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Reduction by CO of NO_x species stored onto Pt–K/Al_2O_3 and Pt–Ba/Al_2O_3 lean NO_x traps

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ABSTRACT

In this paper the results obtained in our previous studies on the reduction of nitrates by CO over Pt–K/Al_2O_3 and Pt–Ba/Al_2O_3 catalysts are compared and discussed in order to define the chemistry that leads to nitrogen evolution during the regeneration of the different lean NO_x traps. The reaction mechanisms and pathways leading to N_2 formation under dry conditions are investigated by complementary transient dynamic experiments and FT-IR analyses.

In both cases, the reduction of stored NO_x by CO occurs according to a Pt catalyzed surface pathway that does not involve the release of NO_x in the gas phase as a first step, is effective already at low temperature and leads to nitrogen. The proposed reaction scheme implies the formation of surface isocyanate species followed by their reaction with residual stored NO_x to give nitrogen. This is the exclusive route for nitrogen formation.

1. Introduction

The need of reducing fuel consumption by employing engines that work under “lean” conditions, i.e. in the presence of excess oxygen, makes lean NO_x trap (LNTs) catalysts a viable technology for reduction of NO_x emissions from the engine exhausts [1]. LNT catalytic systems work under cyclic conditions, alternating lean phases during which NO_x species produced by the engine is adsorbed on the catalyst surface, with short rich phases during which the stored NO_x (mainly nitrates) is reduced to nitrogen.

In LNT catalysts a high surface area carrier like alumina supports a noble metal (Pt), which catalyses both the oxidation of NO_x, CO and unburned hydrocarbons (UHCs) and the reduction of stored NO_x by CO, H_2 and UHCs, together with an alkaline or alkaline-earth oxide that acts as NO_x-storage component: the most common formulations propose barium, but recently potassium is gaining more and more attention, as well [2–4]. Indeed, in the literature many studies analyse the properties and reactivity of Ba containing catalysts [5], but reports on the specific behaviour of K-based catalysts are still rather scarce.

The reduction of NO_x adsorbed species has been addressed in the literature using different reducing agents, such as H_2, CO, C_3H_6 and C_3H_8 [6–8]. In previous works, we have shown that under nearly isothermal conditions the reduction by H_2 of nitrates stored onto Pt–Ba/Al_2O_3 [9–11] and Pt–K/Al_2O_3 [3,12] is not initiated by the thermal decomposition of nitrate/nitrite ad-species with release of NO_x in the gas phase, but involves a Pt catalyzed surface pathway, which is active already at low temperature and leads to nitrogen. Furthermore, it was found that N_2 is formed exclusively via a consecutive reaction scheme that involves the fast reaction of H_2 with stored NO_x to give ammonia followed by a slower reaction of ammonia with residual stored NO_x to give N_2 [3,10,11]. However, it has also been pointed out that this last reaction of ammonia with stored NO_x to give N_2 is slower on Pt–Ba/Al_2O_3 than on Pt–K/Al_2O_3.

Recently, using transient reactivity methods and complementary FT-IR spectroscopy [13], we have performed a systematic and quantitative study of the reaction pathway and mechanism of the reduction by CO of NO_x stored onto a Pt–Ba/Al_2O_3 LNT catalyst under dry conditions: it was shown that the reduction occurs through a Pt-catalyzed surface pathway, like in the case of H_2. In the present paper a comparative study of the reduction steps performed by CO on Pt–K/Al_2O_3 and Pt–Ba/Al_2O_3 systems under dry conditions has been carried out. For this purpose, NO_x has been adsorbed on the catalyst surfaces at 350 °C starting from NO/O_2 mixture; then the reactivity of the stored NO_x with CO has been investigated by means of temperature programmed surface reaction (TPSR), isothermal step concentration reaction (ISCR) and FT-IR spectroscopy to obtain information on the nature, reactivity and evolution of reactants, intermediates and products both in the gaseous phase and on the catalyst surface.

