Experimental demonstration of NO\textsubscript{x} reduction and ammonia slip for diesel engine SCR system

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Abstract
This paper investigates the characteristics of selective catalytic reduction (SCR) with a V\textsubscript{2}O\textsubscript{5}-WO\textsubscript{3}/TiO\textsubscript{2} catalyst by studying the key parameters and determining a method for controlling ammonia injection with a sample test bench. Four parameters are defined and adopted to represent the characteristics of nitrogen oxides (NO\textsubscript{x}) and the ammonia reaction. The effects of NH\textsubscript{3}/NO\textsubscript{x} ratio (NSR), catalyst temperature, and ammonia injection period on NO\textsubscript{x} conversion efficiency and ammonia slip are investigated. The correlation between ammonia slip and ammonia saturation storage level is studied. The experimental results show that the ammonia saturation storage level has a great impact on NO\textsubscript{x} reduction and ammonia slip. The NO\textsubscript{x} conversion efficiency and ammonia slip strongly depend on the ammonia saturation storage level. Under such conditions, the NO\textsubscript{x} conversion efficiency is best when the ammonia saturation storage level is 68.2–73%, and the value reaches 75% before ammonia slip. Pulse injection improves the NO\textsubscript{x} conversion efficiency and limits ammonia slip. Comprehensive comparison shows that the injected ratio of NH\textsubscript{3}/NO\textsubscript{x} is first larger and then smaller than is beneficial for the rapid improvement of NO\textsubscript{x} conversion efficiency; the appropriate NH\textsubscript{3}/NO\textsubscript{x} ratio and continuous injection time must be controlled, or it is easy to cause ammonia slip. Therefore, a control algorithm based on ammonia storage saturation level has been proposed. According to the difference between the actual value of ammonia storage saturation and the target value, the controller corrects the injection of urea to achieve control of ammonia storage saturation level. The period of pulse injection has little influence on the mean value of NO\textsubscript{x} at the outlet; however, it affects the peak level of NO\textsubscript{x} and ammonia slip. Using varied period pulse injection further improves the NO\textsubscript{x} conversion efficiency and restrains ammonia slip. The outlet level of NO\textsubscript{x} can be reduced by adopting a suitable ammonia pulse injection interval.

Keywords Diesel engine · Selective catalytic reduction · Ammonia · Pulsing injection · Ammonia storage and slip

Introduction

Diesel engines are widely used as sources of power owing to their high thermodynamic efficiency, especially when compared with traditional gasoline engines. However, emissions from diesel engines are high, especially for nitrogen oxides (NO\textsubscript{x}) and particulate matter (PM) (Zhao et al. 2011; Xinmei et al. 2015). NO\textsubscript{x} emissions due to transport have been increasing dramatically. During 2000–2010, NO\textsubscript{x} emissions coming from power plants and transport increased over 100% and 200%, respectively, in China (Wang and Hao 2012). To meet stringent regulations calling for lower NO\textsubscript{x} emissions, urea selective catalyst reduction (urea-SCR) technology, a promising approach, has been adopted for diesel engines to reduce nitrogen oxide emissions (Pio et al. 2012; Koebel et al. 2000). As a primary emission control technology in heavy-duty (HD) diesel engines, urea-SCR technology has the advantages of providing good fuel economy and a high level of NO\textsubscript{x} reduction efficiency with a mature production technology (Bin et al. 2014). The urea-SCR system includes a control unit, a urea pump, a dosing module, an SCR catalyst, and other accessories.

Many studies have been focused on the mechanisms for reactions of various active catalyst components (Johnson...
characterization of ammonia adsorption and desorption (Byoung and Man 2013; Colombo et al. 2010; Kleemann et al. 2000; Tan and Lin 2015), and the development of a control strategy for improving NOx conversion efficiency (Zhang et al. 2014; Ofoli 2011). Ciardelli et al. (2004) studied the kinetics of ammonia adsorption-desorption in a 50–450°C temperature range over a commercial powdered V2O5-WO3/TiO2 catalyst loaded in a micro reactor. When ammonia was added, the ammonia outlet concentration showed a dead time, which was due to adsorption onto the catalyst surface. When ammonia was shut off, the ammonia outlet concentration could not be reduced to zero only by performing a temperature programmed desorption (TPD) run. Wallin et al. (2003) investigated the effect of ammonia supply on NOx reduction over zeolite H-ZSM-5 by using step response experiments between 200 and 500°C. They reported that as the inlet NO:NO2 molar ratios exceeded 1, the NOx reduction efficiency transients increased when ammonia was removed from the feed; compared to a process with continuous supply of ammonia, the NOx reduction was enhanced five-fold by pulsing ammonia into the feed. Wu et al. (2019) studied an injection control strategy involving a transient process for a heavy-duty diesel engine. Compared to feed-forward control, the proposed real-time ammonia storage-management control method increased the NOx conversion efficiency by 16%, and the ammonia slip decreased by 75%. Zhao et al. (2011) investigated the characteristics of ammonia storage and slip with a full-sized vanadium-based SCR catalyst under steady and transient conditions. The experimental results showed that the effect of the ammonia storage saturation level on the NOx conversion efficiency became more evident when the temperature was below 280°C.

