

CATALYTIC REDUCTION BEHAVIORS OF NITROGEN OXIDES AND ITS USE IN BIOMASS COMBUSTION

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How to effectively apply NO_x control technology to the biomass combustion process has become a current research hotspot. Aiming at the characteristics that the nitrogen oxides produced in biomass combustion process are mainly from the nitrogen element of biomass fuel, this paper adopts the fuel classification - zone reduction method to control NO_x. Firstly, the reaction behavior, kinetic characteristics and optimal reduction parameters of NO reduction by coke were explored in a fixed-bed reactor. And then, the elemental analysis, pore analysis, scanning electron microscopy and other characterization methods combined with reduction experiments were used to study the changes of physical property parameters, reduction capacity and denitrification efficiency of coke after loading Fe based catalyst. By understanding the NO-carbon heterogeneous reduction process, the optimal conditions for enhancing reduction efficiency will be revealed, such as temperature, oxygen content in flue gas, and catalyst loading. The findings will provide a theoretical basis and technical support for implementing carbon reduction in low-nitrogen biomass combustion.

Key words: biomass boiler; NO_x; reduction efficiency; stratified combustion

1. Introduction

Biomass boiler owns many advantages such as high combustion efficiency, low pollution emissions, use of renewable resources, and combustion residues promoting soil quality and protecting the ecological environment. However, the widespread replacement of existing coal-fired equipment with biomass combustion systems has led to issues such as low combustion efficiency, severe slagging and ash accumulation [1, 2], and high levels of pollutants in exhaust gases, particularly excessive NO_x emissions leading to environmental pollution [3]. Reducing NO_x emissions while efficiently utilizing energy has thus become a topic of widespread attention.

At present, various methods are currently employed to control NO_x emissions. These methods include optimizing boiler operating parameters, fuel grading, air grading, and implementing other low-nitrogen combustion technologies to reduce NO_x emissions during the combustion process.

Additionally, physical and chemical methods are also used to remove NO_x from exhaust gases [4]. Among these methods, direct reduction denitrification technology using charcoal has promising developmental prospects due to its abundant raw material sources, low cost, nontoxicity, ease of preparation, and straightforward handling [5]. Significant differences have been observed between biomass and coal-fired boilers in terms of combustion and NO_x emissions, indicating that traditional denitrification technologies used for coal-fired boilers are not completely suitable for biomass boilers. The combustion temperature is lower than the critical temperature for the formation of thermal nitrogen oxides due to the low calorific value of biomass, and the nitrogen oxides in the flue gas are mainly fuel-type. Therefore, simply controlling the temperature through air grading combustion or flue gas recirculation cannot fundamentally achieve low-nitrogen combustion. Currently, studies on low-nitrogen combustion technology in biomass boilers mainly focus on reducing combustion temperature and generating fuel-type NO_x. However, most boilers face issues such as poor fuel adaptability and low thermal efficiency when applying these technologies. Therefore, the effective application of current NO_x control technologies to biomass boilers has become a prominent research topic and a key factor in promoting clean biomass heating technologies.

This study proposed a technical method for biomass layer combustion coupled with NO_x reduction, investigating the underlying principles and mechanisms. Using various characterization techniques, including elemental analysis, pore analysis, and scanning electron microscopy, along with reduction experiments, the study thoroughly examined the effect of physical parameters, reduction capability, and denitrification efficiency of char after incorporating Fe-based catalysts. By understanding the NO_x-carbon heterogeneous reduction process, the study identified the optimal conditions for enhancing reduction efficiency, such as temperature, oxygen content in flue gas, and catalyst loading. The findings provided a theoretical basis and technical support for implementing carbon reduction in low-nitrogen biomass combustion.

2. Materials and methods

2.1 Materials

This study used biomass pellets as fuel and coke as a reducing agent. The elemental and industrial analyses of these materials are presented in Table 1.

