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Screening of bifunctional water-gas shift catalysts

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Abstract

A large number of different formulations have been recently proposed in the literature as new catalysts for the water-gas shift (WGS) reaction. These formulations typically consist of a metal deposited on a reducible support. As these catalysts have been synthesized and tested by different groups in different operating conditions, a true comparison of their activities is not really possible. The aim of this study is to screen these samples under identical conditions using a model reformate as reaction mixture. A commercial parallel reactor has been used for this task. Comparison of the data obtained for the Pt catalysts from the parallel reactor with those obtained from a single fixed bed reactor showed deviations of 20–30% in the kinetic parameters. Rh and Ru based catalysts produced significant amounts of methane. $Pt/CeO_2/Al_2O_3$ and Pt/TiO_2 were found to be the most active catalysts for the high temperature water-gas shift while gold and copper catalysts showed promising results for low temperature applications, but they require testing at lower CO partial pressures.

Keywords: WGS; Oxides; Methanation; Parallel reactor; Hydrogen purification

1. Introduction

In recent years proton-exchange membrane (PEM) fuel cells have developed into technically and economically viable devices that can provide electricity from hydrogen with very high efficiency and water being the only direct product formed. Currently hydrogen is produced almost exclusively by steam reforming or partial oxidation of hydrocarbons or alcohols. This leads to hydrogen rich mixtures that usually contain varying amounts of carbon oxides. PEM fuel cells have a low tolerance towards CO, depending on their operating temperature. The water-gas shift (WGS) reaction reacts carbon monoxide with water into hydrogen and carbon dioxide, thus lowering the CO content and producing extra hydrogen. The reaction is slightly exothermic and high equilibrium conversions are obtained at low temperatures. The best commercial low temperature water-gas shift (LT-WGS) catalysts are $CuO/ZnO/Al_2O_3$ formulations operated at industrial level in the temperature range between 180 and 250 °C. Besides high activity, other requirements have to be fulfilled for PEM applications: fast response, long lifetime and non-pyrophoric materials. Several new WGS catalysts have been reported in the literature such as Au/Fe_2O_3 [1,2], Au/CeO_2 [2,3], Au/TiO_2 [4], Ru/ZrO_2 [5], Rh/CeO_2 [6] Pt/CeO_2 [2,7–9], Pt/ZrO_2 [10], Pt/TiO_2 [11], Pt/Fe_2O_3 [12], Pd/CeO_2 [13] as promising catalysts for fuel cell applications as they are highly active in the range of 250–400 °C and often less sensitive to air exposure as the industrial ones. These catalyst formulations typically consist of a precious metal (Pt, Rh, Ru, Pd, Au) deposited on a (partially) reducible support (CeO_2, TiO_2, ZrO_2). Some non-noble metal-based catalysts have also been reported: Cu/CeO_2 [14,15], Ag/TiO_2 [4], Cu/TiO_2 [4] and Cu/ZrO_2 [16]. Grenoble et al. [17] and Panagiotopoulou and Kondaridis [18] showed that these catalysts are bifunctional, i.e. both the metal and support have a significant influence on the overall performance. However, comparing these reported catalysts is difficult, as they have been tested under very different operating conditions, in most cases with a mixture of pure carbon monoxide and water. The reaction products, hydrogen and carbon dioxide, have been found to have usually an inhibiting effect on the reaction rate. Moreover, most often, the synthesis method has a large impact on the performance. The first step in a better understanding into

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the bifunctional reaction mechanism is to compare the catalysts under identical conditions. This study uses a commercial high-throughput equipment consisting of a 16 parallel reactor set-up to compare the activity and selectivity of bifunctional WGS catalysts.

2. Experimental

2.1. Catalyst preparation and characterization

In most cases, catalysts were prepared by impregnation (incipient wetness impregnation) of the support with a solution of the corresponding metal precursors $(NH_3)_4Pt(OH)_2$, $Pd(NO_3)_2$, $RhCl_3$, $RuCl_3$ or $Cu(NO_3)_2 \cdot 3H_2O$. The supports used were commercial metal oxide powders chosen to have medium range surface areas (30–80 m^2/g), except for Fe_2O_3 (specific surface area determined by the BET method): CeO_2 (54 m^2/g), TiO_2 (80 m^2/g), ZrO_2 (31 m^2/g), Fe_2O_3 (7 m^2/g). The impregnated materials were dried overnight at 80 °C and calcined at 400 °C for 3–5 h. Rh and Ru catalysts were pre-calcined at 200 °C for 2 h and washed with a diluted NH_4OH solution.

