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ScienceDirect
Catalysis Today 137 (2008) 185–190
CATALYSIS TODAY
www.elsevier.com/locate/cattod

Selective reduction of NO_x by hydrogen and methane in natural gas stationary sources over alumina supported Pd, Co and Co/Pd catalysts
Part B: On the effect of bimetallic catalyst preparation

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Abstract

The aim of the present work is to study the selective reduction of NO_x from natural gas sources using unburned methane or hydrogen as reducing agents. The results suggest that the NO_x are reduced by H_2 at low temperature, when methane is not activated and at higher temperature the methane is then the main reducing agent. Similar results are obtained for alumina supported palladium and alumina supported cobald-palladium catalysts at low temperature in presence of hydrogen suggesting that the active phase for the reaction NO/H_2 is the palladium. However, at high temperature the higher activity is obtained on bimetallic catalyst. The presence of cobalt enhances the catalytic activity. This result suggests that cobalt and palladium both in cationic form are the active sites when the reducing agent is the methane.

Keywords: Stationary sources; Methane; Hydrogen; deNO_x; Bimetallic catalysts

1. Introduction

Emission of nitrogen oxides as a by-product of natural gas high-temperature combustion has been a major environmental concern. The SCR of NO_x by methane is a very attractive technology for the decreasing of NO_x from stationary sources, because natural gas (methane) is readily available. Many years ago, cobalt supported on several zeolites were found to be the best catalysts NO_x removal by methane in lean conditions [1]. However, the presence of water vapor leads to a large deactivation of these catalysts [1,2]. Thus, bimetallic cobalt and palladium loaded zeolites were developed in order to obtain a high resistance to water vapor [3–7]. Over platinum group metal-based catalysts, in a zero valent oxidation state, the mechanism of NO_x reduction is now understood and is well-established in a recent paper of Burch et al. [8]. For other catalytic materials, some mechanisms have been suggested [8,9]. More recently, on cationic metal species, a general three-function model for deNO_x catalysis [10–13] was also proposed. The authors claimed that three functions are necessary for the deNO_x process to occur: (i) oxidation of NO to NO_2; (ii) mild oxidation of methane to alcohol and aldehyde, in the presence of NO_2; (iii) reduction of NO to N_2, assisted by the deep oxidation of the alcohol and aldehyde to CO_2. Simultaneously the active site of the third function is regenerated. The three catalytic cycles should turn over simultaneously in order to obtain deNO_x activity. The detailed process has already been described on alumina supported cobalt palladium based catalysts by Marques et al. [14].

Hydrogen SCR could be applied for NO_x reduction with engines and burners running on traditional fuels. The concentration of hydrogen in exhaust gas of lean-burn natural gas is

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doi:10.1016/j.cattod.2007.11.024

generally too low, which makes external addition of the reducing agent H_2 necessary. Reforming of alcohol occurs below 300 °C, thus the external addition of hydrogen source could be placed directly in the exhaust. Recent investigations [15–18] show that supported noble metal based catalysts can profitably be used for hydrogen SCR of NO_x. Pt and Pd based catalysts have been examined by Ueda et al. [15], for $H_2/NO/O_2$ reaction, under lean burn condition (5% O_2) in presence of 10% of H_2O. All Pd-based catalysts displayed two distinct conversion maximum for the reduction of NO, one centred at 100 °C and the second at 300 °C. At low temperature, the peak results from the reaction between H_2 and NO, while the peak at high temperature would be the result of the reaction between H_2 and NO_2 produces in situ. Engelmann-Pirez et al. [19] investigated the effect of the support on Pd based catalysts in NO + H_2 reaction. The authors have showed that the metal–support interaction is very important to stabilize metallic species. MacLeod and Lambert [20,21] reported the various aspects of system $H_2/CO/NO/O_2$ over platinum and palladium catalysts supported on alumina. In the case of Pt/Al_2O_3, the presence of CO in the reaction mixture $H_2/NO/O_2$ increases the temperature necessary to initiate the reaction. In complete contrast, in the case of palladium, the presence of CO plays a promoting influence for the NO_x conversion. H_2 and CO alone are ineffective reducers for deNO$_x$ under lean burn conditions on the Pd/Al_2O_3 catalyst. Pieterse and Booneveld [22] reported the study of NO_x reduction by the reducing agents H_2, CO, CH_4, in presence and absence of O_2, H_2O and CO_2 on zeolite MOR catalysts impregnated with palladium and cerium. This bimetallic catalyst provides high NO_x conversion showing high nitrogen selectivity (∼90%) with H_2 and CO under lean burn conditions, which is assigned to a synergic co-operation between CO and H_2. The combination of Ce to Pd tends to give higher NO_x conversion with H_2/CO at low temperatures and with CH_4 at high temperatures. In a recent paper, Marques et al. [23] showed that impregnation by palladium tetramine nitrate leads to a more active catalyst in presence of H_2, NO, CH_4, O_2. The aim of this work is to determine the deNO$_x$ activity of catalyst containing Co and Pd in presence of methane alone and hydrogen alone. Then, this activity was compared with the activity obtained in presence of both methane and hydrogen. Finally, the influence of catalyst preparation will then be discussed.

