



Article Kinetics Analysis of the NH₃-SCR Denitration Reaction over Sintered Ore Catalysts

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Abstract: Utilizing sintered ore catalysts (SOC), which are used in the sintering industry for NH₃-SCR denitration, is a feasible and economical way to reduce NO_x emission in sintering flue gas. Therefore, in order to enhance the denitration efficiency of SOC, sintered ore modified by sulfuric acid and sulfated sintered ore catalysts (SSOC-5) were prepared. Kinetic analyses of these two catalysts for denitration were carried out in this study. On the basis of eliminating the influence of internal and external diffusion, the relationship between reactants and reaction rate was studied by a power function kinetic model. This clarified that the adsorption ability of the acid-modified catalyst for reaction gas adsorption was stronger than that of sintered ore catalysts, and the reaction rate was also accelerated. The NO, NH₃ and O₂ reaction orders of SOC were 1, 0.3 and 0.16 at 250~300 °C, while these values of SSOC-5 were 0.8, 0.06 and 0.09, respectively. The apparent activation energy of SOC was 83.66 kJ/mol, while the value of SSOC-5 decreased to 59.93 kJ/mol.

Keywords: sintered ore catalysts; denitration; sulfate-acid modified; kinetics

1. Introduction

The level of NO_x emissions produced by the iron and steel industry in China is over 0.972 million tons every year, half of which are produced in the sintering process [1–3]. At present, the standard of NO_x emission from sintering plants is 300 mg/Nm³ in the general area and 100 mg/Nm³ in the key area, and the average content of NO_x in sintering flue gas is 300–400 mg/Nm³ [4]. However, it is difficult to control the emission of NO_x in the sintering process because of the complex conditions of sintering flue gas. Recently, NH₃-SCR (selective catalytic reduction) technology with the characteristic of a stable operation, high denitration efficiency and relatively high cost performance have been widely used for NO_x control [5–7]. SCR technology is widely used for denitrification in industrial flue gas [8,9]. V₂O₅-WO₃/TiO₂ catalysts with a denitrification efficiency of over 80% at 300~400 °C is a typical SCR catalyst [10–12]. However, the maintenance cost of commercial catalyst is high, and the catalysts are easily vulcanized and deactivated in the presence of SO₂. Moreover, vanadium is harmful to the environment [13,14], so it is necessary to develop new catalysts for denitration.

Iron, as a cheap and environmentally friendly material, is rich in resources and has an excellent thermal stability and sulfur resistance. The valence transformation between the two forms of iron, which are Fe^{2+} and Fe^{3+} , gives these forms a perfect redox ability for denitration. Therefore, iron-based catalysts are broadly applied in the process of NH₃-SCR denitration. Pasel et al. [15] found that Fe_2O_3/AC catalysts exhibited an excellent SCR-DeNO_x performance, the denitration efficiency was 100% at 140–340 °C, and the



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). N_2 selectivity was 100%. Sintered ore is the main product made in a sintering plant, and the main components of sintered ore are Fe₂O₃, CaO, SiO₂ and Al₂O₃. The content of FeO_x is over 50%, which means it can be used for denitration. Both Han et al. [16] and Chen et al. [17,18] discovered that sintered ore has a certain catalytic activity for NH₃-SCR denitration; the denitration efficiency was about 60% at 300 °C. However, with the increasingly stringent emission standards in sintering flue gas, it is difficult for the denitration ability of pure sintered ore to meet the new emission standards, and it is necessary to improve the catalytic activity of sintered ore catalysts. Our previous studies proved that denitration performance was greatly improved after modified sulfuric acid, and the conversion rate of SSOC-5 catalyst for NH₃-SCR denitration reached 92% at 300 °C [19]. This indicated that sintered ore has a certain potential in engineering applications for denitration.

For V₂O₅-WO₃/TiO₂ catalysts, most studies suggest that the process of SCR DeNO_x follows E-R mechanisms. The reaction order of NH₃ is 0, which means that NH₃ was strongly adsorbed on acid sites on the surface of the catalyst without the effect of the reaction rate [20]. Recently, many studies clarified that the reaction kinetics of the SCR process are complex, and the reaction orders of NO and NH₃ vary with the reaction conditions and the type of catalysts [21]. Peng et al. [22] suggested that NH₃ could rapidly activate at the acid sites on the surface of γ -Fe₂O₃ catalysts; its reaction order was 0, and NO was weakly adsorbed on the acid sites with a reaction order of 0.41. When the concentration of O₂ was 0~1%, the reaction order of O₂ was 0.27. However, it had no clear effect on the catalytic reaction when the concentration of O₂ was over 1%. Meanwhile, the apparent activation energy of the catalyst was 28.77 kJ/mol. Li et al. [23] studied the kinetics of Fe-Mo/ZSM-5 catalyst by a power function model. It was found that the reaction orders of NO, NH₃ and O₂ were 0.74~0.99, 0 and 0.01~0.13, respectively, during the process of SCR De-NO_x.

