Study on the removal of NO from flue gas by wet scrubbing using NaClO$_3$

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Abstract: In order to remove nitric oxide (NO) from flue gas, from small coal-fired boilers, it is necessary to exploit the cost-effective wet denitration technology. The absorption of NO with sodium chlorate solution was studied. The effects of experimental conditions, such as temperature, NaClO$_3$ concentration, type of acid, mole ratio of NaClO$_3$ to hydrogen ions, on NO removal rate were investigated, and the optimal conditions were established. As the effect of temperature on denitration was related to the type of acid used, the temperature required for sulfuric acid was high, and the temperature required for nitric acid was low. The optimal mole ratio between NaClO$_3$ and the two types of acids was the same. The reaction products were analyzed by ion chromatography. The reacted solution could be recycled after the removal of sodium chloride. The reaction mechanism and the total chemical reaction equation of NaClO$_3$ denitration were deduced. The thermodynamic derivations showed that this oxidation reaction could proceed spontaneously and the reaction was very thorough. NaClO$_3$ exhibited high NO removal efficiency and its denitration cost was much lower than sodium chlorite.

Keywords: nitrogen oxides; oxidation; absorption; NaClO$_3$; denitration.

INTRODUCTION

Air pollution caused by the burning of fossil fuels is increasingly serious, and the NO$_x$ emitted by the fuels can lead to atmospheric ozone depletion, acid rain[1,2] and visibility problems, through a series of complex reactions with water and oxygen.[3]

Denitration methods such as selective catalytic reduction (SCR) and selective non-catalytic reduction (SNCR) have been extensively studied proved to be a great success in large power plants. However, due to the high cost of SCR and the low efficiency of SNCR, industrial denitration process[4] is still immature in

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the practical application of small coal-fired boilers.[5,6] In addition, the ammonia denitration efficiency is low due to the large temperature difference of flue gas from small boilers. These technologies can not be applied to the treatment of dusty flue gases in steel, ceramics and cement plants. A large amount of dust in the flue gas can easily block the catalyst channel and cause catalyst poisoning. The development of efficient denitration technology is of great significance to environmental protection in various countries.

The wet denitration technology is less affected by temperature in removing dust. It requires less investment and is suitable for coal-fired boilers in oil wells and remote areas. Therefore, it is necessary to develop economical and efficient wet denitration technology.[7–11]

Wet denitration technology is one of the most popular technologies in industrial scale in recent years. The method uses a strong oxidant to oxidize NO to nitrogen dioxide (NO_2),[12,13] which can be absorbed by water or alkaline solution. Up to now, a varity of oxidants, such as ClO_2,[14] $KMnO_4$/NaOH,[15] $NaClO_2$,[16–18] $NaClO_2$/NaOH,[19–21] $NaClO_2$/$(NH_2)_2CO$,[22] H_2O_2[23,24] and water-soluble ferrous-chelating agents,[25] may be included. Among these oxidants, $NaClO_2$ showed better oxidation performance and the industrial application test was carried out. However, the treatment cost of the system was too high to be a practical NO_x control process.

In order to reduce the denitration cost and improve the denitration efficiency, $NaClO_3$ was used to remove NO_x from simulated flue gas in this paper. One molecule of $NaClO_3$ can oxidize more NO than the one of commonly used $NaClO_2$, while the price of $NaClO_3$ is only one third of $NaClO_2$. The reacted liquid could be reused after removing NaCl from the solution. Therefore, developing the denitration technology with $NaClO_3$ has a good prospect for the wet denitration of industrial flue gas. However, there are few basic researches on $NaClO_3$ denitration technology.

In this research, a new denitration solution made of $NaClO_3$ was used for the removal of NO in a bubble reactor. Various influencing factors on the removal efficiency of NO were measured, in order to determine the optimal experimental conditions. The reaction mechanism and the total chemical reaction equation for denitration using $NaClO_3$ were deduced. This method might be applied to an industrial scale denitration process.

EXPERIMENTAL

Materials

The analytical grade reagents used in experiment were H_2SO_4 (95–98 wt. %) and HNO_3 (65–68 wt. %), obtained from Laiyang Chemical Reagent Factory, China. $NaClO_3$ (purity > 99 wt. %) was obtained from Tianjin Chemical Reagent Factory, China. Standard gases, N_2 (99 %) and NO/N_2 (2.02 vol. ‰ NO) span gas, were obtained from Oxygen Co., Ltd., China. Reverse osmosis water was applied to prepare the solutions.

