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Dependence of N₂O/NO Decomposition and Formation on Temperature and Residence Time in Thermal Reactor

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Abstract: Nitrogen dioxide (N₂O) is a greenhouse gas that is harmful to the ozone layer and contributes to global warming. Many other nitrogen oxide emissions are controlled using the selective non-catalytic reaction (SNCR) process, but N₂O reduction methods are few. To avoid future air pollution problems, N₂O reduction from industrial sources is essential. In this study, a N₂O decomposition and NO formation under an argon atmospheric N₂O gas mixture were observed in a lab-scale SNCR system. The reaction rate and mechanism of N₂O were calculated using a reaction path analyzer (CHEMKIN-PRO). The residence time of the gas mixture and the temperature in the reactor were set as experimental variables. The results confirmed that most of the N₂O was converted to N₂ and NO. The change in the N₂O reduction rate increased with the residence time at 1013 and 1113 K, but decreased at 1213 K due to the inverse reaction. NO concentration increased with the residence time at 1013 and 1113 K, but decreased at 1213 K owing to the conversion of NO back to N₂O.

Keywords: nitrous oxide (N₂O); nitric oxide (NO_x); argon (Ar) ambient; thermal decomposition; residence time; rate of progress; GRI-Mech 3.0

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1. Introduction

Nitrous oxide (N₂O), carbon dioxide (CO₂), and methane (CH₄) are considered greenhouse gases. N₂O is very stable in air, and takes an average of 135 years to decompose naturally. Upon reaching the stratosphere, N₂O reacts with an oxygen atom (O) to form nitrogen monoxide (NO). NO reacts with ozone (O₃) again in the ozone layer and destroys O₃ in a chain [1,2]. Therefore, N₂O has 310 times higher global warming potential (GWP) than CO₂.

N₂O was designated as a greenhouse gas by the Kyoto Protocol in 1997. Afterward, N₂O was regulated as a greenhouse gas under the Paris Agreement adopted at the UN Climate Change Conference in 2015. However, in most countries, including South Korea, specific reduction policies for N₂O are still inadequate [3].

N₂O reduction methods can be divided into two methods. The first is the inhibition of N₂O production. The second is N₂O removal at the end of the exhaust [4–6]. In the first method, N₂O generation can be suppressed using a fluid medium in the fluidized bed combustion process (FBC) and by using a catalyst mounted on a generation source such as a vehicle [4–6]. However, this method is greatly affected by the N₂O temperature and pressure conditions. The second method involves reducing the N₂O generated in the incinerator and the industrial process at a subsequent stage [4–6]. This technology of N₂O reduction at the end of the process is largely divided into selective catalytic reduction (SCR) and selective non-catalytic reduction (SNCR). The SCR process uses a catalyst, so the facility can be operated at a lower temperature than that required for SNCR. The SNCR

process operates at a relatively high temperature (approximately 1000–1500 K) compared with the SCR process, and the initial investment cost is lower than other processes [4–8].

The SNCR process is a NO_x reducing process, where the NO_x generated in the combustion process of an incinerator is reduced using a reductant. Specifically, a reductant such as ammonia (NH₃) and urea aqueous solution (NH₂CONH₂) is injected into the chamber at temperatures of 1173–1373 K, thereby reducing NO_x to nitrogen (N₂) and water vapor [9]. In general, NH₃ is more efficient than the urea aqueous solution, but urea is easier to handle and results in a less costly process [10,11].

Figure 1 shows the decomposition mechanism and reaction pathway of NH₃ and urea aqueous solution in the SNCR process [12]. In the process, the Zel'dovich mechanism is used as the basic reaction pathway for oxidation and deoxidation of N₂ [13].

Figure 1. Basic decomposition mechanism of nitrous oxide using ammonia and urea aqueous solution reductants [13].

In a SNCR process that uses urea aqueous solution as a reductant, NO_x is reduced to N₂ and N₂O [14]. Svoboda et al. reported that the reductant employed in the process affects the production of N₂O [15]. Moreover, Kim reported that a small amount of N₂O was generated when urea aqueous solution was injected for NO_x removal in the operation of a waste incinerator [16].

Carbon monoxide (CO) and oxygen (O₂) are typical reaction gases that affect the SNCR process. In the oxygen-free process, the NO_x reducing reaction occurs at temperatures of 1400 K or higher. This results from the NO reduction proceeding at a relatively high temperature due to the low OH concentration, thereby leading to an increase in the reaction temperature. Im reported that the optimal O₂ concentration in the SNCR process is 5–7% [17].