2. Experimental

2.1. Catalysts synthesis

Catalysts were prepared by wet impregnation of crushed and sieved γ-Al_2O_3 (0.8 mm $< d <$ 1.2 mm) (Procatalyse, specific surface area of 190 $m^2\,g^{-1}$, pore volume of 0.7 $cm^3\,g^{-1}$) with $Pd(NH_3)_4(NO_3)_2$ aqueous solution; because this precursor leads to the higher activity on deNO$_x$ in presence of hydrogen and methane [23]; or cobalt(II) nitrate hexahydrate 99%. The precursor's suspension was maintained under stirring at 50 °C for 3 h. After complete removal of water by evaporation under reduced pressure, the catalysts were dried overnight with air at 120 °C and calcined with air at 500 °C for 2 h. The as-prepared catalysts are labelled Pd(*X*)/Al_2O_3 and Co(*X*)/Al_2O_3, in which *X* is content of metal (wt.%). The catalysts preparation procedure is the same as described elsewhere [24]. For bimetallic catalysts, labelled Pd(*X*)/Co(*X*)/Al_2O_3, the cobalt is first impregnated on the support. Then, after the treatments presented above, the palladium is added in a successive impregnation. In the case of Co(*X*)-Pd(*X*)/Al_2O_3, cobalt and palladium are co-impregnated. The prepared catalysts are presented in Table 1.

Table 1
Metal content (wt.%) and BET Surface Area of used catalysts

Catalyst	Content (wt.%)		S_{BET} (m^2/g)
	Co	Pd	
Pd(*X*)/Co(*X*)/Al_2O_3	0.47$^{(a)}$	0.59$^{(a)}$	163.96
Pd(*X*)-Co(*X*)/Al_2O_3	0.51$^{(b)}$	0.50$^{(b)}$	213.89
Co(*X*)/Al_2O_3	0.48	–	183.04
Pd(*X*)/Al_2O_3	–	0.49	182.16

(a) Catalysts prepared by successive impregnations; (b) catalysts prepared by co-impregnation.

2.2. Characterization of catalysts

The catalysts were characterized by the same techniques presented elsewhere [23] such as: transmission electron microscopy (TEM) and UV–visible–near-infrared (NIR), elemental analysis. High-resolution transmission electron microscopy (HRTEM) was performed to determine the particle size of cobalt or palladium particles on alumina and to check their dispersion. HRTEM studies were performed on a JEOL-JEM 100 CXII apparatus associated with a top entry device and operating at 100 kV. EDS analysis was performed with the same apparatus using a LINK AN 10,000 system, connected to a silicon–lithium diode detector, and multichannel analyser. EDS analyses were obtained on large domains of samples (400 nm × 533 nm). Diffuse reflectance spectra were recorded at room temperature between 190 and 2500 nm on a Varian Cary 5E spectrometer equipped with a double monochromator and an integrating sphere coated with polytetrafluoroethylene (PTFE). PTFE was the reference.

2.3. Catalytic measurements

The catalytic reaction was performed with the following reactants.

