

ANALYSIS OF PARALLEL COUPLING OF CHLOR-ALKALI AND ALKALINE WATER ELECTROLYSIS PROCESS WITH COMBINE CATHODE FLUID CIRCULATION

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ABSTRACT

Hydrogen is almost ideal fuel in reducing emissions without CO, CO₂, SOX or NOX. Hydrogen can be used through the combustion or Fuel Cell processes so that emissions are low. One of the hydrogen production processes is through electrolysis, both Chlor-Alkali (CA) and Alkaline Water Electrolysis (AWE). Both processes contained a NaOH solution in the CA and catalyst results in AWE. In this test, a combination of CA and AWE processes will be carried out in the fluid circulation at the cathode of two processes. From this study it was found that there was an increase of 11 percent by weight of gas when CA and AWE cathode fluid were circulated in parallel compared to the CA and AWE processes respectively.

Keywords: Hydrogen, Chlor-Alkali, Alkaline Electrolysis, Parallel Electrolysis, NaOH Circulation

1 INTRODUCTION

Hydrogen is an almost ideal fuel for reducing smog when burned. Hydrogen contains no carbon or sulfur, so no CO, CO₂ or SO_x or soot is produced during combustion (White et al., 2006). Hydrogen enables leaner combustion, resulting in lower combustion temperatures and very low NO_x emissions. Hydrogen is non-toxic, so unburned hydrogen will not pose an immediate health risk (Sapountzi et al., 2017). In terms of emission gas reduction when used electrochemically or in the fuel cell process, Hydrogen in the fuel cell produces zero harmful emissions where nitrogen is completely removed due to low operating temperatures (175 F; 80 C) (Fan et al., 2021).

One of the most widely initiated hydrogen production plans is in offshore rig conditions. The hydrogen production process on offshore rigs has been proposed by D'Amore-Domenech et al., (2018) and (Meier, 2014) where this process is suggested for green hydrogen production on offshore refineries using unused rigs and assisted by wind turbines as a source of energy. In rig conditions that are equipped with seawater purification equipment, electrolysis equipment for Chlor-Alkali and water electrolysis combine process to produce more result. Processed products such as

NaOH, Chlorine and Hydrogen can be directly distributed via ships directly connected to modified rigs. However, this idea is not without drawbacks, because the costs incurred must be calculated again according to annual production results and investment capital (IRENA, 2020).

One method of water electrolysis that has been developed is alkaline electrolysis using the ZeroGap model (Buttler & Spliethoff, 2018). Hydrogen production using the ZeroGap model is a technology that has been developed commercially in the chlor-alkali industry. Thyssenkrupp (2019) has developed this type of electrolyzer to be used in Green Hydrogen production at NEOM Future City in Saudi Arabia (Salameh et al., 2021). Alkaline water electrolysis works at a temperature of 30-80°C with a liquid solvent (Potassium Hydroxide-KOH / Sodium Hydroxide-NaOH). The concentration of the electrolyte used ranges from 20-30% by many industries (D'Amore-Domenech et al., 2018). The process of breaking and joining molecules in alkaline electrolysis occurs with the help of anode and cathode which are powered by DC electricity. The anode, cathode, and membrane also help separate the potential mixing of H₂ and O₂ (Kumar et al., 2021).

Chlor-alkali production is an electrolysis process used to produce chemical compounds that

are widely used in industry. Chlorine, hydrochloric acid, sodium hydroxide, and hydrogen are the products produced by this process. Chlorine and sodium hydroxide are important commodities used in various applications. These two types of compounds support more than 55% of the turnover of the European chemical industry. Until 2012, hydrogen produced from chlor-alkali plants was partially wasted and released into the atmosphere. If recovered and utilized it can play an important role in the heating process. Some plants operate with 47% of the hydrogen produced used in the production of HCl (hydrochloric acid), 10% is used to control the pressure differential, and the rest is not used (i.e. 43% of the hydrogen is discharged into the atmosphere). (Khasawneh et al., 2019)

This research will focus on the analysis of hydrogen mass production by combining the Chlor-Alkali process and water electrolysis. In addition, a gradual increase in the NaCl solution in Chlor-Alkali will be tested to simulate the changing of sea water conditions so that the effect hydrogen mass can be analyzed.

