PEMBeyond

PEMFC system and low-grade bioethanol processor unit development for back-up and off-grid power applications

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Deliverable 5.7

Report on the assembled PSA prototype

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Summary

The present report summarizes the assembly and the initial testing of the pressure swing adsorption (PSA) unit GUS B03 designed by HyGear under the collaboration protocol with UPorto.

The unit was shipped by HyGear to VTT research facilities, where the initial tests were performed under supervision of UPorto (Frederico Relvas) with VTT collaboration (Pauli Koski). A feeding system was assembled to supply a synthetic reformate gas mixture to the PSA; both the feed mixture and the PSA product were analyzed by GCs.

Additionally to Hygear PSA design, a vacuum pump was added to the off-gas line for improving the performance of the separation unit allowing also vacuum swing adsorption (VPSA) operation mode.

The best performing test was obtained operating in VPSA mode, a CO concentration of 4.9 ppm and a recovery of 69.4 % were attained. Operating in PSA mode, 6.8 ppm of CO with a recovery of 68.0 % were reached. To meet the project targets, further optimization is needed.

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Contents

1. Objectives

The purpose of the current document is to report the installation and testing of the PSA unit *GUS B03,* designed by HyGear, under the collaboration protocol with UPorto. The designed PSA aimed at to obtain a hydrogen stream with CO concentration between 20 and 50 ppm with a H_2 recovery > 75 %. A new target for CO concentration, 5 ppm, was set at the midterm assessment, for the same recovery. The goal is to decrease the CO poisoning of the anode catalyst of the fuel cell stack, based on platinum. A vacuum pump was added in the off-gas line to help in the adsorbent regeneration, thus allowing operating also in vacuum pressure swing adsorption mode (VPSA).

2. Test station and PSA/VPSA unit

2.1 Test station

A test station was assembled according to **Figure 1**. The PSA unit comprised 4 columns filled with a pelleted commercial activated carbon. The process lines were built with 10 mm Swagelok stainless steel piping and a 20-liter tank was placed at the PSA feed line to minimize pressure fluctuations. A synthetic reformate gas mixture was prepared and used to supply the PSA unit; theH_2 and CO_2 gases were supplied from the gas grid of VTT while CH_4 and the CO were supplied from a cylinder mixture of 80 % of CH_4 and 20 % of CO. Each feed flowrate was controlled using a Bronkhorst mass flow controller, previously calibrated. Additional H_2 and N_2 lines were also connected to the PSA purge lines. A vacuum pump GD Thomas 118ZC20/24 was placed in the PSA off-gas; this pump can improve the PSA performance and allows operating in VPSA mode. Temperature and pressure in the test bench were monitored using K-type thermocouples and Sensortechnics CTE9000-series pressure sensors, respectively, both in feed and product line.

A gas chromatograph *Agilent 490* Micro GC was used to analyze the feed gas, while a *Hewlett Packard 58090* equipped with a methanizer and a FID detector was used to analyze the product composition.

More detailed specifications of the test station are presented in deliverable D7.1 (Report from the initial testing of the system)

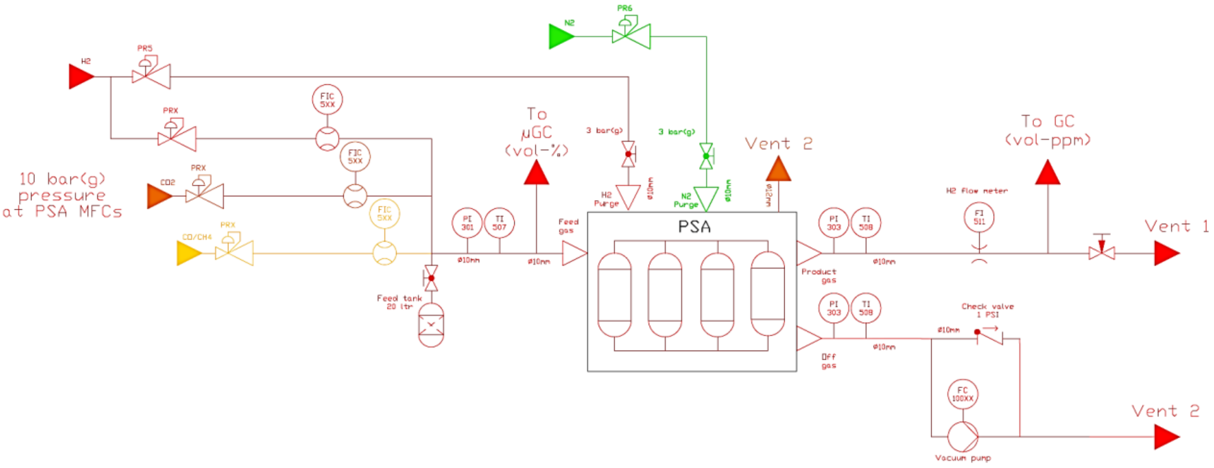


Figure 1 – PSA test station piping and the instrumentation diagram.

