

A Study on the NO Treatment using Dry Oxidant Produced by H_2O_2

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Abstract

In this study, the NO oxidation using dry oxidant produced by catalytic H_2O_2 conversion was conducted. It was shown that Mn-based Fe_2O_3 support catalyst has the best performance in the catalytic H_2O_2 conversion and it's combined-NO oxidation. The reaction characteristics of NO oxidation was investigated by the various operation conditions such as H_2O_2 amount, oxidation temperature and space velocity. As a results, the oxidation efficiency of NO greatly depends on the oxidation reaction temperature, H_2O_2 amount and space velocity. The performance of NO oxidation was increased with increasing the oxidation temperature and H_2O_2 amount. Also performance of NO oxidation was decreased with increasing the space velocity.

Keywords: H₂O₂; Catalytic conversion; NO oxidation; Dry oxidant; Operation conditions

Introduction

 NO_X generated from the internal combustion engine is a representative air pollutant. Various studies have been conducted to effectively remove NO_X , and selective catalytic reduction (SCR) has been widely used [1].

The SCR process is disadvantageous in that the use temperature of the catalyst for reducing NO_X is high and the scale of the equipment is increased according to the applied environment and the process becomes complicated. Therefore, it is disadvantageous in that it is lowered in consideration of economic efficiency due to high complexity of process, enlargement of equipment, and installation and operation cost for operation of established equipment.

The reason why high temperature is required to operate the SCR process is that there is NO which is difficult to convert to NO_X , and a process that can economically convert it into a process able material such as NO_2 is needed. In order to convert NO, a very strong oxidizing agent is required, and it has been confirmed through previous studies that a dry oxidizing agent having a strong oxidizing power can be produced when H_2O_2 is converted on a heterogeneous transition metal catalyst [2-5]. The H_2O_2 conversion process using a transition metal catalyst can effectively produce a dry oxidant even at low temperatures, and it has been confirmed that high oxidation efficiency can be obtained when it is applied to the NO oxidation process [2,5-9].

 H_2O_2 catalyzed dry oxidant formation reaction and the NO oxidation reaction formula are shown in Equation (1)-(5) [10-14]. In this study, to convert NO gas contained in flue-gas, H_2O_2 was converted to a dry oxidant on various catalysts in order to evaluate the effect on NO oxidation, Characteristics the catalytic performance of the prepared catalysts was evaluated through the conversion of H_2O_2 . NO conversion characteristics were also observed depending on the feed rate, space velocity, reaction temperature, NO concentration and ratio of each reactant.

 $H_2O_2+M(I)$ →•OH+•OH (1) NO+•OH→NO₂+HNO₂ (2) NO+•OH→NO₂+H (3) NO+•OH→HNO₃ (4) NO₂+•OH→NO₃⁻+H³ (5)

Experimental Method

Catalyst production

The catalyst used in this experiment was manufactured on a laboratory scale and is shown in Table 1. The prepared catalyst can be classified into a Mn-based catalyst and a Zr-based catalyst. Mn (NO₃)₂ (reagent grade, Aldrich Co., 98% or more) was used as a precursor of Mn-based catalyst, and γ -Al₂O₃ and Fe₂O₃ were used as support. A certain amount of precursor was dissolved in distilled water in a beaker and the carrier was mixed at a certain ratio [2]. Thereafter, the precipitation process was carried out by injecting NH₄OH solution which is a precipitant until the pH value of the slurry mixture prepared above reached a range between 9 and 10.

The slurry mixture was aged at about 110 $^{\circ}$ C for about 4 hours under agitation, and dried at 110 $^{\circ}$ C for 12 hours and then fired at about 600 $^{\circ}$ C for 4 hours. N₂ was injected at 500 mL/min to maintain the inert atmosphere in the firing process. The SO₄₂⁻/ZrO₂ catalyst was prepared by precipitating Zr(OC₃H₇) 4(reagent grade, Aldrich Co., 98% or more) with ammonia water and drying at 110 $^{\circ}$ C for 12 hours.

