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Hydrogen production by glycerol steam reforming over CeZrCo fluorite type oxides

M. Araque[a,b], L.M. Martínez T[a,*], J.C. Vargas[b], A.C. Roger[a]

[a] Laboratoire des Matériaux, Surfaces et Procédés pour la Catalyse LMSPC, UMR CNRS 7515, ECPM – Université de Strasbourg, 25 rue Becquerel, 67087 Strasbourg Cedex 2, France
[b] Departamento de Ingeniería Química y Ambiental, Universidad Nacional de Colombia Ciudad Universitaria, Avenida Carrera 30 No. 45-03, Edificio 453, Bogotá D.C., Colombia

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ABSTRACT

The glycerol steam reforming reaction was studied for selective hydrogen production. The effect of the reaction temperature and of the active phase was evaluated using two catalysts $Ce_2Zr_{1.5}Co_{0.5}O_{8-\delta}$ (CZCo) and $Ce_2Zr_{1.5}Co_{0.47}Rh_{0.07}O_{8-\delta}$ (CZCoRh) mixed oxides. For both catalysts the increment of temperature favoured the glycerol conversion into gaseous products. At 650 °C, 95% of the thermodynamic H_2 production was obtained (6.1 $molH_2\ mol_{Gly.in}{}^{-1}$) using CZCoRh. For the CZCoRh diluted with SiC, the H_2 production increased to 6.7 $molH_2\ mol_{Gly.in}{}^{-1}$ and remained during 16 h. The blank results showed that 0.5 $molH_2\ mol_{Gly.in}{}^{-1}$ is formed using the SiC bed alone.

1. Introduction

Hydrogen production from biomass has become a widely investigated subject [1–3]. The most outstanding processes are gasification, pyrolysis and reforming (steam, dry or auto-thermal) [4]. The H_2 generation from an integrated process has been proposed as an alternative to make economically attractive H_2 production from biomass [5], where the main goal of the processes is the synthesis of high value products and the generation of H_2 comes from the residual fractions. Some of the residual fractions that have been evaluated are trap grease, industrial waste water and crude glycerol [5,6]. Steam reforming of glycerol is an alternative to valorize this by-product [7]. It proceeds according to the glycerol decomposition (Reaction 1) and the water-gas shift reaction (WGSR—(Reaction 2)) [8]:

$$C_3H_8O_3 \rightarrow 3CO + 4H_2 \qquad \text{(Reaction 1)}$$

$$CO + H_2O \Leftrightarrow CO_2 + H_2 \qquad \text{(Reaction 2)}$$

The global reaction of glycerol steam reforming is presented as:

$$C_3H_8O_3 + 3H_2O \rightarrow 3CO_2 + 7H_2 \quad (\Delta H^\circ{}_{298} = +346.4\,\text{kJ/mol}) \qquad \text{(Reaction 3)}$$

Thermodynamically, the effect of temperature, pressure and glycerol:water ratio has been studied for glycerol steam reforming [9–11]. It was shown that the production of H_2 is favoured at high temperatures ($T > 600$ °C), atmospheric pressure and a glycerol:water ratio of 1:9 or higher [12]. It was also predicted that the conditions that favour the H_2 production inhibit the carbon production [11].

Glycerol steam reforming has been performed using different metals (Ru, Rh, Ni, Ir, Co, Pt, Pd, and Fe) over different supports (La_2O_3, ZrO_2, CeO_2, La_2O_3, SiO_2, MgO, Al_2O_3, and CeO_2/Al_2O_3) [13,14]. The production of H_2 has been selectively obtained using Ru/Y_2O_3 at 600 °C [15], Ir/CeO_2 at 400 °C [8], Ni/Al_2O_3 or $Rh/CeO_2/Al_2O_3$ at 900 °C [12,16,17], among others. Nevertheless, the formation of carbon deposits and the aging of the catalysts still remain a problem [14].

