



Enhancing low-temperature NO_x storage and reduction performance of a Pt-based lean NO_x trap catalyst

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Abstract Two lean NO_x trap (LNT) catalysts, Pt/BaO/CeO₂ + Al₂O₃ and Pt/BaO/CeO₂ – Al₂O₃, were prepared and compared for low-temperature (< 250 °C) NO_x storage and reduction performance. The influence of the form of ceria on low-temperature NO_x storage and reduction performance of LNT catalysts was investigated with the focus on NO_x storage capacity, NO_x reduction efficiency during lean/rich cycling, product selectivity and thermal stability. Inductively coupled plasma-atomic emission spectrometry (ICP-AES), Brunner–Emmet–Teller (BET), H₂-pulse chemisorption and X-ray diffraction (XRD) were conducted to characterize the physical properties of LNT catalysts. NO_x storage capacity and NO_x conversion efficiency were measured to evaluate NO_x storage and reduction performance of LNT catalysts. Pt/BaO/CeO₂ – Al₂O₃ catalyst exhibits higher NO_x storage capacity than Pt/BaO/CeO₂ + Al₂O₃ catalyst in the temperature range of 150–250 °C. Meanwhile, Pt/BaO/CeO₂ – Al₂O₃ catalyst shows better NO_x conversion efficiency and N₂ selectivity. XRD results indicate that the thermal stability of CeO₂ – Al₂O₃ complex oxide is superior to that of pure CeO₂. H₂-pulse chemisorption results show that Pt/BaO/CeO₂ – Al₂O₃ catalyst has higher Pt dispersion than Pt/BaO/CeO₂ + Al₂O₃ catalyst over fresh and aged samples. The improved physical properties of Pt/BaO/CeO₂ – Al₂O₃ catalyst are attributed

to enhance the NO_x storage and reduction performance over Pt/BaO/CeO₂ + Al₂O₃ catalyst.

Keywords NO_x storage and reduction; Low temperature; Ceria; Ceria-alumina; LNT

1 Introduction

To meet the stringent China Stage-VI light-duty diesel vehicles' NO_x emission regulations, a promising approach has been explored by a combination of lean NO_x trap (LNT) and selective catalytic reduction (SCR) catalysts [1–3], which can enhance NO_x reduction while avoiding the need for urea injection to the SCR catalyst [4]. However, additional improvements are required in LNT system, especially for low-temperature NO_x conversion [5, 6].

LNT catalysts operate under cyclic lean/rich conditions [7, 8]. During lean conditions, NO_x is stored on the catalyst; while during rich conditions, stored NO_x is released and reduced into N₂ along with possible byproducts, such as NH₃ and N₂O [9–11]. A typical LNT catalyst contains basic components (alkali metal or alkaline earth metal compounds) for NO_x storage, noble metals (Pt, Pd, Rh), and support oxides [7]. In addition, ceria-based materials have been shown to be beneficial for LNT catalysts at low temperatures [12–14]. CeO₂ is a common rare earth metal oxide with special structure and properties [15–17], and many current commercial LNT catalysts have already incorporated ceria [5, 18].

In the present work, the goal was to study the effects of the form of ceria on low-temperature NO_x storage and reduction performance through surface/bulk analysis and activity tests. The first catalyst of Pt/BaO/CeO₂ + Al₂O₃

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used ceria as a support material as well as alumina, and the second catalyst of Pt/BaO/CeO₂ – Al₂O₃ used complex ceria–alumina as a new support material. The low-temperature NO_x storage capacity (NSC), NO_x reduction efficiency, NH₃ production and N₂O emissions were measured. Thermal stability of LNT catalysts was also investigated after aging for 20 h at 800 °C.

