The deactivation of an NH$_3$-SCR Cu-SAPO catalyst upon exposure to non-oxidizing conditions

R. Villamaina[a], I. Nova[a], E. Tronconi[a,*], T. Maunula[b], M. Keenan[c]

[a] Department of Energy, Laboratory of Catalysis and Catalytic Processes, Politecnico di Milano, 20156, Milano, Italy
[b] Dinex Ecocat Oy, Global Catalyst Competence Centre, P.O. Box 20, FI-41331, Vihtavuori, Finland
[c] Ricardo UK Ltd, Shoreham Technical Centre, Shoreham-by-Sea, BN43 5FG, UK

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ABSTRACT

A Cu-SAPO catalyst for NH$_3$-SCR applications showed a significant loss of deNO$_x$ performance after exposure to oxygen-free conditions. The present work aims at elucidating the causes of the observed progressive deactivation by comparing different experimental procedures for the SCR activity tests. The adoption of an experimental protocol, which avoids the exposure to a non-oxidizing environment, ensured a stable activity of the Cu-SAPO catalyst. Moreover, treatment of the deactivated catalyst with an oxidizing mixture at 550 °C for 5 h enabled to partially recover the deNO$_x$ activity.

1. Introduction

Nowadays, the NO$_x$ emissions produced by lean-burn diesel engines are controlled by the NH$_3$ Selective Catalytic Reduction (NH$_3$-SCR) technology, which allows the conversion of nitrogen oxides to harmless N$_2$ and H$_2$O over suitable catalysts through the injection of urea, which provides NH$_3$, the reducing agent of the process, directly in the SCR converter. The common SCR catalysts used until now are based on V-oxides, Fe-zeolites or Cu-zeolites [1–4]. Currently, the state-of-the-art zeolite-based catalysts are made of the small-pore chabazite (CHA) structure, which can exist as SSZ-13 or SAPO-34 on the basis of its chemical composition, exchanged with Cu ions. These catalysts are nowadays largely employed due to their very good activity, already in the low-temperature region, and their outstanding hydrothermal durability [5–11]. Nevertheless, they can be prone to a loss of activity and selectivity if they are exposed to specific conditions. For example, Cu-SAPO-34 catalysts showed poor low-temperature hydro-stability under conditions relevant to NH$_3$-SCR (wet conditions between r.t. and 100 °C) [12] and they were susceptible towards poisoning by SO$_2$ [13–17]. Moreover, hydrothermal treatments (at temperatures higher than 600 °C) may determine changes in their physical and chemical properties, which could negatively affect their NH$_3$-SCR performances [18].

An experimental study of the deNO$_x$ activity over a Cu-SAPO catalyst, however, has recently brought us to observe a progressive loss of its catalytic activity, which seems not connected with the deactivation processes already reported in the literature. Thus, this work aims at investigating the causes of this phenomenon, and at developing a procedure that enables to recover the original catalyst performances.

2. Experimental

SCR runs were performed over a thermally stable Cu-SAPO monolith catalyst, supplied by Dinex Finland [19]. A small amount (< 15 wt-% of the coating) of binder was mixed to the zeolite catalyst in the wash-coat. The tested sample consisted of a cylindrical rolled metallic substrate whose flat and corrugated metal foils were coated with the Cu-SAPO catalyst layer (length = 20 mm, diameter = 12.5 mm, volume = 2453 mm^3, coating = 0.39 g). The catalyst was hydrothermally (HT) aged at 700 °C for 20 h in air flow with 10% of water [19].

The sample, wrapped with a tape of inert quartz and plugged in the central hole with inert quartz wool in order to avoid by-pass of gases, was loaded in a stainless-steel tubular reactor. The catalyst was topped with quartz spheres, and by a quartz wool layer, to increase the turbulence and thus ensure a good mixing of the reactants. The reactor tube (405 mm in length, 15 mm i.d.), containing the catalyst sample, was inserted in a cylindrical electric oven, whose temperature (up to 550 °C) was remotely controlled by a PID controller (Eurotherm model 2132). The reactor was equipped with three K-type thermocouples: one was used to monitor the inlet gas temperature and two were placed in

* Corresponding author.
 E-mail address: enrico.tronconi@polimi.it (E. Tronconi).

contact with the top and the bottom of the catalyst.

