

Influence of the O₂/CO ratio and the presence of H₂O and CO₂ in the feed-stream during the preferential oxidation of CO (PROX) over a CuO_x/CeO₂-coated microchannel reactor

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Abstract

The catalytic performance of a CuO_x/CeO₂ powder catalyst and that of a microchannel reactor or microreactor (MR) coated with the same solid were determined and compared. The catalytic activity measurements were carried out with varying O₂/CO molar ratios in the feed-stream. In addition, the influence of the presence of CO₂ and H₂O in the reaction mixture was studied. Some discrepancies were observed between the performances of the powder catalyst and the MR depending on the O₂/CO ratio. The

MR presented a very good performance with a superior selectivity for CO conversion. This behaviour was due to a more efficient heat removal in the case of the MR that inhibited the H₂ oxidation reaction and the r-WGS. The isothermicity of the microreactor during the process was demonstrated through the monitoring of the MR inlet and outlet temperatures.

Concerning the presence of CO₂ or H₂O in the feed-stream, both compounds gave rise to a decrease of the CO conversion. The negative effect on the catalytic performance was more marked when both compounds were fed together, although the principal inhibitor effect was associated to the CO₂. This seems to be related with the formation of stable carbonates at the catalyst surface.

Keywords

Microchannel reactor; Hydrogen purification; PROX, CO oxidation; Cu-modified ceria

1. Introduction

Global warming caused by the emission of greenhouse gases and the need of alternative energy sources for sustaining the present energy demand, are problems showing the need to rethink our way of life [1, 2]. In this sense, the using of fuels other than the fossil ones is an interesting alternative. For example using renewable H₂ for feeding electric and electronic devices that employ fuel cells such as the proton exchange membrane (PEMFC) type, is a promising option from the environmental point of view [3, 4]. However, the application of PEMFCs requires the use of highly pure H₂ with very low CO contents (<20 ppm) [5]. When H₂ is produced through the reforming of hydrocarbons and alcohols, the preferential oxidation of CO (PROX) is one of the possible processes that can be applied for the final cleaning after preliminary

purification in the high and low temperature water-gas shift (WGS) [6-8]. As for the PROX reaction, several catalysts have been proposed and the solids based on $\text{CuO}_x/\text{CeO}_2$ mixture are among the most studied materials due to their excellent performance for the CO conversion [9, 10]. However a crucial issue for the PROX reaction is broadening the operating temperature window of maximum CO conversion with high selectivity, considering the presence of other reactions that can simultaneously occur (H_2 oxidation and the reverse-water-gas shift (r-WGS)) [11].

On the other hand, from a technological point of view, when the PEMFCs are employed for portable or automotive applications, the use of conventional reactors for producing and cleaning the required H_2 represents several drawbacks such as pressure drop along the catalyst bed, temperature gradients, hot spot formation due to the high exothermicity of the CO and H_2 oxidation reactions and size weight issues [12, 13]. In this regard, microreactors are promising devices that have the advantages of fast response time, easy integration, and small footprint, which are ideal for portable and on-board power systems [14]. Additionally, the mass and heat transport rates are greatly increased in these devices [12, 13, 15, 16], being crucial for the PROX reaction, no matter the catalyst employed, because it requires a careful temperature control due to the exothermicity of the main reactions involved.

In this context, the principal aim of this work is to evaluate the performance of a $\text{CuO}_x/\text{CeO}_2$ coated microreactor for the CO-PROX reaction and comparing it with that of the $\text{CuO}_x/\text{CeO}_2$ catalyst in powder form employing different feed-stream compositions, specifically modifying the O_2/CO molar ratio. The effect of H_2O and CO_2 is also studied in order to establish the catalytic activity of the microreactor under realistic conditions, that is, using PROX feed-stream compositions that simulate the composition of a reformat off-gas after exiting the WGS units.

