainly related to the chemical reaction activity of the char sample. The micro-structure of the char sample, the richness of mineral species, and the content of alkali metals also have a specific impact.
- Metal oxides are used as additives to participate in the char-NO heterogeneous reaction, but the improved efficiency is not obvious. The blending of additives improves the efficiency by about 5%, and it tends to be evident with the increase of temperature.
- The promoting effect of O_2 on the char-NO heterogeneous reduction shows a trend of first increasing and then decreasing. As the temperature increases, the oxygen content, where the peak facilitation occurs, moves towards the lower oxygen content. At 1050 °C, the reduction efficiency under 0.25% oxygen content is 12.7% higher than that under oxygen-free condition.
- The CO is beneficial to remove the C(O) complex adsorbed on the active site of (C) and has a significant promotion effect on the char-NO heterogeneous reduction at high temperature. The heterogeneous reduction efficiency of CO on char-NO was increased by 10.38% at 1050 °C. In contrast, CH_4 has an inhibitory effect on the reaction at high temperatures, due to the coking effect of CH_4 cracking under the catalysis of coke, the internal structure of coke is affected. and the inhibition of char-NO reduction by CH_4 reduced the efficiency by 12.3% at 1050 °C.

Acknowledgement

The present work was supported by the National Natural Science Foundation of China (No. 22008192).

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Paper submitted: December 17, 2021
Paper revised: May 30, 2021
Paper accepted: June 5, 2021