Steady state runs were carried out to investigate the catalytic activity under Standard SCR conditions. The feed concentrations were representative of real after treatment systems: NH_3 = 500 ppm, NO_x = 500 ppm (NO_2/NO_x = 0), O_2 = 5% (v/v), H_2O = 5% (v/v) and balance N_2. H_2O was metered by a volumetric piston pump (Gilson model 305): the feed rate was around 0.025 ml/min +/− 0.0001 for GHSV = 75,000 h^{-1}. Afterwards, the liquid feed was vaporized in a hot pipeline kept at 190 °C, and then mixed with the other gaseous species and fed to the reactor (H_2O feed concentration = 5% v/v).

A wide range of temperatures (150–550 °C) was investigated. All the experiments were carried out at a GHSV of 75,000 h^{-1}. The GHSV was calculated as the volumetric flow rate divided by the overall volume of the monolith catalyst.

All the gaseous species (except N_2) were continuously monitored at the reactor outlet by a FT-IR gas analyzer (Bruker MATRIX MG5).

3. Results and discussion

3.1. Replicated Standard SCR runs: Cu-SAPO deactivation

The catalytic activity of the Cu-SAPO catalyst was investigated evaluating the steady-state NH_3 and NO conversions as well as the N_2O production at fixed temperatures in the range 150–550 °C under Standard SCR conditions (NH_3 = 500 ppm, NO = 500 ppm, O_2 = 5% (v/v), H_2O = 5% (v/v) and balance N_2, GHSV = 75,000 h^{-1}). Specifically, a first set of replicated experiments was carried out following the experimental procedure shown in Fig. 1. The gaseous mixture (containing NH_3, NO, H_2O, O_2 and N_2) was fed to the reactor at the lowest temperature (150 °C) and, once the outlet concentrations reached a steady state, the temperature was increased in steps of 50 °C up to the maximum investigated level (550 °C) in order to collect steady-state concentrations at different temperatures. At the end of the test, the gaseous feed mixture was switched off and the catalyst was cooled down to room temperature in only N_2 stream. Then, the stepwise T-ramp protocol was restarted.

Fig. 2 shows steady-state NO and NH_3 conversions and N_2O concentrations obtained from this first set of nine replicated Standard SCR runs using the experimental procedure presented above.

The deNO$_x$ performance in Run1 (purple lines) (already shown in our previous work [20]) were in line with that typical and well known of a Cu exchanged small pore zeolite catalyst [21], i.e. a very high deNO$_x$ efficiency reached already in the low T-range. Concerning N_2O (Fig. 2C), two temperature regions of production can be identified: a low-T region in which a N_2O peak centered at 250 °C can be detected, and a high-T region in which the N_2O formation increases with

temperature, reaching its maximum production at the maximum investigated temperature. It is already known [22,23] that the N_2O formation over Cu-exchanged zeolites proceeds at low or high temperature according to different mechanisms, which could involve different active sites and different reactions. For example, comparing the N_2O yield obtained from the NH_3 oxidation reaction (not shown) with that one of the Standard SCR reaction, only the high-T branch was found in common, thus indicating that the N_2O pathway at high temperature can be ascribed to the non-selective NH_3 oxidation to N_2O. On the contrary, at low temperature the N_2O evolution was more likely connected to the decomposition of NH_4NO_3 formed below 200 °C, which then decomposed at temperature higher than 200 °C [23]. Even if the role of zeolite pore size in the low-T N_2O formation was confirmed by testing different zeolite frameworks [22,23], the role of Cu ions in the ammonium nitrate deposition and decomposition to gaseous N_2O is still not clarified.

After several tests replicated still using the same procedure (Fig. 1), a significant deactivation was observed when the Standard SCR test was again performed under the same conditions of Run1 (Run 6 – blue lines in Fig. 2). Consequentially, replicated Standard SCR tests were further performed to confirm this deactivation phenomenon: each time the data showed a significant and progressive decline in the activity over the whole temperature range, clearly visible in Fig. 2A and B, Runs 7 - 9. For example, the NO conversion at 300 °C dropped from 85% to less than 20%, from the first to the ninth run (Fig. 2B). It is also worth noticing the differences observed in the N_2O profile (Fig. 2C): the low-T N_2O peak observed in Run1 disappeared in all the replicated runs, while the high-T pattern was preserved but with a progressively lower formation of this by-product. Even though the role of Cu sites in the deposition and decomposition of NH_4NO_3, responsible for the N_2O release at low temperature, is still uncertain, the disappearance of one N_2O formation route together with the pronounced and progressive loss of the deNOx catalytic activity could be jointly ascribed to some loss or modification of the active sites for the SCR reactions at each performed test.

