5 KW_E+ 5 KW_T REFORMER-PEMFC ENERGY GENERATOR FROM BIOETHANOL FIRST DATA ON THE FUEL PROCESSOR FROM A DEMONSTRATIVE PROJECT

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ABSTRACT

A power unit constituted by a reformer section, a H₂ purification section and a fuel cell stack is being tested c/o the Dept. of Physical Chemistry and Electrochemistry of Università degli Studi di Milano, on the basis of a collaboration with HELBIO S.A. Hydrogen and Energy Production Systems, Patras (Greece), supplier of the unit, and some sponsors (Linea Energia S.p.A., Parco Tecnologico Padano and Provincia di Lodi, Italy). The system size allows to co-generate 5 kW_e (220 V, 50 Hz a.c.) + 5 kW_t (hot water at 65°C) as peak output. Bioethanol, obtainable by different non-food-competitive biomass, is transformed into syngas by a pre-reforming and reforming reactors couple and the reformate is purified from CO to a concentration below 20 ppmv, suitable to feed a proton exchange membrane fuel cell (PEMFC) stack that will be integrated in the fuel processor in a second step of the experimentation. This result is achieved by feeding the reformate to two water gas shift reactors, connected in series and operating at high and low temperature, respectively. CO

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concentration in the outcoming gas is *ca*. 0.4 vol% and the final CO removal to meet the specifications is accomplished by two methanation reactors in series. The second methanation step acts merely as a guard, since *ca*. 15 ppmv of CO are obtained already after the first reactor.

The goals of the present project are to test the integrated fuel processor, to check the effectiveness of the proposed technology and to suggest possible adequate improvements.

Keywords: Hydrogen production; Integrated fuel processor; Steam reforming; Bioethanol.

1 - INTRODUCTION

H₂ is one of the most promising energy vectors proposed to supply energy for both stationary and mobile applications, provided that the overall carbon balance can approach zero, in contrast with the current hydrogen production processes, almost fully based on fossil primary sources.

Water electrolysis powered by solar or wind energy, or the direct photo-electro-catalytic water splitting may be seen as long-term forecasts for clean H₂ production. Applicable short-term solutions presently rely on the *on demand* production of H₂ from a source which should be easy to handle, non toxic, with high energy density, easy to produce, store and transport, widely diffused and, of course, derived from renewable sources. Bioethanol is a suitable raw material from this point of view, as witnessed by the increasing scientific and applicative interest in recent years [1-3]. Indeed, its use as primary fuel for FC combines the advantage of the high energy density of a liquid fuel and the high energy conversion efficiency of the FC.

The use of bioethanol as fuel for transportation, in blends with fossil-sources-derived gasoline, has been imposed by the most recent EU regulations (e.g. 2003/30/CE). The U.S. alone produced 13.2 billion US gallons of ethanol fuel in 2010 and the projections on the worldwide production for the same year were almost 22 billion gallons, being ca. 19 billion in 2009 [4].

In the recent past, the so-called second-generation bioethanol was proposed, derived from lignocellulosic biomass, such as sorghum, mischantus or poplars, possibly growing in marginal lands and to which a short rotation forestry can be successfully applied [5].

As for the Italian scenario, in northern Italy (Tortona) a 40 kton/y plant for the fermentation of lignocellulosic materials is presently under construction. The production is based on Arundo Donax, a widely spread wild cane, growing on marginal lands, with minimum water, virtually nil fertilizers input and high ethanol productivity, integrated with wheat straw available locally. The plant requires 4000 hectares of low quality land for cane cultivation.

In spite of the research efforts to improve ethanol conversion and hydrogen yield, from a practical point of view it is compulsory to test the real efficiency of the whole energy conversion technology, from H₂ production and purification to power and heat output. Other key points for the market penetration are system durability, reliability and flexibility.

Despite a broad and growing literature mainly focusing on catalyst formulation [6-11], only a few reports on fuel processors, and even fewer on integrated systems can be found in the literature. Possible system layouts for the reforming of biomass-derived fuels have been recently reviewed [12,13].

Low-temperature ethanol reforming has been carried out on a cobalt catalyst. The collection of kinetic data under different experimental conditions allowed the development of a dynamic model for a tubular reformer loaded with catalytic monoliths for the production of hydrogen to feed a 1 kW PEMFC [14,15]. In another paper, the energy balance of an ethanol-fuelled SOFC has been proposed, hypothesising different system layouts [16].

The optimum design, modelling and simulation of a fixed bed multi-tube reformer was described by Arteaga et al. [17]. The fuel processor size was suitable to feed a 200 kW FC system (>2.19 mol H₂ /s) and the prototype was analysed and optimised using a multiphysics and axi-symmetric model. Simulation of a SR or ATR fuel processor + clean up section and PEMFC was also proposed, in case coupled with a membrane reactor [18]. Simulations were also carried out for an on-board SR or ATR fuelled with biodiesel or bioethanol [19] and for a 5 kW system fuelled with natural gas [20].