2. MATERIALS AND METHODS

2.1 Theory

The research plan is carried out by making an electrolyzer model. The selected model is the ZeroGap model which was first used in 1959 by General Electric. This model has been used

commercially in the Chlor-Alkali (CA) process in the basic chemicals industry. According to Paidar et al., (2016), currently the CA electrolyzer manufacturer is a pioneer in developing a tool similar to the Alkaline Water Electrolysis (AWE) process through Alkaline fluids on a large scale. The companies that develop membranes for CA will become early pioneers in accelerating Hydrogen Economy.

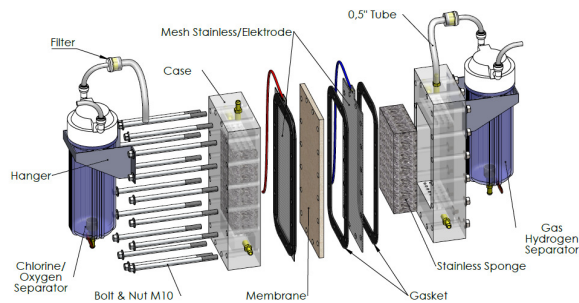


Figure 1. Design Electrolyzer

The product from CA, namely NaOH will be reused in the AWE process as a solvent and reaction catalyst. Hydrogen gas resulting from the CA and AWE processes will be captured in a small tube (Haryati et al., 2014). In this experiment, there is a membrane separator to separate the gas content from electrolysis results. Other gas products from CA such as chlorine will be released into the air as well as gas products from AWE, namely oxygen.

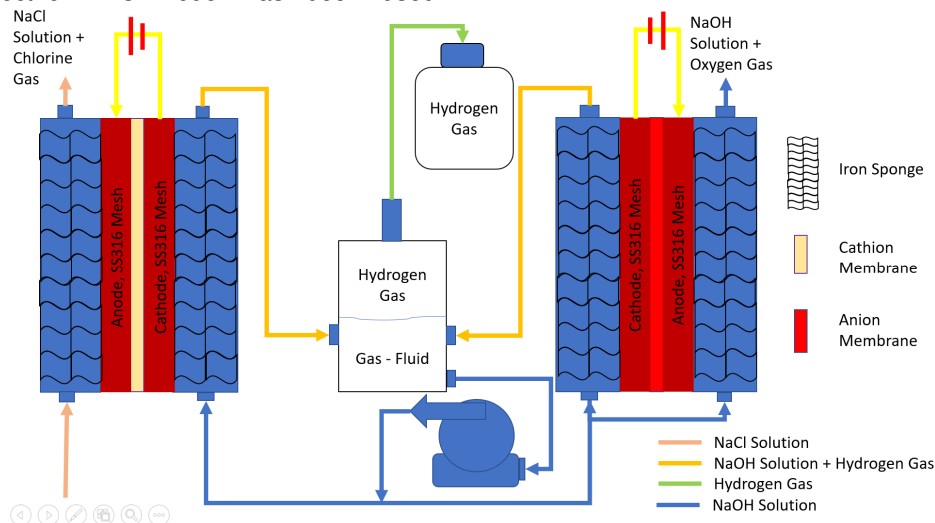


Figure 2. Parallel Coupling of CA and AWE process with combine cathode fluid circulation (Lakshmanan & Murugesan, 2014)

NaOH, with its properties as a solvent, can also be used as an electrolyte in the alkaline electrolysis process (Pein et al., 2021). Hydroxide (OH⁻) will increase the pH of the water, making the electrolysis conditions alkaline. Due to its nature as a solvent, NaOH itself will not experience a

significant reduction in quantity so it can still be collected as an industrial product.

