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Supported gold/MO_x catalysts for NO/H_2 and CO/O_2 reactions

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Abstract

The reduction of NO with H_2 and the oxidation of CO with O_2 were investigated over silica- and alumina supported gold/metal oxide catalysts (M: Co, La, Ce). In general, the silica supported samples were less active than the alumina supported ones, probably due to the presence of large gold particles. The presence of metal oxide was beneficial to the activity in the NO/H_2 reaction and the CO/O_2 reaction over Au/SiO_2. Opposite behavior was observed with the NO reduction over Au/Al_2O_3 where the metal oxide might cover active species. Addition of cobalt oxide and lanthana improves the selectivity towards N_2. ©1999 Elsevier Science B.V. All rights reserved.

Keywords: Gold; Metal oxide; NO reduction; CO oxidation

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1. Introduction

Recently, it has been shown that gold based catalysts exhibit remarkable activity for various reactions [1–8] despite the chemical inertness of this noble metal. For example, Au/TiO_2 [9–13] Au/Fe_2O_3 [9,10,13–15] and Au/NiO [9] have been found to be extremely active for oxidation of CO by O_2, while some zeolite supported Au catalysts have been reported to be capable of reducing NO with H_2 [16,17] or CO [18].

This paper reports on a comparative study of silica and alumina supported gold/metal oxide (MO) catalysts (M = Co, La, Ce), applied to the NO/H_2 and CO/O_2 reactions. Au/Al_2O_3 has been found earlier to be active for NO reduction at 350°C [20] as well as for CO oxidation [9,10,19] The choice of CoO_x is based on our earlier research concerning Pt/MO combinations: the addition of CoO_x to a Pt/SiO_2 catalysts improves the performance in the CO oxidation with NO or O_2 [21]. A similar effect has been reported for $Pt/CoO_x/Al_2O_3$ [22,23]. Ceria is also a well known promoter of noble metals for reactions relevant for automotive catalysis. For example, Burch and Watling reported that CeO_x and LaO_x exhibit a promoter effect on Pt/Al_2O_3 in the NO_x reaction with C_3H_6 under lean conditions [24]. Au/Co_3O_4 showed excellent CO/O_2 activity [9,10,13,14]. Addition of CeO_x improves the activity of Au/MnO_x [25,26]. Many aspects concerning the catalytic activity of gold based systems are yet unclear. The presence of gold as small particles (e.g. <15 nm) appears to be crucial for high activity especially in CO oxidation [12–14,26–28]. Interaction between gold and transition metal oxide appears to be of substantial importance as well, in the case of CO oxidation [12,26,27,30,31] and the NO reduction by C_3H_8 [28]. The oxidation state of Au also may play an important role. According to Minicò et al., unstable oxidic gold species on Fe_2O_3 are the active species for CO oxidation [32]. However, our earlier results

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on Au/TiO_2 [38] and recent results by Boccuzzi et al. on Au/ZrO_2 [34] do not support their conclusion. The possible role and the instability of Au(III) and Au(I) have also been discussed by Nkosi et al. [29] for the hydrochlorination of acetylene. We have investigated silica and alumina supported Au/MO systems by activity and FTIRS measurements in order to elucidate some of the many questions and uncertainties of gold catalysis. More in particular, the questions addressed in this paper are twofold:

1. Is the effect of MO addition the same for SiO_2 and Al_2O_3 supported Au catalysts? and
2. Is the order of activity of Au/MO catalysts similar for NO/H_2 and CO/O_2 reactions?

2. Experimental

2.1. Preparation

The supported Au/MO_x catalysts were prepared by consecutive deposition of gold by homogeneous deposition precipitation and metal oxide by impregnation. First, the required amount of $HAuCl_4{\cdot}3H_2O$ (99.999%, Aldrich Chemicals) to produce a 5 wt.% gold loading was added to a suspension of either γ-Al_2O_3 (Engelhard) or SiO_2 (AeroSil, Degussa) and urea (p.a., Acros). Under vigorous stirring the temperature was kept at 80°C to allow urea to decompose ensuring a slow increase of pH. When a pH of around 8–8.5 was reached the slurry was filtrated and washed thoroughly with water and dried overnight at 80°C. Second, the metal nitrate of the desired deposited metal oxide ($Co(NO_3)_2{\cdot}6H_2O$, $Ce(NO_3)_3{\cdot}6H_2O$ or $La(NO_3)_3{\cdot}6H_2O$, 99.999%, Aldrich Chemicals) was added to a suspension of the freshly supported Au catalyst. The amounts of nitrate were chosen to result in a 10 wt.% MO loading. After the impregnation step the catalyst was ground into powder and kept overnight at 80°C. The samples were characterized by AAS to determine gold loading, XRD (Philips 1050) to estimate average particle size and with a Philips CM30 High resolution transmission electron microscope operated at 300 kV to examine the particle size distribution. Maximum resolution of the HRTEM was 0.5 nm at 5×10^5 magnification. Elemental analysis was performed with Energy Dispersive X-ray Analysis (EDX), by means of a built-in LINK EDX system.