Au/CeO_2 and Au/ZrO_2 were prepared by the deposition–precipitation method. This involves the deposition of gold hydroxide onto the support through a chemical interaction between $HAuCl_4 \cdot 3H_2O$ and NaOH in an aqueous solution at pH 9.5. A CeO_2 support with a high surface area (276 m^2/g) was used. The catalysts were filtrated, washed with water, dried at 80 °C overnight and calcined at 300 °C for 2 h. The theoretical metal loadings of the catalysts thus prepared varied between 2 and 5 wt.%.

Copper catalysts with higher loadings (10 wt.% Cu) were prepared using the co-precipitation method from a mixture of $Cu(NO_3)_2 \cdot 3H_2O$, $Ce(NO_3)_3 \cdot 6H_2O$, $ZrO(NO_3)_2 \cdot xH_2O$ or $Fe(NO_3)_3 \cdot 9H_2O$ and NaOH solution at 50 °C and constant pH of 9. The catalysts were dried overnight at 80 °C and calcined at 500 °C for 3 h.

Commercial catalysts from World Gold Council were used for Au/TiO_2 and Au/Fe_2O_3 catalysts.

A 1% $Pt/CeO_2/Al_2O_3$ catalyst was prepared in a previous study by a sol–gel method based on the Yoldas procedure [19] and used as a reference for noble metal supported catalysts.

The metal content of the catalysts was determined by elemental chemical analysis with a plasma atomic emission spectrometer (Spectroflame-ICP D-SPECTRO, Induced Coupled Plasma).

Specific surface areas of supports and catalysts were measured on a nitrogen adsorption volumetric device (Micromeritics ASAP 2020) according to the BET method.

2.2. Catalytic performance tests

The catalytic screening was realized in a 16 channel-multitubular reactor (SWITCH 16 reactor system by AMTEC GmbH). A detailed description of the experimental apparatus and of the reactor can be found in previous communications [20,21]. Two separate feed lines equipped with mass flow controllers (Brooks MFC-5850) allow to feed one reactor with a specific feed composition (mixture 1) while the other 15 are fed with a second composition (mixture 2), which can be identical to or different from mixture 1. One reactor at a time, the one fed by mixture 1, is analyzed on-line.

The reactors (internal diameter of 7 mm) were charged with 0.1 g of catalyst (particle size: $d = 0.1$–0.2 mm). All catalysts were reduced at 250 °C for 30 min under a 10 vol.% H_2 (balance: He) gas flow. The total gas flow in each reactor was 100 N mL/min.

The catalytic performance of the catalysts for the WGS reaction was investigated at three temperatures: 250, 300 and 350 °C. A feed stream of 100 N mL/min consisting of 10% CO, 10% CO_2, 20% H_2O, 30% H_2 and 30% Ar (typical reformate mixture) was used. A replicate experiment at 250 °C was realized after the catalytic test at 350 °C, to determine the deactivation of the catalysts. An additional test was carried out at 300 °C with 10% CO and 20% H_2O diluted in Ar. The steam was provided by a Shimadzu LC-10 pump connected to a controlled evaporator mixer (CEM-Bronkorst). When a reactor was fed with the reaction gas mixture, the other 15 were fed with He (100 N mL/min). Molar fractions of CO and CO_2 were measured by a chromatograph (Agilent GC6890). All measurements were performed twice. The conversion of carbon monoxide is given by:

$$X_{CO} = \frac{F_{CO}^{in} - F_{CO}^{out}}{F_{CO}^{in}}$$

the carbon dioxide yield by:

$$Y_{CO_2} = \frac{F_{CO_2}^{out} - F_{CO_2}^{in}}{F_{CO}^{in}}$$

and the methane yield by:

$$Y_{CH_4} = \frac{F_{CH_4}^{out}}{F_{CO}^{in} + F_{CO_2}^{in}}$$

where F_j^{in} and F_j^{out} are the molar flow rates (mol s^{-1}) of component j at the reactor inlet and outlet, respectively. The carbon mass balance was always closed within 5%.