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2. Experimental

2.1. Catalyst preparation and characterization

Pt–K/Al_2O_3 (1/5.4/100 w/w/w) and Pt–Ba/Al_2O_3 (1/20/100 w/w/w) catalysts, with the same molar amount of storage component (0.146 mol K/ or Ba/100 g Al_2O_3) were prepared by incipient wetness impregnation of γ-alumina (Versal 250 from UOP). A detailed description of the preparation procedure is reported elsewhere [3,4,10,14]. The following surface areas and pore volumes were measured by N_2 adsorption–desorption at 77 K: 176 m^2/g and 0.9 cm^3/g for the Pt–K/Al_2O_3 sample and 137 m^2/g and 0.81 cm^3/g for the Pt–Ba/Al_2O_3 sample. For the catalysts reduced in H_2 at 350 °C, the Pt dispersion, as determined by H_2 chemisorption at 0 °C, was about 65 and 70% for Pt–K/Al_2O_3 and Pt–Ba/Al_2O_3, respectively, perfectly in line with the mean Pt particle size measured by HRTEM (1.5 nm for both the catalysts) [14,15].

2.2. Reactivity tests

Reactivity studies were performed in a flow micro-reactor system equipped with different on-line analyzer for reagent and product analysis: a mass spectrometer (Omnistar 200, Pfeiffer Vacuum) for the analysis of the following mass-to-charge ratios, 2 (H_2), 18 (H_2O), 28 (N_2 or CO), 30 (NO), 32 (O_2), 44 (N_2O or CO_2), and 46 (NO_2); a micro GC (Agilent 3000A) for the analysis of CO_2, N_2O, and H_2O (Poraplot Q capillary column), and of O_2, N_2, and CO (5 Å molecular sieve capillary column); a UV analyzer (Limas 11HW, ABB) for the analysis of NO, NO_2, NH_3 [3,16]. In each run 60 mg of catalyst with small particle diameter (100–120 μm) and a total flow rate of 100 cm^3/min (at 1 atm and 0 °C, GHVS 10^5 h^{-1}) were employed. In a typical experiment NO_x were adsorbed at 250 °C or at 350 °C from NO (1000 ppm) in He + O_2 (3%, v/v); adsorbed NO_x species were then reduced with CO under temperature programming (temperature programmed surface reaction, TPSR) or at constant temperature (isothermal step concentration reaction, ISCR). In the last case several adsorption/reduction cycles (typically 3–4) were accomplished in order to obtain reproducible catalyst performances. The thermal stability of adsorbed NO_x species was also analyzed by TPD (temperature programmed desorption) experiments in He.

Note that the reduction of stored nitrates has been carried out in the absence of gaseous NO during the rich phase in order to avoid any interference between the nitrate/CO surface reactions and the NO/CO gas phase reactions. However, NO is typically present during the rich phase under actual conditions; preliminary experiments carried out over the Pt–Ba/Al_2O_3 catalyst with CO/NO mixtures pointed out that NO is efficiently reduced to N_2 at temperatures below 200 °C.

2.3. FT-IR study

The reduction of the stored NO_x species was investigated separately by in situ FT-IR spectroscopy on self-supporting discs (10 mg/cm^2) placed in a commercial cell (Aabspec), allowing thermal treatments in controlled atmospheres and simultaneous registration of spectra up to 600 °C. Absorption IR spectra were collected on a Perkin-Elmer FT-IR system 2000 spectrophotometer.

NO_x storage was carried out at 350 °C, over a conditioned catalyst (3–4 adsorption/reduction cycles), by admitting freshly prepared NO/O_2 mixtures (p_{NO} = 5 mbar, $p_{O_2} = 20$ mbar) up to catalysts saturation (ca. 20 min), and outgassing at the same temperature. The reduction was accomplished in CO (p_{CO} = 10 mbar) under isothermal conditions, running the spectra at increasing exposure times. NO (Praxair, purity ≥99.0%) was freshly distilled before use. Conversely, NO_2 (Praxair, purity ≥99.5%), O_2 (Praxair, purity ≥99.999%) and CO (Praxair, purity ≥99.9%) were directly used.

Further details on the experimental apparatus and procedures can be found elsewhere [3,15].