2.2 PSA/VPSA unit operation

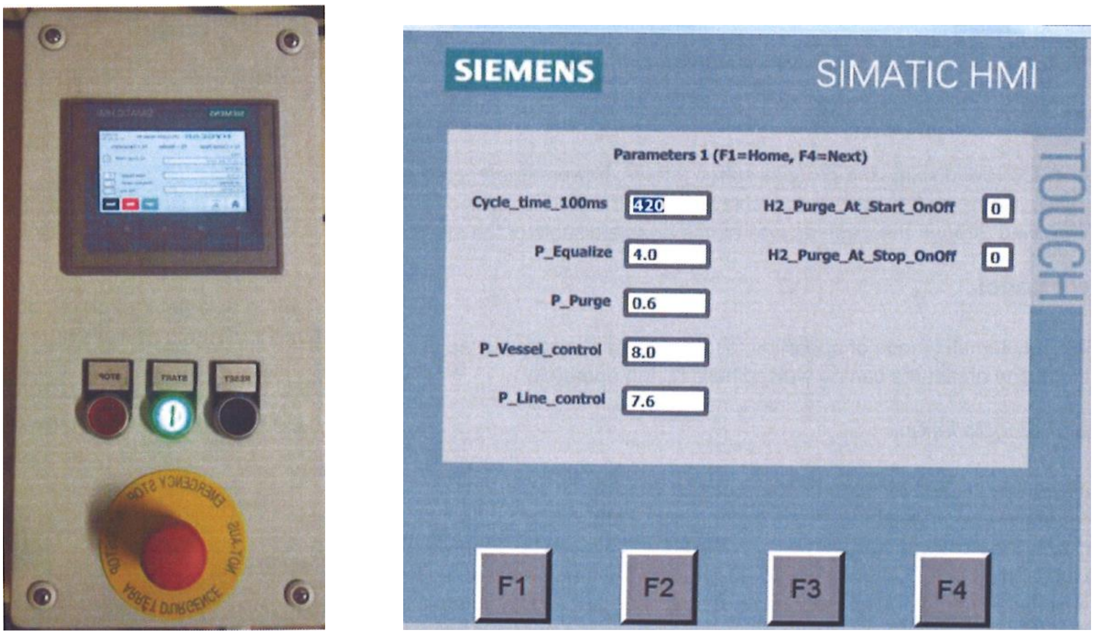
In PSA/VPSA operation neither the feed flowrate or product flowrate are directly controlled. Instead, the unit has a set of proportional valves whose opening fraction is adjusted according to the pressure set-point. The PSA unit is equipped with a *Siemens* HMI display panel, used as interface for controlling and monitoring the system. Up to sixteen parameters, described in **Table 1**, are accessible and can be changed according to the desired set-point; a shorter nomenclature was adopted for the main parameters as presented in the same table. Four additional pushbuttons (*Reset*, *Start*, *Stop* and *Emergency Stop*) are located below the display panel for simple operations, as shown in **Figure 2A**.

Table 1 – Accessible PSA parameters at the HMI display panel.

Parameter		Units	Description
$\boldsymbol{P\_Vessel\_control}$	P	bar	Setpoint of feed pressure during pressure up
$\boldsymbol{P\_Equalize}$	P_{eq}	bar	Setpoint for end of pressure equalization step
$\boldsymbol{P\_Purge}$	P_{purge}	bar	Setpoint for end of purge step
$\boldsymbol{P\_Line\_control}$	P_{line}	bar	Setpoint of line pressure to start production
$\boldsymbol{Cycle\_time\_100ms}$	t	ms	PSA cycle time
$\boldsymbol{H_2PurgeAtStart}$		0 / 1	H_2 purge at startup; off/on selector
$\boldsymbol{H_2PurgeAtStop}$		0 / 1	H_2 purge at stop; off/on selector
$\boldsymbol{ProductLineValve\_Gain}$		-	Product line valve PID controller setting

ProductLineValve_I	s	Product line valve PID controller setting
ProductLineValve_D	s	Product line valve PID controller setting
ProductValve_Gain	-	Product valve PID controller setting
ProductValve_I	s	Product valve PID controller setting
ProductValve_D	s	Product valve PID controller setting
FF_ProductLineValve	%	Preset product line valve for pressure smoothing
FF_ProductValve	%	Preset product valve for pressure smoothing

Figure 2B depicts one of the parameters tabs of the PSA HMI software; the parameters displayed in the figure are the default defined by HyGear.



A B

Figure 2 – A – Control panel, including HMI display panel, pushbuttons and emergency stop button; B - Parameters tab of the PSA HMI.

3. Experimental testing

Before the initial testing, the PSA was purged with N_2 to inert the system, in agreement with the recommendations described in the unit manual; a hydrogen purge was always performed at the end of each test for a faster startup in the next operation. The parameter P_{eq} was defined as half of the maximum P. P_{purge} was kept as defined by Hygear, 1.6 bar, as well as the PID parameters and the presets for pressure smoothing. The results of the experiments performed are depicted in **Table *2***; the feed column regards the feed flowrate to the buffer tank placed prior to PSA.