Gas product measurements are carried out using scales which calculate the weight at 10 minutes intervals. The basic weight calculation is carried out in accordance with mass balance.

$$\sum \dot{m}_{inlet (fluid)} = \sum \dot{m}_{outlet (fluid)} + \sum \dot{m}_{outlet (gas)} \quad (1)$$

Based on Faraday's law, the weight of the gas result from electrolysis will be directly proportional to the electrons used. Therefore, in theory we can estimate the maximum value that can be captured based on the current flowing.

$$\text{Faraday Law} \quad w = \frac{e.i.t}{F} \quad (2)$$

$$\text{Electron Mole} \quad n(elektron) = \frac{w}{e} \quad (3)$$

2.2 Material & Equipment

Tool setup was carried out using Stainless Steel Mesh 400 as electrodes, iron spoons to expand the area of the electrodes, membranes (anions and cations) which were made manually based on Qiao et al., (2010) for Anion Membrane and Shakir et al., (2017) for Cation Membrane. The water flow will be assisted by a pump with a flow rate of 3.6 liters per minute at a pressure of 3 Bar. The electricity for this equipment uses DC 5V at a current of 40 A for 1 set of tools.

Table 1. Matrix of Experiment

No.	Sample Scheme	Sample ID
1	AWE when the electrode uses mesh only. NaOH 10%	A1
2	AWE when electrodes use mesh & spoons. NaOH 10%	A2
3	CA research without AWE. NaCl 10%+NaOH 10%	A3
4	Combination of CA and AWE. NaCl 10% + NaOH 10%	B1
5	Combination of CA and AWE. NaCl 20% + NaOH 10%	B2
6	Combination of CA and AWE. NaCl 30% + NaOH 10%	B3

The research was carried out on each sample A1, A2, and A3 using 1 power supply 5VDC, 40 A. The combine process of Electrolyzer for Chlor-Alkali and water electrolysis will be using 2 sets of 5VDC and 40A power supplies. This combination process is given sample numbers B1, B2 and B3 according to the increase in NaCl levels.

2.3 Research procedure

Before each experiment carried out, the electrolyzer will be run for 1 minute to collect the initial gas as well as clean up the remaining results from the previous research. The initial gas captured was subjected to destructive tests by burning it to check the research results, namely hydrogen without a mixture of oxygen or chlorine.

Hydrogen obtained from both Chlor-Alkali and water electrolysis is collected in a tube and

weighed every 10 minutes for 1 hour (T0, T1, T2, ... T6). The production results of other compounds such as NaOH, Chlorine and Oxygen were not calculated (ignored). The recorded operational data will be used as the basis for the analysis of this research. Testing was carried out with the sample sequence A1, A2, A3, B1, B2, and B3 according to the respective sample variations.

Before the research process, a destructive test was carried out when setting up the tool. The aim is to test the existence of hydrogen as an alternative fuel. The research gas is given fire and heat as conditions for the fire triangle (along with fuel, H₂). In current research, it is proven that the gas obtained is hydrogen fuel as seen in Figure 3.

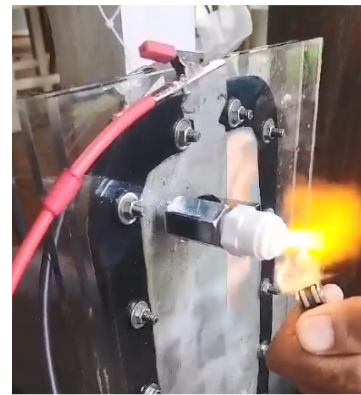


Figure 3. Gas Destructive Test, to test the presence of Hydrogen Gas.

3. RESULTS AND DISCUSSION

3.1 Research result

The research was carried out by taking several samples which were tabulated according to the order of the research samples. Research time data was recorded to see initial temperature data before the research. Changes in temperature also occur as research time increases. Gas data recorded per 10 minutes interval is accumulative from the start of the study.

