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Performance of Co-containing NO_x storage and reduction catalysts as a function of cycling condition

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Abstract

Co-containing NO_x storage and reduction (NSR) catalysts were studied for their NO_x storage and NO_x conversion behavior. It was found that addition of 5% Co increased the NO_x storage capacity of a 1% Rh/15% Ba catalyst by 50% and that of a 1% Pt/15% Ba catalyst by 100%. This promotional effect was attributed to the high oxidizing ability of Co, present in the form of Co_3O_4, providing extra oxidation sites for NO to NO_2 conversion and more contact area for NO_2 spillover to Ba storage sites. Empirical models were developed to predict the NO_x conversion capacity of the catalyst as a function of total cycle time and the fraction spent in the lean phase. Using these models, it was found that 5% Co also improved the performance of 1% Pt/15% Ba and 1% Rh/15% Ba catalysts at higher lean fractions, allowing substantial improvement in overall fuel efficiency. The reduction capacity of Co was much less than that of noble metals, such that noble metals were still necessary (although at a reduced loading) for regenerating the catalyst in the rich phase. Along these lines, it was found that a 0.25% Pt/5% Co/15% Ba-containing catalyst showed better performance than a 1% Pt/15% Ba-containing catalyst. This implies that the cost of NSR catalysts can be substantially reduced by replacing some of the Pt with Co as the active oxidizing metal in NSR catalyst formulations.

Keywords: NO_x storage and reduction; High-throughput experimentation; Combinatorial catalysis; Design of experiments; Response surface design

1. Introduction

The improved efficiency of a continuous lean-burn automobile engine over a traditional stoichiometric engine can be as high as 20–30% [1]. Because NO_x emissions increase significantly when operating under lean conditions, extensive effort has gone into finding catalysts that will reduce NO_x under fuel lean conditions; however, an acceptable catalyst has not yet been discovered [2–4]. To address the apparent conflict of high engine efficiency and low NO_x emissions, NO_x storage and reduction (NSR) catalysts have been developed to store NO_x during a fuel-lean cycle and to reduce the stored NO_x during a subsequent fuel-rich cycle [5–7]. Over the past decade, extensive research has examined the performance of platinum-containing NSR catalysts as an oxidative and reductive component and barium as a NO_x storage component [8–23]. The use of other noble metals in place of, or in addition to, Pt and other NO_x storage components in place of Ba also has been studied [24–27], and the optimization of catalyst composition for best performance at higher lean fractions has been investigated [28, 29]. The use of transition metals to improve the resistance to sulfur poisoning also has been reported [30]. In this report, we focus on the use of transition metals to improve the NO_x storage and conversion efficiency of the catalyst, and also to act as substitute for noble metals, decreasing the cost of the catalyst. In previous work [31], we showed that adding Co leads to a significant increase in the NO_x storage of Pt/Ba catalysts. This suggests that Co can replace Pt as the active oxidizing agent in the conventional NSR catalyst formulation. In the current work, we use a statistical design of experiments approach to further explore the effect of Co on the performance of Pt/Ba and Rh/Ba catalysts under a wide variety of fuel-lean and fuel-rich cycles. This study was performed by varying the total cycle time and the fraction of time spent in the fuel-lean phase. Cata-

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lyst performance was quantified by measuring the overall NO_x conversion, which is the primary criterion for commercial applications. The temperature for these studies was fixed at 648 K, which was shown to be the temperature for optimum performance of Pt/Ba-based NSR catalysts [16].

2. Experimental

2.1. High-throughput experimental setup

All catalytic tests were performed using a 16-channel parallel reactor, details of which have been described previously [32]. The reaction products from all 16 reactors were analyzed simultaneously using Fourier transform infrared (FTIR) imaging [33–37]. The optical setup consists of a Bruker Equinox 55 FTIR spectrometer interfaced with a 64 × 64 pixel mercury cadmium telluride focal plane array detector (Santa Barbara Focal Plane, Goleta, CA), capable of collecting IR spectra of the effluents of all 16 reactors simultaneously with a time resolution of less than 2 s [23]. Details of the optical setup and analytical methods have been provided previously [33,35, 38].

2.2. Catalyst synthesis and characterization

The catalysts were synthesized via incipient wetness on γ-Al_2O_3 (Catalox Sba-200, 200 m^2/g). The naming convention for each catalyst is based on the nominal weight loading. Thus, a catalyst with a nominal weight loading of 1% w/w Pt and 15% w/w Ba is referred to as 1Pt/15Ba. Chloroplatinic acid hexahydrate, barium nitrate, cobalt nitrate, and rhodium (III) chloride hydrate precursors (Strem Chemicals) were dissolved in distilled water before impregnation. Details of the impregnation procedure have been given previously [39]. All catalysts were calcined by heating in air to 473 K for 2 h, holding the temperature at 473 K for 1 h, further heating to 823 K over 3 h, holding at 823 K for 2 h, and then cooling to 298 K. In addition, all catalysts were reduced in the high-throughput reactor for 1 h in 10% v/v H_2 in He at 773 K before the reaction studies were performed. The weight loadings of all catalysts were verified by atomic absorption spectroscopy (S series atomic absorption spectrometer; Thermo Electron). The details of this procedure have been given previously [40] and are not be discussed here.

