Experimental study on the Effect of operating parameters on Hydrogen production from alkaline waste water electrolysis.

MEKELLE UNIVERSITY

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This thesis titled "**Experimental Study on the Effect of Operating Parameters on Hydrogen Production from Alkaline Wastewater Electrolysis**" prepared and submitted by **Yibrah Gebrecherkos Gebreslassie** in partial fulfillment of the requirements for the Master of Science in mechanical engineering (Thermo-fluid Engineering) is hereby accepted.

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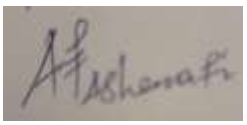
Ashenafi Tessema (M.Sc.)	Signature:	

i. Declaration

I, **Yibrah Gebrecherkos Gebreslassie**, hereby declare that the thesis titled "Experimental study on the Effect of operating parameter on hydrogen production from alkaline waste water electrolysis" is my original work. This work has not been submitted in part or in full for

any other degree or diploma at this or any other university. All sources of information and sources of material have been acknowledged.

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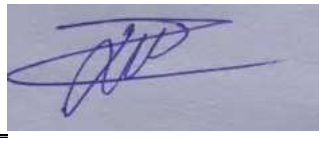
Signature: 

Date: July 22/2025

This is to certify that the above declaration made by the candidate is correct to the best of my knowledge**.**

Tesfaldet Gebregerges (PhD)

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Date: July 22/2025

ii. Abstract

This study investigated the influence of three principal operating parameters on hydrogen production from alkaline wastewater electrolysis. Hence the primary objective of this study was to examine the effects of specific operating conditions on hydrogen yield, employing alkaline wastewater as electrolyte. The key parameters examined included temperature variation, effect of electrolyte concentration variation, and applied current variability.

The findings indicated that electrolysis performance was significantly influenced by these parameters. Specifically, the volume of hydrogen produced rose with rising current. At the tested currents of 0.3A, 0.5A, 0.7A, and 0.8A, the time needed to reach 10 ml of hydrogen was 664.88s, 349.24s, 244.21s, and 230.08s, In addition efficiency rose with each added current, 41.122%, 46.973%, 50.322%, 47.982%, and 44.562%. Higher applied currents initially enhanced the yield. However, beyond a certain threshold, further increases in current lead to a decline in efficiency owing to limitations in mass transport and bubble formation in and around the cell.

Regarding the effect of temperature and electrolyte concentration on the rate of hydrogen production, as the temperature raised at 10 K intervals (303.15, 313.15, 323.15, 333.15, and 343.15 K), the time needed to reach 10 ml of hydrogen by volume reduced in higher order of magnitude (312.61, 267.61, 233.96, 189.72, and 184.18 s, respectively). In addition, the efficiency of the hydrogen production rate improved at each added current: 45.173%, 51.84%, 56.623%, 62.31%, and 67.735%. Although high temperatures improve efficiency, they are not favored because of the higher operating costs. The effect of the electrolyte concentration was also significant in terms of the hydrogen rate. At a current of 0.25 A for all ranges of concentration tested (5, 10 g/l; 15g/l and 20 g/l NaOH), the time(s) to reach 10 ml by volume of hydrogen was 341.58s, 276s, 236.18, and 209.57 s, respectively. This was due to the higher ionic mobility with an enhanced concentration of the electrolyte.

Generally, the hydrogen production yield reached approximately 57% efficiency at a temperature of 323.15 K, current of 0.6A, and concentration of 10 g/NaOH from the alkaline wastewater, highlighting the potential of this method for generating hydrogen from an abundant and environmentally friendly resource. Future research should focus on further optimization strategies, long-term stability of electrodes in wastewater electrolysis, and economic feasibility of scaling up this process for practical applications.

Key words; alkaline electrolysis, Current, electrolyte concentration, temperature difference and efficiency

iii. Acknowledgement

First and foremost, I would like to express my cordial and deepest gratitude to my advisor, **Dr. Tesfaldet Gebregerges**, for your guidance and support. Thank you sir

I am also incredibly grateful to the RBSC company technicians and employees for their assistance with this project. Their willingness to collaborate and share resources was crucial to its success. Specifically, I would like to thank them for facilitating the collection of the alkaline wastewater sample and providing access to company materials for sample characterization.