Although the NH3/NOx ratio and catalyst temperature affect NOx reduction and ammonia slip, the nature of ammonia injection also influences NOx reduction and ammonia slip. Moreover, to meet new stringent exhaust regulations, most diesel engines use updated ammonia injection processes. The reason NOx reduction goes through a minimum when ammonia feed alternates between supply and shut-off should be studied, and the correlation between ammonia storage saturation level and NOx conversion efficiency during pulsing of ammonia into the feed should be investigated.

This study uses an SCR sample test bench to quantitatively investigate the characteristics of NOx reduction when ammonia is supplied and shut off with a vanadium-based SCR catalyst. The effect of temperature on NOx reduction is evaluated when ammonia feed is supplied and shut off. Furthermore, the correlation between the ammonia storage saturation level and the NOx reduction efficiency is studied as the ammonia feed is pulsed. The ammonia storage saturation level is defined from 0 to 100%, and the maximum ammonia storage capacity is defined as the maximum quantity of ammonia stored by the catalyst under steady-state conditions when the NH3/NOx ratio exceeds 1. It is possible to achieve better NOx conversion efficiency and eliminate NH3 slip by adopting a pulsed NH3 feed control strategy. These results can be used for power generation that is consistent with emission regulations.

### Experiment and methods

#### Sample test bench

The SCR system was tested on a test bench, as presented in Fig. 1, which includes four main parts: the gas mixture system, the SCR catalyst reactor, the temperature control system, and the measurement devices for analysis. The gas mixture system realizes mixing of different gases and provides flow of mixed gases through the flowmeter.

The gas mixture system has a chamber for mixing gases such as NO, N2, O2, and NH3, and the gas feed streams are dosed by mass flow controllers to produce different mixture ratios. N2 was used as the balance gas to control both the concentration and the gas hourly space velocity (GHSV). The SCR catalyst was placed into an electric furnace in a tubular cell, and a thermocouple was attached to the surface of the catalyst to measure the reaction temperature. All specifications of the test apparatus are described in Table 1.

The commercial V2O5-WO3-TiO2 catalyst supplied by HUNAN JATECH INC is a full honeycomb monolith, which is made of 4% V2O5, 9% WO3, 6% SiO2, and remaining TiO2 anatase. Table 2 shows the geometrical properties of the SCR monolith and washcoat.

#### The method of data process

The urea-SCR test system directly detects such parameters as the volume flow rate, the temperature, and the volume percent of both NOx and NH3. Based on these metered data, other parameters to evaluate the SCR system are calculated, such as the NOx conversion efficiency, the ammonia storage mass, the ammonia slip, and the ammonia saturation storage level.

The NOx conversion efficiency is calculated by Eq. 1.

$$\eta = \frac{V_{NOx, In} - V_{NOx, Out}}{V_{NOx, In}}$$  

(1)

Here, \(\eta\) is the NOx conversion efficiency, %. \(V_{NOx, In}\) is the NOx volume fraction at the inlet of the SCR catalyst, ppm. \(V_{NOx, Out}\) is the NOx volume fraction at the outlet of the SCR catalyst, ppm.

The ammonia storage mass may be calculated based on the ammonia injection mass rate, the reaction mass of ammonia, and the ammonia slip mass, as shown in Eq. 2.

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The ammonia storage mass may be calculated based on the ammonia injection mass rate, the reaction mass of ammonia, and the ammonia slip mass, as shown in Eq. 2.
Here, \( m_{\text{NH}_3,\text{load}} \) is the ammonia storage mass, g/L. 
\( dm_{\text{NH}_3,\text{inj}} \) is the ammonia injection mass rate, g/s. 
\( dm_{\text{NH}_3,\text{reacted}} \) is the ammonia reaction mass, g/s. 
\( dm_{\text{NH}_3,\text{slip}} \) is the ammonia slip mass, g/s. 
\( V_{\text{Cat}} \) is the volume of the catalyst, L.

The ammonia slip mass rate is deduced based on the ammonia slip and the gas mass flow rate, as shown in Eq. 3.

\[
dm_{\text{NH}_3,\text{slip}} = \frac{V_{\text{NH}_3,\text{Slip}} \cdot dm_{\text{Exh}}}{} \frac{M_{\text{NH}_3} C}{\rho_{\text{Exh}}} \tag{3}
\]

Here, \( V_{\text{NH}_3,\text{Slip}} \) is the ammonia slip volume fraction, ppm. 
\( dm_{\text{Exh}} \) is the exhaust gas mass flow rate, g/s. 
\( \rho_{\text{Exh}} \) is the exhaust gas density, g/L. 
\( M_{\text{NH}_3} \) is the mole mass of ammonia, g/mol. 
\( C \) is the mole volume of ammonia, mol/L.

The ammonia saturation storage level is calculated by Eq. 4.

\[
\varphi_{\text{NH}_3} = \frac{m_{\text{NH}_3,\text{cur}}}{m_{\text{NH}_3,\text{sat}}} \cdot 100 \tag{4}
\]

Here, \( \varphi_{\text{NH}_3} \) is the ammonia saturation storage level, %. 
\( m_{\text{NH}_3,\text{cur}} \) is the ammonia storage, g/L. 
\( m_{\text{NH}_3,\text{sat}} \) is the saturation ammonia storage, g/L.

