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Designing an alkaline water electrolysis test bench

School of Technology and Innovations
Master's Thesis in Energy Technology
Master's Programme in Energy and Information Technology

Vaasa 2024

UNIVERSITY OF VAASA
School of Technology and Innovations

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Title of thesis:	Designing an alkaline water electrolysis test bench
Degree:	Master of Science in Technology
Programme:	Energy Technology
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Year:	2024 **Pages:** 76

ABSTRACT:

In this study, a design of an alkaline water electrolysis test bench is made for VTT. This design work includes making a complete process and instrumentation (P&I) diagram of the test bench, compiling two different safety analyses of the test bench, and finally choosing the necessary instruments according to both the safety analyses and the P&I diagram. Due to confidentiality reasons, the complete P&I diagram of the test bench is not provided in this study.

The purpose of this study is to strengthen Finnish know-how in alkaline electrolysis, as Finland currently lacks extensive practical know-how of this technology at an industrial scale. Utilization of the test bench offers an opportunity for interested companies to be the technology's forerunners, providing agreed research data and educating competent personnel. Interested companies can also utilize the test bench for stack testing and development purposes.

This study is in the style of design research, which also partly involves some empirical research. The study presents the working principle of alkaline electrolysis, and the challenges encountered during the design work of the test bench. Literature and a previous P&I diagram from VTT's earlier test bench have been used as help in the P&I diagram design. Empirical research has been used to illustrate the separation process of the two-phase flow to be able to evaluate the behavior and the amount of unwanted gas (H_2 and O_2) bubbles in real operating conditions. The safety analyzes performed during the design work have been made and documented according to the standards EN IEC 60079-10-1:2021, EN IEC 61511-1:2017 and EN IEC 61511-2:2017. The standards give instructions for classifying potentially explosive zones and how to ensure functional safety for the test bench.

After this study, VTT has a straightforward plan on how to implement an alkaline electrolysis test bench, and what kind of modifications would be needed in different future situations. Moreover, this thesis work will provide VTT a complete list of selected and necessary instruments, and competent personnel to develop the test bench further. The study provides an overview of the measures still needed, after which the assembly and commissioning of the test bench can be started in the upcoming months.

KEYWORDS: hydrogen, electrolysis, P&I diagrams, design research

VAASAN YLIOPISTO
School of Technology and Innovations

Tekijä:	Ilari Laakso
Tutkielman nimi:	Designing an alkaline water electrolysis test bench
Tutkinto:	Diplomi-insinööri
Oppiaine:	Energiatekniikka
Työn valvoja:	Anne Mäkiranta
Työn ohjaajat:	Farhan Ali, Maarit Mäkelä ja Olli Himanen
Valmistumisvuosi:	2024 **Sivumäärä:** 76

TIIVISTELMÄ:

Tutkielmassa suunnitellaan alkalielektrolyysin testipenkki VTT:lle. Suunnittelutyö sisältää täydellisen prosessi- ja instrumentaatiokaavion (PI-kaavio) teon, kahden turvallisuusanalyysin laatimisen testipenkkiä varten, ja tarvittavien instrumenttien valinnan PI-kaavion ja turvallisuusanalyysien esittämien tarpeiden mukaisesti. Salassapitovelvollisuuden vuoksi täydellistä PI-kaaviota ei esitetä tässä tutkielmassa.

Tutkielman tarkoitus on vahvistaa suomalaista osaamista alkalielektrolyysin parissa, sillä tällä hetkellä Suomesta puuttuu laaja alkalielektrolyysin käytännönläheinen osaaminen teollisuusmittakaavassa. Testipenkin käyttö tarjoaa kiinnostuneille yrityksille mahdollisuuden olla alan teknologian edelläkävijöitä, tarjoamalla etukäteen sovittua tutkimusdataa ja kouluttamalla osaavaa henkilökuntaa. Kiinnostuneet yritykset voivat myös hyödyntää testipenkkiä elektrolyysikennoston testaamiseen ja kehittämiseen.

Tutkielma on kehittämistutkimuksen tyylinen, johon liittyy myös osittain kokeellista tutkimista. Tutkielmassa esitellään alkalielektrolyysin toimintatapa, ja kyseisen teknologian testipenkin suunnitteluvaiheen haasteet. Apuna PI-kaavion suunnittelussa on käytetty kirjallisuudesta saatavaa tietoa ja yhtä VTT:n aikaisemman testipenkin PI-kaaviota. Kokeellista tutkimusta on hyödynnetty kaksifaasivirtauksen erotuksen havainnollistamisessa, jotta pystytään arvioimaan haitallisten vety- ja happikaasukuplien käyttäytymistä ja määrä oikeassa tilanteessa. Suunnittelutyön aikana tehdyt turvallisuusanalyysit ovat laadittu ja dokumentoitu standardien EN IEC 60079-10-1:2021, EN IEC 61511-1:2017 ja EN IEC 61511-2:2017 mukaisesti. Standardit ohjeistavat räjähdysvaarallisten tilojen luokitteluun ja toiminnallisen turvallisuuden varmistamiseen.

Tutkielman jälkeen VTT:llä on suoraviivainen suunnitelma, miten toteuttaa alkalielektrolyysin testipenkki, ja millaisia muokkauksia tämä tarvitsee tulevissa tilanteissa. Lisäksi tutkielma tarjoaa VTT:lle täydellisen listan valituista ja tarvittavista instrumenteista, ja teknologiaan perehtynyttä henkilöstöä kehittämään testipenkkiä pidemmälle. Tutkielma tarjoaa katsauksen vielä tarvittavista toimenpiteistä, joiden jälkeen testipenkin rakentamisen ja käyttöönoton voi aloittaa tulevina kuukausina.

AVAINSANAT: vety, elektrolyysi, PI-kaaviot, kehittämistutkimus

Contents

Figures

Tables

1 Introduction

Climate change has been an important topic for years now, but as of lately, we have started to see climate change's effect more clearly. The summer of 2023 was seen to be the hottest summer on record (NASA, 2023), while natural disasters, such as droughts and floodings have become more frequent. To reduce the speed of climate change, action would be needed as soon as possible. This is why we are constantly looking for new ways to produce sustainable energy, and ways to make our energy consumption more efficient.

One potential way is the use hydrogen to decarbonize energy sectors that would otherwise be hard to electrify, like heavy transport. Hydrogen could also decarbonize steel production and be a large contributor to Power-to-X sector coupling. Electricity demand and supply have to match at all times, meaning that sometimes we have to sell the excess electricity produced. Instead of selling all of the excess energy production, could some of this excess electricity be used to produce hydrogen via water electrolysis. This produced hydrogen could then be converted back to electricity, when the electricity demand is larger than the production. This way, hydrogen could be used as an energy storage, limiting our dependency on hydro power reserves.

Production of green hydrogen largely suffers from high electricity prices, and currently cannot compete with other hydrogen production methods, such as steam methane reforming (Office of Energy Efficiency & Renewable Energy, 2021). Green hydrogen is produced via water electrolysis, which can be done by many different methods. Alkaline water electrolysis (AWE) is the most mature technology for green hydrogen production, while proton exchange membrane electrolysis closely follows.

The aim for this thesis is to design an alkaline water electrolysis test bench for VTT, as Finland currently lacks practical alkaline electrolysis knowledge. The designed test bench is aimed to provide knowhow of the technology, while granting other companies the possibility to benefit from practical testing. The test bench provides a shortcut for Finnish companies to be the field’s forerunners, by educating competent personnel to help develop alkaline electrolysis even further.

In this study, four commonly known electrolysis methods are introduced, with alkaline water electrolysis being the main focus. The working principle of alkaline water electrolysis and some of the recent technology improvements are studied, to show why more research on these technologies is necessary.

Design work of the test bench includes designing the process and instrumentation (P&I) diagram, which for confidential reasons, is not fully shown in this study. The P&I diagram was modified several times as new design choices and ideas came up. Some of the test bench’s balance of plant components were also carefully designed, as they are not typical shelf components, and were either made inhouse, or done by a custom order.

During the design work, two different safety analyses were made to ensure the safe operation of our test bench. Safety analyses were done early to expose possible design flaws, as noticing them after the test bench has been in operation would cause unnecessary shutdowns and postpone the operation of the test bench. Safety analyses were a large part of the design work, as many changes were made to the P&I diagram, with safety concerns in mind.

2 Alkaline water electrolysis

This chapter summarizes the literature review, by starting with alkaline water electrolysis' history. Alkaline water electrolysis' working principle is introduced, together with some recent technology developments that have been made. Alkaline water electrolysis is compared to three other commonly known water electrolysis methods. Strengths and weaknesses of each technology are briefly studied, and the suitability of alkaline water electrolysis for renewable energy sources is discussed.

2.1 History of alkaline water electrolysis

There are four commonly known water electrolysis methods: alkaline water electrolysis (AWE), proton exchange membrane electrolysis (PEMEL), solid oxide electrolysis (SOE), and anion exchange membrane water electrolysis (AEMWE). Water electrolysis itself has been known for a long time, as hydrogen production with the help of electricity was first introduced by Adriaan Paets van Troostwijk and Jan Rudolph Deiman in 1789 (Smolinka et al., 2022, p. 1). In the year 1800, Anthony Carlisle and William Nicholson were also able to produce hydrogen from water by using Alessandro Volta's voltaic pile (Katz, 2021, p. 1). Even though electrolysis was discovered early, it took a long time to find practical applications for this newly found technology.

According to Smolinka et al. (2022, p. 1), in the 1900s, about 400 alkaline water electrolyzers were already up and running, with ammonia fertilizers being the most common use for hydrogen. They also mention that other uses for hydrogen were found in the same decades, as in the 1890s Charles Renard introduced hydrogen to be used in French military aviation. Water electrolysis lost its ground as the preferred way to produce hydrogen when steam reforming of methane was introduced as it was a much more cost-effective solution (Kumar & Lim, 2022, p. 3).

Out of the mentioned water electrolysis methods, AWE and PEMEL are more mature technologies and already commercially available, while SOE and AEMWE are still in the research and development phase (IRENA, 2020, p. 31). AWE was already in industrial use in the early 1900s, while PEMEL was developed and worked on in the 1960s, with the main aim to overcome the drawbacks of alkaline water electrolysis (Kumar & Lim, 2022, p. 10). Even though alkaline water electrolysis is widely known and the most mature electrolysis technology, there is still a drive and interest to develop it. Some of the most notable improvements from the recent decades include the zero-gap configuration and diaphragm material overhaul (David et al., 2019, p. 7).

2.2 The working principle of AWE

Every method of water electrolysis follows the same basic principle, where electrical current is used to split water molecules into hydrogen and oxygen, as can be seen in equation 1 (David et al., 2019, p. 3).

$$H_2O \rightarrow H_2 + \frac{1}{2}O_2 \tag{1}$$

However, in water electrolysis, pure water itself is not enough to uphold the process, as its electrical conductivity is not good enough (Phillips & Dunnill, 2016, p. 1). To improve electrical conductivity in the cell, electrolytes are used. The used electrolyte type and how it is applied depends heavily on the used electrolysis method. Notable differences can be seen when comparing the two most mature technologies, as in AWE the electrolyte is pre-mixed to water, before the electrolysis process is started, while in PEMEL, the conductive membrane of the cell works as an electrolyte (Kumar & Lim, 2022, p. 10). The electrolyte used in alkaline electrolysis is usually aqueous potassium hydroxide (KOH) typically at a concentration of 25–30 wt. % KOH (David et al., 2019, p. 4). Alternatively, aqueous sodium hydroxide (NaOH) at similar concentrations can be used (Colli et al., 2019, p. 2).

As in every electrolysis method, the chemical reaction of AWE is a bit more complex than indicated in equation 1. The reaction is divided into two parts, one reaction producing hydrogen, and the other producing oxygen.

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^- \tag{2}$$

$$2OH^- \rightarrow H_2O + \frac{1}{2}O_2 + 2e^- \tag{3}$$

Notably, the produced hydroxide ions in equation 2 are used to produce water and oxygen in equation 3. Equation 2 indicates reaction on the cathode, while equation 3 indicates reaction on the anode.

2.3 AWE cell design

In alkaline electrolysis, the chemical reactions take place inside stack cells. Stack is the main component of every water electrolysis plant, as it is the component upholding the chemical reaction, and is then responsible for hydrogen production. The number of cells inside a stack depends on the desired stack size and its characteristics. Each stack cell has two electrodes, the anode and the cathode. The stack cells are divided into two chambers, each containing one of the electrodes. The cell chambers are separated from each other by a thin diaphragm that prevents the produced gases from mixing, while still allowing hydroxide (OH^-) ions to pass through it (Miller et al., 2020 p. 3).

In AWE process, both cell chambers are filled with liquid electrolyte and an electric potential is applied between the electrodes. This electrical potential causes the chemical reactions of equations 2 and 3. It is worth noting that in alkaline water electrolysis, only water is consumed out of the electrolyte solution, increasing its KOH or NaOH concentration. To keep the electrolyte solution at a desired concentration level, the loss of water needs to be compensated by adding more water into the mix before recirculation.

2.3.1 Zero-gap design

AWE cell designs can follow two main pathways: finite gap design or zero-gap design. In finite gap cell design, the electrodes on each chamber are located on the sides of the cell (Figure 1a). The liquid electrolyte flows in between the electrodes and the diaphragm. Zero-gap design is demonstrated in Figure 1b. In the zero-gap design the electrolyte flows between the outer wall of the cell and the electrodes. Zero-gap design is an improved version of the more traditional finite gap design, which is why it is usually the preferred choice out of the two for new stack designs.

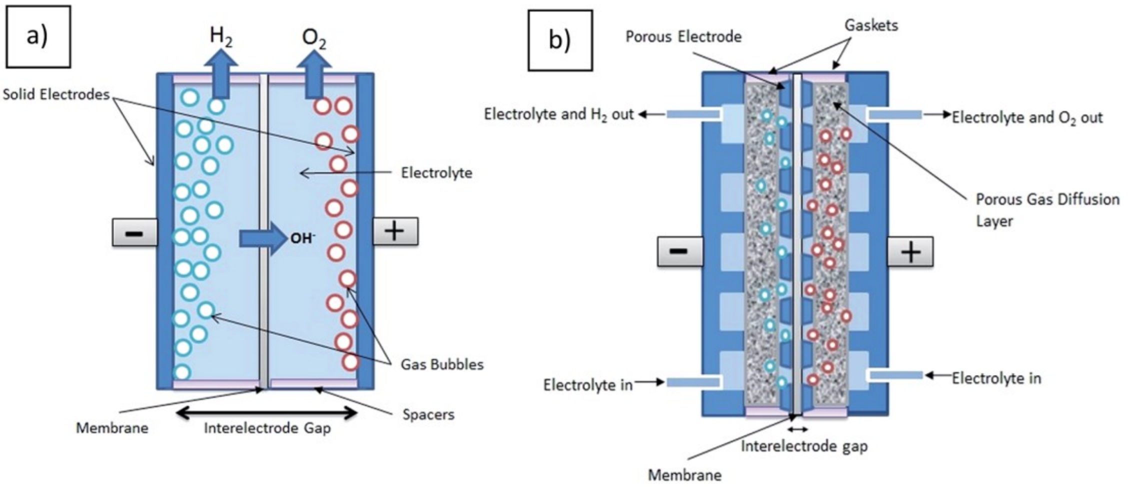


Figure 1. Differences in cell design, (a) finite gap cell design and (b) zero-gap cell design (Adapted from Phillips & Dunnill, 2016, *Zero gap alkaline electrolysis cell design for renewable energy storage as hydrogen gas*).

Zero-gap design reduces the ohmic losses of the finite gap design, allowing for higher current densities. Low current density is usually one of the main flaws of AWE (Phillips et al., 2017 p. 2). Higher current densities are reached by placing the electrodes closer together, only to be separated by the diaphragm. Placing the electrodes closer together greatly reduces the ohmic losses, as the ohmic resistance of the electrolyte will not play such a large role anymore (Phillips & Dunnill, 2016, p. 2). The voltage drop between the electrodes follows ohms law, where the importance of area specific ohmic resistance can be seen (Phillips et al., 2017, p. 3).

$$V = j \times R_{ohmic} \tag{4}$$

In equation 4, V is the voltage drop between the electrodes, j is the current density of the cell, and R_{ohmic} is the area specific ohmic resistance. According to Phillips et al. (2017, p. 3), if gas bubble formation is not considered, area specific ohmic resistance can be divided into area specific resistances for the cell diaphragm and the electrolyte, which is demonstrated in equation 5. They also mention that area specific resistance of the electrolyte can be furthermore divided into electrolyte resistivity, $\rho_{electrolyte}$ and the distance between electrodes l, as seen in equation 6.

$$R_{ohmic} = R_{diaphragm} + R_{electrolyte} \tag{5}$$

$$R_{electrolyte} = \rho_{electrolyte} \times l \tag{6}$$

When the electrolyte and its concentration have been chosen, can only the distance between the electrodes be optimized, to reduce the area specific ohmic losses in the electrolyte (Phillips et al., 2017, p. 3).

During the electrolysis process, gas bubbles are formed on each electrode, which then will have an impact on the electrolyte resistance. To overcome the difficulties of bubble formation, zero-gap design not only aims to minimize the distance between the electrodes, but also allows the removal of formed gas bubbles from the backside of the electrodes (Phillips et al., 2017, p. 3). In zero-gap cell designs the electrodes are usually porous, as seen in Figure 2. This is done to increase the active surface area of the electrode, allowing for higher geometric current densities, while at the same time it allows easier removal of the gas bubbles from the backside (Phillips & Dunnill, 2016, p. 5).