Table 2 – Results and parameters of the PSA/VPSA experiments.

Test	Operation mode	Feed ln·min⁻¹	P bar	P_{line} bar	t s	CO ppm	CO_2 ppm	Rec %	Product ln·min⁻¹	Productivity[1] ln·kg⁻¹·cycle⁻¹
# **0**	PSA	50	7.0	6.6	42	-	-	-	-	-
# **1**	PSA	50	7.0	6.6	42	-	-	-	-	-
# **2**	PSA	50	9.0	8.6	42	6.8	13.0	68.0	23.8	15.0
# **3**	VPSA	50	9.0	8.6	42	0.9	0.8	63.7	22.3	14.7
# **4**	VPSA	50	7.0	6.6	42	56.8	26.4	72.9	25.5	16.0
# **5**	PSA	50	9.0	8.6	42	6.0	0.0	66.9	23.4	14.8
# **6**	VPSA	50	9.0	8.6	48	1.8	2.2	67.1	23.5	16.9
# **7**	VPSA	50	9.0	8.2	48	4.8	0.1	69.4	24.3	17.5
# **8**	VPSA	60	9.0	8.6	42	17.8	3.2	71.9	30.2	19.0
# **9**	PSA	60	9.0	8.6	42	117.1	5.3	73.8	31.0	19.5

The first run (**Test # 0**) was performed with pure N_2 to verify the correct operation of the unit. After one cycle, the system stopped, showing the warning message "*Equalization not reached*". After contacting HyGear it was explained that the valve system was designed for operating with H_2, a much faster diffusing gas, and indeed this problem was not observed whenever H_2 was used.

Before starting the tests with the synthetic mixture, the system was run with pure hydrogen (**Test # 1**). Several leakages were observed, both in the feed system and in the PSA unit itself. All leakages in the feed system were fixed while, upon evaluation, the PSA leaks were not. The leakage in the, the PSA unit were not considered significant and the possible correction would lead to the unit disassembling; a concentration of hydrogen of 1000 to 10 000 ppm was measured in the neighbouring of the PSA unit.

According to the presented results (Test # 2 to Test # 9), a pressure increase leads to a lower CO concentration, while the recovery and productivity decrease; the opposite trend was observed when the cycle time increases. For a higher feed flowrate, an increase in recovery and productivity as well as in CO content were observed. Operating in VPSA mode both recovery and CO decreased. In all experiments, the unit showed to be very effective to remove the carbon dioxide.

In **Tests # 2** and **# 5**, the PSA unit was tested using the default values defined by HyGear. Despite the CO concentration reaches 6 ppm and the recovery was lower than the contracted one (average of 67.5 % % *vs* 75 %). In **Test # 3** a vacuum pump was used for assisting in the blowdown and regeneration of the column beds, in the so-called VPSA operating mode. Despite the recovery decreasing to 63.7 %, a decrease in the CO concentration from 6.8 ppm to 0.8 ppm was achieved. The difference between the VPSA and PSA operating mode is even more evident in **Tests # 8** and **# 9**; operating in VPSA mode 17.8 ppm of CO concentration

[1] The amount of adsorbent was estimated considering 1.9 L per column and an adsorbent bulk density of 0.585 kg·L⁻¹.

and a recovery of 71.9 % where obtained, **Test # 8**, while in PSA mode the CO concentration was 117.1 ppm for a recovery of 73.8 %, **Test # 9**.

Considering the new consortium target of 5 ppm of CO, the best performing parameters correspond to Test # 7, with an average CO concentration of 4.8 ppm, a recovery of 69.4 % for a product flowrate of 24.3 L·min^{-1}.

4. Conclusions

The PSA unit was assembled and tested. The best results were obtained in VPSA operation mode, with a recovery of 69.4 % and a CO content of 4.8 ppm. None of the operating conditions tested was able to meet the targets of the PEMBeyond project.

To meet the requirements of project and the new targets of the consortium, the PSA unit should be further optimized. Accordingly, it is recommended to optimize the PSA parameters to meet required specifications: >75 % recovery; < 20 ppm (DoW target) or < 5 ppm of CO (new consortium target) and a product stream with a hydrogen concentration higher than 98 %. Since a vacuum pump was installed in the PSA unit, allowing a VPSA cycle, the purge quantity should be also adjusted, increasing the time to evacuate the column, increasing the recovery. The operating variables that will be used for optimizing the VPSA unit are: vessel pressure (which should be the highest possible and close to 10 bar), cycle time and purge time (and consequently evacuation time). Although the product flowrate cannot be directly manipulated, it can be optimized changing product line pressure. The product line pressure, however, can only be changed until reaching the hydrogen storage tank pressure. The optimization results will be presented in future documents.