### Results and discussion

**NH\(_3\)-NO\(_x\)-O\(_2\) standard reaction processing**

In the standard SCR reaction, NO, O\(_2\), and NH\(_3\) undergo oxidation and reduction reactions under the influence of the catalyst. There is a connection between NH\(_3\) injection and the concentration of reduced NO\(_x\). Figure 2 shows the relationship between NH\(_3\) and NO\(_x\) at the catalyst outlet when the NO concentration at the catalyst inlet is 738 ppm (the unit of volumetric concentration, in parts per million) and the NH\(_3\) concentration at the catalyst inlet is 1000 ppm, so the NH\(_3\)/NO\(_x\) ratio is 1.4. This shows that as the SCR catalyst is fed with NH\(_3\), NO at the catalyst outlet sharply declines to its minimum value (492 ppm), which is marked as point A. After point A, although the NH\(_3\) is continually fed into the SCR system and the outlet NH\(_3\) concentration remains at 800 ppm, NO begins to increase slightly up to 506 ppm. Once the NH\(_3\) feed is shut off, the NO concentration at the outlet first decreases to the minimum value (490 ppm), marked as point B, and then it rapidly increases up to the same high value as inlet NO concentration (738 ppm). This relationship between NO and NH\(_3\) may be explained by the reaction involving NO and NH\(_3\). When both the temperature and the GHSV are constant, over-injection of NH\(_3\) may obstruct the catalyzed reduction reaction occurring between NO and NH\(_3\). Furthermore, the level of ammonia slip at the SCR catalyst outlet increases

### Table 1 Specifications of the test apparatus

| Apparatus                  | Type                        | Range                  | Uncertainty |
|----------------------------|-----------------------------|------------------------|-------------|
| Electric furnace           | CNT QSK-3-12                | 0–1500°C               | ±1°C        |
|                                           | T-ramp: 0–20°C/min          |                        |             |
| Flowmeter                  | SINCERTITYDMF-1-1-AB        | 0–17 L/min             | ±0.1%       |
| Thermocouple               | Nickel chromium-nickel silicon | −200–1300°C          | ±0.75%      |
| NO\(_x\),NH\(_3\) analyzer | ECM NO\(_x\)/NH\(_3\)       | NO\(_x\):0–5000 ppm    | NO\(_3\): ±5 ppm |
|                                           | NH\(_3\):0–1000 ppm        | NH\(_3\): ±5 ppm       |             |
due to NH₃ over-injection, which causes secondary pollution. Therefore, it is necessary to effectively control NH₃ injection. Figure 3 shows the effect of the NH₃/NOₓ ratio on the reaction between NO and NH₃. Figure 3a describes the results seen for an NH₃/NO ratio = 1, in which the NO outlet concentration is steady at 516 ppm, and the outlet NH₃ concentration is steady at 540 ppm. Figure 3b considers an NH₃/NOₓ ratio of 0.7, the outlet NO concentration is steady at 527 ppm, and the outlet NH₃ concentration is steady at 320 ppm. The NH₃/NO ratio is 1.35, as shown in Fig. 2. With these figures, we found that NH₃ injection concentration decreases as the NH₃/NOₓ ratio decreases. The higher the NH₃/NOₓ ratio is, the quicker the rate of NO decrease, and the higher the level of ammonia slip.

Figure 4 illustrates the effect of the NH₃/NOₓ ratio on NO conversion efficiency and NH₃ slip. The outlet ammonia concentration drops sharply from 790 to 310 ppm when the NH₃/NOₓ ratio decreases from 1.4 to 0.7. It is clear that the level of NH₃ slip decreases with decreasing NH₃/NOₓ ratio. However, the NO conversion efficiency drops slightly with a decrease in the NH₃/NOₓ ratio. Therefore, a lower NH₃/NOₓ ratio is helpful in decreasing NH₃ slip, but it does so with lower NO conversion efficiency. It is necessary to investigate the NO and NH₃ reaction in detail to define parameters that effectively describe NO conversion and outlet NH₃ slip.

![Fig. 2](image)

**Table 2** Geometrical properties of urea-SCR monolith and urea-SCR washcoat

| Property                  | Value                      |
|---------------------------|----------------------------|
| Catalyst type             | V₂O₅-WO₃/TiO₂              |
| Supported metal           | Cordierite                 |
| Cell density              | 600CPSI                    |
| Diameter × length/mm      | ϕ23 × 24                   |
| Specific surface area cm²/cm³ | 34.3                   |

![Fig. 3](image)  

**Fig. 3** Effects of NH₃/NOₓ ratio on NO and NH₃ reactions. a NH₃/NOₓ ratio =1, b NH₃/NOₓ ratio =0.7
Key parameters of NH$_3$-NO$_x$-O$_2$ reacting processing

During the SCR test, NH$_3$ is fed to the catalyst via supply and shut-off steps, and the effects of the NH$_3$/NO$_x$ ratio and temperature on the catalytic reaction were carefully studied to determine effective parameters. Figure 5 shows the test results. Under these operating conditions, which used a catalyst temperature of 300°C and a space velocity of 30,000 h$^{-1}$, NO (744 ppm) was first supplied at the catalyst inlet for 50 s, and then NH$_3$ (523 ppm) was fed into the catalyst. Then, NH$_3$ was shut off once the NH$_3$ concentration at the catalyst outlet became stable.

There were three periods in the total NO$_x$-NH$_3$ catalytic reaction according to the results shown in Fig. 5: I, NO$_x$ decline period; II, NO$_x$-NH$_3$ stable period; III, NH$_3$ shut-off period.

