

HYGRID

FLEXIBLE HYBRID SEPARATION SYSTEM FOR H₂ RECOVERY FROM NG GRIDS

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1. EXECUTIVE SUMMARY

1.1. Description of the deliverable content and purpose

The HyGrid system is planned to extract hydrogen from a mixture of natural gas and hydrogen, combining a Pd membrane module, Electrochemical Hydrogen Purification (EHP) and Temperature Swing Adsorption (TSA). The development, engineering and assembly of the EHP sub-system are described in this deliverable. The EHP sub-system will extract the remaining hydrogen from the retentate gas flow coming from the Pd membrane module. HyET is responsible for this part of the project and has cooperated with HyGear for smooth integration of the EHP sub-system later on in the HyGrid system and the operation of the complete system.

1.2. Brief description of the state of the art and the innovation brought

The main traditional separation systems for hydrogen recovery from a hydrogen-methane stream are Pressure Swing Adsorption (PSA) units and cryogenic systems. The electric power consumption of both techniques is high due to the PSA compression step and the low sub-zero temperatures for cryogenic separation. The HyGrid project has the purpose to separate the hydrogen with lower electric consumption and cheaper capital costs, by using membrane technology.

HyET has built systems in the past for compressing pure hydrogen up to 875 bar and up to a flow rate of 120 kg H₂/day or 56 Nm³/h. Systems for both hydrogen extraction and purification have only been built by HyET up to 10 Nm³/hr. The HyGrid challenge for HyET was 1) to design the EHP sub-system for 85 Nm³/h and 2) at a low pressure drop, preferably below 100 mbar.

2. System overview and specifications

Electrochemical performance: The EHP sub-system has been designed to extract the remaining H₂ that is still present in the mixture of natural gas and H₂ coming from the Pd membrane module retentate outlet. The retentate concentration of the residual H₂ is expected to be around 6%. The targets for the EHP stacks are to operate at an electrical energy demand of 4 kWh/kg H₂ and extract at least 60% of the remaining H₂. The whole EHP sub-system should operate at an energy demand of 5 kWh/kg H₂.

The target set for the EHP sub-system is to extract 65 mol H₂/h or 3.1 kg H₂/day.

Operation parameters: The feed gas has a flow rate of 85 Nm³/h, and the operating pressure is 8 bar(g). The operating temperature is at nominal 35 °C and can be increased to 65 °C. The EHP stacks will extract the hydrogen, but they are also capable of simultaneously compressing the hydrogen to 400 bar. This was not needed for the HyGrid project, so the hydrogen pressure is delivered at 15 bar(g) at maximum.

3D dimensions: The skid has been fitted in a shipping container as housing for the HyGrid system. At an early stage in the project, it was determined that the skid should not be wider or higher than the opening of a shipping container (roughly 2.3 by 2.0 m) and could be 1.5 m deep. The system should be able to be transported to and from HyGear with a forklift truck.

Process integration: The feed gas mixture coming from the Pd membrane module is at a few hundred degrees. The gas cooling and conditioning system to cool down the gas to the operating temperature of the EHP stack while simultaneously humidifying the feed gas mixtures has been built by HyGear as part of the complete HyGrid system.

3. System design and engineering

A first version of the P&ID was set up, based on the process flow diagram (PFD) and flow sheets set up by HyGear. The philosophy of the EHP sub-system safety discussed between HyGear and HyET and agreed upon is that it should be safe autonomously and not rely on safety measures also implemented in the rest of the HyGrid system built by HyGear. This decision increased costs as some additional components are needed, but will prevent complicated discussions and delays in the unfortunate case damage would be caused from one part of the system to another part of the system.