The detailed design of a microreactor for methanol reforming has been recently depicted [21] and the detailed life cycle assessment of methanol fuelled SOFC system has been evaluated [22]. Furthermore, honeycomb type reactors were tested for the reforming of synthetic biomass gas on both lab and pilot scale [23].

Bower et al. [24] described an on-board reformer-FC system designed for gasoline, but also adaptable to ethanol. It was based on auto-thermal reforming (ATR) coupled with high- and low-temperature water gas shift (HT- and LT-WGS) for hydrogen purification. The final purity specification was below 100 ppm CO, accomplished by preferential oxidation (PROX).

Furthermore, a micro-structured fuel processor has been developed to convert different fuels, including ethanol, to hydrogen [25], also in this case substantially based on ATR and PROX. Kinetic modelling on microchannel reactors is also available [26].

More recently, Aicher et al. [27] showed very interesting data on a demonstrative 250 W power system based on ATR of ethanol to produce hydrogen. That system was integrated with a H₂ purification train so to attain CO concentration of *ca*. 20 ppmv, by coupling WGS and methanation.

A hybrid plant based on an ethanol reformer has been also studied, using a concentrating solar power plant for reactor heating [28].

The ethanol steam reforming reaction has been considered for producing pure hydrogen for feeding a 4 kW PEMFC [29]. A Pd–Ag membrane reactor was taken into account for an energy efficiency analysis of the processes, comparing that solution with a traditional process. The latter consisted of an ethanol reformer followed by two WGS and one PROX reactors, to reduce the CO concentration below 10 ppm before feeding a PEMFC.

Finally, a few hours operation of a 5 kW SOFC system with ethanol as fuel instead of natural gas has been described by Lanzini et al. [30], together with a thorough simulation of the native NG-fuelled plant. A balance of plant of a similar system has been also simulated [16]. In spite of these few reports, often based on simulated data only, and various project descriptions, there is a fundamental lack of experimental data on integrated fuel processors. Therefore, we started a demonstrative experimentation on an assembled reformer-H₂ purification system, sized for 5 kW_e + 5 kW_t cogeneration by a PEMFC. In the present paper we report the first data on the performance of the fuel processor, to define its suitability as for the targeted power size and H₂ purity requirement.

The size selected for the present demonstrative unit is suitable for small houses (*ca.* 5 kW_e + 5 kW_t), but a perspective goal of the project will be to extend the experimentation to a larger unit (*ca.* 200 kW_e + 200 kW_t). One of the main novelties here proposed relies on a basic difference with respect to some other proposals of biomass valorisation. Indeed, at the moment biofuels are mainly used as blends for gasoline or diesel, combustion being intrinsically a low-efficiency process, in addition characterised by noxious emissions. By contrast, a theoretical 50% efficiency (increasing to 80% for combined heat and power production, CHP) could be envisaged for the here proposed coupling of fuel processor with a

PEMFC system. Taking into account the efficiency of the fuel processor, one may expect *ca*. 40% efficiency on electrical power generation, raising to 60-65% for CHP, to be compared with the *ca*. 30% efficiency of a common internal combustion (IC) system. Of course, the overall energetic efficiency may be increased by CHP also in the latter case, but the contribution of the highly valued electric energy term remains much higher for FC than for IC power units. In spite of the attractiveness of this technology, poor or no data on the fully integrated process are still available, this topic representing one of the final goals of the present experimentation.

The research has been mainly concentrated on the adaptation of some consolidated refinery processes to different biomass-derived feed, such as gasification and reforming, both SR and ATR [31]. Gasification can successfully transform solid biomass, but it is preferentially applied to larger-size plants and it requires direct H₂ consumption on-site, due to storage and distribution problems. By contrast, reforming of liquid fuels has been proposed for different power demand and it is also viable for mobile applications. Due to its versatility, both in terms of size and feed, this project focused on SR. ATR has been often proposed in the literature, but it may lead to lower volumetric concentration of hydrogen with respect to SR, requiring high volumetric flow through the FC.

2 - EXPERIMENTAL

The integrated fuel processor has been developed by HELBIO SA. It consists of six reactors connected in series, as sketched in Fig. 1 (auxiliary equipment, interconnections and heat recovery systems were not reported for simplicity).