I. NO$_x$ decline period The time when NH$_3$ injection began was defined as the start time or point 0, $t_0$=50 s. Once NH$_3$ was fed into the catalyst and reacted with NO on the catalyst, NO concentration decreased sharply. The time when the NO concentration declined to 95% of inlet NO concentration is marked as point 1, and the time from point 0 to point 1 is defined as TC. This NO decline period represents the response of the SCR catalytic reduction reaction. A shorter TC corresponds to a faster response of the SCR system.

II. NO$_x$-NH$_3$ stable period After point 1, the SCR system was still supplied with NH$_3$, and both NO and NH$_3$ concentrations became relatively stable. This is the stable period, and it ended once NH$_3$ was shut off at point 2. The interval from point 1 to point 2 constitutes the second period. At the beginning of the second period, NH$_3$ rapidly increased, which means that NH$_3$
slip occurred. During this NH$_3$ rapid increase period, the decrease in NO concentrations lowered. Furthermore, once the NH$_3$ slip level stabilized, NO stopped decreasing and increased slightly. In the second period, NH$_3$ slip occurred, which is marked as point 4. The time from NH$_3$ feed to NH$_3$ slip is defined as $T_A$, which represents ammonia absorption by the catalyst.

III. NH$_3$ shut-off period

Point 2 to end point 3 represents the last period, the NH$_3$ shut-off period. Just after the NH$_3$ feed was shut off, NO decreased slightly at first. The time when NO$_x$ decreased to its minimum after NH$_3$ shut-off was defined as $T_{D}$, which represents the system response after NH$_3$ shut-off. However, NO increased soon after $T_D$ and reached a value equal to the inlet NO concentration because there was a reduction reaction caused by the lack of NH$_3$. There is another time parameter in period III. After NH$_3$ input was shut off, NH$_3$ declined rapidly. The time when NH$_3$ concentration declined to 95% is marked as point 5, and the time from point 2 to point 5 is defined as $T_B$, which represents the ammonia desorption capacity.

In this work, four parameters were defined and adopted to represent the characteristics of the NO$_x$-NH$_3$ reaction, i.e., $T_A$, $T_B$, $T_C$, and $T_D$. To achieve better NO$_x$-NH$_3$ reaction characteristics, $T_A$ and $T_B$ should be increased, and $T_C$ and $T_D$ should be decreased. In the following sections, these parameters are discussed in detail based on the experimental results.

Figure 6a shows the relationship between $T_A$ and NH$_3$/NO$_x$ ratio with different catalyst carrier temperatures. $T_A$ declined with increasing NH$_3$/NO$_x$ ratio. In addition, the catalyst temperature had little influence on $T_A$. Figure 6b shows the ammonia saturation storage level when $T_A$ occurred. With different catalyst temperatures, the ammonia saturation storage was within the range 73–80%. As the NH$_3$/NO$_x$ ratio increased, the effective NH$_3$ injection per unit time increased, and at the same time, the ammonia saturation storage level rose at a higher rate, which resulted in the advancement of $T_A$. Therefore, $T_A$ decreased as the NH$_3$/NO$_x$ ratio increased. In addition, the influence of the catalyst carrier temperature on $T_A$ fell on two sides. First, when the catalyst carrier temperature rose, the ammonia saturation storage declined, and the time $T_A$ for saturation storage decreased. Second, catalyst activity was enhanced to boost the NO$_x$ conversion efficiency, which caused more consumption of NH$_3$ due to the active catalyst reaction, and less ammonia was stored; thus, the time $T_A$ for saturation storage increased. These results indicate that $T_A$ may remain stable with increasing catalyst temperature.

Figure 7 shows the effects of NH$_3$/NO$_x$ ratio on $T_C$. In Fig. 7a, $T_C$ decreased with the increase in the NH$_3$/NO$_x$ ratio. In Fig. 7b, the ammonia saturation storage level remained nearly stable (within the range of 68–77%) as the NH$_3$/NO$_x$ ratio increased. Furthermore, the ammonia saturation storage level increased slightly as the catalyst temperature rose. When the catalyst temperature was 250°C, the ammonia saturation storage level varied within the range 68.2–71.1%. When the catalyst temperature was 300°C, the ammonia saturation storage level varied within the range 73.1–76.8%. When the catalyst temperature was 350°C, the ammonia saturation storage level varied within the range 73.4–75.9%. These results show that at a low catalyst temperature, a higher NH$_3$/NO$_x$ ratio favored the NO catalytic reaction and led to a shorter $T_C$. Additionally, the ammonia saturation storage level was related to $T_C$; thus, the time when $T_C$ occurred may be estimated based on the ammonia saturation storage level.

Figure 8 shows the effects of NH$_3$/NO$_x$ ratio on $T_D$. In Fig. 7a, $T_D$ increased slightly with increasing NH$_3$/NO$_x$ ratio. In Fig. 7b, the ammonia saturation storage level declined as the NH$_3$/NO$_x$ ratio increased. Furthermore, when the catalyst temperature increased, $T_D$ decreased, but the ammonia saturation storage level increased. Thus, a higher catalyst temperature is helpful for a shorter $T_D$ and better NO$_x$ conversion efficiency.

The results from Figs. 6, 7, and 8 show that the ammonia saturation storage level had a great impact on the reaction of NO$_x$ and NH$_3$. For this case, $T_A$ was in the range 73–80%, and $T_C$ was in the range 68.2–76.8%. This means that when the ammonia saturation storage level was maintained at 68.2–76.8%, the NH$_3$ slip was maximally controlled, and the NO$_x$ conversion efficiency was further improved. Therefore, the ammonia saturation storage level directly affected the efficiency of NO$_x$ conversion.