2 Experimental

2.1 Catalyst preparation

Catalysts were prepared by incipient wetness impregnation technique, using aqueous solutions of Ba(CH₃COO)₂ and Pt(NO₃)₂. In the preparation of Pt/BaO/CeO₂ + Al₂O₃ (1 wt% Pt and 9 wt% BaO), CeO₂ (specific surface area 152 m²·g⁻¹) and γ-Al₂O₃ (150 m²·g⁻¹) were mechanically mixed at the weight ratio of 3:7. The impregnation was carried out in a sequential manner: The well-mixed CeO₂ + Al₂O₃ support was firstly impregnated with the Ba acetate solution followed by Pt nitrate solution. On the other hand, Pt/BaO/CeO₂ – Al₂O₃ (1 wt% Pt and 9 wt% BaO) was prepared similarly, but using a CeO₂ – Al₂O₃ complex oxide (30 wt% CeO₂ and 70 wt% Al₂O₃, 154 m²·g⁻¹) as the support. After each impregnation step, the catalyst was dried at 100 °C in air for overnight and then calcined in air at 500 °C for 5 h. The obtained fresh catalysts were denoted as F-1 and F-2, respectively. In order to compare their thermal stability, the catalysts were further calcined in air at 800 °C for 20 h. The aged catalysts were denoted as A-1 and A-2.

2.2 Catalyst characterization

The actual elemental compositions of catalysts were analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES, Optima 2100 DV, PerkinElmer). The Brunner–Emmet–Teller (BET) specific surface areas were measured by N₂ adsorption–desorption at –196 °C on a Micromeritics ASAP 2000 analyzer. Prior to measurements, the samples were pre-treated at 150 °C under vacuum for 3 h to eliminate the adsorbed species. The Pt dispersions were determined by H₂-pulse chemisorption at –80 °C by a Micromeritics AutoChem II Analyzer. The crystalline phases of the catalysts were characterized by X-ray diffractometer (XRD, XRD-600, Shimadzu) with a Cu Kα radiation (λ = 0.154056 nm) at 36 kV with a graphite monochromator.

2.3 Activity measurements

In NO_x storage capacity (NSC) and NO_x conversion efficiency measurements, 0.25 g Pt/BaO/Al₂O₃ catalyst was mixed with 0.75 g quartz sand. The total flow rate is 1 L·min⁻¹, corresponding to a space velocity of 60,000 h⁻¹. Before each experiment, the sample was oxidized in 10% O₂/N₂ balance at 350 °C for 30 min, and then reduced in 5% H₂/N₂ balance at 450 °C for 20 min. The reactor was then cooled in N₂ to the target test temperatures at 150, 200 and 250 °C. The outlet gas of the reactor was maintained at 140 °C to avoid condensation and NH₃ hold-up. A MKS MultiGas 2030 FT-IR analyzer was used to monitor NO, NO₂, N₂O, NH₃, CO, C₃H₆, CO₂ and H₂O concentrations of the outlet gas.

NO_x storage capacity (NSC) tests were conducted by exposing the catalyst to the flowing gas containing 250 × 10⁻⁶ NO, 8% O₂, 5% H₂O, 5% CO₂ at 150, 200 and 250 °C. The storage time was 10 min at each temperature during NSC measurement. NO_x conversion efficiency tests were also measured at 150, 200 and 250 °C, and LNT performance was evaluated in a lean/rich (100 s/17 s) cycle under the gas composition detailed in Table 1. The average value of the last 5 cycles was calculated after 15 lean/rich cycles.

3 Results and discussion

3.1 Catalyst characterization

The contents of Pt, Ba, Ce and Al in catalysts are summarized in Table 2. F-1 and F-2 samples have similar Pt and Ba contents. Ce content in F-1 is slightly lower than that in F-2 (26.24 wt% vs. 27.54 wt%).

BET specific surface areas and Pt dispersions of catalysts are listed in Table 3. The specific surface areas of F-1 and F-2 samples are 117 and 122 m²·g⁻¹, respectively. Pt dispersion of F-1 is 60%, slightly lower than 62% of F-2. After thermal aging at 800 °C for 20 h, their surface areas decrease to 99 and 103 m²·g⁻¹, respectively. The Pt dispersions reach significantly low values, only 12% for A-1 and 19% for A-2, indicating that the sintering of Pt particles occurs during aging due to the mobility of Pt crystallites and eventually merged to form larger particles [19].