2. Materials and methods

The synthesis of the $\text{CuO}_x/\text{CeO}_2$ powder catalyst was carried out by coprecipitation employing suitable amounts of $\text{Cu}(\text{NO}_3)_2$ and $\text{Ce}(\text{NO}_3)_3$ in order to achieve a 9:1 $\text{Ce}(\text{OH})_3:\text{Cu}(\text{OH})_2$ weight ratio after the addition of NaOH (2M). The obtained solid washed with distilled water and dried at $60\text{ }^\circ\text{C}$ overnight, and finally calcined for 2 h at $300\text{ }^\circ\text{C}$ [17, 18].

The details of the MR manufacturing (micromachining and joining of the steel plates), and assembly have been recently reported [17]. Washcoating was selected for coating the microchannels with the $\text{CuO}_x/\text{CeO}_2$ catalyst using a slurry with the following composition: 76 wt.% powder $\text{CuO}_x/\text{CeO}_2$ catalyst, 7 wt.% polyvinyl alcohol (PVOH) and 17 wt.% colloidal alumina. The pH of the suspension was adjusted to 4 with diluted HNO_3 [17, 19, 20].

Once the microchannels were immersed in the slurry, the elimination of excess was done by air blowing (2 L/min). After each coating process, the microchannel block was dried at $120\text{ }^\circ\text{C}$ for 30 min and finally calcined at $300\text{ }^\circ\text{C}$ for 3 h ($1\text{ }^\circ\text{C}/\text{min}$). This method resulted in a microchannel block loading of $5.46\text{ mg}/\text{cm}^2$ after eight washcoating processes, with a total load of 300 mg of catalyst [17].

Concerning to the catalytic activity measurements, prior to every test the catalyst (both in the powder form and in the microreactor) was activated under 30 mL/min total flow of 21% O_2 in N_2 at $300\text{ }^\circ\text{C}$ for 2 h.

As for the powder catalyst, the PROX reaction was carried out at atmospheric pressure on a cylindrical stainless steel reactor (9 mm inner diameter), with 100 mL/min total flow of the mixture of reaction. The catalyst powder (100 mg, particle size $\phi = 100\text{-}200\text{ }\mu\text{m}$) was diluted with crushed inert glass with the same particle size until a reactor bed

height of 5 mm. The temperature of the reaction was recorded by a thermocouple K-type whose top was in contact with the top of the fixed bed.

In the case of the microreactor, the temperature was continuously monitored by 4 K-type thermocouples. Two of them were placed in contact with the metallic block at the inlet and outlet positions of some central microchannels. The other two sensors recorded the temperature at lateral positions in the walls of the microblock [18].

For the two evaluated catalytic systems, the products and reactants were analyzed by online gas chromatography (Agilent® 7890 equipped with a Porapak® Q, two Molecular Sieve 5A, and two Hayesep® Q columns) and then quantified using a TCD detector. The CO conversion and the selectivity to CO conversion were calculated according to Equations 1 and 2, respectively, where F_{in} and F_{out} refer to molar flow rates at the reactor entry and outlet, respectively [21].

$$\text{CO conversion (\%)} = \frac{(F_{CO,in} - F_{CO,out}) \times 100}{F_{CO,in}} \quad (\text{Eq.1})$$

$$\text{Selectivity to CO conversion (\%)} = \frac{(F_{CO,in} - F_{CO,out}) \times 100}{2(F_{O_2,in} - F_{O_2,out})} \quad (\text{Eq.2})$$

In order to compare the catalytic activity of the $\text{CuO}_x/\text{CeO}_2$ powder catalyst (100 mg) and that of the microreactor, the variation of the O_2/CO molar ratio and the presence of H_2O and CO_2 feed-stream were studied. To this end, the several feed-stream compositions included in Table 1 were employed (N_2 was used as balance). The oxygen excess in the reaction mixture was defined as a factor (λ), according to Eq. 3, taking into account that the stoichiometric amounts of CO and O_2 present a $\lambda = 1$. This λ factor has been previously applied by other authors in the study of different feed-stream composition for PROX reaction [22].

$$\lambda = \frac{2 \times F_{O_2, in}}{F_{CO, in}} \quad (\text{Eq.3})$$

Total flow rate was 100 mL·min⁻¹ for the powder catalyst and 300 mL·min⁻¹ for the microreactor in order to achieve the same total flow (L·h⁻¹) to weight of catalyst (g) ratio in both cases (60 L·h⁻¹·g⁻¹).

