

Experimental analysis of a 2 kW LPG-based fuel processor for polymer electrolyte fuel cells

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Abstract

The paper describes the research activities carried out at the CNR Institute for Advanced Energy Technologies “Nicola Giordano”, aimed to develop, assemble and test a hydrogen generator, named HYGen I, to integrate with a PEFC (polymer electrolyte fuel cell) for residential applications. The unit, capable to convert light hydrocarbons (methane, propane, LPG, butane), with a nominal hydrogen production of $2 \text{ N m}^3 \text{ h}^{-1}$ and a maximum hydrogen production of $5 \text{ N m}^3 \text{ h}^{-1}$, can be considered the only prototype of this size in Italy. Preliminary experimental results, with a proprietary Pt/CeO₂ catalyst for the autothermal reforming (ATR) and commercial catalysts for intermediate water gas shift (ITWS) and preferential oxidation (PROX) have been reported.

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1. Introduction

Worldwide attention has been focused in recent years on the development of fuel cells as alternative energy conversion devices. The polymer electrolyte fuel cell (PEFC) fuelled by hydrogen appears to be the most promising option for both vehicular and small scale combined heat and power applications, due to its compactness, modularity, high power density and fast response [1]. The actual fuel cells market, as number of companies involved, (stack manufacturers, distributors, integrators or otherwise), is in continuous expansion [2]. A growing interest for small stationary applications (0.5–10 kW electrical output range), with a large increase in the number of installed units in the world, as a decentralised power supply, grid support, peak shaving, power back-up or uninterruptible power supply (UPS), can be derived [3]. The trend is confirmed by some fuel cells and hydrogen strategy plans and Government programmes, focused to improve the efficiency of fossil-based technologies, both in the near (to 2010) and in the medium term (to 2020) [4–9]. The absence of a hydrogen refuelling infrastructure and the problems concerning the hydrogen storage, has led to the development

of fuel processors able to convert available fuels (hydrocarbons and or alcohols) into hydrogen rich reformat gas [10]. The choice of a suitable fuel processor and fuel, during the transition phase to a hydrogen economy, are the key aspects to the successful implementation of direct-hydrogen fuel cell systems.

Key requirements for a fuel processor include rapid start-up, good dynamic-response to change in hydrogen demand, high fuel-conversion, small size and weight, simple design (construction and operation), stable performance for repeated start-up and shutdown cycles, maximum thermal integration, low cost and maintenance, high reliability and safety [11].

The most investigated reforming processes for hydrogen generation are steam reforming, partial oxidation and autothermal reforming [12–14]. Conventional steam reforming gives highest H₂ product, but requires considerable heat input. The complex relation between the fuel-to-water ratio, available heat, thermal-mass balance, etc., can result in relatively slow start-up characteristics and poor response to transient demands for steam reformers. Partial oxidation is an exothermic process characterized by relative compactness, potentially good start-up and load-following; it can be considered less energy efficient than steam reforming ones because of the higher temperatures involved and the problem of heat recovery. Autothermal reformers, coupling partial oxidation and steam reforming process, combine the compactness and load following capabilities of

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partial oxidation reactors with the efficiency of steam reformers. The heat generated by the partial oxidation reaction provides the heat required for the steam reforming reaction, without external burner or heat source. Therefore, the autothermal reformers typically offer higher system efficiency of partial oxidation systems, where excess heat is not easily recovered [6,15].

However, to utilize the reformat gas as reactant for PEFC systems, clean-up steps (water gas shift and preferential oxidation) must be considered to reduce the CO concentration at acceptable level (10 ppm) [16,17]. In small scale applications natural gas remains the fuel most employed for its wide availability and related infrastructure. For some niche markets, such as electricity production in remote sites, propane could be an interesting optional fuel [18,19].

Based upon the above considerations a 2 kWe hydrogen generator unit, (capable to reform methane, propane, LPG, butane) has been developed, assembled and tested. The aim of this paper is to describe the research activities carried out at the CNR Institute for Advanced Energy Technologies “Nicola Giordano” for the design and testing of the system.

