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# Non-thermal plasma-assisted $NO_x$ reduction over alkali and alkaline earth ion exchanged Y, FAU zeolites

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## Abstract

The catalytic activities of a series of alkali and alkaline earth cation exchanged Y, FAU zeolites were investigated in the non-thermal plasma-assisted $NO_x$ reduction reaction using a simulated diesel engine exhaust gas mixture. The catalytic activity of the Y, FAU zeolite showed significant variations with both the nature of the charge compensating cation, and the method of catalyst preparation. Our results show that conventional multiple solution ion exchange is insufficient to prepare the most active catalyst for the given cationic form. The highest $NO_x$ conversion level was achieved over a Ba-Y, FAU which was prepared by a multiple ion exchange method, in which each solution ion exchange step was followed by a high temperature calcination. A systematic change in the catalytic activity was observed as a function of the charge density around the charge compensating cation. For both catalyst series (alkali and alkaline earth ion exchanged Y, FAU), the specific activity decreased with increasing electrostatic field around the charge compensating cation. The large difference in the $NO_x$ reduction activity at a given $e/r$ ratio, however, may suggest different reaction mechanisms for the two sets of catalysts. Indeed, there is a noticeable difference in the product distribution (selectivity) for the alkali and alkaline earth series of catalysts. Our results also reveal that extreme care must be taken when catalytic activities are compared for seemingly similar materials. We found that two base zeolite materials with identical Si/Al ratios, obtained from the same manufacturer but from different synthesis batches show significantly different catalytic behavior.

*Keywords:* Plasma-assisted $NO_x$ reduction; Alkali and alkaline earth ion exchanged Y, FAU; Catalyst preparation

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## 1. Introduction

Increased fuel efficiency of internal combustion engines and controlling vehicle exhaust emissions are the two major concerns in automotive engine development. An important way to enhance fuel efficiency is to operate engines under ‘lean’ conditions, i.e., in an excess of oxygen. Today diesel engines are the only ones that operate under truly oxidizing conditions. Unfortunately, ‘lean’ engine operation makes the efficient catalytic exhaust control a major challenge. In particular, traditional three-way catalysts are unable to reduce $NO_x$ under net oxidizing conditions. Therefore, there is a large effort worldwide to develop new concepts and catalysts for automotive after treatment to comply with the increasingly stringent environmental regulations [1]. One of the new, promising technologies is plasma-assisted $NO_x$ reduction. In this process a non-thermal plasma reactor is inserted between the engine and the catalyst bed. The chemistry inside the plasma reactor is complex, but fairly well understood [2,3]. It is concluded that the two most important reactions initiated by the plasma are the practically complete conversion of NO into $NO_2$, and the partial oxidation of unburned hydrocarbons in the exhaust gas. Thus, the important catalytic chemistry involves the reaction of $NO_2$ with these partially oxidized hydrocarbons. We have previously shown that aldehydes are particularly effective reductants for the catalytic conversion of $NO_2$ [4].

Balmer et al. [5] have recently reviewed the literature comparing various catalysts found to show some activity for $NO_x$ reduction when combined with a non-thermal plasma. More recently, two sets of catalysts have been found to be especially effective for $NO_x$ conversion in a plasma-treated exhaust gas stream: zeolite-based catalysts for light duty diesel engines [5–7] and alumina-based catalysts [8] for heavy-duty diesel engines. These catalysts have been shown to be active in different temperature regimes. The zeolite-based catalysts show the highest activity in the 473–523 K range, while the alumina-based materials work well above 623 K. Among the zeolite-based material Na-

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and Ba-ion exchanged Y, FAU materials showed the most promising activities in $NO_x$ reduction.

Here we report the results of a systematic study on the alkali and alkaline earth ion exchanged Y, FAU catalysts. The two key questions we sought answers to are: which cationic forms are the most active catalysts for plasma-assisted $NO_x$ reduction; how does the catalyst preparation influence the catalyst performance.