For SOC and SSOC-5, the denitration kinetics of these two catalysts are still unknown. In this study, in order to fully reveal the mechanism of NO_x reduction by SOC and SSOC-5, the denitration kinetics of these two catalysts are analyzed based on the results of previous study [19].

2. Experimental Section

2.1. Catalyst Preparation

The sintered ore used in this study was collected from Wuhan Iron and Steel Works. The process of catalyst preparation was introduced in detail in our previous study [19]. In previous research, the method of catalysts preparation and catalysts characterization, such as X-ray fluorescence spectroscopy, X-ray diffraction, BET surface area, X-ray photoelectron spectroscopy and in situ DRIFTS, are already included. The De-NOx ability of SOC and SSOC, as well as its mechanism, were all analyzed in previous research. Therefore, SOC and SSOC-5 were selected for investigation in this study.

2.2. Kinetic Measurements

The experiments were carried out on a fixed bed reactor. The prepared SOC and SSOC-5 catalysts were packed on a sieve plate in the middle of a quartz tube reactor with a diameter of 20 mm and length of 1000 mm. Referring to the components of sintering flue gas, the experimental conditions were as follows: 0~15% O₂, 0~300 ppm NO, 0~300 ppm NH₃, and N₂. The flue gas flow rate was controlled by mass flow controllers. The total flow rate was 1 L/min in each experiment. The gas was introduced into the quartz reactor after mixing in a mixing tank. The concentration of NO_x was measured by flue gas analyzer (PG-350, Horiba, Japan). The conversion of NO_x was calculated by Equation (1):

$$\eta = \frac{\text{NO}_{\text{x,in}} - \text{NO}_{\text{x,out}}}{\text{NO}_{\text{x,in}}} \times 100\%$$
(1)

where, η is the conversion efficiency of NO_x (%), NO_{x,in} is the concentration of NO_x at the inlet of the reactor (ppm), NO_{x,out} is the concentration of NO_x at the outlet of the reactor (ppm).

The NO reaction rate was calculated by Equation (2):

$$R_{\rm NO} = \frac{\eta \cdot C_{\rm NO} \cdot V}{22.4 \cdot W} \tag{2}$$

where, R_{NO} is the reaction rate of NO in the reaction (μ mol·g⁻¹min⁻¹), η is the conversion efficiency of NO_x (%), C_{NO} is the NO concentration (ppm), V is the flow rate of gas in the feedstock (L/min), W is the weight of the used catalyst (g).

In the study of kinetics, it is necessary to clarify the relationship between reaction rate and variables by means of a kinetic model. At present, there are two main models in the study of SCR reaction kinetics, the mechanism model and empirical model. Compared with other kinetic models, the empirical power-function reaction kinetic model has a simple form, and the parameters have a direct input to ASPEN Plus, Fluent, CFX and PRO II, etc. Therefore, the following power function kinetic models (Equation (3)) are used in this study:

$$R_{\rm NO} = -K[\rm NO]^{\alpha}[\rm NH_3]^{\beta}[\rm O_2]^{\gamma}$$
(3)

where, R_{NO} is the reaction rate of NO in the reaction (μ mol·g⁻¹min⁻¹), *K* is the apparent rate constant, α is the reaction order of NO, β is the reaction order of NH₃, γ is the reaction order of O₂.

The NH₃-SCR catalytic reaction only occurs after the diffusion and transfer process of reactants. It is necessary to exclude the influence of diffusion and transfer processes before the experiment, so the catalytic reaction rate only depends on the reaction temperature and concentration of reaction components. Hence, the accurate and reliable intrinsic reaction rate can be obtained. In the fixed bed reactor, the elimination of the external diffusion effect is generally maintained at constant gas hourly space velocity through the method of changing the flow rate and catalyst quality at the same time. When the conversion efficiency of NO_x no longer changes with the change in the flow rate, the external diffusion effect could be neglected.