Figure 1 shows XRD patterns of fresh and aged catalysts. The results show that Pt-related phases in all four samples were not detected because of either the low loading ratio or its small size. The main crystallite phases detected are CeO₂, BaCO₃ and γ-Al₂O₃ in all samples. The BaAl₂O₄ peak only appears after aging [20, 21]. Applying Scherrer equation, the average CeO₂ crystal size was calculated based on CeO₂ diffraction peak at 2θ = 28.5°. The

Table 1 NO_x conversion efficiency measurement: gas composition

Gas composition	NO	CO	C ₃ H ₆	CO ₂ /%	H ₂ O/%	O ₂ /%	H ₂ /%	N ₂
Lean	250 × 10 ⁻⁶	1800 × 10 ⁻⁶	370 × 10 ⁻⁶	5	5	10.00	–	Bal.
Rich	250 × 10 ⁻⁶	3.00%	960 × 10 ⁻⁶	5	5	0.60	0.25	Bal.

Table 2 Elemental composition of fresh catalysts (wt%)

Catalyst	Pt	Ba	Ce	Al
F-1	1.05	8.06	21.36	32.29
F-2	1.07	7.98	22.42	31.72

F-1—fresh Pt/BaO/CeO₂ + Al₂O₃ catalyst; F-2—fresh Pt/BaO/CeO₂ – Al₂O₃ catalyst

Table 3 BET specific surface areas and Pt dispersions of catalysts

Catalyst	Surface area/(m ² ·g ⁻¹)	Pt dispersion/%
F-1	117	60
F-2	122	62
A-1	95	12
A-2	103	19

CeO₂ crystal size in F-1 is estimated to be 10 nm, while 6 nm in F-2, 16 nm in A-1, and 7 nm in A-2, respectively. These XRD results suggest that the thermal stability of CeO₂ – Al₂O₃ complex oxide is superior to that of pure CeO₂ + Al₂O₃ with regard to CeO₂ crystal size.

3.2 NO_x storage capacity (NSC)

The results of NO_x storage capacity tests are reported in Table 4. All samples' NSCs increase substantially as the test temperature increases from 150 to 250 °C due to

Table 4 NO_x storage capacity (250 × 10⁻⁶ NO, 8% O₂, 5% H₂O, 5% CO₂, N₂ bal., GHSV = 60,000 h⁻¹) of catalysts (μmol·g⁻¹)

Temperature/°C	F-1	F-2	A-2	A-2
150	36.4	39.6	32.3	36.1
200	88.2	98.5	75.4	88.0
250	226.7	243.3	164.4	188.2

enhancing NO oxidation activity to produce NO₂ which is known to be more effective than NO to be adsorbed on LNT catalysts [22]. F-2 sample displays higher NSC compared to F-1 sample at 150, 200 and 250 °C. NSCs of aged samples are lower than those of the fresh samples. A-2 sample still has higher NSC than A-1 sample. The NSC results indicate that CeO₂ – Al₂O₃ complex oxide as the support loaded with Ba and Pt can trap NO_x amount to more extent in comparison with CeO₂ + Al₂O₃.

3.3 NO_x conversion efficiency

NO_x concentration profiles during the last 5 lean/rich cycles are shown in Fig. 2. Average NO_x conversions during the last 5 cycles are presented in Table 5. For F-1 sample, NO_x breakthrough is immediately observed after the feed gas switched into lean condition at 150 °C, and the outlet NO_x concentration decreases with time and gradually approaches the inlet NO_x concentration. When the feed gas is subsequently switched to the rich condition, a sharp and intense NO_x release peak appears and then intensity

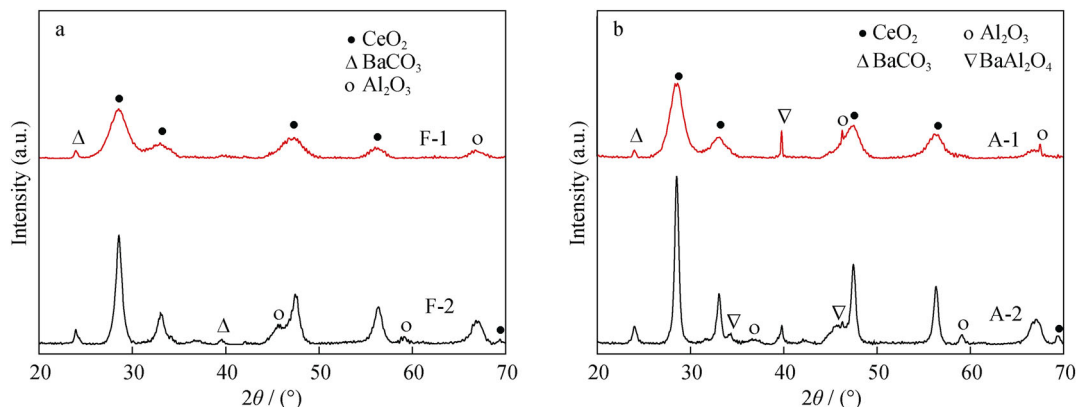


Fig. 1 XRD patterns of fresh and aged catalysts: **a** F-1 and F-2 catalysts and **b** A-1 and A-2 catalysts