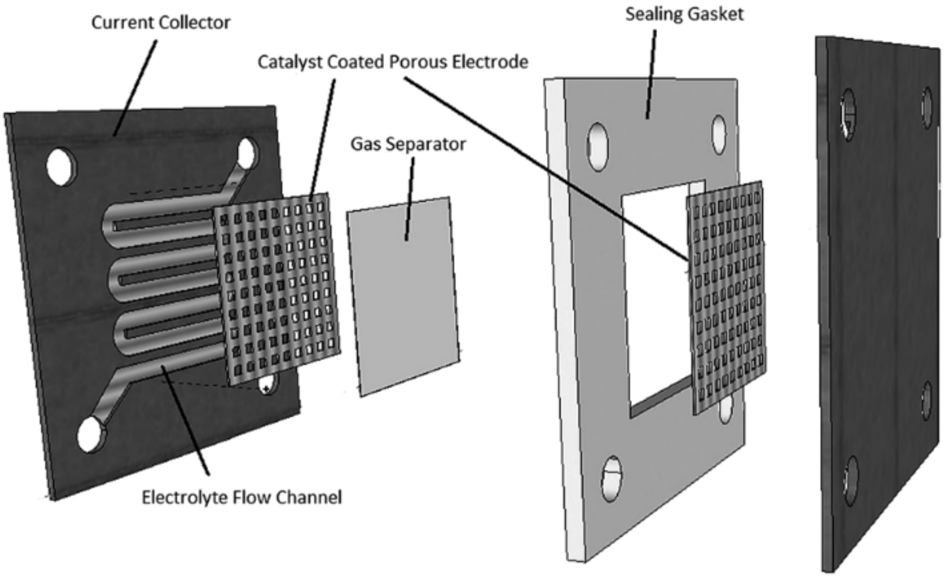


Figure 2. 3D model of a zero-gap cell (Phillips & Dunnill, 2016, *Zero gap alkaline electrolysis cell design for renewable energy storage as hydrogen gas*).

2.3.2 Diaphragm of the cell

Diaphragm is a crucial part of the AWE cell design, as it not only separates the product gases from each other, but also allows ions to pass through it. Previously AWE diaphragms were made of asbestos (David et al., 2019, p. 7), but as asbestos has been found harmful, and many countries have limited the use of asbestos, other design choices have been explored, such as Zirfon or nickel (Kumar & Lim, 2022, p. 4).

As seen in equation 5, the area specific resistance of the diaphragm is also a main contributor to the ohmic resistance of the cell. The thickness of the diaphragm is mostly responsible for its area specific resistance, and by thinning the diaphragm, conductivity can be improved (Ehlers et al., 2023 p. 4–5). However, the diaphragm cannot be thinned too much, as this will increase the rate of gas crossover.

One of the main topics of improved diaphragm designs is to allow higher temperatures in the stack. According to Ehlers et al. (2023, p. 3), higher temperatures would increase the overall efficiency of the stack, as the electrolyte solution conductivity increases with higher temperatures. They also mention that higher temperatures contribute to the water splitting process, as less energy is needed in the form of electricity. Increasing temperature also has its limits, as current commercial diaphragms can handle temperatures roughly up to 110 degrees Celsius, to avoid cell damage (Ehlers et al., 2023, p. 4). To find the ideal design for a diaphragm, it is important to find a balance between diaphragm characteristics, and its structural integrity. According to Ehlers et al. (2023, p. 4), diaphragm designs that would allow for increased cell temperature usually suffer either from poor hydrophilicity, reduced conductivity, or higher gas crossover rates.

Finding ways to improve diaphragm strength is not easy, as polymers that have good hydrophilic properties are usually vulnerable to the corrosive environment of the electrolyte (Ehlers et al., 2023, p. 4). According to Zhang et al. (2018, p. 3), the tensile strength of the diaphragm can be improved by reinforcing it with stronger additional materials, or by using improved cross-linking for the ion conducting polymers. They also mention that these methods decrease the conductivity of the diaphragm, as adding more material adds more mass to the diaphragm and cross-linking polymers lowers the polymer flexibility.

2.3.3 Electrodes

Catalysts are used in electrolysis to increase cell efficiency, as they lower the activation energy needed for chemical reactions. According to Phillips and Dunnill (2016, p. 5–6), in zero-gap alkaline cell designs, the catalysts can be directly applied to the electrodes, or to the diaphragm. They also mention that the zero-gap designs were firstly based on the more traditional setup, where electrodes were coated with the catalysts, and electrode designs were kept simple and affordable. Recently, new varieties for electrodes have been studied, such as carbon paper and nickel foam designs (Phillips & Dunnill, 2016, p. 5).

Electrodes in AWE are usually either nickel or steel, as there is no need for noble metals in non-acidic conditions. Catalyst coated nickel and stainless steel electrodes fulfill the most important features wanted from the electrodes: corrosion resistance, high catalytic activity, and high conductivity (Colli et al., 2019, p.2). According to Colli et al. (2019, p. 9), nickel electrodes with nickel-based catalysts, such as Raney nickel, have been found to have the best performance for hydrogen evolution reaction (HER). They also mention that other alloys, such as nickel-copper and nickel-cobalt, were tested and found to have promising catalytic results. On the oxygen evolution reaction (OER) side, stainless steel has been found to be formidable option for nickel as the electrode material. Cells with Raney nickel coated stainless steel anodes have been found to outperform cells with symmetric nickel electrodes in long term tests (Colli et al., 2019, p. 12).

2.4 Comparison of AWE with other electrolysis methods

Due to the liquid electrolyte and the diaphragm’s high ohmic losses, alkaline water electrolyzers suffer from lower current densities when compared against other water electrolysis technologies, such as PEMEL (Carmo et al., 2013, p. 4). According to Carmo et al. (2013, p.4), one of the reasons for PEMEL development in 1960s was in fact, to overcome the drawbacks of AWE. The main idea was to have a solid electrolyte, tackling the difficulties that liquid electrolyte brings. In the concept idealized by Thomas Grubb, solid sulfonated polystyrene membrane is used as an electrolyte (Carmo et al., 2013). As PEMEL does not use liquid electrolyte, the cell characteristics are not restricted by the strong base conditions of KOH or NaOH. Proton exchange membranes have many advantages when compared to the ones used in AWE, such as lower gas permeability, better conductivity, and lower thickness (Kumar & Himabindu, 2019, p. 4). Proton exchange membranes also differ from AWE’s membranes, as they transfer protons (H^+) instead of hydroxide ions. Advantages of having better conductivity and lower thickness eventually reduces the ohmic losses of the diaphragm, granting larger current densities in each cell. Anode and cathode reactions for PEMEL can be seen in equation 7 and 8. Equation 7 indicates the reaction on the anode, while equation 8 indicates reaction on the cathode.

$$H_2O \rightarrow 2H^+ + \frac{1}{2}O_2 + 2e^- \tag{7}$$

$$2H^+ + 2e^- \rightarrow H_2 \tag{8}$$

As a drawback, proton exchange membrane (PEM) electrolyzers contain more rare minerals, such as platinum and iridium metals (Hu et al., 2022, p. 2). Rare minerals are required due to highly acidic PEMEL operating conditions (Carmo et al., 2013, p. 5). Iridium and platinum are one of the rarest elements, and the demand for them is already high from other technologies. Iridium, for example, is used for smart device LEDs, and the addition of large-scale PEMEL could increase the already hefty price (Carmo et al., 2013, p. 5–6). An example of how much more platinum would be needed by hydrogen technologies to meet the net zero by 2050 scenario, can be seen in Figure 3. The chart indicates platinum demand to increase until 2040, when the total platinum demand is 0,2 kt (IEA, 2023).

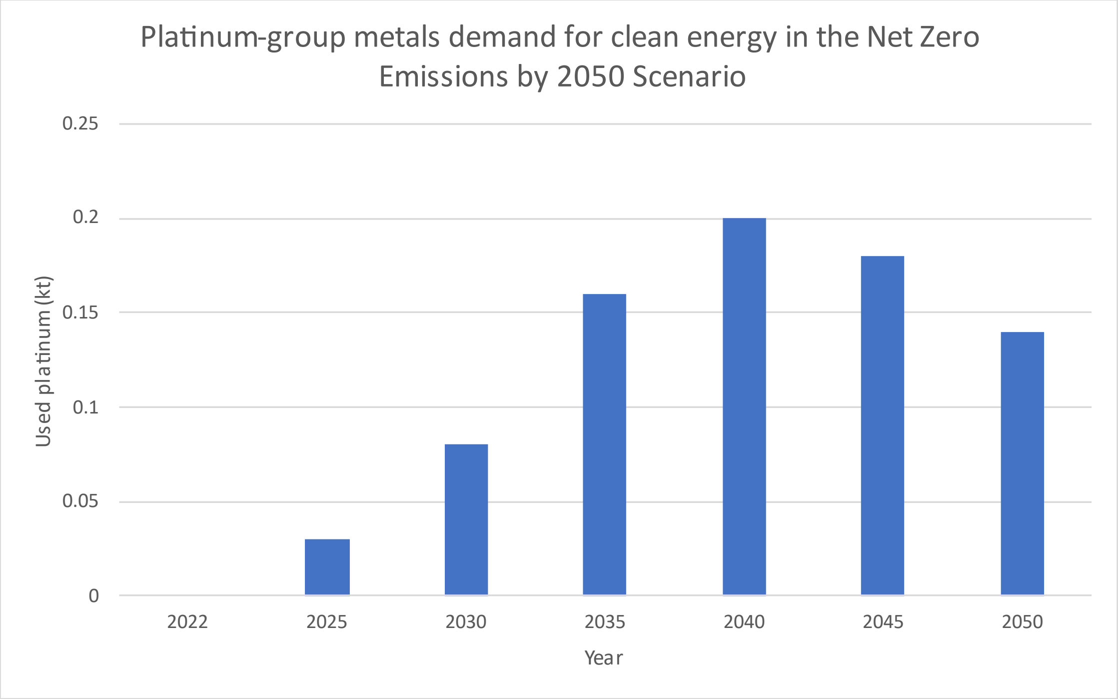


Figure 3. Growing platinum demand by hydrogen technologies if net zero by 2050 scenario is to be met. (Adapted from International Energy Agency, 2023, *Critical Minerals Data Explorer*).

Alkaline water electrolyzers have been improved since the days PEMEL was developed, but still there are the same drawbacks when compared to PEMEL. As PEMEL also has its drawbacks, therefore, anion exchange membrane water electrolysis is developed, to combine the advantages of these two technologies (Tee et al., 2022, p. 2). AEMWE somewhat resembles PEMEL, as it has a thin non-porous membrane. Anion exchange membrane, however, transfers hydroxide ions as AWE does, instead of protons (H^+) like proton exchange membrane does (Henkensmeier et al., 2020, p. 2). AEMWE can reach high current densities, like PEMEL, by having a thin non-porous membrane, while avoiding the acidic conditions of PEMEL. According to Henkensmeier et al. (2020, p. 2), this is done by using both alkaline media and a solid electrolyte, increasing the pH value of the system. They also mention that by getting rid of the acidic conditions, there is no need to use platinum on the electrodes or titanium on the bipolar plates, and these materials can be replaced with nickel and steel. Chemical reactions on anode and cathode for AEMWE are the same as in AWE (Kumar & Lim, 2022, p. 7). AEMWE is still in the research and development (R&D) phase, and it has its own drawbacks as well. These drawbacks include low ionic conductivity (Park et al., 2019, p. 2), and poor membrane stability (Miller et al., 2020, p. 4).

AWE, PEMEL and AEMWE are low temperature electrolysis methods. The last of the remaining four commonly known water electrolysis methods, solid oxide electrolysis, is an outlier, as it is classified as a high temperature electrolysis method. In SOE, the needed electrical energy is reduced, by substantially increasing the system temperature. SOE operates at temperatures similar to 800 degrees Celsius, and the feed water is fed into the stack as steam (Brauns & Turek, 2020, p. 2). As some of the energy needed for the electrolysis process is gained from the high temperature, SOE does not need as much electrical power, improving the electrolysis process' efficiency. According to Hu et al. (2022, p. 2), SOE has the efficiency rate of >90 %, while PEMEL would have efficiencies between 65 % to 82 %. Brauns and Turek (2020, p. 2), also mention that the theoretical SOE stack efficiencies could go near 100 %.

SOE has two porous electrodes and a dense ceramic electrolyte, which works as a membrane in the cell (Kumar & Lim, 2022, p. 13). Instead of hydroxide ions or protons (H^+), the electrolyte membrane in SOE needs to conduct oxygen ions (O^{2-}). The chemical reactions on the anode and cathode can be seen in equation 9 and 10. Equation 9 is the chemical reaction on the cathode, while equation 10 is the reaction on the anode.

$$H_2O + 2e^- \rightarrow H_2 + O^{2-} \tag{9}$$

$$O^{2-} \rightarrow \frac{1}{2}O_2 + 2e^- \tag{10}$$

The electrolyte membrane needs to be dense, as it cannot allow hydrogen to pass through, as this would lead to the recombination of hydrogen and oxygen (Ni et al., 2008, p. 3). The main drawbacks for SOE include rapid material degradation and short stack lifetime (Hu et al., 2022, p. 2). SOE is also currently in research and development phase, and the stack sizes available are below 10 kW (Brauns & Turek, 2020, p. 2). Due to the small stack sizes and short lifetimes, the high efficiency rate of SOE is overshadowed by the high manufacturing costs of the stack.

Each water electrolysis method has its own strengths and weaknesses. Some water electrolysis methods were specifically designed to overcome the other's weaknesses, such as PEMEL to AWE and AEMWE to PEMEL. The overall conclusion of the main four different water electrolysis methods and their main operating parameters can be seen in Table 1.

Table 1. Conclusion of the four known electrolysis method's parameters (Adapted from Kumar & Lim, 2022, *An overview of water electrolysis technologies for green hydrogen production*).

	AWE	**AEMWE**	**PEMEL**	**SOE**
Electrolyte	KOH/NaOH	DVB polymer + KOH/NaOH	Solid polymer electrolyte	Yttria stabilized Zirconia (YSZ)
Separator	Asbestos/Zirfon	Fumatech	Nafion	Solid electrolyte YSZ
Electrode	Nickel/Stainless steel	Nickel	Iridium oxide	Nickel/YSZ
Current density	0.2 – 0.8 A/cm^2	0.2 – 2 A/cm^2	1 – 2 A/cm^2	0.3 – 1 A/cm^2
Voltage	1.4 – 3 V	1.4 – 2.0 V	1.4 – 2.5 V	1.0 – 1.5 V
System temperature	70 – 90 °C	40 – 60 °C	50 – 80 °C	700 – 850 °C
Status	Mature	R&D	Commercial	R&D
Gas purity	>99.5 %	>99.99 %	>99.9999 %	>99.9 %
Efficiency	50 – 78 %	57 – 59 %	50 – 83 %	89 %

When compared to the other water electrolysis method’s performance and characteristics, AWE seems to lack behind them. AWE suffers from low current density values due to high ohmic resistances, and it produces chemical waste as lye. AWE is however, the most mature and well-known water electrolysis method, and much cheaper than the other alternatives. Alkaline water electrolysis can comfortably be expanded to megawatt (MW) scale, while providing long term stability and long stack lifetime. AWE’s large scale and long lifespan prove it to be a considerable option for green hydrogen production, as it is cheaper than its closest alternative PEMEL.

When paired with renewable energy sources, AWE should be optimized for dynamic operation. AWE suffers from gas impurities with partial loads due to increased gas crossover (Ursúla et. al, 2013, p. 5), which makes AWE hard to implement to the fluctuating renewable energy sources. Fluctuating energy sources could lead to additional shutdowns and startup cycles, which are not very suitable for AWE (Brauns & Turek, 2020, p. 15), as reverse current degrades the nickel electrodes (Kojima et. al, 2023, p. 6). Additionally, AWE has a long startup time. Brauns and Turek (2020, p. 2) mention, that due to these complications, PEMEL is usually the preferred water electrolysis choice to pair with renewable energy sources, as this has shorter startup time than AWE, and provides better load flexibility. However, AWE could provide larger capacities for cheaper price, if the alkaline electrolyzer is optimized for dynamic operation, and predictive control is used. Using different renewable sources together could also help steady the fluctuating load.

3 Design of the test bench

One of the reasons for building this test bench is to grant a possibility for companies to test and develop their stack designs. This will provide an opportunity to study the electrode materials or develop a completely different stack design.

As our aim is to provide the use of the test bench for interested companies, it is important to design our test bench applicable for many different stacks. As stacks vary largely from one another, it was decided, that changing a stack should lead to minimal changes in our test bench. This would mean that our test bench should be able to withstand hydrogen and oxygen pressures up to 30 bar and have the possibility to provide more power, even if our initial stack would not be rated up to those values.

3.1 Balance of Plant

Before starting any of the design work, it is important to be familiar with alkaline electrolyzer's balance of plant (BoP). Based on literature review, a simple BoP diagram was made, which can be seen in Figure 4. The diagram drawing includes the most common BoP elements of an alkaline system, and some of the earliest design discussions and questions that were pondered in the design meetings.

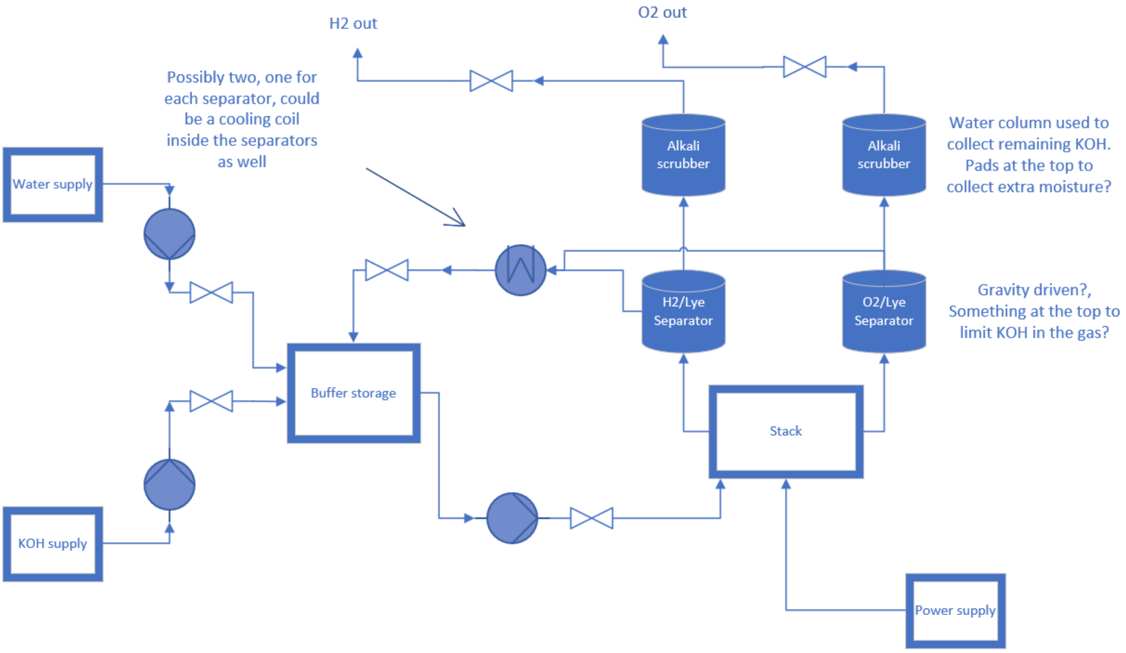


Figure 4. Initial diagram of Balance of Plant.