For the single fixed bed experiments the catalyst samples (200 mg) were tested in a 4 mm inner diameter quartz tube reactor with a total flow rate of 200 N mL/min with the same composition as for the parallel reactor.

3. Results and discussion

The metal loading and the surface area of the catalysts studied are reported in Table 1. Only in the case of catalysts prepared by impregnation or deposition–precipitation, a measurement of the change in surface area before and after calcinations was possible. No significant variations in the surface area of the supports were observed after calcinations of the catalysts except for the Au/CeO_2. After calcination the surface area of the Au/CeO_2 decreased from 276 to 232 m^2/g. As mentioned above, the expected metal content should be in the range of 2–5 wt.%. This is not the case for the Rh and Ru catalysts where significantly lower contents have been measured. The washing procedure used

Table 1
List of catalysts studied, ICP and surface area characterizations

Catalysts	Synthesis	Calcination temperature	Metal loading (wt.%)	Specific surface support area (m^2/g)
$Pt/CeO_2/Al_2O_3$	Sol–gel (Yoldas)	500 °C/5 h	0.9	250
Pt/ZrO_2	IWI	400 °C/4 h	1.5	30
Pt/CeO_2	IWI	400 °C/4 h	2	54
Pt/TiO_2	IWI	400 °C/4 h	1.9	78
Pt/Fe_2O_3	IWI	400 °C/5 h	1.5	6
Au/CeO_2	DP	300 °C/2 h	5	232
Au/ZrO_2	DP	300 °C/2 h	1.2	30
Cu/CeO_2	IWI	400 °C/4 h	2.1	56
Cu/ZrO_2	IWI	400 °C/4 h	2.1	29
Cu/TiO_2	IWI	400 °C/4 h	2.5	74
Cu/CeO_2	CP	500 °C/3 h	8.9	87
Cu/ZrO_2	CP	500 °C/3 h	10.5	55
Cu/Fe_2O_3	CP	500 °C/3 h	9.1	21
Rh/CeO_2	IWI	400 °C/4 h	1.5	55
Rh/ZrO_2	IWI	400 °C/3 h	1.1	28
Rh/Fe_2O_3	IWI	400 °C/3 h	0.4	6
Pd/CeO_2	IWI	400 °C/3 h	1.7	56
Ru/Fe_2O_3	IWI	400 °C/3 h	2.8	6
Ru/CeO_2	IWI	400 °C/3 h	1.5	54
Au/TiO_2	World Gold Council*		1.5	57
Au/Fe_2O_3	World Gold Council*		5	39

IWI: incipient wetness impregnation.
DP: deposition-precipitation.
CP: co-precipitation.
* Commercial catalyst.

for Rh and Ru catalysts probably eliminated a part of metal deposited on the support and it is also known that volatile ruthenium oxides could be formed at a temperature as low as 150 °C under oxidizing atmosphere.

The CO conversions expressed as a function of the temperature for all the samples run in the parallel reactor set-up are shown in Figs. 1–4. Each data point in the graphs is the average value of two analyses. The data is plotted as a function of the temperature set points, not the actual bed temperatures.

Rh/ZrO_2, Rh/CeO_2 and Ru/CeO_2 catalysts are highly active with CO conversions up to 90% at 350 °C. This value exceeds the maximal CO conversion value predicted by the thermodynamics for the WGS reaction only (X_{CO} = 75% at 350 °C) for these operating conditions. In this case, deviations from thermodynamics stem from the formation of methane through the methanation reactions:

$$CO + 3H_2 \leftrightarrows CH_4 + H_2O \quad (1)$$

$$CO_2 + 4H_2 \leftrightarrows CH_4 + 2H_2O \quad (2)$$

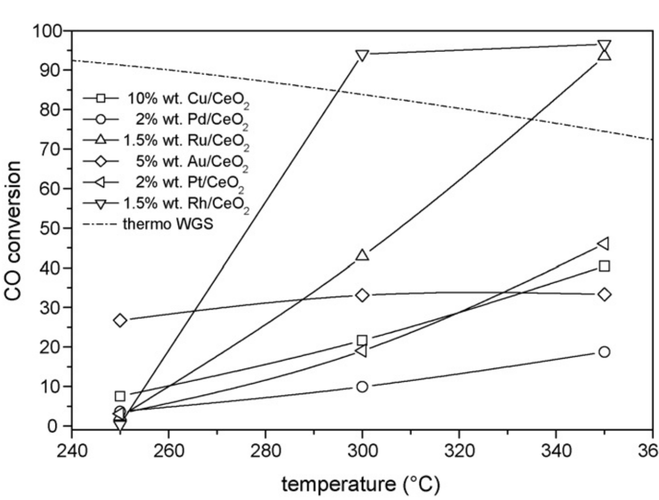

Fig. 1. CO conversion as a function of the temperature for different metals supported on CeO_2, 100 mg of sample, 100 N mL/min of 10% CO, 10% CO_2, 20% H_2O, 30% H_2, 30% Ar. The CO conversion calculated according the thermodynamics take into account only the reaction $CO + H_2O = CO_2 + H_2$.

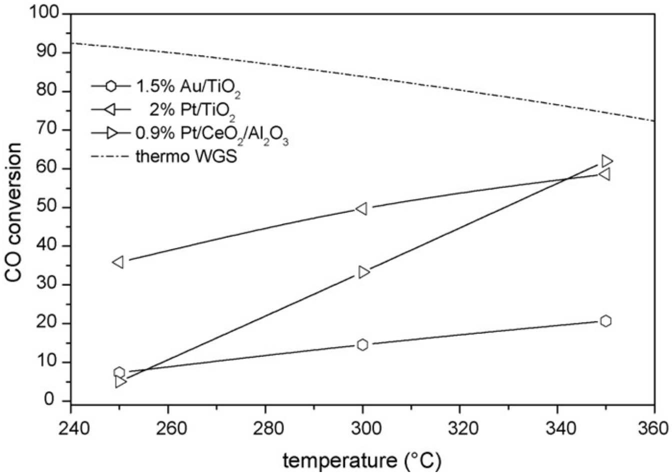

Fig. 2. CO conversion as a function of the temperature for different metals supported on TiO_2 and CeO_2/Al_2O_3. 100 mg of sample, 100 N mL/min of 10% CO, 10% CO_2, 20% H_2O, 30% H_2, 30% Ar. The CO conversion calculated according the thermodynamics take into account only the reaction $CO + H_2O = CO_2 + H_2$.

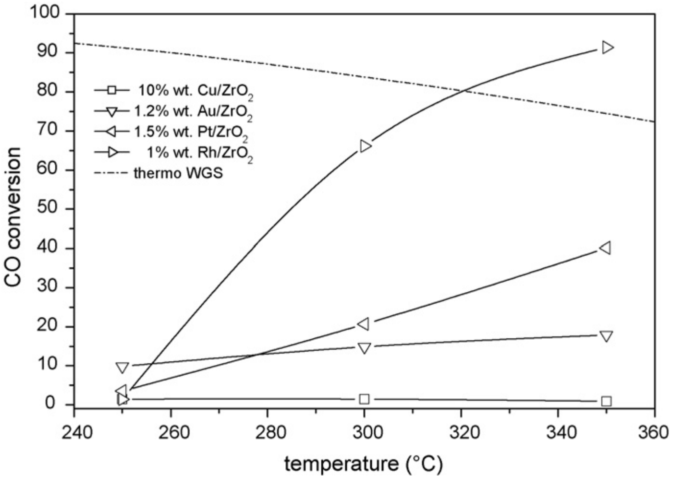

Fig. 3. CO conversion as a function of the temperature for different metals supported on ZrO_2, 100 mg of sample, 100 N mL/min of 10% CO, 10% CO_2, 20% H_2O, 30% H_2, 30% Ar. The CO conversion calculated according the thermodynamics take into account only the reaction $CO + H_2O = CO_2 + H_2$.