2.3. Experimental cycling design and model development

The catalysts were initially screened for long-cycle conditions, with 15 min fuel-rich and 30 min fuel-lean conditions, to check their total NO_x storage and reduction behavior. These studies are referred to as long-cycle studies throughout this report, because the lean and rich cycle times were relatively long. The CO and NO_x concentrations were calculated from a moving average of nine spectra, whereas the N_2O concentrations were calculated from a single spectrum. The second studies, in which the total cycle time was kept relatively short to simulate actual exhaust conditions and the catalysts were screened for average NO_x conversion capacity, are called short-cycle studies. For all of the short-cycle data, the CO and NO_x concentrations were calculated from a moving average of five spectra, whereas the N_2O concentrations were calculated from a single spectral scan. This was done to improve the signal-to-noise ratio of the experimental data, because the absorbance bands for the reaction gases (CO and NO_x) in the IR spectra are weak. The total cycle time is defined as the time between the beginning of one lean phase and the beginning of the next lean phase, and the lean fraction is defined as the fraction of the total cycle time spent in the fuel-lean phase. A graphical representation of the total cycle time and lean fraction is shown in Fig. 1. For both the long-cycle and the short-cycle studies, the feed gas consisted of 0.15% v/v NO, 6% v/v O_2, 0.9% v/v CO, and 0.15% v/v C_2H_4 in He for the fuel-lean phase at a space velocity of 42,000 mL/h/g catalyst. The fuel-rich phase was simulated by replacing the oxygen with an equal volume of helium while maintaining all other flow rates constant. For all experiments, 150 mg of catalyst was loaded into each reactor, and all reactions were performed at $T = 648$ K and atmospheric pressure. The total cycle time and the lean fraction, for short-cycle studies, were varied according to a response surface design for two variables, as indicated by the design points shown in Fig. 2.

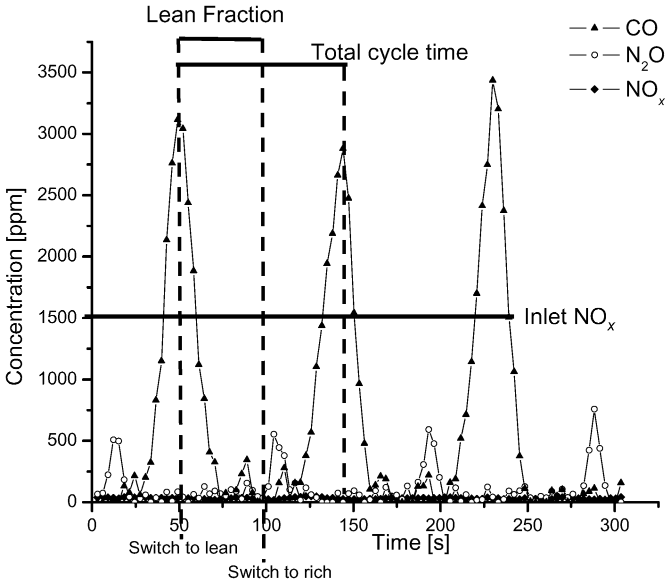

Fig. 1. Definition of performance criteria for catalysts tested under realistic short-cycle conditions.

The center cycling conditions (90 s total cycle time and a lean fraction of 0.675) were repeated at least three times to estimate the variability in the measurements. All of the design conditions, including the repeats for the center cycling conditions, were tested in random order. The total cycle time and lean fraction were fit to the catalytic response (R), determined by average NO_x conversion, using an empirical model of the form of Eq. (1), where C, α, and β are constants derived by fitting to the experimental data and CT and LF are variables representing cycle time and lean fraction, respectively:

$$R = C + \alpha_1(\mathrm{CT}) + \alpha_2(\mathrm{LF}) + \alpha_{12}(\mathrm{CT})(\mathrm{LF}) + \beta_1(\mathrm{CT})^2 + \beta_2(\mathrm{LF})^2. \quad (1)$$

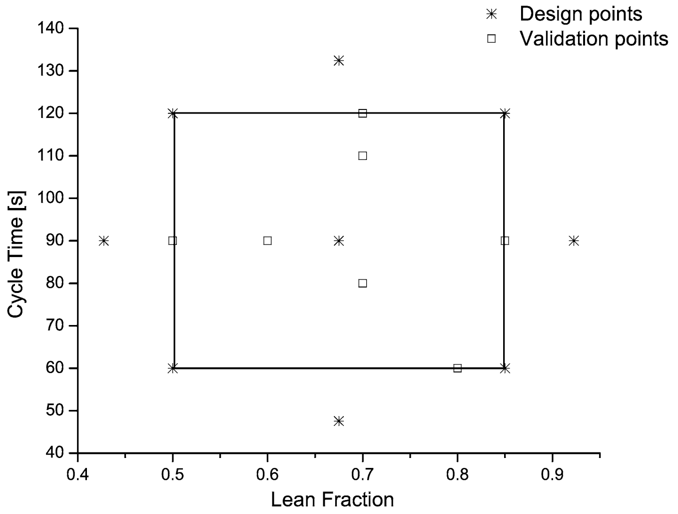

Fig. 2. Response surface design for cycle time and lean fraction used for testing the catalysts.

2.4. Performance criteria

As mentioned above, the catalysts were initially screened for NO_x storage capacity via long-cycle studies. The total NO_x storage is determined by setting a cutoff NO_x concentration of 300 ppm. The area between the inlet NO_x concentration and the outlet NO_x concentration until reaching the cutoff NO_x limit was integrated and converted to moles of NO_x in the same manner as described previously [39]. Further details on the testing procedure are available elsewhere [41].