In a study on the residence time, Liang et al. reported that the optimum temperature for N₂O decomposition and NO_x formation decreased with increasing residence time of the mixture [18]. Various studies considering the dependence of NO reduction on the temperature and residence time have used ammonia as a reductant in the SNCR process [19–22]. Duo et al. reported that the optimum temperature for NO reduction decreased with increasing residence time [23].

Previous studies have confirmed that the reduction efficiency of N₂O is determined by complex factors such as the amount of reductant, mixture composition, and residence time. Most of those studies focused on controlling the amount of N₂O generated during the NO_x reduction process in the SNCR. Additionally, studies of N₂O decomposition have mostly used catalysts. [24–26]. In the SNCR system, N₂O is less regulated than NO_x; hence, the reduction of N₂O has rarely been investigated. Therefore, in this study, we used a laboratory-scale thermal reactor that simulated an actual SNCR system, and observed the N₂O and NO behaviors that were generated in combustion furnace.

The reaction characteristics of N_2O and NO were studied through thermal decomposition experiments, where the temperature and residence time in an argon (Ar) atmosphere gas mixture were varied. Furthermore, the reaction equation and reaction rate (rate of progress) of N_2O and NO were examined using a chemical reaction program (CHEMKIN-PRO) that simulated the same conditions as the experiment. Correlations between the reduction of N_2O and NO, and the reaction rate based on the temperature and residence time, were investigated.

2. Experimental Methods

2.1. Experimental Setup

Figure 2 shows a schematic of the experimental setup used to investigate the N_2O thermal decomposition characteristics. The setup consisted of a gas inlet part, a thermal reactor, a measuring part, and an exhaust port. The flow of experimental gases Ar, O_2 , and N_2O into the thermal reactor were controlled using a mass flow controller (MFC). The reactor had a cylindrical structure (inner diameter: 140 mm, and length: 1000 mm), was an electric furnace capable of heating up to 1273 K, and was heated with four internal heating coils. The temperature inside the reactor was measured using an R-type thermocouple placed in the center of each coil. After the thermal reaction, the exhaust gas passed through a heat exchanger. A non-dispersive infrared (NDIR) type gas meter (Sensonic IR-1, for the N_2O measurement) and a gas analyzer (MK 6000+, com GmbH, for the NO_x and O_2 measurement) were used to measure the gas components. The measurement error of the NDIR gas meter (equipped with an electrochemical sensor) was 3%. The measurement error of NO and N_2O of the gas analyzer (also equipped with an electrochemical sensor) was 2% [27].

Figure 2. A scheme of the experimental setup [27].

Figure 3 shows a photograph of a laboratory-scale SNCR apparatus for N_2O thermal decomposition. To prevent heat loss and to maintain the temperature employed in the experimental conditions, the reactor was surrounded by an insulator.

Figure 3. The laboratory scale selective non-catalytic reaction (SNCR) system.

2.2. Experimental Conditions

Table 1 shows the composition of the gas mixture used in the N_2O thermal decomposition experiment [27]. The gas mixture was composed of 94.96% Ar , 5% O_2 , and 400 ppm of N_2O to simulate the exhaust gas in a selective non-catalytic reduction process. The actual combustion exhaust gas is N_2 and CO_2 atmosphere. However, in this experiment, an Ar atmosphere was employed to minimize the involvement of nitrogen components from sources, in addition to the N_2 generated by thermal decomposition of N_2O . Moreover, the molecular weights of N_2O and CO_2 were the same, and hence measurement errors occurred in the NDIR measurement. Therefore, no hydrocarbon-based material was added. The chemical reaction time of NO_x and N_2O depends on the residence time and temperature. Accordingly, the residence time of the gas mixture in the thermal reactor was selected as an experimental variable. The dependence of the N_2O thermal decomposition on the residence time was investigated by fixing the composition of the gas mixture and varying the residence time (times of 10, 20, and 40 s were employed). The gas mixture flow rate corresponding to each residence time was calculated from the equation of the reactor volume and residence time (Equation (1)). In this equation, Q is the gas mixture flow rate, D is the inner diameter of the reactor, L is the length of the reactor, and t is the residence time.