3.2. Identification of the causes for the deactivation phenomenon

Being the ammonia storage capacity a fundamental parameter for the deNO$_x$ performance of SCR catalysts and, moreover, also relevant for the estimation of the amount of the active acid sites involved in the SCR reactions, we verified if the declined SCR efficiency might be correlated to a drop of this parameter. At the beginning of each Standard SCR test, after feeding the mixture containing 5% O_2 and 5% H_2O (+ balance N_2) at 150 °C, 500 ppm of NH_3 were fed to the reactor. When the saturation of the catalyst was reached (namely when the NH_3 outlet concentration was equal to NH_3 feed level), NO (500 ppm) was further fed to light off the Standard SCR reaction. With this procedure, the evaluation of the NH_3 storage capacity under wet conditions at 150 °C was possible prior to each Standard SCR test. The NH_3 adsorption capacity evaluated at the beginning of the Standard SCR test is illustrated in Fig. 3 for all the nine runs of the first set of Std SCR experiments, together with the corresponding steady state NH_3 conversions collected at several temperatures, regarded as an index of the SCR activity at each run. In comparison to the strong and continuous catalyst deactivation towards NO$_x$ reduction, the NH_3 storage capacity did not seem affected as much as seen for the activity. Indeed, passing from Run1 to Run6 there was only a slight decrease of this parameter, from 1.12 mmol/g_{cat} to 0.93 mmol/g_{cat}. Then, even when the activity drop was more important, the adsorption capacity stayed quite constant, assuming the final value of 0.86 mmol/g_{cat}. Since the ammonia storage capacity was still significant and in line with the one estimated on commercial Cu zeolite catalysts [21,22], the loss of activity does not seem correlated to a loss of the active acid sites. Moreover, the comparison of NH_3-TPD profiles (recorded after NH_3 adsorption at 150 °C under dry conditions) collected on the fresh sample and after its deactivation did not provide evidence of possible changes in the nature of
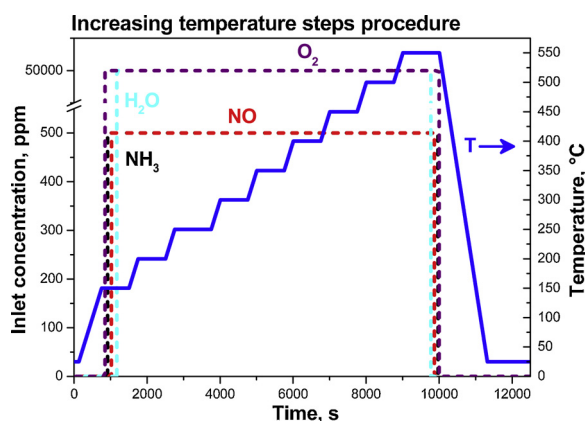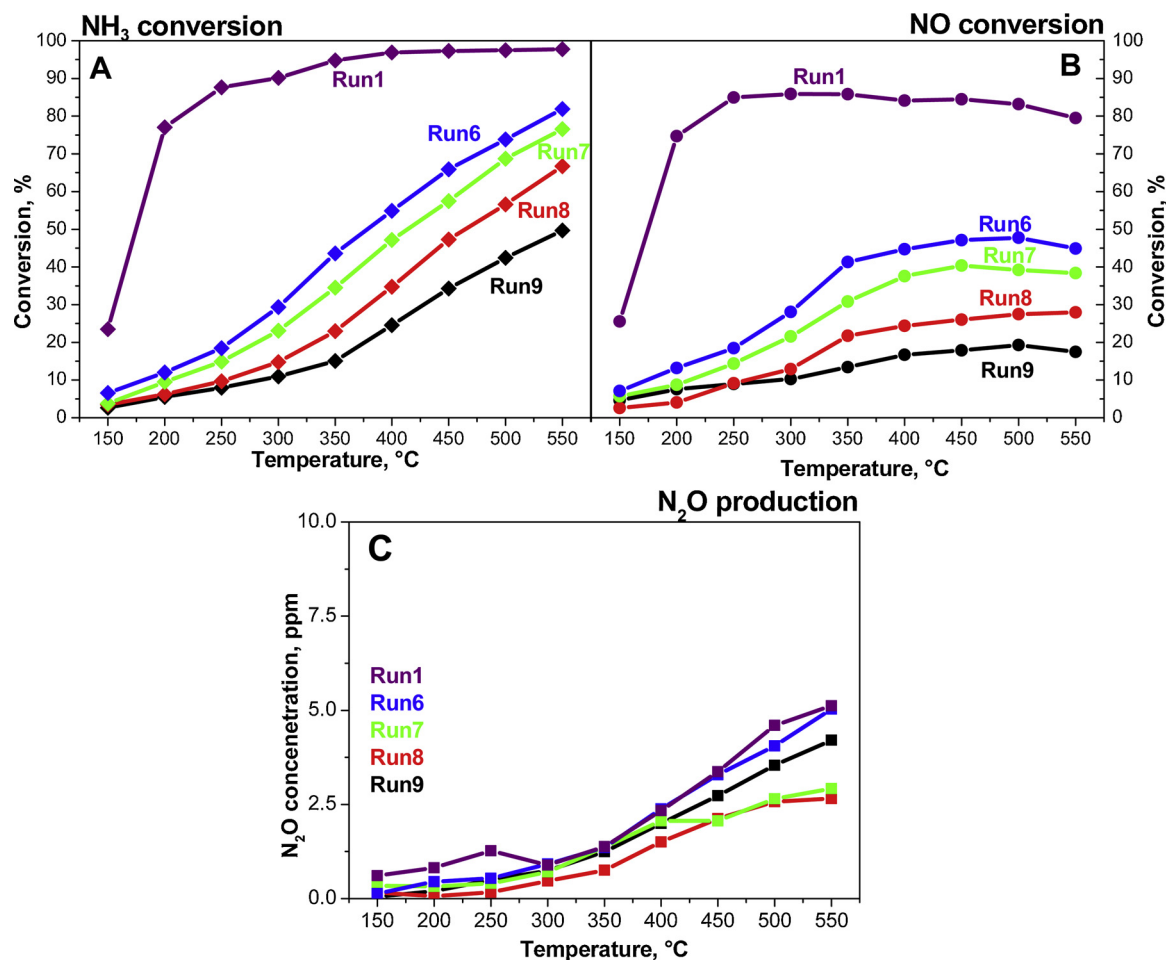