For short-cycle studies, the catalysts were screened for NO_x conversion, which is a much more relevant criterion for the use of catalysts in commercial applications. All cycling conditions were preceded by running the catalysts under fuel-rich conditions for 15 min to ensure that all catalysts began each cycle free of reversibly stored nitrates [28]. After completion of the long fuel-rich phase, the reaction gases were cycled between fuel-rich and fuel-lean conditions according to the cycle time and lean fraction specified by the cycling design. The catalysts were exposed to multiple lean/rich cycles until their behavior became reproducible, typically within two to three cycles. The NO_x concentration was then averaged over multiple cycles and used to calculate NO_x conversion using Eq. (2) for that particular combination of cycle time and lean fraction,

$$NO_x\text{conv} = 100 \times \left[1 - \frac{NO_x\text{avg}}{NO_x i}\right], \tag{2}$$

where NO_xavg is the average NO_x effluent concentration and $NO_x i$ is the inlet NO_x concentration.

3. Results and discussion

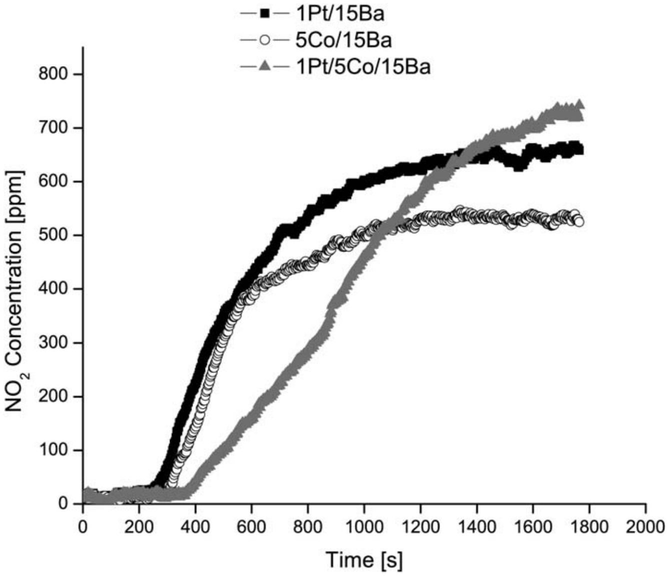

Fig. 3. NO_2 concentration profiles for 5Co/15Ba, 1Pt/15Ba, and 1Pt/5Co/15Ba catalysts after a switch from rich to lean during long-cycle studies.

Table 1
Characterization of catalysts using atomic absorption (AA) spectroscopy

Nominal loading (% w/w)	Actual weight loadings measured by AA			
Catalyst	Pt (%)	Rh (%)	Co (%)	Ba (%)
1Pt/15Ba	0.81	–	–	13.19
1Rh/15Ba	–	0.62	–	12.88
5Co/15Ba	–	–	4.8	11.17
1Pt/5Co/15Ba	0.6	–	5.02	12.44
1Rh/5Co/15Ba	–	0.7	5.08	13.5
0.25Pt/5Co/15Ba	0.25	–	5.1	12.2
0.5Pt/5Co/15Ba	0.37	–	5.04	12.40

In a previous communication [31], we showed that adding 5% Co doubles the NO_x storage of a 1Pt/15Ba catalyst and that the total NO_x storage, from long-cycle studies, of a noble metal-free 5Co/15Ba catalyst is equivalent to that of a 1Pt/15Ba catalyst. Fig. 3 shows the NO_2 concentration profiles for three different catalysts after switching from the rich to the lean phase for long-cycle studies. The steady-state NO_2 concentration for 5Co/15Ba catalyst was similar to that of 1Pt/15Ba, demonstrating that the oxidizing ability of 5% Co is comparable to that of 1% Pt. Thus, the promotional effect of Co is associated with its strong oxidizing ability, present in the form of Co_3O_4 [31], providing high oxygen content to increase the nitric oxide oxidation to NO_2 and also more contact area for NO_2 spillover to the Ba NO_x storage sites. It was then suggested that Co could replace Pt as the active oxidizing metal, thereby reducing the total noble metal content in NSR catalysts. However, it was also claimed that a small amount of noble metal is needed to quickly reduce the stored NO_x during regeneration of the catalysts under fuel-rich conditions, because Co is a poor reducing agent compared with noble metals like Pt or Rh. To confirm this, we further explored the effect of Co in combination with either Pt or Rh. Table 1 shows the actual weight loadings, measured using atomic absorption spectroscopy, for all catalysts tested in these studies. Fig. 4 compares the performance of several NSR catalysts containing 5% Co in combination with Pt or Rh in terms of total NO_x storage under long-cycling conditions. The addition of 5% Co doubles the NO_x storage of the 1Pt/15Ba catalyst and also increases the NO_x storage of the 1Rh/15Ba catalyst by ∼50%. This behavior can again be attributed to the

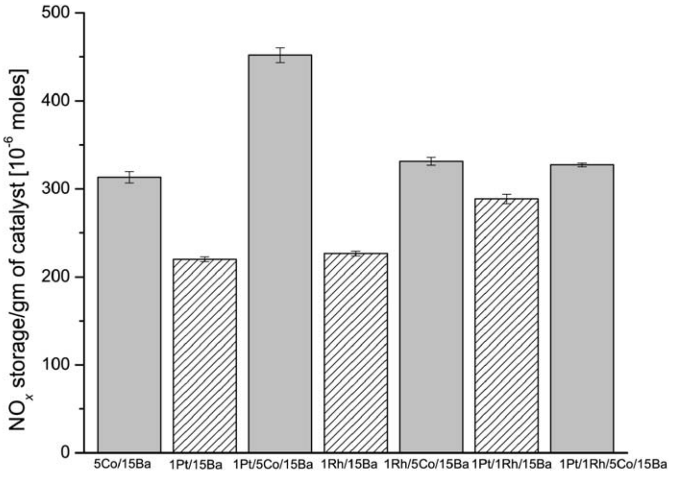

Fig. 4. Lean NO_x storage results for various Co-containing NSR catalysts for a total cycle time of 45 min, with lean phase of 30 min, and rich phase of 15 min.