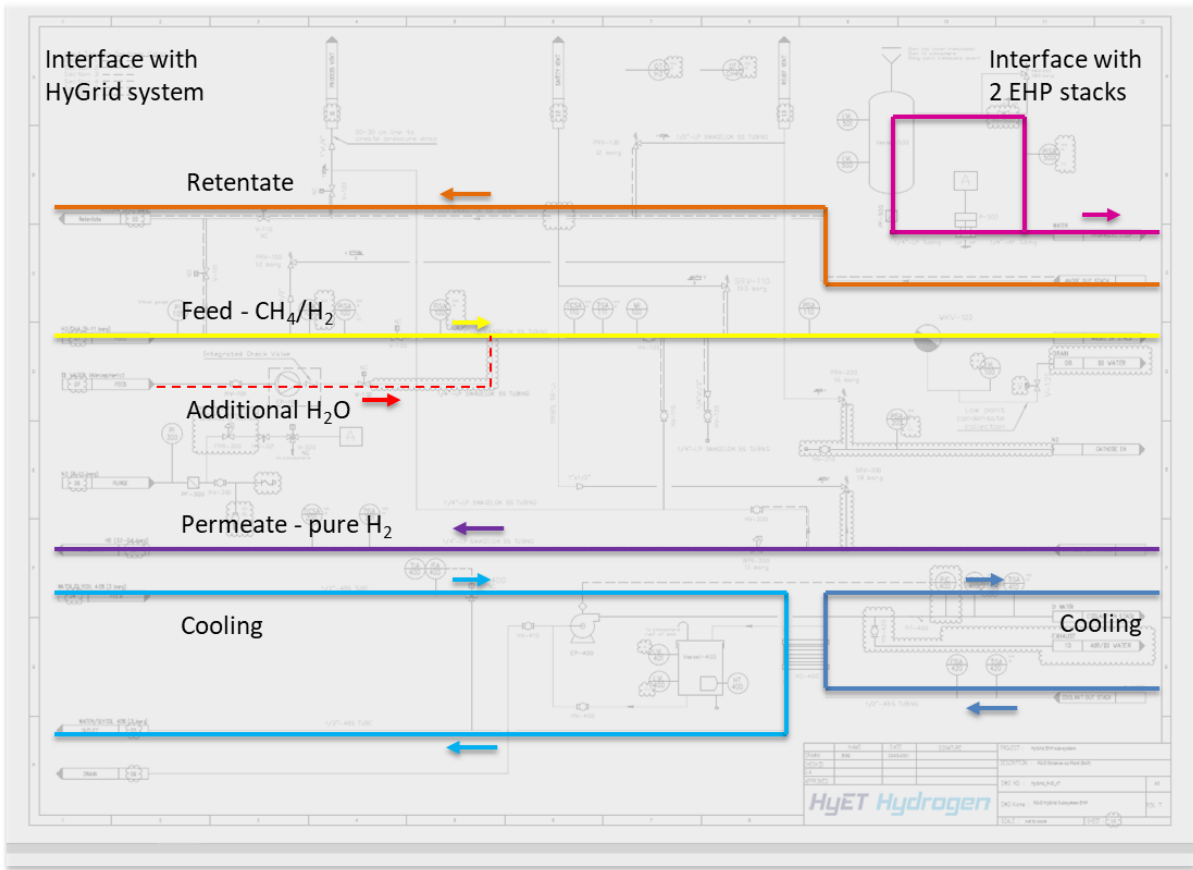


Figure 1. P&ID (main sheet) of the EHP sub-system. Separate sections are coloured.

3.1 Piping and Instrumentation Diagram (P&ID)

The main sheet of the resulting P&ID is shown in Figure 1. The lines in this figure have been coloured to indicate the different sections. Two sections are discussed and explained in more detail.

HyGear has offered to use the cooling loop present at their premises (light blue lines). This cooling load is used to cool the EHP stacks during operation. The EHP stacks can only be cooled

with demin water as a coolant so a secondary circuit is needed with a heat exchanger (dark blue lines). In the case of malfunction of the EHP stacks, the feed side of the EHP sub-system can be immediately rerouted to the retentate side, bypassing the EHP stacks. For maintenance purposes, the whole system needs to be purged manually with nitrogen before sections can be opened (green lines). No hydrogen or methane/hydrogen mixture must be presented when performing any maintenance.

3.1.1 Hydraulic circuit required for EHP stack gas sealing

The EHP stack design that has been chosen requires hydraulic pressure internally to ensure leak tightness. For this, a hydraulic circuit has been added to the P&ID (pink lines). This circuit is pressurized by a hydraulic pump which is powered by the pneumatic pressure of the nitrogen line. This nitrogen is supplied to the EHP sub-system by HyGear as a utility.

3.1.2 Dosing pump for feed gas humidification

The feed gas of the EHP stacks enters the EHP sub-system fully humidified with a dew point temperature close to the operating temperature of the EHP stacks. From the testing with small multicell EHP stacks, it turned out that more humidification would be needed to increase the performance and reach the targets set in this project for the EHP. Because the gas stream was already fully humidified by water vapour, it was decided to inject liquid water droplets by a digitally controlled dosing pump (red dotted line).