Fig. 1. Experimental procedure adopted for the Standard SCR runs: increasing temperature steps procedure. GHSV = 75,000 h^{-1}, NH_3 = 500 ppm, NO = 500 ppm, H_2O = 5% v/v, O_2 = 5% v/v, T_{range} = 150–550 °C, N_2 balance.

Fig. 2. Progressive deactivation of Cu-SAPO catalyst under Standard SCR conditions adopting increasing T-steps (first sample): A) NH_3 steady state conversions, B) NO steady state conversions, C) N_2O steady state concentrations. GHSV = 75,000 h^{-1}, NH_3 = 500 ppm, NO = 500 ppm, H_2O = 5% v/v, O_2 = 5% v/v, T_{range} = 150–550 °C, N_2 balance. (For interpretation of the references to colour in the text, the reader is referred to the web version of this article.)
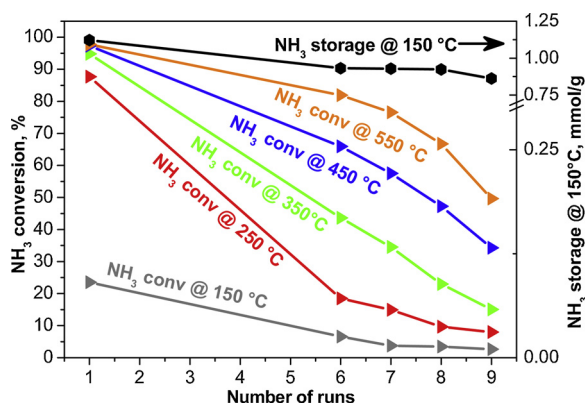
Fig. 3. NH_3 conversions at fixed temperatures (150, 250, 350, 450, 550 °C) and NH_3 storage capacity (mmol/g) at 150 °C evaluated from the first set of replicated Standard SCR reactions on the first catalyst sample. GHSV = 75,000 h^{-1}, NH_3 = 500 ppm, NO = 500 ppm, H_2O = 5% v/v, O_2 = 5% v/v, T_{range} = 150–550 °C, N_2 balance.

the catalyst acid sites. Indeed, only a slight decrease of the intensity of the NH_3 desorption peak was noted, while its area, representative of the NH_3 storage capacity on the Cu-SAPO catalyst, remained essentially similar after deactivation.