3. Results and discussion

3.1. Influence of the O₂/CO molar ratio in the feed-stream

A comparison between the catalytic behaviour of the microchannel reactor and that of the CuO_x/CeO₂ powder catalyst as a function of the λ factor was carried out employing the feed-stream compositions 1-4 (Table 1). The results of CO conversion and selectivity to CO oxidation are presented in Figs. 1 and 2, respectively.

Both systems show similar general trends. CO conversion increases with temperature until reaching a plateau at 160 °C irrespective of the O₂/CO ratio employed.

The powder catalyst shows a slightly superior catalytic activity than the microreactor at temperatures below 160 °C at λ factors (1.5) slightly above the stoichiometric one (1). However, the microreactor gives higher CO conversions when the proportion of O₂ increases up to $\lambda \geq 4$. For that reason the results are presented in two figures (Figure 1a and 1b), because the higher excess of O₂ seems to improve the performance of the microreactor.

Considering that not significant changes in the chemical nature of the catalyst were noticeable after the washcoating, as demonstrated by the characterization of the microreactor presented in [17], the observed superior catalytic activity of the microreactor under O₂ excess conditions, could be related with changes of the mass and

heat transfer rates when using the microreactor compared with the packed bed of powder catalyst. It has to be pointed out that the exothermicity of the CO and H₂ oxidations does not depend on the extension of these reactions. Thus, the amount of heat released is a function only of the catalytic performances of the catalyst and not of their nature.

On the other hand, the microreactor allows a more efficient contact between the catalyst surface and the reaction mixture. This favours products release, which is continuously shifting the equilibrium to the CO₂ formation. It means that the mass transport phenomena can be enhanced for the microreactor with respect to the fixed bed reactor. However, this cannot be directly established with an *in-situ* characterization technique because the access to the microchannels requires the destruction of the block, which implies the modification of the catalyst. For that reason the mass transport phenomena are not deeply described in the present work. However complementary works are being carried out with the aim of establishing a correlation between the amount of loaded catalyst and the catalytic activity of the microreactor. On the other hand, an *in-situ* DRIFT study under reaction PROX conditions has been carried out over some plates of the same material than that of the microreactor (ferritic stainless steel-Fecralloy[®]) and coated with the CuO_x/CeO₂ catalyst. The obtained results are currently under analysis and will be published in the near future.

Concerning the selectivity for the oxidation of CO, it decreases with the temperature (see Figure 2), which demonstrates that the H₂ oxidation is improved at higher temperatures. These results agree with previous studies where the behaviour of the CuO_x/CeO₂ catalysts in this reaction has been investigated [7, 8]. In addition, the activation energy values obtained in the kinetic study by Arzamendi et al. [6] of the CO-PROX reaction over the CuO_x/CeO₂ catalyst loaded in the catalytic-wall microreactor of

the present study where $110 \text{ kJ}\cdot\text{mol}^{-1}$ for the H_2 oxidation reaction and $36.9 \text{ kJ}\cdot\text{mol}^{-1}$ for the oxidation of CO. This explains the fact that as the temperature increases, the oxidation of H_2 is much more favoured than that of CO thus leading to a selectivity decrease.