To further investigate the influence of the ammonia saturation storage level on the NO$_x$ conversion efficiency and NH$_3$ slip, switching of the NH$_3$ injection process is discussed. Figure 9 shows the effects of starting NH$_3$ injection. At the beginning of NH$_3$ injection, the NO$_x$ conversion efficiency was still zero, which means that the reaction between NO$_x$ and NH$_3$ did not occur during the initial period of NH$_3$ injection. This is because the catalyst first absorbed NH$_3$, and there was no NH$_3$ available to affect the chemical reaction during this NH$_3$ absorption period. Once the ammonia saturation storage level was over 17%, the NO$_x$ conversion efficiency increased rapidly. This illustrates that after NH$_3$ absorption, NH$_3$ began to desorb and reacted with NO$_x$. Thus, the NO$_x$ conversion efficiency rose with increasing ammonia saturation storage level. Moreover, the maximum NO$_x$ conversion efficiency was 34% when the ammonia saturation storage level was 85%, but the NH$_3$ concentration at the catalyst outlet increased after the ammonia saturation storage level exceeded 80%. This means that NH$_3$ began to slip as the ammonia saturation storage level exceeded a threshold level. As a result of NH$_3$ slip, the NO$_x$ conversion efficiency dropped as the ammonia saturation storage level continued to increase.

Figure 10 shows the results obtained after stopping NH$_3$ injection. This indicates that the ammonia saturation storage level began to decline as NH$_3$ injection was stopped. While
the ammonia saturation storage level began to drop, the NO\textsubscript{x} conversion efficiency increased slightly. The NO\textsubscript{x} conversion efficiency reached its maximum level at 33% when the ammonia saturation storage level decreased to 38%. After that, the NO\textsubscript{x} conversion efficiency dropped with decreasing ammonia saturation storage level. The outlet NH\textsubscript{3} concentration declined overall as the ammonia saturation storage level decreased. At the beginning, the outlet NH\textsubscript{3} concentration decreased slightly, but it dropped sharply as the ammonia saturation storage level fell below 70%. This is because NH\textsubscript{3} slip was diminished as NH\textsubscript{3} injection was stopped.

These results reveal that NO\textsubscript{x} conversion efficiency reached its maximum level at the beginning of NH\textsubscript{3} injection and maintained it until the ammonia saturation storage level reached a high threshold; when NH\textsubscript{3} injection was stopped, the NO\textsubscript{x} conversion efficiency reached a maximum level until the ammonia saturation storage level decreased to a small threshold. Therefore, during NH\textsubscript{3} injection, the NO\textsubscript{x} conversion efficiency exhibited two peak values reflecting two ammonia saturation storage levels. It is possible to improve NO\textsubscript{x} conversion efficiency and reduce NH\textsubscript{3} slip by effectively controlling the ammonia saturation storage level within a reasonable range. This may be achieved by pulsing NH\textsubscript{3} injection.

**Variable NH\textsubscript{3}/NO\textsubscript{x} ratio and ammonia slip**

When the NH\textsubscript{3}/NO ratio was greater than the maximum NO\textsubscript{x} conversion efficiency corresponding to the NH\textsubscript{3}/NO\textsubscript{x} ratio realized under the current temperature and space velocity conditions, the NO\textsubscript{x} conversion efficiency no longer increased as the NH\textsubscript{3}/NO\textsubscript{x} ratio increased. Therefore, the effect of the fixed NH\textsubscript{3}/NO\textsubscript{x} injection ratio on the final efficiency of NO\textsubscript{x} conversion is predictable, but the influence of the variable NH\textsubscript{3}/NO\textsubscript{x} ratio injection method on NO\textsubscript{x} conversion efficiency needs further study. Figure 11 shows the relationship between NO\textsubscript{x} conversion efficiency and ammonia storage in the variable NH\textsubscript{3}/NO\textsubscript{x} ratio injection mode. The catalyst bed temperature was 250°C, and the gas hourly space velocity was 2.5×10\textsuperscript{4} h\textsuperscript{-1}.

Under normal injection conditions, it takes 8.4 s from the start of injection to the start of efficient NO\textsubscript{x} conversion. As
**Fig. 7** Effects of NH$_3$/NOx ratio on T$_C$ based on different temperature

![Graph showing the effects of NH$_3$/NOx ratio on T$_C$](image)

**Fig. 8** Effects of NH$_3$/NOx ratio on T$_D$ based on different temperature

![Graph showing the effects of NH$_3$/NOx ratio on T$_D$](image)
the injection NH₃/NOₓ ratio first increased and then decreased, the delay time was shortened to 8.2 s and then 6.7 s. According to the size and duration of the NH₃/NOₓ ratio, there was an inflection point between ammonia storage and NOₓ conversion efficiency. To facilitate the comparison of these effects, the change in NOₓ conversion efficiency and the change in ammonia storage within a time period after ammonia injection were used for evaluation, and the detailed parameters are shown in Table 3.