2.2. Activity experiments

Activity tests of the catalysts were performed in a micro reactor. Typically 0.2 g of catalyst was used and the total gas flow was maintained at 40 ml min^{-1} (GHSV $\approx$ 2500 h^{-1}). Prior to the activity experiments, the catalysts were reduced in situ by 4 vol% H_2 in He for 2 h at 400°C. Gas mixtures used were NO + H_2 (ratio 0.2) and CO + O_2 (ratio 1). All gases were 4 vol% in He (Air Products). The effluent stream was analyzed by a mass spectrometer (Balzers) for the NO reduction by H_2 and by a gas chromatograph (Chrompack CP-2002) in experiments concerning the CO oxidation reaction. With the NO/H_2 reaction *m*/*z* values of 17 (NH_3), 18 (H_2O), 28 (N_2), 30 (NO) and 44 (N_2O) were monitored with the mass spectrometer. CO and O_2 were detected with a Molsieve 5 Å column and CO_2 with an Haysep A column, both mounted in the gas chromatograph. The reaction was temperature programmed with a ramp of 5° min^{-1} and in a range between 25 and 350°C. Both heating and cooling stages were recorded to establish possible differences. The process of a heating and cooling stage was repeated to detect deactivation or activation.

2.3. FTIR measurements

Catalyst powder was pressed into a disc that was mounted in a vacuum cell (base pressure $<10^{-5}$ mbar) and was reduced in situ by H_2 or oxidized by O_2 for 1 h at 350°C. Infrared spectra were recorded with a single-beam spectrometer (Mattson Galaxy 2020) operated at a resolution of 4 cm^{-1}. To reduce the noise/signal ratio 32 scans were taken per spectrum and the applied infrared range was 4000–1300 cm^{-1}. Background spectra were recorded before admitting reaction mixtures. Reactant gases used were NO (99.5%), H_2 (99.999%), CO (99.997%) and O_2 (99.998%, Messer Griesheim), and were admitted between 1 and 100 mbar. Finally, the spectra were corrected for gas phase bands of NO or CO and backgrounds were subtracted.

3. Results

3.1. Characterization

The silica supported Au catalysts contained around 4.5 wt.% Au and the alumina based samples around