## 2. Experimental

The catalysts used in this study were prepared from a Na-Y, FAU zeolite (Si/Al ∼ 2.6), obtained from Zeolyst International Co. (CBV 100), by solution ion exchange. The Na-Y, FAU extrudates (1/16 in.) (containing approximately 10% alumina binder) were ion exchanged with aqueous solutions of the corresponding nitrate salts. For the preparation of Ba-Y samples, a $Ba(CH_3COO)_2$ solution was used due to the low solubility of $Ba(NO_3)_2$. The ion exchanges were carried at room temperature (295 K) for 48 h with solutions of 0.5 mol/l. Following the solution ion exchange the samples were thoroughly washed with distilled water and then dried at 393 K in air. Prior to each catalytic experiment, the samples were calcined at 773 K for 4 h in air.

Two basic catalyst preparation methods were established: in one method the starting Na-Y material was solution ion exchanged one to four times with the corresponding cation containing aqueous solution, washed with water and then dried and calcined. These materials are labeled as M-Y($x-1$), where $x$ represents the number of solution ion exchange ($1 \leq x \leq 4$), and M the alkali or alkaline earth cation. In the other method, after each solution ion exchange step the catalyst was washed, dried and calcined at 773 K for 4 h in air. These samples are designated as M-Y($x - y$), where $x$ represents the number of solution ion exchange and $y$ the number of high temperature calcinations ($1 \leq x = y \leq 4$). Catalyst compositions and ion exchange levels were estimated from ICP analysis of the prepared samples.

The $NO_x$ reduction activities of the catalysts were measured in a two-stage non-thermal plasma/catalytic reactor system described in detail previously [9]. Briefly, the system consisted of a dielectric barrier discharge between an array of oppositely polarized electrodes followed by a packed bed catalytic reactor. The electrical power source consisted of a high voltage sinusoidal wavefront, an audio amplifier and a high voltage transformer. A simulated exhaust gas mixture of 8% $O_2$, 2% $H_2O$, 245 ppm NO, and 520 ppm $C_3H_6$ in $N_2$ balance was used in the catalytic tests. The total flow rate was kept at 2.1 l corresponding to a space velocity of approximately 12000 $h^{-1}$. In the bottom portion of a quartz reactor, 10 ml catalyst was placed on top of quartz wool, and then the empty upper part of the reactor was filled with glass beads for efficient heat transfer. Typically, the reactor was first heated up to 443 K in flowing nitrogen without turning on the plasma. Upon reaching the desired reactor temperature, the gas flow was switched to the reactant gas mixture, and the $NO_x$ level was monitored with a chemiluminescent $NO_x$ analyzer (Rosemount, Model 955). As soon as the $NO_x$ levels stabilized, the plasma was turned on at a power level of 10 J/l (0.35 W). (The protocol for the measurement and adjustment of the plasma power were performed according to Tonkyn et al. [9].) Throughout the catalytic measurements, the $NO_x$ level ($NO + NO_2$) was monitored. Periodically the NO level was measured, allowing us to estimate the quantity of unconverted $NO_2$. During the catalytic tests the reaction temperature was raised form 443 to 563 K in 30 K increments. At each temperature, sufficient time was allowed to reach steady state (or near steady state).