My sincere appreciation extends to all the laboratory technicians at Mekelle University who helped with the experimentation and analysis. Their support and assistance were invaluable during the laboratory work.

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vi. Abbreviations

AWE Alkaline water electrolysis

NaOH Sodium hydroxide

HTAWE High temp alkaline water electrolysis

PEM Polymer electrolyte membrane

SOEC Solid oxide electrolysis cell

STP Standard temperature and pressure

TDS Total dissolved solid

BOD Biological oxygen demand

COD Chemical oxygen demand

RBSC Raya brewery Share Company

Chapter one

1. Introduction

1.1 Background

Global energy demand is on continual rise due to population growth, improving living standard, and industrial development in economically emerging countries (1). In addition, conventional energy sources are becoming increasingly rare. Research has shown that the estimated fossil fuel reserve potential of the world is limited (2). In addition, fossil fuels are the main cause of global warming and climate change. Report showed that in the first decade of the 21st century, greenhouse gases in the atmosphere have grown rapidly compared to the previous decades (3).To moderate the challenges of climate change and the exhaustion of conventional fuel sources, our energy system needs to be resilient, reliable, and sustainable. In this case, renewable energy systems can/will play a greater role in generating clean and sustainable energy resource (4). In the coming twenty years, the share of renewable energy sources as the major energy supply is expected to rise globally (5). This implies that a better share of energy will be generated from renewable energy resources such as wind, solar, and hydro. After electricity is generated from these abundant renewable energy resources it needs to be integrated with energy storage systems to make it useful and economically feasible.

The key to unlocking the full potential these renewable energy resources lies in the development of efficient methods for conversion, energy storage, and intermittent electricity load management of the unsteady resources. Hydrogen often referred as the energy carrier of the future can be used to store these unsteady renewable energy sources. Additionally, hydrogen can be the first candidate to replace conventional fuels to provide a green energy for the future due to its suitability, abundance, and environmentally friendly nature. The idea of creating sustainable energy systems has led to several hydrogen energy demonstration projects worldwide over the past decade. Hence, hydrogen has emerged as a potential solution for storing clean energy from renewable energy resources (6). Therefore, a favorable method is to combine renewable energy sources with hydrogen production. This not only improves the renewable energy capacity but also allows for environmentally friendly energy generation, minimal pollutant emission and better energy load control.

Naturally hydrogen as a gas has high energy density. This energy can be exploited by combusting hydrogen with oxygen. The combustion of hydrogen with oxygen is clean reaction which produces solely water vapor as a byproduct. On the other hand, the combustion of conventional fuels (fossil fuels) generates greenhouse gases which are the foremost source of environmental pollution (7). In addition hydrogen combustion has other environmental importance; it can be used both as fuel to generate electricity and in electrical energy storage which in consequence replaces fossil fuels, these by implication reduce greenhouse gas emissions and important electricity energy carrier (8). Hydrogen gas (molecule) is made up of two hydrogen atoms connected by covalent bond. Breaking of this covalent bond generates energy. But, in contrast to other gases, hydrogen is naturally non abundant, constituting less than 1% of in the earths' atmosphere. Because of its value as energy storage and electricity source, we need to produce it from different resources.

There are two major methods to extract hydrogen: one is through a process called methane reforming using conventional fuel sources such as coal, oil, and natural gas. The second one is by splitting water in to hydrogen and oxygen molecules with electrolysis process using renewable sources such as hydropower, wind, and solar power as sources of electricity. Electrolysis (splitting water into hydrogen and oxygen) is considered a clean energy source, because it can proceed through friendly processes that do not produce environmentally harmful products or byproducts. Several studies have been published on the production of hydrogen from renewable energy resources using electrolysis (9). Moreover other methods can be employed for hydrogen production: including electrical, thermal photonic and biological methods. Many researchers are attracted to alkaline electrolysis processes, because of their environmentally friendly, simplicity and ease of maintenance.