The prototype is based on three catalytic reaction steps: the autothermal reforming (ATR), the intermediate water gas shift (ITWS) and the preferential oxidation (PROX). The system has been engineered, focusing the design of every single component and the integration of the system, to meet the requirements of small size and volume, improving thermal and mass transfer and the system efficiency. An automation system, to monitor the operating conditions and control the plant equipment, has been developed [20]. Experimental tests developed with a Pt/CeO₂ catalyst for the autothermal reforming (ATR) step coupled with commercial catalysts for intermediate water gas shift (ITWS) and preferential oxidation (PROX), are reported. The main aim of the tests has been to define the operating parameters in order to assure high activity and stability for prolonged start-up and shutdown cycles, indicating the drawbacks of the system for a future implementation of a commercial unit. Besides, the tests were aimed to validate heat and mass balance and evaluate the reactors and system performance (in steady state and in transient response) in terms of fuel conversion, hydrogen content in the product gas and efficiency.

2. Prototype design

HYGen I can generate hydrogen for polymer electrolyte fuel cell for small stationary applications. The prototype is able to convert light hydrocarbons (methane, propane, LPG, butane) with a nominal hydrogen production of 2 N m³ h⁻¹ and a maximum hydrogen production of 5 N m³ h⁻¹. The net size (mm) of the integrated fuel processor, shown in Fig. 1, is 870 (width) × 880 (length) × 970 (height).

2.1. Catalysts

The reaction conditions and the catalyst formulation, that can assure high activity, good thermal, mechanical and chemical stability have been derived from previous micro-scale tests [21,22].

ATR catalyst is a CNR-ITAE proprietary catalyst; the Pt/CeO₂ sample has been previously selected and the operating parameters such as temperature, O₂/C₃H₈ and H₂O/C₃H₈ molar ratios and gas hourly space velocity (GHSV) have been evaluated. The catalyst, prepared by combustion-synthesis and pressed into pellets 3 mm × 3 mm, operates at 600 °C (light-off temperatures of 300 °C) under atmospheric pressure with GHSV of 5000 h⁻¹, O₂/C₃H₈ and H₂O/C₃H₈ ratios of 1.5 and 3.0, respectively.

Two commercial catalysts have been selected for ITWS and PROX reactions, and used in their manufactured shapes and sizes. The ITWS catalyst (pellets form 3 mm × 3 mm), supplied by NexTech Materials, operates at 320 °C (light-off temperature of 260 °C) under atmospheric pressure with GHSV = 30,000 h⁻¹ and H₂O/CO ratio of 3.0. The choice of the operating temperature of ITWS step is a compromise between thermodynamics requirements, that severely restricts conversion at higher temperatures [23] and the need to achieve an optimized unit heat integration.

The PROX catalyst (pellets form 3 mm × 3 mm), supplied by Engelhard, operates at 120 °C (light-off temperature of 90 °C) under atmospheric pressure with a gas hourly space velocity of 4000 h⁻¹ and O₂/CO ratio of 2.

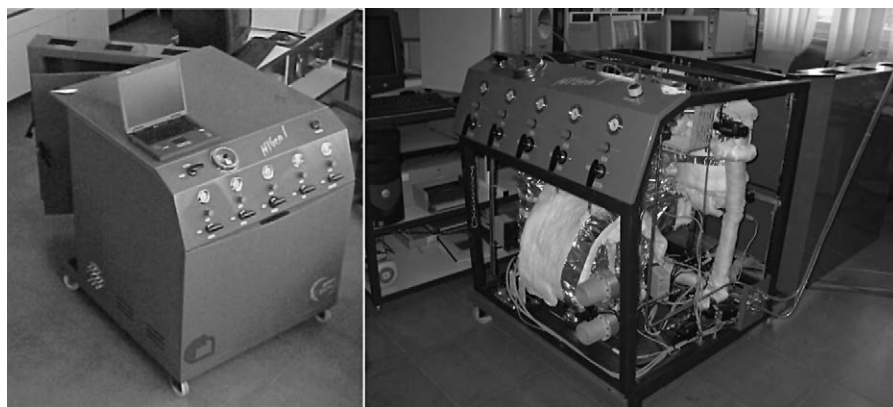


Fig. 1. View of the 2 kWe fuel processor (HYGen I).