In order to understand what else had contributed to the observed marked drop of deNOx efficiency, several operating procedures were

then tested to confirm or exclude some of the already known deactivation causes of Cu-SAPO catalysts. Indeed, previous literature works had already identified the causes that could adversely affect the deNO$_x$ performance of Cu-SAPO catalysts, such as the exposure of the catalyst either to water at low temperature [12] or to hydrothermal treatments at temperature above 600 °C [18]. Concerning the latter, Ma et al. [18] showed that Cu-SAPO 34 catalysts were more robust than Cu-SSZ13 catalysts towards the exposure to high temperatures (600–850 °C) in a stream containing water, oxygen and CO_2. Indeed, from replicated Standard SCR tests they observed no big changes in the deNO$_x$ performance and in the formation of side products, such as N_2O, as well. Considering that our catalyst sample was not exposed to those harsh aging conditions and that, even if it was, its catalytic activity would not have been negatively affected [18], we examined other possible origins for the observed deactivation. To this purpose, two more sets of Standard SCR experiments were performed adopting two different experimental protocols, in order to discriminate if the observed deactivation could be linked to: i) the exposure of the catalyst to water at low temperature, as proposed by Leinstar et al. [12], or ii) the exposure of the catalyst to non-oxidizing conditions during cooling and heating phases.

The first possible cause was verified following the same protocol as in Fig. 1A, but this time avoiding the experimental investigation in the low temperature range (T < 250 °C). Accordingly, the Cu-zeolite sample was exposed to water only at temperatures high enough to ensure the absence of liquid water in the zeolite pores, thus preventing the deterioration of the crystalline structure by the action of water [24].
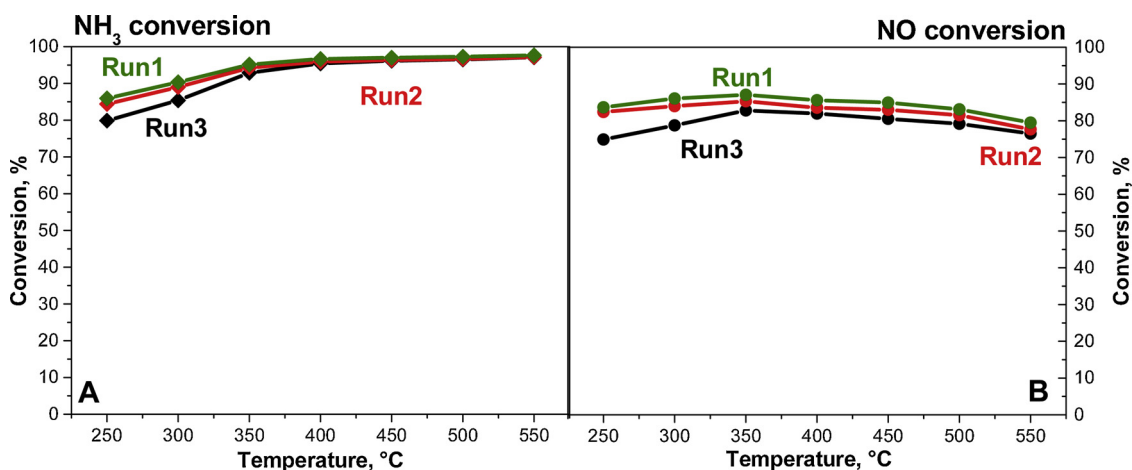
Fig. 4. Progressive deactivation of Cu-SAPO catalyst under Standard SCR conditions avoiding the exposure of H_2O at T $<$ 250 °C and adopting the increasing T-steps procedure (second sample): A) NH_3 steady state conversion, B) NO steady state conversion. GHSV = 75,000 h^{-1}, NH_3 = 500 ppm, NO = 500 ppm, H_2O = 5% v/v, O_2 = 5% v/v, T_{range} = 250–550 °C, N_2 balance.

The results from three consecutive replicated tests under Standard SCR conditions in the 250–550 °C T-range are displayed in Fig. 4: it is apparent that this careful procedure did not prevent the drop in the activity. Indeed, both NO and NH_3 consumptions decreased from run to run, then ruling out H_2O exposure at low temperatures as a deactivation cause. However, Leinstar et al. [12] observed an activity loss above all when the temperature was below 100 °C, where the catalyst is certainly in presence of liquid water. Moreover, they also verified that the catalyst deactivation did not involve a decline in the ammonia storage capacity, proposing, at the end, that the likely cause of the loss of SCR activity could be the transformation of the copper sites into inactive form [12].

It is worth noticing that the activity loss apparent in Fig. 4 is associated with three consecutive Standard SCR experiments. On the other hand, the greater activity loss observed between the first three tests in Fig. 2 is not related to three consecutive tests. In fact, between Run 1 and Run 6 in Fig. 2 other Standard SCR tests were carried out using a different space velocity (therefore not included in Fig. 2), which further contributed to the catalyst deactivation.