Table 2
Simulated diesel exhaust conditions used for catalyst testing

Reaction gas	Lean cycle	Rich cycle
O_2 (%)	9	1
NO (%)	0.08	0.08
CO (%)	0.05	3
C_2H_4 (%)	0.04	1
H_2 (%)	0.03	1
CO_2 (%)	5	5

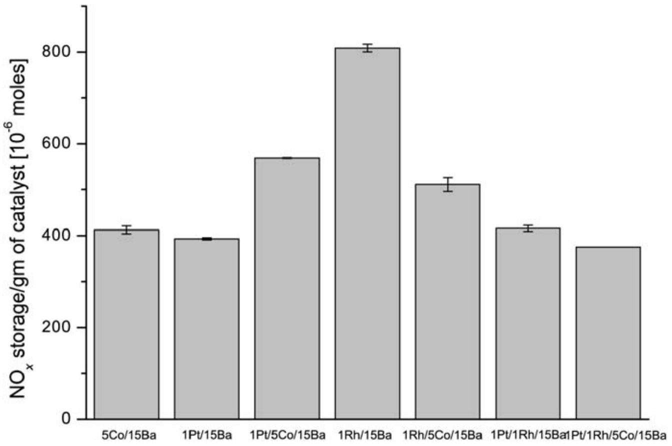

Fig. 5. Lean NO_x storage results for Co-containing NSR catalysts tested for simulated diesel exhaust conditions using long-cycle studies.

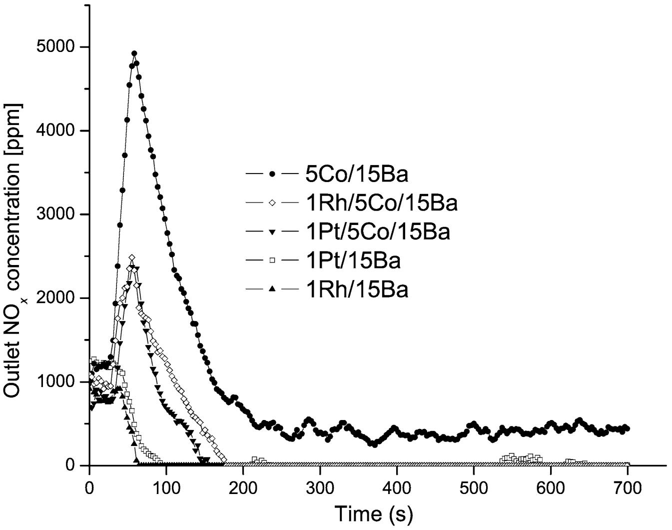

Fig. 6. NO_x concentration profiles during a lean to rich switch, for a total cycle time of 45 min, with lean phase of 30 min, and rich phase of 15 min.

high oxidizing capacity of Co, with increased conversion from NO to NO_2 and increased contact area with Ba NO_x storage sites for NO_2 spillover, because there is 5% Co compared with 1% Pt. However, when both Pt and Rh are present and there is twice the amount of noble metal, there is no significant change in the storage, implying a net decrease in the ratio of NO_x storage to noble metal content. Similar results have also been reported by Amberntsson et al. [42], who found a net decrease in the NO_x storage capacity when Pt and Rh were combined together while the total noble metal content was kept constant. This finding could be due to the decreased contact area between Pt and Ba, resulting from Rh being in close contact with Pt, which is believed to be an important factor governing the NO_x storage capacity of the catalyst [14,24,29,40,41,43,44]. The results given in the remainder of this report are limited to those for NSR catalysts containing only one of the noble metals; the effect of adding more than one noble metal will be addressed in more depth in a future publication. In addition, to further verify the promotional effect of Co, we also tested the aforementioned catalysts for simulated diesel exhaust conditions via long-cycle time studies. The reaction conditions used are given in Table 2, and the results of this testing are shown in Fig. 5. Helium was used as the carrier gas, and the testing was done at 648 K. The feed stream contained three different reducing agents and 5% CO_2. The results shown in Fig. 5 clearly demonstrate that the Co-containing catalyst (5Co/15Ba) still outperformed the noble metal catalysts (1Pt/15Ba and 1Rh/15Ba). Moreover, adding 5% Co to 1Pt/15Ba catalysts once again doubled the catalyst's NO_x storage capacity. Thus, the results of these tests are similar to those shown in Fig. 3 and can be explained by the same reasoning.