$$Q = \frac{\pi \times (D/2)^2 \times L}{t} \quad (1)$$

Table 2 shows the experimental conditions [27]. The experimental temperature was increased in 20 K intervals for temperatures ranging from 1023 to 1223 K, which is the general operating temperature of the SNCR process. In order to maintain the residence time of the gas mixture on each temperature section, the flow rate was adjusted based on Charles's law, which is given as follows:

$$\frac{Q_1}{T_1} = \frac{Q_2}{T_2} \quad (2)$$

where Q_1 is the inlet flow rate, T_1 is the inlet temperature, Q_2 is the flow rate for each residence time, and T_2 is the reactor temperature. The gas concentration was measured in 1 min intervals after 10 min of supplying the gas mixture. For gas measurements, the concentration of exhaust gas from the rear end of the reactor was measured. The measured data are reported as the average value of five measurements performed for each experimental condition. Laboratory ambient conditions were maintained at atmospheric air (298 K, 1 atm).

Table 1. Test gas condition [27].

Residence Time (s)	Gas Composition
10, 20, 40	Ar (94.96%), O ₂ (5%), N ₂ O (400 ppm)

Table 2. Experiment conditions [27].

Condition	Value
Temperature (K)	1013–1213
Flow Rate (L/min)	5.44–25.81
Ambient Temperature (K)	298
Ambient Pressure (atm.)	1

2.3. CHEMKIN Calculation Condition

CHEMKIN-PRO [28] calculations were performed by simulating the thermal reactor used in the N₂O thermal decomposition experiment. The 1D reaction structure analysis plug flow reactor model, a cylindrical reactor model that assumes a steady state during flow, was used for the calculations. The input gas was assumed to be perfectly mixed, and continuous chemical reactions inside the reactor were observed. The model of a reaction furnace with an 140 mm inner diameter and a 1000 mm length was built, which was the same as the actual experimental apparatus. The temperature of the thermal reactor was divided into 11 intervals, ranging from 1023 to 1223 K. The residence time of the gas mixture was calculated in the same manner as the experimental conditions at 10, 20, and 40 s. Furthermore, the N₂O chemical reaction (see Table 3) was calculated from the chemical reaction equation included in GRI 3.0 [29], and the chemical reaction route was confirmed via reaction path analysis.

Table 3. GRI-Mech 3.0.

Reaction		k = AT ^b exp(−E/RT)		
		A (cm ³ /gmol)/s	b	E (kJ/gmol)
N ₂ O(+M) <=> N ₂ +O(+M)	(1)	7.9 × 10 ¹⁰	0	56,020
N ₂ O+O <=> N ₂ +O ₂	(2)	1.4 × 10 ¹²	0	10,810
N ₂ O+O <=> 2NO	(3)	2.9 × 10 ¹³	0	23,150
NO ₂ +O <=> NO+O ₂	(4)	3.9 × 10 ¹²	0	−240.00
NO+O+M <=> NO ₂ +M	(5)	1.06 × 10 ²⁰	−1.41	0
N+NO <=> N ₂ +O	(6)	2.7 × 10 ¹³	0	355
N+O ₂ <=> NO+O	(7)	9.0 × 10 ⁹	1	6500

3. Results and Discussion

3.1. Formation of Nitrogen and Nitrogen Monoxide by Thermal Decomposition of Nitrous Oxide

Figure 4 shows the gas concentration calculated using CHEMKIN-PRO. Calculation conditions of residence time: 1 s, gas composition: 99.96% Ar, 400 ppm N₂O, and reactor temperature: 1020–1220 K, were employed. The results confirmed that the concentration of N₂O in the reactor generally decreased with increasing temperature. In the initial temperature condition (1013 K), the N₂O concentration was 397 ppm. Concentrations of 375 and 253 ppm occurred at 1113 and 1213 K, respectively. The concentration of N₂ was 2 ppm under the initial temperature conditions. The concentration of N₂ increased from 23 ppm at 1113 K to 138 ppm at 1213 K. This resulted from N₂O thermally decomposing into two N and one O (see Reaction (1) of Table 3) with increasing reactor temperature. The decomposed two N collided with each other, thereby generating an activation energy, and stable N₂ was formed. NO was not formed under the initial temperature condition of 1013 K, but ~8 ppm formed when the temperature increased to 1213 K. This resulted from

N₂O and N being converted into NO (see Reactions (3) and (7) of Table 3). Therefore, with the reduction of N₂O, a small amount of NO was generated.

Figure 4. Dependence of the calculated gas concentration on the temperature.