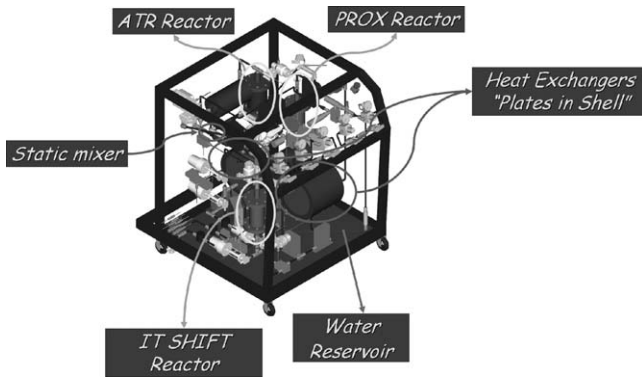


Fig. 2. Diagram of the 2 kWe fuel processor (HYGen I).

2.2. Components features and system integration

The processing unit consists of three coupled reactors, filled with the above-cited catalysts.

The ATR reactor is an Incoloy 800 HT tube with an internal volume of 0.4 l. The Incoloy 800 HT was selected as material because it can withstand hydrogen embrittlement, high temperatures and thermal cycling. The ITWS and the PROX reactors are AISI 316 L tubes, with an internal volume of 0.2 and 2.5 l, respectively.

The diagram of the integrated system is reported in Fig. 2; the main three fixed bed reactors are coupled with heat exchangers, a water reservoir, static mixers, manual/automatic valves, pressure regulator/transducers, flow meters/regulators, electrical heating elements and ancillaries.

The management of heat recovery is assured by two “plates in shell” heat exchangers (PSHE), supplied by Vahterus, made in AISI 316 L and with an internal volume of about 4.3 and

9.2 l, respectively; a fully welded pack of circular plates, housed within an outer pressure vessel in the PSHE exchanger, offers a durable, compact and gasket free solution with high heat transfer, high pressure (up to 100 bar) and high temperature capability (up to 900 °C). The high turbulence created by the complex geometry of each plate determines an high PSHE efficiency.

Four static mixers, in AISI 316 and designed at CNR-ITAE, are used to mix the different fluids. The water reservoir, made in AISI 316 L, is located inside the support. The design of the prototype included an analysis (3D CAD modeling) of the accessibility for maintenance work, assured for every component.

Moreover various connections allow the analysis of reactants and products from each reactor using both gas chromatograph (Agilent 6890 Plus), equipped with a thermal conductivity and a flame ionization detectors and a mass spectrometer (VG Pro-labo).

2.3. Management system

The prototype includes a completely automated acquisition data and control system. Control of plant parameters (fluxes, temperatures, absolute pressures, differential pressures, electro valves state, electrical heating elements) and storage of data process, at user-defined intervals, were realized by coupling the management software and the measurement hardware. The integrated software and hardware platform can control the unit in two operation modes: a semi-automatic control, to test the performance of the prototype with manual procedures, and a fully automatic screening.

In particular, the monitoring of operating parameters and the control of plant equipment, is carried out by Field Point distributed I/O hardware, managed by a dedicated graphical