For the elimination of the external diffusion influence on SOC catalysts for denitration, the experimental conditions were as follows: the ratio of the mass of the catalyst and flow rate (W/F) was maintained at 0.9 kg s L⁻¹, the reaction temperature was 250–300 °C, NO concentration was 300 ppm, NH₃/NO ratio was 1.0, and O₂ content was 15%. To eliminate the influence of external diffusion on the SSOC-5 catalyst for denitration, the experimental condition were as follows: the ratio of W/F was 0.36 kg s L⁻¹, the reaction temperature was 250–300 °C, NO concentration was 300 ppm, NH₃/NO ratio was 1.0, and the O₂ content was 15%.

The internal diffusion was eliminated by changing the particle size of the catalyst until the conversion of NO_x did not change with the particle size. Five groups of catalysts with different particle sizes (0.1, 0.16, 0.21, 0.3 and 0.6 mm) were selected to eliminate the effect of internal diffusion. The reaction temperature was 250~300 °C. The ratio of NH₃/NO was 1.0, O₂ content was 15% and the flow rate was 1 L/min.

3. Results and Discussion

3.1. Elimination the External and Internal Diffusion Effect

As shown in Figure 1a, the variation in NO_x conversion with a flow rate of three temperatures was approximately the same. When the flow rate increased from 0.6 L/min to 0.9 L/min, the conversion efficiency of NO_x significantly increased. Because the ratio of catalyst mass to gas flow rate was constant, the catalyst mass in the reactor also increased as the flow rate increased. With the increase in flow rate and catalyst mass, the contact area between gas and catalyst also increased, which reduced the diffusion process of reactants to the surface of catalyst. It was shown that at the condition of lower gas flow rate, the diffusion rate of reaction components from the main stream to the surface of catalyst was

less than catalytic reaction rate, and the external diffusion was the reaction control step. When the flow rate was reached to 0.9 L/min, the external diffusion rate was increased with the increase in flow rate. The external diffusion rate was faster than the catalytic reaction rate. Therefore, the external diffusion was no longer the control step of catalytic reaction, the NO_x conversion ability of catalyst seemed to be stable, and the external diffusion could be neglected.



Figure 1. Effect of flow rate on NO_x conversion over the SOC and SSOC-5 (**a**) SOC, $W/F = 0.9 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$; (**b**) SSOC-5, $W/F = 0.36 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$.

As shown in Figure 1b, the effect of external diffusion on SSOC-5 in denitration was approximately the same as that of SOC. When the flue gas flow rate was higher than 0.9 L/min, the NO_x conversion ability of SSOC-5 showed no clear change. Therefore, a flow rate of 1 L/min was selected to conduct the kinetic experiments.

Figure 2 was the effect of the catalyst particle size on NO_x conversion at different temperature. It was shown that, when the catalyst particle size rose from 0.6 mm to 0.3 mm, the NO_x conversion increased. This was because the reaction components from the catalyst surface were diffused to the inside of the catalyst faster than the reaction rate under the condition of larger catalyst particle size. Additionally, previous research proved that both Brønsted and Lewis acid sites, which are the main active sites for NO and NH₃ adsorption [19], took part in this process. When the catalyst particle size was between 0.6 mm and 0.3 mm, the number of active sites increased as the catalyst particle size decreased. Hence, internal diffusion can be considered as the control step of catalytic reaction. However, when the particle size was 0.1–0.3 mm, both the NO_x conversion of SOC and SSOC-5 catalysts did not change. This was because, when the particle size decreased, the time taken for the reaction components to diffuse from the catalyst surface to the inner channel also decreased, and the internal diffusion did not become the control step of the catalytic reaction. Therefore, a particle size of 0.2 mm was selected to conduct the kinetic experiments.

3.2. Effect of NO Concentration on the Rate of NO Conversion

Under the above conditions of eliminating internal and external diffusion, the reaction order of each reactant in the catalytic reaction was studied by changing the concentration of each reactant. As shown in Figure 3, the reaction rate linearly increased with the concentration of NO at various temperatures. The increase in NO concentration meant that the amount of NO reacted in the catalytic reaction also increased, and the reaction rate of NO simultaneously increased. The NO reaction order of SOC for denitration was 1.12, 0.84 and 0.92 at 250 °C, 275 °C and 300 °C. Additionally, the NO reaction order of SSOC-5 for denitration was 0.77, 0.83 and 0.83 at 250 °C, 275 °C and 300 °C. It was clear that the reaction order of NO was decreased after sulfuric acid modification, which indicated that sulfuric