150 ppm NO, 7 vol.% O_2, 0 vol.% CO_2, 9000 ppm CH_4, 0 vol.% H_2O, 0 ppm H_2, in Ar as balance. 150 ppm NO, 7 vol.% O_2, 0 vol.% CO_2, 0 ppm CH_4, 0 vol.% H_2O, 1500 ppm H_2, in Ar as balance. 150 ppm NO, 7 vol.% O_2, 0 vol.% CO_2, 9000 ppm CH_4, 0 vol.% H_2O, 1500 ppm H_2, in Ar as balance. The NO mixture was supplied by Air Liquide as 1 vol.% NO and 99 vol.% Ar (<10 ppm other gases). The O_2 mixture contained 100 vol.% O_2 (Air Liquide). The CH_4 mixture contained 5 vol.% CH_4 and 95 vol.% Ar (Air Liquide). The total gas flow was maintained at 0.25 L min^{-1} NTP. Each of the gas mixtures was metered using calibrated electronic mass flow

controllers (Brooks, Model 5850E). The gas hourly space velocity (GHSV) was chosen at 40,000 h^{-1}. Catalytic experiments were carried out in a glass microreactor containing quartz wool supporting the sample. The bed temperature was measured using K-type thermocouple affixed to the outer reactor surface. The temperature was controlled using an electronic controller (Eurotherm 2408). The reactor outflow was analyzed using a set of specific detectors. An Eco Physics CLD 700 AL NO_x Chemiluminescence analyzer (for NO and total NO_x (i.e. NO + NO_2)) allowed the simultaneous detection of NO, NO_2 and NO_x. An Ultramat 6 IR analyzer was used to monitor N_2O and a FID detector was used to follow the total concentration of hydrocarbons (HC). Temperature programmed desorption (TPD) experiments were carried out in Ar/O_2 (7 vol.% O_2 in Ar as balance, 0.250 L min^{-1}) with a heating rate of 5 °C min^{-1}, up to 500 °C, over pretreated samples. Before TPD gas mixture was pre-adsorbed (150 ppm NO, 7 vol.% O_2 in Ar as balance, 0.250 L min^{-1}). Isothermal steady-state reaction was measured at different temperatures, ranging from 200 to 500 °C, in decreasing the temperature to avoid all the desorption effects. The NO_x and methane conversions were calculated from measured concentration of NO_x and methane, respectively.

3. Results and discussion

3.1. Highly dispersed cationic materials

The materials, listed in Table 1, are characterized TEM. No crystallized phases are detected by HRTEM, although EDS showed Pd/Al and Co/Al ratios are constant. Cobalt and palladium species are then highly dispersed on the support. UV–visible–NIR diffuse reflectance is performed to characterized theses latter species. The UV–visible–NIR diffuse reflectance spectra of Co(X)/Al_2O_3, Pd(X)/Al_2O_3, Pd(X)/Co(X)/Al_2O_3 and Pd(X)-Co(X)/Al_2O_3 catalysts are displayed in Fig. 1. The spectra of the support consists mainly of several bands in the near infrared (NIR) due to: (i) $\nu_{(OH)}$ overtones of surface hydroxyl groups (1364 nm) and (ii) a combination of $\nu_{(OH)}$ and $\delta_{(OH)}$ (1885 and 2207 nm) [25]. After exchange of cobalt, the spectra show a set of three bands at 540, 580 and 633 nm. As already reported in literature [26], these three bands are indicative of tetrahedral Co^{2+} ions, as found in the compound $CoAl_2O_4$. For Pd(X)Co(X)/Al_2O_3 catalyst a new band is observed at 420 nm, this band is characteristic of isolated Pd^{2+} in an oxygen environment [27]. As conclusion, no particles of Co_3O_4 and PdO were detected. More over, in our samples, the cobalt and the palladium are in cationic form Co^{2+} and Pd^{2+}, respectively, well dispersed on the support.