This paper shows the study of glycerol steam reforming using $Ce_2Zr_{1.5}Co_{0.5}O_{8-\delta}$ (CZCo) and $Ce_2Zr_{1.5}Co_{0.47}Rh_{0.07}O_{8-\delta}$ (CZCoRh) mixed oxides as catalysts. The mixed oxides allow the insertion of transitions metals (Co, Ni), or/and noble metals (Rh, Ru) into the oxide structure (CeZr), increasing the interaction between the active phase and the support [18–20]. For CZCo and CZCoRh the effect of temperature and the presence of Rh in long duration tests (24 h) were studied. For long duration tests the catalysts were diluted with 50 wt% of SiC.

2. Experimental

2.1. Preparation and characterization of the mixed oxides

The CZCo and CZCoRh mixed oxides were synthesized by a pseudo sol–gel method, which is based on thermal decomposition

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* Corresponding author.
E-mail addresses: leidy@icmse.csic.es (L.M. Martínez T), annececile.roger@unistra.fr (A.C. Roger).

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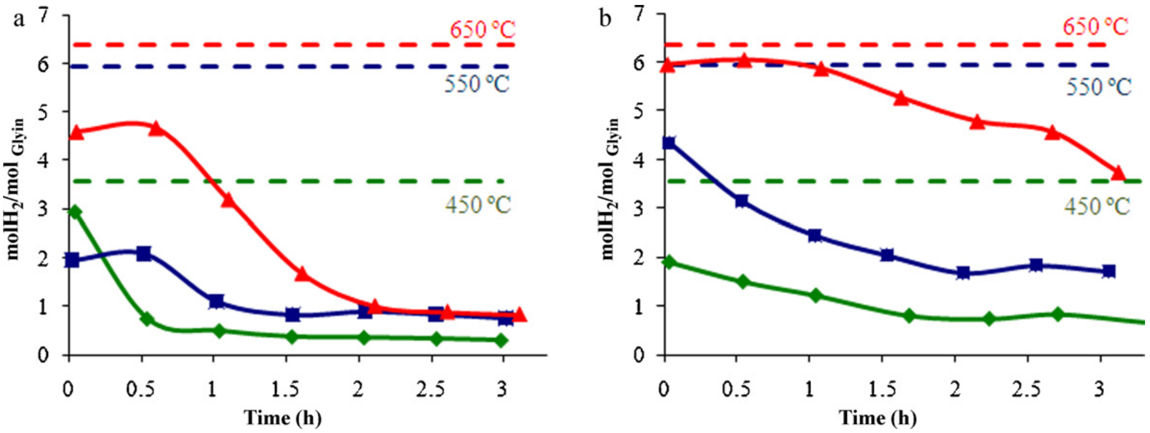

Fig. 1. Mol of H_2 produced per mol of glycerol introduced at 450 °C (♦), 550 °C (■), 650 °C (▲) for: (a) CZCo and (b) CZCoRh.

of metallic propionates [18,19,21]. The mixed oxides were calcined at 700 °C during 6 h, with a heating rate of 2 °C min^{-1}.

The characterization before test was performed by X-ray diffraction (XRD), and thermoprogrammed reduction (TPR). The experimental details have been published elsewhere [21].

After test the morphology of the catalysts was studied by SEM experiments on a JEOL JSM-6700F apparatus.

Analysis of the carbonaceous deposits formed during the glycerol reforming was carried out by thermoprogrammed oxidation (TPO) with 0.05 g of catalyst after test, in a 1% O_2 flow diluted in He (total flow of 50 mL min^{-1}). The temperature was increased at 15 °C min^{-1} from room temperature up to 900 °C. The oxidation gases were followed by mass spectrometry in a PFEIFFER vacuum equipment.

2.2. *Catalytic tests*

The glycerol steam reforming was performed in a tubular reactor (8 mm ID) at atmospheric pressure. The reactant solution (glycerol:water molar ratio 1:9) was pumped into the system using a 305 Gilson micropump and it was introduced into the reactor by a dosage system. The reactant liquid flow used was 0.0213 g min^{-1}. It was vaporized (1.13 L h^{-1} gas flow) and diluted with a N_2:Ar gas flow mixture (1:4 molar ratio) of 1.87 L h^{-1}.