The most important BoP elements are the gas-liquid separators, heat exchangers, and pumps (Rizwan et. al, 2021, p. 3). As every alkaline system will be different, can the amount of these key components, such as pumps, change from system to system. Gas scrubbers are also often seen in BoP drawings, as these are important when clean hydrogen is needed for a certain application, or the hydrogen will be pressurized and stored. The first BoP diagram included all of the most common elements, as well as the scrubber units.

Alkaline electrolysis can be done without these BoP elements, but if continuous operation with an electrolyzer is desired, would a system without these elements be a safety hazard for both the environment and the personnel. Heat exchangers, for example, are crucial components for heat managing, as without them, will the stack overheat. In worst cases, overheating could lead to damage of the stack’s internal components, making the stack unusable. With heat exchangers, stack temperature can be controlled by having continuous flow of cooled electrolyte into the stack. To ensure a continuous flow of electrolyte into the stack, a circulation pump would be a necessary BoP component.

Gas-liquid separators are responsible for separating the product gases from the two-phase flow coming from the stack. Remains of electrolyte can still escape with the product gases, together with water vapor. Usually, the remaining electrolyte would be washed away from the product gases inside a scrubber unit, which is used to clean the hydrogen stream before its application.

3.2 Development of the P&I diagram

As the necessary BoP elements and the stack manufacturer had been considered, the actual design process was started. To help design the first version of the P&I diagram, a complete diagram of another VTT test station was studied. This diagram would provide knowledge on how the actual finished P&I diagram should look like, while giving an example for different used valve types and their locations. Studying the purpose of each valve in its location would help us decide whether such a valve would be needed in our new test station. A simplified version of the first P&I design can be seen in Figure 5. As the diagram is a simplified version, it does not include the most common instruments, such as valves and transmitters, which are present in the actual design. Additional lines that cannot be seen on Figure 5, include the cooling lines and emergency flushing lines.

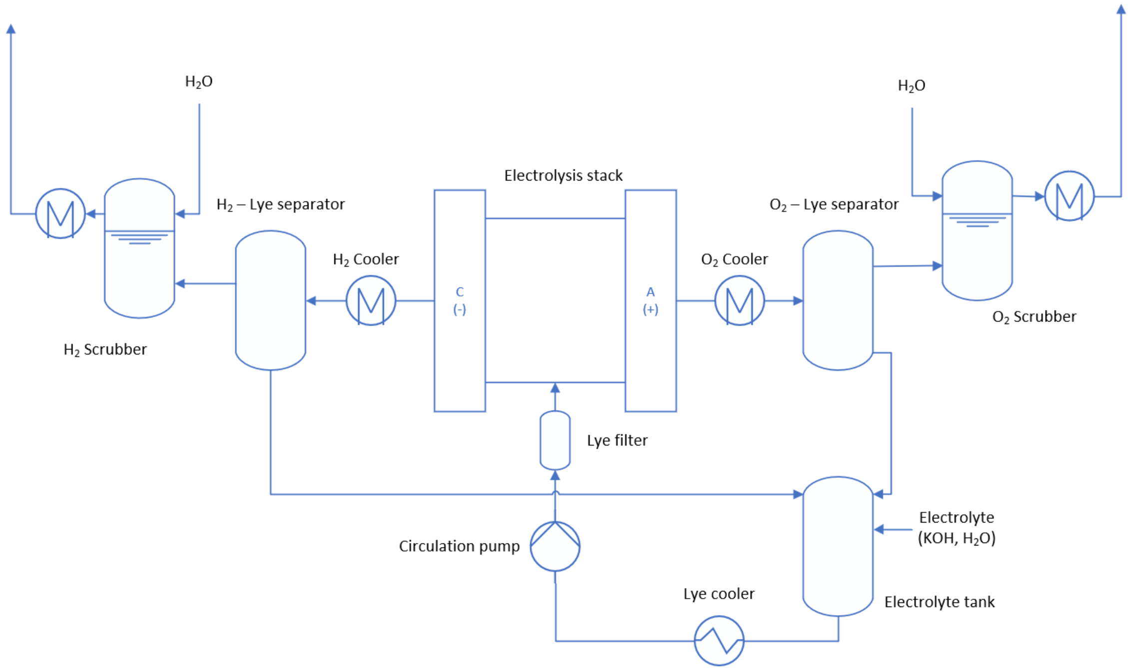


Figure 5. Simplified version of the initial P&I diagram.

The first P&I diagram design includes all of the found common BoP elements, but only one pump, which would feed the electrolyte liquid into the stack with a continuous flow. Between the pump and the stack is a lye filter, that would ensure there would not be any lumps in the liquid electrolyte. The need for this filter was not a certainty, as it was more of a “what if” -thought, and there were only a handful of mentions for such units in the literature. For example, IRENA’s Green hydrogen cost reduction report (2020, p. 34), includes a lye filter, but does not explain its use. The electrolyte would be fed to the stack, and it would come out of it together with the product gases. The stack has two outlets, as the product gases have their own flow channels to prevent the gases from mixing together and forming possible explosive mixtures. Each of the outlet lines have their own BoP elements and the outlet lines are identical. The two-phase flow would be run through a heat exchanger, to cool down the mixture, before entering a gas-liquid separator. A heat exchanger at this point would be necessary, as the planned coalescer type separators have their own temperature limits for the alkaline media.

The separator units would separate the product gases from the electrolyte liquid, allowing the product gases to be directed to their own lines. The separated liquid would be fed back to the storage tank, where it would be re-circulated through the pump. The concentration of the liquid electrolyte would be measured to ensure that a desired 30 wt. % KOH concentration is not exceeded too much. If this happens, the KOH concentration would be diluted by letting purified water flow into the mixture inside the storage tank. Before the pump, the electrolyte would be again cooled down with a lye cooler, to make sure that the temperature would not exceed the limits for the pump's safe use.

The separated product gases could still have the possibility to carry KOH with water vapor. To eliminate the possible remaining KOH from the gas stream, the gas is passed through a scrubber unit that would use purified water to trap the remaining KOH particles into itself. Purified water with the trapped KOH would stay at the bottom of the scrubber unit. Clean gas streams would exit the scrubbers to enter heat exchangers, which are meant to dry the product gases. Gas drying was implemented to reduce the gas moisture, as some of the instruments used at the end of these lines could be vulnerable to liquid KOH vapors. Passing through the sensitive instruments, the dry gases would eventually be vented out to the atmosphere, as there were no plans for hydrogen use at this stage of the test bench.

A second iteration of the P&I diagram was made with the idea to skip a scrubber on the oxygen side. This idea came from further studying the literature, as some of the cited diagrams for alkaline systems included a scrubber only on the hydrogen side. An example of a diagram only containing one scrubber unit can be found on well-known electrolyzer supplier NEL Hydrogen's brochures (2021, p. 3). This second version of the diagram was quickly discarded, as we were not familiar with how to make sure KOH would not escape into the environment while venting oxygen into the atmosphere. The possibility of potentially releasing KOH particles into the atmosphere and to the surrounding environment was deemed not to be worth of the financial benefits that eliminating a scrubber unit would provide.

The third iteration of the P&I diagram saw a change in the scrubber designs, as the planned spray tower scrubber design used in previous diagram versions, was hard to design in our test bench size range. The thought of building an in-house spray tower scrubber came up, but was eventually discarded, as there was not enough time to build such complex vessels in-house. The third iteration of the diagram saw a return of the first scrubber design, where the scrubber would just have a purified water column to clean the gases. This third version also saw a change in heat exchanger location, as some of the end-side instruments were moved between the separator and scrubber units. The performed changes can be seen in Figure 6.

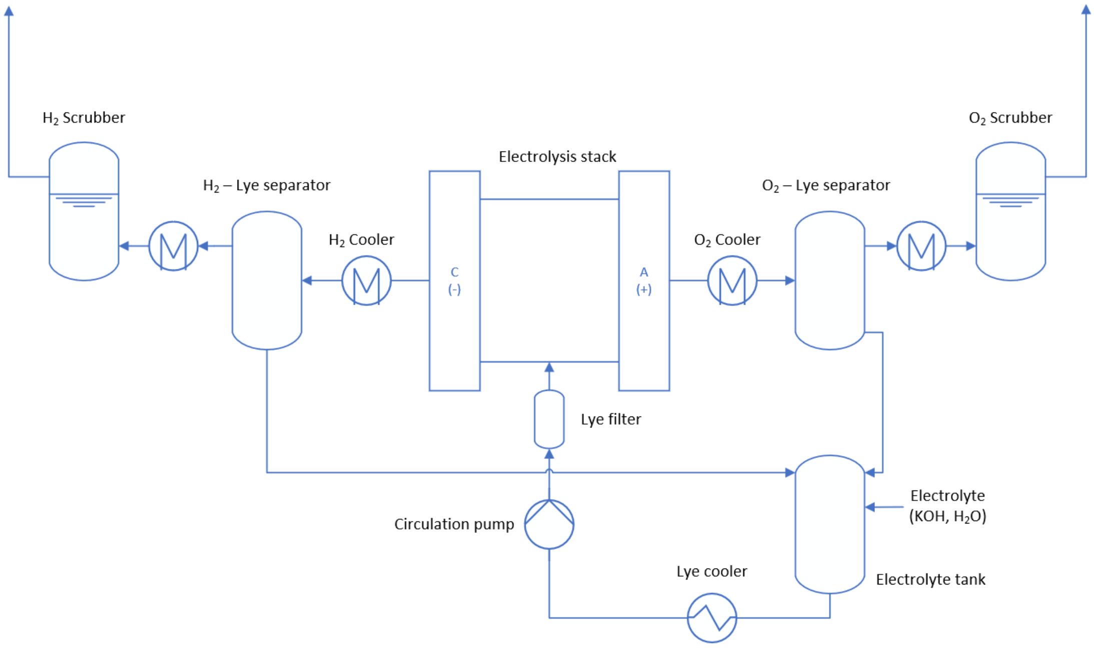


Figure 6. Third iteration of the P&I diagram, which has the end instruments before scrubbers.

The final P&I diagram design saw simplifications to the main BoP elements, as the separator and scrubber units were combined. The diagram also saw a reduction in the number of heat exchangers, as there was no need to cool down the fluid temperature before the separator anymore, as the separator would not be coalescer type design. A simplified version of the final diagram can be seen in Figure 7. The final design would work like the first design, if the separator and scrubber designs are not considered.

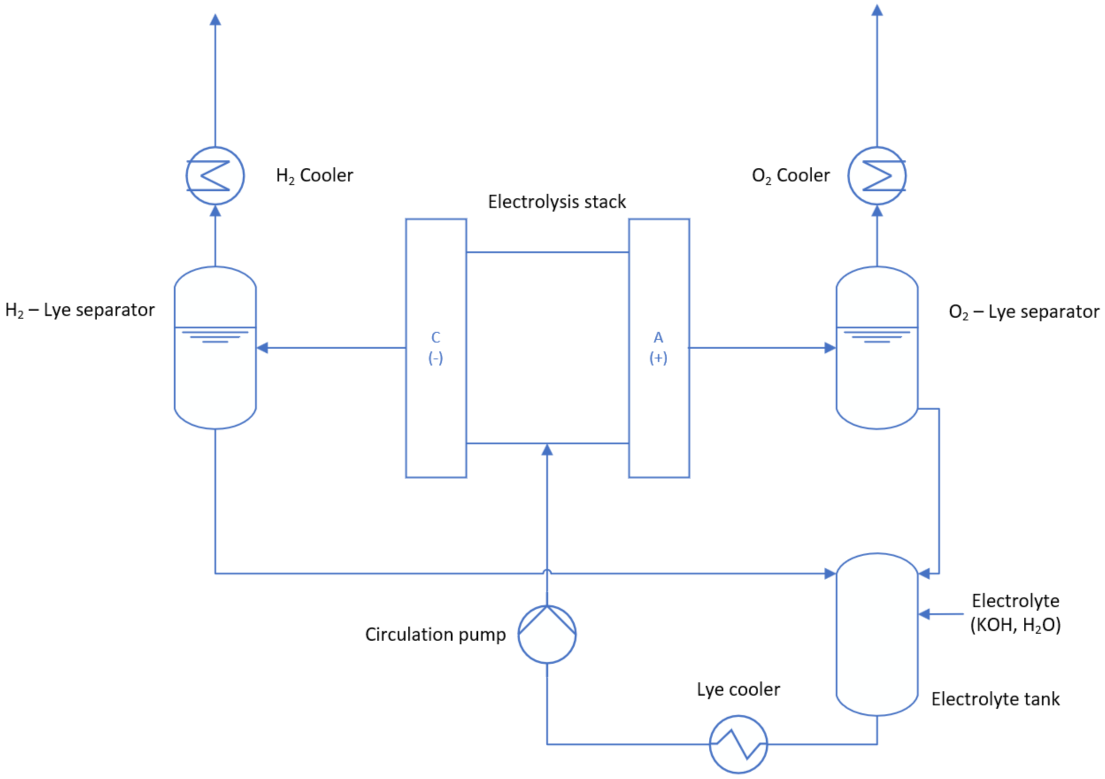


Figure 7. Final version of the P&I diagram, which does not have scrubber units.

The final design also saw adjustments on the emergency lines, and an increase in the number of used instruments. Other changes that cannot be seen in Figure 7, include improved level control for the separator vessels, and reworked cooling lines that lead to the heat exchangers. Cooling line's rework was one of the most significant changes made to the test bench, as the idea to use laboratory room's tap water would not be enough to cool down the product gases coming from the separators. This meant that we would need to implement a chiller unit to maximize the condensation of KOH vapours. The water circulation to the lye cooler would still be managed by the laboratory's tap water, as this cooling process would not be as demanding as the ones after the separator unit.

3.3 Scrubber design

The first planned scrubber idea was adapted from the literature. The scrubber design done by Sandeep et al. (2017, p. 3–4), is meant to be a simple column of water in a vessel. The product gases would bubble through purified water, dissolving the remaining KOH particles from the gas stream. The design from Sandeep et al. (2017, p. 3–4), included a mist eliminator at the top, to dry up the gas before entering the heat exchangers. An illustration of the adapted design for the first scrubber, can be seen in Figure 8.

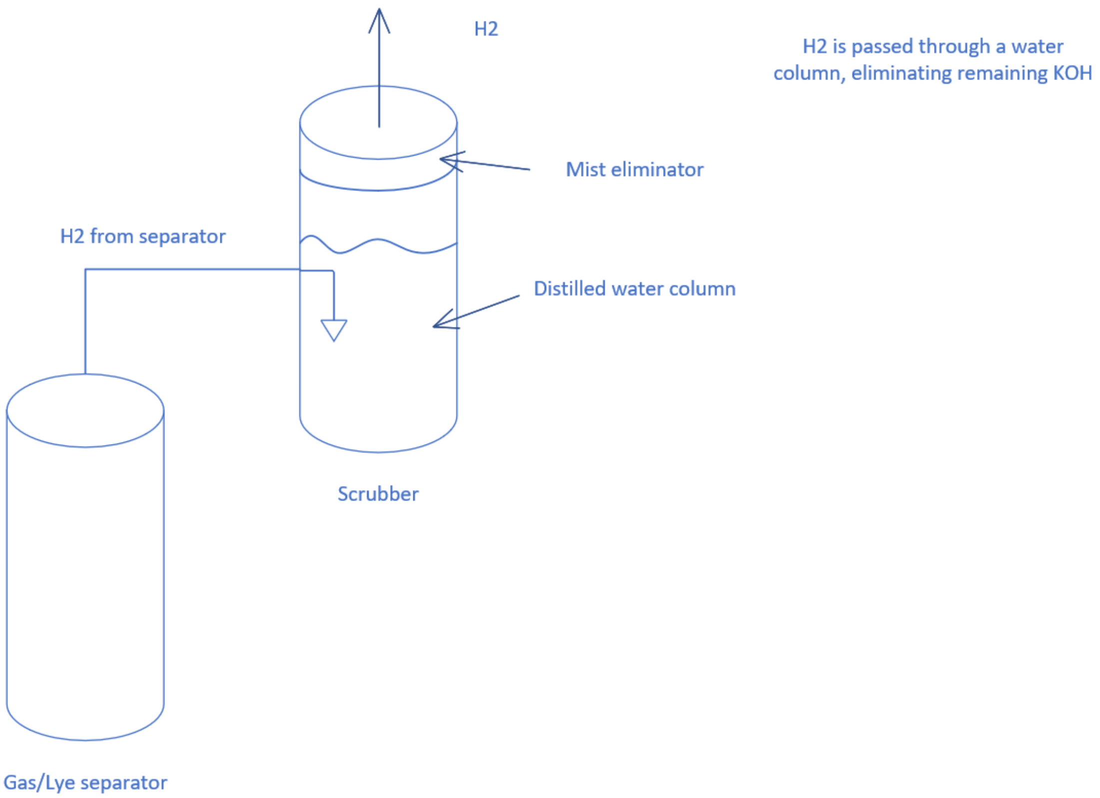


Figure 8. Illustration of the adapted first scrubber design, in which gas bubbles through a column of distilled water.