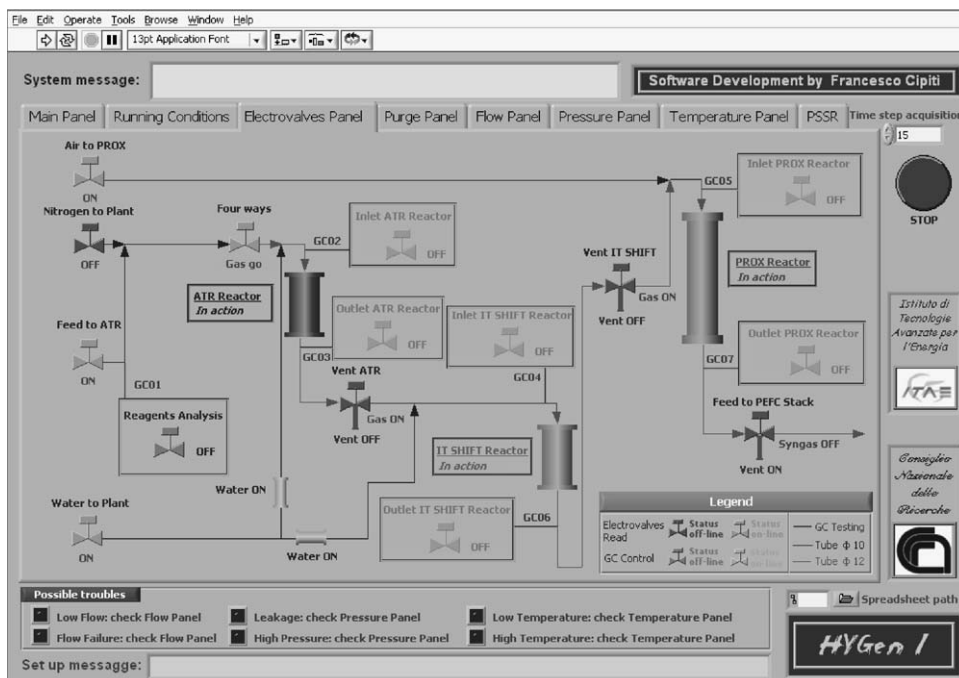


Fig. 3. Software screen, developed using Labview, depicting a working state of the system.

software, developed at CNR-ITAE using Labview; the graphical environment is able to create flexible and scalable tests and measurement/control applications. Labview programs can easily be used from simple data-logging applications to highly sophisticated control systems depending on needs; a picture of the software screen is shown in Fig. 3.

Field Point is a measurement and control hardware, designed for distributed I/O, and embedded control and measurement systems. A wide operating temperature range, watchdog timers, defined power-up states, automatic diagnostic, and many other industrial features make Field Point ideal for reliable process automation and monitoring with a connection to a supervisory computer.

Labview and Field Point are based on open architectures, and can be easily integrated with other software and hardware through industry-standard interfaces.

Based upon the above considerations, the control instrumentations (flow meters/regulators, thermocouples, absolute and differential pressure transmitters, power solid state relays with analog control) have been identified. Furthermore, we provided the plant with the electronically controllable components, such as electro-valves and contactors, obtaining the automation of the process control.

In order to have complete information about every parameter, we developed all the possible operation modes that allowed us to analyze the detailed workings of each process phase.

In particular two main working configurations have been investigated, in order to assure highest operating flexibility of the unit: the system can run in a single stage mode named “individual order” (ATR, or ITWS using a proper simulated gaseous feed, or PROX, or ATR/ITWS) or in the normal configuration named “sequential order” with the above-cited reactors in series connected. In this last configuration, the ITWS and the PROX are heated using the product’s heat of the previous catalytic reaction step.

The safety system consists of different action cycles for some different events (P_{max} : maximum pressure, P_{min} : minimum pressure, T_{high} : high temperature, T_{low} : low temperature, Q_{max} : maximum flow rate, Q_{min} : minimum flow rate). The safety levels with the monitoring instrumentations and the controlled parameters are shown in Table 1. Two action cycles were generally defined for each controlled parameter: a pre-alarm (visual and sound signal) and alarm, that modifies the working configuration of the system to isolate any malfunctions or, alternatively, switch on the shutdown phase.

The management of the action cycles is based on the following control levels in order to guaranteed safety and reliability:

Table 1
HYGen I safety levels

Safety levels	Monitoring instrumentations	Controlled parameters
Manual	Manometer, monitor	Pressures, temperatures
Active	Electro-valves	Pressures, temperatures
Passive	Safety valves, gas sensor	Pressures, leakages

- *manual level*. The monitoring of temperatures and pressures is carried out by the operator;
- *active level*. Operative when the controlled parameters reach a value out of the setup ranges. The monitoring is carried out by the management system software;
- *passive level*. Activated by controlled fluid and maintains temperature and pressure in the fixed range. The monitoring is carried out by safety valves and gas sensors. The management system, protects also against danger sources related to elements not easily identifiable, such as high pressure or leakage.

3. Results

The P&I of the system, is reported in Fig. 4. During the start-up, nitrogen is sent to the ATR reactor and heated by electrical heating elements. When the temperature of the ATR catalytic bed is ca. 300 °C the nitrogen is stopped and air–propane–steam mixture is sent to the ATR reactor: at regime conditions, the air–propane–steam mixture is preheated by using the reaction products in the jacket of the ATR reactor. External heating is supplied only during the start-up; on regime operations the global heat balance is smoothly exothermic. During the shutdown, the air–propane–steam mixture is shut-off and nitrogen is sent as a purge. The electrical modulation of the power solid state relays is automatically regulated to reach and maintain the temperatures set, relieved by thermocouples. Table 2 reports the main functions of some prototype components.