Table 1 Combustion characteristics of fuels used in the experiment

Sample	Elemental analysis (wt%)						Industrial analysis (wt%)				
	C _{ad}	H _{ad}	O _{ad}	N _{ad}	S _{t,ad}	M _{ad}	A _{ad}	V _{ad}	FC _{ad}	Q _{net,ad} (MJ/kg)	
Biomass	45.22	5.51	39.46	4.63	0.21	1.23	3.98	79.67	15.12	18.74	
Char	76.20	1.44	7.30	0.87	0.48	10.46	9.53	18.73	61.28	23.12	

According to the data in the table, the following differences between biomass fuel and coke materials were as follows: The carbon element and fixed carbon contents in biomass fuels were generally low. Typically, the carbon content in biomass fuels did not exceed 50%, similar to that of younger lignite. Compared with coke, the fixed carbon content of biomass fuels was significantly lower, resulting in a relatively lower calorific value and characteristics such as low burn resistance. Biomass fuels had high oxygen and volatile contents. They contained a large amount of hydrocarbons, which decomposed into volatile matter at certain temperatures. This characteristic made biomass fuels easy to ignite and burn completely.

2.2 Experimental apparatus

A NO_x-char heterogeneous reduction experimental system (shown in Fig. 1) was constructed to fully meet the requirements for NO_x-char heterogeneous reduction research. The system mainly consisted of a fixed-bed reactor, a temperature measurement and control system, a gas distribution system, and a flue gas analysis system.



Figure 1 NO_x-carbon heterogeneous reduction experimental system

(1) Fixed-bed reactor

The fixed-bed reactor consisted of a vertical tube electric heating furnace. The main body of the furnace comprised an insulation material, a refractory layer, silicon carbon rod heating elements, and a quartz furnace tube, with a power of 1.2 kW. The heating section of the furnace was 20 cm long, ensuring a constant-temperature zone of more than 10 cm so that the material layer in the middle of the quartz tube remained within this zone. The quartz tube reactor was 60 cm long with an inner diameter of 2 cm. The gas outlet was located at the top of the reactor, whereas the gas inlet was at the bottom.

(2) Temperature measurement and control system

The temperature measurement and control system of the fixed-bed reactor primarily consisted of a temperature controller. This controller, characterized by its high responsiveness, ensured that the furnace temperature changes with small amplitude and frequency. The PID control system of the temperature controller could achieve heating in up to 12 zones. Each zone underwent a heating phase and an insulation for a specific duration, then moving to the next zone. Before starting the experiment, the initial temperature, heating time, final temperature, and insulation time for each zone needed to be set. During the heating process, the temperature controller continuously monitored the actual furnace temperature and compared it to the set temperature. The maximum rated temperature of this system was 800°C, with a temperature control accuracy of $\pm 1^\circ\text{C}$.

(3) Air distribution system

The gas supply system primarily consisted of gas cylinders, gas mixing cylinders, mass flow meters, gas flow meters, and gas connecting pipelines. Its main function was to accurately regulate the gas flow required during the reaction process. All gas connecting pipelines were made of 316 stainless steel, linking the gas cylinders with the mass flow meter, gas mixing cylinder, and experimental platform.

The reaction gases included high-purity N₂, NO, and O₂, with their flow rates controlled by mass flow meters to ensure precise atmospheric control during the reaction. In the NO_x-carbon heterogeneous reduction experimental system, the gases were mixed at room temperature by the gas distribution system and then introduced into the quartz tube reactor. This introduction impacted the temperature field of the entire reactor contents. To reduce this impact, a heat tracing belt was installed outside the inlet section of the pipeline to preheat the mixed gases to 200°C before they entered the reactor.

(4) Smoke gas analysis system

The gases before and after the reaction were discharged from the top of the quartz tube reactor through a sufficiently long rubber hose, which was connected to a flue gas analyzer. The concentrations of the flue gas components before and after the reaction were monitored online using the TESTO 350 flue gas analyzer. This device could simultaneously monitor six components in the flue gas: O₂, CO, NO, CO₂, SO₂, and N₂O. However, the amounts of N₂O and SO₂ were extremely small, so they were not considered in the analysis.

2.3 Experimental procedures

This study employed characterization methods such as pore analysis and scanning electron microscopy, combined with reduction experiments, to conduct an in-depth exploration of the physical properties, reduction ability, and denitrification efficiency of coke after loading Fe-based catalysts. This will explore the nature of the catalytic action, providing a theoretical basis and technical support for biomass layer combustion coupled with NO_x reduction experiments. To ensure the reliability of test data, each NO_x reduction experiment was repeated three times, and the results were in good agreement. The data reported in this paper are average values of three times.