3.2. Catalytic performance of catalysts

3.2.1. Co/Pd/Al_2O_3

The $deNO_x$ catalytic behaviour was studied in steady-state conditions. The results are presented in Fig. 2a. In presence of hydrogen alone, the NO_x conversion is observed at low temperature between 200 and 300 °C. The maximum of conversion (23%) is observed at 250 °C. At low temperature, the hydrogen is the reducing agent scavenging the "O" left by NO during the NO dissociation [10,11]. Above 300 °C, the hydrogen is totally consumed and oxidized in H_2O. At low temperature, a kinetic coupling exists between NO/O_2 and H_2/O_2 reactions. In presence of methane alone, a NO_x conversion into N_2 is observed between 300 and 500 °C. The maximum is observed at 390 °C (42%). As for hydrogen at low temperature, the methane scavenges the "O" atoms on the active sites, but only at high temperature. Indeed, the methane reacts only for temperatures higher than 300 °C. At high temperature, a kinetic coupling exists between NO/O_2 and CH_4/O_2 reactions.

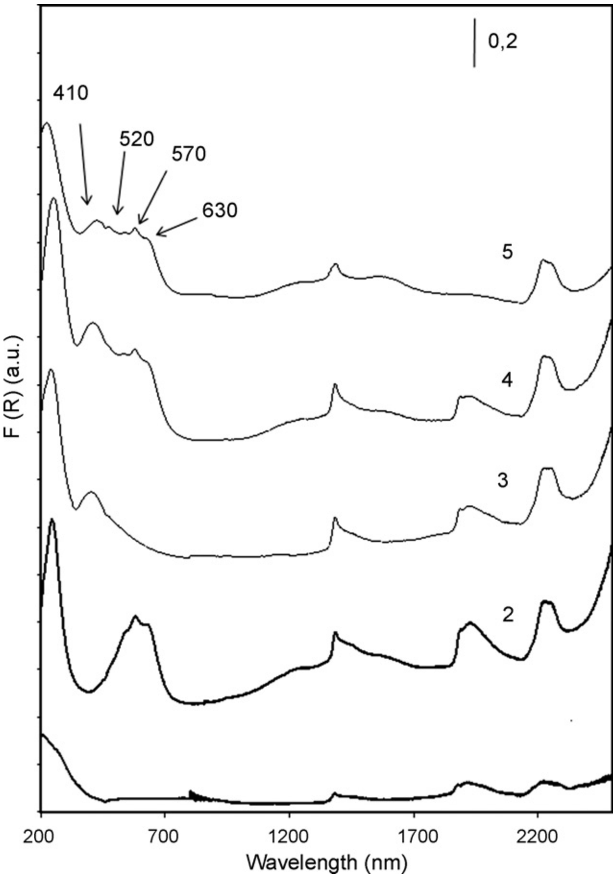

Fig. 1. UV–visible–NIR diffuse reflectance spectra of Co(X)Pd(X)/Al_2O_3 catalysts: 1, Al_2O_3; 2, Co(0.49)/Al_2O_3; 3, Pd(0.48)/Al_2O_3; 4, Co(0.47)/Pd(0.59)/Al_2O_3; 5, Co(0.51)-Pd(0.50)/Al_2O_3.

Thus, the catalysts are active at low temperature (200–250 °C) and at high temperature (350–500 °C). These two catalytic domains can be explained by the NO TPD (figure not shown). As reported elsewhere [14,23], the NO TPD can predict the temperature range in which the $deNO_x$ can occur. On Co/Pd/Al_2O_3 catalyst, we found two desorption peaks centered at 210 and 400 °C. These temperatures are in agreement with those obtained previously on Co-Pd based catalysts, and can be linked to those associated to $deNO_x$ reaction.

A complete reaction mixture was then realized using simultaneously methane and hydrogen as reducing agents. This experiment was performed to observe an additive or synergetic effect of the two reducing agents on the $deNO_x$ reaction. In