## 3. Results and discussion

The maximum $NO_x$ conversion (defined as [total $NO_x$ out of the catalytic reactor]/[total NO into the plasma reactor] × 100) of the base Na-Y catalyst (as received from the manufacturer) is 55% and it is measured at a reactor temperature of 473 K. With increasing temperature the activity of Na-Y decreases gradually, and it drops to below 40% at 563 K. Substitution of $Na^+$ ions by $Ba^{2+}$ ions brings about a large increase in $NO_x$ reduction activity. The $NO_x$ conversion on Ba-Y(1-1) catalyst at 473 K is 74%, almost 20% higher than that of the base Na-Y. The $NO_x$ conversion as a function of reactor temperature is shown for Na-Y and a series of Ba-Y($x - 1$) ($1 \leq x \leq 4$) in Fig. 1. The temperature dependence of the $NO_x$ conversion is very similar for all these catalysts, going through a maximum at 473 K. Besides the large increase in $NO_x$ conversion upon $Ba^{2+}$ ion exchange, the most important observation from Fig. 1 is that the $NO_x$ conversion is practically the same for all $Ba^{2+}$-ion exchanged Y, FAU samples. This may suggest that, under

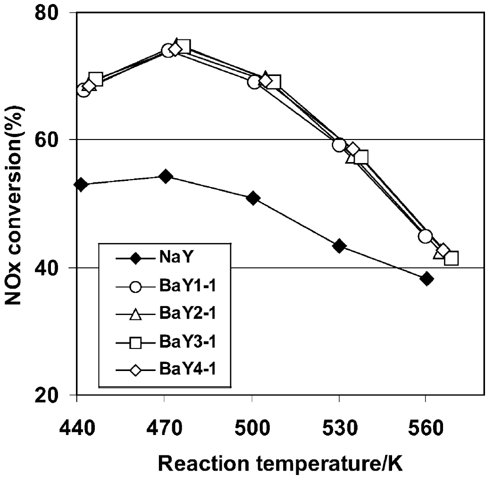

Fig. 1. The effect of multiple solution ion exchanges on the catalytic activities of Ba-Y, FAU zeolites. The Na-Y starting material was ion exchanged with $Ba(CH_3COOH)_2$ solution one to four times, and then the samples were calcined at 773 K for 4 h.

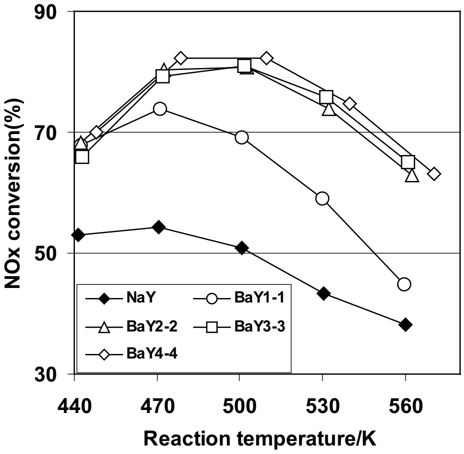

Fig. 2. The effect of solution ion exchange/high temperature calcination cycles on the catalytic activities of Ba-Y, FAU zeolites. (After *each* solution ion exchange step the catalyst was calcined at 773 K for 4 h.)

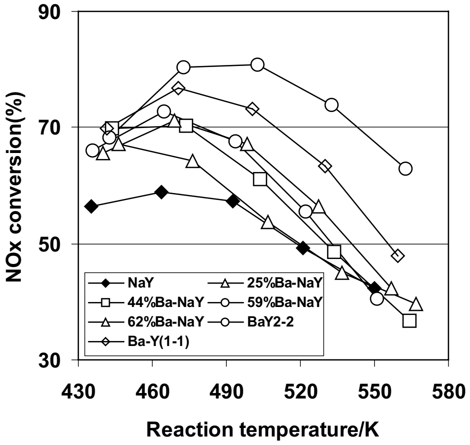

Fig. 3. The effect of ion exchange level in Ba (Na)-Y, FAU zeolites on the $NO_x$ conversion activity. The catalysts were prepared from solutions containing various concentrations of $Ba^{2+}$ ions.

the given ion exchange conditions, the first ion exchange is effective in replacing most of the accessible $Na^+$ ions with $Ba^{2+}$ ions.