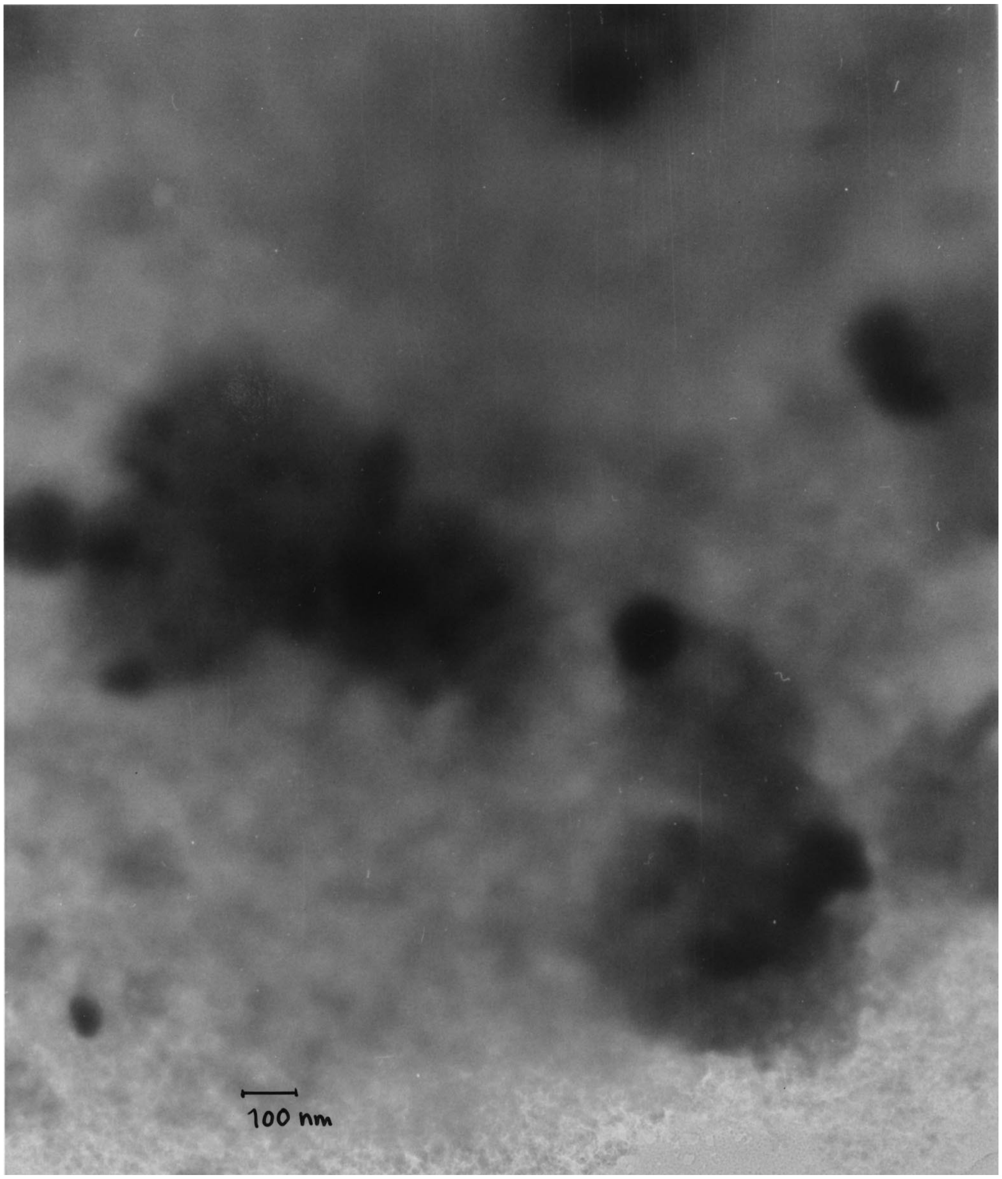

Fig. 1. HRTEM image of a freshly prepared Au/CoO_x/SiO_2 catalyst. Magnification: 1×10^5.

4.7 wt.%. XRD indicated that the average particle size of fresh Au/SiO_2 samples was 10–15 nm but considerably larger after reaction (~50 nm). The gold particles in the Au/Al_2O_3 samples were 5–10 nm and stable during the reaction. The XRD measurements of the MO containing samples did not indicate the presence of crystalline MO nor M.

The particle size distribution of gold particles on Au/MO_x/SiO_2 was determined by HRTEM. The following HRTEM images were made of catalysts that were used in the CO/O_2 reaction following prereduction. Fig. 1 shows an image of Au/CoO_x/SiO_2. Clearly visible are gold particles as spherical dark particles embedded in lighter patches of cobalt oxide,

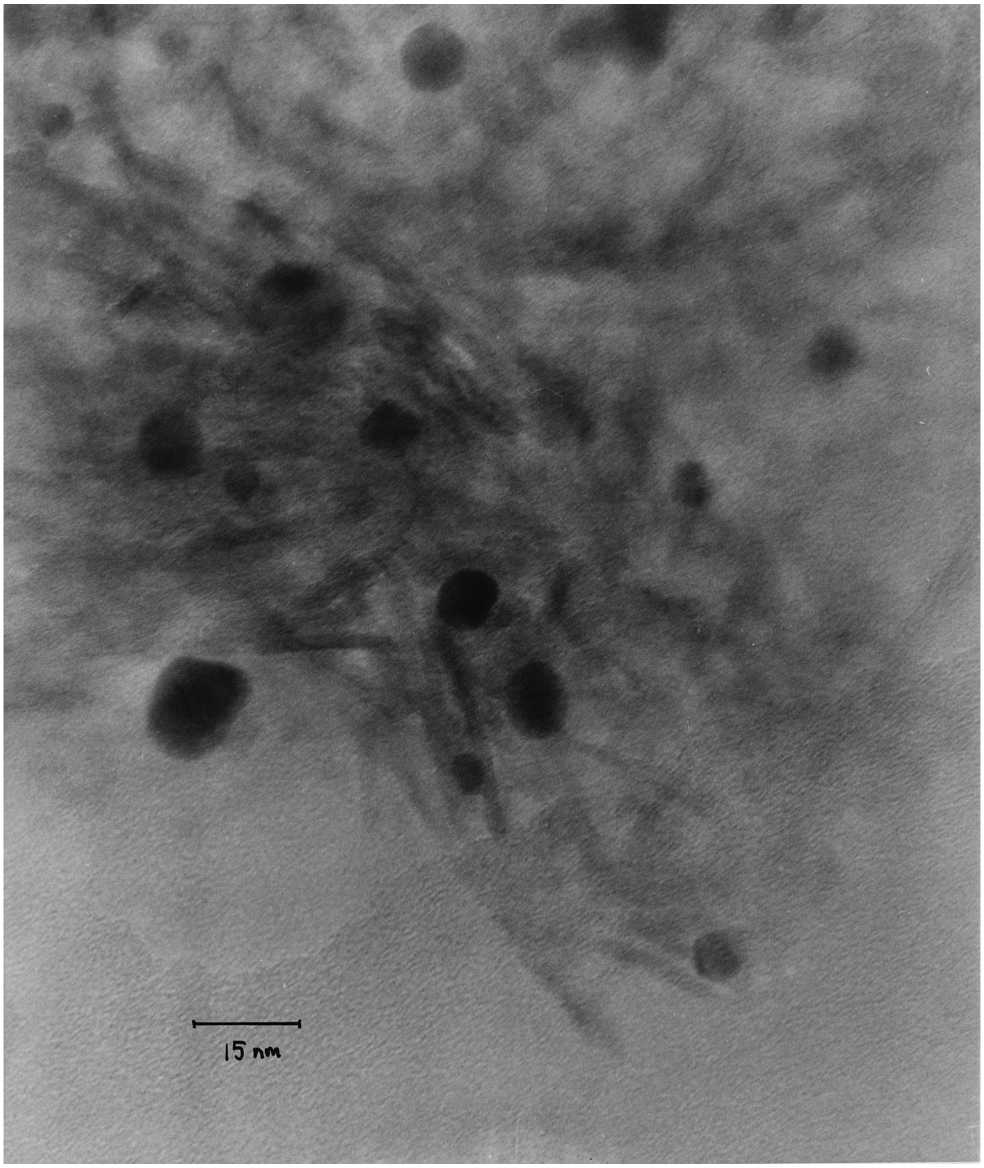

Fig. 2. HRTEM image of a used Au/CoO_x/Al_2O_3 catalyst. Magnification: 5.35×10^5.

as identified by EDX. No isolated areas of cobalt oxide were found CoO_x was rather homogeneously distributed on the surface. The particle size of gold increased considerably upon pretreatment, despite the presence of cobalt oxide. Au/LaO_x/SiO_2 and Au/CeO_x/SiO_2, were partly present on the surface in large whiskers or chunks (not shown). However, a large part was evenly distributed on the surface and in close contact with gold, as was the case with Au/CoO_x/SiO_2.