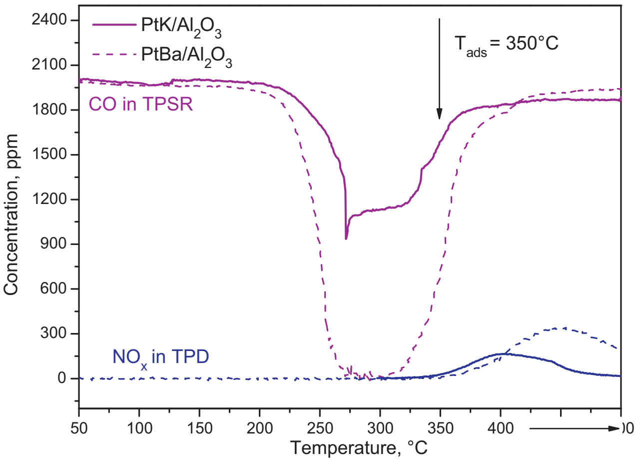

Fig. 1. NO_x and CO concentration during He-TPD during CO-TPSR over the Pt–K/Al_2O_3 (solid lines) and Pt–Ba/Al_2O_3 (dashed lines) catalysts.

3. Results and discussion

3.1. Stability and reactivity of stored NO_x

NO_x species adsorbed at 350 °C were heated up under temperature programming in inert atmosphere (He-TPD) and in the presence of 2000 ppm of CO under dry conditions (CO-TPSR) in order to analyse their stability/reactivity. Fig. 1 compares the NO_x (NO + NO_2) outlet concentration profiles measured during the TPD experiment with the CO concentration traces measured during the TPSR run over Pt–K/Al_2O_3 (solid lines) and over Pt–Ba/Al_2O_3 (dashed lines) catalysts.

During TPD experiments the stored NO_x species decompose to NO, NO_2 and O_2. As apparent from Fig. 1, the temperature onset for decomposition of the stored NO_x is close to 350 °C, i.e. close to the adsorption temperature [3,13,16].

On the other hand, during the TPSR experiment, CO consumption starts at lower temperature, i.e. near 210 °C in the case of the Pt–K/Al_2O_3 sample and 180 °C in the case of the Pt–Ba/Al_2O_3 sample, in both cases with evolution of N_2 and CO_2 (not shown in the figure). Note that in the case of the Pt–K/Al_2O_3 sample a shoulder in the CO consumption peak is observed near 315 °C, in line with the presence, on the catalyst surface, of both ionic and bidentate nitrates having different reactivities (for Pt–Ba catalyst mainly ionic nitrates are present), as shown by CO reduction at increasing temperature followed by FT-IR spectroscopy [16].

The comparison of the results of TPD and TPSR experiments clearly indicates that the reduction of the stored nitrates is not initiated by their thermal decomposition, but proceeds through Pt catalyzed surface reactions. Indeed, over both the catalyst samples, the CO consumption is observed at temperatures well below those corresponding to the decomposition of the stored NO_x. Furthermore, the results of CO-TPSR experiments carried out over the corresponding Pt-free samples [13,16], showing that the reduction by CO of nitrates stored at 350 °C occurs only at high temperature (330–350 °C) and in very limited extent, is a clear indication of the catalytic role of Pt in the reduction of the stored NO_x.

On the basis of the amounts of stored NO_x (4.33×10^{-4} mole/g_{cat} for K-containing system and 5.76×10^{-4} mole/g_{cat} for Ba-containing system) and N_2 evolved (1.90×10^{-4} mole/g_{cat} and