Bioethanol from marc, the widespread byproduct of wine production (Alcoplus, 99.0 vol%) was diluted to 96.0 vol% with deionised water before feeding the unit. Indeed, 96 vol% ethanol was considered as the usual fuel, due to its lower cost. However, the effect on reforming catalyst of the possible different impurities present in the commercial 96% alcohol will be studied in the near future. The alcohol is fed to a heat-exchange reactor (shell and tubes) containing both the steam reforming catalyst and a commercial combustion catalyst. After evaporation ethanol is mixed with air for feeding the combustion side and with steam to feed the hydrogen production line. The latter reacting mixture is first fed to a pre-reformer, then to the main reforming reactor. Heat recovery is provided between these two units. Two water gas shift reactors and then two methanation reactors follow the reformer. Heat exchange is provided between each of them to progressively lower the reaction temperature. Excess water is condensed at the outlet of the last reactor by cooling at ca. 80°C. A pressure relief valve allows to set the reformate discharge pressure so to match the FC requirement. Gas sampling points are available after each reactor. Products analysis was carried out by means of an Agilent 7890 GC equipped with FI and TC detectors and with Poraplot Q and Molecular Sieves columns. The analytical method allows the quantification of ethanol, water, H₂, CO, CO₂, CH₄, acetaldehyde, ethylene, ethane and acetic acid. A methanation device was mounted before the FID, to quantify accurately the CO (last steps of the purification section).

3 – RESULTS AND DISCUSSION

The catalytic combustion of ethanol is here separated from the hydrogen production line, but optimal heat exchange to the reformer is ensured by proper reactor configuration as shell and tube heat exchanger. Hence, the combustion and reforming catalysts are physically separated, but thermally coupled. Details on this technology may be found elsewhere [32]. Steam reforming (SR) is an endothermal process [33,34] requiring a heat input. The reaction may be schematically represented as:

 $C_2H_5OH + H_2O \rightarrow 2 CO + 4 H_2$

and it is usually coupled with water gas shift:

$$CO + H_2O \leftrightarrows CO_2 + H_2$$

leading ideally to 6 mol of H₂ per mol of ethanol converted. The latter reaction is useful both to improve H₂ yield and to convert CO, a poison for fuel cell catalysts. However, due to the opposite reaction enthalpy, the operating conditions of SR are poorly compatible with the exothermal WGS, so that H₂ yield and CO concentration in the reformate gas strongly depend on the reaction temperature. Acetaldehyde can be produced from ethanol dehydrogenation and usually it reforms or decomposes under relatively mild conditions. Methane may also form by ethanol decomposition and it is further reformed. However, the reforming of CH₄ requires higher operating temperature than ethanol reforming.

In the fuel processor here tested a first pre-reforming step is present, operating with a HELBIO SA proprietary catalyst at 300-350°C, GHSV = 1900 h⁻¹, leading to almost full ethanol conversion into H₂, CO and CH₄. By contrast, the main reformer is operated between 550 and 750°C, GHSV = 7700 h⁻¹, allowing the conversion of the produced CH₄ besides the residual ethanol.

Reactor heating is accomplished by combustion of part of the ethanol over a commercial combustion catalyst. The latter is loaded as mentioned before, in the tubes of a heat exchanger type reactor, hosting the reforming catalyst in the shell side.

The water/ethanol ratio in this case has been set to *ca.* 5.5 mol/mol, to prevent extensive coking and to drive the WGS equilibrium towards CO removal.

The reformate gas composition depends on the operating conditions and the catalyst employed. The pressure at the outlet of the processor has been firstly adjusted to 0.8 bar(g) by using two relief valves, in order to feed the FC module. Additional tests have been also carried out at higher pressure, *i.e.* 0.95 bar(g), with similar results.

Typical gas composition after each reactor is reported in Table 1, as vol% on a dry basis. H₂ concentration was *ca.* 60% after the first pre-reforming step, as expected since a high methane yield was achieved by ethanol decomposition, but reaction temperature was too low to achieve its full reformation. The latter was accomplished in the subsequent reforming reactor, where methane was fully converted. However, the CO/CO₂ ratio increased due to the thermodynamically unfavourable conditions for the WGS reaction.

H₂ purity is a fundamental task for its use in fuel cells, to prevent rapid and irreversible deactivation of the FC catalyst. For the present application a PEMFC meets the power requirements. Indeed, these cells are considered suitable since their development is in a semi-commercial stage and their power density is comparatively higher than other FC. They operate at low temperature (*ca.* 80°C) and hence they do not require long start-up periods, showing adaptable also for emergency devices. Nevertheless, CO poisoning of the Pt-based electrodes is a serious drawback when H₂ is derived from organic feedstocks. The fuel processor here adopted is designed to decrease CO concentration below 20 ppm in the reformate, suitable to feed PEMFCs operating around 80°C. However, our future

experimentation will be carried out also on a recently developed high temperature PEM fuel cell (HT-PEMFC), operating at 160-180°C. The increase of the FC working temperature represents an advantage for a higher quality heat output and mainly for a much lower sensitivity to CO (depending on power loading, even 2 vol% CO or more may be tolerated). This would allow to sensibly simplify the present reactors layout, by eliminating both the methanation units. Major drawbacks could be a lower reliability of HT-PEMFC, due to lacking experience and more critical operation when frequent start-up and shut down occur.