Although lean NO_x storage capacity is an important criterion for comparing catalysts, the performance under realistic conditions also depends on the reduction capacity of the catalyst, that is, how much and how fast the catalyst can regenerate in the fuel-rich phase. To check the catalysts' overall reduction capacity, they were exposed to 30 min of lean phase, such that the catalyst surface was saturated with nitrates. The conditions were then switched to the rich phase, and the outlet NO_x concentration was monitored. Fig. 6 shows the NO_x concentration profiles for five different catalysts after switching from the fuel-lean phase to the fuel-rich phase. It shows that for 5Co/15Ba catalysts, a large NO_x breakthrough peak initially occurs, after which the final outlet concentration stabilizes at a higher value than for the other catalysts. However, for 1Pt/15Ba and 1Rh/15Ba catalysts, not only does the final outlet NO_x concentration drop to zero, but also the initial NO_x breakthrough disappears. It has been suggested in the literature that the initial breakthrough peak in the rich phase is associated mainly with the release of NO and NO_2, which are produced due to the partial reduction of nitrates formed in the lean phase [14, 16,27,43,45–47]. The higher breakthrough peak for 5Co/15Ba compared with 1Pt/15Ba or 1Rh/15Ba implies that the reduction capacity of Co is not comparable to that of noble metals. Most of the NO and NO_2 generated in the rich phase is released without being completely reduced to N_2 or N_2O, unlike the case when Pt or Rh is present. Moreover, the higher steady-state NO_x concentration in the fuel-rich phase further confirms that a Co-containing catalyst was not able to match the reduction capacity of a noble metal-containing catalyst, even at more than five times the weight loading. However, adding 1% Pt or 1% Rh to a 5Co/15Ba catalyst not only reduces the initial NO_x breakthrough peak, but also causes the steady-state NO_x concentration to go to zero. Because the performance of a catalyst at high lean fraction is governed largely by its regeneration capacity [29], the above results show that noble metals are still necessary along with Co for complete catalyst regeneration in the rich phase.

Along with NO_x storage and reduction capacity, the catalysts were also tested for NO_x conversion in short-cycle studies. Fig. 7 shows the average NO_x conversion for different Co-containing catalysts tested for a particular combination of cycle time (60 s) and lean fraction (0.85). 5Co/15Ba showed only 25% NO_x conversion, which could be due to the low reduction capacity of Co, the absence of noble metals, and the short time for regeneration. However, the catalysts containing a combination of Co with a noble metal showed 70–75% NO_x conversion under the same operating conditions. This can be attributed to a combination of higher reduction capacity due to the presence of noble metals, which improves regeneration in the rich phase, and increased NO_x storage in the lean phase due to the greater oxidizing ability of Co.

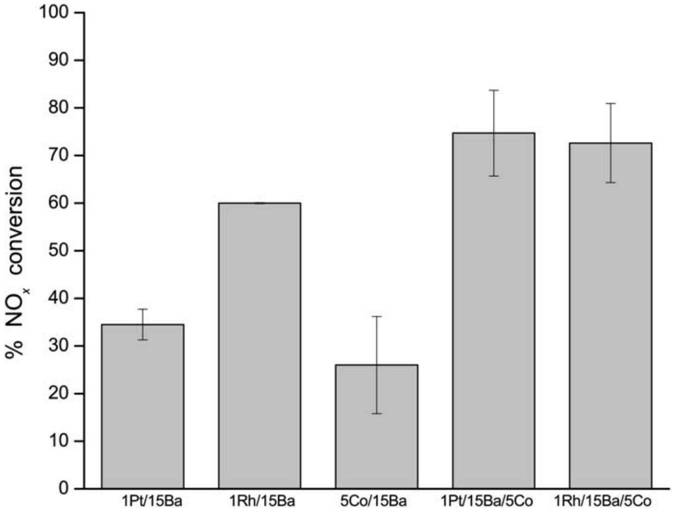

Fig. 7. NO_x conversion for a total cycle time of 60 s and a lean fraction of 0.85.

The Co-containing catalysts were further tested for different combinations of lean fraction and cycle time, chosen based on the response surface central composite design shown in Fig. 2. The average NO_x conversion was used to develop an empirical quadratic model relating cycle time and lean fraction to NO_x conversion, as in Eq. (1). Table 3 shows the coefficients for the quadratic models developed for 1Pt/15Ba, 1Rh/15Ba, 1Pt/15Ba/5Co, and 1Rh/15Ba/5Co NSR catalysts. To verify the consistency of the empirical models, Fig. 8 shows predicted and actual NO_x concentrations for the model developed for 1Pt/15Ba/5Co. The error bar in the figure is calculated from averaging over the three repeats of the center cycling condition tested randomly at different times, as well as with the same catalysts tested under the same conditions but in different reactor tubes. Also included in this figure are seven additional experimental conditions, as shown in Fig. 2, which were not included in the initial design but were used to validate the empirical model. From the figure, it can be seen that the model adequately predicts the experimental data over the entire region

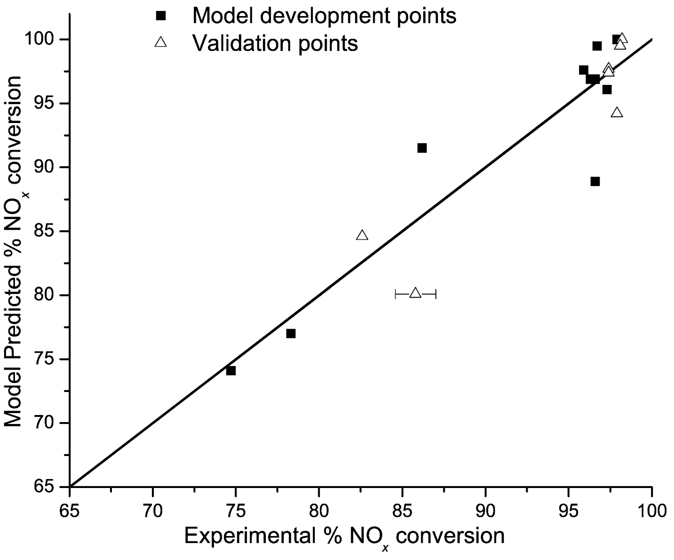

Fig. 8. Comparison between the experimental NO_x conversion and model predicted NO_x conversion for 1Pt/5Co/15Ba catalysts.