Modifying the catalyst preparation conditions such that each solution ion exchange is followed by a high temperature calcination step results in significant changes in the $NO_x$ conversion levels for the thus prepared Ba-Y catalysts. $NO_x$ conversion as a function of reactor temperature is displayed in Fig. 2 for these Ba-Y($x - y$) ($1 \le x = y \le 4$) catalysts. The activities of the multiply ion exchanged Ba-Y($x - y$) catalysts are significantly improved when $x = y \ge 2$. $NO_x$ conversion over these catalysts is above 80% in the 473–513 K temperature range. As we have discussed in detail in our previous publication [10], the most probable explanation for this large activity increase is the cation redistribution inside the zeolite structure upon the high temperature calcination step of the catalyst preparation process. The consequence of this high temperature cation redistribution is that additional $Na^+$ ions become accessible for $Ba^{2+}$ ion exchange in successive ion exchange steps. The final outcome is an increased $Ba^{2+}/Na^+$ ratio, and higher $NO_x$ conversion. Note that the largest increase in activity is observed for the Ba-Y(2-2) sample. Additional solution ion exchange/calcination cycles result in only marginal improvements in $NO_x$ conversion. It is also worth mentioning that the multiple ion exchange/calcination preparation procedure produced catalysts with vastly improved catalytic performance in the high temperature regime studied here.

The results just presented demonstrate a vastly improved catalyst performance when Ba ions are substituted for $Na^+$ in Na-Y, FAU zeolites. To study the effect of the $Ba^{2+}/Na^+$ ratio on the $NO_x$ conversion in more detail, we prepared a series of Ba-Y/Na-Y catalysts where the extent of exchange was controlled. The samples were prepared by a single solution ion exchange followed by a high temperature calcination. The concentration of the $Ba^{2+}$ ions in the aqueous solution was changed in order to prepare a series of Ba-Y samples with different $Ba^{2+}/Na^+$ ratios. The percentages in the figure refer to the $Ba^{2+}$ ion exchange levels estimated for the results of ICP analysis. The $NO_x$ conversion as a function of reaction temperature for this series of catalysts is illustrated in Fig. 3. The figure also shows the results for Ba-Y(1-1) and Ba-Y(2-2) for comparison.

A more quantitative comparison of the reactivity of a series of Ba-Y catalysts for plasma-assisted $NO_x$ reduction can be made by plotting the specific catalytic activities, expressed as the number of $NO_x$ converted per supercage per second, as a function of the number of $Ba^{2+}$ ions per unit cell. Fig. 4 shows this comparison of the specific activities at a reaction temperature of 473 K. The catalytic activity of Ba-Y linearly increases with the average number of $Ba^{2+}$ ions present in a unit cell. This figure clearly shows that the Ba-form of the Y, FAU zeolite is intrinsically more active for plasma-assisted $NO_x$ reduction than the parent Na-Y.

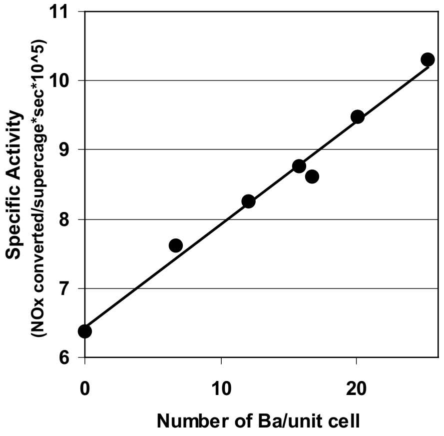

Fig. 4. Specific $NO_x$ reduction activity as a function of the number of $Ba^{2+}$ ions per unit cell. Activities are compared at a reaction temperature of 473 K.