H₂ purification is here accomplished by water gas shift (WGS), an exothermic reaction, which is however kinetically limited at low temperature [31,35]. Therefore, it is common to split it in two, high-temperature (HT) and low temperature (LT), WGS steps. The former is operated around 350°C and it converts *ca.* 90% of CO, the remaining portion being abated in the LT step, operated at 280°C, to shift the equilibrium towards *ca.* 0.3-1% CO.

Typical gas composition outflowing from both WGS reactors (Table 1) shows that some methane forms due to methanation of CO or CO_2 and H_2 concentration is higher than 70% on a dry basis. However the most important datum is CO level, kept below 2 vol% after the former WGS stage and below 0.5 vol% after the latter. Such purity level is perfectly suitable to feed the HT-PEMFC.

The further purification required to meet the higher H₂ purity demand of low temperature PEMFC (CO<20 ppm), is accomplished by the two selective methanation reactors, operated at 215 and 210°C respectively. Preferential oxidation (PROX) could be used after WGS to reach the desired hydrogen purity. However, this would require air addition to the reformate, with increasing system complexity. Otherwise a pressure swing adsorption (PSA) system could be used, which however would require to operate the entire system at higher pressure, with increasing cost and safety issues, or to provide a compressor to increase significantly the

reformate pressure at the outlet of the last WGS stage. This would also imply a more rigid control of the FC operating pressure. Therefore, methanation seemed the most suitable solution for the present application. The CO amount was quantified in the last three reactors by the methanation-FID device of the GC, which allows a detection limit down to 2 ppmv. CO concentration decreased from 0.4 vol% after the LT-WGS stage, to *ca.* 15 and 10 ppmv, respectively, after the first and second methanation reactors. Therefore, the present fuel processor showed fully suitable for H₂ production with enough purity for both a LT-PEMFC (CO < 20 ppmv) and a newly developed HT-PEMFC.

The system requires a relatively long start-up period, making it suitable for continuous operation and stationary applications. Indeed, the whole start-up procedure, mainly consisting in reactors heating and catalyst activation, takes *ca*. 3.5 h. The operation is fully controlled and emergency shut-down protocols are also implemented to avoid safety issues and to prevent catalyst damaging.

We may underline that the presently adopted layout is less compact than possible commercial units, due to the need of gas sampling and internal inspection during the testing campaign. A picture of the fuel processor is reported in Fig. 2.

The fuel processor presently operated under fixed reaction conditions only. At 100% capacity the hydrogen production to feed a 5 kW_e PEM-FC is *ca*. 6.5 Nm³/h H₂. This requires 44 and 98 g/min as feeding flow rates of 96 vol% ethanol and of demineralised water, respectively. Based on these values, the total system efficiency under the selected operating conditions would be *ca*. 22.5 %, calculated on electrical power output only and according to ethanol LHV and it could be further optimised. This result is much better than 17.5 % reported in the literature for a smaller system (250 W), with a similar layout [27] and it should be compared

with the simulated 35% net efficiency, but based on ethanol HHV, estimated by Francesconi et al. [36].

4 - CONCLUSIONS

A demonstrative project is active c/o Università degli Studi di Milano to check the reliability of a bioethanol reforming-WGS-(methanation)-PEMFC integrated system. The power size is suitable for small residential applications (5 kW_e + 5 kW_t). The current experimentation on the fuel processor showed that the reformate quality is suitable to feed low temperature PEMFC (CO concentration after the second methanation step <20 ppmv). If a high temperature PEMFC stack is used, a much more compact and simpler layout may be foreseen, avoiding any final methanation steps. The relatively long start up period of the fuel processor makes it suitable for stationary continuous operation only.

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Table 1: Products distribution (vol%) after every reactor on a dry basis. No significant trace of other by-products was ever detected. Ethanol conversion = 100% after the first reactor.

Compound	Prereformer	Reformer	HT-WGS	LT-WGS
H ₂	59	73	73	74
СО	1	10	1.5	0.4
CO ₂	21	17	24	24.4
CH ₄	19	-	1.5	1.2

Fig. 1: Schematic flowsheet of the Fuel processor. Heat recovery not shown.

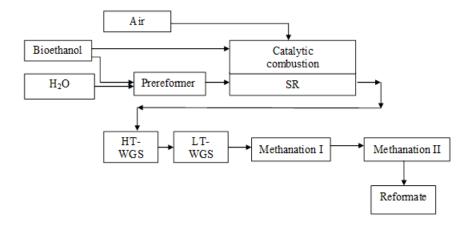


Fig. 2: Picture of the fuel processor (external FC unit not included). Size in cm: 70 x 100 x 180.