Experimental setup

The experimental system of this research consists of three parts: a flue gas simulation section, a bubble reactor and a nitrogen oxide analyzer. A schematic diagram of the experimental device is shown in Fig. 1.
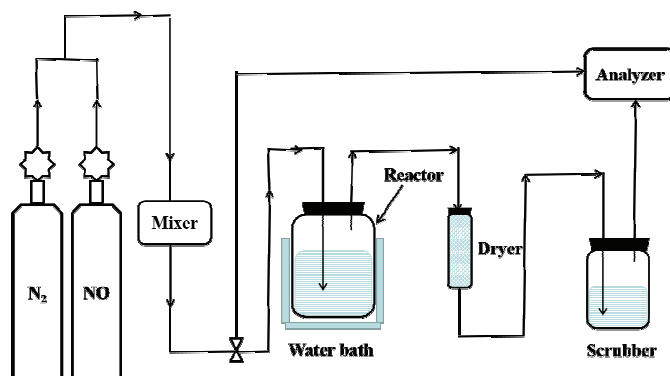

Fig. 1. Schematic diagram of the experimental setup.

Nitrogen and NO/N$_2$ were obtained from cylinders and metered through mass flow controllers. The total flow of blended gas was kept at 160 mL/min. The simulated flue gas was then introduced into the mixer and NO was diluted to 800 ppm. The absorption experiments were performed in a bubbling reactor. The temperature of the absorbing liquid (solution volume = 500 mL; liquid height = 12 cm) was controlled by a water bath. The solution was stirred magnetically and the simulated flue gas entered the bubbling reactor. The concentration of NO$_x$ was continuously recorded by flue gas analyzer.

In order to protect the flue gas analyzer, the gas coming from the reactor passed through a drying tube containing anhydrous calcium chloride and a scrubber with phosphoric acid.

Data analysis

When the simulated flue gas flowed through the aqueous solutions of NaClO$_3$, NO reacted with the oxidant and was removed. The removal efficiency of NO in % was defined as:

$$E_f = 100 \frac{C_{NO_{in}} - C_{NOx_{out}}}{C_{NO_{in}}} \tag{1}$$

where E_f is the efficiency of NO removal, $C_{NO_{in}}$ and $C_{NOx_{out}}$ represent the inlet and outlet gas concentrations, respectively. The denitration rate was calculated by the integral method.

RESULTS AND DISCUSSION

Contrast study in different systems

NaClO$_3$ was used to remove NO in a bubble reactor and a series of comparative experiments were carried out under different conditions. The result is shown in Fig. 2. NO removal efficiency was 29.11 % in water, 28.82 % in H$_2$SO$_4$ solution and 29.63 % in HNO$_3$ solution. The removal efficiency of NO was only 29.52 % by NaClO$_3$ solution without the presence of acid, which was just similar to water. However, the removal efficiency of NO achieved 84.40 % by the com-

bination of 0.50 % $NaClO_3$ and 0.60 mol/L HNO_3. Similarly, the removal efficiency of NO in the mixed solution of $NaClO_3$ and H_2SO_4 reached 86.17 %. The results showed that $NaClO_3$ combined with acid significantly promoted the removal of NO.
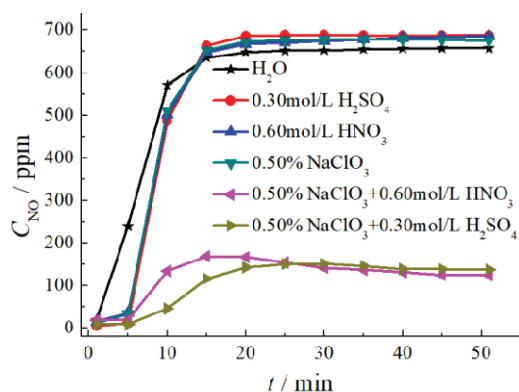

Fig. 2. Contrast study of denitration in different systems.