The catalyst sample (0.160 g) was pre-reduced before test with 3 mL min^{-1} of pure H_2 during 12 h at 450 °C, at a heating ramp of 2 °C min^{-1}. After that, the reaction system was purged with the inert gas flow (N_2:Ar) and it was heated at 2 °C min^{-1} up to the desired reaction temperature (450 °C, 550 °C, and 650 °C). A thermal blank was carried out during 6 h at 650 °C on 0.160 g of SiC.

Long duration tests were performed for both catalysts at 650 °C during 24 h. The catalyst sample (0.160 g) was diluted with 0.160 g of SiC and it was pre-reduced at 450 °C.

The reaction products were divided into gaseous and liquid products. The gaseous products (H_2, CO, CO_2, CH_4, and C_2H_4) were analysed by on line gas chromatography using a Carbosieve II column with N_2 as internal standard. The liquid products were recovered every determined time (3 h, 8 h, and 24 h) using a system of two traps, the first one at room temperature and the second one at 0 °C. The analysis of the liquid phase products was performed by gas chromatography using a ZB-Wax Plus (Zebron) column with n-butanol as internal standard. The products analysed in liquid phase were acetone, acetaldehyde, acrolein, methanol, ethanol, hydroxyacetone, acetic acid, propionic acid, propyleneglycol, ethyleneglycol and glycerol.

2.3. *Analytical methodology*

The catalyst activity was evaluated by three different conversions: the global conversion of glycerol, obtained from the glycerol recovered in the liquid phase (X_G – Eq. (1)); the conversion to gaseous products (X_{GP} – Eq. (2)); and the conversion to liquid products (X_{LP} – Eq. (3)).

$$X_G\,(\%) = \left[1 - \frac{g_{\mathrm{Gly.out}}}{g_{\mathrm{Gly.in}}}\right] \times 100 \qquad (1)$$

$$X_{GP}\,(\%) = \frac{F_{CO_2} + F_{CO} + F_{CH_4} + 2F_{C_2H_4}}{3F_{\mathrm{Gly.in}}} \times 100 \qquad (2)$$

$$X_{LP} = \frac{\sum F_X}{3F_{\mathrm{Gly.in}}} \times 100 \qquad (3)$$

where F_X is the molar flow of x; $g_{\mathrm{Gly.out}}$ is the mass of exhaust glycerol for a given period of time, and $g_{\mathrm{Gly.in}}$ is the mass of glycerol introduced to the system for the same period of time.

The production of H_2 obtained experimentally ($H_2^{(a)}$ – $molH_2\ mol_{\mathrm{Gly.in}}{}^{-1}$) was compared to the production obtained according to the thermodynamic calculus at the same reaction conditions ($H_2^{(b)}$ – $molH_2\ mol_{\mathrm{Gly.in}}{}^{-1}$). The thermodynamic model used for the simulation was the UNIQUAC.

3. Results and discussion

3.1. *Effect of the reaction temperature*

Fig. 1 shows the profiles of H_2 production for CZCo (Fig. 1a) and for CZCoRh (Fig. 1b). The results are compared to the thermodynamic production (dashed lines).

For both catalysts the increment in temperature favours the H_2 production. The moles of H_2 obtained increase and the time at higher H_2 production is prolonged. For CZCo, after 0.5 h the production of H_2 is 0.75 $molH_2\ mol_{\mathrm{Gly.in}}{}^{-1}$ at 450 °C, 2.1 $molH_2\ mol_{\mathrm{Gly.in}}{}^{-1}$ at 550 °C, and 4.7 $molH_2\ mol_{\mathrm{Gly.in}}{}^{-1}$ at 650 °C. The thermodynamic value was not reached at any of the temperatures. For CZCoRh, the production corresponds to 1.5 $molH_2\ mol_{\mathrm{Gly.in}}{}^{-1}$, 3.1 $molH_2\ mol_{\mathrm{Gly.in}}{}^{-1}$ and 6.1 $molH_2\ mol_{\mathrm{Gly.in}}{}^{-1}$, respectively. At 650 °C the moles of H_2 formed correspond to 95% of the thermodynamic value. At 550 °C and 450 °C, these values correspond only to 73% and 53%, respectively.