Table 3
Model coefficients for the quadratic model relating NO_x conversion as a function of cycle time and lean fraction

Model parameters	NO_x conversion coefficients				
	1Pt/15Ba	1Rh/15Ba	0.25Pt/5Co/15Ba	1Pt/5Co/15Ba	1Rh/5Co/15Ba
Constant (C)	4.86	42.12	35.5433	102.80	34.8
Cycle time (CT)	0.1	−0.58	0.0384	−0.26	−0.47
Lean fraction (LF)	345.13	303.95	235.77	48.9	308.93
Cycle time × cycle time	−0.01	0.0007	−0.0009	−0.002	0.0006
Lean fraction × lean fraction	−464.14	−331.02	−226.53	−141.22	−320.82
Cycle time × lean fraction	1.91	0.78	0.15	−1.07	0.61

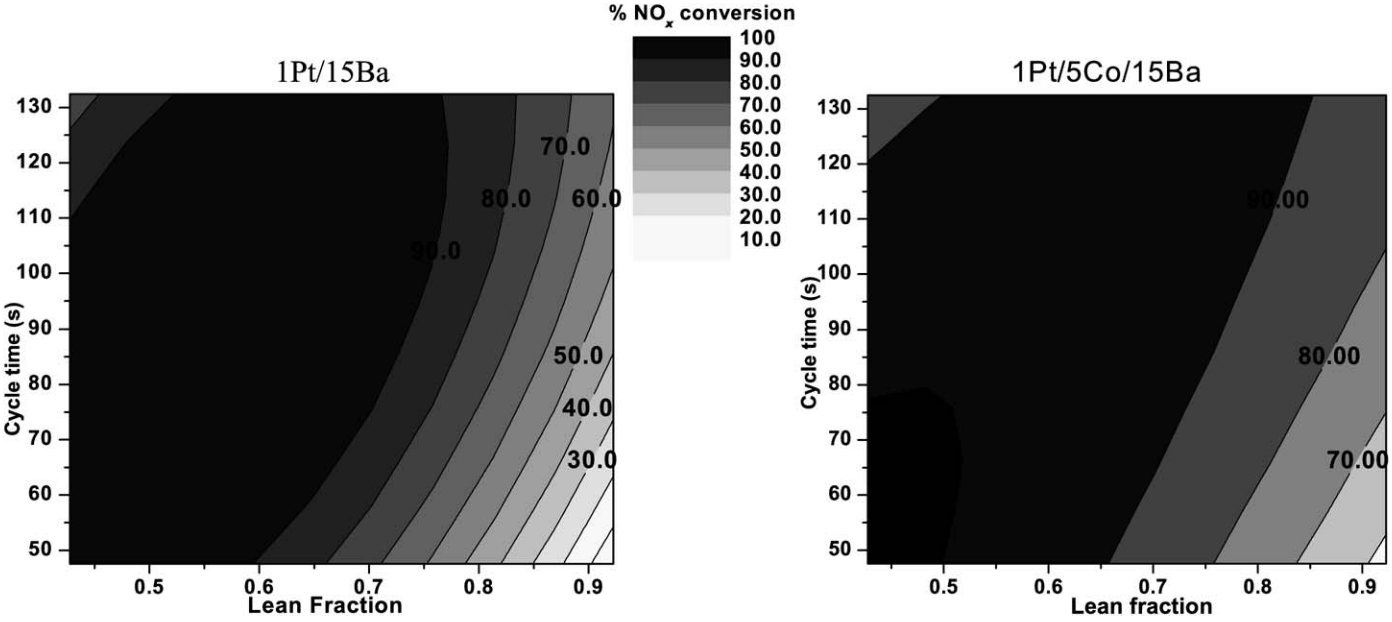

Fig. 9. Model predictions for NO$_x$ conversion as a function of cycle time and lean fraction for 1Pt/15Ba and 1Pt/15Ba/5Co NSR catalysts.