Effect of temperature

The reaction temperature was an important factor affecting the denitration effect. Experiments were carried out under the conditon of room temperature to 80 °C. It could be seen from Fig. 3a that the concentration of outlet NO_x gradually decreased when the temperature rose from 30 to 80 °C in $NaClO_3/H_2SO_4$ system. Considering the accelerating evaporation of water with the increase of temperature, the optimal temperature in practical application was chosen to be 80 °C.
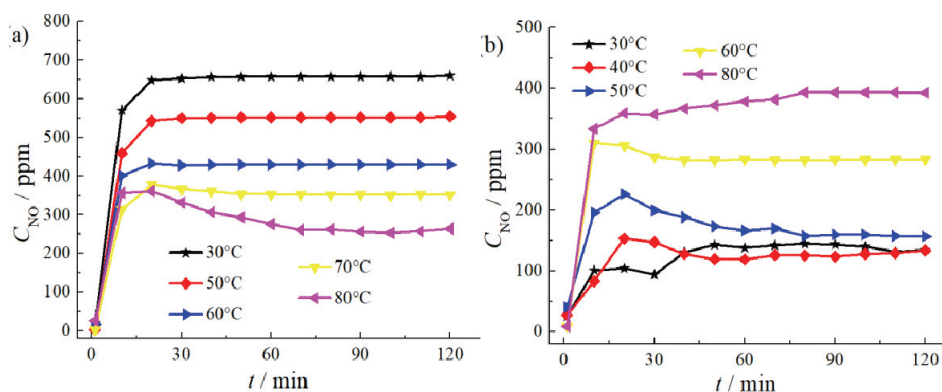

Fig. 3. Effect of temperature on NO removal efficiency.

However, for $NaClO_3/HNO_3$ system, the denitration efficiency decreased rapidly from 85.14 to 54.83 % with the temperature from 40 to 80 °C (Fig. 3b), which was contrary to the $NaClO_3/H_2SO_4$ system. Therefore, 40 °C was selected as the optimal temperature for $NaClO_3/HNO_3$ system. The optimal temperature

was close to the operating temperature of wet flue gas desulfurization (WFGD), which was very conducive to the joint operation of desulfurization and denitration process.

Effect of NaClO$_3$ concentration

The effect of NaClO$_3$ concentration on NO absorption efficiency was carried out to ascertain the optimal NaClO$_3$ concentration and the results were shown in Fig. 4. The removal efficiency of NO in NaClO$_3$/H$_2$SO$_4$ system increased sharply with an increment of NaClO$_3$ concentration at first, and then grew slowly. When the concentration of NaClO$_3$ was 0.50 %, the NO removal efficiency reached 83.53 %. This can be explained from two aspects: chemical reaction and physical properties. The chemical reaction can improve the removal efficiency of NO, but the viscosity of denitration liquid increases with the increment of NaClO$_3$ concentration, therefore, the liquid diffusion coefficient and solubility of NO decrease.
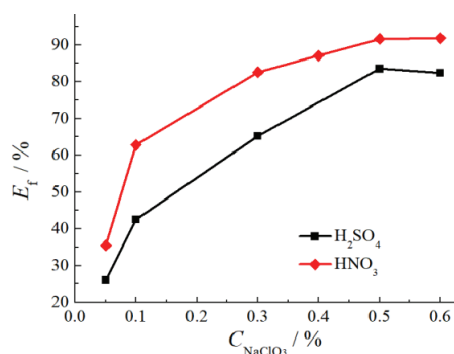

Fig. 4. Effect of NaClO$_3$ concentration on NO removal efficiency: H$_2$SO$_4$ concentration, 0.30 mol/L, HNO$_3$ concentration, 0.60 mol/L.

The effect of NaClO$_3$ concentration on NO absorption efficiency in NaClO$_3$/ /HNO$_3$ system indicated that NO absorption efficiency increased sharply from 35.48 to 91.65 % with NaClO$_3$ concentration rising from 0.05 to 0.50 %. But the change was not obvious when NaClO$_3$ concentration exceeded 0.50 %. So the optimal NaClO$_3$ concentration was chosen to be 0.50 %.