3.2. Dependence of Nitrous Oxide Reduction Rate on the Residence Time

Figure 5 shows the dependence of the N₂O reduction rate on the residence time in the reactor. The rate is expressed as the ratio of the inlet N₂O concentration (400 ppm), and the reduced N₂O concentration after the reaction, i.e.,

$$N_2O \text{ Reduction}(\%) = \frac{\text{Reduced } N_2O \text{ Concentration}}{\text{Inlet } N_2O \text{ Concentration}} \times 100 \quad (3)$$

The gas mixture composition was 94.96% Ar, 5% O₂, and 400 ppm N₂O. Residence times of 10, 20, and 40 s, and reaction temperatures of 1013, 1113, and 1213 K were employed. For each residence time, the N₂O reduction rate increased with increasing reaction temperature because both variables (i.e., the time and the temperature) affect the forward reaction of the N₂O thermal decomposition process. However, in an actual reactor, increasing both variables infinitely is inefficient. Therefore, determining the relationship between the residence time and the reaction temperature and finding an optimum condition between the two variables are essential.

Figure 5. Dependence of N₂O reduction on the residence time.

Table 4 shows the N_2O reduction rate in each temperature section of the reactor based on the change in residence time shown in Figure 5. In residence time change Section 1, the change in percentage of the reduction rate increased linearly from 9.75% to 21.25% with increasing temperature in the reactor. However, the change in percentage decreased from 27.5% to 15.5% when the temperature increased from 1113 to 1213 K in change Section 2.

Table 4. Reduction rate of N_2O depending on change in residence time and reactor temperature.

s \ T	1013 K	1113 K	1213 K
Residence time Change Section 1 (10 s $\rightarrow$ 20 s)	9.75 %	16 %	21.25 %
Residence time Change Section 2 (20 s $\rightarrow$ 40 s)	21.25 %	27.5 %	15.5 %

Figures 6 and 7 show the calculation results obtained from the reaction path analyzer. These results explain the trends observed in Figure 5 and Table 4. The reaction rate of conversion from N_2O to N_2 based on the residence time was calculated with CHEMKIN-PRO. Table 5 shows the specific reaction rate (rate of progress) values plotted in Figure 6.

Figure 6. Rate of progress from N_2O to N_2 ($\text{M}/\text{cm}^3\text{s}$) at 1013 and 1113 K.

Figure 7. Rate of progress from N_2O to N_2 ($\text{M}/\text{cm}^3\text{s}$) at 1213 K.

Table 5. Rate of progress between N₂O and N₂.

$\begin{matrix} \text{K} \\ \text{s} \end{matrix}$	10	20	40
1013 K	Forward reaction rate: 4.6×10^{-11} Reverse reaction rate: -1.54×10^{-17}	Forward reaction rate: 3.64×10^{-11} Reverse reaction rate: -2.32×10^{-17}	Forward reaction rate: 2.37×10^{-11} Reverse reaction rate: -2.74×10^{-17}
1113 K	Forward reaction rate: 7.52×10^{-11} Reverse reaction rate: -2.14×10^{-16}	Forward reaction rate: 1.89×10^{-11} Reverse reaction rate: -7.94×10^{-17}	Forward reaction rate: 1.21×10^{-11} Reverse reaction rate: -5.42×10^{-17}
1213 K	Forward reaction rate: 6.62×10^{-13} Reverse reaction rate: -4.1×10^{-17}	Forward reaction rate: 1.49×10^{-15} Reverse reaction rate: -3.63×10^{-17}	Forward reaction rate: 5.29×10^{-17} Reverse reaction rate: -3.63×10^{-17}

As shown in Figure 6, the reaction rate of the N₂O decomposition decreased with increasing residence time (see Reactions (1) and (2) of Table 3). This resulted from the chemical reaction rate being related to the concentration of the reactants. The reaction time of the N₂O introduced in the reactor increased with increasing residence time, and hence the concentration of exhausted N₂O after the reaction decreased. When the N₂O concentration decreased, the number of molecules per unit volume in the reactor and, consequently, the collisions between these molecules decreased. Therefore, the number of chemical reactions and the reaction rate were reduced, leading to an increase in the N₂O reduction rate with increasing residence time and temperature, as shown in Figure 5.

Figure 7 shows that the forward reaction rate decreased with increasing residence time (see Reaction (1) of Table 3). In addition, the inverse reaction rate, corresponding to the formation of the N₂O, appeared with increasing residence time at 1213 K. Accordingly, the N₂O reduction rate decreased significantly since N₂ and O recombined to form N₂O again. This observation was consistent with the previous experimental result.