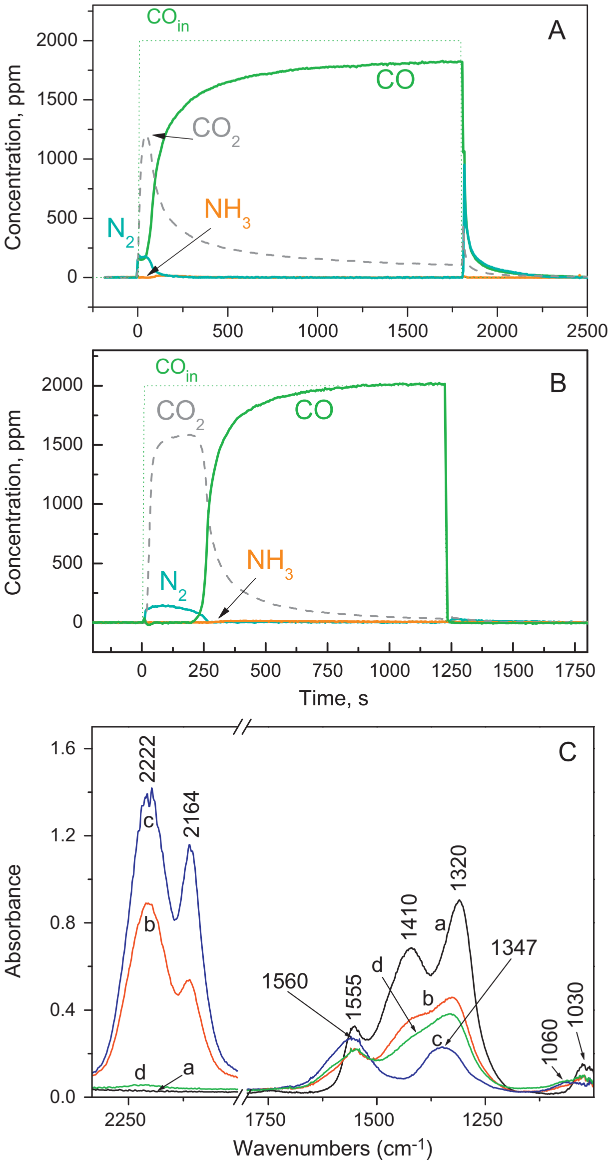

Fig. 2. ISCR experiments with CO (2000 ppm in He) at 250 °C (A) and 350 °C (B) over the Pt–Ba/Al_2O_3 catalyst after NO/O_2 adsorption at the same temperature. (C) FT-IR spectra of NO_x reduction in CO (10 mbar) at 350 °C and subsequent oxidation at the same temperature. Curve a, spectrum of NO_x stored at 350 °C by NO/O_2 adsorption; curves b and c, spectra after reduction for 30 s and 2 min, respectively; curve d, spectrum after admission of O_2 at 350 °C.

0.93×10^{-4} mole/g_{cat}, respectively) during the TPSR experiment, it was estimated that in the case of Pt–K/Al_2O_3 ~90% of the stored nitrates are reduced to gaseous N-containing products, whereas in the case of Pt–Ba/Al_2O_3 only ~43% of the stored nitrates are removed by CO as gaseous N-containing products. Consequently, N-containing species are still present at the catalyst surface at the end of the CO-TPSR experiments, in particular in the case of the Pt–Ba/Al_2O_3 catalyst. This point will be further addressed below.

3.2. Reduction by CO under isothermal conditions

The reduction of stored NO_x by CO was also performed at constant temperature (250 °C and 350 °C, ISCR experiments) after NO_x adsorption at the same temperatures. Fig. 2 shows the results obtained in the experiments carried out at 250 °C (Fig. 2A) and 350 °C (Fig. 2B) upon imposing a CO step over Pt–Ba/Al_2O_3 catalyst, on which NO_x were adsorbed at the same temperature mainly as nitrates. The results obtained in similar experiments over Pt–K/Al_2O_3 are reported in Fig. 3A (250 °C) and B (350 °C).