acid modification strengthened the ability of NO adsorption and activation. It enabled NO to more rapidly participate in the catalytic reaction. The in situ diffuse reflectance infrared spectroscopy (DRIFTS) results from our previous study [19] prove that the adsorption intensity of nitrate species and nitro compounds on the SOC surface is weaker than that of SSOC-5. It was found in previous research that the bands at 1602 and 1618 cm⁻¹ were ascribed to bridged nitrate species; bands at 1490 and 1498 cm⁻¹ were ascribed to bidentate nitrates; bands at 1413 and 1419 cm⁻¹ were ascribed to monodentate nitrates; and bands at 1290, 1295, 1020 and 1011 cm⁻¹ were ascribed to nitro compounds [19]. As for nitro compounds at 1290 cm⁻¹ and the nitrate species at 1490 cm⁻¹, the adsorption intensity (the height of infrared spectroscopy absorption peak) of SSOC-5 was much higher than that of SOC, indicating a stronger NO adsorption ability of SSOC-5, which was consistent with the NO reaction order results of these two catalysts.



Figure 2. Effect of particle size on NO_x conversion over the SOC and SSOC-5 (a) SOC, $W/F = 0.9 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$, (b) SSOC-5, $W/F = 0.36 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$.

3.3. Effect of NH₃ Concentration on the Rate of NO Conversion

Figure 4 showed the effects of NH₃ concentration on NO reaction rate. As shown in Figure 4a, the variety of NH₃ concentration had a certain effect on the NO reaction rate. With the increase in NH₃ concentration, more NH₃ was supplied to the SCR DeNOx reaction, so the NO reaction rate gradually increased at all three temperatures. The NH₃ reaction order of SOC for denitration was 0.36, 0.25 and 0.27 at 250 °C, 275 °C and 300 °C, respectively. In Figure 4b, the effect of NH₃ concentration on the NO reaction rate over SSOC-5 was reduced compared with SOC, which indicated that NH₃ could react faster on the surface of the catalyst. The NH₃ reaction order of SSOC-5 was 0.09, 0.06 and 0.04, respectively, at 250 °C, 275 °C and 300 °C, which could be considered as 0.06. It could be concluded that the catalytic reaction was promoted by sulfuric acid modification. The decrease in the reaction order of SSOC-5 for denitration indicated that NH₃ could participate in the reaction more quickly, which further proved that sulfuric acid modification improved the catalytic performance of SOC. The FTIR spectra results of SOC and SSOC-5 demonstrated that more Brønsted acid sites were generated on the surface of SSOC-5 [19], which further promoted NH₃ adsorption on the catalyst surface.

3.4. Effect of O₂ Concentration on the Rate of NO Conversion

The effect of O_2 concentration on NO reaction rate is shown in Figure 5. When the volume concentration of O_2 increased from 0% to 3%, the reaction rate of NO rapidly increased because of the introduction of O_2 to the process of the SCR reaction. When the concentration of O_2 was more than 3%, the concentration of O_2 was much higher than that of NO and NH₃, so the reaction rate of NO conversion slowly increased. However, the NO

reaction rate of SSOC-5 catalyst was higher than that of SOC. Similarly, it was found that at 250 °C, 275 °C and 300 °C, the reaction order of SOC catalyst was 0.13, 0.18 and 0.16, respectively, while that of the SSOC-5 catalyst was 0.08, 0.11 and 0.07, approximating 0.09.



Figure 3. Effect of NO concentration on the rate of NO conversion (a) SOC, $W/F = 0.9 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$, (b) SSOC-5, $W/F = 0.36 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$.



Figure 4. Effect of NH₃ concentration on the rate of NO conversion (**a**) SOC, $W/F = 0.9 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$, (**b**) SSOC-5, $W/F = 0.36 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$.



Figure 5. Effect of O₂ concentration on rate of NO conversion: (a) SOC, $W/F = 0.9 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$, (b) SSOC-5, $W/F = 0.36 \text{ kg} \cdot \text{s} \cdot \text{L}^{-1}$.

3.5. Reaction Rate Constant and Apparent Activation Energy

As mentioned in the previous study [19], the mechanism of selective catalytic denitration process over SOC and SSOC was as follows:

$$NO(g)+O(a) \rightarrow NO_2(a)$$
-Bridge nitrites (4)

$$NO(g)+O(a) \rightarrow NO_2(a)$$
-Monodentated nitrites (5)

$$NO_2(g) + O(a) \rightarrow NO_3(a)$$
-Bidentate nitrites (6)

$$NH_2(a) + NO(g) \rightarrow NH_2NO \rightarrow 2N_2 + 2H_2O$$
(7)

$$2NH_4^+(a) + NO(g) \rightarrow \{NH_3HNO \rightarrow NH_2NO + H_2O\} \rightarrow N_2 + H_2O$$
(8)

$$NO_2(a) + NH_3(a) \rightarrow NO_2[NH_3]_2(a) + NO \rightarrow 2N_2 + 3H_2O$$
(9)

$$NO_2(a) + 2NH_4^+(a) \to NO_2[NH_4^+]_2(a) + NO \to N_2 + 3H_2O$$
 (10)