The main problem of this scrubber design was the column of purified water. Eventually this water would need to be replaced occasionally, which would add complications to our pipelines and water waste management. KOH content would make the purified water into chemical waste, which would need regular disposal. There were also questions whether this kind of design would be effective, as the KOH concentration of the purified water would rise during the test bench use. There were also uncertainties on how we would know when it was necessary to change the purified water of the vessel.

To overcome the difficulties of the first design, a spray tower like scrubber was seen as a better design choice. This second design would work by having spray nozzles inside the vessel, spraying purified water into the incoming gas stream. This purified water spray would absorb the remaining KOH particles in the gas stream, making it accumulate at the bottom of the vessel. However, this design did not fix all the first designs problems, such as the chemical waste.

As spray tower scrubbers are usually used in marine applications, it was hard to find a supplier who could provide us spray tower scrubber units this small. This kind of design would also be difficult to build locally by ourselves, which ultimately lead to the abandonment of this design, as more complex design did not fix all the problems of the first design.

Scrapping the spray tower design meant the reintroduction of the first scrubber design. As the original design was not convincing, a quick idea draft for its modification was introduced, where instead of a vessel, the scrubber unit could be a larger pipeline with wave like bends containing purified water. The illustration of the idea can be seen in Figure 9. The design would have problems, such as how the purified water could be easily changed, and how we could overcome the large pressure drops caused by the design. The latter problem was solved with the idea of placing the scrubber at atmospheric pressure, after the pressure controlling instruments.

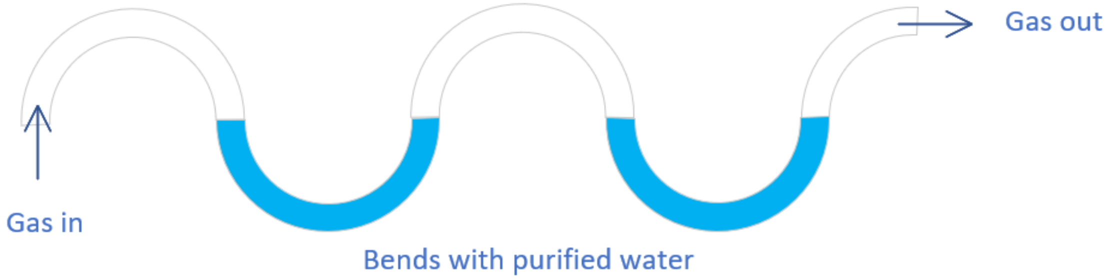


Figure 9. Pipeline scrubber design with multiple patches of purified water.

This new bent pipe scrubber design did not see a final polished design plan, as eventually the use of scrubbers was deemed unnecessary. Collaborative third-party meetings were held to help us tackle the test bench's design complications, and with the help of these collaborative meetings, we became familiar on how to make sure our oxygen was KOH free, without passing it through a scrubber, allowing us to vent it straight to the atmosphere. As there was no plan to use or store the produced hydrogen in our test bench, there was no longer any reasons to have scrubber units on either side of the test bench.

3.4 Separator design

The first separator design idea was also adapted from the found literature, and rather simple. The test bench would have two separators, as the product gases are in their own respective channels. In the design done by Sandeep et al. (2017, p. 3–4), the separators would be vessels, into which the two-phase flow (gas and liquid) from the stack would be fed. The separator would be gravity driven, meaning that the product gas, whether it is oxygen or hydrogen, would rise to the top of the vessel, while any liquid flow would drop to the bottom of the vessel. At the top there could be a steel demister to limit moisture in the outgoing gases. A diagram of the adapted design idea is presented in Figure 10.

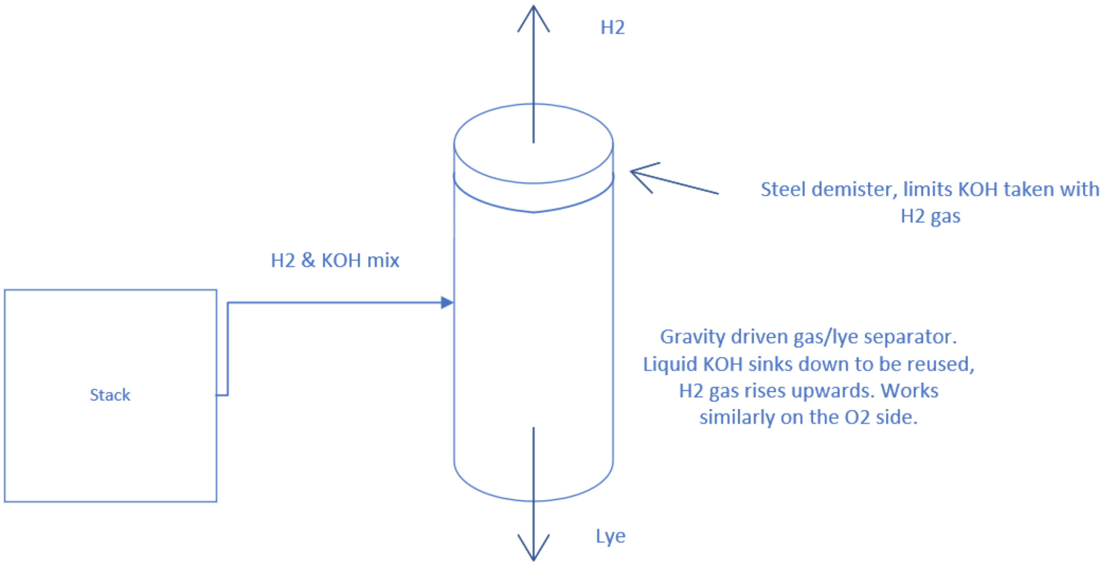


Figure 10. Adapted first design of the separator, with gravity driven operation and a steel demister on top.

This adaptive design did not see much further planning, as there were doubts whether this kind of design would be effective or efficient enough. The second design idea was to use coalescer type separators, as they are very effective in removing even the smallest liquid droplets from gas flows. Coalescer separators work by combining smaller droplets into larger ones, eventually separating most of the liquid from two-phase flows. Coalescers are usually not designed to be used in hydrogen applications, but there could be a possibility to adapt a coalescer unit for hydrogen use with the help of its manufacturer. However, coalescer units are expensive with overall cost point of view.

An illustration of the second design can be seen in Figure 11. The second design was somewhat similar to the first design, but the vessel would have a filter inside it. The two-phase flow would be fed into the vessel from the bottom inlet. After the inlet, the two-phase flow would enter the filter inside the vessel. Product gases would then percolate through the filter and leave the vessel from the gas outlet. The liquid electrolyte would be collected at the bottom of the vessel, where it would be fed back to the storage tank, to be re-circulated and re-used.

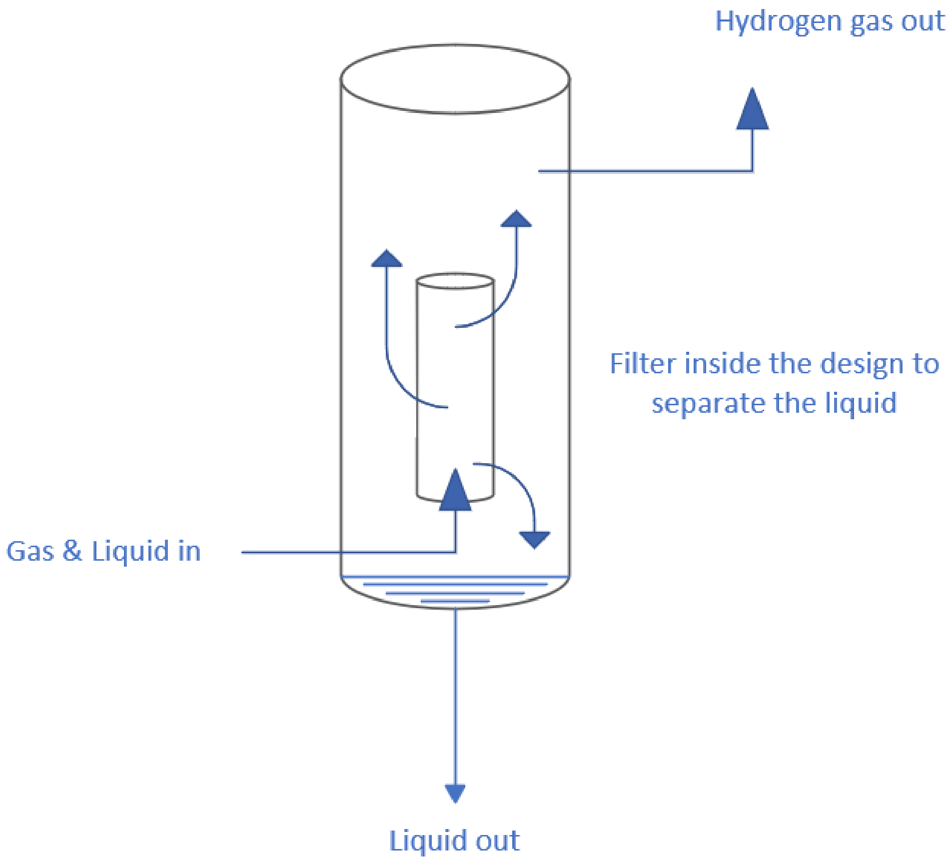


Figure 11. Coalescer type separator design, with a filter element separating gas from liquid.

As coalescer type separators are not designed for hydrogen use, there was no certainty on how clearly their usual high separation rates would translate to our new wanted application. If the separation rate would be similar to those seen in their designed applications, there was a thought, whether a scrubber unit would be even necessary, as the used separator already has such high separation effectiveness.

Unfortunately, the design was rather large when compared to the available laboratory space. Other reasons why this coalescer design was not chosen included the filters, as there would need for regular filter changes, which would increase the already high price of a single separator unit. Also, it was thought that this kind of separator unit would be an excellent choice, if we were to store and use our produced hydrogen. Because there were no such plans, the design was deemed a bit too expensive for our application.

The final separator design was more like the first design than the second one. The final design is a combination of the separator and scrubber unit's first designs. The separator would be a gravity driven vessel, where liquid electrolyte would be stored. A certain level of electrolyte would be maintained inside the vessel, allowing the product gases to bubble through an electrolyte column, instead of a purified water column, as in the scrubber's first design. The product gases would be led into a heat exchanger unit, which would eliminate the remaining moisture in the gases by cooling the gas down to a certain temperature. The condensed lye will be collected at the bottom of the separator, or straight at the lye storage tank. The separator units are designed and constructed in-house. An illustration of the combined units design can be seen in Figure 12.

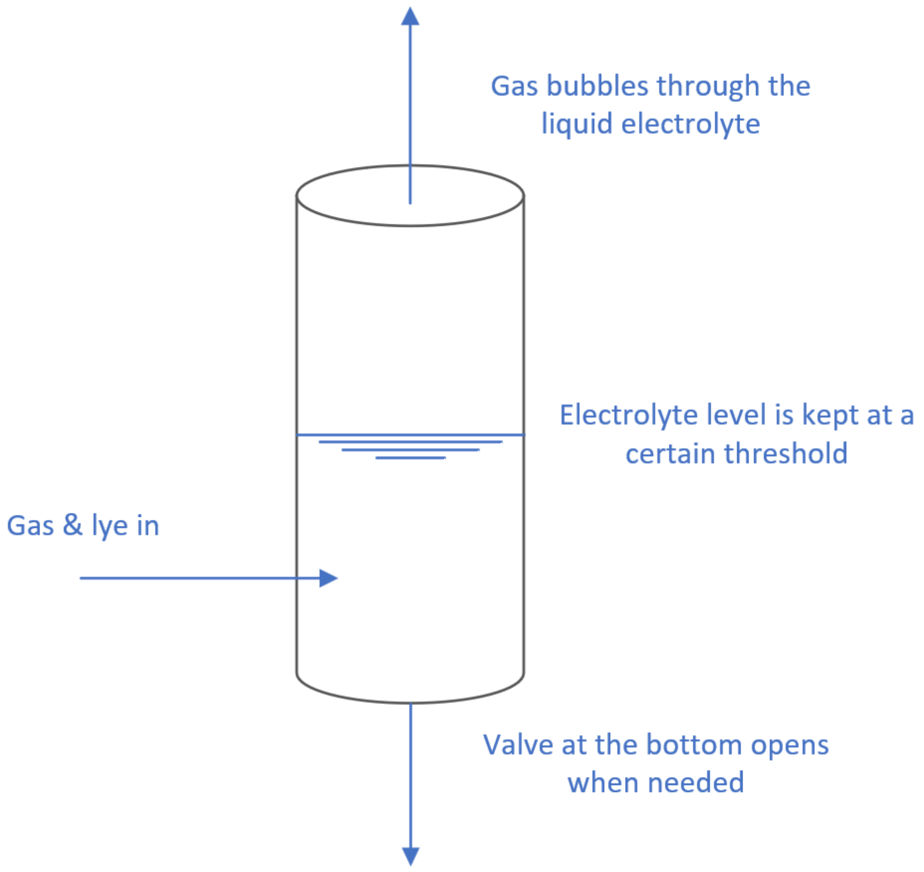


Figure 12. Final separator design with a gas/lye inlet, gas would bubble through a lye column.

To design a compact test bench and address the problem of limited space, the separator design should be compact too. Another advocacy for compact design was pressure vessel classification. If a vessel is pressurized, there exist a certain volume limit, before the vessel design needs to fulfill technical safety measurements of European Commission's Pressure Equipment Directive (PED) (EU 68/2014, p. 19–20). The vessel will still be tested to ensure its safe operation. Designing the vessel according to PED would add additional steps and timeline to get the required certificates. Therefore, the separator was designed at a smaller operating pressure and volume to avoid further delays in the project timeline.

The separators were designed to have a continuous level of lye during the operation, which meant that the liquid level would need to be monitored at all times. The liquid levels inside the separators are monitored by having level switches at certain volume points of the vessel, each sending a signal if the liquid level is at required height. If the liquid level is seen at one of the uppermost level switches, it will send a signal to a solenoid valve at the outlet of the separator. This solenoid valve will remain open until the liquid level is back at the desired threshold levels.

The solenoid valve was selected by choosing the desired amount of time it would take for the liquid level to drop down from the thresholds upper limit to the lower limit. As the liquid volumes at the threshold limits are known, the needed volumetric flow can be calculated using equation 11.

$$Q_{out} = \frac{V_2 - V_1}{t} \tag{11}$$

In equation 11, t indicates the desired period of time it takes to lower the liquid level from the threshold's upper limit to the lower limit, V_2 indicates the volume of liquid inside the separator when the threshold's upper limit is reached, and V_1 the volume of liquid inside the separator when the threshold's lower limit is reached. It is important to note that this calculation assumes that there is no lye flow into the vessel during the time when the solenoid valve is open. This will not be the case during normal operation, which is why it is important to take notice of the maximum lye flow into the vessel.

To take a more practical approach to see if our separator design would work, a simple test was arranged. In this test, a clear cylinder-like glass container was filled with water to observe the bubbling of an inlet gas. A camera picture of the used test setup can be seen in Figure 13. The size of the glass container did not represent the actual separator design dimensions, but it was good enough to give a rough estimation on how the gas would behave.

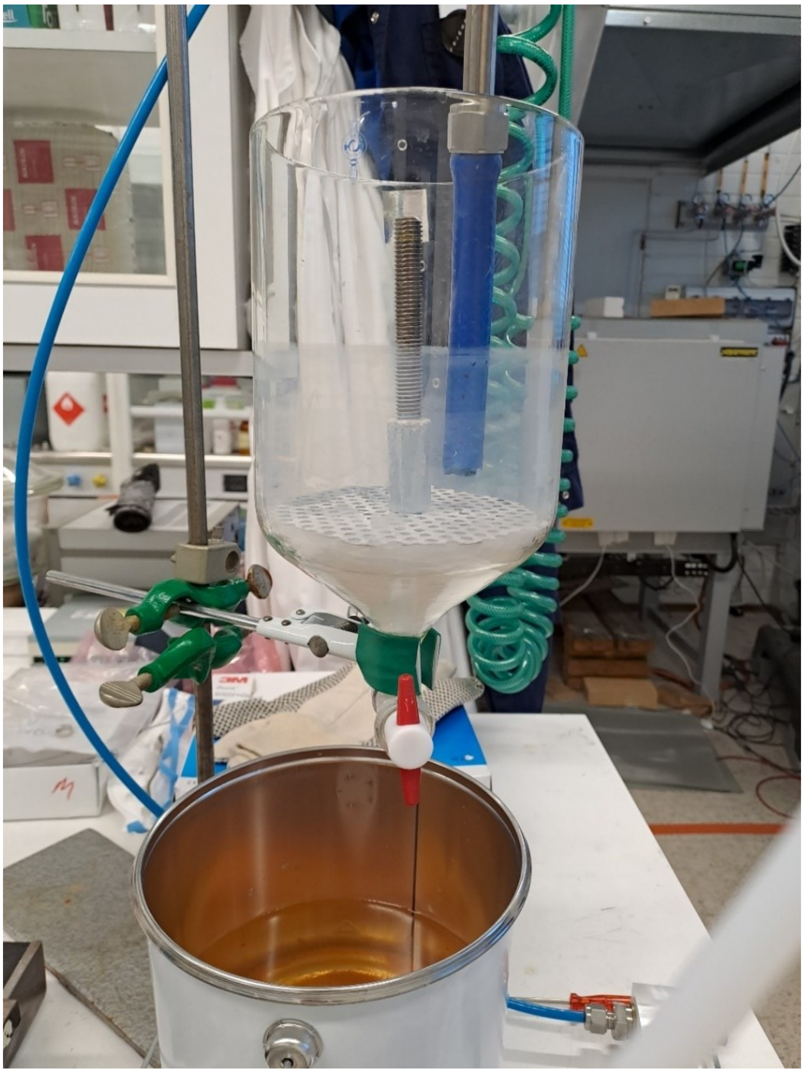


Figure 13. Setup used to test gas bubbling and bubble accumulation in the separator vessels.