The dried precipitate was mixed with 0.5 $\rm NH_2SO_4$ Aged for 12 hours, dried at 105 °C for 12 hours and then calcined at 500 °C. The catalytic performance of the catalysts was investigated by pulverizing the solid product obtained by the two methods and using the particles in the range of about 100-150 through the sieving process (Figure 1 and Table 1).

	Α	В	C	D	E
Catalyst	Mn/Fe ₂ O ₃	70 wt % Mn/Al ₂ O ₃	10 wt % Mn/Al ₂ O ₃	Fe ₂ O ₃	SO ₂ -/ZrO ₂

Table 1: Preparation of the various catalysts for the catalytic H₂O₂ conversion.

Laboratory scale NO oxidation characterization

Experiments were conducted to investigate the NO conversion characteristics of the dry oxidant produced through the H2O2 conversion reaction on the solid phase catalyst respectively. The experimental setup consisted of a fixed-bed catalytic reactor which converted H₂O₂ on the catalyst to produce a dry oxidant and an oxidation process which oxidized NO by the injection of the generated dry oxidant. The fixed-bed catalytic reactor was a stainless steel tubular reactor with an outer diameter of 1/2 inch (H₂O₂ injection amount of 0.3 g/min) and the catalyst was filled with 0.5 g of the catalyst at the center of the reactor. The temperature of the reactor was controlled using an automatic temperature controller after locating the thermocouple (K-type) connected to the electric furnace at the same point as the catalyst filling layer. H₂O₂ was injected into the catalytic reactor with a metering pump to produce a dry oxidant, and a 1/4 inch stainless steel tube between the metering pump and the catalytic reactor was heated to 150 °C.



Figure 1: Schematic diagram of the experimental setup for the catalytic H_2O_2 conversion and NO oxidation.

The raw materials used for the production of dry oxidants were aqueous H_2O_2 solutions (Duksan Co., 30%), which are commercially available as generic reagent grade. For the NO conversion experiment using the generated dry oxidant, the reactor was constructed as follows.

The reactor was made of stainless steel having an outer diameter of 1/2 inch and a length of about 50 cm, and an external heater was installed to control the reaction temperature.

The temperature of the heater was adjusted by placing a thermocouple (K type) in the reactor corresponding to the center of the reactor and then using an automatic temperature controller. As a reactant, NO was injected to simulate NO in the flue gas.

The flow rate of the gas was adjusted by the corrected mass flow controller (Brooks MFC 5850E) to the $[H_2O_2]/[NO]$, And the basic reaction conditions are shown in Table 2. The concentration of NO_X before and after the reaction was analyzed in real time using a gas analyzer (Greenline 9000, Eurotron). NO conversion rates and the like were calculated using the equation (6) before and after the reaction (Table 2).

Factors	Values	
Temperature	40-200 °C	
Space Velocity	5,000-30,000 cm ³ /g-cat.h	
[H ₂ O ₂]/[NO]	1.0-3.5	
NO concentration	1,000-5,000 ppm(Balance gas: N ₂)	

Table 2: Basic reaction conditions.

NO Conversion (%)= $[NO]_{in}$ - $[NO]_{out}/[NO]_{in} \times 100$

Results and Discussion

Evaluation of applicability of catalyst for H₂O₂ Conversion

The characteristics of the NO oxidation reaction on the oxidation process which can be connected at the downstream are investigated by using the dry oxidant generated through the H_2O_2 conversion reaction. Figure 2 shows the NO conversion rate by simulating the dry oxidant obtained after the H_2O_2 conversion reaction performed according to the type of the produced catalyst and simultaneously injecting it with the exhaust gas.

The reaction temperature was controlled to 150 °C, and the amount of H_2O_2 injected and the amount of catalyst charged were 0.3 g/min and 0.5 g, respectively, in the H_2O_2 conversion process. The oxidation process also controlled the reaction temperature to 150 °C, and the flow rate of the injected simulated flue gas and the NO concentration were 300 mL/min and 1000 ppm, respectively.