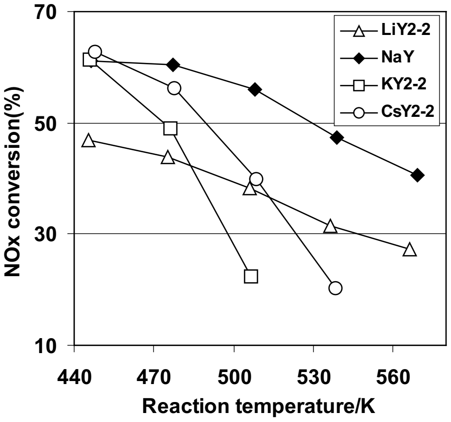

Fig. 5. $NO_x$ conversion for Li-, Na-, K-, and Cs-Y, FAU zeolites in the 443–563 K temperature range. All catalysts were prepared by two solution ion exchange/high temperature calcination cycles with the exception of Na-Y used as received.

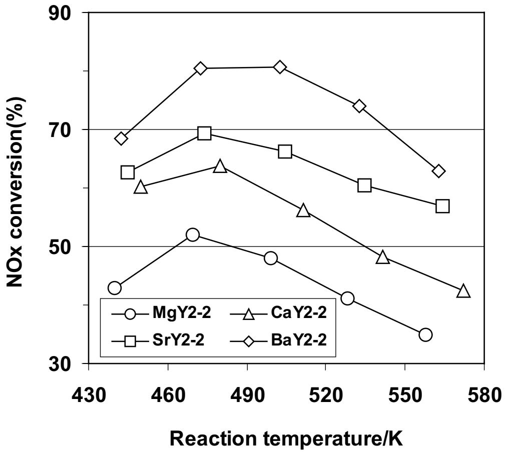

Fig. 6. $NO_x$ conversion for Mg-, Ca-, Sr-, and Ba-Y, FAU zeolites in the 443–563 K temperature range. All catalysts were prepared by two solution ion exchange/high temperature calcination cycles.

So far we have shown a clear correlation between the quantity of the cations present in cationic positions of Y, FAU zeolites and the activity of a given catalyst in the $NO_x$ reduction process studied. We have also demonstrated that the Ba-form of the Y, FAU is a much better catalyst in this process than the base Na-Y zeolite. Next we systematically investigated how the nature of the cation in the zeolite affects the $NO_x$ reduction activity. To this end we prepared two series of catalysts from the parent Na-Y material: alkali and alkaline earth ion exchanged ones. We chose the preparation method that gave very good results for Ba-Y, i.e., two solution ion exchanges, with high temperature calcination after each ion exchange step. The catalysts are designated as M-Y(2-2).

The $NO_x$ conversion as a function of temperature is shown in Fig. 5 for Li-, Na-, K-, and Cs-Y(2-2) catalysts. Practically complete ion exchange was achieved in the Li- and K-Y(2-2) preparation, while in the Cs-Y(2-2) case the ion exchange level was below 50%. At the lowest temperature studied (443 K) Na-, K-, and Cs-Y show practically the same activity toward $NO_x$ conversion, while Li-Y is much less active. With increasing reaction temperature the $NO_x$ conversion decreases for all four, alkali ion exchanged materials, however their trends are very different. Li-, and Na-Y show very similar trends, their $NO_x$ conversions decrease almost linearly with temperature. The $NO_x$ conversion plots for these two materials are parallel to each other in the entire temperature range studied. On the other hand, the trends in reactivity with temperature observed for the $K^+$ and $Cs^+$ ion exchanged catalysts are distinct. Their $NO_x$ reduction activities are high at low temperature, but drop very rapidly with increasing reaction temperature.

The $NO_x$ conversion results for the Mg-, Ca-, Sr-, and Ba-Y(2-2) samples are displayed in Fig. 6 as a function of reaction temperature. The trends for the alkaline earth ion exchanged zeolites are very different from that discussed above for the alkali ion exchanged ones. Here there is a clear activity trend in the conversion of $NO_x$, i.e., Mg $<$ Ca $<$ Sr $<$ Ba-Y(2-2) at all temperatures. The Mg-Y sample shows very low $NO_x$ conversion over the entire temperature range studied; its maximum activity of ∼52% is measured at 473 K. On the other hand, the Ba-Y catalyst is the most active among all the catalysts we have studied. As noted before, $NO_x$ conversion level over this catalyst in the 473–503 K temperature region is above 80%. The activities of the Ca-, and Sr-Y samples are between those of the Mg- and Ba-Y zeolites. The activity trend observed for the alkaline earth ion exchanged catalysts seem to correlate with the basicity of the cations in charge compensating positions of the zeolite framework.