The test was done with oxygen and helium gases to observe how the bubbling can differ in each separator, while the gas flows were adjusted to meet the real maximum conditions of our actual test bench. The main idea of the test was to see how many small gas bubbles would be trapped in the liquid, near the bottom of the vessel. Small gas bubbles were a concern, as they could escape with the lye when the solenoid valve at the separator outlet is opened.

One of the tested aspects was the effect of the separator inlet, which in this test was made from a regular hose. The hose was flexible material, allowing to test different bends and locations for the inlet. By pointing the inlet straight down, some accumulation of gas bubbles could be seen at the bottom of the vessel. To get some indication how the bubbling could change with a horizontal inlet, the hose was bent almost to a 90°-degree angle. The bend did not seem to have a considerable effect on the amount of small gas bubbles accumulating at the bottom, but it was still deemed to be the better design choice, as a straight pipe from the side of the vessel would be easier to implement. Horizontal inlet would not allow a straight path to the bottom of the vessel, which should decrease the amount of small gas bubbles accumulating there. The location of the inlet played a significant role in the accumulation of the gas bubbles, as inlet closer to the surface accumulated less small gas bubbles at the bottom.

To limit the accumulating gas bubbles at the bottom, a perforated steel disk was put to the bottom of the glass container. The disk could be of simple design, as seen in Figure 14. The mesh would disturb the flow from the straight-down pointed inlet by not allowing a straight-line path for the flow at the bottom of the vessel. This worked as thought, as there were clearly less small gas bubbles at the bottom. The mesh did, however, add other points of concern, as when tested with helium, larger gas bubbles were trapped underneath it. This might have been caused by the inlet being, at some points, directly on top of the mesh, but it indicated a real flaw, as the gas bubbles could not rise up if they get underneath the mesh. There was also a possibility that the holes of the mesh would be blocked by the solidified KOH when the test bench is not running.

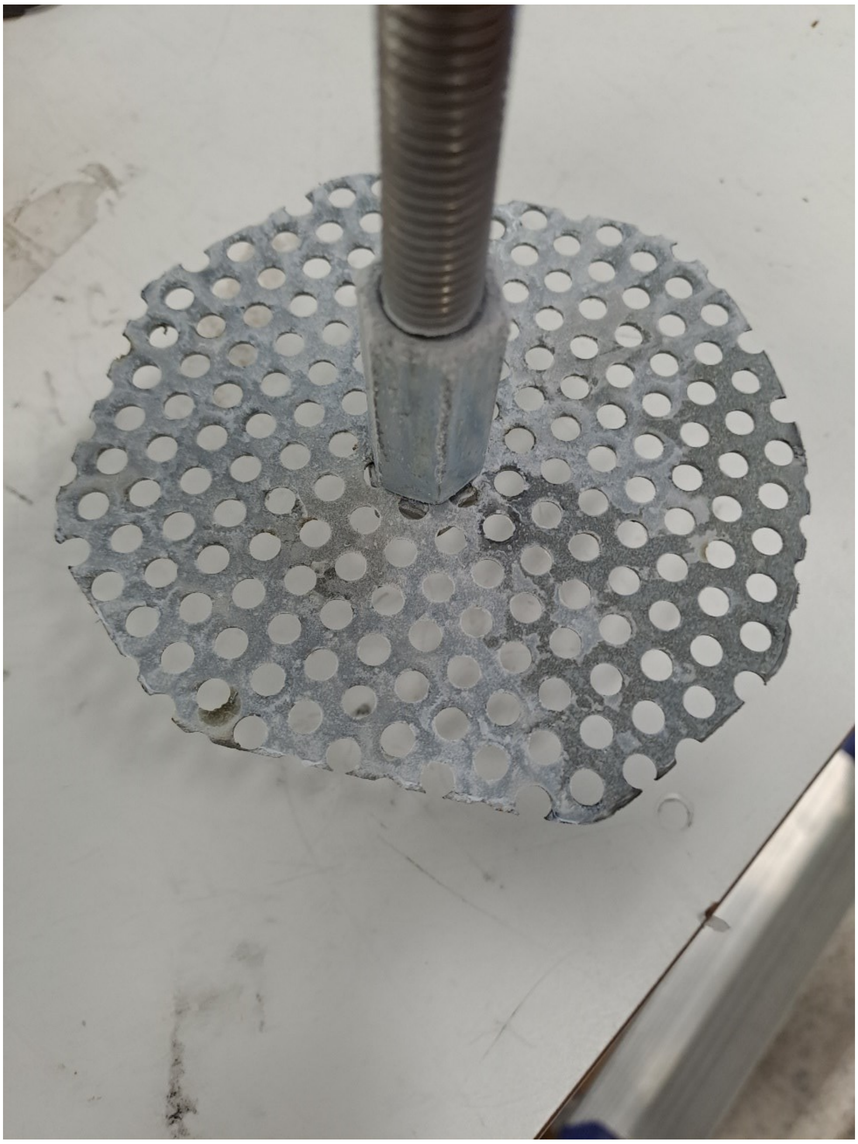


Figure 14. Perforated disk used to disturb gas flow.

During the test, holes were made on the side of the hose, to see how it could affect the bubbling. The inlet with an increased number of holes, which can be seen in Figure 15, had a positive impact, as this made the bubbling process less violent. The size of the holes was also studied, and smaller holes were seen to make the bubbling even more calm. However, smaller diameter holes did increase the number of small gas bubbles, which would not escape to the liquid’s surface quite as fast as the larger ones, trapping them in the liquid for a longer time. This was not seen as a desired outcome, as the smaller gas bubbles could then eventually find their way to the bottom of the vessel and escape with lye, when the solenoid valve opens.

Figure 15. Hose inlet of 15 mm, with increased number of holes.

3.5 Test bench parameters

Stack was the most important component when designing our test bench, as it defines the test bench’s characteristics by giving us the most limiting parameters. These parameters include the required power, temperature limit, pressure limit, amount of gases produced, and the needed electrolyte flows in and out of the stack. Our test station was planned to be adaptable with minimal changes to be able to fit a different stack, which meant we would also need to design our test bench for a secondary set of parameters, such as higher pressures and larger flow rates.

Our test bench's stack is an LBE-30C-F unit made by a South Korean company Lightbridge, which can be seen in Figure 16. The stack has a total of 30 cells, with a power rating up to 4.8 kW. When operated at maximum conditions, the stack will produce hydrogen at a rate of 21 nl/min, and oxygen at a rate of 10.5 nl/min. To achieve these gas production rates, the stack would need a 1 l/min feed flow rate of electrolyte. The full list of stack parameters can be seen from Table 2.

Table 2. Stack parameters (Adapted from Lightbridge, *n.d., Alkaline Water Electrolyzer Stack Install Guide (LBE-30C-F) User Manual*)

Parameter	**Value**
Type	LBE-30C-F
Power	4.8 kW
Current density	0.3 – 0.5 A/cm^2
Cell voltage	1.8 V
Design pressure	Max 7 bar
Design temperature	Max 80 °C
Membrane material	Porous polymer
Electrode material	Nickel based compound
Electrode active area	177 cm^2
Electrolyte	30 wt. % KOH
Hydrogen production rate	Max 21 nl/min

The gas and liquid flow rates were the most important parameters of the stack when the separator, scrubber, and pump units were considered. As the planned coalescer type separators and the pump already had their own temperature limits, the maximum temperature limit of the stack has no issues, as this was within a similar temperature range.

Figure 16. Stack received from Lightbridge (Model no. LBE-30C-F).

As the stack is not designed to withstand pressure differences between the anode and cathode compartments, a certain pressure must be maintained on both sides of the test bench. To maintain the same pressure on both sides of the test bench, a pressure control loop has been implemented, with a pressure controller on each side.

From the stack onwards, both hydrogen and oxygen lines of the test bench are mostly identical. Main differences come from the instrument characteristics, as the instruments and the pipeline on the oxygen side need a special cleaning to avoid oxygen system fires, as materials are more easily ignited in oxygen-enriched environments (Wood et. al, 2021, p. 2). It was also decided that the oxygen side's pressure controller acts as the master controller to the hydrogen side's slave pressure controller. A pressure setpoint would be given to the master controller, which then sends a signal to the slave controller, to match its pressure with the master controller.

The lines have mostly the same instruments, one of the only differences being the absence of a mass flow transmitter on the oxygen side. Flow of produced oxygen is not measured, as we are more interested in the stack's hydrogen production rate on various occasions. The used mass flow transmitter is a Bronkhorst EL-FLOW Select, which can be seen in Figure 17.

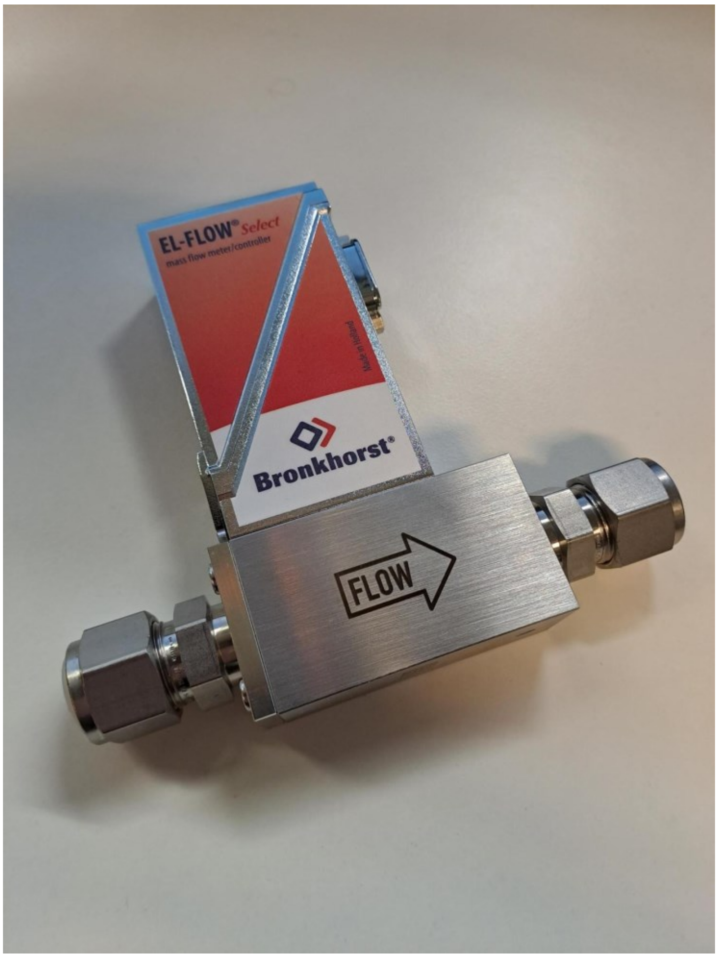


Figure 17. Hydrogen mass flow transmitter made by Bronkhorst.

The selected flow transmitter works with thermal mass flow measuring principle, in which part of the gas flow will be redirected through heating elements and bypass sensors. The heating elements warm up the gas to different temperatures, creating a temperature difference, which is proportional to mass flow through the sensors (Bronkhorst, n.d., p. 3). An illustration on how the valve works can be seen in Figure 18. In our application, the used mass flow transmitter will measure hydrogen flow rate up to 30 nl/min, withstanding pressures up to 51 bar, allowing us to use the same transmitter with different stack characteristics.

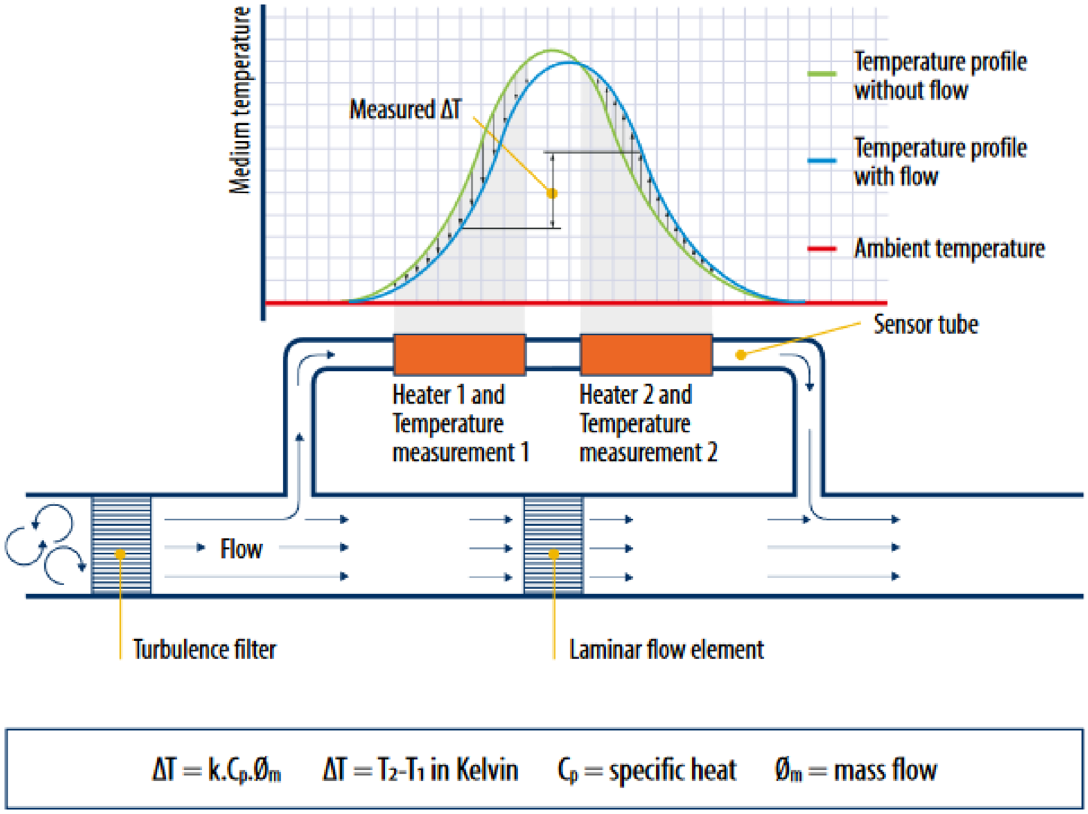


Figure 18. Working principle of the mass flow transmitter (Bronkhorst, n.d., *EL-FLOW Select Digital Thermal Mass Flow Meters and Controllers for Gases*).

Each of the main lines would have a set of valves, such as manual valves, check valves, and relief valves. Manual valves in the main pipelines are mainly meant for manual control of the flow. These valves could be, for example, used when a change of stack would be desired, without the need to empty the whole system of the liquid electrolyte. Check valves would act as directional flow control, and are used to prevent unwanted flow directions, such as backflow of lye from the separator vessels to the stack. Relief valves are purely for the safety of the test bench, as uncontrolled rise of pressure could lead to significant damages to both personnel and to the test bench. The main lines would also have transmitters to check temperature and pressure levels at certain points of the test bench, and more specific transmitters, such as gas analysers to ensure there would not be explosive gas mixtures in the lines.

Feed line leading to the stack would also have its own share of instruments. As a difference to the main lines, the feed line would have a concentration transmitter to measure the wt. % of KOH in purified water. Concentration is measured with a Coriolis type flow transmitter, which can measure the flowing media’s density. The feed line would also have flow controllers to divide the electrolyte flow evenly between the anode and cathode sides of the stack.

Other significant lines in the test bench are the cooling lines, and the emergency lines. The cooling lines were split into two channels, one channel managing tap water feed to the lye cooler, and the other managing water flow from the chiller to the heat exchangers for product gases. To get the stack faster to operation conditions, it was decided that the same lye heat exchanger would be used to preheat the electrolyte at the test bench’s start up. This heat exchanger would then switch to cooling the electrolyte, as the power is applied to the stack. Emergency lines in the test bench include emergency flushing with nitrogen gas, and emergency flushing with purified water. In case of emergency, nitrogen gas would flush the system from hydrogen and oxygen gases, while the purified water would clean lye out of the lines.

Like oxygen gas, hydrogen can bring complications and requirements for the used instruments. As hydrogen atoms are very small, hydrogen can leak through many solid materials, and even cause embrittlement on steels (Dwivedi & Vishwakarma, 2018, p. 3). This steel embrittlement could eventually lead to instrument failures, creating unwanted leaks. The main construction material for hydrogen should, for example, be austenitic stainless steel, as this is less liable for hydrogen embrittlement (Dwivedi & Vishwakarma, 2018, p. 2). Likewise, the used valves should be specifically designed to be fit for hydrogen, as regular stainless steel valves could still allow hydrogen leakage from their seals.

As lye is an environmental hazard, it cannot be drained down in a regular drain. The test bench is designed to have drain lines to prevent lye from escaping to the laboratory room floor if problems occurred, such as, if the storage tank would threaten to overflow. Highly corrosive base KOH brings its own difficulties. Potassium hydroxide especially affects the used instruments, such as valves, as KOH can have an effect even on some stainless steel grades. To prevent material-based problems on the pipelines and instruments, stainless steel grade of 316 was chosen, because it is resistant to most aggressive medias, such as KOH. SS 316 is a part of a 300 series stainless steel group, which consists of austenitic stainless steels, making the selected grade suitable for hydrogen use.

3.6 Design challenges

Initial difficulties of designing the test bench for two different set of operational parameters came up when discussions were started for a circulation pump. As we do not know the electrolyte flow rate needed for the future stacks, it would be hard to choose a pump suitable for every situation. Because of this, the pump was chosen by sizing it for our current stack. If a stack with a different necessity for feed flow was to be installed to the test bench, a change of pump would be necessary. Finding a pump to fit our stack was also a bit difficult, with 7 bar(g) pressure limit and a relatively slow flow rate of 60 L/h further limiting our choices. The chosen pump is an IX-C060 dosing pump from IWAKI, together with a M002 membrane type pulsation dampener. The pulsation dampener is used to reduce the pressure differences caused by the pump pulses, which could prove to be problematic in our application. An example of discharge pressure with and without the provided pulsation dampener can be seen in Figure 19.