The conversion efficiency of NO in the oxidation process was different depending on the kind of the catalyst, and the NO conversion rate varied depending on the performance of the catalyst obtained in the H_2O_2 conversion process.

As a result, the catalysts with the highest NO conversion efficiency were Mn/Fe_2O_3 , 70 wt % Mn/Al_2O_3 catalyst in the oxidation process of each type of catalyst used in the H_2O_2 conversion process, and the NO conversion rate obtained in the oxidation process reached about 100% (Figure 2).

The NO conversion rates of the catalysts used in the H_2O_2 conversion process were $Mn/Fe_2O_3 \approx 70$ wt.% $Mn / Al_2O_3 >> 10$ wt % $Mn/Al_2O_3 > Fe_2O_3 > SO_4^{2-}/ZrO_2$. In the combined operation of the H_2O_2 conversion process and the oxidation process the NO concentration in the simulated exhaust gas injected into the oxidation process was increased to about 5000 ppm and the efficiency of the combined operation and the oxidation process was investigated (Figure 3).

The NO conversion efficiency was investigated by injecting the dry oxidant obtained after the H_2O_2 conversion reaction performed according to the catalyst type into the oxidation process simultaneously with the exhaust gas.



Figure 2: Effect of the various catalysts on the NO oxidation (A: Mn/Fe_2O_3 , B: 70 wt % Mn/Al_2O_3 , C: 10 wt % Mn/Al_2O_3 , D: Fe_2O_3 , E: SO_4^{2-}/ZrO_2 , NO concentration: 1,000 ppm).

The reaction temperature was controlled at 150 °C and the amount of H₂O₂ injected and the amount of catalyst charged were 0.3 g/min and 0.5 g, respectively, in the H₂O₂ conversion process. The oxidation process also controlled the reaction temperature to 150 °C, and the flow rate of injected simulated flue gas and NO concentration were 300 mL/min and 5000 ppm, respectively. The conversion efficiency of NO in the oxidation process was different depending on the kind of the catalyst, and the NO conversion rate varied depending on the performance of the catalyst obtained in the H₂O₂ conversion process. First, in the case of the combined operation of the oxidation process the NO conversion rate is slightly changed as the NO concentration in the simulated exhaust gas injected into the oxidation process increases from 1,000 ppm to 5000 ppm. The NO conversion efficiency of the catalysts used in the H₂O₂ conversion process was Mn/Fe₂O₃, and the conversion of NO obtained in the oxidation process reached about 100%. The NO conversion rates of the catalysts used in the H_2O_2 conversion process were in the order of Mn/Fe₂O₃ \approx 70 wt % Mn/ Al₂O₃>>10 wt % Mn/Al₂O₃> Fe₂O₃> SO₄²⁻/ZrO₂ (Figure 3).



Figure 3: Effect of the various catalysts on the NO oxidation (A: Mn/Fe_2O_3 , B: 70 wt % Mn/Al_2O_3 , C: 10 wt % Mn/Al_2O_3 , D: Fe_2O_3 , E: SO_4^{2-}/ZrO_2 , NO concentration: 5,000 ppm).

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Laboratory scale H₂O₂ conversion and NO oxidation process linked operation

Oxidation of NO by oxidation/production of dry oxidant was investigated in a laboratory scale H_2O_2 catalytic reaction using Mn/ Fe₂O₃ catalyst. In this process, the NO oxidation reaction characteristics after the laboratory scale H_2O_2 conversion to treat NO gas were investigated. In the oxidation process, the treatment capacity was less than about 1 L/min scale and the effect of oxidation rate on linear velocity, residence time, etc. In the H_2O_2 conversion reaction, the H_2O_2 feed rate was controlled by the flow rate and concentration of the NO gas supplied to the oxidation reaction.

The Mn/Fe₂O₃ catalyst which had the highest NO oxidation efficiency in the subsequent oxidation process, was selected using the dry oxidant produced according to the H₂O₂ conversion reaction characteristics according to the catalyst type, and the reaction temperature of the downstream oxidation process And the reaction characteristics were investigated. Figure 4 shows the NO conversion efficiency by simultaneously injecting the dry oxidant obtained after the H₂O₂ conversion reaction performed in accordance with the catalyst type into the oxidation process and injecting it together with the exhaust gas.