3. Results and Discussion

3.1 Characterization of coke-loaded Fe-based catalysts

The pore structure parameters of coke loaded with different concentrations of Fe-based catalysts, obtained using the low-temperature nitrogen adsorption method, are presented in Table 2. Loading Fe significantly altered the pore structure parameters of coke, resulting in a richer specific surface area, micropore area, and pore volume. These parameters were closely related to the chemical reactions involved in coke combustion and gasification processes, as well as the diffusion of gas phases within the coke pores. The coke loaded with Fe-based catalysts exhibited higher reactivity. When the Fe loading in coke ranged from 1% to 10%, the data showed that coke loaded with 1% Fe had a higher specific surface area, micropore area, and pore volume. However, these parameters were the lowest at a loading amount of 10%, suggesting that excessive loading may cause partial pore blockage in coke, thereby reducing the specific surface area and negatively impacting NO reduction.

Table 2 Pore structure parameters of coke with different Fe loadings

sample	BET Specific surface area (m ² /g)	Micropore area (m ² /g)	Pore volume (cm ³ /g)	Micropore volume (cm ³ /g)	Mean aperture (Å)
Coke original	77.4	46.7	0.062	0.023	35.67
load 1% Fe	308.9	268.2	0.176	0.124	32.76
load 2% Fe	301.4	259.1	0.173	0.121	32.98
load 5% Fe	293.7	248.9	0.169	0.117	33.42
load 10% Fe	285.0	241.1	0.167	0.112	33.76

The original coke sample and the coke samples loaded with 1% and 10% Fe-based catalysts were selected for observation under the scanning electron microscope. Two to three random areas were selected for each sample, and two typical photos from each were selected for analysis. The morphology of the material is clearly shown in Figures 2.

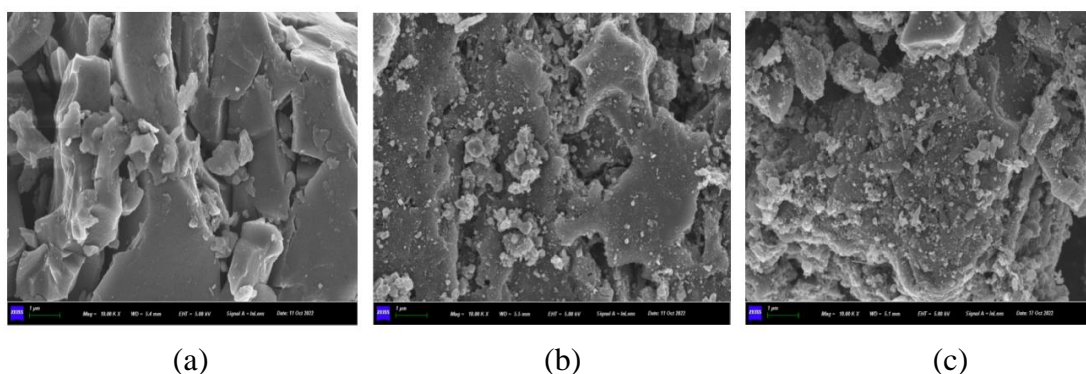


Figure 2 Surface morphology characteristics of coke (a), coke loaded with 1% (b) and with 10% Fe catalyst (c)

The surface of the coke exhibited a smooth texture with pores of various sizes distributed across it, although no obvious open pore structure was observed. This was due to the irregular and disordered arrangement of basic microcrystals in the coke, which resulted in the formation of gaps of different shapes and sizes on the surface, leading to the creation of pores with varying diameters. Volatile matter rapidly precipitated from within the coke particles during pyrolysis, producing gas that escaped the particle structure, ultimately leading to the formation of a porous surface structure on the coke. In addition, the images revealed the distribution of some brighter particles on the smooth surface of the coke. Literature research [6] indicated that these particles were ash particles.