Table 2. Experiment result of Sample

Sample ID	Interval	Time	Weight (gr)	Temperature (°C)
A1	T0	16:20:16	0,00	26,2
	T1	16:30:25	0,14	45,0
	T2	16:40:46	0,30	54,3
	T3	16:51:26	0,47	61,4
	T4	16:01:20	0,67	67,5
	T5	17:11:24	0,84	69,9
	T6	17:22:33	1,01	71,2
A2	T0	9:20:06	0,00	26,2
	T1	9:30:20	0,21	45,0
	T2	9:40:36	0,43	54,3
	T3	9:51:22	0,68	61,4
	T4	9:01:30	0,90	67,5

Table 2. Experiment result of Sample

Sample ID	Interval	Time	Weight (gr)	Temperature (°C)
A3	T5	10:11:22	1,13	72,6
	T6	10:22:03	1,35	74,8
	T0	11:32:00	0,00	26,2
	T1	11:41:13	0,21	49,5
	T2	12:52:05	0,43	59,7
	T3	12:02:21	0,67	67,5
	T4	12:11:46	0,92	74,3
B1	T5	12:22:22	1,19	79,9
	T6	12:32:03	1,44	82,3
	T0	15:23:00	0,00	26,2
	T1	15:33:20	0,43	49,5
	T2	15:43:32	0,89	59,7
	T3	15:53:32	1,37	67,5
	T4	16:03:32	1,91	74,3
B2	T5	16:13:32	2,54	76,9
	T6	16:22:32	3,14	78,3
	T0	9:10:00	0,00	24,2
	T1	9:20:00	0,44	50,0
	T2	9:30:33	0,90	60,3
	T3	9:41:02	1,40	68,1
	T4	9:50:48	1,95	75,0
B3	T5	10:01:22	2,47	77,1
	T6	10:12:03	2,97	78,1
	T0	11:40:50	0,00	28,2
	T1	11:51:23	0,46	52,0
	T2	12:01:05	0,95	62,7
	T3	12:12:25	1,49	70,9
	T4	12:21:56	2,01	78,0
	T5	12:32:22	2,52	80,2
	T6	12:42:00	3,02	81,2

3.2.1 Hydrogen Gas Production

The increase in hydrogen production in the research was very clear, samples A2 and A3 which were tested separately had different production results when compared to the results from Research B1. There is a yield difference of close to **11%** in gas mass. The second difference in research lies between the temperature fluctuation between each sample.

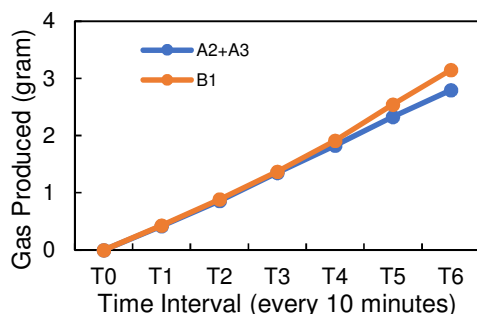


Figure 4. Variation in hydrogen yield under CA and AWE conditions at 40 A each compared to CA and AWE where the cathode solution was combined.

The initial hypothesis that there was an increase in the amount of dissolved OH⁻ which increased from the results of combining of cathode fluid in CA and AWE simultaneously (Sapountzi et al., 2017). From Figure 4, it can be seen that the increase in production occurred with the combination fluid cathode of CA and AWE by each experiment interval.

3.2.2 Electrode Area Expansion

Figure 5 shows the differences from samples A1, A2, and A3, where research conditions were carried out with the same solvent of 10% by mass of water. It can be seen that there has been an increase of more than 30% in the mass of gas produced. The additional area provides more space for hydrogen to form at the cathode with unchanged current and voltage (Hodges et al., 2022).