On the other hand, although more points would be required for the generation of more complete trends, the MR provides higher selectivities than the powder catalyst whatever the O_2/CO ratio (see Figure 2). This evidences that the MR is more efficient for the PROX reaction because similar CO conversion levels were obtained with less H_2 consumption, especially at low temperatures and high O_2/CO molar ratios. The possibility of getting a more efficient heat transport during the PROX reaction can be considered as a possible cause contributing to achieve a higher selectivity in the case of the MR.

The positive effects of using the catalytic microreactor are more evident if the selectivity is represented as a function of the CO conversion (see Fig. 3).

In the case of the powder catalyst, it can be observed that the excess of O_2 decreases both the maximum CO conversion and the selectivity for CO oxidation. However the MR keeps the maximum selectivity levels even at high oxygen excess in the feed-stream, and CO conversion as high as 98%. A further increase of the CO conversion leads to a marked decrease of the selectivity; these values correspond to reaction temperatures near $200 \text{ }^\circ\text{C}$ that strongly enhance the H_2 oxidation. In general, the MR allows working with a wider operating temperatures window for acceptable CO conversion and selectivity.

A similar behaviour was observed by Ouyang et al. [23] on a silicon microchannel reactor fabricated by means of a micromachining process [24] and employing a $\text{Pt}/\text{Al}_2\text{O}_3$ catalyst synthesized by a sol-gel method. In this work, it was demonstrated

through simulations generated from a kinetic model, that for mini-packed reactors some temperature gradients may be noticeable during the CO-PROX reaction. These gradients favour the reverse water-gas-shift (r-WGS) reaction, thus narrowing the range of temperatures where a high CO conversion is achieved with high selectivities. But in the case of the silicon microchannel reactors, more efficient heat removal was appreciated, which prevents the r-WGS reaction. This means that, as in our case, the generation of a catalytic thin layer covering the walls of the microchannels allows an efficient heat release during the reaction and this would be the key for achieving not only a high CO conversion but also a high selectivity.

The exothermicity of the CO and H₂ oxidation reactions (-283 and -242 kJ·mol⁻¹, respectively) determines that the thermal management is a key aspect of the design of the PROX reactors as confirmed by studies such as the one reported by Roberts et al. [25], in which the r-WGS reaction occurrence was analysed in an adiabatic monolith reactor during the CO-PROX. In this work it was observed that the complete conversion of O₂ generates an important increase of the temperature until 300 °C for an inlet temperature of 170 °C. A similar behaviour could be considered for the powder catalysts that present a poor thermal conductivity. However, in the microreactor, the small thickness of the catalytic layer allows reducing the distance for the radial heat conduction [23]. In this regard, the selection of the material for manufacturing the microchannel reactor is also crucial in order to ensure a rapid heat transfer during the reaction. In our case the ferritic stainless FeCrAlloy[®] results adequate for this purpose.

In a recent report [6] we developed a computational fluid dynamics (CFD) model for the same MR considered in this study that was successfully validated with experimental data in [18]. With this model the temperature profiles of the fluidic and solids domains were obtained considering the possibility of cooling the reactor with air passing through

a portion of the channels. A very uniform temperature profile is established along the solid domain, without big differences between the inlet and outlet temperatures.

The isothermicity of the MR during the PROX reaction can be confirmed experimentally through the monitoring of the inlet and outlet temperatures with the two thermocouples that are placed at these microreactor positions. As representative examples, the inlet and outlet temperatures recorded during a PROX reaction at 160 °C (as the set temperature) using two different λ factors are presented in Fig. 4.

The data recording started with the feed-stream passing through the reactor bypass. The reaction started after switching a valve that allowed the access of the feed-stream to the microchannels is indicated by the dashed lines of Fig. 4. The initial difference between the inlet and outlet temperatures is less than 1 °C and it is attributed to the precision of the sensors. The reaction increases both temperatures but the difference respect to the set value (160 °C) is not superior to 2 or 3 °C and the system tends to be quickly stabilized at the set temperature value. The heat is rapidly released which allows achieving a strict control of the reaction temperature.