The reaction temperature was controlled at 150 °C and the amount of H_2O_2 injected and the amount of catalyst charged were 0.3 g/min and 0.5 g, respectively in the H_2O_2 conversion process. In the oxidation process, the reaction temperature was changed to 40-150 °C, and the flow rate of injected simulated flue gas and the NO concentration were 300 mL/min and 1000 ppm, respectively. The NO conversion efficiency varied depending on the reaction temperature of the oxidation process.

As the reaction temperature increased, the NO conversion rate was increased and the NO conversion rate reached about 100% at about 150 °C. Also, as the reaction temperature increases NO concentration which is the main conversion target gradually decreases, and the concentration of NO₂ which is the main product gradually increases.

As the reaction temperature increases, the NO conversion rate increases and the NO₂ selectivity also increases. As the reaction temperature increases the reactivity of the dry oxidant and NO as the main conversion material increases (Figure 4). Figure 5 controlled the amount of H_2O_2 fed to the H_2O_2 catalytic conversion reaction system for dry oxidant formation/feed while maintaining the NO concentration and flow rate constant.

The temperature condition in which the H_2O_2 conversion reaction in the H_2O_2 catalytic conversion system was stabilized was about 150 °C. In the NO oxidation process using the dry oxidant generated in the H_2O_2 conversion process, the reaction temperature was set at about 150 °C.

Through this process, the conversion rate of NO the gas to be treated, and the selectivity of NO₂ which can be generated mainly in the oxidation reaction, were investigated according to the amount of H_2O_2 injected. In addition, we tried to elucidate the factors and conditions maximizing the NO conversion and NO₂ selectivity by observing the changes of various conditions in the oxidation process due to the H_2O_2 dose. The flow rate of NO gas supplied to the oxidation process was about 300 mL/min and the concentration was about 1000 ppm (Figure 5).



The amount of H₂O₂ injected may be varied based on the amount of NO gas injected into the oxidation process, and the amount of NO gas may vary depending on the total amount and concentration of the injected exhaust gas. As the amount of H2O2 injected differs the amount of diluted H₂O₂ varies so the amount of residual steam which is a diluent may also vary. Figure 6 shows a typical correlation of factors influencing the oxidation process depending on the amount of H₂O₂ injected. The required amount of H₂O₂ converted into a dry oxidizing agent and the amount of steam supplied further by injecting diluted H₂O₂ In order to investigate the effect, the value is expressed as the ratio of the amount of total gas injected into the oxidation process and the amount of NO as the main target reactant $[H_2O_2(s)/flow$ rate of total gas] ratio to the total flue gas injected into the oxidation process varied from 0.3 to 0.9 g/L-Total, and the ratio of the amount of pure H₂O₂ in the diluted H₂O₂ aqueous solution to the total amount of exhaust gas changes from about 0.05 to 0.035 g/L-total. It can also be seen that the amount of water vapor remaining in the feeder chain relative to the total flue-gas injection volume increases from 0.2 to 0.6 g/L-total. When the diluted H2O2 supply amount, the pure H2O2 supply amount and the steam supply amount are shown as a ratio to the pure NO gas in the exhaust gas supplied to the oxidation process, as the diluted H₂O₂ supply increases from 0.045 to 0.3 mL/min, NO increased from about 188 to 1032 g/L-NO, and from 72 to 319 g/L-NO

increased from 144 to 741 g/L-NO. Therefore, it can be seen that various variables are changed according to the amount of NO gas contained in the exhaust gas as well as the total amount of exhaust gas injected into the oxidation process (Figure 6). Figure 7 shows the efficiency of the oxidation process obtained by varying the space velocity in the H_2O_2 conversion process.





The NO concentration in the oxidation process varied according to the space velocity in the H_2O_2 conversion process and the NO conversion rate. As the space velocity increased, the NO conversion rate in the oxidation process was reduced from about 91 to about 70%.