The activity results for both sets of catalysts studied are summarized in Fig. 7. Here the specific catalytic activity, defined as the number of $NO_x$ converted per supercage per second, measured at 473 K reaction temperature is plotted as a function of charge density (charge/ionic radius, *e*/*r*). In this presentation of the activity data, there are clear trends

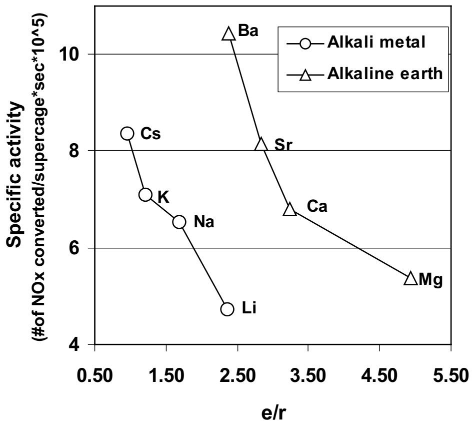

Fig. 7. Specific $NO_x$ reduction activity as a function of charge density around the charge compensating cations in the Y, FAU framework. Activities are compared at a reaction temperature of 473 K.

for both sets of catalysts studied here, i.e., the activity increases with decreasing *e*/*r*. This correlation seems to emphasize again the importance of the basic character of the catalysts in the plasma-assisted $NO_x$ reduction process. Although there is correlation between activity and *e*/*r* for each set of catalysts, there is a disconnect between the two sets of catalysts. This suggests that charge density around the charge compensating cation is not the only factor influencing the overall $NO_x$ reduction activity.

Another clear difference between the alkali and alkaline earth ion exchanged zeolites is the strength of $NO_2$ adsorption. As we have discussed previously [10] for the Na- and Ba-ion containing catalysts, $NO_2$ adsorbs much more strongly on the Ba-Y sample. For this latter catalyst material, we observed two desorption features in the $NO_2$ TPD, one at around 350 K and the other at 450 K. On the other hand, for Na-Y there was only one desorption feature at the same temperature as the low temperature desorption feature for Ba-Y. There are other possibilities that may contribute to the very different behavior observed for the two sets of catalysts. In the reactant gas mixture there is a significant amount (2%) of $H_2O$ present. It is well known, that alkali and alkaline earth ion exchanged zeolites interact with water differently. While alkali ion exchanged zeolites do not dissociate adsorbed water, alkaline earth ion exchanged zeolites do, consequently creating Brönsted acidic sites inside the zeolite channels under reaction conditions. This may significantly influence the interaction between the (partially oxidized) hydrocarbons and the zeolite. These differences between the two sets of catalysts may suggest a somewhat different mechanism by which these materials work in the $NO_x$ reduction process.

So far we have discussed the total $NO_x$ conversion levels for the two sets of catalysts, and drew comparisons on that basis only. It is also very useful to take a look at the $NO_2$ conversion levels (defined as [$NO_2$ out of the catalytic reactor]/[total NO into the plasma reactor] × 100) on these two catalyst series. For this, it is assumed, and previously confirmed by experiments [2], that NO is completely converted into $NO_2$ in the non-thermal plasma reactor. This means that the only $NO_x$ species that reaches the catalyst bed is $NO_2$. $NO_2$ is a very strong oxidizing agent and can directly oxidize hydrocarbons/partially oxidized hydrocarbons, while itself is converted back into NO. This is an undesirable process, since the thus formed NO will pass through the catalyst bed without taking part in the $NO_x$ reduction process. The desired processes for $NO_2$ are the ones in which it interacts with hydrocarbons on the catalyst and forms intermediates that eventually lead to the formation of $N_2$, and also some other N-containing compounds (e.g., HCN) that might be easily converted into harmless compounds in additional catalytic steps. Thus at a minimum, we can define two pathways for $NO_2$ over the M-Y zeolites: (1) catalytic $NO_2$ reduction to $N_2$, $N_2O$, HCN, etc.; (2) $NO_2$ to NO via reaction with the catalyst and/or a 'direct' reaction with hydrocarbonaceous species.