Concerning the influence of the presence of CO₂ in the feed-stream, we have previously discussed results for the same MR that is being studied in this work. Using 10 vol.% CO₂ it was pointed out that this compound was the principal inhibitor of the CO-PROX reaction. In the present study, different proportions of CO₂ have been considered (compositions 3, 5-7 – see Table 1) and the results are shown in Fig. 5. In this case the catalytic activity is presented not as the CO conversion but as the CO content (ppmv) at the MR outlet.

These results demonstrate that there is a temperature or even a range of temperatures (160-200 °C) where the CO content presents a minimum value for all the studied feed-stream compositions. However, when CO₂ is present in the feed-stream, the CO content

at temperatures below 200 °C increases and the operating temperature window for acceptable CO conversion becomes narrow. There are not big differences irrespective of the CO₂ proportion, especially above 200 °C. However, this behaviour is modified after the introduction of H₂O in the feed-stream (compositions 8-10 in Table 1). The results are presented in Fig. 6.

As in the previous case, the CO content at the MR outlet increases with the amount of CO₂. However, the emitted CO concentration is higher in presence of H₂O, suggesting that when both CO₂ and H₂O are fed there is an increased inhibition of the CO conversion.

Recently, we found that the presence of 10 vol.% H₂O protects the CuO_x/CeO₂ coated MR of the inhibition caused otherwise by the presence of 10 vol.% CO₂ in the feed-stream [18]. However, these experiments were carried out employing a higher oxygen excess with respect to the CO content ($O_2/CO = 2$), than that used in the catalytic tests presented here ($O_2/CO = 1$, see Table 1). This means that the O_2/CO ratio in the feed-stream also influences the loss of activity of the catalyst by the adsorption of H₂O and CO₂, which could compete with the CO for being adsorbed on the active sites. The negative effect of H₂O and CO₂ has been discussed by other authors such as Lee et al. [26] in their kinetic study with a CuO_x/CeO₂ powder catalyst of the CO-PROX reaction. In this report it was demonstrated that the rate of the CO and H₂ oxidation reactions are dependent of the partial pressure of CO₂ and H₂O resulting in negative reaction orders with respect to the partial pressures of these compounds.

On the other hand, considering the highly reducing character of the PROX environment, the presence of metallic copper and Ce³⁺ species is expected according to the reducibility studies carried out with the powder catalysts. The reduced cerium species

may give rise to the formation of stable carbonates and this can be the reason of the strong inhibition of the CO conversion caused by CO₂.

According to these results, a more efficient desorption of CO₂ from catalyst would improve the catalytic performance prolonging the life time of the microreactor. It is possible that the enhancement of the mass transport rates during the reaction owing to the very short diffusion distances prevailing in the catalytic wall MR compared with the powder catalyst may allow a faster release of the CO₂ species. However this point has to be confirmed with complementary studies that are currently in progress in our laboratories.

Conclusions

After the comparison of the catalytic performance of the CuO_x/CeO₂ catalyst in powder and that of a microreactor that was coated with this catalyst, the second one system presents similar CO conversion levels than the powder but with a superior selectivity. Under high oxygen excess in the feed-stream ($\lambda = 4$ and 6.7), the microreactor not only presents a superior selectivity but also a high CO conversion at temperatures below 160 °C. Although the improvement of the catalytic activity and selectivity in the microreactor can also be associated to some changes in the mass transport phenomena, the more evident contribution is the achieving of a very efficient heat transport during reaction, which is improving the CO oxidation and inhibits the H₂ oxidation and the R-WGS reactions. The microreactor allows obtaining a good control of the set temperature of the reaction, which in the case of this highly exothermic process results crucial for the avoiding of hot spots in the reaction environment.