Effect of NaClO$_3$ to H$^+$ mole ratio and type of acid

The influence of NaClO$_3$ to H$^+$ mole ratio on the removal efficiency of NO was studied, and the result was shown in Fig. 5. A significant increment of the NO removal efficiency from 53.30 to about 87.32 % was observed when the mole ratio of NaClO$_3$ to H$^+$ varied from 1:2.13 to 1:8.50. When the mole ratio of NaClO$_3$ to H$^+$ was 1:12.76, the maximum absorption efficiency of NO was about 91.68 %. This indicated that the oxidizing ability of NaClO$_3$ gradually increased with the increase of acid intensity.[26,27] With the further increase of molar ratio, the removal efficiency of NO did not change much.
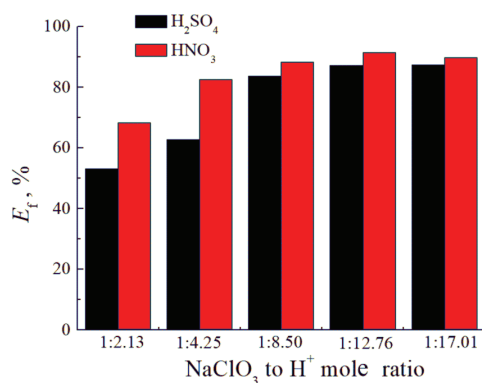
Fig. 5. Effect of molar ratio of $NaClO_3$ to H^+ and type of acid on NO removal efficiency.

The type of acid was also a factor affecting NO absorption. It could be seen from Fig. 5 that the absorption efficiency of NO by H_2SO_4 was slightly higher than by HNO_3 at the same molar ratio.

Analysis of products in solution

In order to deduce the reaction mechanism, the results of ion chromatography (IC) analysis of the products are given in Table I. Nitrites were not found, while the main anions in the absorbent solution were nitrates and chlorides. This indicates that the NO_x directly oxidize to nitrates. The Cl^- comes from the reduced chlorate.
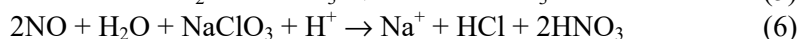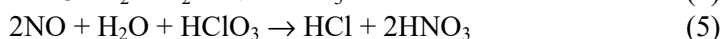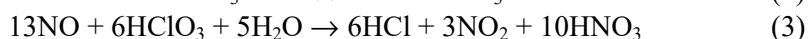
Table I. Products of denitration; A1 – 1:12.76 (H_2SO_4); A2 – 1:17.01 (H_2SO_4); A3 – 1:12.76 (HNO_3); A4 – 1:17.01 (HNO_3)

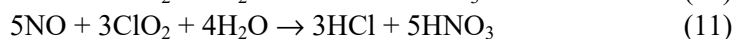| Medium	$C_{NO_3^-}$ / mg L^{-1}	C_{Cl^-} / mg L^{-1}
A1	85.03	24.243
A2	80.12	21.205
A3	–	24.240
A4	–	22.066

Denitration reaction mechanism

It was worth pointing out that the solution remained clear and transparent rather than yellow-green during the denitration process using $NaClO_3$ solution, indicating that no chlorine dioxide (ClO_2) was generated. This was different from the phenomenon of removing NO_x from simulated flue gas by acidic $NaClO_2$ solution.[28,29] ClO_2 is a very harsh gas and even very small amount of ClO_2 can be detected. In a mass concentration of 0.025 % $NaClO_2$ and 0.12 % H_2SO_4 solution, it could be observed clearly that the solution was yellow-green. However, the solution of a mass concentration of 0.40 % $NaClO_3$ and 2.55 % H_2SO_4 remained colourless, proving that no ClO_2 existed. The process involved numerous

chemical reactions. The mechanism of NO absorption by $NaClO_3$ may be as follows:

$$NaClO_3 + H^+ \leftrightarrow Na^+ + HClO_3 \tag{2}$$
$$13NO + 6HClO_3 + 5H_2O \rightarrow 6HCl + 3NO_2 + 10HNO_3 \tag{3}$$
$$3NO_2 + H_2O \rightarrow 2HNO_3 + NO \tag{4}$$
$$2NO + H_2O + HClO_3 \rightarrow HCl + 2HNO_3 \tag{5}$$
$$2NO + H_2O + NaClO_3 + H^+ \rightarrow Na^+ + HCl + 2HNO_3 \tag{6}$$
$$NaClO_3 + 2NO + H_2O \rightarrow 2HNO_3 + NaCl \tag{7}$$