As shown in Figure 2 (b) and (c), the surface morphology of coke underwent significant changes after loading with Fe-based catalysts. Clear residual traces were observed after loading, with a large number of tiny fragment attachments and abundant pore structures distributed across the surface, leading to an increase in specific surface area. Compared with Figure 2 (a), (b) and (c) reveal the formation of a honeycomb structure on the coke surface, which substantially enhanced the specific surface area of the carbon carrier. This structural modification improved the catalyst's absorption capacity for tar molecules, created more space for metal active sites, enhanced the interaction between tar molecules and active sites, and prolonged the reaction time [7]. These observations aligned with the results of the previous BET analysis, indicating a significant increase in the specific surface area of coke loaded with Fe-based catalysts.

Two weaker diffraction peaks were observed when the loading concentration was 1%, indicating

an increase in the specific surface area of coke after loading with 1% Fe, which enhanced dispersion to some extent (Fig. 3). The generated Fe_xO_y and $\text{Fe}(0)$ were well dispersed, resulting in weaker diffraction peaks representing the formation of aggregated crystal phases in the XRD spectrum. When the loading amount increased to 2%, three diffraction peaks representing Fe_2O_3 , $\text{Fe}(0)$, and Fe_3O_4 were detected, with smaller peak intensities. As the loading amount further increased, more pronounced diffraction peaks appeared in the XRD spectrum, including peaks representing $\text{Fe}(0)$ at 44.7° and 65.1° . The peak intensity was the highest at 44.7° , and a diffraction peak of Fe_3O_4 was also detected, which overlapped with the diffraction peak of $\text{Fe}(0)$ at 44.7° and was not quite distinct.

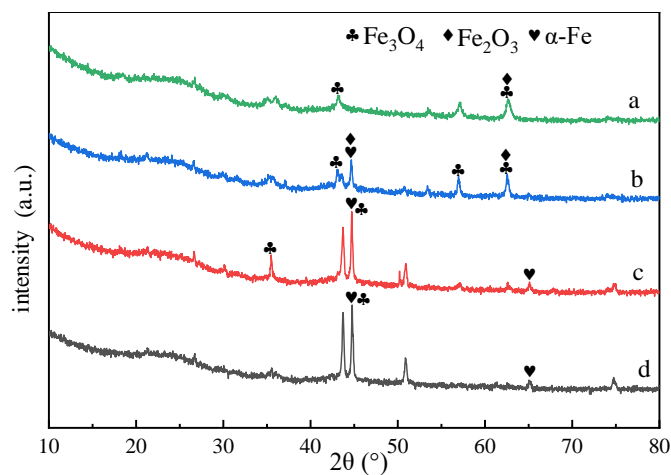


Figure 3 XRD spectra of coke, a) loaded 1% Fe, b) loaded 2% Fe, c) loaded 5% Fe, d) loaded 10% Fe

The aforementioned analysis revealed that when loading Fe-based catalysts onto coke, the main species detected in the sample were Fe_3O_4 , Fe_2O_3 , and $\text{Fe}(0)$. In addition, excessive loading could result in considerable crystal phase aggregation of the loaded iron species, potentially impacting the NO reduction.

3.2 NO_x -carbon reduction behavior

In the vertical fixed-bed reactor, the behavior characteristics of NO_x -char heterogeneous reduction reaction under 1% O_2 concentration were investigated using the programmed heating method. The experimental procedure was as follows: 1 g of coke particles were weighed and placed in a quartz tube reactor. The mass flow rates of N_2 , O_2 , and NO were adjusted to meet the concentration requirements. Once the NO outlet concentration measured by the flue gas analyzer reached the required level and stabilized, the temperature control cabinet was adjusted to heat up at a rate of $15^\circ\text{C}/\text{min}$. At 100°C , the gas concentrations at the reactor outlet were recorded and the experimental data subsequently recorded every 25°C increase in temperature.