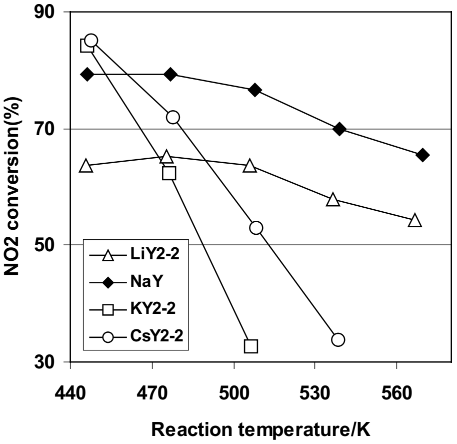

Fig. 8. $NO_2$ conversion for Li-, Na-, K-, and Cs-Y, FAU zeolites in the 443–563 K reaction temperature range.

The $NO_2$ conversion as a function of reaction temperature for the alkali ion exchanged zeolites is shown in Fig. 8. The trends observed for $NO_2$ conversion are strikingly similar to those seen for $NO_x$ conversion in Fig. 5. At low reaction temperature (473 K) both K- and Cs-Y(2-2) converts more than 80% of $NO_2$. At this high $NO_2$ conversion level we observed the highest $NO_x$ conversion activity as well. However, as the reaction temperature increased the $NO_2$ conversion over these two catalysts falls sharply, and above 500 K most of the $NO_2$ passes through the catalyst bed unreacted. Again, we see a very different trend for the other two alkali ion exchanged zeolites, Li- and Na-Y. Their $NO_2$ conversion activity is the highest at 473 K, and gradually decreases with increasing reaction temperature. The similarity between Li- and Na-Y on the one hand, and for K- and Cs-Y on the other hand is striking. It is currently not clear why these two sets of catalysts (all belonging to the alkali metal ion exchanged zeolite group) behave so differently in this reaction. The very high activity for the K- and Cs-Y sample at low temperature may suggest that the intrinsic activities of these cationic forms are very high. In fact, they might be so active, that they form some type of low reactivity surface intermediate at higher temperature, which ultimately leads to their very fast deactivation. Current in situ FT-IR reactor experiments are aimed at addressing this issue.

The $NO_2$ conversion data for the alkaline earth ion exchanged Y zeolites are displayed in Fig. 9. The trends for these catalysts are very different from those we just discussed for the alkali ion exchanged ones. The most important observation is the very high level of $NO_2$ conversion for all of the alkaline earth ion exchanged samples. Except for Mg-Y, all the other catalyst show $NO_2$ conversion levels above 90% over the entire temperature range studied. Also note that the $NO_2$ conversion is very stable, it hardly changes with increasing reaction temperature. These results show that the interaction between $NO_2$ and the alkaline earth ion exchanged Y zeolites is strong. From the results of Figs. 6 and 9, we can deduce that a significant fraction of the $NO_2$