The detailed engineering process of the EHP sub-system had already started, but it turned out that there was sufficient space to add a dosing pump for demin water.

3.2 Interfacing with complete HyGrid system

During the detailed engineering phase, HyGear and HyET have been in close contact to define the number of interfaces between the EHP sub-system and the HyGrid system. Interfaces are all connections, electrically and mechanically, between the systems. The location, tubing size, and connection type have been documented as well as operating temperatures, pressures, flow rates and fluid composition.

Furthermore, ATEX zoning has been discussed with HyGear. HyGear will apply ventilation in the shipping container needed to comply with an ATEX zone 2NE. If the EHP sub-system would leak, the failure mode of the sealings has been identified together with the substance leaking and the release rate has been calculated. This result has been communicated with HyGear.

The EHP sub-system once placed at HyGear will be operated as part of the whole HyGrid system and the control system of the EHP sub-system will work as a slave to the control system

of the whole HyGrid system. For this, HyET and HyGear have agreed to use Modbus communication over TCP and HyET has defined the exact protocol and signals that are needed to be exchanged. HyGear and HyET also agreed that during the commissioning phase, HyET can make changes to the control software to optimize, for example, the efficiency of the system. During operation, HyET can read out the logging data generated by the EHP sub-system in real-time for early analysis. In that phase, HyET cannot change any parameter.

During the finishing of the assembly in December 2020 and January 2021, the EHP sub-system has been placed temporarily in the complete HyGrid system at HyGear for about a month to prepare all mechanical and electrical connections from and to the EHP sub-system. This helped to speed up the final installation of the EHP sub-system later on.

3.3 Safety assessment and certification

An important aspect of system engineering is the hazard and operability (HAZOP) study of the system. First, an analysis was performed on a preliminary version of the P&ID. Later on, the risks and operational issues have been formulated and mitigating actions have been defined for the final P&ID in four different sessions with a team of specialists at HyET. The measures needed are implemented for safe operation within the scope of what needs to be tested for this project.

The analysis has been updated continuously with the changes and additions to the EHP sub-system.

One of the aspects from the HAZOP that has been discussed with TU/e and HyGear is the risk of CO or CO₂ being present in the gas that will be used in the tests. Without proper measures, the performance of the EHP cells inside the stacks would be affected within minutes due to catalyst poisoning. This appeared to be no issue.

The sensors for the detection of methane and hydrogen gas released to the environment of the shipping container had not been selected yet in the second period. HyET has put effort into sourcing two separate gas sensors that are selective for hydrogen and methane, the flammable substances inside the EHP sub-system. The selected sensors do not show cross-sensitivity, which means that the detection of methane is very small in the hydrogen sensor, and *vice versa*.

An important aspect needed for the CE certification of the EHP sub-system is a manual describing the installation, operation and maintenance of the system. HyGear and HyET have discussed what documents on the safety assessment need to be added as appendices to the manual. This manual is shared by HyET with HyGear and discussed between them where

necessary. Because of the R&D nature of the whole HyGrid system, the experiments of the EHP sub-system were done with the constant supervision of both employees of HyGear and HyET.

3.4 Implementation

To implement layers of safety identified in the HAZOP, components have been added to the P&ID. Based on that revision of the P&ID, the first version of the Bill of Materials (BoM) has been created. From the systems built in the past, HyET has gained experience on which components work best in their systems. The BoM is easily populated with these more standard items.

However, as this is the first system for HyET for extraction of hydrogen out of gas mixture at a flow rate higher than 10 Nm³/hr, some attention was needed to new types of components that are able to process the higher gas flow.

From the BoM, the sourcing of components has been done. Most of the larger components and equipment had the longest lead time, so sourcing of these components had priority. The components sourced at first were the heat exchanger, pump, and control valve, all located in the cooling circuit. Besides those, special attention was given to the actuated valves in the feed and retentate section of the EHP sub-system. All components in these sections should not induce a large pressure drop by a narrow opening functioning effectively as an orifice. Therefore, ball valves have been selected which have a large opening.

Most systems of HyET built so far, do not have an ATEX zone 2NE but zone 2. This implies that all components with some kind of electrical connection need to be certified for the ATEX directive. For the EHP sub-system, this is not necessary which has led to better availability of components, lower prices and shorter lead times in general.