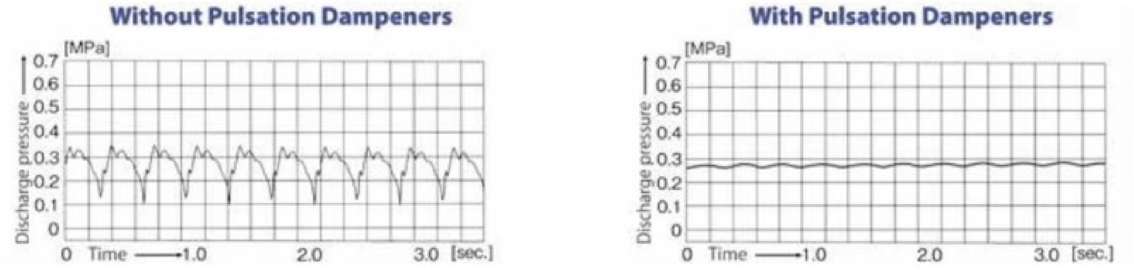


Figure 19. Discharge pressure from the pump (T. Pennanen, IWAKI, 31.8.2023, personal conversation).

Unknown flow rates were also a difficulty when the design of separators and scrubbers was ongoing. To design the coalescer type separator units, the maximum flow rate of gas and liquid would need to be known to correctly size the filter inside. As the possible flow rates of our future stacks were not known, it was decided that the coalescer separators would also be sized for our current stack's flow rates. Going for larger filters could have caused problems with the limited laboratory space, and even if, there would be no guarantee that the chosen filter size would be enough for future stacks. Flow rates are not as large of a problem for the final separator designs, as these separators do not have a filter element. Most important factor would be the electrolyte flow into the separator, as this could reduce the time to fill up the separator vessel. If the electrolyte accumulates, it increases the risk of overflowing the separator. This problem can be fixed by changing the size of the solenoid valve at the outlet of the vessel.

For the final separator design, pressure would be the most limiting factor, as they are not designed to be classified as pressure vessels. Even if the final product could withstand pressures above the test bench's maximum pressure rating, still their use at higher pressures is prohibited, as they do not have the necessary certifications. This means that the separator units would still need to be changed, if the test bench would be run at higher pressures in the future. Designing the test bench for two different pressure parameters would also bring difficulties for certain instruments, like the back pressure controllers, as there were no 30 bar(g) rated controllers that could go down to our current desired pressure ranges.

The same pressure problems would arise when the relief valves were chosen, but this was to be expected, as relief valves are set to start opening at a certain pressure level when ordered. This would mean that even if a relief valve is able to work on both desired pressure ranges, it would need to be recalibrated, when pressure is increased. Ultimately, the list of components that would need to be replaced, when a new stack is introduced, grew larger than originally expected.

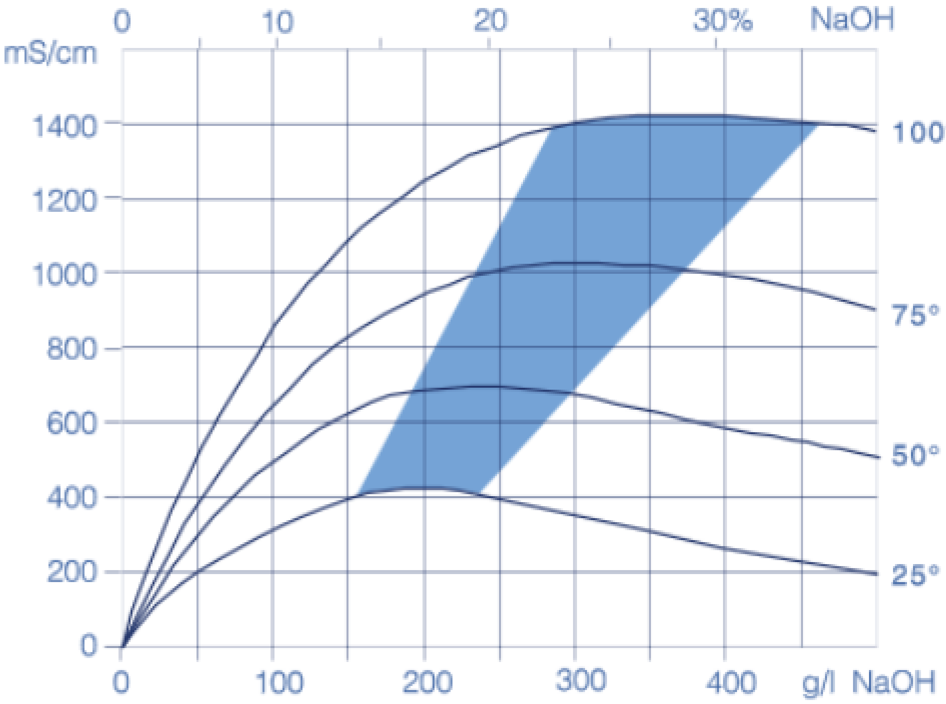
Instrument seal materials were also a challenge, as potassium hydroxide is corrosive for the most common O-ring materials, such as Viton and FKM. Most types of O-rings can see wear from corrosive media, but different kind of materials would endure KOH better and hold their durability longer than others. As leaks from seal fractures were not a desired outcome, seal materials like EPDM and PTFE, that have an excellent KOH resistance, were chosen. As these materials are often not the default seal materials on valves, it was important to find instruments with one of these materials as an option. Even if the chosen seal materials are resistant to KOH, it is important to make regular checks to detect possible fractures as soon as possible. Early detection of fractures and other seal defects helps to prevent the occurrence of leaks.

Some of the more challenging problems are still partially unsolved, such as the chemical waste disposal, and the existence of gas bubbles in liquid flows. The amount of chemical waste was greatly reduced as the scrubber units were removed, making chemical waste a smaller issue. Before the start of operation, thorough planning for its disposal would still be necessary, as the whole chemical waste problem has not vanished. Ideas for the disposal have already been discussed, as this could be done by dilution or by water evaporation.

As the electrolyte and the product gases come out of the stack from the same outlets, there will be a two-phase flow on the following pipelines. To get these two phases separated, the separator units are implemented. The efficiency of our separators is largely unknown, as there could be small gas bubbles at the bottom of the separator vessels, which was demonstrated to be the case in our tests. Different ways to reduce the amount of small gas bubbles at the bottom were introduced in the earlier chapter, but most likely even with the preventive measures taken, some of the gas bubbles will be escaping with the liquid electrolyte. The gas bubbles possess a threat, as the outlet lines from both separators lead to the same storage vessel, allowing the gases to mix. This mixing is to be avoided, as there is a possibility that the gases can form an explosive mixture. To neutralize possible explosive gas mixtures inside the storage vessel, a pipeline for nitrogen has been introduced into the storage tank, which could purge nitrogen gas in timed intervals to the possible gas mixture inside the storage tank. The storage tank would have an escape line for the gases to allow them to escape to the atmosphere. The same escape line could be used by the gas bubbles, reducing the likelihood of explosive mixtures.

Some design challenges have proved to be easier to fix than first expected. An example can be found on the KOH concentration transmitter, which was at first thought to be hard to implement. Mainly two different transmitters were looked at, which both had their own methods to indicate the KOH concentration. The first transmitter candidate was Valmet's Valmet Concentration Measurement 3300, which measures the flowing liquid media's conductivity and temperature, while the second transmitter candidate was Bronkhorst's mini CORI-FLOW M15, which would measure the KOH electrolyte density and temperature.

Valmet's transmitter would have a specific pre-configured recipe profiles that it could use, to directly show the KOH concentration from the measured conductivity and temperature values (Valmet, 2019, p. 2). While the transmitter did sound extremely good at first, it had its own problems if adapted into our application. These problems were related to the media's conductivity, as going beyond KOH concentration of 30 wt. %, the conductivity curve would start to go down, as it previously went upwards when concentration was increased. An illustrative chart for NaOH can be seen in Figure 20, which has the same conductivity problem at similar concentration values. This would mean that the transmitter would show us the correct KOH concentration up to 30 wt. % KOH, and then start to go down again.



Conductivity of NaOH at various temperatures.Concentration measurement cannot be used in the shadow area.

Figure 20. Conductivity chart for NaOH (Valmet, 2019, *Valmet Concentration Measurement 3300*).

There would have been a possibility to order a custom profile that would go a bit over 30 wt. % KOH upper limit, but this profile would have needed an increase in the media's temperature, which was already at the limit. As our concentration will most likely swing between 25–35 wt. % KOH while the test bench is in use, the idea of using this transmitter was discarded.

Bronkhorst's transmitter is a Coriolis type flow transmitter. Coriolis flow transmitters measure flow by directing the liquid flow through a vibrating pipe. Liquid flow will cause additional twist due to the fluid's inertia (Bronkhorst, 2023a), which can be measured to get the mass flow value. Such transmitters can measure the flowing media's density by measuring the vibrations frequency (Bronkhorst, 2023a), the denser the fluid, the lower the vibration frequency.

Unfortunately, this transmitter does not have a straight indicator on the flowing media's concentration, as it does not have programmed profiles to know what type of media is flowing through it. Instead, by checking the media's density and temperature values, it is possible to compare the given values to the common density values of KOH concentrations between 25–35 wt. % at the given temperature. In our application, this density value should be around 1.26 g/cm^3 (Akerlof & Bender, 1941, p. 4).

As this transmitter is mostly limited by temperature build-up (Bronkhorst, 2023b, p. 18), this transmitter was chosen to be used in our test bench. The transmitter's safe operating temperature was a concern, as the transmitter is self-heating due to its electrical components. Fortunately, Bronkhorst provides clear guidance on which temperatures their transmitters can be safely used, providing a chart with ranges for both the ambient and fluid temperatures. This chart can be seen in Figure 21.

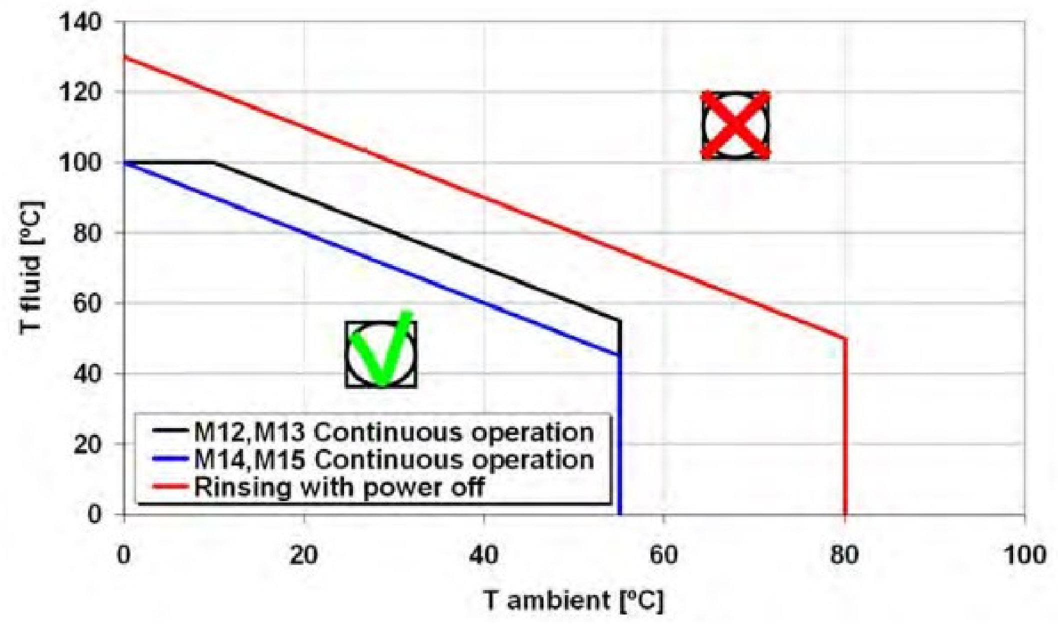


Figure 21. Temperature limits for Coriolis flow transmitter (Bronkhorst, 2023, *Instruction Manual mini CORI-FLOW™ M1x series Compact Coriolis Mass Flow Meters/Controllers for Liquids and Gases*).

Our transmitter is type M15, which means its continuous operation is safe below the blue line. Even if the ambient temperature of the laboratory room would go up to 30 degrees Celsius in the summer, the transmitter should still be fine, as the fluid temperature is kept at 70 degrees Celsius at maximum. Therefore, temperature limits for the transmitter did not cause an issue.

4 System safety

Hydrogen (H_2) gas is classified as extremely flammable and can easily ignite from heat and sparks (National Center for Biotechnology Information, 2023a). Hydrogen can also form explosive mixtures with air and has a lower flammable limit of 4% (National Center for Biotechnology Information, 2023b). As hydrogen is very easily ignited, it possesses serious safety hazards. Safety measures' main task is to make the likelihood of hydrogen fires as low as possible.

Potassium hydroxide (KOH) is classified as highly corrosive and it releases poisonous gas in fire, while potassium hydroxide itself does not burn (New Jersey Department of Health, 2010, p. 1). Potassium hydroxide has a 2 mg/m^3 workplace exposure limit (ThermoFisher Scientific, 2023, p. 5), above which it can cause lung damage when inhaled (New Jersey Department of Health, 2010, p. 2). Contact to skin and eyes causes severe irritation, burns and damage (LabChem, 2018, p. 1). To avoid any contact with the lye, the whole test bench will be in its own separate compartment.

As earlier mentioned, environments with higher oxygen levels are more susceptible to fire hazards, allowing pressurized oxygen gas to cause fires inside oxygen systems, if the instruments are not properly cleaned. Fire inside a pressurized oxygen pipeline can be caused by impurities, which have a low autoignition temperature. Autoignition could be caused by friction, particle impact, or by compression heating (Swagelok, 2017, p. 1). If the impurities with low autoignition temperatures catch fire, the rising temperature level will be able to ignite other materials in the pipeline as well. To avoid any impurities in the oxygen sides instruments, they should be specially cleaned for oxygen applications.

Safety analysis is an important part of the design work, as the test bench will operate with hazardous chemicals and flammable gases, which could cause harm to the equipment, the environment, and the operating personnel. Two different safety analyses were done to ensure the test bench's safe operation, layers of protection analysis and ATEX zone classification. Standards, such as SFS-EN IEC 61511-1:2017, SFS-EN IEC 61511-

2:2017 and SFS-EN IEC 60079-10-1:2021, were used to help us make these analyses, as they provide relevant guidance on these topics.

4.1 Layers of Protection Analysis

Layers of protection analysis (LOPA) is done to ensure the functional safety of our test bench. LOPA is done according to the European Standards EN 61511-1:2017 and EN 61511-2:2017, which have the status of a Finnish national standard, and provide guidance on how to identify management activities to ensure the test bench's functional safety. The standard states that the aim for LOPA is to find hazards and hazardous events of the process and the used equipment, find the events leading to these hazardous events, assess risks of these events, and find the safety functions to reduce their risks (Finnish Standards Association SFS ry, 2017a, s. 140). LOPA was documented on an excel sheet, and started by identifying the hazards that could be encountered while operating the test bench. These hazards can range from simple cases, like too high system pressure, to bit more complex cases, like fire inside the laboratory.

Each documented deviation or hazard in LOPA has a cause and a consequence, or even multiple of these. The causes include possible scenarios that could lead to the hazard, for example, a clogged valve could lead to an increase in system pressure. Different sections of the test bench can have different causes for the same hazard. Pressure increase could have been caused, in the regular hydrogen line, by a clogged valve, while in the nitrogen line, this could have been caused by a faulty pressure regulator. An example of a consequence for excessive pressure in the lines, could be damage to pressure sensitive components, such as the stack. Before the risk levels of each hazard can be evaluated, the likelihood of each hazard must be estimated (Finnish Standards Association SFS ry, 2017a, s. 140).

By defining each hazard's consequences and their likelihoods, the hazard's risks can be valued. The likelihood and the severity of consequences are further divided into three categories to estimate material, personnel and environmental risk of each hazard individually. This is done as some hazards might have more severe consequences for personnel than environment, while in some cases, events leading to damage for the environment could be more likely to happen than the events leading to damage for the operating personnel. A risk level for each of these subcategories ultimately decides if protective measures are necessary, and how many of them are needed to bring this risk level down. Protective measures can be mechanical, automated, or protective and preventive. Relief valves are an excellent example of an effective mechanical protective measure, which are used to prevent pressure levels from rising too much. Gas analyzers could be automated protective measures against gas mixing, while flushing the pipelines after operation could be a preventive measure against valve clogging. Protective measures aim to decrease the likelihood of hazardous events, bringing down the hazards' risk levels.

LOPA is done while the design progress of the test bench is ongoing, to ensure that the possible safety measures can be implemented to the test bench. After the test bench's design has been completed, it is important to evaluate whether a new and more in depth risk assessment would be needed (Finnish Standards Association SFS ry, 2017b, s. 24). As the test bench has been divided into different sections, such as the oxygen and hydrogen lines, each section is valued in the LOPA separately. This would mean that hazards, such as pressure and temperature increase, are analyzed and estimated multiple times in LOPA, each time for a different section of the test bench.

4.2 ATEX zone classification

As the test bench works with explosive and flammable gases, ATEX (atmosphèes explosibles) zone classification must be made to ensure the safe operation of the test bench. These space classifications decide if used electrical components need to have an ATEX certification. An instrument with an ATEX certification can be safely used in spaces that could contain explosive mixtures, whether it can be gases or liquids. If possible, our laboratory space should be in the non-hazardous category, as this would make working with our test bench safer. Our aim with ATEX zone classification is to then ensure good enough ventilation for the hydrogen side of the test bench, allowing explosive gases to be vented out before they can form an explosive mixture. ATEX zone classification was done according to the European standard EN IEC 60079-10-1:2021.

ATEX zone classification starts by defining possible sources of leakage, or “sources of release” (Finnish Standards Association SFS ry, 2021, s. 129). In our case, sources of release are different instruments that could leak hydrogen gas into the surrounding environment. Each instrument that contains explosive gases can, and should be, assumed to be a source of release, even if they are designed to be well sealed (Finnish Standards Association SFS ry, 2021, s. 20). After all the sources of release have been identified, it is important to know where this hydrogen gas could be diffused. In our case, there were two different sections for the hydrogen gas to leak: the emergency exhaust lines and the test bench’s fume hood.