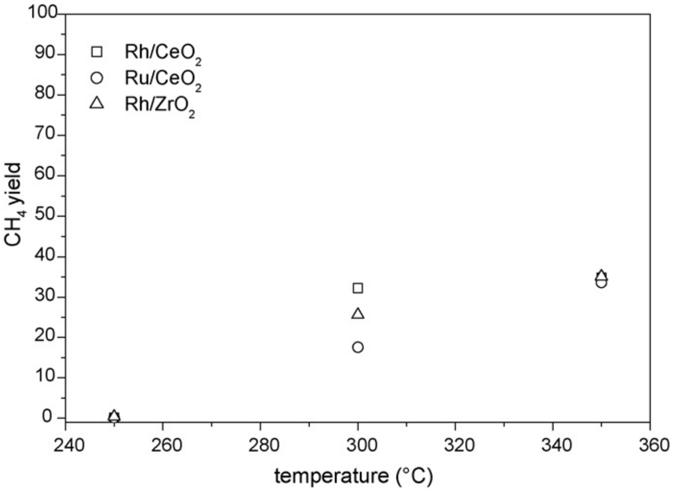

Fig. 5. CH_4 yield as a function of temperature with a reformate mixture (100 N mL/min of 10% CO, 10% CO_2, 20% H_2O, 30% H_2, 30% Ar) for Rh/CeO_2, Ru/CeO_2 and Rh/ZrO_2 catalysts, 100 mg of sample.

Fig. 5 shows the production of methane as a function of temperature for Rh/ZrO_2, Rh/CeO_2 and Ru/CeO_2 samples. An important methane yield was observed from 300 °C onwards. Rh/Fe_2O_3, Ru/Fe_2O_3 catalysts showed much lower quantities of methane produced (less than 1%), but showed hardly any activity at all. In many studies, Rh and Ru catalysts were found to promote the methanation reaction [22,23]. Utaka et al. [24] reported that Ru catalysts supported on CeO_2 or ZrO_2 demonstrated high activity for the methanation from CO and CO_2. A small production of methane, less than 1%, was also observed for Pt/CeO_2, Pd/CeO_2 and Pt/ZrO_2 at 350 °C.

Pd/CeO_2 catalyst was the less active of all catalysts supported on ceria (Fig. 1), although Wang et al. [13] reported 25% of CO conversion at 250 °C under a reaction mixture with CO and H_2O.

Pt and Au catalysts show high activity in comparison with other noble metal supported catalysts. At 250 °C, Pt/TiO_2 and Au/CeO_2 catalysts exhibit high activity with 40% and 30% of CO conversion, respectively. The Pt catalysts at 300 °C can be classified in the following order: $Pt/TiO_2 > Pt/CeO_2/Al_2O_3 > Pt/ZrO_2 \geq Pt/CeO_2 > Pt/Fe_2O_3$.

The CO conversion over the Au/CeO_2 catalyst amounted to approximately 30%, almost independent of the temperature. The other Au catalysts have much lower activities that varied only slightly with temperature.

Copper catalysts were tested for comparison with noble metal catalysts. Activities of 10% Cu/CeO_2 and 10% Cu/Fe_2O_3 follow the activities of Pt/CeO_2 and Au/Fe_2O_3, respectively. Increasing the Cu loading from 2.5% (not shown) to 10% did not have a drastic effect on the catalytic activity. The CO conversion was identical at 250 and 300 °C for both catalysts. The 10% Cu/CeO_2 catalyst became more active at 350 °C with an increase of 10% of the CO conversion. The Cu/ZrO_2 catalyst showed very low activity. Only Au/Fe_2O_3 and Pt/TiO_2 showed a significant deactivation (10% drop in the CO conversion) during the catalytic tests.

Hydrogen and carbon dioxide have generally a negative effect on the activity and they can also be the reactants for the methanation reactions. The comparison between the catalytic tests at 300 °C under reformate type gas and pure reactants mixture is reported in Fig. 6. The data in Fig. 6 are presented in terms of the carbon dioxide yield, as the CO conversion includes the reaction to methane. For a majority of the catalysts, their activity was enhanced when the reaction gas mixture was composed of 10% CO and 20% H_2O. The CO conversion of Rh/ZrO_2, Rh/CeO_2 and Ru/CeO_2 dropped strongly due to the suppression of the methanation reactions. Despite the methanation activity the carbon dioxide yield is higher over Rh/CeO_2 for the reformate mixture than for the reactants only, indicating that this sample catalyzes the WGS reaction very well. This is in line with the high carbon monoxide conversion observed over this sample (Fig. 1). Surprisingly, the activity of Au/ZrO_2 and Pd/CeO_2 catalyst was lower at 300 °C with a CO and H_2O gas mixture.