It must also be noticed that for the two catalysts the production of H_2 drops in time at the three temperatures. For CZCo (Fig. 1a), a minimum production is reached after 1 h at 450 °C and at 550 °C;

and after 2 h at 650 °C. For the catalyst with Rh (Fig. 1b), the minimal production is reached after 1.5 h at 450 °C, 2 h at 550 °C, and more than 3 h at 650 °C. In all cases, the decrease in the H_2 production is accompanied by a decrease of conversion to gas products (results not shown), indicating a progressive deactivation of the catalyst.

The introduction of Rh clearly promotes both the activity and the stability of the catalyst compared to the catalyst with only Co. However, the stability of CZCoRh in glycerol steam reforming decreases faster than in ethanol steam reforming [21], where total conversion of ethanol was obtained during 150 h at 550 °C. The stability of the catalyst is then affected by the increase in the number of –OH functions of the reactant alcohol.

3.2. Effect of the active phase

The effect of Rh introduction was evaluated by comparing the CZCo and CZCoRh catalytic performance at 650 °C during 24 h. The average conversions and moles of H_2 produced per mol of glycerol introduced are presented in Table 1. From the global results (0–24 h), it is clearly evidenced that Rh favours the production of gaseous products (X_{GP}), at the expense of liquids products (X_{LP}). Also the production of H_2 is favoured, increasing from 1.6 $\mathrm{molH_2\,mol_{Gly.in}}^{-1}$ for CZCo up to 5.8 $\mathrm{molH_2\,mol_{Gly.in}}^{-1}$ for CZCoRh.

For CZCo, the global conversion of glycerol is total during the first 3 h ($X_G = 100\%$). X_{LP} is 1%, due to the formation of some traces of acetaldehyde, acetone and acrolein. During this period, X_{GP} is 89% and the production of H_2 is 5.7 $\mathrm{molH_2\,mol_{Gly.in}}^{-1}$. Between 3 h and 24 h, X_G remains total (only some traces of glycerol are detected), but X_{GP} highly diminishes down to 33%, and X_{LP} increases up to 11%.

Table 1
Average conversions and moles of H_2 produced for 24 h of glycerol steam reforming on CZCo and CZCoRh at 650 °C.

	CZCo			CZCoRh			
	0–24 h	0–3 h	3–24 h	0–24 h	0–3 h	3–8 h	3–24 h
X_G	99	100	99	100	100	100	100
X_{GP}	42	89	33	92	97	98	89
X_{LP}	10	1	11	1	0	0	1
H_2[a]	1.6	5.7	1.0	5.8	6.7	6.7	5.3
H_2[b]	6.5	6.5	6.5	6.5	6.5	6.5	6.5

[a] Experimental $\mathrm{molH_2\,mol_{Gly.in}}^{-1}$.
[b] Thermodynamic $\mathrm{molH_2\,mol_{Gly.in}}^{-1}$.

For the CZCoRh catalyst, glycerol is not detected for any of the liquid fractions recovered ($X_G = 100\%$). For the first 8 h, the conversion to gaseous products is around 100% and the production of H_2 remains constant at the thermodynamic value (6.5 $\mathrm{molH_2\,mol_{Gly.in}}^{-1}$). For the fraction from 8 h to 24 h, traces of liquid products are detected ($X_{LP} = 1\%$), mainly hydroxyacetone and acrolein.

During the period of deactivation part of the incoming glycerol is transformed into carbonaceous deposits, which is observable in the reactor after the two tests. The presence of carbon filaments was observed for both catalysts by SEM. The proportion of filaments was higher for the CZCo (Fig. 3a) in comparison to the CZCoRh catalyst (Fig. 3b).