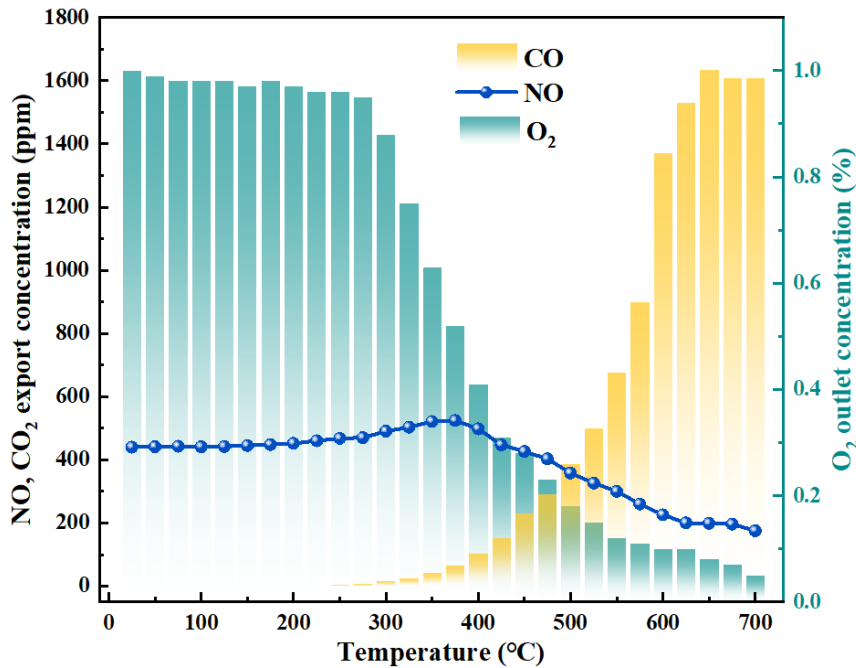


Figure 4 Variation of inlet and outlet gas concentration of NO_x-carbon reduction reaction with temperature

Figure 4 illustrates the variations in gas concentration during the NO_x-char reduction reaction at the inlet and outlet with temperature. The outlet gas primarily consisted of NO, introduced O₂, and generated CO. The diverse changes in gas composition at different temperatures indicated the variations in the NO_x-char reaction mechanism. Based on the concentration change patterns, the reaction process was divided into two main stages: the gas adsorption stage and the coke gasification reduction NO stage.

The first stage spanned from 0°C to 250°C. In this stage, the main reactions were physical and chemical adsorption owing to the low temperature[8]. O₂ and NO could be adsorbed onto the active sites on the surface of the coke, resulting in a lower concentration of NO at the outlet compared with the initial concentration (500 ppm). In this temperature range, the reactivity of coke with O₂ and NO was relatively low, and the active carbon sites (C^{*}) were occupied by initially adsorbed NO and O₂. Then, the NO entering the coke did not undergo any reaction before being discharged. In addition, the generation of CO was not detected in the gas outlet owing to the low reaction temperature, indicating the absence of gasification reactions in the coke at this temperature, which was consistent with the findings of Pevida et al. [9].

The second stage occurred from 250°C to 700°C. In this stage, the O₂ content decreased with the increase in reaction temperature, and CO began to appear in the outlet gas, with its concentration increasing with temperature. This was because of the formation of oxygen-containing complexes C(O) by NO on the coke surface via chemical adsorption. These complexes were thermally desorbed with the increase in reaction temperature, generating CO or CO₂. This process also created a new active C^{*} for the chemical adsorption of NO and O₂ on the coke surface. These observations aligned with the research findings of IllanGomez et al. [9], which suggested that high temperatures promoted the generation of CO.

The NO concentration initially increased and then decreased. It gradually peaked at 498 ppm between 250°C and 400°C and then gradually decreased to a low of 175 ppm between 400°C and 700°C.

This pattern could be explained by the increasing reaction rates (Sections 3.1 and 3.2) with the increase in temperature. Within the range of 250°C–400°C, the consumption rate of O₂ significantly exceeded the generation rate of CO, indicating that the reaction between O₂ and C was mainly chemical adsorption. As the temperature increased, the amount of O₂ adsorbed on the coke surface C* decreased. Therefore, the chemical adsorption of NO on the coke surface competed with the chemical adsorption of O₂ on the coke surface. Studies found that the reaction order of coke with O₂ was greater than that of coke with NO [10], indicating that the reaction of coke with O₂ dominated at this temperature, thereby inhibiting the reduction of NO and leading to an increase in NO concentration. When the temperature continued to increase (>400°C), the rate of CO generation accelerated, producing more active sites C* with high activity. At this point, the oxygen content in the outlet gas was already extremely low, allowing more NO to participate in the reaction. The formation and decomposition rates of C(N) and C(O) increased leading to a decrease in the concentration of NO. When the temperature reached 700°C, the NO outlet concentration dropped to 175 ppm.