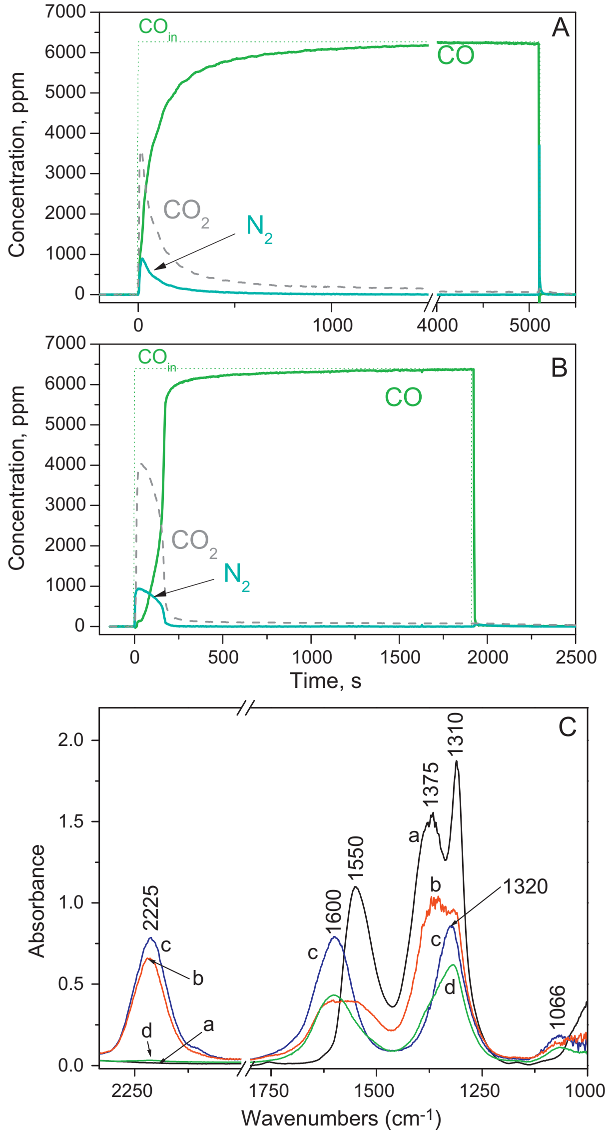

Fig. 3. ISCR experiments with CO (6500 ppm in He) at 250 °C (A) and 350 °C (B) over the Pt–K/Al_2O_3 catalyst after NO/O_2 adsorption at the same temperature. (C) FT-IR spectra of NO_x reduction in CO (10 mbar) at 350 °C and subsequent oxidation at the same temperature. Curve a, spectrum of NO_x stored at 350 °C by NO/O_2 adsorption; curves b and c, spectra after reduction for 30 s and 2 min, respectively; curve d, spectrum after admission of O_2 at 350 °C.

Over Pt–Ba/Al_2O_3 upon CO admission at 250 °C (Fig. 2A) at $t = 0$ s, N_2 (about 180 ppm) and CO_2 (near 1200 ppm) are immediately produced. After 120 s, the N_2 concentration decreases accompanied by that of CO_2, and evolution of very small amounts of ammonia are also observed; at the same time CO concentration increases reaching the inlet value. When CO is removed from the inlet feed flow (at 1800 s), a second nitrogen peak is observed. This is in line with the hypothesis of a self-inhibition effect of CO due to its strong adsorption on the Pt active sites [13].

A quantitative analysis of the amounts of evolved N-containing products indicates that at the end of the reduction run only a fraction (near 10%) of the initially stored NO_x are removed from the catalyst and reduced to N_2.

It is worth of note that during the experiment N_2 formation and CO consumption do not obey the stoichiometry of the expected reduction of nitrates by CO:

$$Ba(NO_3)_2 + 5CO \rightarrow N_2 + BaCO_3 + 4CO_2 \qquad (1)$$

In fact the amounts of CO consumed during the experiment are higher than those expected from reaction (1) on the basis of the quantity of released N_2. This point will be addressed later on.

CO-ISCR experiments were also performed at 350 °C, and results are presented in Fig. 2B [13]. The results are qualitatively similar to those obtained at 250 °C: after CO admission, evolution of N_2 and CO_2 (and NH_3) is observed. Then N_2 and the CO_2 concentrations decrease, and CO breakthrough is observed. As in the case of the experiment carried out at 250 °C, a nitrogen production peak is observed upon CO shutoff. Notably, this peak is smaller than that observed at 250 °C, whereas N_2 evolution during the overall experiment occurs primarily after the CO admission. A quantitative analysis of the amounts of evolved N-containing products indicates that at the end of the run roughly 60% of the initially stored NO_x are removed from the catalyst and reduced to N_2 (and NH_3).