TPO experiments were also performed for the catalysts after test. The CO, CO_2, CH_4 and H_2O signals were followed, but only the CO_2 profiles (mass 44) are reported in Fig. 4. A single peak of high intensity was observed at 680 °C for the catalyst with only Co, while two peaks were noticed, the first one at 250 °C and the second one at

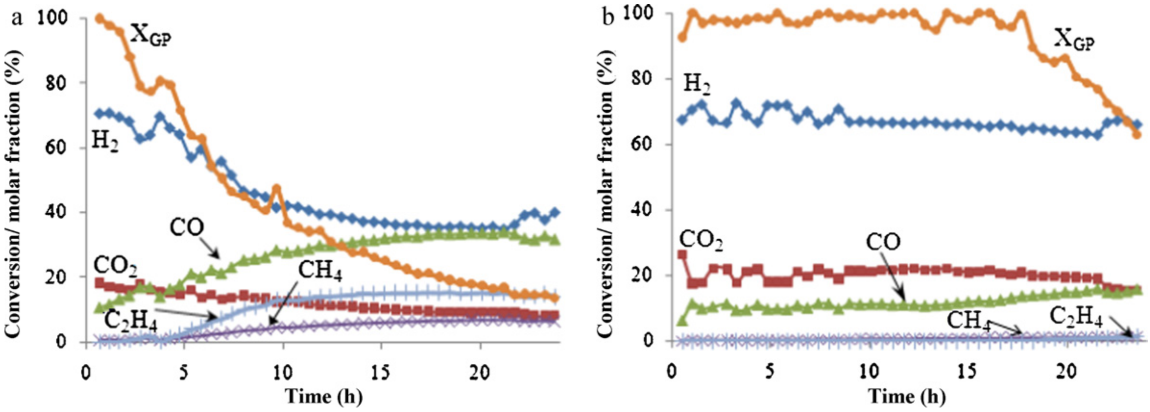

Fig. 2. Profiles of X_{GP} and gas molar fraction for: (a) CZCo, and (b) CZCoRh diluted with 50 wt% of SiC, at 650 °C.

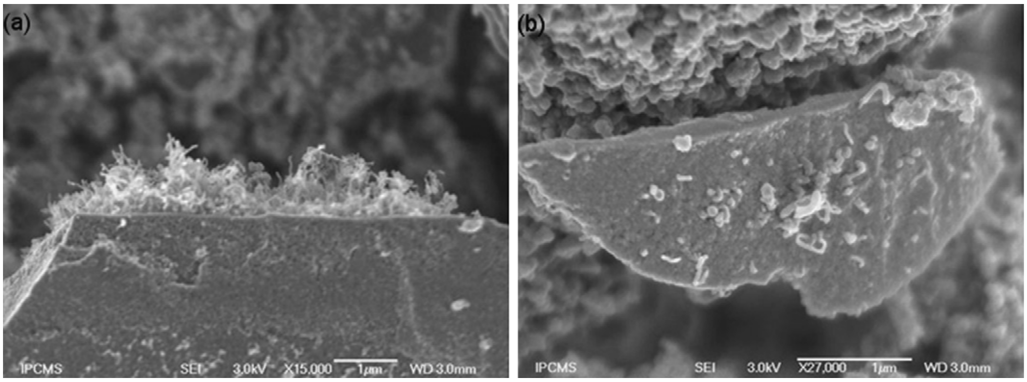

Fig. 3. SEM micrographs of the catalysts after test: (a) CZCo, (b) CZCoRh.

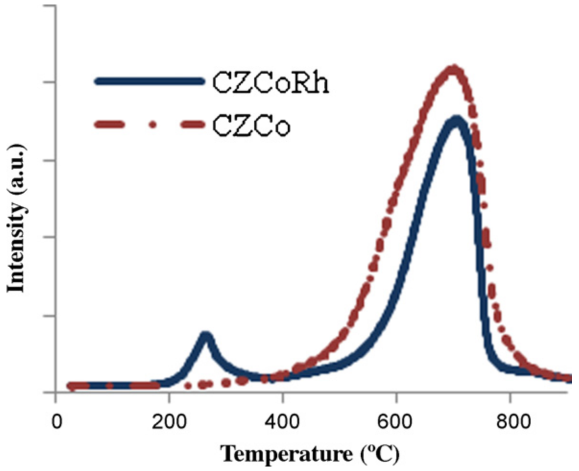

Fig. 4. CO_2 profiles from TPO experiments for the CZCo and CZCoRh catalysts after catalytic test.