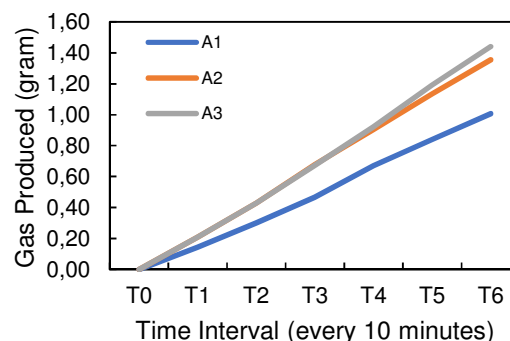


Figure 5. The difference in hydrogen gas production with the addition of spoons of iron as an expansion of the electrode area.

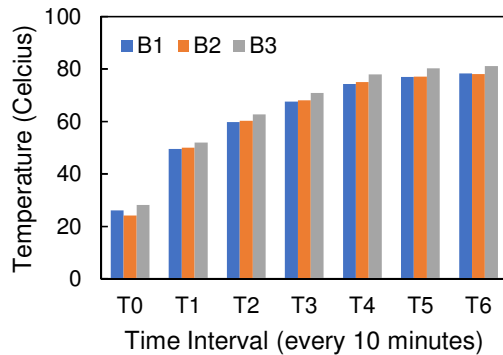
Figure 5 also shows that sample A3 at 10% NaCl and 10% NaOH has a greater increase in mass when compared to sample A2. It can be concluded that the reaction speed of sample A3 is faster than A2, which has a solubility of NaOH of 10% per 1,000 liters. Meanwhile, in sample A3, two types of fluids were circulated, 10% NaCl in 1,000 liters of water and 10% NaOH in 1,000 liters of water.

3.2.3 Temperature Control

Figure 6 shows a rapid increase in the temperature of the test equipment. The trend of increasing temperature goes significantly up to the T4 interval or at around 70 degrees Celsius in proportion to the speed of reaction gas production (Buelvas et al., 2014). Different things occur at temperatures of 70 degrees Celsius and above, where there is a decrease in the mass of gas captured in the final tube.

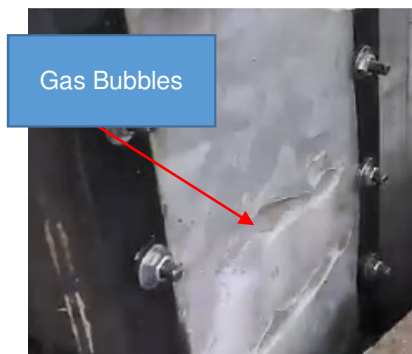
Table 3. Fluctuation mass of Gas at different NaCl concentration

Interval	B1 v B2 (gr)	B1 v B3 (gr)
T0	0,00	0,00
T1	0,02	0,07
T2	0,02	0,07
T3	0,02	0,11
T4	0,02	-0,04
T5	-0,18	-0,19
T6	-0,17	-0,17

**Figure 6.** Simultaneous Temperature Increase in the CA-EA Process.

The potential decrease in gas production occurs at different temperatures depending on the concentration of the dissolved NaCl solution. Sample B3 experienced a faster decline in production, followed by samples B2 and B1. From the Figure 6, a decrease in production occurs at temperatures above 70 degrees Celsius.

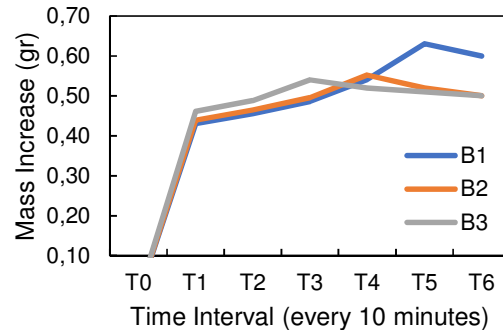
In table 3, the mass increase values between B1-B2 and B1-B3 increase every 2 to 3% per every 10% increase in NaCl. The increase occurs per each electrolyte solution up to the saturation temperature level as shown in figure 6. It is necessary to pay attention to the increase in the percentage of gas mass compared to the electrolyte mass variations.