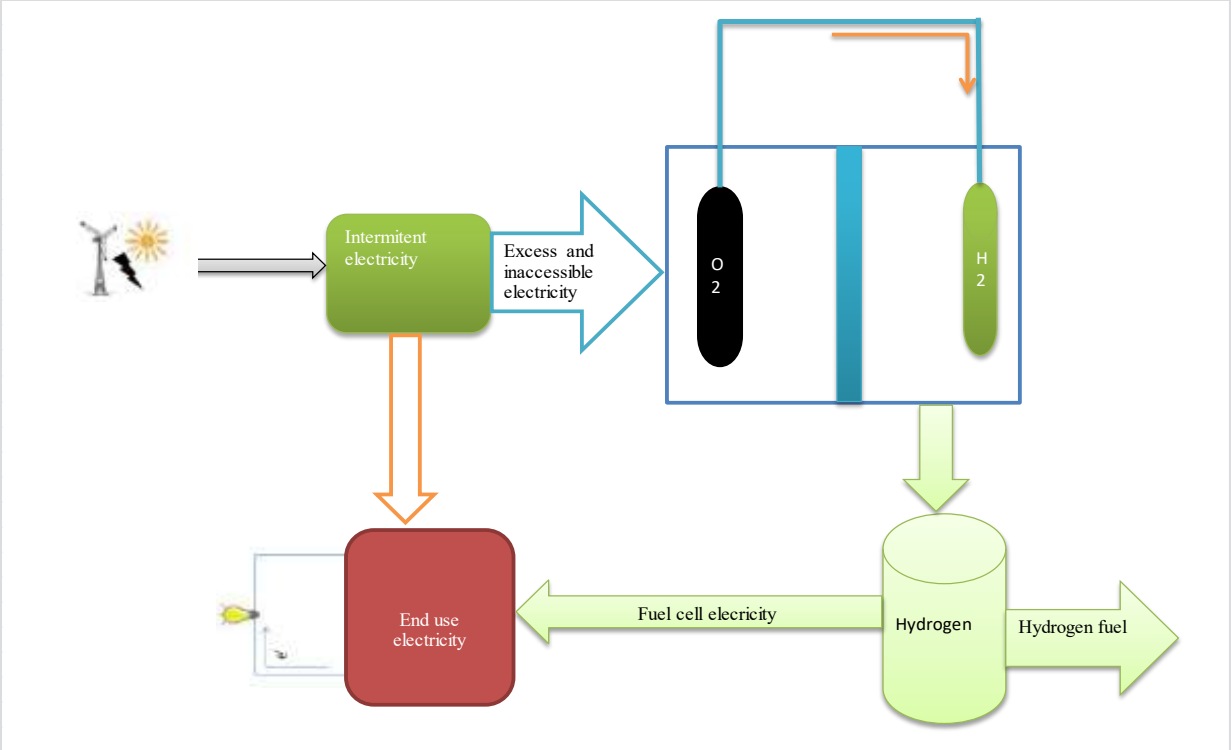

Figure 1: schematics of water electrolysis in hydrogen production as a fuel gas and energy storage.

In general during electrolysis exchange of atoms and ions, or removal addition of electrons takes place at an electrolyzer. During electrolysis, two electrodes know as cathode and anode – negative electrode at which reduction takes place and positive electrode where oxidation takes place - are connected to a power source are immersed in water. Then, hydrogen evolution reaction takes place at cathode while oxygen evolution reaction at anode. In addition electrolysis can be simply expressed as in the following equation:

$$H_2O \rightarrow H_2 + \frac{1}{2}O_2 \qquad (1)$$

Water molecule is broken in to its constituent elements at a minimum voltage of 1.229 V (10). This is the minimum voltage required to break the covalent bond between hydrogen and oxygen in water molecule. Not only voltage but also several other factors affect volume, purity and rate of hydrogen evolution. Some of them are cell current density, electrolyte temperature, cell pressure, electrolyte concentration, and electrode material etc. Several experiment studies revealed that these factors have an important effect in the performance of the electrolysis cell. Some studies revealed that:

- A report showed that applied current and electrolyte concentration/pH affect the hydrogen evolution reactions. It revealed that as the amount of current, and concentration of the electrolyte rises, the hydrogen evolution from electrolysis grows (11).
- Another report indicated that as temperature raises ionic mobility is enhanced, ionic conductivity gets better, and electrolyte surface reaction increases, these leads to an increasing hydrogen evolution (12).