In this EHP sub-system, a few novelties on system engineering have been implemented that are described in the following sub-sections.

3.4.1 Frame for easy placement of EHP stacks

The skid of the EHP sub-system consists of a large steel frame with a smaller frame inside it for the EHP stacks. This smaller stack frame is designed in such a way that both EHP stacks can be removed easily for maintenance.

The two EHP stacks can be placed next to each other in this frame.



Figure 2. EHP stack frame for easy placement of stacks in the skid of the EHP sub-system.

3.4.2 Monitoring cell potentials

The electrical potentials of all cells in the two EHP stacks need to be monitored to prevent that the cell potentials can reach levels of about 0.8 V which, when applied during long periods of time, could damage the catalyst. HyET has developed its own cell voltage monitoring system (CVM), based on the learnings of the MEMPHYS project (GA 735533). The system can communicate all cell voltages to the central PLC over a CAN network, multiple times per second. The CVM box is shown in the middle at the bottom of the stack frame in Figure 2.

3.4.3 Heat tracing

The tubing on the stack frame needs to be heat traced to prevent water condensation from the humidified gas streams. As can be seen in Figure 2, the tubing consists of many branches to supply each package of 24 cells in the EHP stacks of the feed gas and cooling liquid and to remove the permeate (cathode outlet) and retentate (anode outlet) gas streams from both stacks. There are in total 72 branches in the stack frame.

HyET has used self-limiting parallel heat tracing in the past and that is also applied to the tubing in the main frame. One disadvantage of parallel heat tracing is the limited and inhomogeneous flexibility, which is needed with that many branches. HyET has decided to use serial heat tracing instead for this frame. That heat tracing is much more flexible. The exact length of the sections has been determined and based on that the specific ohmic resistance of the serial heat tracing is selected that leads to an average power of 30 W/m.

3.4.4. Modular PLC software for fast development

HyET has developed its own method to program the PLC in a modular form. This development was outside the scope of the HyGrid project but has led to significant time reduction for

programming the PLC because most modules were already developed. Only the communication with some sensors that have not been used in one of HyET's systems in the past, has been added to the control software. Also, the external cooling circuit that HyGear supplies needs to be controlled and had to be programmed from scratch.

For this system, a custom-made Graphical User Interface (GUI) has been set up to check and control a few parameters. The main screen of that GUI is shown in Figure 3.