From Fig. 3a, we found that when the NH₃/NOₓ ratio was 0.45, the rate for change in the level of ammonia storage was 0.82 mg·L⁻¹/s within 17.6 s after ammonia injection. The timing started at 8.4 s after ammonia injection, and within 22.2 s, the change rate for NOₓ conversion efficiency was 0.49%/s. Figure 3b showed that the NH₃/NOₓ ratio in the first stage was 0.60, the duration was 19.2 s, and the NH₃/NOₓ ratio in the second stage was 0.44. Within 19.7 s after ammonia injection, the rate of change in ammonia storage level was 0.98 mg·L⁻¹/s. The time started at 8.2 s after ammonia injection, and within 21.3 s, the change rate for NOₓ conversion efficiency was 0.89%/s. Figure 3c shows that the NH₃/NOₓ ratio in the first stage was 0.97, the duration was 22.6 s, and the NH₃/NOₓ ratio in the second stage was 0.34. Within 22.8 s after ammonia injection, the rate of change in ammonia storage level was 1.48 mg·L⁻¹/s. The time started at 6.7 s after ammonia injection, and within 26.7 s, the change rate of NOₓ conversion efficiency was 1.08%/s. Comprehensive comparison shows that the ammonia-injected method with which the NH₃/NOₓ ratio was first larger and then smaller was beneficial for the rapid improvement of NOₓ conversion efficiency, but the appropriate NH₃/NOₓ ratio and continuous injection time must be controlled, or it is easy to cause ammonia slip.

It is important to improve SCR conversion efficiency to reasonably control NH₃ slip. In this work, ammonia slip test experiments were performed with different NH₃/NOₓ ratios and catalyst temperatures.
Figure 12 shows the effect of the NH$_3$/NO$_x$ ratio on ammonia slip when the catalyst temperature was 300°C. At a time of 50 s, NH$_3$ was injected into the catalyst carrier. As soon as NH$_3$ was supplied, the ammonia saturation storage level began to increase. For each NH$_3$/NO$_x$ ratio, the ammonia saturation storage level became saturated and constant. The curve for ammonia saturation storage level exhibited a sharp gradient with increasing NH$_3$/NO$_x$ ratio. As the ammonia saturation storage level increased up to a threshold, NH$_3$ slip occurred at the outlet. For different NH$_3$/NO$_x$ ratios, this ammonia slip threshold varied. For NH$_3$/NO$_x$ ratio values of 0.7, 1.0, and 1.4, the ammonia slip thresholds were 79%, 75%, and 72%.
76%, respectively, with an average of 76.7%. The time when the ammonia slip threshold occurred was defined as the time threshold of the ammonia slip and marked by \( t_s \). This time threshold \( t_s \) advanced with increasing NH\(_3\)/NO\(_x\) ratio. Thus, \( t_s \) could be predicted based on the ammonia saturation storage level for a certain NH\(_3\)/NO\(_x\) ratio.

Figure 13 shows the effects of catalyst temperature on NH\(_3\) slip. During this experiment, the NO gas at the carrier inlet had a concentration of 750 ppm, the NH\(_3\)/NO\(_x\) ratio was 1.0, and the catalyst space velocity was 30,000 h\(^{-1}\). The catalyst carrier temperature was adjusted to 250°C, 300°C, or 350°C. This shows that NH\(_3\) slip occurred at \( t_s=120 \) s, and NH\(_3\) slip decreased with increasing catalyst temperature, which demonstrates that a higher catalyst temperature favors the NH\(_3\)-NO\(_x\) reaction and better NO\(_x\) conversion efficiency. In addition, the catalyst temperature had little effect on the NH\(_3\) slip threshold time \( t_s \). Furthermore, as the catalyst temperature increased, the ammonia saturation storage level increased slightly before NH\(_3\) slip occurred, but it decreased once NH\(_3\) slip occurred. In fact, the ammonia saturation storage level changed slightly as the catalyst temperature was varied. With these three temperatures, the ammonia saturation storage level exhibited values of 77.2%, 75%, and 75.2%, with an average of 75.8%. Compared with the effects of the NH\(_3\)/NO\(_x\) ratio on NH\(_3\) slip, those of the catalyst temperature were smaller.

In summary, the results showed that the NH\(_3\)/NO\(_x\) ratio and the catalyst temperature influenced NH\(_3\) slip, and the NH\(_3\)/NO\(_x\) ratio had a greater effect. Furthermore, the NH\(_3\) slip threshold time \( t_s \) remained constant, although the catalyst temperature changed. When the catalyst temperature was 250–350 °C and the NH\(_3\)/NO\(_x\) ratio was 0.7–1.4, the ammonia saturation storage level corresponding to the NH\(_3\) slip threshold was approximately 75–79%. The results also revealed that NO\(_x\) conversion efficiency was best when the ammonia saturation storage level was approximately 68.2–73%, and within this range for ammonia saturation storage level, there was no NH\(_3\) slip. Thus, it is possible to achieve better NO\(_x\) conversion efficiency together with no NH\(_3\) slip by adjusting the SCR system to maintain the ammonia saturation storage level in the range 68.2–73%. This is possible by adopting an NH\(_3\) pulsed feed control strategy.