The reported denitration mechanism with $NaClO_2$ is shown in Eqs. (8)–(12), in which ClO_2 is produced in the reaction process, which possesses a potential hazard for the actual use:

$$8NaClO_2 + 8H^+ \rightarrow 6ClO_2 + Cl_2 + 4H_2O + 8Na \tag{8}$$
$$5NO + 2ClO_2 + H_2O \rightarrow 2HCl + 5NO_2 \tag{9}$$
$$5NO + ClO_2 + 3H_2O \rightarrow HCl + 5HNO_3 \tag{10}$$
$$5NO + 3ClO_2 + 4H_2O \rightarrow 3HCl + 5HNO_3 \tag{11}$$
$$3NaClO_2 + 4NO + 2H_2O \rightarrow 4HNO_3 + 3NaCl \tag{12}$$
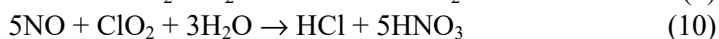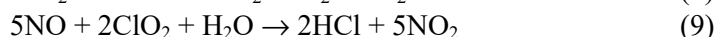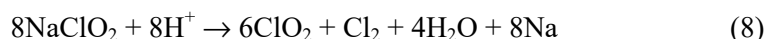
Reacting 1.00 kg of NO requires 2.26 kg $NaClO_2$, but only 1.77 kg $NaClO_3$ calculated with Eqs. (7) and (12). In addition, the price of $NaClO_3$ is one third of $NaClO_2$. In other words, the cost of $NaClO_3$ process is about a quarter of the current cost of $NaClO_2$ wet denitration technology. Due to the high rate of denitration and low cost, the denitration technology with $NaClO_3$ has a good industrial application prospect for the wet denitration of industrial flue gas.

Chemical thermodynamics

The thermodynamic data of chemical reactions are important for assessing the extent of the reaction (Eq. (7)). Data calculations of the thermodynamic parameters were conducted and the enthalpy change of reaction ($\Delta_r H_m(T)$), Gibbs energy change of reaction ($\Delta_r G_m(T)$) and chemical reaction equilibrium constant ($K^\ominus$) of Eq. (7) were obtained. The thermodynamic data of related substances in this research were given in Table II.[30]

TABLE II. Standard formation enthalpy, standard formation Gibbs function, standard entropy and calorific capacity

Substance	$\Delta_f H_m^\ominus$ / kJ mol^{-1}	$\Delta_f G_m^\ominus$ / kJ mol^{-1}	$S_m^\ominus$ / J mol^{-1} K^{-1}	$C_p^\ominus$ / J mol^{-1} K^{-1}
HNO_3 (aq)	–207.36	–111.34	146.4	–86.6
$NaClO_3$ (aq)	–344.09	–269.91	221.3	111.3
NaCl (aq)	–407.27	–393.17	115.5	–90.0
NO (g)	91.29	87.60	210.76	29.85
H_2O (l)	–285.83	–237.14	69.95	75.35

$\Delta_r H_m(T)$ at different temperatures have been calculated based on the Eqs. (13) and (14):

$$\Delta_r H_m(T) = \sum \gamma \Delta_f H_m^{\ominus}(\text{products}) - \sum \gamma \Delta_f H_m^{\ominus}(\text{reactants}) \qquad (13)$$

$$\Delta_r H_m(T) = \Delta_r H_m^{\ominus} + \int_{289.15}^{T} \Delta_r C_{p.m} dT \qquad (14)$$

$$\Delta_r C_{p.m} = \sum \gamma C_{p.m}^{\ominus}(\text{products}) - \sum \gamma C_{p.m}^{\ominus}(\text{reactants}) \qquad (15)$$

The results of Eq. (14) were $\Delta_r H_m^{\ominus} = -374.65$ kJ/mol, $\Delta_r H_m$ (313.15 K) = = –382.29 kJ/mol and $\Delta_r H_m$ (353.15 K) = –402.68 kJ/mol.