Each source of release is categorized by the frequency of release. If the gas release would be continuous in normal conditions, the source of release is categorized as a continuous source. If the release of explosive gas has a possibility to happen in normal conditions, the source of release would be classified as a primary source. If the release of gas is not expected to happen in normal conditions, the source of release is classified as a secondary source. It is important to note that not every source of release that should not release gas in normal conditions is automatically a secondary source. Being secondary source also means that the leak duration is short, and the leak is quickly detected (Finnish Standards Association SFS ry, 2012, s. 11).

Each source of release will have a rate of release, which indicates how much of the explosive gas is released to the surroundings per second. This rate of release is calculated differently, depending on the type of released fluid. In our application, the released fluid is pressurized gas, and our system pressure goes higher than the European Standard’s critical pressure p_c. The standard provides an estimation for critical pressure which can be seen in equation 12. This equation can give a general approximation of critical pressures for the majority of gases.

$$p_c = 1.89 \times p_a \tag{12}$$

In equation 12, p_a indicates normal air pressure of 101 325 Pa. The standard also provides a more complex equation, if there is need to calculate the critical pressure more accurately. This given formula can be seen in equation 13.

$$p_c = p_a \left(\frac{\gamma+1}{2}\right)^{\frac{\gamma}{\gamma-1}} \tag{13}$$

In which γ is the index of adiabatic expansion. Both equations confirm that our system pressure is higher than the critical pressure, indicating that we need to calculate the release rate of gas with choked gas velocity. This rate of release can be calculated with equation 14.

$$W_g = C_d S p \sqrt{\gamma \frac{M}{ZRT} \left(\frac{2}{\gamma+1}\right)^{\frac{(\gamma+1)}{(\gamma-1)}}} \tag{14}$$

In which, C_d is the coefficient of discharge, S the cross section of the discharge hole, p gas pressure inside the instrument, M molar mass of the gas, Z compressibility factor of the gas, R the molar gas constant, and T the operating temperature.

Discharge hole size and source radius determine the release rate of hydrogen and are therefore the most significant factors to be estimated (Finnish Standards Association SFS ry, 2021, s. 39). For secondary sources, such as valve sealing elements, the standard provides a table meant to help estimate the cross-section size. For primary and continuous sources, the cross-section needs to be estimated by your own calculations.

In our test bench there were two possible locations for the release to discharge. Because of this, the sources of release are divided into two categories, as each discharge location will need its own degree of dilution calculations. Calculating the degree of dilution for each source of release is unnecessary, as it is enough to only consider the source with the largest rate of release for each category.

As the first discharge location is inside the fume hood, there will not be any sufficient natural sources of ventilation. To overcome the lack of ventilation, a centrifugal fan is used to create artificial ventilation. Dilution calculations were used to help us find the optimal fume hood size for a certain type of fan, as we wanted to have as much room as possible for us to locate the instruments containing hydrogen. This design choice was made to ensure there is enough room for future stacks and other possible instrument changes, like the separators, on the hydrogen side of the test bench. As the test bench was designed for two pressure values, the dilution calculations were done assuming it was already operating with 30 bar pressure.

Degree of dilution can be assessed by using a chart provided in the standard. The chart takes into consideration the calculated values for volumetric release characteristics of the source Q_c, and the ventilation velocity u_w. Needed volumetric release characteristics of the source can be calculated using equation 15.

$$Q_c = \frac{W_g}{\rho_g \times LFL} \tag{15}$$

In which, ρ_g indicates the gas density and LFL the lower flammable limit. Ventilation velocity is simply calculated by dividing the centrifugal fans volumetric flow, with the cross-section area of the fume hood. By calculating these two values, the standard's provided chart could be used to see the degree of dilution.

Just having high dilution is not enough, as for secondary sources, we would need at least a fair availability of ventilation. Availability of ventilation is classified as good, if the ventilation is continuous, without any disturbances. If the ventilation is present during normal operation but had discontinuity which lasted for a short time, the availability of ventilation is fair. As our centrifugal fan will always be on during the operation, it will most certainly fit both categories. As a precaution, our fan will be run through uninterruptible power supply, allowing it to be on during power outages as well.

In Table 3, we can see an example of one of the considered sources of release in our test bench. Each of the parameters in the table are calculated by the given equations or estimated by using the tables provided in the standard.

Table 3. Source of release considered in degree of dilution calculations.

Characteristic	**Value**
Source of release	Pipe fittings
Release location	Inside the fume hood
Grade of release	Secondary
Size of the release hole S (mm^2)	0.25
Rate of release (kg/s)	0.000373
Rate of release (m^3/s)	0.00415
Fluid temperature (°C)	0
Fluid pressure (bar)	30
Fluid phase	Gas

The pipe fittings are classified as secondary sources, as the fittings are not expected to leak hydrogen under normal conditions. Possible leaks from the fittings would also be quickly detected, allowing us to stop the operation of the test bench. Size of the possible release hole is graded to be 0.25 mm^2, as we are using Swagelok tube fittings. Swagelok tube fittings are classified as small bore connections, and by looking at table B.1 from the standard, the leak source size can be assessed to 0.25 mm^2 (Finnish Standards Association SFS ry, 2021, s. 149). The leak source could enlarge due to corrosion, which is why this value has been chosen.

Rate of release has been calculated with equation 14. In our application, coefficient of discharge is 0.75, and index of adiabatic expansion 1.41. Compressibility factor Z has been found out by using Excel's "solveZ" function. Volumetric rate of release has been calculated by dividing the previous rate of release with hydrogen gas density in NTP conditions.

Operation conditions for the gas, such as the temperature and pressure are assessed by worst case scenario. The hydrogen gas flowing through the pipe fittings will not be zero degrees Celsius, but this was used as a precaution. Pressure of 30 bar was chosen for reasons previously explained.

To find out the degree of dilution, volumetric release characteristics of the source were calculated by equation 15. A centrifugal fan was preselected to match a similar test bench's fan, which has a maximum volumetric flow rate of 1.078 m^3/s. As the velocity of ventilation in the ventilated space is calculated by dividing the volumetric flow with the cross-section area of the ventilated space, we could size our fume hood to match the fans limitations. Ventilation velocity u_w would change, as we would change the fume hood's cross-section area. To help us detect that we still are within high dilution limits, the chart seen in Figure 22 was used.

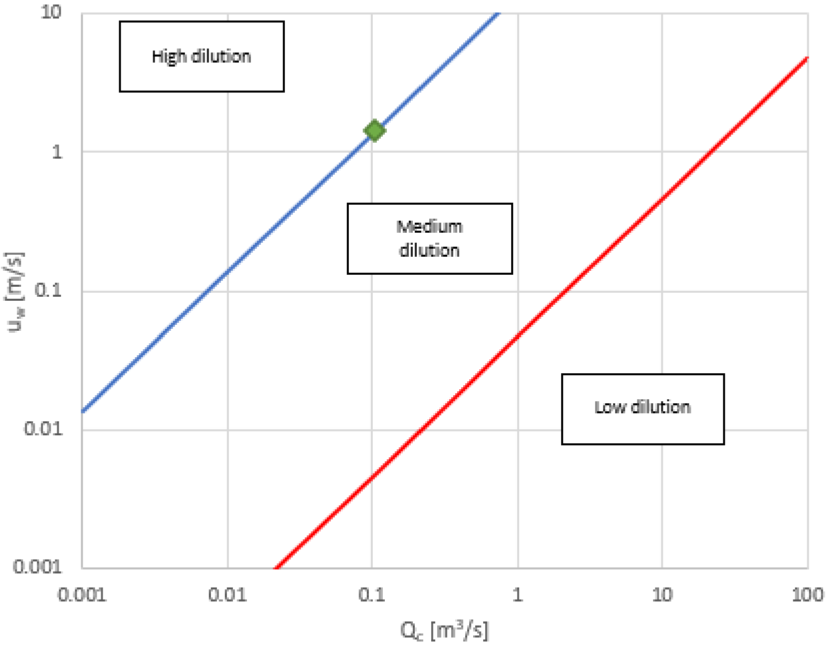


Figure 22. Chart for degree of dilution.

Volumetric release characteristics Q_c would stay the same, while u_w would change depending on the fume hood's size. By having volumetric flow rate of 1.078 m^3/s for the fan, we could size our fume hood's cross-section area to be 0.76 m^2.

Complete list of ventilation characteristics for the fume hood can be seen in Table 4. Zone classification has been done by using the standard's table D.1. Zone 2 NE is a theoretical zone, which is not considered under normal conditions (Finnish Standards Association SFS ry, 2021, s. 174)

Table 4. ATEX zone classification for the fume hood.

Characteristic	**Value**
Degree of dilution	High dilution
Ventilation availability	Good
Zone type	Non-hazardous (Zone 2 NE)

By having a high degree of dilution and good availability of ventilation, our test bench's fume hood is a non-hazardous zone, as can be seen in Table 4. The same calculations were done for the second discharge location. As the emergency exhaust lines lead the gases out of the laboratory building, the correct area to be addressed for zone classification is on the building's roof. As the building's roof is an open outdoor space, it differs the calculation a bit from earlier. Instead of calculating the ventilation velocity, the wind speed in calm weather is used as u_w for Figure 22. Following the standards table C.1 (Finnish Standards Association SFS ry, 2021, s. 53), a wind speed of 0.5 m/s was deemed to be the most suitable in our case. The same minimum wind speed can be classified to have good availability (Finnish Standards Association SFS ry, 2021, s. 52). As the degree of dilution was not high, and the low degree of dilution does not usually apply for outdoor spaces (Finnish Standards Association SFS ry, 2021, s. 54), the degree of dilution was classified as medium. Degree of dilution was also calculated, which put it at the same medium level. Release sources for the second calculation were classified as primary, making the outdoor space within Zone 1 + Zone 2 classification.

4.3 Protective measures for the test bench

The test bench has a lot of protective equipment to ensure its safe operation. Some of the more typical test bench instruments, such as pressure transmitters and check valves act as safety measures, that can reduce the risk levels in LOPA. Even though LOPA introduces many new potential hazards, not all the test bench's safety measures are implemented because of it. For example, some safety measures that are commonly used in VTT's other test benches, were already implemented to this test bench, such as nitrogen flushing.

The most important safety measures that were implemented because of LOPA include vessel liquid level control and drains for the lye. Main safety concern was the possibility of overflowing the separator and tank vessels, which would cause the lye to enter lines with open ends and sensitive instruments. Vessel level control was taken care of by implementing level switches with automated solenoid valves to the vessels. Drains in the test bench would differ from typical test bench drains, as we cannot release lye to the laboratory room’s own drains. From the test bench’s drain lines, lye could be collected into separate containers, from which the lye would be disposed of, or put back to the circulation.

Other notable safety measures that were implemented through LOPA are flow indicators and fire alarms. Flow indicators are especially needed in cooling lines, to make sure there is liquid flow to the heat exchangers. Without liquid flowing to the heat exchangers, we risk overheating the stack, and our gas drying would not work.

Like in many test benches, emergency purging for the gases is done with nitrogen. The test bench differs from others, as it needs another emergency flushing line to clean the pipeline from KOH. After shutdown, the test bench’s pipelines are flushed with purified water to eliminate the lye remains. If lye is not flushed from the pipelines, KOH can solidify as the water evaporates from the lye. An example of why this flushing is done can be seen in Figure 23, where leftover KOH from the stack manufacturer’s test runs has leaked from the stack inlets. This type of solidification could easily clog the check valves on the main pipelines, possibly leading to safety hazards, such as uncontrolled pressure increases.

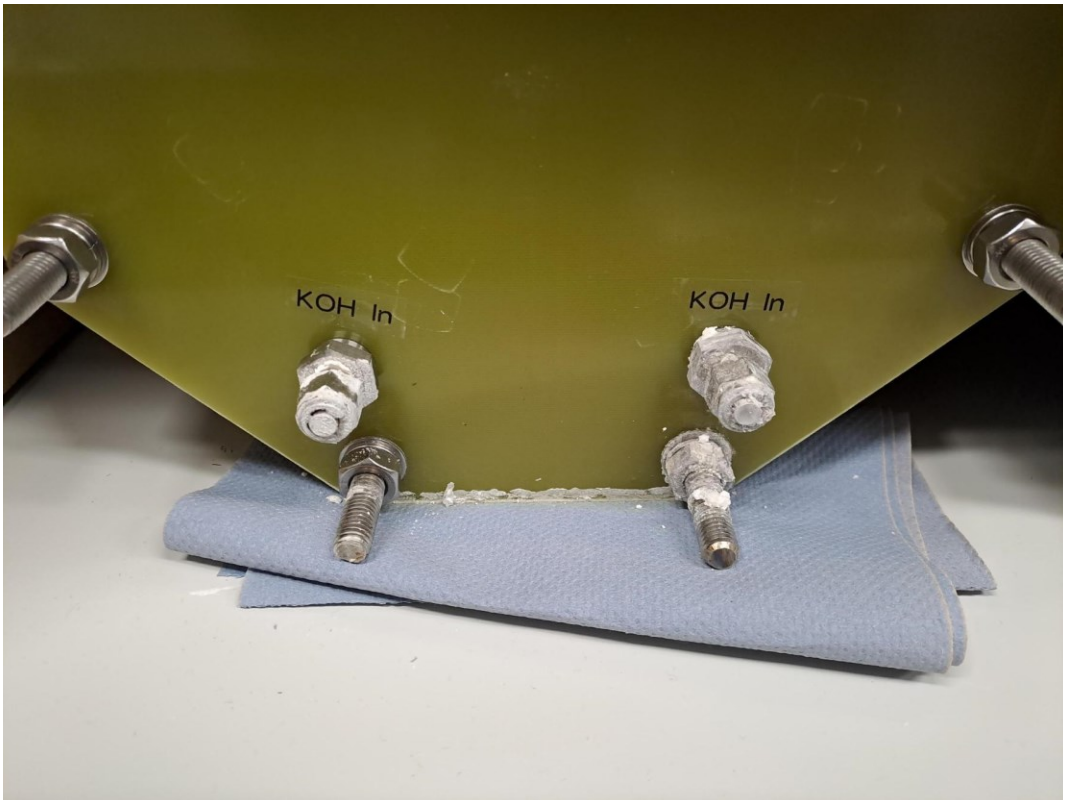


Figure 23. Solidified KOH on the stack inlets.

Gas analyzers are important instruments for the test bench's safety, as these detect if there are any explosive gas mixtures inside the pipelines. While the hydrogen side of the test bench is under continuous ventilation to avoid explosive mixtures inside the fume hood, nitrogen gas is responsible for eliminating the explosive mixtures inside the pipelines. Nitrogen gas will enter into the pipes during emergency shutdown, which will be triggered by the gas analyzers.

4.4 Emergency shutdown

Emergency shutdown can be triggered by a couple of important instruments. These instruments include the uppermost level switches on each separator and tank vessel, as these indicate that the vessel is going to overflow. The uppermost level switch has been located to the vessels by calculating the time it takes for the liquid level to rise from the highest acceptable value to the lines meant for gases only. Shutdown can also be triggered by the gas analyzers, if they detect that the allowed upper limit for mix of product gases has been exceeded.

Emergency shut down cuts the power to the stack and allows the emergency lines to open. The product gases will be neutralized by the nitrogen gas coming from the purge lines, and the system will be flushed with purified water. The circulation pump will continue its operation, as this will help us to cool the stack down. Similarly, the shutdown will open the valves regulating cold tap water, so that the lye cooler has access to water. Pressure in the system will be released, as the pressure controllers will lose power, and emergency exhaust lines will open, allowing gases to escape faster.

If the laboratory room lost access to power, the same operating procedure will start as in emergency shutdown. The valves controlling the emergency lines are chosen to be normally open, meaning that the valve will be fully open, if it does not receive any signal. Instruments that should continue operating, such as the circulation pump, are connected via UPS, to guarantee their operation for a certain amount of time after a power outage.

Emergency shutdown will also be triggered if any of the laboratory rooms own safety measures detect hazards. Such hazards could be a risen hydrogen content inside the laboratory room, or a possible fire alarm. Each test bench in the same room has its own fire alarm, to detect local fires as soon as possible. The laboratory room itself will have its own fire alarm as well, just in case a test bench fire alarm is faulty, or the fire takes place inside the room, rather than inside a certain test bench.

5 Future work

Before the test bench can be used for research purposes, it still needs to be assembled and tested. After this study, the test bench design has been completed, and the related safety analyses have been done. During the design phase, consideration for every instrument has been done, allowing us to now purchase the chosen instruments. Most of the crucial instruments have already been ordered, with the aim being to have everything ordered on time.

Some important missing components are the aluminum frame, where the test bench will be built, the separators, and the feed storage vessel. An aluminum frame would be needed as soon as possible, as the assembly work cannot fully start without it. The aluminum frame is custom ordered, which will be built and delivered to us according to our design needs. The design drawings for the separator vessels have been done, allowing us to start building these as soon as we get the needed materials.

To help us assemble the test bench, a 3D computer-aided design (CAD) model is being done of the test bench. The CAD model would help to plan the layout of the test bench. The CAD model could be improved as the assembly work is ongoing, as it is enough that the model would give an initial layout to start the assembly work.

Assembly work will start in Q1 of 2024 and the final ordered instruments should arrive at the same time. The test bench assembly can be delayed if some instruments are not delivered in time. Most likely the absence of a singular instrument would not cause any delays, as the missing instrument can be installed later. Delays could occur if the delivery is late for multiple instruments at the same time. Absence of a significant component, such as the storage vessel, could hinder the assembly work worse, but even the absence of these significant components should not be able to completely put a stop to the assembly work. Separator vessels will be built while the test bench assembly is ongoing, unless the vessels can be built before the aluminum frame arrives.