Being the catalyst sample cooled down to room temperature in N_2 flow at the end of each experiment, and observing the deactivation at the beginning of each successive test, the experimental procedure was changed in order to evaluate if the exposure to non-oxidizing condition could affect the catalyst performance. To this purpose, the catalyst was firstly heated up to 550 °C in a $O_2 + N_2$ mixture, then the Std SCR was carried out between 550 and 150 °C with decreasing temperature steps, and finally the catalyst was cooled down to room temperature again feeding again $O_2 + N_2$, according to the experimental protocol illustrated in Fig. 5. Through this protocol, the catalyst was never exposed to a non-oxidizing atmosphere.

When tested according to this new protocol, the catalyst was found to be stable in terms of activity: a very good reproducibility of deNOx efficiency was noted at all the investigated temperatures in replicated Standard SCR tests (Fig. 6).

To confirm that the non-oxidizing conditions played a key role in the observed catalyst deactivation, another set of Standard SCR runs was performed alternating each activity test with a pre-treatment in N_2 flow (Fig. 7). Run1 shows the deNO$_x$ performance of the fresh catalyst. Then the catalyst was subjected to a heating treatment in N_2 flow from room temperature to 550 °C (heating rate: 15 °C/min) followed by a cooling transient to r. t. in the same gas flow (Run 2). As a result, the decline of the catalyst activity after this pre-treatment resulted to be more pronounced (Run 3 - Fig. 7) than observed repeating the SCR reaction after only the cooling in N_2 (Run 2 - Fig. 4). To confirm the instability of the catalyst in the absence of O_2 another pre-treatment in
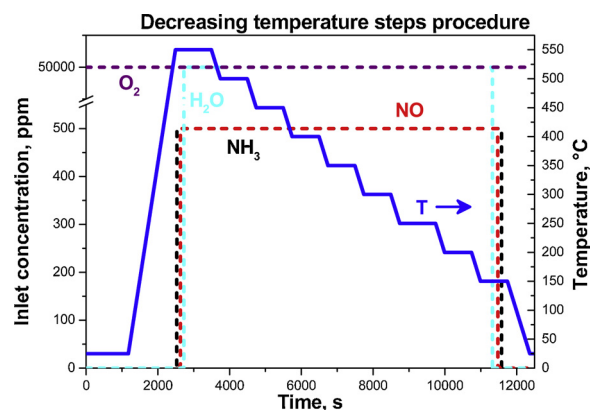

Fig. 5. Experimental procedure adopted for the third set of Standard SCR runs: decreasing temperature steps. GHSV = 75,000 h^{-1}, NH_3 = 500 ppm, NO = 500 ppm, H_2O = 5% v/v, O_2 = 5% v/v, T_{range} = 150–550 °C, N_2 balance.

N_2 was repeated (Run 4): the following Standard SCR test (Run 5) showed a further deactivation.

Other dedicated transient tests were carried out to assess the deactivation of the catalyst when oxygen was not present in the gas mixture. Specifically, under Standard SCR conditions oxygen was turned off at different temperatures to expose the Cu-SAPO catalyst to reducing conditions ($NH_3 + NO$ mixture) for different time lengths.

When the exposure to the $NH_3 + NO$ mixture lasted no longer than 5 min, the catalyst was able to restore its initial activity, reaching again the initial reactant conversions. Fig. 8 shows the dynamics of the outlet concentrations during one of the reducing/oxidizing cycles no longer than 5 min at 550 °C. During the experiment the catalyst was kept at 550 °C, feeding continuously 500 ppm of NH_3, 500 ppm of NO, 5% (v/v) H_2O, while oxygen (5% v/v) was alternatively switched off and on. At this high temperature, NH_3 was totally converted due to both the SCR and the oxidation reactions. NO, instead, was still present at that temperature as its conversion was stoichiometrically limited due to the NH_3 oxidation. After several switches from oxidizing conditions to oxygen-free conditions, the deNOx efficiency was restored: indeed, the NO concentration leveled off to its starting value, corresponding to almost 80% conversion.