Fig. 2 shows HRTEM images made of Au/CoO_x/Al_2O_3 after pretreatment and reaction. Comparing to images of the fresh sample (not shown) the sample had been subject to sintering of gold particles The majority of the particles agglomerated from 0.5–3 nm to 10–20 nm while a few particles were

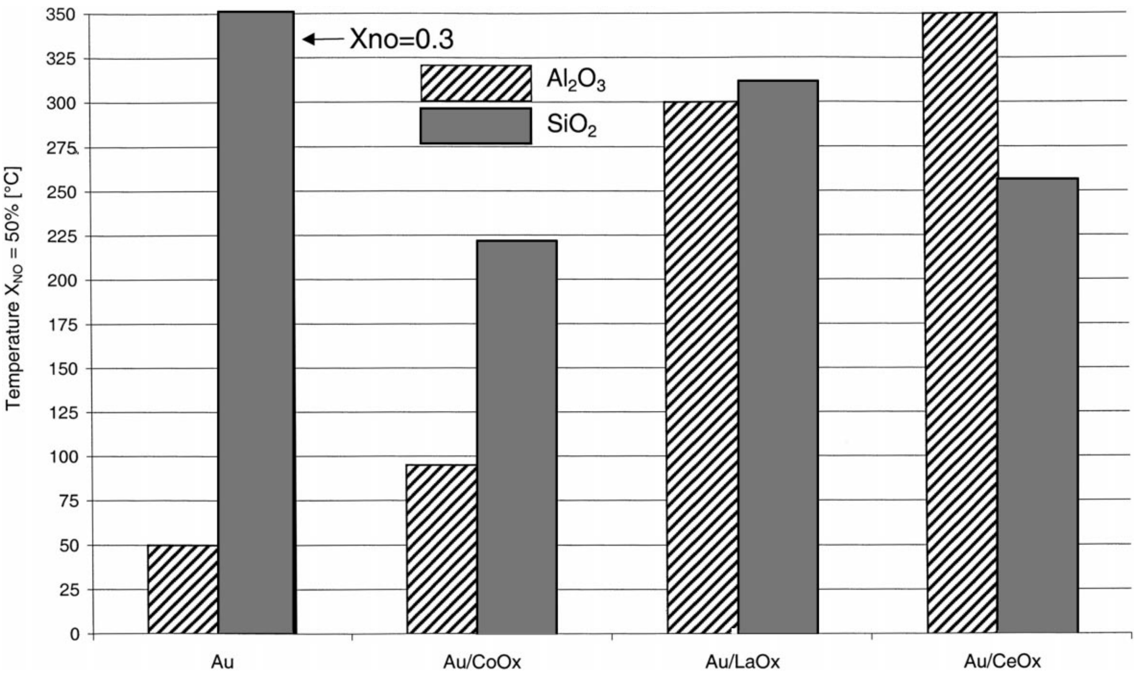

Fig. 3. Temperatures of 50% NO conversion for the NO/H_2 reaction (ratio 0.2) of Au, Au/CoO_x, Au/LaO_x,, and Au/CeO_x, supported on SiO_2 and Al_2O_3.

as large as 50 nm in diameter. Like the freshly prepared catalyst, the cobalt oxide was not visible. EDX analysis not only established its presence but also its rather inhomogeneous distribution on the sample.

3.2. NO reduction by H_2

Fig. 3 shows the temperature of 50% NO conversion over prereduced Au/SiO_2 and Au/Al_2O_3 catalysts. The capability of NO reduction improved upon adding the MOs to Au/SiO_2 and vice versa. The supported metal oxides without gold present did not reach 50% NO conversion below 350°C (see Table 1). These results point to a large synergistic effect: the activity of the Au/MO_x/SiO_2 catalysts is much better than those of the Au/SiO_2 and MO_x/SiO_2 catalysts. Most active SiO_2 supported sample was Au/CoO_x/SiO_x showing a NO conversion of 50% at 220°C. This sample eventually reached full NO conversion above 250°C whereas Au/LaO_x/SiO_2 and Au/CeO_x/SiO_2 only converted NO at 350°C for 60 and 80%, respectively. With Au/CeO_x/SiO_2 the conversion of 80% was already reached at 300°C. It appeared to be the maximum conversion possible for this sample. In Table 1 the selectivities toward NH_3 and N_2 at 350°C are listed for a NO/H_2 ratio of 0.2. Au/SiO_2 only produced NH_3 under these conditions. The presence of MO's increased the N_2 formation to about 30% of the NO converted.