Electrolysis utilizes pure water to produce hydrogen. Though hydrogen has shown potential as future source of electricity and energy storage, exploiting fresh water has other side effects. Freshwater is a scarce resource that is often insufficient in terms of quality and quantity for industrial level electrolysis. Moreover, globally freshwater sources are in limited supply. Population growth and industrialization are making freshwater sources scarce and polluted. According to report published by United Nations World Water Development in 2018, it predicted that by 2050, an estimated 6 billion people are going to have difficulty accessing clean water (13). Many of the pollutants pose risks to the environment and human health. Environmental protection strategies focus on developing cleaner industrial processes that minimize environmental impact and treat unavoidable waste.

Rising fresh water costs and stricter wastewater discharge regulations have motivated industries to implement water conservation programs and seek new methods for fresh water resource optimization, resource recycling and recovery. Hence Wastewater electrolysis may have the potential for decentralized hydrogen production with simultaneous on site wastewater treatment (14). Various studies have focused on hydrogen production from wastewater in the open literature. However, although reviews have been conducted on various specific topics regarding hydrogen production from wastewater, a comprehensive review and comparative assessment of all available options for producing hydrogen from alkaline wastewater using nickel and graphite as electrodes have not been found in the literature. Therefore, examining the methods and effects of parameters for hydrogen production from alkaline wastewaters under a single source is important.

1.2.Problem statement

The escalating global reliance on intermittent renewable energy sources necessitates cost-effective and sustainable large-scale energy storage solutions to ensure grid stability and reliable energy supply. Concurrently, significant volumes of alkaline wastewater generated by industries (as observed at RBSC) pose an environmental burden and represent an unexploited resource with inherent chemical energy. Current practices often treat this wastewater as a waste product, thereby missing a critical opportunity for both resource recovery and clean energy generation.

While the electrochemical production of hydrogen from alkaline wastewater offers a promising pathway to address both energy storage needs and waste management challenges, its full potential remains unrealized. This is primarily due to a lack of comprehensive understanding regarding the influence of key operating parameters—including applied current, electrolyte concentration and temperature — on the efficiency and hydrogen yield when utilizing cost-effective electrode materials (e.g., nickel and graphite).

In addition there is an urgent need to precisely define the intricate trade-offs between these parameters to achieve efficient, economically viable hydrogen production, especially considering the balance between improved yield and electrode materials. Therefore, the core problem lies in the underutilization of alkaline wastewater as a viable and economically attractive feedstock for green hydrogen production and valuable resource recovery, hindering sustainable energy transition and waste valorization efforts. Hence the key research question – this thesis primarily tries to address - is how do electrolyte concentration, temperature, and current affect hydrogen production rates and energy efficiency in alkaline waste water electrolysis?

1.3.Objective of the study

1.3.1. General objective

The general objective of this research is to investigate the efficiency of hydrogen production through alkaline wastewater electrolysis using nickel and graphite as electrodes with major objective to understand the key factors influencing hydrogen yield from alkaline waste water.

1.3.2. Specific Objective of Study

1. **Characterization of Alkaline Wastewater**: Analyze the composition and properties of alkaline wastewater from the selected site using advanced analytical techniques.

2. **Impurity Assessment**: Evaluate the treatment of alkaline wastewater for dissolved solids and their impact on electrolysis efficiency.
3. **Current Variability Analysis**: Investigate the effect of varying electric current on hydrogen production efficiency in alkaline electrolyte solutions.
4. **Electrolyte Concentration Effects**: Assess how different electrolyte concentrations influence hydrogen yield during wastewater electrolysis.
5. **Temperature Influence**: Explore the impact of temperature on hydrogen production rates in alkaline wastewater electrolysis.

1.4. Limitation of Study

The alkaline waste water was exclusively collected from RBSC washer room. This may limit to make the necessary generalization for other industries. In addition this research is focused only on the hydrogen production efficiency. Other byproducts are not included in the analysis.

The analysis is based on a laboratory test which does not account for some practical challenges of scaling up the process. Issues like electrode degradation, long-term stability in wastewater environments, and maintenance needs cannot be overlooked. In general no pilot plant test was made.

The analysis is limited to specific electrodes, nickel as anode and graphite as cathode. Although this is limited by the capacity of our lab, exploring a wider range of materials for electrode, including their compatibility with wastewater and cost-effectiveness, is beneficial.