Concerning the presence of CO₂ and H₂O, both compounds generate a decrease of the catalytic activity of the coated CuO_x/CeO₂ solid, may be because they are competing with CO for becoming adsorbed on the active sites. Nevertheless, the CO₂ produces a

stronger inhibitor effect, probably due to the formation of carbonaceous species over the catalytic surface, especially metallic carbonates that would be blocking the active sites.

Acknowledgments

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TABLE

Table 1. Feed-stream compositions (vol.%) used in the catalytic tests

Composition	CO	O ₂	CO ₂	H ₂ O	H ₂	N ₂	λ
1	0.3	1.0	--	--	50.0	48.7	6.7
2	0.5	1.0	--	--	50.0	48.5	4
3	1.0	1.0	--	--	50.0	48.0	2
4	2.0	1.5	--	--	50.0	46.5	1.5
5	1.0	1.0	2.0	--	50.0	46.0	2
6	1.0	1.0	5.0	--	50.0	43.0	2
7	1.0	1.0	10.0	--	50.0	38.0	2
8	1.0	1.0	2.0	10.0	50.0	36.0	2
9	1.0	1.0	5.0	10.0	50.0	33.0	2
10	1.0	1.0	10.0	10.0	50.0	28.0	2

FIGURE CAPTIONS

Figure 1. CO conversion as a function of the λ factor in the feed-stream and the reaction temperature for the powder catalyst and the microreactor (MR) [λ : (a) 2 and 1.5; (b) 6.7 and 4].

Figure 2. Selectivity for CO oxidation as a function of the λ factor in the feed-stream and the reaction temperature for the powder catalyst and the microreactor (MR) [λ : (a) 2 and 1.5; (b) 6.7 and 4].

Figure 3. Selectivity for CO oxidation as a function of the λ factor in the feed-stream and the CO conversion for the powder catalyst and the microreactor (MR)

Fig.4. Monitoring of the inlet and outlet temperatures during a catalytic test with the MR at 160 °C using different λ factors: (a) 4; (b) 2.

Figure 5. Effect of the presence of CO₂ in the feed-stream and the reaction temperature on the CO content at the CO-PROX microreactor outlet.

Figure 6. Effect of the presence CO₂ and H₂O in the feed-stream and the reaction temperature on the CO content at the CO-PROX microreactor outlet.