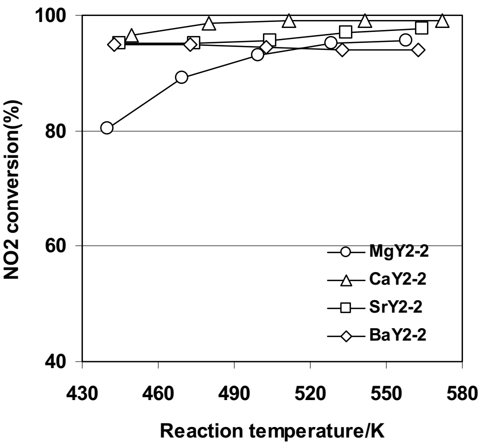

Fig. 9. $NO_2$ conversion for Mg-, Ca-, Sr-, and Ba-Y, FAU zeolites in the 443–563 K temperature range.

that arrives onto the alkaline earth ion exchanged Y zeolites gets converted back to NO. Since the $NO_2$ conversion is almost 100% over these catalysts, we can conclude that with increasing reaction temperature an increasing fraction of the incoming $NO_2$ is converted back to NO, resulting in a decreased overall $NO_x$ conversion at higher temperatures.

In the limited literature on plasma-assisted catalysis, significant variations can be seen for apparently similar materials and reaction conditions. To this end we evaluated two Na-Y, FAU zeolites (CBV 100) of Si/Al $\sim$ 2.55, received from the same manufacturer (Zeolyst). Chemical composition, Al and Si NMR analyses showed no difference between the two materials. However, they behaved significantly differently for the plasma-assisted reduction of $NO_x$. The $NO_x$ conversion, NO, and $NO_2$ yields for these two Na-Y samples are compared in Fig. 10. The Na-Y catalysts from batch number 1825-59 shows an average of 10% higher $NO_x$ conversion than the one for batch number SX 0499-002. Although the trends in $NO_x$ conversion, NO and $NO_2$ yields are very similar for these two Na-Y samples, the absolute values are significantly different. We also prepared, under the same conditions, the alkali and alkaline earth ion exchanged Y zeolite catalyst series from both base materials, and for each pair of samples we observed the same differences in $NO_x$ conversion, NO and $NO_2$ yields (results are not shown). At present the root cause of this variance from batch to batch is unknown, however, these results serve as a caution to those attempting to quantitatively compare catalytic results obtained from materials that are thought to be very similar.

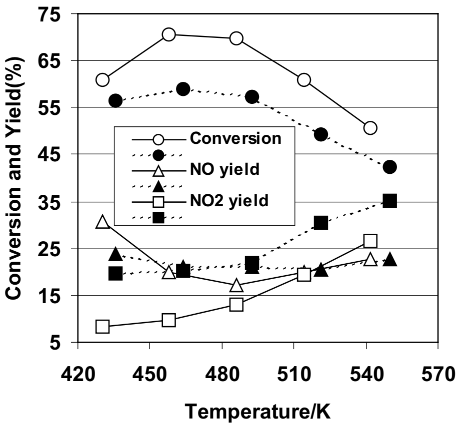

Fig. 10. Comparison of $NO_x$ conversion, NO and $NO_2$ yields for two Na-Y, FAU starting materials. The chemical compositions of the two catalysts are the same.

## 4. Conclusions

Alkali and alkaline earth ion exchanged Y, FAU zeolites are effective catalysts in the plasma-assisted $NO_x$ reduction process. In particular, Na-Y in the alkali series, and Ba-Y in the alkaline earth series show very promising activity for this reaction. The significant differences observed for both $NO_x$ and $NO_2$ conversions for the two series of catalysts suggests that the reaction pathways may differ depending on the nature of the charge compensating cation in the zeolite. We have shown that that the performance of a catalyst may be significantly influenced by the preparation method. Multiple solution ion exchange is insufficient for the preparation of the most active catalysts. High temperature calcination following each solution ion exchange step is necessary to obtain the highest catalytic activities. Our results also suggest that quantitative comparison among the activities of seemingly similar catalysts must be handled with extreme care. Here we presented results for two “identical” Na-Y materials that exhibited very different catalytic activities.

## Acknowledgements

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