3.3 Effect of O₂ on NO_x–carbon reduction

Experiments were conducted with five different O₂ concentrations for comparison to investigate the effect of adding O₂ to the reaction atmosphere on the reduction rate of NO. The fixed-bed reactor was used for programmed temperature rise experiments, following the same procedure as described in Section 3.2. The trend of NO reduction rate and reaction temperature at different O₂ concentrations is depicted in Figure 5. Without adding O₂, the conversion rate of NO gradually increased with the increase in temperature; however, with the addition of O₂, the reduction rate of NO initially decreased and then increased.

In the temperature range of 400°C–700°C, the average conversion rate of NO was significantly higher when 1% and 3% O₂ were added compared with the rate under anaerobic conditions, with the most pronounced promoting effect observed at 1% O₂. According to the reaction mechanism between coke and NO, coke underwent oxidation in the presence of O₂ in the reaction system, generating C(O) and C* continuously on the surface and increasing the active sites compared with the surface area. Therefore, NO was more easily adsorbed on the coke surface, thereby promoting the reduction of NO. Many scholars in China and abroad have also conducted research on this topic. Wang et al. [11] examined the reaction of two coke materials (graphite and coal coke) with NO in the temperature range of 30°C–900°C using a thermogravimetric analyzer and found that when a small amount of O₂ was present (at a concentration of 1.2%), the reaction temperature of coke and NO decreased, increasing the reaction rate. Rodriguez-Mirasol et al. [12] observed that the reaction between coal coke and NO significantly accelerated upon adding O₂, reducing the initial reaction temperature. They attributed this to the high content of CO produced during the combustion of coal coke, which changed the chemical properties of the coal coke surface and promoted the reduction of NO.

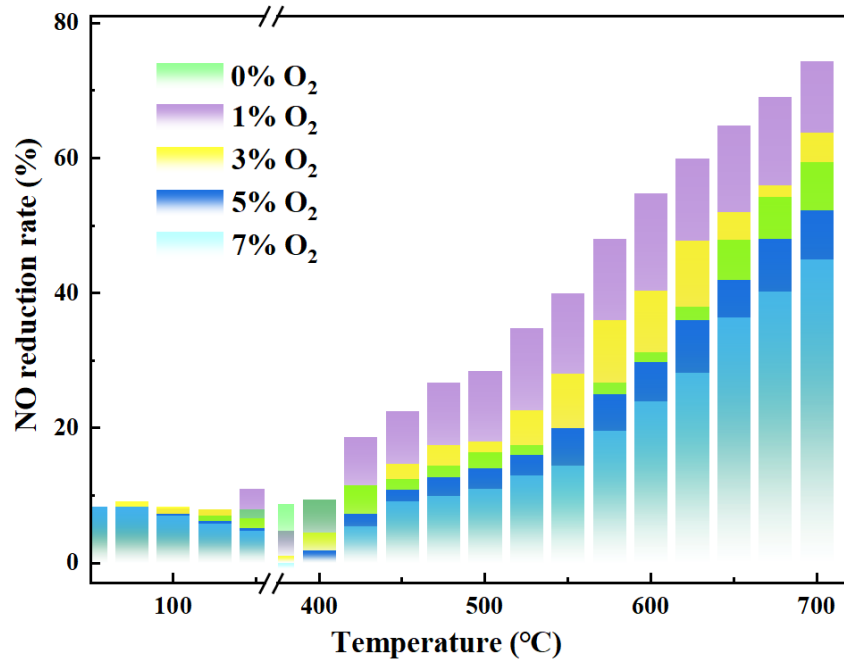


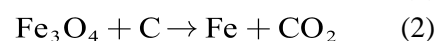
Figure 5 The change of NO reduction rate with reaction temperature under different O₂ concentration in the temperature programmed process

When the O₂ concentration was 5% and 7%, the overall reaction rate significantly decreased, even falling below the rate observed under anaerobic conditions. At a concentration of 7%, the denitrification efficiency at 700°C was only 45%, which was a 15% decrease compared with that under anaerobic conditions. This was because a high oxygen concentration interfered with the reaction between coke and NO, reducing the reactivity of both and thus lowering the NO reduction rate. Gupta suggested that a high O₂ concentration might lead to the occupancy of more active C*, resulting in a decrease in denitrification efficiency.