Fig. 2a, one can observe that in presence of two reducing agents acting at different temperatures, two NO conversion maxima are observed. The maximum at high temperature (32% of NO_x conversion at 400 °C) is assigned to the methane as reducing agent. The maximum at low temperature (26% at 250 °C) is assigned to hydrogen. However, we observe a decrease of the $deNO_x$ at high temperature. Thus, there is no additive effect on this catalyst. To confirm the loss of activity, a *"back point"* in $CH_4/NO/O_2$ mixture was realized after all the other experiments on the same catalyst (noted by used). The results of methane total oxidation are presented in Fig. 2b. The CO_2 concentration is plotted versus temperature. On fresh catalyst (first experiment in presence of methane alone), 100% of methane is converted whereas only 84% is converted on the used catalyst. The CO_2 comes from the oxidation of oxygenated species created by the interaction between CH_4 and NO_2 (function 2 of $deNO_x$ model) [11] by NO or by O_2. This deactivation can be explained by a change in the state of the active sites. In Fig. 2c are plotted the results of UV–vis spectroscopy on fresh and used catalysts. No significative change is observed for Co^{2+} species. However, a change in palladium species is observed. The peak centered at 420 nm corresponding to Pd^{2+} species surrounded by oxygen atoms is shifted to lower wave numbers (370 nm). This shifted peak corresponds to palladium species Pd^{2+} in interaction with PdO clusters. Thus, this change in palladium species leads to a lower activity in $deNO_x$. By these experiments, we can conclude that there is a change in palladium species in the course of the reaction with hydrogen and methane used as reducing agents. More over, the active species for $deNO_x$ reaction should be palladium highly dispersed as Pd^{2+} species surrounded by oxygen atoms "PdO_x".

To confirm this latter point and for sake of comparison alumina supported catalysts were tested. The catalyst exhibits no activity in presence of hydrogen as reducing agent at low temperature. However, in presence of methane, the NO_x conversion leads to 24% at 500 °C. The cobalt Co^{2+} in tetrahedral form can be one active species in the course of SCR of NO_x by methane at high temperature. The same experiments were thus performed on palladium catalysts.

3.2.2. Pd/Al_2O_3

The $deNO_x$ catalytic behaviour was studied in steady-state conditions. As already presented for bimetallic catalyst, in presence of methane as reducing agent, a NO_x conversion is observed from 300 to 500 °C with a maximum of 50% at 400 °C (Fig. 3a). In the presence of hydrogen alone, a maximum of 26% of NO_x conversion is observed at 250 °C. The methane is also in that case the reducing agent at high temperature whereas the hydrogen is the reducing agent at low temperature. For this monometallic catalyst, the activity is higher than for bimetallic catalyst leading to conclude that palladium species are the