Table 1
NO conversion at 350°C of supported metal oxides[a]

Supported MO_x	SiO_2 (%)	Al_2O_3 (%)
CoO_x	15	35
LaO_x	~0	20
CeO_x	25	25

[a] Note: NO conversions are approximate (+/−5%).

It has been shown before [33] that Au/Al_2O_3 is also capable of reducing NO by H_2 at low temperatures. The effects of the addition of metal oxides are strikingly different compared to the silica supported catalysts. The addition of MO's suppressed the NO conversion over the Al_2O_3 supported catalysts. Over

Au/Al_2O_3 and $Au/CoO_x/Al_2O_3$ full conversion was attained above 200°C but the use of $Au/LaO_x/Al_2O_3$ and $Au/CeO_x/Al_2O_3$ resulted in a conversion of only around 50% in the applied temperature range. An oxidative pretreatment led to partly the same results but the differences in behavior of the four catalysts were much smaller than observed after prereduction. The $T_{50\%}$s of Au/Al_2O_3, $Au/CoO_x/Al_2O_3$ and $Au/CeO_x/Al_2O_3$ were in close range of each other, only LaO_x addition appeared to be beneficial to the activity. Au/Al_2O_3 was selective towards N_2O at low temperatures (up to 100°C), formed N_2 between 100 and 250°C and produced mainly NH_3 at higher temperatures [33]. All samples produced amounts of N_2O at temperatures under 100–150°C. CoO_x and LaO_x containing samples increased the N_2 formation at high temperatures, as shown in Table 2. The pretreatment did not influence the selectivities much.

Infrared spectra did not reveal any evidence of NO adsorbed on Au, as observed on Au/TiO_2 [33]. Fig. 4 shows spectra observed for NO/H_2 mixtures on preoxidized $Au/CoO_x/Al_2O_3$. No absorption bands were visible (spectrum a) when only NO was present. However, when an equal amount of hydrogen was added (b) bands at 1863 and 1790 cm^{-1} appeared. Those bands can most likely be assigned to NO adsorbed on cobalt oxide [35,36]. More addition of H_2 (c) induced shifts in the above mentioned bands as well as a new small band at 1979 cm^{-1}. Similar spectra were obtained when the sample was prereduced. $Au/LaO_x/Al_2O_3$ and $Au/CeO_x/Al_2O_3$ showed no absorption bands except of nitrate species, exhibited by all samples.

Table 2
Selectivities towards NH_3 and N_2 at 350°C for the NO/H_2 reaction (ratio 0.2)

Sample	S_{NH3}	S_{N2}
Au/SiO_2	1	0
$Au/CoO_x/SiO_2$	0.65	0.35
$Au/LaO_x/SiO_2$	0.70	0.30
$Au/CeO_x/SiO_2$	0.80	0.20
Au/Al_2O_3	1	0
$Au/CoO_x/Al_2O_3$	0.70	0.30
$Au/LaO_x/Al_2O_3$	0.65	0.35
$Au/CeO_x/Al_2O_3$	0.95	0.05

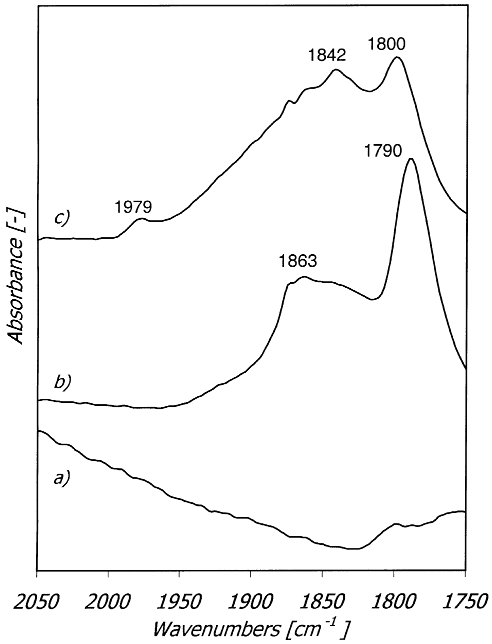

Fig. 4. Infrared spectra at 50°C of $Au/CoO_x/Al_2O_3$ with (a) 20 mbar NO, (b) 20 mbar NO + 20 mbar H_2 and (c) 20 mbar NO + 60 mbar H_2.

3.3. CO oxidation by O_2

Fig. 5 shows an overview of the catalytic performance of the prereduced catalysts for the CO/O_2 reaction. Generally, the temperature giving 50% CO conversion ($T_{50\%}$) decreased when a metal oxide was added to Au/Al_2O_3 or Au/SiO_2. Table 3 shows the CO conversion at 350°C for the supported MO_x only. The MO_x samples were not able to convert CO for more than 50% below 350°C except CoO_x/Al_2O_3. Its $T_{50\%}$ was at 1 70°C, much higher than Au/Al_2O_3 and $Au/CoO_x/Al_2O_3$.

The largest effect was established with silica-based catalysts. The Au/SiO_2 catalyst showed a $T_{50\%}$ of

Table 3
CO conversion at 350°C of supported metal oxides

Supported MO_x	SiO_2 (%)	Al_2O_3 (%)
CoO_x	15	100[a]
LaO_x	~0	20
CeO_x	25	25

[a] $T_{50\%}$: 170°C (see text).