3.4 Effect of Fe-loaded catalyst on the NO_x-carbon heterogeneous reduction reaction

NO reduction experiments were conducted on coke loaded with different concentrations of Fe-based catalysts. Five different loading amounts of coke were subjected to programmed temperature rise experiments, following the same procedure as described in Section 3.2. The Fe-based catalyst showed almost no catalytic effect within the low-temperature range (<400°C), and the change in NO followed the same pattern as the unloaded coke. However, as the temperature increased, the reduction ability of the coke improved to varying degrees depending on the amount of Fe-based catalyst loaded.

The reaction mechanism of NO reduction by coke catalyzed by Fe loading was expressed as follows:



The smooth progress of NO reduction by coke loaded with Fe-based catalyst required that reactions (1) and (2) occurred simultaneously. Reaction (1) was relatively easy to carry out, so the rate at which coke reduced high-valent Fe to low-valent Fe became the dominant factor in the denitrification rate. XRD phase detection revealed that the main components on the surface of the coke were Fe₃O₄, Fe₂O₃, and Fe(0). The coke surface provided a reducing atmosphere, and hence Fe₃O₄ should be the primary oxide component in the reaction process.

The figure shows that the addition of an Fe-based catalyst increased the reduction activity of coke and NO, lowering the ignition temperature of the reaction. A comparison of the effects of different catalyst concentrations revealed that the NO reduction curves of 1% and 2% loadings were almost identical, indicating a similarity in their catalytic activities. The mechanistic analysis showed that the rate of NO reduction by coke mainly depended on the speed at which coke reduced high-valent Fe₃O₄ to low-valent Fe. Although 2% loading provided more low-valent Fe, the steady-state reaction rate of NO reduction by coke depended on the rate of reaction (2) producing low-valent Fe. The reduction curve showed a significant downward trend when the loading amount increased to 5% and 10%, with the largest decrease observed at 10%. Combined with the BET analysis results, it was clear that excessive loading blocked the pores on the coke surface, decreasing the specific surface area, reducing the contact area between Fe₃O₄ and coke, and thus lowering the NO conversion rate. In conclusion, 1% loading was sufficient to catalytically reduce NO using coke.

4. Conclusions

BET analysis found that the specific surface area and pore volume of the coke after loading the Fe catalyst increased significantly, and the specific surface area and pore volume reached the maximum when the loading was 1%. Overloading will lead to the occurrence of partial pore blockage of the coke, resulting in a decrease in the specific surface area. SEM analysis also further verified this conclusion. The supported Fe catalyst produced a richer pore structure on the surface of coke particles, which was conducive to the subsequent reduction reaction. XRD analysis found that the species loaded on coke were mainly Fe₂O₃, Fe₃O₄ and Fe(0). When the loading amount was too high, it was found that the loaded iron species had obvious crystal phase aggregation, which was not conducive to the reduction of NO. XPS analysis shows that there is more stable high-priced Fe₃O₄ on the surface of coke.

The introduction of O₂ has a significant impact on the reaction. It is found that adding 1% concentration of O₂ has the most obvious promotion effect on coke NO reduction reaction, and too high O₂ concentration will reduce the reactivity of coke and NO.

The denitrification efficiency of coke loaded with Fe catalyst is significantly improved, and the NO reduction efficiency is the highest when 1% Fe is loaded, Excessive loading will lead to a reduction in reduction efficiency, which is consistent with the previous physical property analysis results, that is, excessive loading concentration will lead to a decrease in the specific surface area of coke, and a decrease in the effective contact area between Fe₃O₄ and coke, which is not conducive to the reduction of NO.

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