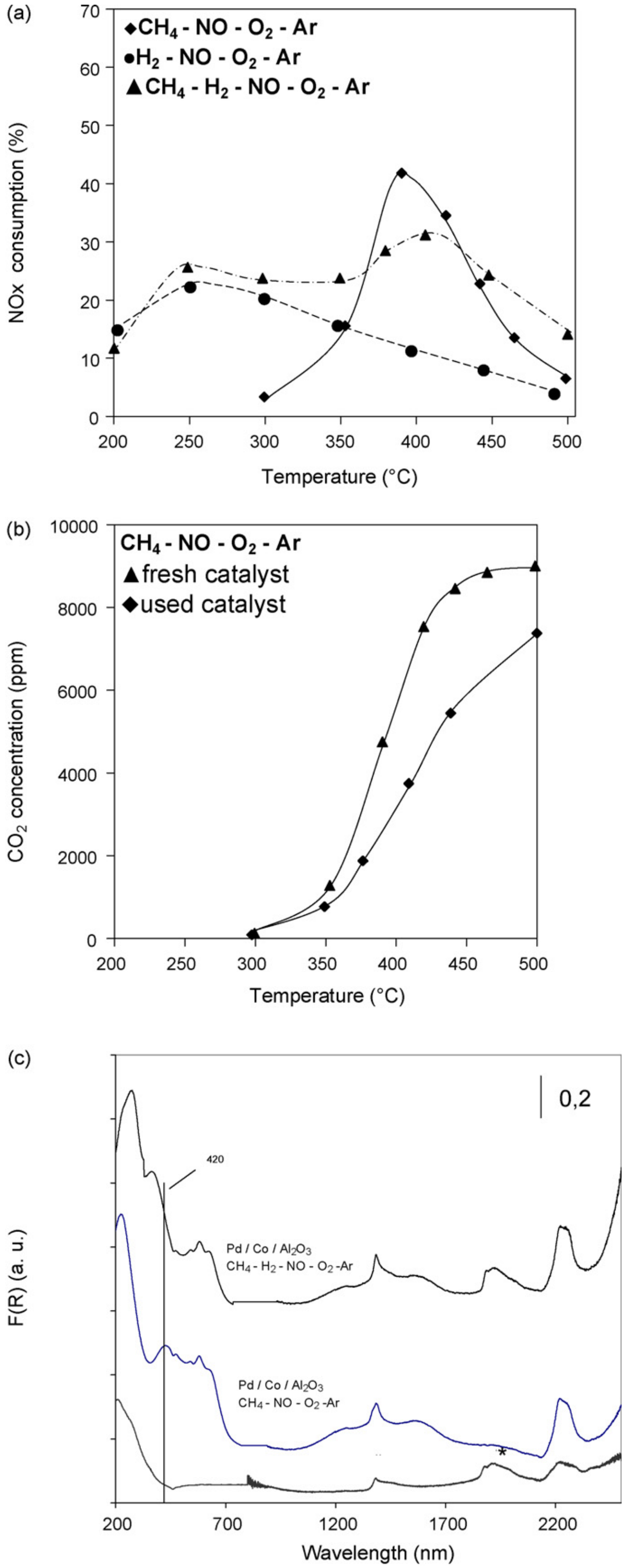

Fig. 2. (a) Isothermal steady-state consumption of NO_x in the course of CH_4–NO–O_2 (9000 ppm–150 ppm–7 vol.%) or H_2–NO–O_2 (1500 ppm–150 ppm–7 vol.%) or H_2–CH_4–NO–O_2 (1500 ppm–9000 ppm–150 ppm–7 vol.%), Ar as balance over: Co (0.47 wt.%)/Pd (0.50 wt.%)/Al_2O_3. (b) Evolution of CO_2 concentration during the isothermal steady-state consumption of NO_x in the course of CH_4–NO–O_2 (9000 ppm–150 ppm–7 vol.%), Ar as balance over: Co (0.47 wt.%)/Pd (0.50 wt.%)/Al_2O_3 before and after a series of runs. (c) UV–visible–NIR diffuse reflectance spectra of Co(*X*)/Pd(*X*)/Al_2O_3 catalyst after CH_4–NO–O_2 and H_2–CH_4–NO–O_2 runs.