After the reduction a new lean phase was performed (not shown). The O_2 and NO admission causes the evolution of non negligible amounts of N_2 along with CO_2. To better investigate these aspects, complementary FT-IR experiments were performed to analyse the nature and the evolution of the surface species originating upon CO admission at 350 °C after NO_x adsorption at the same temperature. The results are presented in Fig. 2C: curve a is the spectrum of NO_x previously stored at 350 °C (mainly ionic nitrates with $\nu_{NO_3,asym}$ mode split at 1410 and 1320 cm^{-1} and $\nu_{NO_3,sym}$ mode at 1030 cm^{-1}; minor amount of bidentate nitrates with $\nu_{N=O}$ mode at 1555 cm^{-1}, the only one visible [15]); curves b and c correspond to the spectra recorded during interaction with CO at increasing exposure times at 350 °C. The reduction of nitrates by CO is very fast since the intensity of the nitrate bands is reduced markedly after 30 s of CO contact (curve b). In parallel, carbonates are formed (bands at 1560, 1347 and 1060 cm^{-1} fully superimposed to the nitrate peaks, corresponding to $\nu_{C=O}$, $\nu_{O-C-O,asym}$ and $\nu_{O-C-O,sym}$ modes, respectively) [13]. Furthermore, intense bands appear at 2222 and 2164 cm^{-1}, related to formation of isocyanate species [13,16]. Upon further CO contact with the catalyst (2 min, curve c), the bands of nitrates almost disappear, whereas those of isocyanate and carbonate species increase. Subsequently, the spectrum recorded after 5 min of contact with O_2 at 350 °C (trace d) shows that the bands corresponding to NCO species are completely eroded; in parallel, the bands in the region 1700–1000 cm^{-1} reveal the formation of carbonate and nitrate species as surface products of NCO oxidation.

These results are in line with the CO-ISCR data presented in Fig. 2A and B, and pointed out that N-containing species (isocyanates) are left on the catalyst surface at the end of the reduction process. These species are oxidised to N_2 and CO_2, which are evolved in the gas phase during the subsequent lean phase, as pointed out by the CO-ISCR experiments previously described (Fig. 2A and B), along with formation of surface carbonates (and minor amounts of nitrates) as well.

Slightly different features are observed when K is present as storage component instead of Ba. The results of CO-ISCR experiment over Pt–K/Al_2O_3 catalyst are reported in Fig. 3A (250 °C) and B (350 °C). In this case 6500 ppm of CO has been used instead of 2000 ppm, but dedicated experiments showed that the concentration of CO does not affect the features of the reduction mechanism.

Upon CO admission at 250 °C (Fig. 3A), N_2 and CO_2 are immediately observed. The reduction is very fast and, already after 200 s, nitrogen (and CO_2) decreases to zero and CO approaches its inlet value. When the CO feed is stopped (at 5090 s), a small amount of nitrogen is produced, along with CO_2. As in the case of the Ba-based catalyst, this is due to the availability of vacant Pt sites which are partially poisoned by CO during the CO feed.

From the amounts of CO consumed and of evolved N_2 and CO_2 it appears that the reduction of the stored NO_x roughly obeys the stoichiometry of reaction (2):

$$2KNO_3 + 5CO \rightarrow N_2 + K_2CO_3 + 4CO_2 \qquad (2)$$

Besides, the N-balance is almost closed thus suggesting that in the case of the K-containing catalyst, as opposite to what observed in the case of Pt–Ba/Al_2O_3, almost all the stored NO_x have been removed by CO.

CO-ISCR experiment performed at 350 °C (Fig. 3B) is qualitatively and quantitatively very similar to those collected at 250 °C. Also at high temperature, reaction (2) accounts for the formation of nitrogen and CO_2.

Fig. 3C shows the results of FT-IR experiment at 350 °C: curve a is the spectrum of NO_x species previously stored at 350 °C (ionic nitrates with $\nu_{NO_3,asym}$ and $\nu_{NO_3,sym}$ modes at 1375 and 1040 cm^{-1}, respectively, and bidentate nitrates with $\nu_{N=O}$, $\nu_{O-N-O,asym}$ and $\nu_{O-N-O,sym}$ modes at 1550, 1314 and 1000 cm^{-1}, respectively [14]); curves b and c correspond to the spectra recorded during interaction with CO at increasing exposure times at 350 °C. The reduction of nitrates is very fast since the band intensities of both bidentate and ionic nitrates are reduced markedly after 30 s of CO contact (curve b). At 2 min of contact (curve c) nitrates are completely consumed. Simultaneously to the nitrate reduction, the carbonate species appear (bands at 1600, 1320 and 1066 cm^{-1}) along with NCO species (band at 2225 and very small absorption at 2163 cm^{-1}). As observed for ISCR experiments, also FT-IR isothermal measurements confirm that isocyanate species formed on Pt–K/Al_2O_3 catalyst at 350 °C are in very low amounts if compared with those formed under the same experimental conditions by reduction with CO of NO_x stored on the Pt–Ba/Al_2O_3 catalyst [13,16]. Actually, this quantitative evaluation could be done comparing the integrated intensities of absorption bands related to NCO, assuming that the NCO species having the same spectroscopic features on Pt–K/Al_2O_3 and Pt–Ba/Al_2O_3 systems, have also the same absorption coefficients [13,16]. As reported elsewhere [16], it is possible to directly compare the integrated intensities (normalized to the same surface area) of the isocyanate species present at the end of the reduction on the two catalysts to make an evaluation of their different amounts: the quantity of isocyanates on Pt–K catalyst is roughly 5 times lower than that on Pt–Ba catalyst, in agreement with ISCR data.