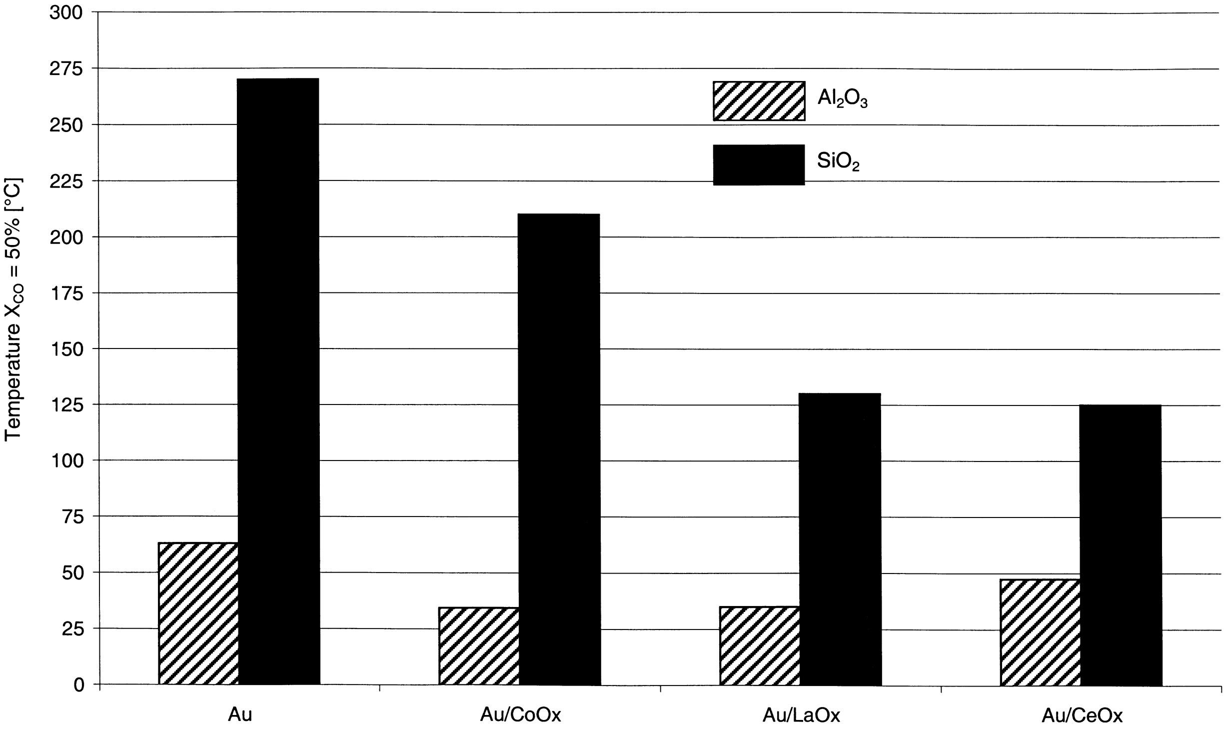

Fig. 5. Temperatures of 50% CO conversion for the CO/O_2 reaction (ratio 1) of Au, Au/CoO_x/Au/LaO_x and Au/CeO_x, supported on SiO_2 and Al_2O_3.

240°C, but with metal oxide present it decreased dramatically. Addition of CoO_x ($T_{50\%} = 185°C$) and LaO_x ($T_{50\%} = 135°C$) were beneficial but adding CeO_x had the largest effect ($T_{50\%} = 115°C$). Also shown are the results obtained for alumina based catalysts. Clearly, Au/Al_2O_3 is more active than Au/SiO_2, as has been shown elsewhere [33], but the effects resulted by adding metal oxide were less pronounced as for Au/SiO_2. However, in this context it should be emphasized that all the Al_2O_3 supported catalysts exhibit a much lower $T_{50\%}$ than the most active SiO_2 supported samples. Contrary to Au/SiO_2, the presence of CoO_x had the largest positive effect while addition of CeO_x produced little improvement in activity. The preoxidized samples exhibited different behavior. It appeared that during the first heating stage of the activity experiment, the CO conversion remained considerably lower than after prereduction. At around 70°C the conversion increased to levels similar after a reductive pretreatment. The three remaining stages all resembled the activity pattern of prereduced Au/MO_x/Al_2O_3. The negative effect of preoxidation on activity has already been reported for Au/TiO_2 [38] and for Au/Al_2O_3 [33] and it is referred to as initial 'inactivity'. The initial inactivity was total with Au/Al_2O_3 but only partly with Au/MO_x/Al_2O_3 (see Table 4).