**Figure 7.** Gas bubbles trapped at temperatures above 70 degrees Celsius

The phenomenon of decreased production also seen by the accumulation of gas bubbles on several parts of the tool surface. In the interval T1 to T3, the bubble gas that accumulates on the surface of the tool wall will flow out quickly at the outlet at the top. But the gas bubbles are increasingly accumulating as the temperature increases above 70 °C. This may occur due to pump performance which has decreased at temperatures beyond its capacity limits.

3.2.4 Gas Bubble Trap

Referring to Figure 8, there is a decrease in the speed of gas mass production with the variation of NaCl from sample B1, B2, and B3. This shows that the membrane is made to work, which means that there is an addition of (OH⁻) ions in the cathode area. The reaction speed of the gas in the electrode cannot be matched by the circulation of the fluid and gas transfer rates.

**Figure 8.** The decrease in hydrogen mass (B1, B2, B3) caused by the performance of the bubble trap and pump limit.

A bubble trap phenomenon occurred at intervals T6 in sample B1, T5 in sample B2 and T4 in sample B3 as shown in table 4. There was a time difference of 10 minutes between samples in the process of decreasing gas mass accumulation. The gas products that accumulate on the electrode surface result in less gas because the reaction area is filled with gas bubbles that remain on the electrode. In sample B3 which had the most NaCl content in the CA process, the mass loss occurred faster and sequentially in samples B2 and B1.

Table 4. Increase of gas mass per interval

	B1 (gr)	B2 (gr)	B3 (gr)
T0	0,00	0,00	0,00
T1	0,43	0,44	0,46
T2	0,46	0,47	0,49
T3	0,49	0,50	0,54
T4	0,54	0,55	0,52
T5	0,63	0,52	0,51
T6	0,60	0,50	0,50

This phenomenon has been described by Atsushi & Nagakazu, (1997) as a bubble trap. Gas reaction speed that does not match the release of gas from the electrode. The increase in temperature causes the performance of the pump to circulating the working fluid and gas discharge to decrease. In addition, the accumulation of gas bubbles hinders the reaction of forming new gas on the electrode surface, thereby reducing the mass rate of gas formed.

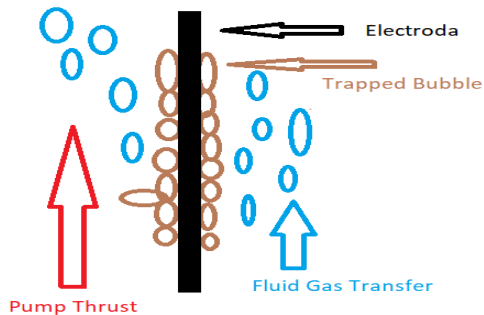


Figure 9. Gas bubbles inhibit new reactions from occurring so that gas accumulation decreases.

Temperature control is required through a heat transfer mechanism from the working fluid before it enters the electrolyzer. This will continue to maintain the performance of the electrolyzer and pump so that potential reductions in flow and hydrogen production can be avoided. It should also be noted that the size of the reaction area adapts to the current flowing in the electrolysis process.

3.2.5 Overpotential

This research uses a static adapter by providing 5 V and 40 A makes the process run with excess voltage. Under ideal conditions, CA electrolyzers use a voltage of around 2.2 V and around 1.86 V for AWE electrolyzers (Kumar et al., 2021). Providing high voltage in this research result in gas production but is also directly proportional to the excess energy wasted. The speed of temperature increased in Figure 6 it takes up to 40 minutes for the gas reaction speed to become saturated. It is estimated that some of the applied voltage turns into heat, thus accelerating the increase in the reaction rate before it becomes saturated (Buelvas et al., 2014).