Electrical connections for each necessary instrument are already designed, before the assembly work starts. Some of the electrical work decisions have already been made during the test bench's design, such as which instruments would need to be run on UPS. Automation design for the test bench will also start early this year. Automation would include topics like pressure control and the automatic shutdown procedures.

After the test bench has been assembled, the test bench will be tested to ensure its safe operation. When green light for the safety has been show, the test bench can be set to run long term tests, which could measure the stack behavior at certain current densities, check the changes in hydrogen production rates, and estimate the stack durability and lifetime. Other tests could for example be accelerated stress tests, where the stack is put under changing conditions. Changes could be made to the stack power, temperature, and pressure mid-run. These tests would show us how stable our stack is with changing operating conditions. The stress test could for example simulate the alkaline stack's use in a grid, where power is received from fluctuating renewable sources.

6 Results & discussion

Alkaline electrolysis is the most mature water electrolysis technology, but its development is still important, as AWE has its own flaws which could be improved. AWE has already seen some major improvements, indicating that further development can, and should be made. To pair alkaline electrolysis with renewable energy sources, it is important to steady the fluctuating loads with different renewable sources.

To build an alkaline electrolysis test bench, it is important to know the most important balance of plant components for your test bench. In our application, the most important balance of plant components are the gas-liquid separators, lye storage tank, heat exchangers, and the circulation pump. To make sure our test bench’s instruments are suitable for the test bench application, they all need to be made from grade 316 stainless steel. Instruments on the hydrogen side of the test bench should be specifically designed for hydrogen use, ensuring that hydrogen embrittlement will not cause issues, and the valves are leak tight. Instruments on the oxygen side should be chosen to be variants with special cleaning, to avoid oxygen system fires.

Cooling down the lye is an important factor when designing the cooling for the test bench. Cooling down the lye makes sure that the used stack, or other important instruments, do not overheat. Overheating can lead to decreased lifetime for the instruments or outright break them. When designing the cooling lines of the test bench, it is important to note whether normal tap water could be enough for the heat exchange or if other instruments, such as chillers would be needed. In our application, tap water can be used for the lye cooling, but product gas drying would need a chiller unit for the cooling water.

Separator units can be custom ordered from their manufacturers if a certain level of separation efficiency is desired. Separator units could also be made inhouse, as in our design. When designing simple separator units, such as the ones used in this study, it is important to have monitored level control for the vessels, to make sure lye will not overflow. If scrubber units are desired, spray tower scrubbers look like the most promising option. Small spray tower scrubbers were hard to find, which makes it important to reserve enough time for the design and assembly work, if they are to be made inhouse.

When designing a test bench, it is important to start the necessary safety analyses early on, so that the changes can be made as early as possible, when the design work is still ongoing. Layers of protection analysis and ATEX zone classifications are important analyses to be done, to ensure the safe operation of the designed test bench. Layers of protection analysis helps to find all of the possible events that could lead to different safety hazards, while ATEX zone classification is done to ensure no explosions can be caused by the possibly leaked hydrogen. With ATEX zone classifications, it can be seen whether explosive mixtures can be prevented from forming altogether or if the used electrical instruments would need ATEX certifications, to guarantee there would not be sparks to ignite the possible explosive gas mixture. Both of these analyses have their corresponding standards that need to be followed.

As the safety analyses have been made, it is important to implement the necessary protective measures to the test bench. In this study, ATEX zone classification for the fume hood was seen to be non-hazardous, so we did not need to have ATEX certified instruments inside it. Other protective measures can come from LOPA, as in our case, the separator vessel level control. It is also important to include protective measures for the laboratory room where the test bench will be placed, to shut down the test bench if hazards are detected from other sources.

7 Conclusion

This study includes the necessary information, which VTT needs to build an alkaline water electrolysis test bench. Design work provides a pathway to reach the goals set for the test bench and has already fulfilled one of the test bench goals, by educating competent personnel inside VTT. Once the test bench is fully operational, this knowledge and education can be expanded outside of VTT.

Designing an alkaline water electrolysis test bench is not a simple task and cannot be done without any previous experience or guidance. Literature does not usually have complete P&I diagrams for confidentiality reasons, which makes designing a new P&I diagram extremely difficult. Having a P&I diagram of a similar system available was a crucial circumstance, as I did not have any P&I design experience beforehand. Guidance was also a crucial part for the safety aspect, as safety analyzes should have a high priority and done by competent personnel.

As the test bench still needs some work before it can be fully assembled and operational, it is likely that the test bench never reaches its final form. The design of the test bench is continuously evolving during the operation, as tests can expose possible design flaws or mistakes. During the operation, new ideas for simplifications or transmitters could come up and be added to the test bench, as we gain more experience in operating the test bench in question.

8 Summary

Alkaline water electrolysis is the most mature water electrolysis method. New water electrolysis methods have been developed to overcome the older technologies' weaknesses, causing AWE to lack behind their performance characteristics. As AWE is relatively cheap, has a long lifespan and it can be comfortably expanded to MW scale, there is a desire to develop AWE further. Low current density is one of the main flaws of AWE, and many developments, such as zero-gap configuration, focus on reducing the area specific ohmic resistance of the cell, resulting in higher current density values.

AWE suffers from gas impurities with partial loads, making AWE hard to pair with fluctuating renewable energy sources. Use of renewable energy sources could also mean an increase in shutdown and startup cycles, which tend to degrade the nickel electrodes, reducing their lifetime. Compared to the other water electrolysis methods, AWE also has a relatively high startup time. To use AWE with renewable energy sources, specific optimization for dynamic operation should be done.

To allow VTT to build an alkaline water electrolysis test bench, a complete P&I diagram design of the test bench was made. The test bench could be run with different stacks, allowing interested companies to develop their products and knowledge. The test bench was designed to have a secondary set of parameters for the future stacks, allowing us to choose instruments that could be used for both the future and current operating parameter sets. The current design parameters for the test bench are determined by the stack provided to us by Lightbridge.

Two different parameter sets proved to be problematic, when certain instruments were chosen and designed, such as the separator vessels. The most limiting factor of the designed test bench is its design pressure limit. Most of the necessary instrument changes to the test bench can be avoided if the possible new stacks will run on similar pressure levels.

The safe operation of the test bench was guaranteed by making two different safety analyses, one for the protective layers of the test bench, and the other to check ATEX zone classifications for our test bench. Done layers of protection analysis allowed us to see possible safety hazards of the test bench, their consequences, and precautionary measures. ATEX zone classification helped us to size our test bench's fume hood, so that it will stay at the non-hazardous zone.

The test bench will still need a 3D model layout, after which the test bench assembly can be started. Common alkaline water electrolysis design challenges have become familiar to VTT during this design process, and this knowhow of alkaline electrolysis will continue to grow, as the test bench is assembled and operational. This gathered knowhow will expand outside of VTT, as different companies can start utilizing the results gained from the test bench.

Sources

Akerlof, G. & Bender, P. (1941). The Density of Aqueous Solutions of Potassium Hydroxide. *Journal of the American Chemical Society*, *63*(4), 1085–1088. https://doi.org/10.1021/ja01849a054 [Restricted availability]

Brauns, J. & Turek, T. (2020). Alkaline Water Electrolysis Powered by Renewable Energy: A Review. *Processes, 8*(2), 248. https://doi.org/10.3390/pr8020248

Bronkhorst. (2023a). *Coriolis flow meters: how do they work?*. Retrieved 28.12.2023 from https://www.bronkhorst.com/int/service-support-1/technologies/coriolis-mass-flow-measuring-principle/

Bronkhorst. (2023b). *Instruction Manual mini CORI-FLOW™ M1x series Compact Coriolis Mass Flow Meters/Controllers for Liquids and Gases*. Retrieved 28.12.2023 from https://www.bronkhorst.com/int/products/liquid-flow/mini-cori-flow/?page=1#downloads

Bronkhorst. (n.d.). *EL-FLOW Select Digital Thermal Mass Flow Meters and Controllers for Gases*. Retrieved 28.12.2023 from https://www.bronkhorst.com/int/products/gas-flow/el-flow-select/?page=1#downloads

Carmo, M., Fritz, D., Mergel, J., & Stolten, D. (2013). A comprehensive review on PEM water electrolysis. *International Journal of Hydrogen Energy, 38*(8), 4901–4934. https://doi.org/10.1016/j.ijhydene.2013.01.151 [Restricted availability]

Colli, A., Girault, H. & Battistel, A. (2019). Non-Precious Electrodes for Practical Alkaline Water Electrolysis. *Materials, 12,* 1336. https://doi.org/10.3390/ma12081336

David, M., Ocampo-Martínez, C. & Sánchez-Peña, R. (2019). Advances in alkaline water electrolyzers: A review. *Journal of Energy Storage, 23,* 392–403. https://doi.org/10.1016/j.est.2019.03.001 [Restricted availability]

Dwivedi, S. & Vishwakarma, M. (2018). Hydrogen embrittlement in different materials: A review. *International Journal of Hydrogen Energy, 43,* 21603–21616. https://doi.org/10.1016/j.ijhydene.2018.09.201 [Restricted Availability]

Ehlers, J., Feidenhans'l, A., Therkildsen, K. & Larrazábal, G. (2023). Affordable Green Hydrogen from Alkaline Water Electrolysis: Key Research Needs from an

Industrial Perspective. *ACS Energy Letters, 8*(3), 1502–1509. https://doi.org/10.1021/acsenergylett.2c02897

Finnish Standards Association SFS ry (editor). (2012). *SFS-käsikirja 59 Räjähdysvaarallisten tilojen luokittelu: palavat nesteet ja kaasut.*

Finnish Standards Association SFS ry. (2017a). *Functional safety – Safety instrumented systems for the process industry sector – Part 1: Framework, definitions, system, hardware and application programming Requirements*. (SFS 61511).

Finnish Standards Association SFS ry. (2017b). *Functional safety – Safety instrumented systems for the process industry sector – Part 2: Guidelines for the application of IEC 61511-1*. (SFS 61511).

Finnish Standards Association SFS ry. (2021). *Explosive atmospheres. Part 10-1: Classification of areas. Explosive gas atmospheres*. (SFS 60079).

Henkensmeier, D., Najibah, M., Harms, C., Žitka, J., Hnát, J. & Bouzek, K. (2020). Overview: State-of-the Art Commercial Membranes for Anion Exchange Membrane Water Electrolysis. *Journal of Electrochemical Energy Conversion and Storage, 18*(2). https://doi.org/10.1115/1.4047963

Hu, S., Guo, B., Ding, S., Yang, F., Dang, J., Liu, B., Gu, J., Ma, J. & Ouyang, M. (2022). A comprehensive review of alkaline water electrolysis mathematical modelling. *Applied Energy, 327*. https://doi.org/10.1016/j.apenergy.2022.120099 [Restricted availability]

IEA. (2023). *Critical Minerals Data Explorer*. Retrieved 28.12.2023 from https://www.iea.org/data-and-statistics/data-tools/critical-minerals-data-explorer

IRENA. (2020). *Green hydrogen cost reduction*. Retrieved 28.12.2023 from https://www.irena.org/publications/2020/Dec/Green-hydrogen-cost-reduction

Katz, E. (2021). Electrochemical contributions: William Nicholson (1753–1815). *Electrochemical Science Advances, 1*. http://dx.doi.org/10.1002/elsa.202160003

Kojima, H., Nagasawa, K., Todoroki, N., Ito, Y., Matsui, T. & Nakajima R. (2023). Influence of renewable energy power fluctuations on water electrolysis for green hydrogen production. *International Journal of Hydrogen Energy, 48,* 4572–4593. https://doi.org/10.1016/j.ijhydene.2022.11.018

Kumar, S. & Himabindu, V. (2019). Hydrogen production by PEM water electrolysis – A review. *Materials Science for Energy Technologies, 2,* 442–454. https://doi.org/10.1016/j.mset.2019.03.002

Kumar, S. & Lim, H. (2022). An overview of water electrolysis technologies for green hydrogen production. *Energy Reports, 8,* 13793–13813. https://doi.org/10.1016/j.egyr.2022.10.127

LabChem. (2018). *Potassium Hydroxide Safety Data Sheet*. Retrieved 28.12.2023 from https://www.labchem.com/tools/msds/msds/LC19190.pdf

Lightbridge. (n.d.). *Alkaline Water Electrolyzer Stack Install Guide (LBE-30C-F) User Manual*.

Miller, H., Bouzek, K., Hnat, J., Loos, S., Bernäcker, C., Weißgärber, T., Röntzsch, L. & Meier-Haack, J. (2020). Green hydrogen from anion exchange membrane water electrolysis: a review of recent developments in critical materials and operating conditions. *Sustainable Energy & Fuels, 4,* 2114–2133. https://doi.org/10.1039/C9SE01240K

NASA. (2023). *NASA Announces Summer 2023 Hottest on Record*. Retrieved 28.12.2023 from https://www.nasa.gov/news-release/nasa-announces-summer-2023-hottest-on-record/

National Center for Biotechnology Information. (2023a). PubChem Compound LCSS for CID 783. *Hydrogen*. Retrieved 28.12.2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Hydrogen#datasheet=LCSS&section=Fire-Hazards

National Center for Biotechnology Information. (2023b). PubChem Annotation Record. *Hydrogen*. Retrieved 28.12.2023 from https://pubchem.ncbi.nlm.nih.gov/source/hsdb/5026#section=Flammable-Limits

Nel Hydrogen. (2021). *Alkaline and Proton PEM Electrolysers*. Retrieved 28.12.2023 from https://nelhydrogen.com/resources/electrolysers-brochure/

New Jersey Department of Health. (2010). *Hazardous Substance Fact Sheet: POTASSIUM HYDROXIDE*. Retrieved 28.12.2023 from http://www.nj.gov/health/eoh/rtkweb/documents/fs/1571.pdf

Ni, M., Leung, M. & Leung, D. (2008). Technological development of hydrogen production by solid oxide electrolyzer cell (SOEC). *International Journal of Hydrogen Energy, 33,* 2337–2354*.* https://doi.org/10.1016/j.ijhydene.2008.02.048 [Restricted availability]

Office of Energy Efficiency & Renewable Energy. (2021). *Hydrogen Production: Electrolysis*. Retrieved 28.12.2023 from https://www.energy.gov/eere/fuelcells/hydrogen-production-electrolysis

Park, J., Kang, S., Oh, S., Kim, J., Lim, M., Ahn, C., Cho, Y. & Sung, Y. (2019). High-performance anion-exchange membrane water electrolysis. *Electrochimica Acta, 295,* 99–106. https://doi.org/10.1016/j.electacta.2018.10.143 [Restricted availability]

Phillips, R., Edvards, A., Rome, B., Jones, D. & Dunnill, C. (2017). Minimising the ohmic resistance of an alkaline electrolysis cell through effective cell design. *International Journal of Hydrogen Energy, 42,* 23986–23994. https://doi.org/10.1016/j.ijhydene.2017.07.184 [Restricted availability]

Phillips, R. & Dunnill, C. (2016). Zero gap alkaline electrolysis cell design for renewable energy storage as hydrogen gas. *RCS Advances, 6,* 100643–100651. https://doi.org/10.1039/C6RA22242K [Restricted availability]

Pressure Equipment Directive 17.7.2014/68. EUR-Lex. Retrieved 28.12.2023 from http://data.europa.eu/eli/dir/2014/68/2014-07-17

Rizwan, M., Alstad, V. & Jäschke, J. (2021). Design considerations for industrial water electrolyzer plants. *International Journal of Hydrogen Energy, 46,* 37120–37136. https://doi.org/10.1016/j.ijhydene.2021.09.018

Sandeep, K., Kamath, S., Mistry, K., Kumar, A., Bhattacharya, S., Bhanja, K. & Mohan, S. (2017). Experimental studies and modeling of advanced alkaline water electrolyser with porous nickel electrodes for hydrogen production. *International*

Journal of Hydrogen Energy, 42, 12094–12103. https://doi.org/10.1016/j.ijhydene.2017.03.154 [Restricted availability]

Smolinka, T., Bergmann, H., Garche, J. & Kusnezoff, M. (2022). Chapter 4 - The history of water electrolysis from its beginnings to the present. *Electrochemical Power Sources: Fundamentals, Systems, and Applications,* 83–164, https://doi.org/10.1016/B978-0-12-819424-9.00010-0 [Restricted Availability]

Swagelok. (2017). *Oxygen System Safety*. Retrieved 28.12.2023 from https://www.swagelok.com/downloads/webcatalogs/En/ms-06-13.pdf

ThermoFisher. (2023). *SAFETY DATA SHEET*. Retrieved 28.12.2023 from https://www.fishersci.se/shop/products/potassium-hydroxide-ca-85-analysis-pellets-thermo-scientific/10575355

Tee, H., Park, H., Shah, P., Trindell, J., Sugar, J. & Kohl, P. (2022). Self-Adhesive Ionomers for Alkaline Electrolysis: Optimized Hydrogen Evolution Electrode. *Journal of The Electrochemical Society, 169*. https://doi.org/10.1149/1945-7111/acab8a [Restricted availability]

Ursúla, A., San Martín, I., Barrios, E. & Sanchis, P. (2013). Stand-alone operation of an alkaline water electrolyser fed by wind and photovoltaic systems. *International Journal of Hydrogen Energy, 38,* 14952–14967. https://doi.org/10.1016/j.ijhydene.2013.09.085 [Restricted availability]

Valmet. (2019). *Valmet Concentration Measurement 3300*. Retrieved 28.12.2023 from https://www.valmet.com/automation/analyzers-measurements/conductivity-kemotron/inline-concentration-measurements/

Wood, M., Hailwood, M. & Koutelos, K. (2021). Reducing the risk of oxygen-related fires and explosions in hospitals treating Covid-19 patients. *Process Safety and Environmental Protection, 153,* 278–288. https://doi.org/10.1016/j.psep.2021.06.023

Zhang, K., McDonald, M., Genina, I. & Hammond, P. (2018). A Highly Conductive and Mechanically Robust OH^- Conducting Membrane for Alkaline Water Electrolysis. *Chemistry of Materials, 30*(18), 6420–6430. https://doi.org/10.1021/acs.chemmater.8b02709 [Restricted Availability]