This is because as the space velocity is increased, the contact time between the injected H_2O_2 and the catalyst is reduced, so that the efficiency of generating the dry oxidant in the H_2O_2 conversion process and the supply efficiency to the oxidation process are reduced, and the oxidation reaction efficiency.

This is probably due to the decrease in as the space velocity in the H_2O_2 conversion process changes, the amount of H_2O_2 injected may be varied depending on the factors of the oxidation process and the amount of NO gas may vary depending on the total amount and concentration of the injected flue gas.

Since the amount of diluted H_2O_2 varies, the amount of steam, the diluent that is the residual material, may also vary. Figure 8 shows the effect of diluted H_2O_2 supplied by the oxidation process on the space velocity in the H_2O_2 conversion process and the amount of steam supplied by the addition of diluted H_2O_2 which is a value calculated for

the total amount of gas and the amount of NO as the main target reactant $[H_2O_2(s)/flow$ rate of total gas] ratio to the total flue gas injected into the oxidation process, and the diluted H_2O_2 .

The ratio of pure H_2O_2 in the aqueous solution to the total flue gas amount and the ratio to the amount of the residual gas vapor were all slightly reduced, but showed a slight change. When the injected diluted H_2O_2 aqueous solution feed rate, pure H_2O_2 feed rate, steam feed rate, and the like are expressed as the ratio of the pure NO gas in the exhaust gas supplied to the oxidation process, when the space velocity rises by the same variation width, 12 g/L-NO and 118 to 49 g/L-NO, respectively, from 173 to 94 g/L-NO.

Therefore, the H_2O_2 supply per unit time decreases as the space velocity increases in the H_2O_2 conversion process compared to the total amount of the exhaust gas injected into the oxidation process. Therefore, various variables are changed depending on the amount of NO_X gas supplied and the amount of NO_X gas supplied to the oxidation process (Figures 7 and 8).



Figure 7: Effect of the space velocity on the NO oxidation (H2O2 amount: 0.045 g/min, NO concentration 1,000 ppm).

Based on the above experimental results, it was found that the amount of H_2O_2 injected into the H_2O_2 conversion process was controlled at 0.3 g/min and the flow rate and concentration of the simulated flue gas injected into the oxidation process were maintained at 1 L/min and 1000 ppm. Figure 9 shows the NO concentration in the oxidation process, which was changed according to the space velocity in the H_2O_2 conversion process, and the NO conversion rate.

As the space velocity increased, the NO conversion rate in the oxidation process was reduced from about 100 to about 88%. This is because as the space velocity is increased, the contact time between the injected H_2O_2 and the catalyst is reduced, so that the efficiency of generating the dry oxidant in the H_2O_2 conversion process and the supply efficiency to the oxidation process are reduced, and the oxidation reaction efficiency.

This is probably due to the decrease in. However, it can be seen that the efficiency of the oxidation process is maintained at almost 100% under the condition of the space velocity of about 72,000 mL/g-cat.·h. Figure 10 shows the effect of diluted H_2O_2 supplied by the oxidation

process on the space velocity in the $\rm H_2O_2$ conversion process and the amount of steam supplied by the addition of diluted $\rm H_2O_2$.



Figure 8: Effect of the space velocity on the NO conversion and design factor (H2O2 amount: 0.045 g/min, NO concentration: 1,000 ppm).

Which is a value calculated for the total amount of gas and the amount of NO as the main target reactant. ($[H_2O_2(s)/flow$ rate of total gas] ratio to the total flue gas injected into the oxidation process, and the diluted H_2O_2 .

The ratio of pure H_2O_2 in the aqueous solution to the total flue gas amount and the ratio to the amount of the residual gas vapor gradually decreased from 0.98 to 0.42, from 0.36 to 0.21 and from 0.68 to 0.38 respectively.

When the injected diluted H_2O_2 aqueous solution feed rate, the pure H_2O_2 feed rate, the steam feed rate, and the like are expressed as the ratio to the pure NO gas in the exhaust gas supplied to the oxidation process, when the space velocity increases with the same variation width, 294 g/L-NO, 228 to 98 g/L-NO to 749 to 208 g/L-NO.