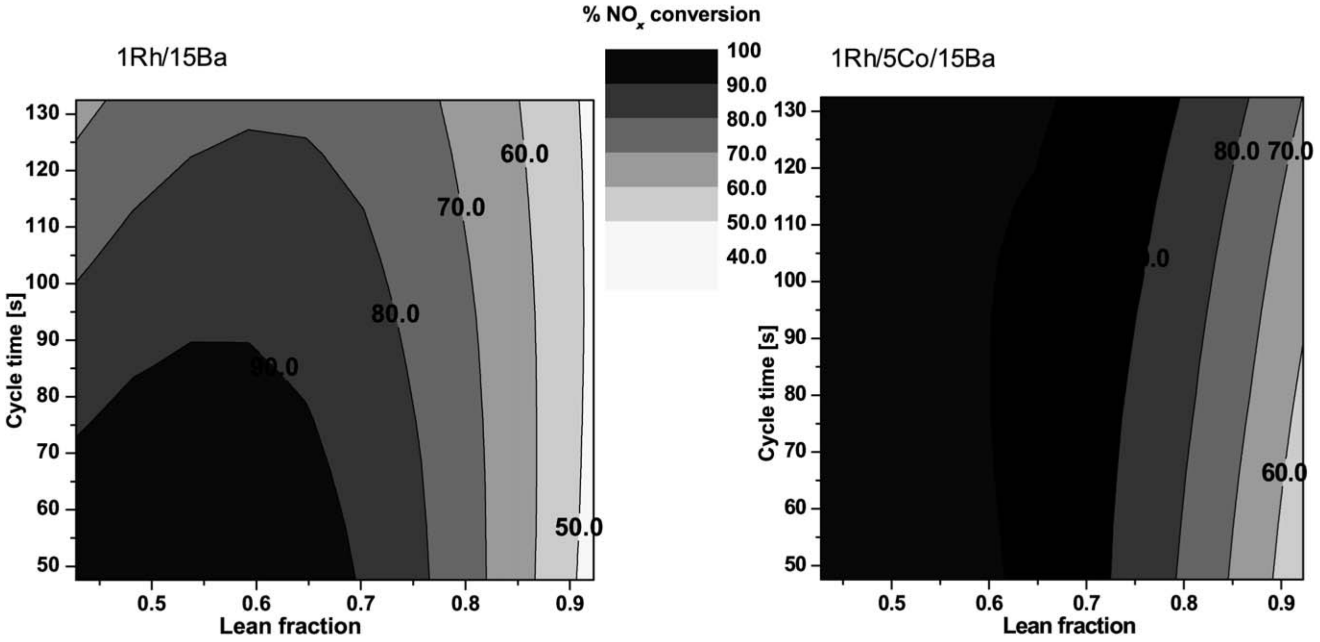

Fig. 10. Model predictions for NO$_x$ conversion as a function of cycle time and lean fraction for 1Rh/15Ba and 1Rh/15Ba/5Co NSR catalysts.

of investigation. Fig. 9 graphically represents the model predictions for 1Pt/15Ba and 1Pt/15Ba/5Co for NO$_x$ conversion as a function of cycle time and lean fraction. The NO$_x$ conversion values were obtained from the NO$_x$ conversion model, using Eq. (2) and the coefficients given in Table 3. A large increase in the region of >95% conversion occurred after the addition of Co. In addition, NO$_x$ conversion at short-cycle times and long-lean fractions showed a marked improvement of around 30–40%. For lower lean fractions, when the rich phase is sufficient for the catalyst to completely regenerate, performance is governed by NO$_x$ storage capacity, that is, the catalyst's oxidation capacity. Similarly, for large lean fractions, when the rich phase is not long enough for complete regeneration, the reduction capacity of the catalyst governs the performance [29]. Co, with its high oxidizing capacity and subsequently high NO$_x$ storage capacity, substantially improves catalyst performance in the oxidation-governing regime, that is, at lower lean fractions. In addition, Co reduces NO$_x$ to a certain extent, as shown in Fig. 6, thus improving the performance in the reduction-governing regime (i.e., long lean fraction). Similar behavior can be seen when comparing model predictions for 1Rh/15Ba and 1Rh/15Ba/5Co, as shown in Fig. 10. The figure shows significantly improved performance in the oxidation-governing regime and some enhancement in the reduction-governing regime. Adding Co to both 1Pt/15Ba and 1Rh/15Ba catalysts extends the region of optimum operation to lower cycle times and higher lean fractions, which has potential impact in terms of improving the fuel efficiency of lean-burn automobile engines.

In addition to the desired improvement in fuel efficiency, there also exists motivation to find a substitute for expensive noble metals in catalytic converters. It was previously suggested that using Co will allow for reduced noble metal loadings [31]. To evaluate the extent to which the noble metal can be reduced, catalysts with 5Co, 15Ba, and a various weight loadings of Pt (all $<1\%$) were synthesized. The catalysts were tested for lean NO_x storage under long-cycle conditions and for NO_x conversion under short-cycle conditions. In practical applications, we want to operate at a high-lean fraction, where the reduction capacity governs the performance and noble metal plays the most important role in regeneration. Hence, comparing catalyst performance at high-lean fractions provides more insight into the actual amount of noble metal that can be replaced without affecting catalyst performance. Fig. 11 shows the NO_x conversion efficiency for the combination of 90 s of cycle time and a 0.92 lean fraction; Fig. 12 shows the results in terms of NO_x storage for long-cycle studies for all of the different catalysts. It can be seen that the NO_x storage capacity and NO_x conversion efficiency of the 0.25Pt/5Co/15Ba catalyst were better than those of 1Pt/15Ba, even with a greatly reduced noble metal content (three times less based on actual weight loadings). In general, the performance of the 0.25Pt/5Co/15Ba catalyst for NO_x conversion was better than that of 1Pt/15Ba under all operating conditions tested. Fig. 13 compares the model predictions of NO_x conversion for 1Pt/15Ba with 0.25Pt/5Co/15Ba catalyst at different cycle times and lean fractions; as can be seen, the model also predicts that the 0.25Pt-containing catalyst will outperform the 1Pt-containing catalyst. These results can again be attributed to the improved oxidative ability of the catalysts due

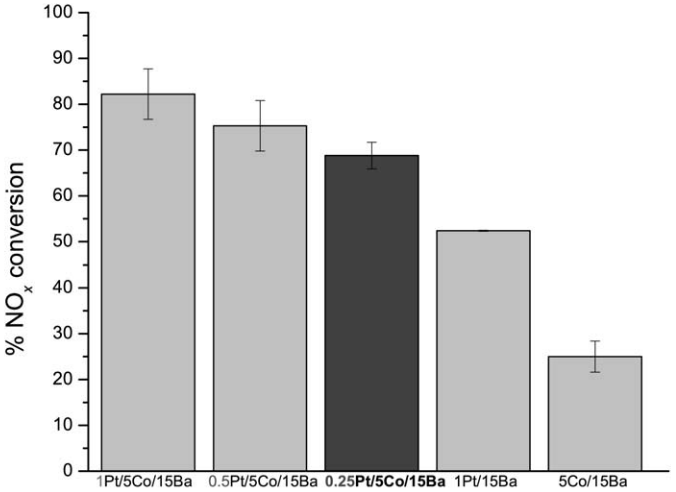

Fig. 11. NO_x conversion efficiency for different Co-containing NSR catalysts with lower weight loadings of Pt tested for a total cycle time of 90 s and a lean fraction of 0.92.