When, instead, the reducing period was extended to at least 30 min, the catalyst was not able to recover its previous deNOx efficiency when the oxygen was fed again. Fig. 9 illustrates how the NH_3 and NO concentrations changed upon exposures of the catalyst to reducing conditions of different lengths. After only 30 min in absence of O_2, the NO
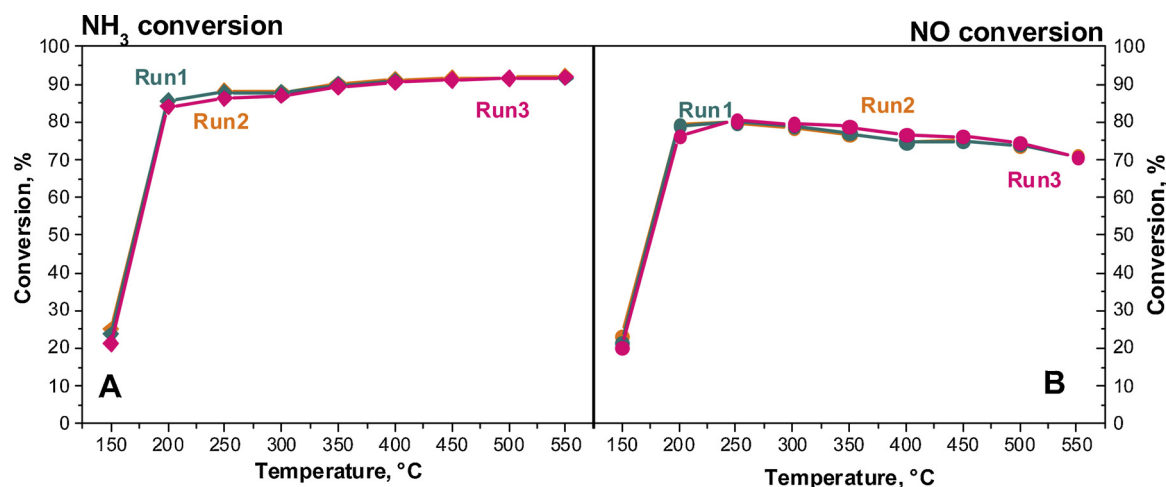
Fig. 6. Replicated Standard SCR reactions adopting the decreasing T-steps procedure (third sample): A) NH_3 steady state conversion, B) NO steady state conversion. GHSV = 75,000 h^{-1}, NH_3 = 500 ppm, NO = 500 ppm, H_2O = 5% v/v, O_2 = 5% v/v, T_{range} = 150–550 °C, N_2 balance.

conversion dropped from 80% to 72% while the NH_3 conversions stayed close to 100%. Further exposure to an O_2-free gas mixture for 1 h led this time to a greater deactivation: the NO conversion dropped to 60%. An even longer reducing treatment did not affect further the deNO$_x$ efficiency.

Finally, the last part of the experiment was an attempt to regenerate the catalyst by feeding a high concentration of oxygen (15% w/v) for 5 h. As shown in Fig. 9, the exposure to oxidizing conditions enabled the almost complete recovery of the activity, reestablishing a NO conversion very close to that one measured before the reducing treatment.

The observed catalytic activity loss is possibly linked to a change in the nature of the active Cu sites, no longer active towards the SCR reactions, when Cu-SAPO is exposed to reducing conditions or to only N_2 flow. In particular, a greater activity loss was detected when the catalyst was exposed to inert conditions during heating/cooling phases with respect to that observed exposing the catalyst to reducing conditions at fixed temperature. Accordingly, temperature cycles could contribute to further stressing the catalyst and promoting the deactivation of its active sites. Of course, in order to elucidate what happens in those conditions and to understand the mechanism behind this deactivation, further catalyst characterization tests will be necessary.

However, inert N_2 flow is an extreme condition to study the Cu-SAPO properties. It was found in another study that slightly rich conditions (λ = 0.98, 650 °C, 30 min) did not deactivate the same Cu-SAPO
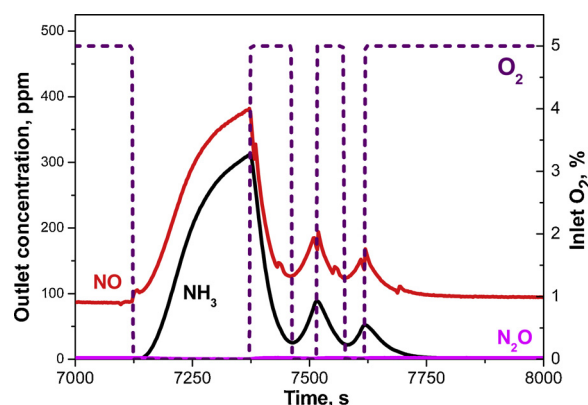