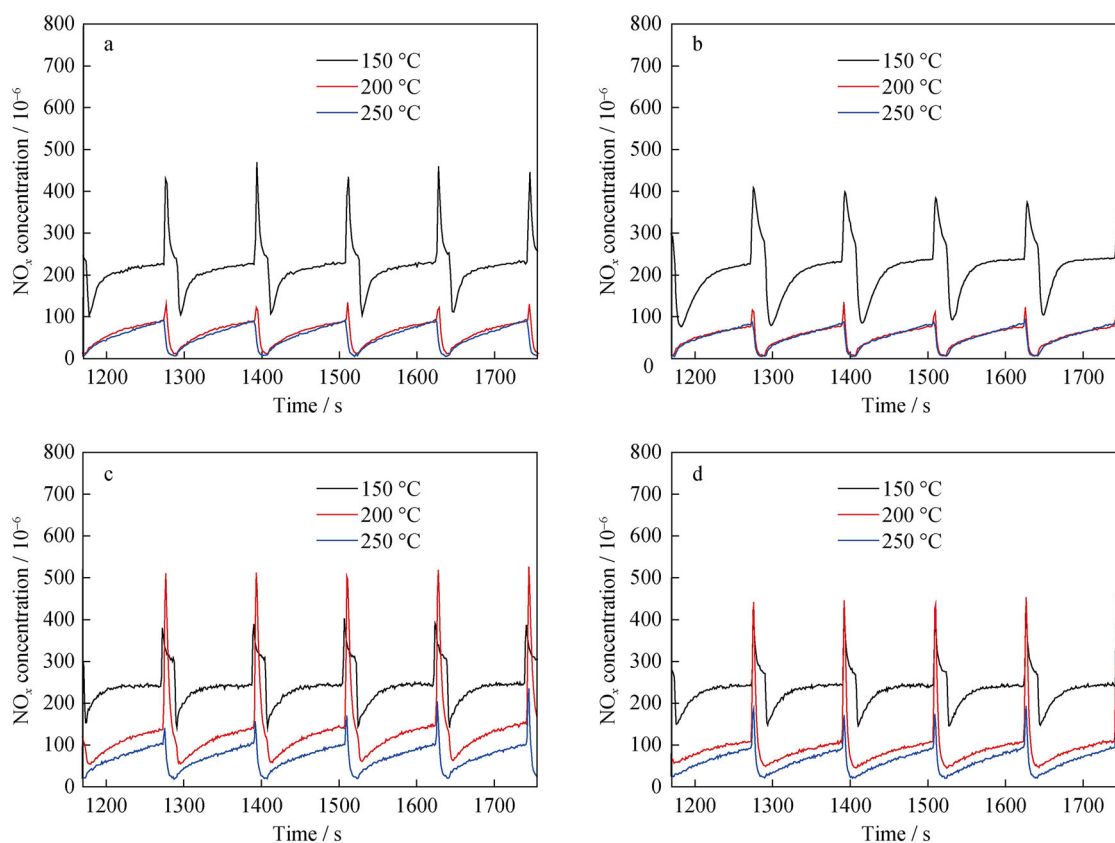


Fig. 2 NO_x concentration profiles during last 5 lean/rich cycles: **a** F-1 catalyst, **b** F-2 catalyst, **c** A-1 catalyst, and **d** A-2 catalyst

Table 5 NO_x conversion and selectivity of N-containing products during last 5 lean/rich cycles