3.3 Discussion

Apart from the performance side, considerations for using CA and AWE simultaneously must take into account the CAPEX (Capital Expenditure) and OPEX (Operational Expenditure) of production. In the Chlor-Alkali industry, Chlorine, NaOH and Hydrogen have standard prices. Meanwhile, in the AWE process, the main product is hydrogen. The OPEX value of a hydrogen plant is very dependent on the commodity price of the hydrogen itself. For comparison, the current commodity prices for CA products can be seen in the Table 5.

Table 5. Production of 1 Kg Chlorine & CA Derivatives (Garcia-Herrero et al., 2017)

	Production (kg)	Price (Euro/Kg)	Profit (Euro)
Chlorine	1	0.165	0.165
Sodium Hydroxide (50%)	2.256	0.224	0.505
Hydrogen	0.02	1.7	0.048
Total Price			0.719

As a reference, 1 kg of chlorine production, Sodium Hydroxide (50%) is 2,256 Kg, and 0.02 Kg of Hydrogen requires around 1 kwh of energy (Fesenko et al., 2017). This means that the energy required to produce 1 kg of hydrogen is around 50 kwh based on current technology. The energy required is directly proportional to the increase in the price of making the Chlor-Alkali model. Research models are needed to obtain energy savings that can reduce energy.

Based on Table 4, the highest increase of each interval of was in T5 from sample B1, 0.63 gr per 10 minutes operation. Based on this assumption the optimal operating condition was in T5-B1 for each hour operation, the electrolyzer can produced 3,78 gr of hydrogen gas. The production time to reach 1 kilogram of hydrogen shall be reduced to 265 hour compare to 318 hour in B1 without optimal condition. The energy consumption shall be reduced to 105 kwh to get 1 kilogram of hydrogen gas.

In Table 6 you can see the differences between each sample where you can see the performance of the electrolyzer made. There are still many improvements that need to be made so that the electrolyzer models created become more competitive.

Table 6. Comparison of Gas Mass Production Efficiency for each Sample

	A1	A2	A3	B1	B2	B3	Unit
Voltage	5	5	5	5	5	5	volt
Current	40	40	40	80	80	80	Ampere
Energy/hr	200	200	200	400	400	400	wh
W H ₂ /hr	1,01	1,35	1,44	3,14	2,97	3,02	gr




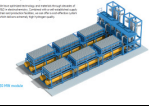
For kg H ₂	993,61	738,20	694,44	318,05	336,46	331,20	hour
per kg H ₂	198,72	147,64	138,89	127,22	134,59	132,48	kwh
Electrolyte	NaOH 10%	NaOH 10%	NaOH 10% +NaCl 10%	NaOH 10% +NaCl 20%	NaOH 10% +NaCl 20%	NaOH 10% +NaCl 20%	

Therefore, the combination of the CA-AWE electrolyzer which working in parallel with an increase of 11% in gas mass have good potential to developed. Current research still uses materials and processes that are not fully effective with a consumption of 127 kwh per kg H₂. In industry more detailed and structured investment calculations are required. The development of CA-AWE can be carried out from electrodes, membranes, fluids with

higher levels of purity, compare with electrolyzers that have been developed to date.

In table 7, the B1 test equipment is compared with several commercial electrolyzer. Several improvements from this Research such as the addition of a heat exchanger, catalyst control, electrode area, membrane sensitivity, as well as improving the performance of the electrode shall be implemented to reduce up the gap of electrolyzer.

Table 7. Comparison of Test Equipment Performance with commercial Electrolyzers

Reference Value	Unit	Current	 Enapter	 Bloomenergy	
Photograph		B1			
Source			(Enapter, 2020)	(Bloom, 2020)	(Thyssenkrupp, 2019)
Prod Rate H ₂	kg/h	0,00314	0,04	7,8	3.595,2
Hydrogen Pressure Rate	Bar		up to 35	0,05	300
Consumption	kwh/kg	105 to 127	57,60	54	48
Operating Power	kW	0,4	2,40	360	20.000
Operational Temperature	celcius	20 to 85	5 to 45	130 to 180	Up to 90
water consumption	kg/h	>0,02	0,40	102	4.000
Water pressure input	Bar	1,2	1 to 4	-	-
Weight	kg	3	55,00	6000	20 MW Module
Type		Alkaline	Anion Exchange Membran	Solid Oxide Fuel Cel (High Temp.)	Alkaline
Model		ZeroGap	Asymmetric /Solid State	Asymmetric	ZeroGap