In Fig. 3C, curve d represents the spectrum recorded after 5 min of contact with O_2 at 350 °C. Also in this case the erosion of NCO species is complete. However, differently from the Pt–Ba system, only very small amounts of nitrates are formed (shoulders at 1380 and 1545 cm^{-1}) and carbonates formation cannot be appreciated. Indeed, the intensity of their bands at 1600, 1320 and 1066 cm^{-1} decreases, due to their lower thermal stability [14] with respect to that formed on Pt–Ba system [15].

4. Conclusive remarks

The results of CO-TPSR (Fig. 1), CO-ISCR and FT-IR study (Figs. 2 and 3) indicate that the reduction by CO of nitrates stored onto Pt–K/Al_2O_3 and Pt–Ba/Al_2O_3 catalysts occurs according to a Pt catalyzed surface pathway, with the formation of N_2 and CO_2 in the gas phase and NCO and carbonate species on the surface. During the NO_x reduction by CO, N_2 is primarily formed according to an in series two steps process where NCO species are formed first and then converted to nitrogen upon reaction with NO_x stored species.

In analogy with the reaction pathway proposed for the reduction of stored nitrates by H_2 [10], the reduction under dry conditions by CO of nitrates stored onto the Pt–Ba/Al_2O_3 and Pt–K/Al_2O_3 catalysts occurs according to the stoichiometry of the following overall reactions:

$$2NO_3^- + 8CO \rightarrow 2NCO^- + 6CO_2 \quad (3)$$

$$6NO_3^- + 10NCO^- \rightarrow 8N_2 + 8CO_3^= + 2CO_2 \quad (4)$$

Hence, NCO species are considered intermediates in the formation of N_2 (as NH_3 in the reduction by H_2) whose formation occurs exclusively according to the stoichiometry of the reactions (3) + (4), the sum giving the overall reaction (1) or (2).

In addition, it is shown that over Pt–Ba/Al_2O_3 catalyst [13] NCO species can be oxidized to give N_2 during the subsequent lean phase either by oxygen or by NO + O_2 (and NO_2), according to the overall reaction:

$$Ba(NCO)_2 + 3/2O_2 \rightarrow N_2 + BaCO_3 + CO_2 \quad (5)$$

This reaction accounts for the evolution of N_2 upon oxygen admission.

Note that over both the Pt–Ba/Al_2O_3 and Pt–K/Al_2O_3 catalytic systems the reaction between surface nitrates and surface NCO species to give nitrogen (reaction (4)) is slower than that responsible for NCO species formation (reaction (3)). In fact, on both catalysts NCO species were already formed at temperatures lower than those corresponding to N_2 evolution, as revealed by TPSR experiments [13,16]. Furthermore, reaction (4) is faster and more efficient over K-containing system than over Ba-containing system. Indeed, lower amounts of isocyanates species are left at the end of the reduction over Pt–K/Al_2O_3 than over Pt–Ba/Al_2O_3.

In the pathway suggested for the reduction of stored NO_x by CO catalyzed by Pt, the rate determining step is the slow reaction between nitrates and isocyanates (reaction (4)) which involves two surface species; hence the surface mobility of these species is expected to play a major role on the reaction. Along these lines, K-nitrates and K–NCO species might have a higher surface mobility that facilitate their spillover from the K component onto the Pt particles and as consequence their reaction. As a result, the CO reduction is more efficient over Pt–K/Al_2O_3 catalyst than over Pt–Ba/Al_2O_3 catalyst.

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