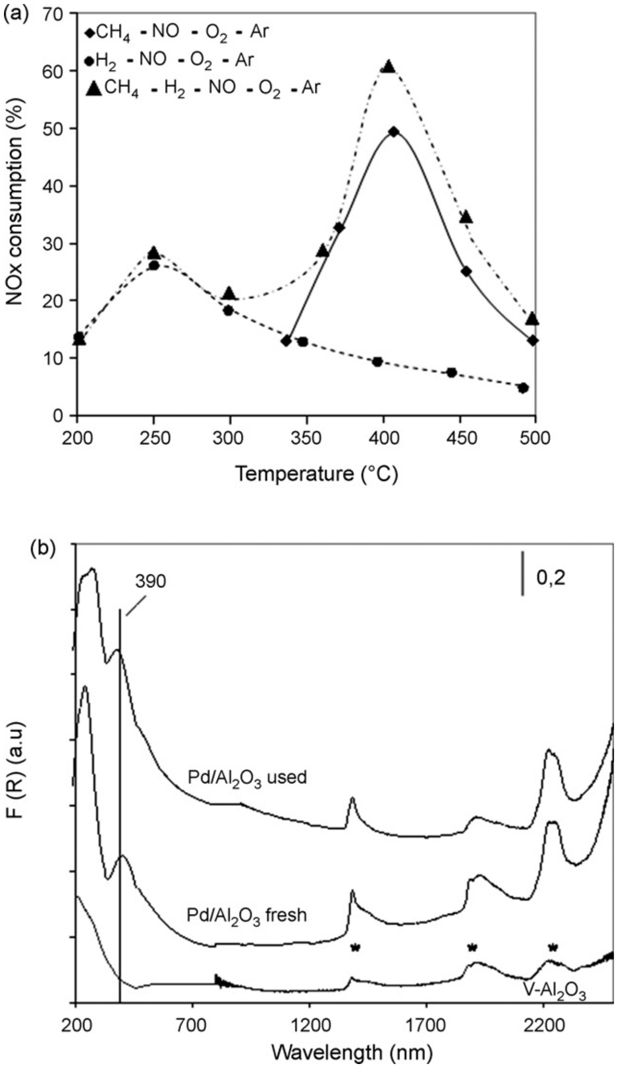

Fig. 3. (a) Isothermal steady-state consumption of NO_x in the course of CH_4–NO–O_2 (9000 ppm–150 ppm–7 vol.%) or H_2–NO–O_2 (1500 ppm–150 ppm–7 vol.%) or H_2–CH_4–NO–O_2 (1500 ppm–9000 ppm–150 ppm–7 vol.%), Ar as balance over: Pd (0.49 wt.%)/Al_2O_3. (b) UV–visible–NIR diffuse reflectance spectra of Pd(X)/Al_2O_3 catalyst after CH_4–NO–O_2 and H_2–CH_4–NO–O_2 runs.

active species for SCR of NO_x by hydrogen and methane. Using simultaneously methane and hydrogen as reducing agents (Fig. 3a), two NO_x conversion peaks are observed. 61% of conversion is obtained at 400 °C and 28% is obtained at 250 °C. For this catalyst, no deactivation is observed and an additive effect is observed using two reducing agents instead of one. The CO_2 formation was followed on fresh and used catalyst (figure not shown). No difference is observed. One can conclude that the palladium species do not change in the course of the deNO_x reaction. To confirm this result, we characterized this catalyst fresh and after run (used) by UV–vis spectroscopy. Similar results are obtained on fresh and used catalysts (Fig. 3b). A band characteristic of palladium Pd^{2+} surrounded by “O” is observed on both catalysts. In the course of deNO_x reaction, there is no change in the active sites and no deactivation of the alumina supported palladium catalysts.

Finally, we try to synthesize a bimetallic catalyst which does not deactivate with time on stream or with reaction mixture.

3.2.3. Co-Pd/Al_2O_3

A co impregnated Co-Pd catalyst was tested in the same conditions (Fig. 4a). We observed again in presence of methane a conversion of NO_x from 300 to 500 °C, with a maximum at 400 °C (40%). More over, in presence of hydrogen, a NO_x conversion is observed from 200 to 300 °C, with a maximum of 23% at 250 °C. Using the two reducing agents, two deNO_x maxima are observed at 250 °C (25%) and 400 °C (44%). On this catalyst, we also observed an additive effect of the two reducing agents and no deactivation with time of stream. A back point was realized in order to confirm this result. No difference is observed between fresh and used catalyst. The active sites do not change during the reaction in presence of hydrogen and methane. To confirm this fact, UV–vis spectroscopy was performed (Fig. 4b).