### Table 3  Effect of variable NH\(_3\)/NO\(_x\) ratios on the SCR system

| Property of the NH\(_3\)/NO\(_x\) ratio (-) | Change of NO\(_x\) conversion efficiency (%/s) | Change of ammonia storage (mg·L\(^{-1}\)/s) | Ammonia slip peak volume concentration (×10\(^{-6}\)) |
|------------------------------------------|-----------------------------------------------|---------------------------------------------|-------------------------------------------------|
| NH\(_3\)/NO\(_x\) ratio kept at 0.44     | 0.49                                          | 0.82                                        | 3                                               |
| NH\(_3\)/NO\(_x\) ratio varied from 0.6 to 0.44 | 0.89                                          | 0.98                                        | 6                                               |
| NH\(_3\)/NO\(_x\) ratio varied from 0.97 to 0.34 | 1.08                                          | 1.48                                        | 12                                              |

![Fig. 12](image-url)  
**Fig. 12** Effects of NH\(_3\)/NO\(_x\) ratio on ammonia slip
Control strategy and NH₃ pulsing feed

The results described above show that the saturated storage capacity for ammonia is closely related to temperature. The higher the catalyst temperature, the smaller the saturated storage capacity for ammonia. The theoretical ammonia storage saturation and the actual ammonia storage saturation will not be identical. The difference between the two is called the safety factor. A greater safety factor means a smaller risk of ammonia slip, and a smaller safety factor means a higher risk of ammonia slip. To quickly reach the ammonia storage saturation level corresponding to the critical point for ammonia leakage, it is necessary to increase the NH₃/NOₓ ratio. It is also necessary to consider the influence of temperature change rate on ammonia storage saturation when designing the control algorithm. When the temperature rises, the ammonia storage saturation should be appropriately reduced, and the safety factor should be increased. When the temperature decreases, the ammonia storage saturation should be appropriately increased, the safety factor should be reduced, and the NOₓ conversion efficiency should be improved. Figure 14 shows a schematic diagram of ammonia storage saturation control. To compensate for system control error, the actual level for ammonia storage saturation changes within a controllable error band, and the error band is related to the rate of temperature change. The purpose is to control ammonia slip at the different temperatures.

The NH₃/NOₓ ratio and temperature also impact ammonia slip, but the ammonia storage saturation corresponding to the critical point of ammonia slip does not change with changes in the NH₃/NOₓ ratio and temperature.

When designing the control strategy, it is necessary to design a safety factor situated between the theoretical value and the actual value of the ammonia storage saturation level. As the temperature change rate increases, the safety factor increases, and as the temperature change rate decreases, the safety factor decreases. Experiments show that controlling the ammonia storage saturation level between 75 and 80% gives the best synergistic control of NOₓ conversion efficiency and ammonia slip. In actual control, a certain error is considered, and a correction coefficient is introduced for adjustment. The formula for calculation of the ammonia storage saturation value is shown in Eq. 5.

\[
r_{NH3, Set} = r_{NH3, Opt} \cdot fac_{T, VS}
\]  

Here, \( r_{NH3, Set} \) is the storage saturation setting value, %. \( r_{NH3, Opt} \) is the storage saturation theoretical value, %. \( fac_{T, VS} \) is the correction factor based on different working point.

Fig. 13 Effects of catalyst temperature on ammonia slip

Fig. 14 Schematic diagram of ammonia storage saturation control
Based on the SCR reaction rate model, temperature prediction model, and exhaust gas flow model, the ammonia storage saturation level can be calculated, and the ammonia storage saturation of each unit module can be determined based on the law of conservation of mass. The formula is shown in Eq. 6.

\[
\frac{d\theta}{dt} = \frac{r_{ads} - r_{des} - 4r_{std} - 8r_{sbe} - 6r_{ox,s}}{\theta_{NH3}}
\]  

Here, \(\theta\) is the ammonia storage saturation, \%. 
\(r_{ads}\) is the ammonia adsorption rate, mol·m\(^{-3}\)/s. 
\(r_{des}\) is the ammonia desorption rate, mol·m\(^{-3}\)/s. 
\(r_{std}\) is the reaction rate of NO in standard reaction, mol·m\(^{-3}\)/s. 
\(r_{sbe}\) is the reaction rate of NO\(_2\) in slowly reaction, mol·m\(^{-3}\)/s. 
\(r_{ox,s}\) is the reaction rate of ammonia oxidation, mol·m\(^{-3}\)/s. 
\(\theta_{NH3}\) is the Saturated ammonia storage capacity, mol/m\(^2\).

Figure 15 presents a schematic diagram of the principles of the control algorithm, in which the ammonia storage saturation is calculated based on the NO\(_x\) concentration upstream of the catalyst, the exhaust gas flow, the urea injection volume, and the ammonia slip volume. The control algorithm is based on the calculation of ammonia storage saturation and takes it as the control target. According to differences in ammonia storage saturation, the controller corrects the urea injection to achieve control of ammonia storage saturation.

To achieve better NO\(_x\) conversion efficiency, an NH\(_3\) pulsed feed experiment was conducted. During the experiment, NH\(_3\) was fed via pulse injection into the catalyst carrier, and the pulse injection interval was controlled to adjust the NH\(_3\) injection volume flow rate per unit time. The catalyst temperature was controlled at 350°C, and the catalyst space velocity was 30,000 h\(^{-1}\).

Figure 16 shows the results for different pulse injection intervals. The NH\(_3\) pulse injection interval, marked as \(t_p\), was set to 5 s, 10 s, 15 s, 30 s, 60 s, and 90 s. The outlet NH\(_3\) concentration (i.e., the NH\(_3\) slip) decreased as the pulse injection interval declined. When the pulse injection interval was 90 s, the NH\(_3\) slip was the highest (over 80 ppm). When \(t_p\) was 10 s, the NH\(_3\) slip dropped to 9.6 ppm. As \(t_p\) was shortened to 5 s, there was zero NH\(_3\) slip at the outlet. The ammonia saturation storage level decreased when the pulse injection interval decreased, but as NH\(_3\) slip disappeared, the storage level increased slightly. Moreover, the NH\(_3\) pulse injection interval had little influence on the mean concentration of NO at the outlet; however, it affected the peak value of NO concentration. The mean value for NO outlet concentration rose slightly with increasing \(t_p\). The NO mean value was 446 ppm when \(t_p\) was 5 s, and it became 452 ppm and 460 ppm at \(t_p\) values of 10 s and 15 s, respectively.