Temperature/°C	Catalyst	NO _x conversion/%	Selectivity/%		
			NH ₃	N ₂ O	N ₂
150	F-1	13.8	8.6	51.1	40.3
	F-2	14.6	7.4	51.5	41.1
	A-1	6.6	0	66.9	33.1
	A-2	9.0	4.9	55.6	39.5
200	F-1	72.0	38.5	34.7	26.8
	F-2	73.4	29.6	33.8	36.6
	A-1	49.0	46.4	38.3	15.3
	A-2	58.9	36.4	38.2	25.4
250	F-1	75.2	22.6	23.8	53.6
	F-2	76.1	18.9	20.7	60.4
	A-1	68.4	42.3	28.6	29.1
	A-2	71.7	31.4	25.5	43.1

quickly decreases. The similar profile was also observed in a previous study [23]. The average NO_x conversion at 150 °C is 13.8%. At 200 °C, the outlet NO_x concentration reaches only about 90×10^{-6} at the end of the lean duration. The amount of NO_x release during switching to

rich condition becomes significantly lower compared to that at 150 °C. Therefore, the average NO_x conversion increases to 72.0%. At 250 °C, NO_x concentration profile during lean duration is almost coincided with that at 200 °C. However, there is no NO_x release detected during switching to rich condition. Average NO_x conversion reaches 75.2%. Raising reaction temperature increases NO_x storage capacity, and on the other hand, high temperature is beneficial to promoting NO_x reduction ability. As a result, the average NO_x conversion increases with reaction temperature increasing from 150 to 250 °C.

In the case of F-2 sample, NO_x evolutions at three temperatures are similar to those of F-1 sample, but F-2 sample has slightly higher average NO_x conversions, 14.6% at 150 °C, 73.4% at 200 °C, and 76.1% at 250 °C, in accordance with the results of NSC measurements.

After thermal aging, the amount of NO_x trapped on the aged catalyst at lean phase at 150 °C becomes smaller in comparison with fresh catalysts and the outlet NO_x concentrations gradually reach a constant level (around 240×10^{-6}) for both aged catalysts. Higher NO_x slip is observed during rich phase, and average NO_x conversions of A-1 and A-2 samples are only 6.6% and 9.0%, respectively. At 200 °C, NO_x-spill-out over A-1 is relatively larger in comparison with that over A-2 during switching to

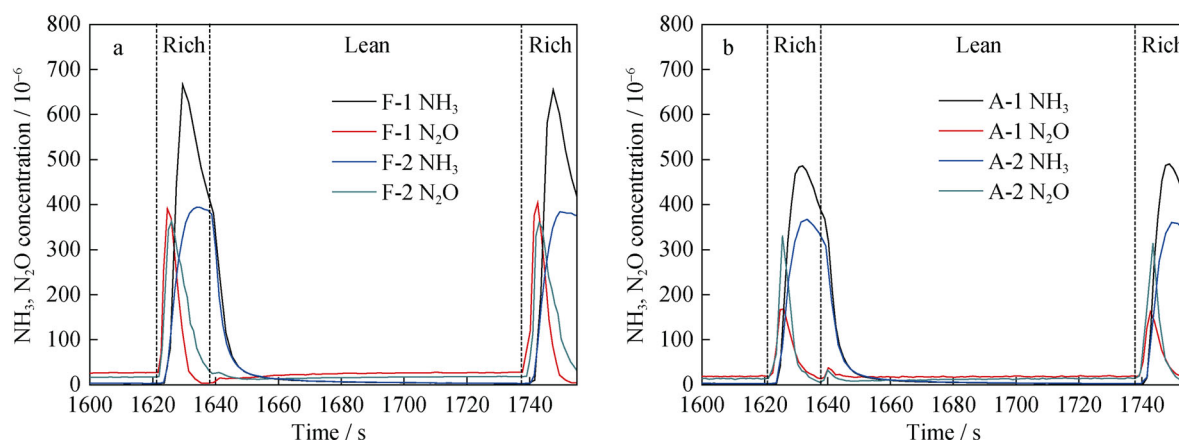
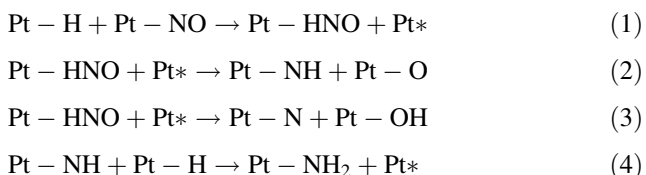