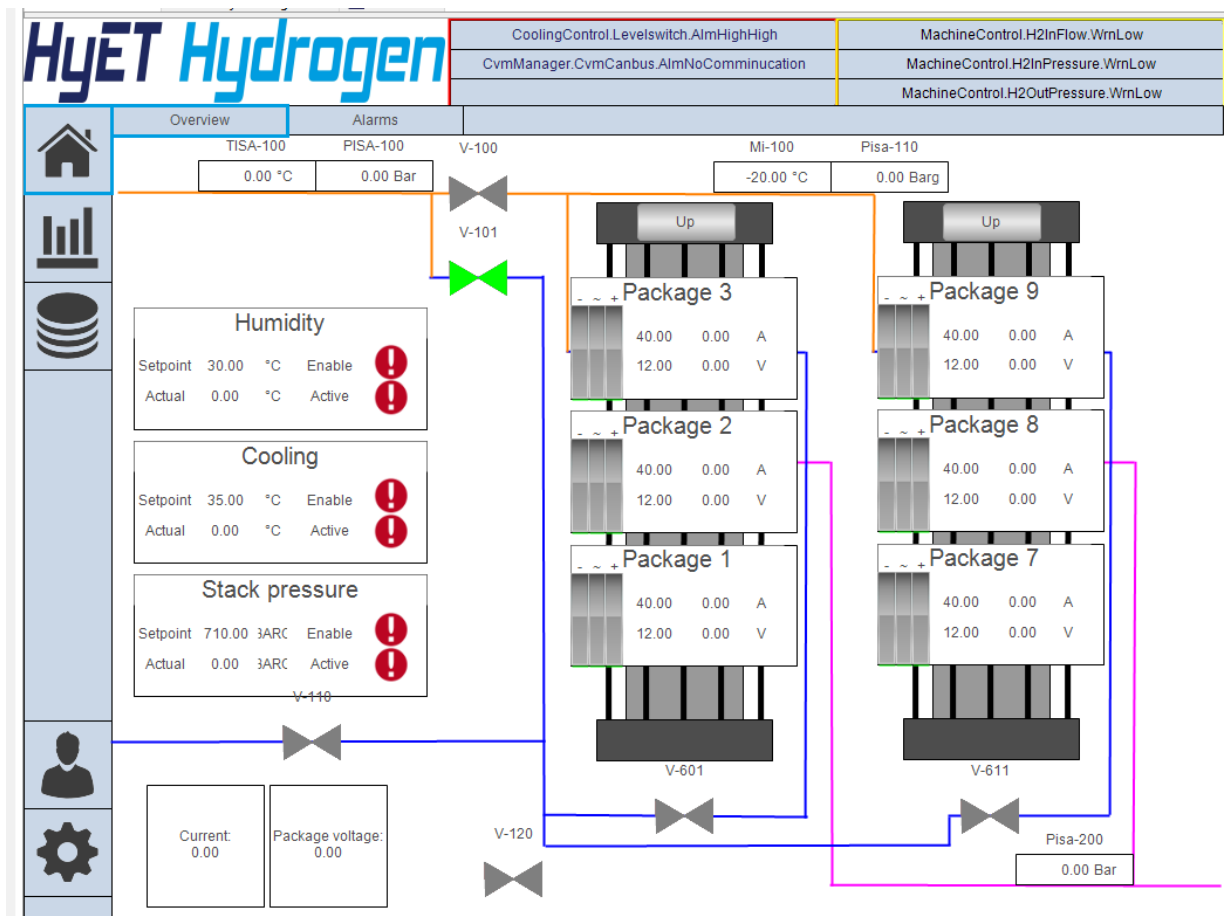


Figure 3. Main screen of GUI to check and set value on the PLC control system.

4. Assembly and commissioning

The EHP stacks have been developed from the start of the project by HyET. Two full-size EHP stacks consisting of in total of 288 cells have been assembled for the EHP sub-system. These stacks have been tested on leak tightness and performance with pure hydrogen only in HyET's standard Factory Acceptance Test (FAT) protocols. The test results were within the required ranges.

The two full-size EHP stacks have been placed in a dedicated stack frame (see Figure 2 on the right) and all 76 mechanical connections and all 324 electrical connections to the stacks have been made and checked. This dedicated stack frame reduced the mechanical connections to be made in the main frame to only 7. After this, the stack frame was ready to be placed in the main frame of the EHP sub-system.

After the assembly of the main equipment and connecting the tubing, the work focused on minor items, initial testing and solving small issues that were found during commissioning. Besides that, the PLC control software of the EHP sub-system has been implemented and tested thoroughly. After assembly of the EHP sub-system was completed and during a preliminary Factory Acceptance Test (FAT) a variety of small issues have been found, both mechanical as electrical. They have been resolved prior to the shipment of the EHP sub-system to HyGear.

On the electrical and software side, testing continued on an I/O check. In the control software, different machine states have been defined like 'Starting' and 'Running'. Switching between the machine states have been tested. Besides that, control loops for heating and cooling the EHP stacks, pressurization of the hydraulic circuit and the heat tracing of the tubing have been tested.

The assembly and testing were finished at the end of February 2021 and the system was transported from HyET to HyGear in April 2021, see Figure 4. On delivery, the electrical safety circuit integration between the EHP sub-system and the HyGrid system has been tested. Operational testing of the complete HyGrid system, including the EHP sub-system started in August 2021 and finished in that month. Testing and test results are described in a non-public report.



Figure 4. Final EHP sub-system integrated into the container of HyGrid system at HyGear.

5. Conclusions

The EHP sub-system has been engineered, built and commissioned with HyET as the leading partner. This custom-made system is capable of processing the 85 Nm³/h of a natural gas / hydrogen mixture and at an operating pressure of 8 bar(g) and an operating temperature of 35°C. The expected extraction rate of the hydrogen out of the natural gas is at least 65 mol H₂/h or 3.1 kg H₂/day.

Two stacks, consisting of in total 12 packages with 24 cells per package that can be operated individually, have learned HyET about how to operate each package at it is optimal condition.

The system is made ready for integration at HyGear in the HyGrid system and has been delivered there in April 2021.

As planned, HyGear operates the complete HyGrid system and test results are described in a non-public report.