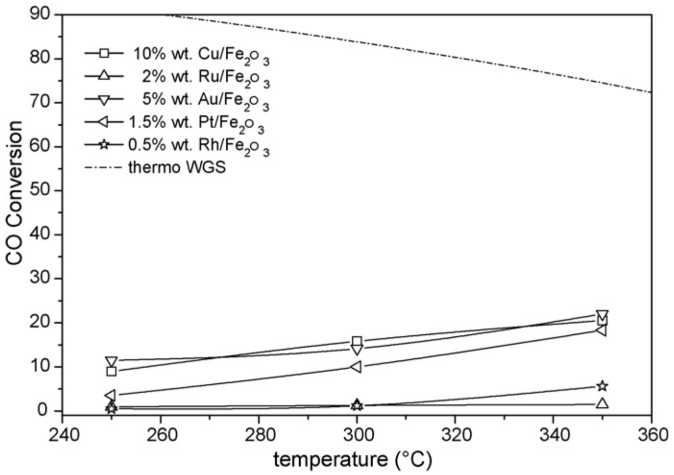

Fig. 4. CO conversion as a function of the temperature for different metals supported on Fe_2O_3, 100 mg of sample, 100 N mL/min of 10% CO, 10% CO_2, 20% H_2O, 30% H_2, 30% Ar. The CO conversion calculated according the thermodynamics take into account only the reaction $CO + H_2O = CO_2 + H_2$.

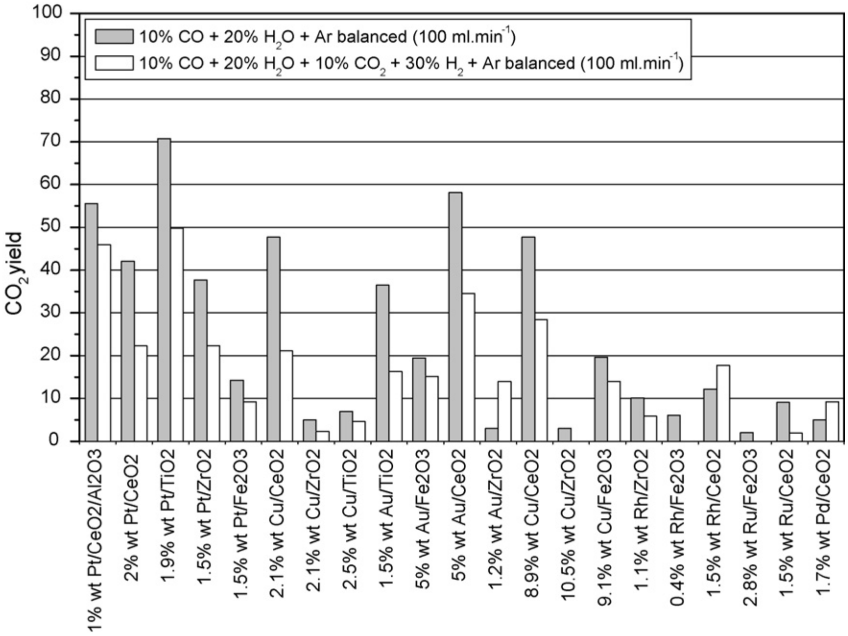

Fig. 6. CO_2 yield at 300 °C with a reformate mixture (100 N mL/min of 10% CO, 10% CO_2, 20% H_2O, 30% H_2, 30% Ar) and a pure reactant mixture (100 N mL/min of 10% CO, 20%H_2O, 70% Ar) 100 mg of sample.

Three samples (Pt/ZrO_2, Pt/CeO_2, Au/TiO_2) were tested twice but in different channels in the parallel reactor. Fig. 7 shows the good reproducibility of the results.

The activities of two Pt catalysts that were tested in the parallel reactor have been tested more extensively in a single tubular fixed bed reactor. Fig. 8 compares the results for the two samples in the two tests. The activity of the $Pt/CeO_2/Al_2O_3$ sample was significantly higher in the parallel reactor than in a single fixed bed reactor. The difference in conversion corresponds to a 30% difference in the pre-exponential factor. A better agreement was observed for the Pt/ZrO_2 catalyst, although in this case a lower activation energy (−25%) was estimated from the parallel reactor data. The discrepancy between the reactors might be due to slightly different partial pressures of water vapor as it is a process variable that is difficult to control precisely in the parallel reactor set-up [20].