Therefore, the H_2O_2 supply per unit time decreases as the space velocity increases in the H_2O_2 conversion process compared to the total amount of the exhaust gas injected into the oxidation process. Therefore, various variables are changed depending on the amount of NOX gas supplied and the amount of NO_X gas supplied to the oxidation process (Figure 10).

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Figure 10: Effect of the space velocity on the NO conversion and design factor (H2O2 amount: 0.3 g/min, NO concentration: 1,000 ppm).

Based on the above experimental results, it was confirmed that the amount of H_2O_2 injected into the H_2O_2 conversion process was controlled at 0.3 g/min, and the concentration of the simulated flue gas injected into the oxidation process was maintained at 5000 ppm. Figure 11 shows the NO concentration and the NO conversion rate in the oxidation process changed according to the H_2O_2 conversion process and the oxidation process connection operation according to the space velocity change. As the space velocity increased from 36,000

to 84,000 mL/g-cat, the NO conversion rate in the oxidation process was reduced from about 100 to about 97%. This is because as the space velocity is increased, the amount of the dry oxidant supplied from the H₂O₂ catalyst conversion is kept constant, while the flow rate including NO to be treated is increased and the contact efficiency is decreased. This is because the oxidation efficiency of NO is decreased. However, it can be seen that the efficiency of the oxidation process is maintained at almost 100% under the condition of the space velocity of about 72,000 mL/g-cat.h (Figure 11). Figure 12 shows the effect of diluted H₂O₂ supplied by the oxidation process on the space velocity in the H₂O₂ conversion process and the amount of steam supplied by the addition of diluted H₂O₂. Which is a value calculated for the total amount of gas and the amount of NO as the main target reactant. [H2O2(s)/flow rate of total gas] ratio to the total flue gas injected into the oxidation process as the space velocity increases more than twice from 36,000 to 84,000 mL/g-cat.h.



Figure 11: Effect of the space velocity on the NO oxidation.(H2O2 amount : 0.3 g/min, NO concentration 5,000 ppm).

The ratio of the amount of pure H_2O_2 in the aqueous H_2O_2 solution to the total amount of exhaust gas and the ratio of the amount of the residual gas to water vapor gradually decreased from 1.12 to 0.51, from 0.37 to 0.18 and from 0.68 to 0.32 respectively other changes were shown.

When the injected diluted H_2O_2 aqueous solution feed rate, the pure H_2O_2 feed rate, and the steam feed rate are expressed as a ratio to the pure NO gas in the exhaust gas supplied to the oxidation process, when the space velocity rises by the same variation width, 422 g/L-NO, from 244 to 132 g/L-NO, from 782 to 312 g/L-NO.

Therefore, the H_2O_2 supply per unit time decreases as the space velocity increases in the H_2O_2 conversion process, compared to the total amount of the exhaust gas injected into the oxidation process. Therefore, various variables are changed depending on the amount of NO_X gas and the amount of NO_X gas supplied to the oxidation process (Figure 12).



Figure 12: Effect of the space velocity on the NO conversion and design factor. (H2O2 amount: 0.3 g/min, NO concentration : 5,000 ppm).

Conclusion

In this study, NO oxidation characteristics were investigated by injecting dry oxidant obtained through catalytic H_2O_2 conversion into NO oxidation process. Screening of various catalysts revealed that the Mn-based catalysts containing Fe₂O₃ as the support showed the highest H_2O_2 conversion efficiency, and the NO oxidizing effect was confirmed when the dry oxidants produced were injected into the coupled oxidation process. As a result of the investigation of NO oxidation characteristics under various operating conditions, the NO oxidation rate increased as the H_2O_2 injection amount and the oxidation reaction temperature increased, and decreased as the space velocity increased. The NO conversion rate obtained from the optimal operating conditions such as the amount of diluted H_2O_2 supplied during the oxidation process and the amount of steam supplied further by injecting diluted H_2O_2 according to the space velocity in the H_2O_2 conversion process obtained through the experiment is about 100%.

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