FIGURES

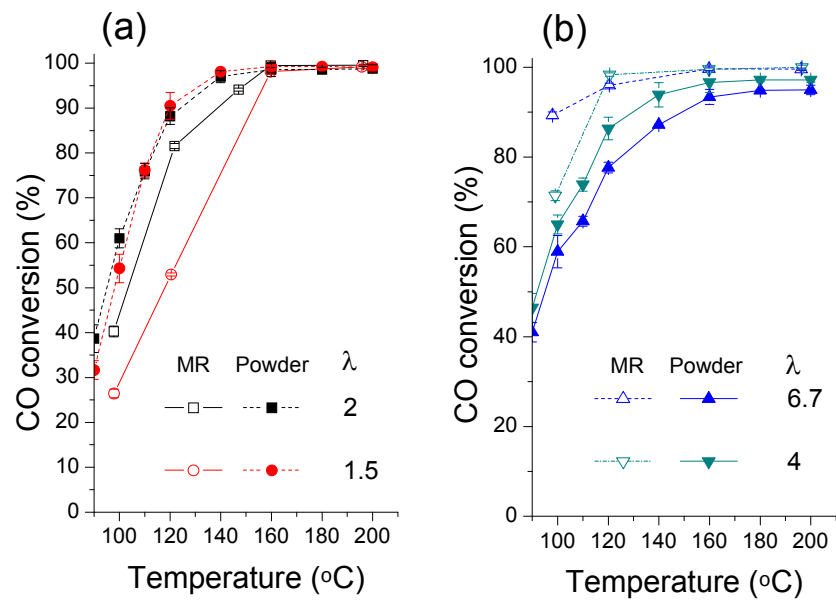


Figure 1

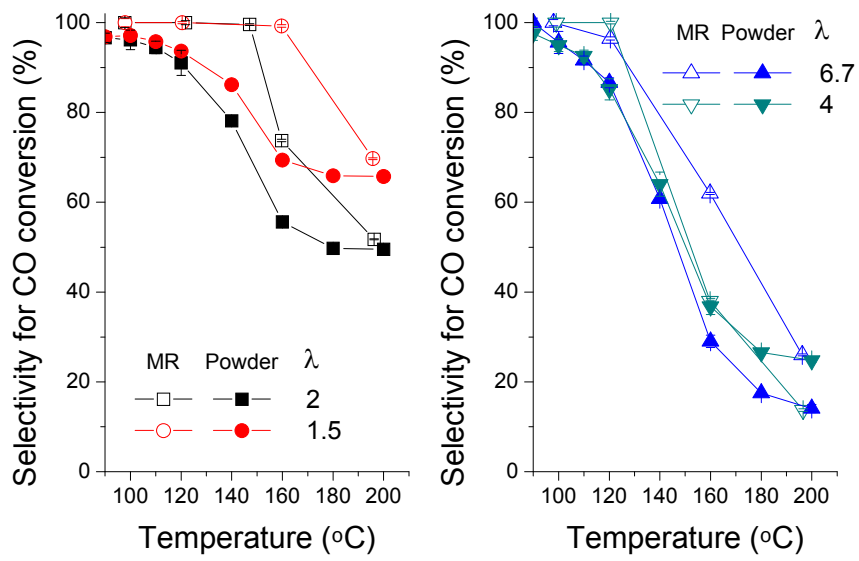


Figure 2

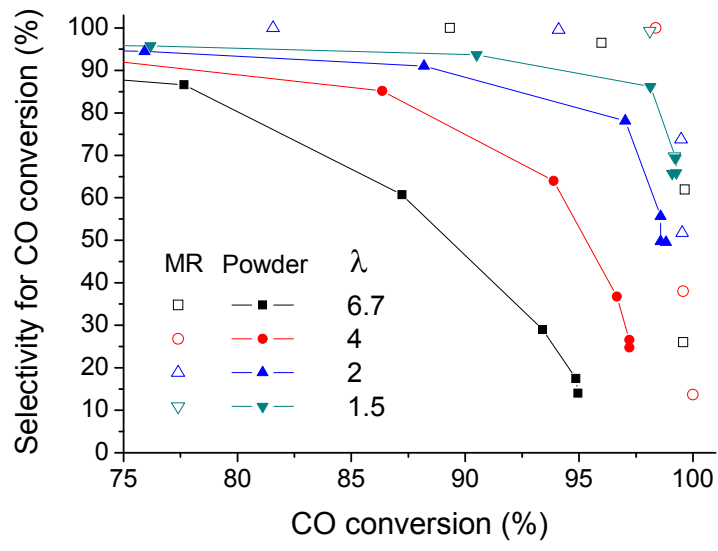


Figure 3