For the kinetics analysis, the following formulas could be obtained by substituting the reaction orders of the two catalysts obtained from the above experiments into Formula (3):

$$R_{\rm NO}(\rm SOC) = -K[\rm NO][\rm NH_3]^{0.3}[\rm O_2]^{0.16}$$
(11)

$$R_{\rm NO}(\rm SSOC) = -K[\rm NO]^{0.8}[\rm NH_3]^{0.06}[\rm O_2]^{0.09}$$
(12)

In a study of the reaction constants of SOC catalysts for denitration, Formula (11) can be simplified as follows:

$$R_{\rm NO} = -K^*[\rm NO] \tag{13}$$

The relationship between the apparent reaction rate constant (K^*) and NO_x conversion (x) could be obtained using the integral of Equation (6):

$$K^* = -\frac{\operatorname{Fln}(1-x)}{W} \tag{14}$$

where, *x* is NO_x conversion (%); F is the flow rate of gas (mL/min); and W is the weight of used catalysts (g).

On the premise of a flow rate of 1 L/min, the trends of NO_x conversion under different space velocity conditions were obtained by changing the used catalyst weights, which are shown in Figure 6. After data fitting, the apparent reaction rate constants (K^*) of SOC catalysts at 250 °C, 275 °C and 300 °C were obtained by changing the used catalyst weight; the above eC-5 were 29.83, 31.71 and 33.96 at 250 °C, 275 °C and 300 °C, respectively.



Figure 6. Trend chart of $-\ln(1 - x)$ with W/F at different temperatures: (a) SOC, (b) SSOC-5.

The apparent activation energy of the SCR reaction could be calculated from the Arrhenius equation as follows:

$$K = Ae^{-\frac{La}{RT}}$$
(15)

where A is the pre-factor; *Ea* is the apparent activation energy (kJ/mol); *R* is the ideal gas constant (8.314 J/(mol·K)); and T is the reaction temperature ($^{\circ}$ C).

Through the logarithm of both sides of Equation (8), the following formula obtains:

$$\ln K = \ln A - \frac{E_a}{RT} \tag{16}$$

The relationship of ln*K* and 10^3 /T is shown in Figure 7. By fitting and calculating the data, as shown in Figure 7a,b, the apparent activation energy of the SOC catalyst for denitration is 83.66 kJ/mol, and the pre-exponential factor A is 143.51. The apparent activation of the SSOC-5 for denitration is 59.93 kJ/mol, and the pre-exponential factor A is 114.54. The results show that the apparent activation energy of the catalyst for denitration was reduced after sulfuric acid modification, which promoted the catalytic reaction and improved the denitration efficiency. The results were consistent with the previous study since both the amount of Brønsted acid sites and Lewis acid sites increased after SOC was treated by sulfuric acid [19]. Both L-H and E-R mechanisms were present during the process of denitration reaction with SOC and SSOC [19], and the amount of Brønsted acid sites and Lewis acid sites significantly affected the process of NH₃ and NO adsorption on the catalyst surface. Therefore, the sulfuric acid treatment of SOC significantly reduced the apparent activation of these catalysts for denitration.



Figure 7. The relationship of lnK and $10^3/T$ (**a**) SOC, (**b**) SSOC-5.

4. Conclusions

According to the kinetic experiment results, the NO reaction order of the SOC was 1, the NH₃ reaction order was 0.3, and the O₂ reaction order was 0.16 at 250~300 °C. The SSOC-5 had a NO reaction order of 0.8, an NH₃ reaction order of 0.06, and an O₂ reaction order of 0.09. The reaction order of each reactant reduced after modification, and the reaction rate accelerated, indicating that sulfuric acid promoted the denitration performance of the sinter ore and reduced the activation energy of the catalytic reaction. The apparent activation energy of the SOC was 83.66 kJ/mol, and the apparent activation energy of the sulfuric acid-modified SSOC-5 reduced to 59.93 kJ/mol, make the catalytic reaction easier. It could be seen that sulfuric acid modification greatly enhanced the catalytic ability of the sintered ore in the SCR and improved denitration efficiency.

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