The preliminary tests were carried out to analyze:

- heat and mass balance of the prototype;
- optimization of start-up and shutdown methodology (light-off temperature, start-up time, etc.);
- ATR catalytic activity (propane conversion, product distribution);
- ATR catalytic stability (steady state regime, influence of frequent start-up and shutdown cycles, etc.);
- carbon deposition phenomena;
- by-products formation;
- evaluation of the control and check systems.

Table 2
Main functions of some prototype components

Components	Services
Heat exchanger EH2	Evaporating water to be used in ATR reactor, using heat from ATR product gas
Heat exchanger EH3	Evaporating water to be used in ITWS reactor, using heat from ITWS product gas
Mix 1	Mix air and propane
Mix 2	Mix the air–propane mixture with steam from EH2 to be used in ATR reactor
Mix 3	Mix the ATR product gas with steam from EH3 to be used in ITWS reactor
Mix 4	Mix ITWS product gas with air to be used in PROX reactor
Water reservoir	Store water to be used in the ATR and ITWS reactors

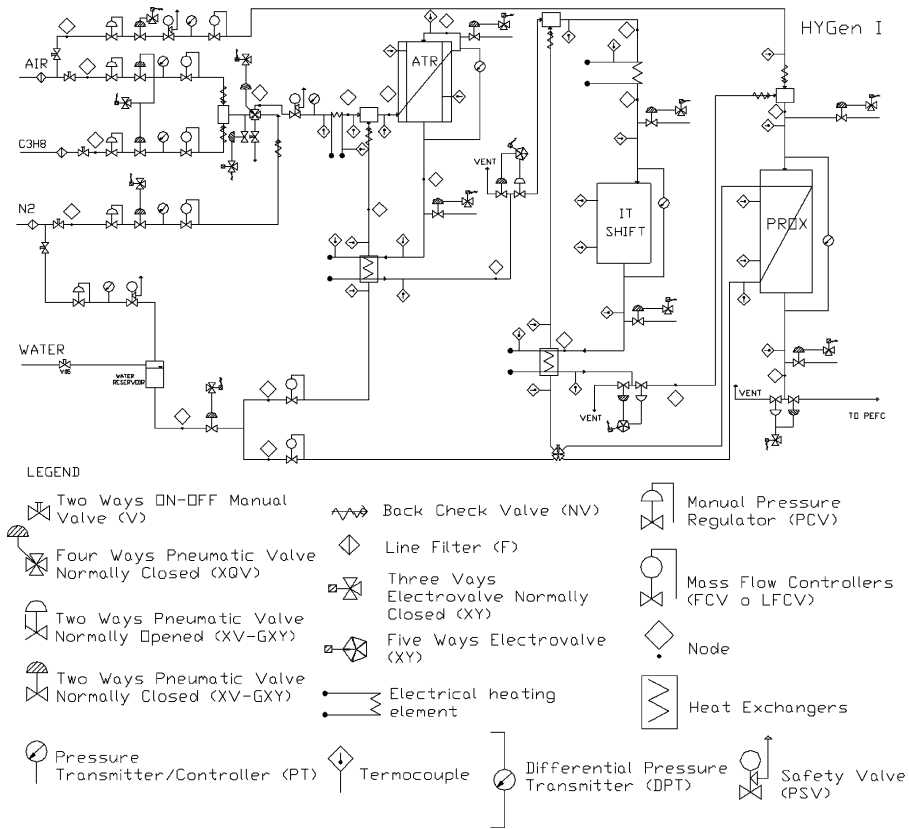


Fig. 4. P&I of the 2 kWe fuel processor.

The experimental tests have been carried out with four daily cycles of 6 h continuous test and one daily cycle of 3 h continuous test for a total of 27 h, using propane as fuel at constant flow rate of the gaseous feed in order to analyze the performance of the system in steady state; at the end of each test the reactor has been maintained at ambient temperature.

The experimental conditions of each reaction step are sensitized in Table 3.

3.1. Autothermal reforming

The autothermal reforming propane was performed by heating the catalytic bed sending N₂ flow at 300 °C. Subsequently N₂ flow was stopped, and the air–propane–steam mixture was sent to the reactor; a rapid increase in temperature was observed that stabilised within some minute at 600 °C.