As was discussed in the introduction, it is difficult to compare these results with those reported in the literature due to the very different operating conditions. In the case of platinum based catalysts, it has been reported that the order with respect to the partial pressure of CO is close to zero [9]. Thus, the reaction rate does not depend on the partial pressure of CO in the mixture. However, the order of the water partial pressure is between 0.5 and 1. Therefore, the rate will still depend on the H_2O/CO ratio. The activities of different Pt catalysts tested in this study have been calculated at 300 °C and have been

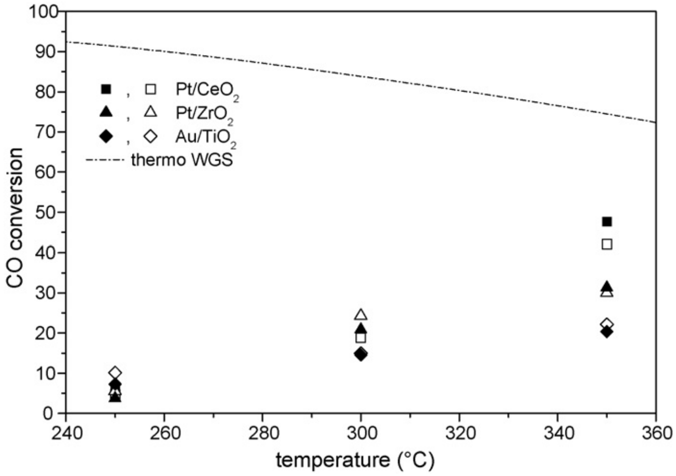

Fig. 7. Replicate tests in the parallel reactor for Pt/CeO_2, Pt/ZrO_2, Au/TiO_2, 100 mg of sample, 100 N mL/min of 10% CO, 10% CO_2, 20% H_2O, 30% H_2, 30% Ar.

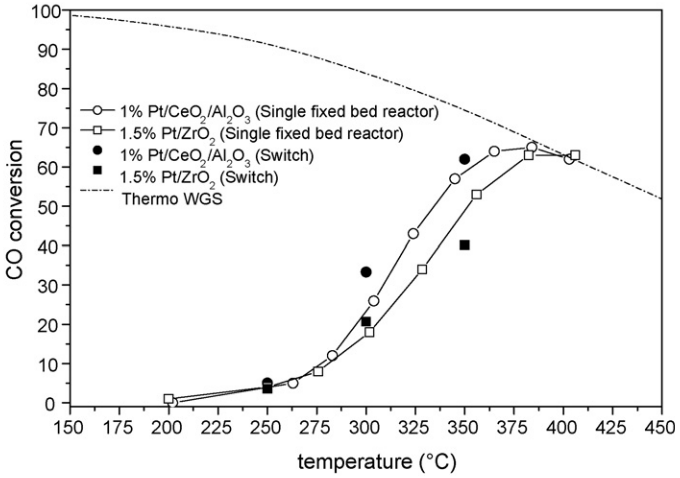

Fig. 8. CO conversion as a function of temperature with a reformate mixture (100 N mL/min of 10% CO, 10% CO_2, 20% H_2O, 30% H_2, 30% Ar) in both a parallel reactor and a single tubular reactor.

compared to the values obtained in other studies. The following equation has been applied:

$$\text{activity}\,(\text{mmol/s}\,\text{kg}_{\text{cat}}) = F_{\text{CO}} \times \frac{X_{\text{CO}}}{W_{\text{cat}}}$$

where F_{CO} (mol/s) is the molar flow rate of CO, X_{CO} is the CO conversion and W_{cat} (kg) is the weight of the catalyst. Note that this catalytic activity does not equal the actual reaction rate as for the latter differential conditions are necessary. Table 2 compares the activities for Pt based formulations for the reformate mixture with activities estimated from literature studies. The value in parentheses gives the H_2O/CO ratio. Given the variation in the H_2O/CO ratio and the fact that most studies do not take into account the inhibiting effect of the products, the catalytic activities of the different studies compare rather well with the current study. For example, Basińskaa et al. [12] reported interesting results regarding their Pt/Fe_2O_3 catalyst, obtaining 80% conversion at 350 °C. However, they were using a $W_{\text{cat}}/F_{\text{CO}}$ 1.5 times the value than the one used in this study and a more favorable H_2O/CO ratio. Thus their catalyst activity is similar to most other Pt supported formulations but at 50 °C higher. This reinforces the superior activity of Pt/TiO_2 and $Pt/CeO_2/Al_2O_3$ catalysts.