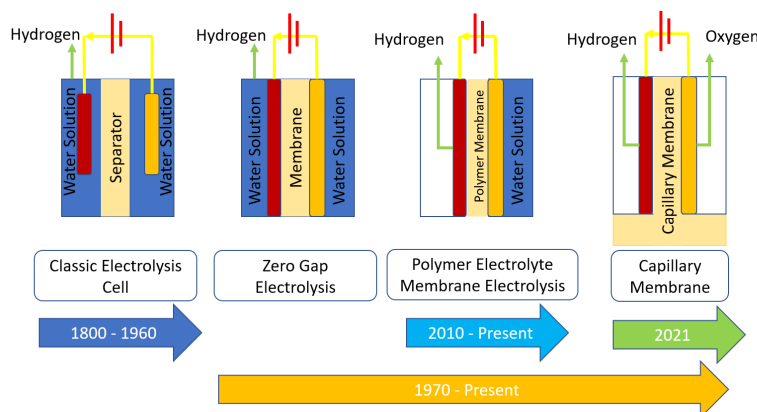


Figure 10. Development of electrolyzers to date (Buelvas et al., 2014)

Currently, the development of the model and geometry of the electrolyzer is research that continues to develop. The design used in the current research based on ZeroGap model which has been used by CA manufacturer. Improvements in the asymmetric Membrane Cell Model have made electric cars from hydrogen possible due to their high performance and small size.

In the latest research by Hodges et al., (2022) using water capillarity in membranes allows performance to be increased to 40.4 kWh kg⁻¹ H₂. This model also makes the components of the electrolysis plant more compact, thereby reducing energy use and resulting in a reduction in factory CAPEX.

The development of hydrogen as an environmentally friendly fuel has been developed for many applications. Hydrogen with a fuel cell module has been used in hydrogen electric cars (Hochgraf, 2009). There are also other means of transportation such as ships by Baldi et al., (2019), aviation by Hoelzen et al., (2022) and even countries Cheng et al., (2022). In addition, industries with high levels of carbon pollution such as steelmaking according to (Marchand & Strawderman, 2020) have tried to use hydrogen as combustion fuel to greatly reduce the emission.

4. CONCLUSION

In the research that has been carried out, several conclusions have been obtained that can be used in the process of developing further research.

1. The combine process between CA and AWE in parallel where the electrode fluid is circulated has the potential to increase the mass production of hydrogen gas by close to 11%.
2. Increasing NaCl concentration in the Chlor-Alkali process has the effect of increasing gas production up to a temperature of 65 degrees Celsius. The value of increase per 10% electrolyte will increase the mass increase

between 2-3.5% of hydrogen gas. However, the increase of electrolyte must consider the reaction area, temperature and pump discharge to avoid bubble traps.

3. A temperature control mechanism is needed at the electrolyzer input to overcome the decrease in reaction performance and pump thrust,. It is best if the temperature controlled between 40 and 65 degrees Celsius.
4. Bubble traps from gas and fluid reactions can be avoided by expanding the area. There are 2 methods of expanding the area currently being developed, namely widening the electrode and membrane, and stacking the electrode and membrane in a parallel design.

5. NOTATION LISTS

W	= Mass Fluid/ Gas	Gr
I	= Electrical Current	ampere
V	= Voltage	Volt
e	= Electron	electron
F	= Faraday constant	coulomb
n	= Number of moles	mol
R	= Gas Constant	J/K.mol
T	= Temperature	kelvin
t	= Time	Second / hr
M	= Molar Mass	gr/mol
ρ	= Density	gr/liter

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