Table 2
Distribution of products in liquid phase (molar fraction) for 24 h of glycerol steam reforming on CZCo and CZCoRh at 650 °C.

	CZCo	CZCoRh
Acetaldehyde	18	25
Acetone	2	6
Acrolein	11	9
Methanol	12	9
Ethanol	1	2
Hydroxyacetone	46	41
Acetic acid	6	5
Propionic acid	2	3
Ethylene glycol	1	1

690 °C, for the catalyst with Rh. In general, the peak at low temperature is ascribed to CO_2 coming from the oxidation of intermediary species and superficial carbon; and the peak at high temperature is associated to the oxidation of more structured carbon (filaments). However, in TPO the variation in the other masses (mass 29 for CO, mass 18 for H_2O, mass 32 for O_2) follow the same tendency for both peaks (low and high temperature), indicating the oxidation of similar carbon deposits. This behavior could be explained by recent works on Pt/CeZr catalysts, where it was noticed that the different peaks of CO_2 in TPO results are related to different locations of the carbon rather than to different forms of carbon deposits (filaments, superficial carbon, and adsorbed species) [22,23]. The peak at low temperature has been ascribed to carbon formations or adsorbed species close to the metallic particles, while the peak at high temperature has been ascribed to carbon or adsorbed species deposited over the support (CeZr). Then, the peak at high temperature, present for both catalysts, would represent carbon formations over the CeZr oxide, and the peak at low temperature, only present for the catalyst with Rh, will be related to the presence of this metal in the catalyst.

The quantification of the carbon formed during the glycerol reforming was calculated from the TPO results and it is presented in Table 3. The formation of carbon deposits highly diminished with the presence of Rh, from 4.2 mmolC $g_{cat}{}^{-1}$ for CZCo to 2.8 mmolC $g_{cat}{}^{-1}$ for CZCoRh. The selectivity to carbonaceous deposits was 0.55 mmolC $mol_{carbon\ converted}{}^{-1}$ for CZCo and 0.36 mmolC $mol_{carbon\ converted}{}^{-1}$ for CZCoRh. This corroborates that the presence of Rh decreases the carbon deposition, by increasing the ability of breaking the C–C bond [24].

The evolution of X_{GP} and the distribution of gas products for both catalysts are shown in Fig. 2. For CZCo (Fig. 2a), X_{GP} diminishes after only 1 h of reaction. The distribution of gaseous products at 100% of X_{GP} is 70% H_2, 18% CO_2, 11% CO and 0.6% CH_4. After this period, the production of H_2 and CO_2 decreases and the production of CO and CH_4 increases. The production of C_2H_4 is detected after 2 h. After 20 h the distribution of products is: 35% H_2, 9% CO_2, 34% CO, 7% CH_4 and 15% C_2H_4.

For the catalyst with Rh (Fig. 2b), X_{GP} remains at 100% during 16 h. The distribution of gaseous products changes only slightly during this period, being around 67% of H_2, 21.5% of CO_2, 11% of CO, and 0.5% of CH_4. The presence of C_2H_4 is noticed after 18 h. After the 24 h of reaction, X_{GP} falls down to 59%, and the distribution is the following: 65% H_2, 16% CO_2, 16% CO, 1.4% CH_4 and 1.7% C_2H_4.

For the catalysts diluted with SiC (long duration tests), the production of H_2 is higher than for the catalysts with no dilution (effect of the temperature (Fig. 1 (▲) 650 °C)). The dilution enhances the activity and as a consequence the stable production of H_2. According to the results of the thermal blank, 0.5 molH_2 $mol_{Gly.in}{}^{-1}$ is produced on 0.160 g bed of SiC. At this temperature, the gas product distribution is 49% of C_2H_4, 36% of CO, 10% of H_2, 5% of CH_4 and traces of CO_2 (<1%). The hydrogen production is not negligible compared to the production of H_2 obtained on the catalysts at 650 °C (6.1 molH_2 $mol_{Gly.in}{}^{-1}$). The formation of high amounts of carbon deposits was observed in the reactor and in the SiC bed after test. The effect of the dilution with SiC is currently being performed.