The activities of Au and Cu supported catalysts are largely reported in the literature [1–4,14,16]. However, it is difficult to compare the literature data with the data we obtained in this study, because the activity of the catalysts is particularly affected by the experimental conditions and the method of preparation [25,26]. Burch discusses in great detail the comparison of gold catalysts and he has extrapolated the rate for CO conversion to 200 °C for a large number of samples reported in the literature [26]. Only for the Au/CeO_2 and 9% Cu/ CeO_2 catalyst a comparison with the data by Fu et al. [27] and Koryabkina et al. [28] is included due to the rather close composition of the reaction mixtures. In the case of the gold catalyst, the activities are rather close taking all uncertainties into account, but in the case of the copper sample an order of magnitude larger activity is observed in this study compared to the data in [28]. Koryabkina et al. [28], in fact, show that the rate over different Cu catalysts is proportional with the Cu surface area, which can vary significantly due to the differences in the preparation methods. Unfortunately, for both studies the Cu surface areas are not available.

Table 2
Apparent activation energies and catalytic activities of the tested catalysts

Catalysts	E_a (kJ/mol)	Activity ($\text{mmol/kg}_{\text{cat}}$ s) at 300 °C	
		This study (2)[a]	
0.9% $Pt/CeO_2/Al_2O_3$	70	27	18 (2.4)[a] [9]
1.5% Pt/ZrO_2	58	20	7 (1.3)[a] [10]
2% Pt/CeO_2	65	15	27 (3.3)[a] [7]
1.9% Pt/TiO_2	23	39	32 (3.3)[a] [11]
1.5% Pt/Fe_2O_3	44	6	17[b] (2.5)[a] [12]
1.7% Pd/CeO_2	43	8	
5% Au/CeO_2	9	27	12 (2.4)[a] [27]
1.5% Au/TiO_2	29	12	
5% Au/Fe_2O_3	21	12	
1.5% Au/ZrO_2	15	12	
2.1% Cu/CeO_2	43	16	
8.9% Cu/CeO_2	49	18	2 (3.0)[a] [28]
9.1% Cu/Fe_2O_3	23	13	

[a] In parentheses: the H_2O to CO ratio.

[b] Extrapolated from the data in [12] to 300 °C using an activation energy of 44 kJ/mol.

Apparent activation energies were calculated for the most active WGS catalysts and reported in Table 2. These activation energies were calculated by integration of the following rate equation:

$$r = k^0 \exp\left(\frac{E_{\text{act}}}{RT}\right) P_{\text{CO}}^{0.5} P_{H_2O}^{0.5}$$

The reaction orders of CO and H_2O were fixed for all catalysts at 0.5. Lower reaction orders for the partial pressure of CO (typically for Pt and Pd) will give slightly lower activation energies (2–4 kJ/mol) whereas higher orders (typically for Au and Cu) will give slightly higher activation energies than reported in Table 2. The values of the activation energies for a large number of catalysts are rather low. One source of error stems from the small amount of data points as shown above (see Fig. 8). Another source of error is that the inhibiting effect of the products on the reaction rate is neglected in the above rate equation, leading to too low activation energies [29]. The Pt based catalysts show the highest values for the activation energies, Cu based catalysts intermediate values and Au low values. This trend is consistent with other studies [9,15,27,28]. These low activation energies for the gold catalysts should make those catalysts especially suited for the low temperature WGS. However, the lower partial pressures of carbon monoxide applied in the low temperature WGS will decrease the activity of the gold and copper catalysts significantly as they have orders for CO close to 1 [15,26].

4. Conclusions

Over 20 different bifunctional catalysts have been screened for their activity and selectivity for the WGS reaction between 250 and 350 °C in a commercial parallel reactor set-up. The objective of this study was to compare the large number of different formulations recently proposed in the literature under identical conditions using a representative reformate reaction mixture. Replicate tests in the parallel reactor showed good reproducibility. Comparison of the data from the parallel reactor with a single fixed bed reactor showed deviations of 20–30% in the kinetic parameters. Rh and Ru based catalysts showed significant amounts of methane formation. The platinum based formulations compare rather well with results from literature. $Pt/CeO_2/Al_2O_3$ and Pt/TiO_2 are the most active catalysts for the HT WGS while gold and copper catalysts show promising results for LT applications, but they require testing at lower CO partial pressures.

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