Fig. 8. Effect of reducing/oxidizing cycles on Standard SCR activity at 550 °C: NH_3, NO, N_2O outlet concentrations. GHSV = 75,000 h^{-1}, NH_3 = 500 ppm, NO = 500 ppm, H_2O = 5% v/v, O_2 = 0–5% v/v, N_2 balance, T = 550 °C.
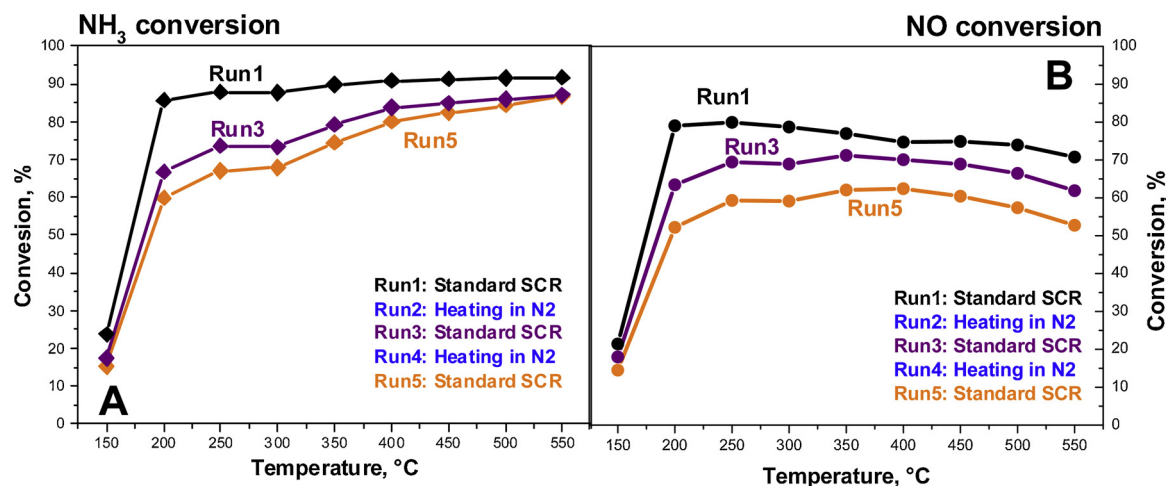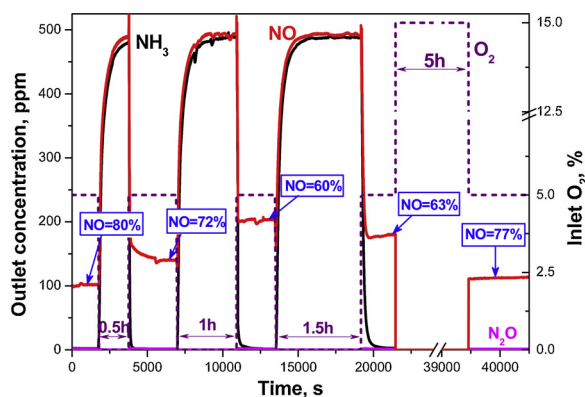

Fig. 7. Progressive deactivation of Cu-SAPO catalyst under Standard SCR conditions after each heating/cooling phase in N_2 flow (third sample): A) NH_3 steady state conversion, B) NO steady state conversion. GHSV = 75,000 h^{-1}, NH_3 = 500 ppm, NO = 500 ppm, H_2O = 5% v/v, O_2 = 5% v/v, T_{range} = 150–550 °C, N_2 balance.

Fig. 9. Effect of reducing conditions on Standard SCR activity at 550 °C and recovery of deNO$_x$ activity after a following stronger oxidative treatment: NH$_3$, NO, N$_2$O outlet concentrations. GHSV = 75,000 h^{-1}, NH$_3$ = 500 ppm, NO = 500 ppm, H$_2$O = 5% v/v, O$_2$ = 0–15% v/v, N$_2$ balance, T = 550 °C.

catalyst, but instead desulfated the catalyst, which almost fully recovered its initial activity without sulfur [19].

4. Conclusions

We have replicated Standard SCR reaction tests several times over a Cu-SAPO catalyst adopting different experimental protocols to discriminate the causes of a significant progressive activity loss observed after each SCR experiment. Only maintaining the catalyst in an oxidizing environment (O$_2$ = 5% v/v) it was possible to preserve its well-known high deNO$_x$ performance. Instead, when the Cu-SAPO catalyst was exposed either to reducing conditions (oxygen-free NO + NH$_3$ gaseous mixture at high temperature) or to inert conditions (N$_2$ flow) during heating or cooling transients, deactivation was observed. Feeding a high concentration of oxygen (15% v/v) for 5 h at 550 °C enabled however recovery of the activity almost to its original level.

While the fundamental implications related to the mechanism of the observed Cu-SAPO deactivation remain to be elucidated, our results emphasize that some caution should be taken in designing how to run catalytic activity experiments over this class of catalysts: depending on how you do the cool down in between experimental runs, different results can be expected.

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