Table 2 shows the distribution of products in liquid phase over the 24 h of reaction. For both catalysts, the main products are hydroxyacetone and acetaldehyde. Methanol and acrolein are also detected, together with traces of acetone, ethanol, acetic acid, propionic acid, and ethylenglycol. Hydroxyacetone (Reaction 3′), acrolein (Reaction 4) and acetaldehyde (Reaction 5) are products associated with the glycerol dehydration [25]. In the case of acetaldehyde the rupture of a C–C bond is also involved.

$$C_3H_8O_3 \rightarrow CH_3C(O)CH_2OH + H_2O \qquad \text{(Reaction 3′)}$$

$$C_3H_8O_3 \rightarrow CH_2{=}\ CHCHO + 2H_2O \qquad \text{(Reaction 4)}$$

$$C_3H_8O_3 \rightarrow HCHO + CH_3CHO + H_2O \qquad \text{(Reaction 5)}$$

The proportion of hydroxyacetone is slightly higher for CZCo than for CZCoRh. On the other hand, the proportion of acetaldehyde is lower for CZCo than for the catalyst with Rh, showing the higher capacity of Rh to cleave the C–C bond.

For both catalysts, the deactivation pattern is the same. The production of H_2 and CO_2 decreases and the production of CO, CH_4 and C_2H_4 increases. The formation of CH_4 proceeds probably by methanation (Reactions 6 and 7), on the contrary of the ethanol steam reforming, where it is formed directly from ethanol decomposition [26]. The formation of C_2H_4 comes from the hydrogenation/dehydration of acetaldehyde (Reaction 8) [13], and it

Table 3
Carbonaceous deposits for the CeZrCoRh catalyst after 50 h and 350 h of reaction.

Catalyst	Amount of carbonaceous deposits (mmolC $g_{cat}{}^{-1}$)	Production of carbonaceous deposits (mmolC $g_{cat}{}^{-1}$ h^{-1})	Selectivity to carbonaceous deposits (mmolC $mol_{carbon\ converted}{}^{-1}$)
CZCo	4.2	0.17	0.55
CZCoRh	2.8	0.12	0.36

indicates that the capacity for cleaving the C–C bond is also affected.

$$CO + 3H_2 \Leftrightarrow CH_4 + H_2O \quad \text{(Reaction 6)}$$

$$CO_2 + 4H_2 \Leftrightarrow CH_4 + 2H_2O \quad \text{(Reaction 7)}$$

$$CH_3CHO + H_2 \rightarrow C_2H_4 + 2H_2O \quad \text{(Reaction 8)}$$

The increase of the CO fraction, along with the decrease of the CO_2 fraction, reveals that the capacity of the catalyst to oxygenate the surface carbon is progressively lost. Nevertheless, the increase in the CO fraction can be related to the formaldehyde decomposition (Reaction 9) or the reverse WGSR (Reaction 2).

$$HCHO \rightarrow CO + H_2 \quad \text{(Reaction 9)}$$

From these results we propose that for CZCoRh, the Rh capacity for breaking the C–C bond helps the catalyst to further convert the glycerol. However, after 16 h, the deactivation signs are also observable.

4. Conclusions

Glycerol steam reforming was performed using CZCo and CZCoRh mixed oxides as catalysts. The increase of the temperature and the introduction of Rh favoured the activity and stability of the catalysts. At 650 °C the CZCoRh showed the best performance towards H_2 production reaching a 95% of the predicted thermodynamic value (6.5 $\text{molH}_2\ \text{mol}_{\text{Gly.in}}{}^{-1}$). For the long duration tests, the introduction of Rh favoured the production into gaseous products at the expense of the liquid products (<1%). For this catalyst an average production of H_2 of 6.7 $\text{molH}_2\ \text{mol}_{\text{Gly.in}}{}^{-1}$ was obtained during over 16 h, while CZCo catalyst was stable only during 1 h. When the deactivation occurs the formation of C_2H_4 is observed, indicating the loss of the C–C bond breaking capacity.

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