The results show that $\Delta_r H_m^{\ominus}$, $\Delta_r H_m$ (313.15 K) and $\Delta_r H_m$ (353.15 K) values of the chemical reaction are negative, which indicates that the reaction between NO and $NaClO_3$ is exothermic. Therefore, the chemical equilibrium for this reaction is unfavourable at high temperatures from the thermodynamics point of view. However, the reaction rate is generally affected by the temperature, and the reaction rate increases at high temperatures. The effect of temperature on the removal efficiency of NO showed there was an optimal reaction temperature at which NO could be removed quickly. This was the result of a combination of chemical thermodynamics and kinetics. $\Delta_r G_m(T)$ at 298.15, 313.15 and 353.15 K was calculated by Eqs. (16) and (17):

$$\Delta_r G_m^{\ominus} = \sum \gamma \Delta_f G_m^{\ominus}(\text{products}) - \sum \gamma \Delta_f G_m^{\ominus}(\text{reactants}) \qquad (16)$$

$$\Delta_r G_m(T) = \Delta_r H_m^{\ominus} - T \Delta_r S_m^{\ominus}(T) =$$
$$= \Delta_r H_m^{\ominus} - T \Delta_r S_m^{\ominus} 298.15 - T \int_{298.15}^{T} \frac{\Delta_r C_{p.m}}{T} dT \qquad (17)$$

The results of Eq. (17) were $\Delta_r G_m^{\ominus} = -284$ kJ/mol, $\Delta_r G_m$ (313.15 K) = = –279.33 kJ/mol and $\Delta_r G_m$ (353.15 K) = –267.21 kJ/mol.

The chemical reaction equilibrium constant ($K^{\ominus}$) of Eq. (7) was given as follows:

$$\Delta G^{\ominus} = -RT \ln K^{\ominus} \qquad (18)$$

The results of Eq. (18) for $K^{\ominus}$ are $10^{49.78}$, $10^{46.60}$ and $10^{39.52}$.

The results showed that $\Delta_r G_m^{\ominus}$, $\Delta_r G_m$ (313.15 K) and $\Delta_r G_m$ (353.15 K) of the chemical reaction were high negative values, compared to –40 kJ/mol, and $K^{\ominus}$ values calculated were far greater than 10^5.[31] From these thermodynamic derivations, we can suggest that this oxidation reaction can proceed spontaneously and the reaction is very thorough. In the actual operation, the reason why the denitration rate did not reach 100 % should be that the residence time was short, resulting in insufficient reaction.

CONCLUSIONS

$NaClO_3$ is a promising absorbent for the removal of NO from flue gas. The denitration efficiency increased with temperature from 30 to 80 °C in $NaClO_3$//H_2SO_4 system. However, for $NaClO_3$/HNO_3 system, the denitration efficiency decreased with temperature from 40 to 80 °C. The NO removal efficiency increased with $NaClO_3$ and acid concentrations. The optimal mole ratio of $NaClO_3$ to H^+ was 1:12.76. The NO removal efficiency was 91.86 %. The cost of $NaClO_3$ process was about a quarter of the $NaClO_2$ wet denitration technology. The reaction product was NaCl and could be recovered. Therefore, $NaClO_3$ method may be an alternative way to solving the problem of denitration of dusty flue gas small boilers produced intermittently.

ИЗВОД

СТУДИЈА О УКЛАЊАЊУ NO ИЗ ЕМИСИЈЕ МОКРИМ СКРУБИНГОМ ПОМОЋУ NaClO₃

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За уклањање азот-моноксида (NO) из гасне емисије малих котлова на угаљ, неопходно је употребити економичну технологију влажне денитрације. Проучавана је апсорпција NO помоћу раствора натријум-хлората. Установљени су утицаји експерименталних услова као што су температура, концентрација NaClO₃, врста киселине, молски однос NaClO₃ према водоничним јонима – на брзину уклањања NO, и утврђени оптимални услови. Како је утицај температуре био повезан са врстом киселине, температура потребна за сумпорну киселину (H₂SO₄) била је висока, а температура потребна за азотну киселину (HNO₃) ниска. Оптимални молски однос NaClO₃ према овим двема киселинама био је исти. Продукти реакције анализирани су јонском хроматографијом. Реакциони раствор се могао рециклирати након уклањања натријум-хлорида. Изведен је закључак о механизму и укупној једначини реакције NaClO₃ денитрације. Добијени термодинамички резултати показали су да је ова оксидациона реакција спонтана и врло темељна. NaClO₃ је показао високу ефикасност уклањања NO, а трошак денитрације је био много нижи него помоћу натријум-хлорита.

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