3.3. Nitrogen Monoxide Concentration According to Residence Time

Figure 8 shows the dependence of the NO concentration on the residence time. The gas composition and the experimental conditions are the same as those shown in Figure 5. The results confirmed that for a given residence time, the amount of NO formed increased with increasing temperature. This resulted from NO forming via thermal decomposition of N₂O (see Reaction (3) in Table 3). At a reactor temperature of 1013 K, only a small amount of NO formed at residence times of 10 and 20 s, but the amount increased at 40 s. Increasing the residence time at a reactor temperature of 1113 K yielded only a slight increase in the NO concentration. However, at a reactor temperature of 1213 K, the NO concentration decreased with increasing residence time.

Figure 8. Dependence of the NO concentration on the residence time.

Figures 9 and 10 show the calculated reaction rates of conversion from N_2O to NO as determined by the CHEMKIN-PRO reaction path analyzer. The calculation conditions were the same as those employed in the experiment. The specific reaction rate values are shown in Table 6.

Figure 9. Rate of progress from N_2O to $N_2(M/cm^3s)$ at 1013 and 1113 K.

Figure 10. Rate of progress from N_2O to $NO(M/cm^3s)$ at 1213 K.

Table 6. Rate of progress between N_2O and NO.

K \ s	10	20	40
1013 K	Forward reaction rate: 2.38×10^{-12} Reverse reaction rate: -1.23×10^{-21}	Forward reaction rate: 1.63×10^{-12} Reverse reaction rate: -3.47×10^{-21}	Forward reaction rate: 8.01×10^{-13} Reverse reaction rate: -7.56×10^{-21}
1113 K	Forward reaction rate: 2.35×10^{-12} Reverse reaction rate: -3.0×10^{-19}	Forward reaction rate: 1.92×10^{-13} Reverse reaction rate: -3.39×10^{-19}	Forward reaction rate: 2.33×10^{-15} Reverse reaction rate: -3.43×10^{-19}
1213 K	Forward reaction rate: 7.45×10^{-16} Reverse reaction rate: -4.98×10^{-18}	Forward reaction rate: 1.48×10^{-18} Reverse reaction rate: -4.98×10^{-18}	Forward reaction rate: 5.27×10^{-20} Reverse reaction rate: -4.98×10^{-18}

At reaction temperatures of 1013 and 1113 K, the reaction rate decreased with increasing residence time. Times of 10 and 20 s yielded only slight differences in the reaction rate, but a time of 40 s led to a significant decrease in the reaction rate. Therefore, the concentration of NO at a residence time of 40 s was higher than that at 10 and 20 s, indicating that more N_2O was converted into NO. This result corresponds to the change in NO concentration shown in Figure 8, where the experimental results are presented.

As shown in Figure 10, the overall reaction rate was further reduced when the temperature increased. This confirmed that the forward reaction rate decreased with increasing

residence time, whereas the inverse reaction rate remained the same (see Reaction (3) in Table 3). Therefore, at a temperature of 1213 K, the inverse reaction rate appeared with increasing residence time and NO was converted back to N₂O; hence, the NO concentration decreased at a time of 40 s.

4. Conclusions

In this study, N₂O reduction and NO formation were observed through thermal decomposition experiments performed in a high-temperature reactor. The dependence of the chemical reaction on the residence time and temperature was confirmed by comparing the calculated reaction equation and the reaction rate with experimental results. The major findings of this work are summarized as follows:

1. The results confirmed that most of the N₂O was converted to N₂ and a small amount of NO via thermal decomposition in Ar atmosphere due to the high operating temperature of SNCR. Therefore, the thermal decomposition temperature must be controlled appropriately to prevent the generation of NO.
2. The change in the N₂O reduction rate at temperatures of 1013 and 1113 K increased with the residence time, but decreased at 1213 K and a time of 40 s. This resulted from the inverse reaction rate and the regeneration of N₂O at 1213 K. Therefore, exaggerated residence times and thermal decomposition temperatures are inefficient in terms of the energy consumption compared to the reduction of N₂O.
3. The NO concentration increased with residence time at temperatures of 1013 K and 1113 K, but decreased with the time at 1213 K. This resulted from the inverse reaction rate associated with the conversion of NO back to N₂O. Therefore, the optimal operating conditions for the highest reduction efficiency must be studied.

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