Table 4
CO conversion for CO/O_2 reaction (ratio 1) at room temperature over preoxidized Au/MO_x/Al_2O_3 catalysts

Catalyst	1st Stage	2nd, 3rd and 4th Stage
Au/Al_2O_3	~0	0.40
Au/CoO_x/Al_2O_3	0.85	0.60
Au/LaO_x/Al_2O_3	0.20	0.85
Au/CeO_x/Al_2O_3	0.30	0.80

Fig. 6 shows FTIR spectra in the presence of a CO/O_2 mixture both for prereduced and preoxidized Au/Al_2O_3 and Au/CeO_x/Al_2O_3 catalysts at 50°C. Carbonate species were detected (1650 and 1435 cm^{-1}) on all four samples. The capability of CO oxidation of these catalysts was clearly visible by the appear-

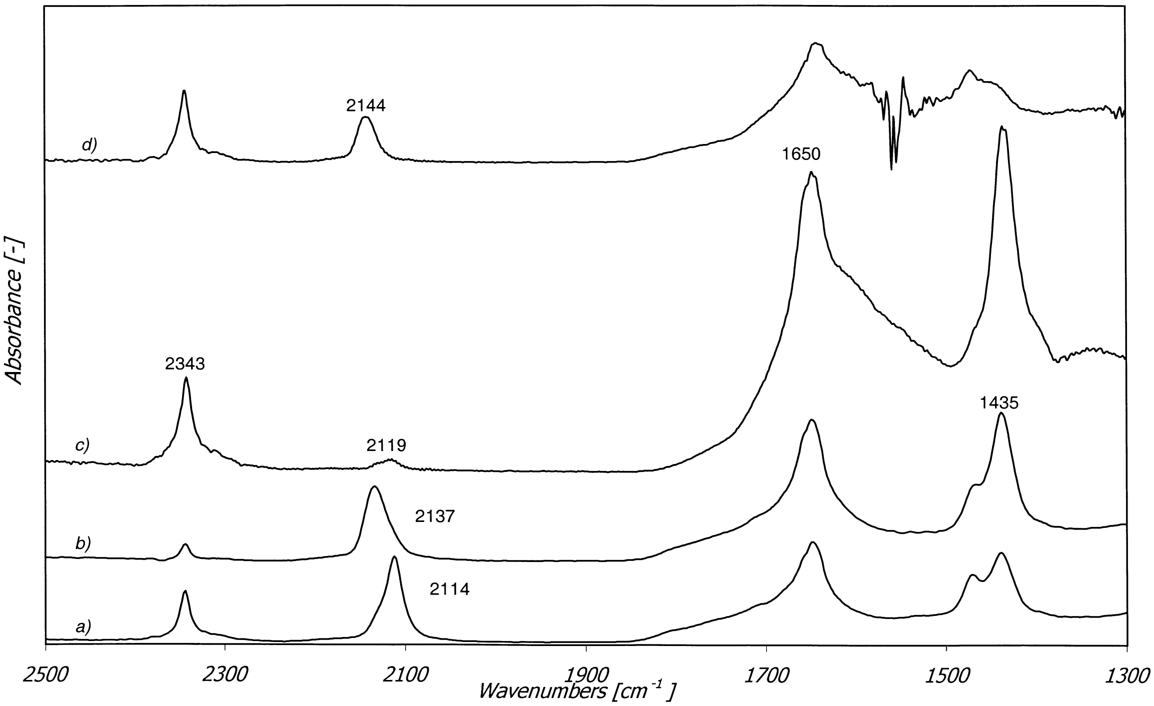

Fig. 6. Infrared spectra at 50°C of a CO/O_2 mixture (30 mbar, ratio 1) with (a) prereduced Au/Al_2O_3, (b) preoxidized Au/Al_2O_3, (c) prereduced $Au/CeO_x/Al_2O_3$ and (d) preoxidized $Au/CeO_x/Al_2O_3$.

ance of a band at 2343 cm^{-1}, which is characteristic of CO_2. The spectra of the prereduced samples (a and c) show a absorption band at ~2116 cm^{-1} that can be assigned to CO adsorbed on metallic gold (Au^0). On the preoxidized samples, however, this CO band appeared to be shifted towards ~2140 cm^{-1}. The differences between Au/Al_2O_3 and $Au/CeO_x/Al_2O_3$ were the larger CO_2 bands and the smaller bands at 2116 and 2140 cm^{-1} which were also slightly shifted towards higher wavenumbers compared with Au/Al_2O_3. The carbonate bands appeared to be larger with prereduced $Au/CeO_x/Al_2O_3$ but smaller when preoxidized. The samples containing CoO_x or LaO_x (not shown) showed similar behavior with the following exceptions. On prereduced $Au/CoO_x/Al_2O_3$ the CO absorption band at 2115 cm^{-1} was broader and in 30 min the band split up into two small bands at 2121 and 2167 cm^{-1}. Preoxidation resulted in an intense band at 2135 cm^{-1}. $Au/LaO_x/Al_2O_3$ exhibited a broad band at 2119 cm^{-1} when prereduced and a large band at 2135 $cm^{-1}$when preoxidized.

4. Discussion

The mechanism of the high activity of gold based catalysts is still unclear. It has been established that very small gold particles are required for high activity [1,12–14,26–28]. The mechanisms proposed include

1. very small Au particles are needed because of a different electronic structure [41],
2. the active species is ionic gold [32],
3. low co-ordination sites in the form of specific ensembles of gold atoms act as active sites.

In this light, the beneficial role of MO addition may be related to stabilization of small gold particles or to stabilization of ionic gold species. In addition, it has been suggested that the reaction may only take place at the gold/MO interface. It also cannot be excluded a priori that the catalytic action mainly takes place on special MO sites which are created by the presence of gold.