Fig. 3 Evolution of NH₃ and N₂O concentrations during lean/rich cycles at 200 °C: **a** fresh samples and **b** aged samples

the rich condition, leading to a lower NO_x conversion for A-1 (49.0% vs. 58.9% for A-2). NO_x release still appears over aged samples at 250 °C, resulting in that NO_x conversion decreases to some extent. Average NO_x conversion sharply declines at 150 and 200 °C. Pt is known to have impacts on NO oxidation under lean conditions, NO_x storage under rich/lean conditions and NO_x reduction under rich conditions [7]. The difference in NO_x conversion may be correlated with different Pt dispersions of catalysts. Clayton et al. [24] also found that the differences in storage and reduction activity were the largest among three catalysts which have different Pt dispersions at low temperatures (< 200 °C).

From the perspective of study on the law and production of NH₃ and N₂O, their concentrations evolution at 200 °C during lean/rich cycles is taken as an example and shown in Fig. 3. For fresh and aged samples, NH₃ and N₂O are both formed in lean and rich phases. During lean phase, NH₃ and N₂O are subsequently formed, NH₃ intensity sharply decreases to below 10 × 10⁻⁶ at the initial lean phase, while a small N₂O peak appears, and then the N₂O intensity gradually becomes stabilized. A sharp N₂O peak is immediately observed at the beginning of rich phase, and NH₃ peak delays about 2 s. In comparison with the results obtained over fresh samples, the amount of NH₃ and N₂O formation decreases over aged samples. In the lean and rich phase, NH₃ is mainly formed via the reduction in NO_x by surface hydrogen, although NH₃ is also a product of isocyanate hydrolysis reaction [25, 26], Dasari et al. [25] summarized the following reaction mechanism:



N₂O formation in lean phase is related to the reactions between surface-deposited reductants/intermediates (CO, HC, NH₃, isocyanate) and gaseous NO/O₂, and N₂O release peak during rich phase may be attributed to that NO_x partially reduced over platinum group metal (PGM) sites [26–28].

Concerning the product selectivity, N₂O is the main product at 150 °C. At 200 °C, selectivity of NH₃, N₂O and N₂ is similar for fresh samples, but NH₃ and N₂O become the main products for aged samples. By increasing the temperature to 250 °C, N₂ selectivity of all samples shows a substantial enhancement. Moreover, N₂O selectivity decreases as the temperature increases from 150 to 250 °C. N₂ selectivity decreases in the following order: F-2 > F-1 > A-2 > A-1. Thermal aging results are in an increase in NH₃ and N₂O selectivity, except NH₃ selectivity at 150 °C. Compared to fresh samples, the higher NH₃ selectivity over aged LNT catalysts above 200 °C was also found by Chatterjee et al. [29] and Easterling et al. [30]. Chatterjee et al. [29] suggested that the lower noble metal and oxygen storage activities correspond to the shifted light-off of the ammonia oxidation reactions to higher temperatures, which supports the faster NH₃ breakthrough.

4 Conclusion

The influence of the form of ceria on low-temperature NO_x storage and reduction performance of LNT catalysts was investigated, using CeO₂ – Al₂O₃ complex oxide or CeO₂ + Al₂O₃ mixed oxide as the support for BaO and Pt. CeO₂ – Al₂O₃ support exhibits the improvements on NO_x storage capacity, NO_x conversion efficiency (especially for aged samples at 200 °C) and N₂ selectivity in comparison

with $\text{CeO}_2 + \text{Al}_2\text{O}_3$ support in the temperature range of 150–250 °C. After aging for 20 h at 800 °C, Pt/BaO/ $\text{CeO}_2 - \text{Al}_2\text{O}_3$ catalyst exhibits better thermal stability and chemical distribution than Pt/BaO/ $\text{CeO}_2 + \text{Al}_2\text{O}_3$ catalyst. Overall, $\text{CeO}_2 - \text{Al}_2\text{O}_3$ -based catalysts show superior NO_x storage and regeneration performance over $\text{CeO}_2 + \text{Al}_2\text{O}_3$ -based catalyst at low temperatures.

Concerning NH_3 and N_2O selectivity of NO_x reduction, N_2O is the main product at 150 °C, and its selectivity decreases as the temperature increases from 150 to 250 °C. Thermal aging results are in an increase in the NH_3 and N_2O selectivity at 200 and 250 °C.

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