This work also involved experiments with varied pulse injection intervals. During the experiment, NH\(_3\) was first injected with a larger injection interval, and the injection stopped once the outlet NO\(_x\) concentration declined to the minimum level needed to achieve good NO\(_x\) conversion efficiency; then, the injection interval was adjusted a shorter time, and the outlet NO\(_x\) and NH\(_3\) concentrations were recorded. Figure 17 shows results for various pulse injection intervals \(t_p\). Here, we introduced 4 kinds of operation cases, marked as A, B, C, and D. In case A, the pulse injection interval \(t_p\) was constant, \(t_p = 5\) s. In case B, first \(t_p=45\) s, and then \(t_p\) was adjusted to 5 s. In case C, first \(t_p=60\) s, and then \(t_p=5\) s. In case D, first \(t_p=90\) s, and then \(t_p=5\) s. This shows that variations in \(t_p\) had little influence on either the outlet NO concentration peak value or the outlet NO concentration mean value. However, compared with case A with constant \(t_p\), the minimum NO levels for cases B, C, and D were all decreased. Furthermore, the minimum NO concentration declined with increasing first pulse time. In addition, there was no NH\(_3\) slip.
in case A or B. However, as the first pulse time was increased (cases C and D), NH₃ slip occurred at the catalyst outlet, as marked by C1 and D1. In case C1, the maximum NH₃ slip was 4 ppm, but in case D1, it increased sharply up to 75 ppm. Additionally, compared with case A with constant $t_p$, the ammonia saturation storage level increased with increasing first pulse time. Therefore, these results reveal that it is possible to reduce outlet NO concentration by using varied NH₃ pulse injection intervals, but an excessively long first pulse time tends to induce NH₃ slip. Therefore, NOₓ conversion efficiency may be improved by adopting a suitable NH₃ pulse injection interval.

**Conclusions**

In this work, the SCR catalyst standard reaction process and the effects of key parameters on the reaction are investigated...
experimentally, and NH$_3$ pulse injection is further discussed in detail. The main observations and conclusions are as follows:

The total NO$_x$-NH$_3$ catalytic reaction was divided into three periods: I, NO$_x$ decline period; II, NO$_x$-NH$_3$ stable period; and III, NH$_3$ shut-off period. During the NO$_x$-NH$_3$ stable period, NH$_3$ slip occurs. In the three periods, four parameters are defined and adopted to represent the characteristics of the NO$_x$-NH$_3$ reaction, i.e., T$_A$, T$_B$, T$_C$, and T$_D$. Although the NH$_3$/NO$_x$ ratio and catalyst bed temperature have a great impact on T$_A$, T$_B$, T$_C$, and T$_D$, the variations in ammonia saturation storage level with changes in the NH$_3$/NO$_x$ ratio and catalyst bed temperature are very small. The value of the ammonia saturation storage level when T$_A$ occurs is within the range 73~80%; when T$_C$ occurs, the value is within the range 68.2~76.8%. The time at which NH$_3$ slip occurs and the ammonia saturation storage level are strongly correlated. When NH$_3$/NO$_x$ ratio is 0.7, 1.0, or 1.4 and the catalyst temperature is set to 250~350°C, the ammonia saturation storage level is within the range 75~79% at the onset of NH$_3$ slip. It is helpful to improve the NO$_x$ conversion efficiency and reduce NH$_3$ slip by effectively controlling the ammonia saturation storage level within a reasonable range.

The process of pulsing NH$_3$ injection affects the NO$_x$ conversion efficiency and NH$_3$ slip. When the NH$_3$ injection starts, the NO$_x$ conversion efficiency increases rapidly until the ammonia saturation storage level reaches 17%. NH$_3$ begins to slip as the ammonia saturation storage level exceeds 80%. Moreover, the maximum NO$_x$ conversion efficiency is 34% when the ammonia saturation storage level is 85%. After stopping NH$_3$ input, the NO$_x$ conversion efficiency reaches its maximum of 33% when the ammonia saturation storage level decreases to 38%.

Pulsed injection can improve the efficiency of NO$_x$ conversion and reduce NH$_3$ slip. The period of pulse injection has little influence on the mean value of NO$_x$ concentration at the outlet; however, it affects the peak values of NO$_x$ concentration and NH$_3$ slip. The use of various periods for pulse injection further improves the NO$_x$ conversion efficiency and restrains NH$_3$ slip. It is possible to achieve better NO$_x$ conversion efficiency together with no NH$_3$ slip by adopting an NH$_3$ pulsed feed control strategy.

**Author contribution** All authors contributed to the study conception and design. Material preparation, design of experiment, and analysis were performed by Yue Guangzhao and Qiu Tao. The first draft of the manuscript was written by Lei Yan. All authors read and approved the final manuscript, no competing interests.

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**Data availability** All relevant data are within the manuscript and available from the corresponding author upon request.

**Declarations**

**Ethics approval** All analyses were not based on animal and human, thus no ethical approval is required.

**Consent for publication** All authors agree to publish.

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