The Pt group metals are very efficient catalysts both for the NO/H_2 and CO/O_2 reactions. It is accepted that on these metals NO reduction proceeds by NO dissociation and CO oxidation by a Langmuir-Hinshelwood mechanism with adsorbed CO and adsorbed O. For gold catalysts, however, alternative mechanisms cannot be excluded in particular for the NO/H_2 reaction since there is no experimental evidence that NO dissociation occurs on Au. Galvagno and Parravano proposed that the reaction proceeds via an Eley-Rideal mechanism with molecularly adsorbed NO and gas phase H_2 [42]. Nitrate species have been detected on a number of Au based catalysts [33] which might point to a possible role of NO_2 as reaction intermediate. It was hoped that the study described in the present paper — a comparison of NO reduction with H_2 and CO oxidation with O_2 over Au/SiO_2, Au/Al_2O_3, three $Au/MO_x/SiO_2$ and $Au/MO_x/Al_2O_3$ catalysts ($MO = CoO_x$, LaO_x and CeO_x) — could elucidate some of the problems concerning catalysis by gold.

Our results show that Au based catalysts which are active in the CO/O_2 reaction are also active in the NO/H_2 reaction. Alumina supported catalysts are much more active than the silica supported samples. Addition of CoO_x, LaO_x and CeO_x results in an improved activity for the less active SiO_2 supported catalysts in both reactions. For the most active Al_2O_3 supported Au catalysts MO addition results in better activity at low temperatures for CO oxidation. However, MO addition has a detrimental effect on the activity for the NO/H_2 reaction, in particular for LaO_x and CeO_x. For the NO/H_2 reaction the highest activity has been found for the non-promoted Au/Al_2O_3 catalysts.

The difference in activity for CO oxidation and NO reduction between silica supported and alumina supported catalysts can be related to different gold particle sizes [33]. As the HRTEM images of the Au/MO catalysts show the presence of metal oxides cannot prevent the initially small gold particles on silica from sintering. The images also reveal close contact between MO and gold, which may be required to obtain enhanced activity. A Langmuir-Hinshelwood mechanism could be the main reaction mechanism of the CO/O_2 reaction on Au/Al_2O_3 with both CO and O adsorbed on Au. The beneficial effect of CeO_x and CoO_x addition may be interpreted in terms of a role of MO as additional oxygen supplier, similar to what was proposed for $Pt/CoO_x/SiO_2$ [37]. Such a mechanism was also suggested for Au/TiO_2 and Au/Co_3O_4 [10]. Srinivas et al. [13] reported inhibition of CO oxidation by carboxylate and carbonate species on Au/Co_3O_4, as observed with FTIRS. The infrared measurements described in this study also indicate formation of carbonates but it did not point to an inhibition of CO oxidation by carbonates.

It can be suggested that, based on the FTIRS results, surface gold is present as $Au^{\delta+}$ after preoxidation, and that oxidic gold is not active for CO oxidation. When the temperature reaches 60–70°C the reactant CO reduces $Au^{\delta+}$ to metallic gold, thus enabling the CO/O_2 reaction to proceed [38]. In this light, the absence of initial inactivity in case of the NO/H_2 reaction might be related the fact that hydrogen is a more powerful reducing agent than CO. In addition, the presence of oxygen in the gas phase may retard the reduction of $Au^{\delta+}$ by CO below $\sim$60°C.

Adsorption of NO on Au/Al_2O_3 was not detected by FTIRS. Apparently, the Au–NO bond is too weak to make the NO concentration sufficiently high at room temperature. The improvement of Pt/SiO_2 for nitric oxide conversion to nitrogen by the presence of CoO_x [40] was suggested to originate from an enhancement of NO dissociation. Although no direct evidence is found for NO adsorption of gold, cobalt oxide could have a similar effect on gold based catalysts. It would then explain the increased N_2 selectivity for $Au/CoO_x/Al_2O_3$. It has also been reported that lanthana can promote nitric oxide reduction on Pt and Rh [39]. Possibly, LaO_x depresses low temperature activity by partly covering active Au sites but promotes N_2 formation at higher temperatures.

The ambivalent activity results for silica and alumina based Au/MO_x catalysts in the NO/H_2 reaction may originate from site blocking by the metal oxide on Au/Al_2O_3. Yet $Au/MO_x/Al_2O_3$ has been found to be very active for CO oxidation. It might be that NO reduction by H_2 will require larger ensembles of gold atoms than needed for the CO/O_2 reaction. The metal oxide may perhaps partly cover the active sites at low temperatures resulting in ensembles of Au atoms that can oxidize CO but that are too small for NO reduction.

5. Conclusions

With respect to the two questions stated in the introduction we can conclude the following:

Silica supported gold catalysts are poorer in both NO reduction and CO oxidation compared to alumina supported samples, due to increased sintering of gold particles on silica.

The addition of CeO_x, CoO_x or LaO_x improves the activity in the NO/H_2 reaction on Au/SiO_2, and in the CO/O_2 reaction on Au/Al_2O_3 and Au/SiO_2.

CoO_x and LaO_x addition improves the selectivity towards N_2 formation.

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