Fig. 5 showed the catalytic stability for the reforming step at the reaction conditions of 600 °C and molar ratios of O₂/C₃H₈ = 1.5 and H₂O/C₃H₈ = 3 over 25 h. The catalyst exhib-

ited good stability over the investigated time, indicating the resistance to coke deposition, as confirmed by characterization of spent catalyst. Total C₃H₈ and O₂ conversion (not showed) was reached; the H₂ and CO composition (dry basis) was almost 28–30% and 10–8%, respectively, associated with 10–12% of CO₂ and 8–6% of CH₄ in absence of lower hydrocarbons. The outlet composition approaches the predicted thermodynamic equilibrium value at the current experimental conditions: H₂/CO/CO₂/CH₄ = 32/8.9/11.15/3.1, except for the CH₄ amount that results more high than the thermodynamic value.

Table 3
Operating conditions employed during the tests

Reaction	Experimental conditions
ATR	GHSV = 5000 h ⁻¹ , T = 600 °C, O ₂ /C ₃ H ₈ = 1.5, H ₂ O/C ₃ H ₈ = 3
ITWS	GHSV = 30,000 h ⁻¹ , T = 330 °C, H ₂ O/CO = 3
PROX	GHSV = 4000 h ⁻¹ , T = 120 °C, O ₂ /CO = 2

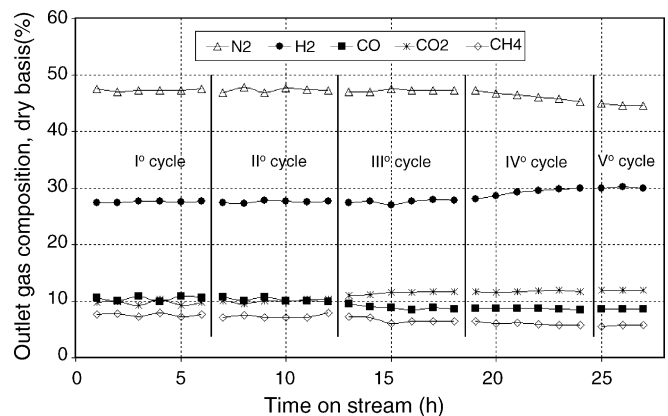


Fig. 5. Products gas compositions from the autothermal reforming of propane at 600 °C, GHSV = 5000 h⁻¹ with O₂/C₃H₈ = 1.5 and H₂O/C₃H₈ = 3.

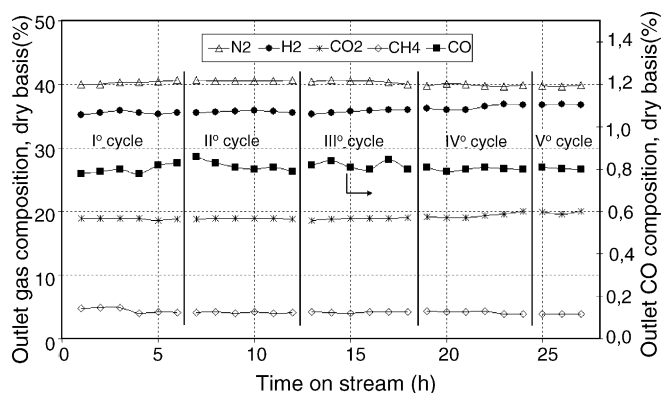


Fig. 6. Products gas compositions from the water gas shift step at 320 °C, GHSV = 30,000 h⁻¹ with H₂O/CO = 3.

The light increase in the H₂ and CO₂ output, and conversely the CO and CH₄ decrease, during the time on stream was due to the slow stabilization in the stream supply to the setup value. The resulting increase in the H₂O/C₃H₈ to the setup value during time, can determine a more high contribution of water gas shift and steam reforming reactions. Thermal cracking of C₃H₈ may be responsible for the high amount of CH₄; previous investigation [22] have been evidenced that increasing the O₂/C₃H₈ ratio (from 1.4 to 3) results in a decrease of CH₄ produced. Tests, carried out by a fine tuning of O₂/C₃H₈ and H₂O/C₃H₈ molar ratios in the feed, to obtain a reformate with the highest possible amount of hydrogen and CO minimizing the CH₄, are in progress. The main trouble of this experimentation regards the high start-up time (from ambient temperature to light-off temperature, about 50 min).