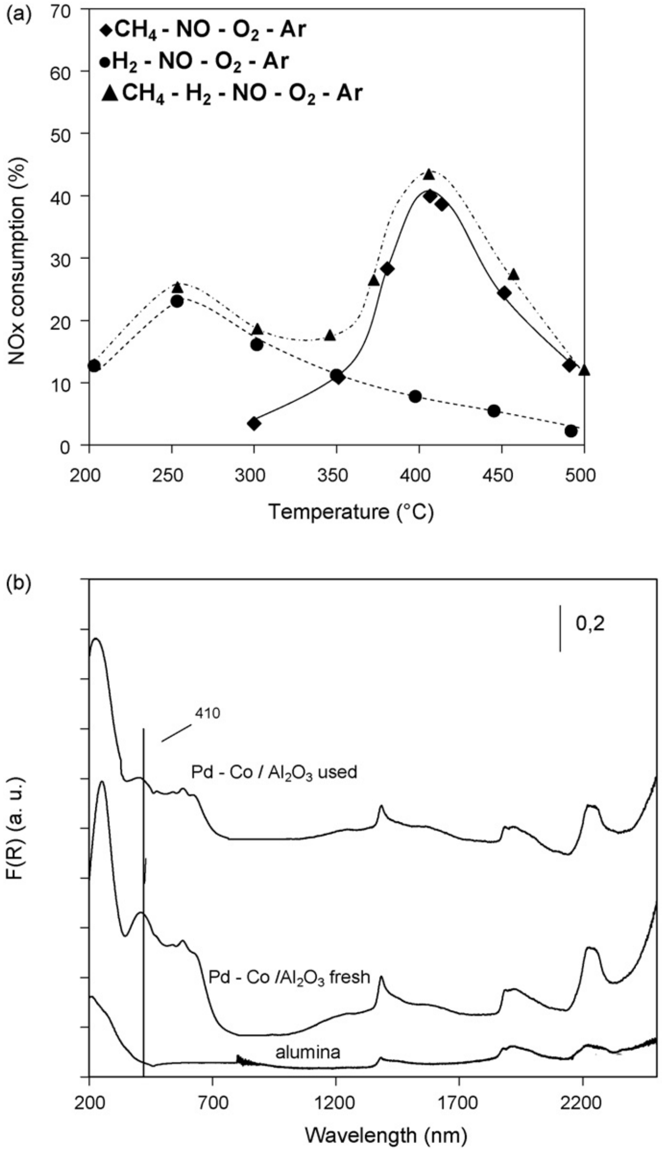

Fig. 4. (a) Isothermal steady-state consumption of NO_x in the course of CH_4–NO–O_2 (9000 ppm–150 ppm–7 vol.%) or H_2–NO–O_2 (1500 ppm–150 ppm–7 vol.%) or H_2–CH_4–NO–O_2 (1500 ppm–9000 ppm–150 ppm–7 vol.%), Ar as balance over: Co (0.51 wt.%)-Pd (0.50 wt.%)/Al_2O_3. (b) UV–visible–NIR diffuse reflectance spectra of Co(X)-Pd(X)/Al_2O_3 catalyst after CH_4–NO–O_2 and H_2–CH_4–NO–O_2 runs.

On the spectra, one can see 3 bands corresponding to Co species in tetrahedral positions as already observed on the Co/Pd catalysts [14]. These bands do not change. More over, another band is observed at 410 nm. This band corresponds to Pd^{2+} species that do no change with time on stream. Thus, we synthesized a bimetallic active catalyst for SCR NO_x by methane and hydrogen. More over, the Co-Pd catalysts are stable with time on stream. Finally, on can conclude that the active species for deNO_x assisted by methane are Co^{2+} or Pd^{2+} species and that active species for deNO_x assisted by hydrogen are Pd^{2+} species.

4. Conclusions

Reduction of NO_x by hydrogen and methane was performed on Pd/Co/alumina, Pd-Co/alumina, Pd/alumina and Co/alumina catalysts. At low temperature the methane is inert whereas the hydrogen is the reducing agent. At high temperature, the hydrogen is totally oxidized in water and the methane becomes the reducing agent (>300 °C). Co/alumina catalyst is only active with methane whereas Pd/Co/alumina, Pd-Co/alumina, Pd/alumina are active in presence of methane and hydrogen. The major active sites for deNO_x are Pd^{2+} species. However, depending on the preparation, these species are not stable and change with time on stream. Thus, on Co/Pd catalyst a deactivation is observed where as no deactivation is observed on Pd and Co-Pd catalysts. Furthermore, the co-impregnation method is the way of impregnation leading to stable catalyst for deNO_x reaction assisted by methane and hydrogen. On this bimetallic catalyst an additive effect of methane and hydrogen is observed for deNO_x reaction. UV–vis characterization of fresh and used catalysts coupled with methane oxidation reaction lead us to conclude that the active sites for deNO_x depend on the reducing agents. In presence of methane, Co^{2+} and Pd^{2+} species are the active sites whereas only Pd^{2+} species are the active sites if hydrogen is used as reducing agents.

The deNO_x reaction was performed in presence of 3% water. Similar results are obtained with a shift of 20 °C to higher temperature. This indicates that water just plays a role of ligand in the deNO_x process. More over, runs in presence of 10 ppm of SO_2 are in course to test the thioresistance of alumina supported catalysts before Syngas runs in pilot plant.

Acknowledgements

This work was carried out in the framework of the European program “EUREKA” STATIONOCAT. Rui Marques acknowledges the financial support of ADEME and Gaz de France in the form of an “ADEME” grant. Prof. Bozon-Verduraz and Dr. P. Beaunier are greatly acknowledged for UV–vis and TEM experiments, respectively.

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