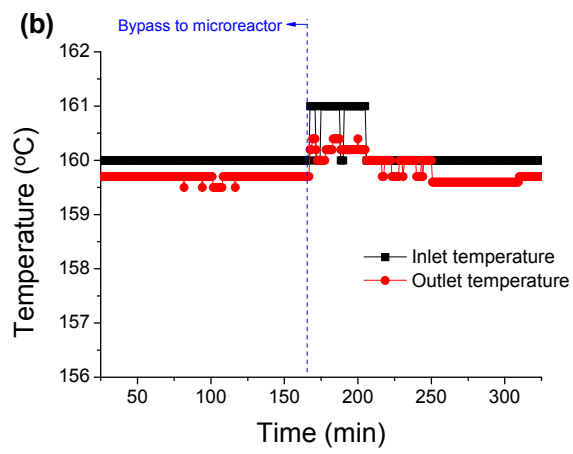
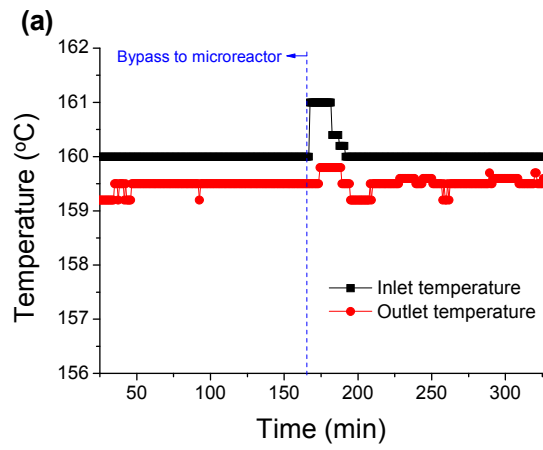


Figure 4

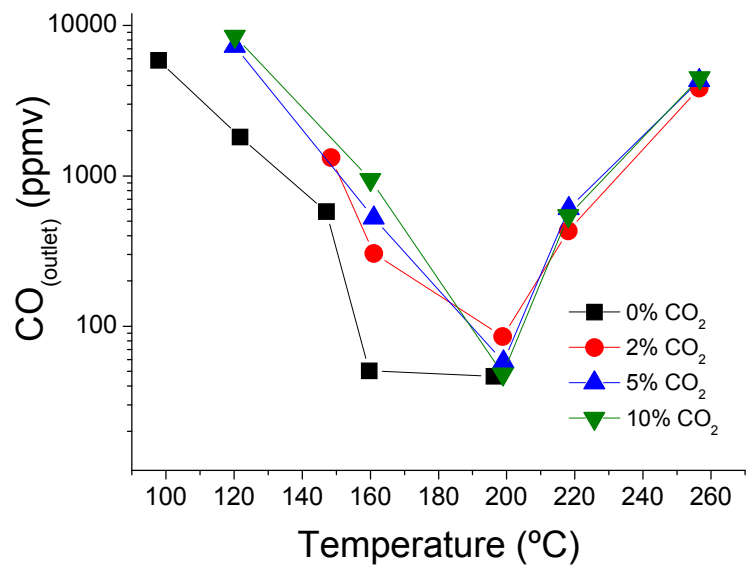


Figure 5

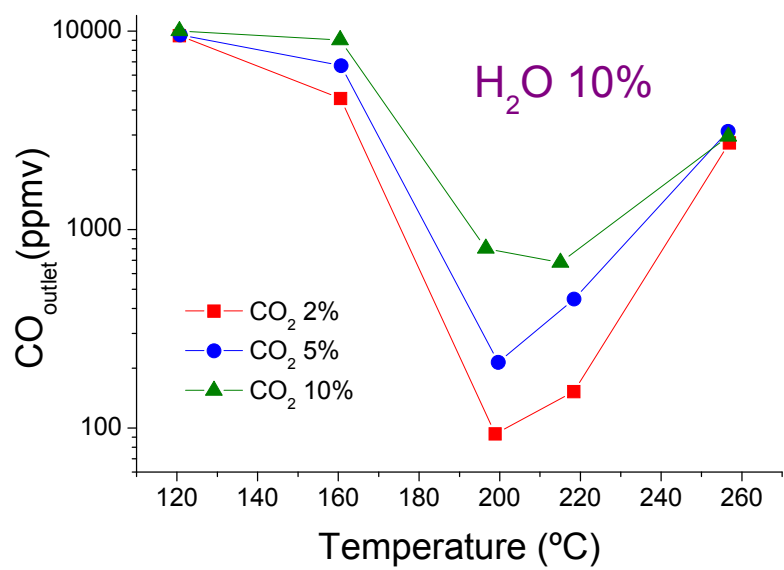


Figure 6