On the basis of experimental results, the related ATR efficiency $\eta_{\text{H}_2\text{HHV}}$ (%) (higher heating value based) reaches a value of about 50% and is defined as:

$$\eta_{\text{H}_2\text{HHV}} (\%) = \frac{n_{\text{H}_2}\text{HHV}(\text{H}_2)}{n_{\text{C}_3\text{H}_8}\text{HHV}(\text{C}_3\text{H}_8)} \times 100$$

where n_{H_2} (mol h⁻¹) denotes the hydrogen molar flow output after ATR; $n_{\text{C}_3\text{H}_8}$ (mol h⁻¹) denotes the propane molar flow input to the reformer; HHV(H₂) (kcal mol⁻¹) and HHV(C₃H₈) (kcal mol⁻¹) denote the higher heating values of H₂ and C₃H₈, respectively.

3.2. Intermediate water gas shift

The mildly exothermic water gas shift reaction was performed by preheating the catalytic bed at 260 °C, connecting the cooled effluent from ATR reactor and changing the steam ratio to the value of H₂O/CO = 3. The bed temperature quickly reached 330 °C by the exothermic CO conversion. At the reaction temperature of 330 °C and a gas hourly space velocity of 30,000 h⁻¹, the fuel gas mixture from the exit of the water gas shift reactor is enriched with hydrogen giving a low concentration of CO. The related products composition is shown in Fig. 6; H₂ and CO content of about 34 and 0.8% (dry basis) were, respectively, observed.

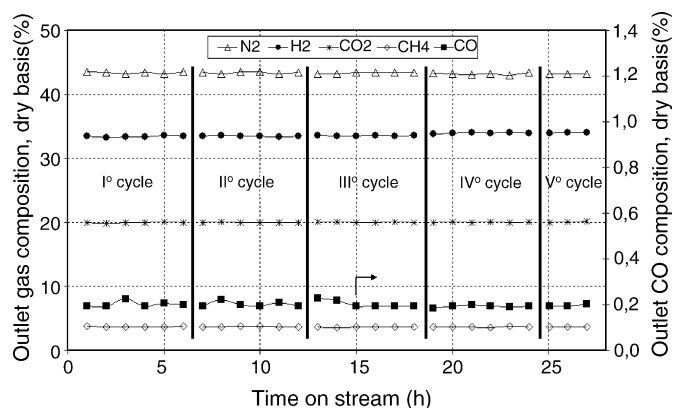


Fig. 7. Products gas compositions from the preferential oxidation step at 120 °C, GHSV = 4000 h⁻¹ with O₂/CO = 2.

3.3. CO preferential oxidation

The CO preferential oxidation was performed by heating the reactor at 90 °C with a gas hourly space velocity of 4000 h⁻¹ under excess of O₂ (O₂/CO = 2); this value, slightly higher than the stoichiometric value, was used considering that the CO content at the ITWS exit can fluctuate incidentally during the transient operation. During the tests, the temperature control along the catalytic bed was not satisfactory; the thermal balance between the exothermic nature of the oxidation reaction and the used techniques to cool the catalyst bed, caused not isothermal conditions. As a consequence the residual CO content in the output of the reactor reaches about 0.2% (dry basis). The obtained products composition is reported in Fig. 7; the CH₄ amount was substantially unchanged, whereas a low H₂ consumption, respect to ITWS outlet, was noted.

4. Conclusions

The proposed prototype satisfies some of the requests of a proper hydrogen generator, like good propane conversion and H₂ production, small unit size, improved thermal and mass transfer, very simple system management. The automation system results suitable for laboratory tests, due to its flexibility and to the possibility of integrating the management software with automation hardware and personal computer into a single system.

The main drawbacks evidenced includes high start-up time and the high CO level in the PROX output stream associated with an high CH₄ content.

Based upon this experience, additional tests to verify the performance in terms of chemical stability (for prolonged start-up and shutdown cycles) and to overcome the limit factors of the unit, are in progress. However, the results show the need of improving the prototype engineering, focusing on further efforts to achieve optimized design of the PROX subsystem.

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