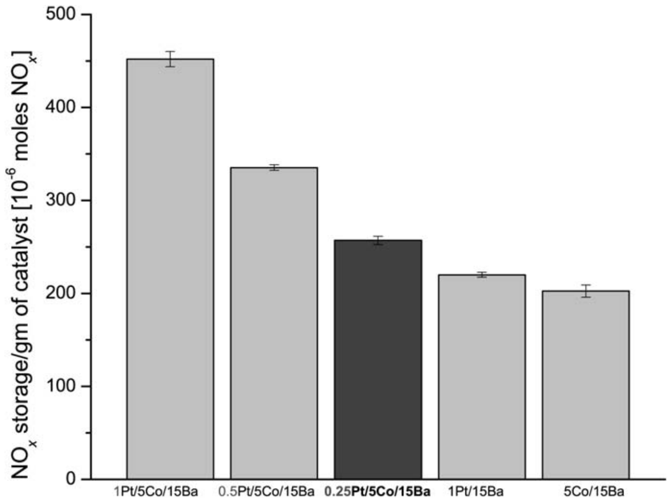

Fig. 12. Lean NO_x storage for different Co-containing NSR catalysts with varying Pt content for total cycle time of 45 min, with lean phase of 30 min, and rich phase of 15 min.

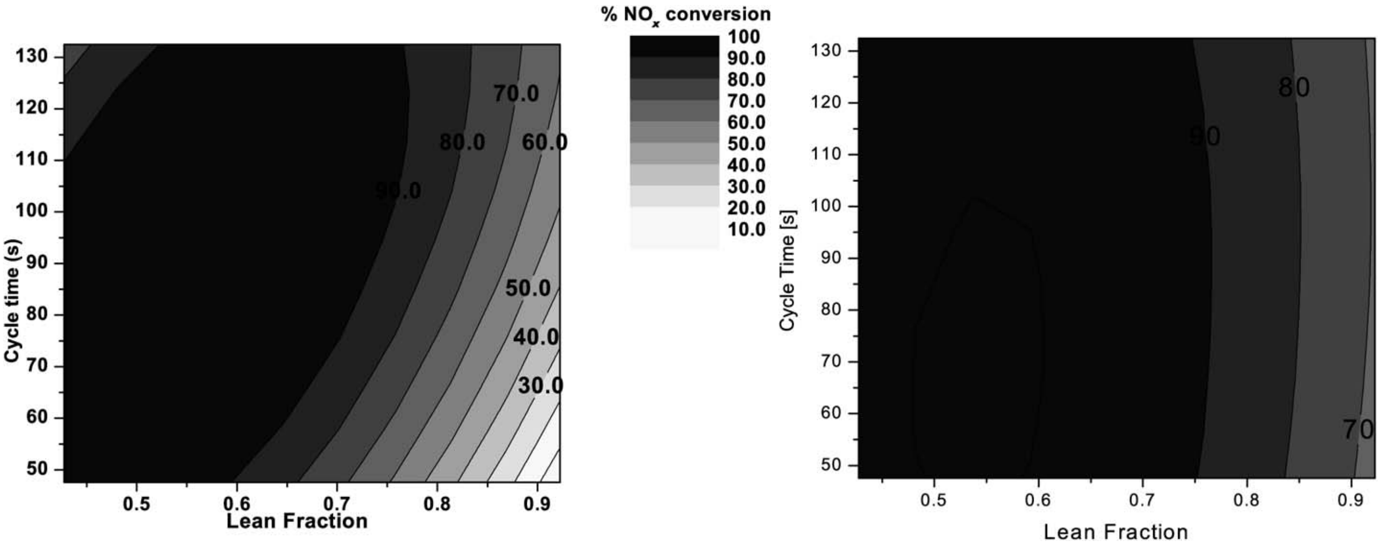

Fig. 13. Model predictions for NO_x conversion as a function of cycle time and lean fraction for 1Pt/15Ba and 0.25Pt/5Co/15Ba NSR catalysts.

to the presence of 5% Co, which compensates for the decreased storage due to the reduced noble metal content. Just 0.25% Pt was sufficient for catalyst regeneration in the rich phase. From the above results, it can be inferred that adding 5Co to 1Pt/15Ba catalysts allows for the reduction of noble metal content to 0.25% without affecting the overall catalyst performance, implying a substantial decrease in the cost of catalysts.

4. Conclusion

Co-containing NSR catalysts were found to outperform traditional Pt/Ba-based catalysts under both long-cycle and short-cycle conditions. Empirical models, developed using statistical design of experiments, predicts that adding 5% Co to 1Pt/15Ba or 1Rh/15Ba catalysts will expand the region of 100% conversion to higher lean fraction. It was also found that the reduction capacity of Co is not comparable to that of noble metals, such that noble metals are still necessary for catalyst regeneration in the rich phase. Adding 5% Co to 1Pt/15Ba allows for a reduction of the noble metal content to 0.25% without affecting the performance of the catalysts. Therefore, the addition of Co to NSR catalysts is expected to have a significant impact in terms of improving fuel efficiency as well as reducing the cost of catalysts.

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