The economic viability of the process, including capital costs, operational expenses, and hydrogen production value, are not explicitly factored into the analysis.

1.5. Organization of the thesis

Chapter 1 is a short introduction of the drive for producing hydrogen via alkaline waste water electrolysis together with the objectives of the study and structure of the thesis.
Chapter 2 encloses the fundamentals of electrolysis, waste water electrolysis, hydrogen production by electrolysis including the main principle, the thermodynamics and an overview of the main cause for an efficiency loss in an electrolysis cell. In addition the technologies used for electrolysis of water and waste water were thoroughly reviewed. The

research gap for producing hydrogen from alkaline waste water is materialized.

In **Chapter 3** gives an overview the methodologies and materials used to characterize the waste water. The methodology to investigate the electrolysis process variables was also included under this topic.

In **Chapter 4** the main outcomes related to the experiment was analyzed for their respective outcomes.

In **Chapter 5** Overall conclusions of the findings reported in the thesis and the appended data are in this chapter. In addition recommendations and further areas of study that can strengthen the outcome (further research) are included in this chapter.

Chapter two

2.0. Fundamentals of water electrolysis

2.1. **Principle of water electrolysis**

Water electrolysis is the process of splitting water molecules into its constituent elements - hydrogen gas and oxygen gas - using electricity. The overall chemical reaction of water electrolysis without required thermodynamic energy values can be written as:

$$H_2O\ (l) \rightarrow H_2\ (g) + \frac{1}{2} O_2(g) \qquad (2)$$

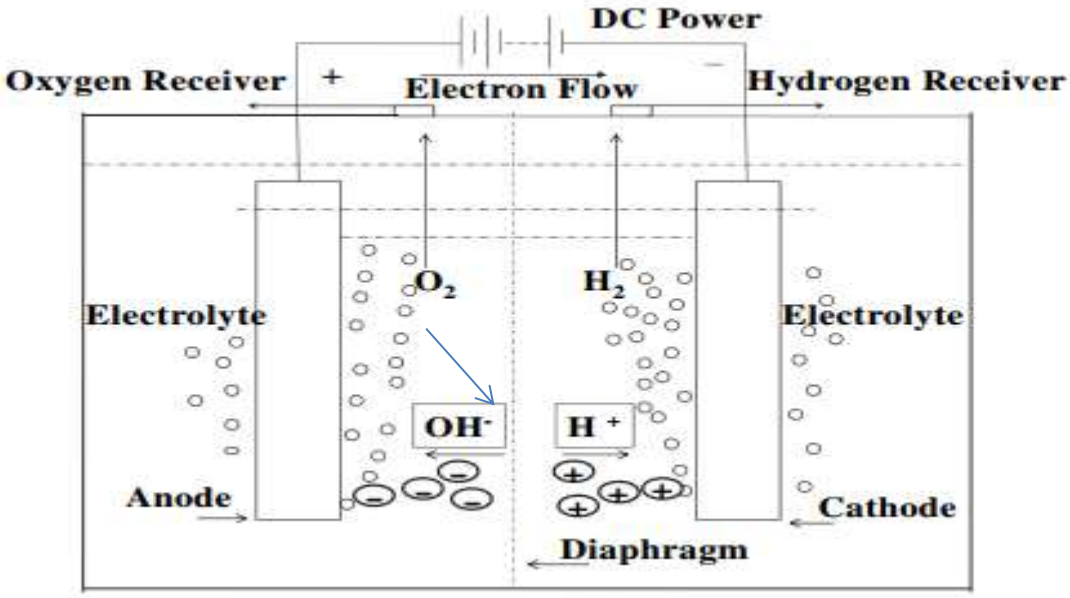

Figure 2 : A schematic illustration of a basic water electrolysis system (15).

This show as current passes through the system hydrogen evolution reaction takes place at the cathode while oxygen evolution is at anode. On the other hand the diaphragm separates these electrodes, preventing gas crossover or mixing while it allows transfer of free ions. These pure gases are collected as they rise from their respective electrodes. As will be described in the sections below, specific chemical reactions that take place at each electrode are different with different water electrolysis methods employed.

2.1.1. Thermodynamics

As figure-2 (below) indicates electrolysis process is not thermodynamically favorable. It requires input of energy as indicated by the power supply curve. Under standard conditions to decompose one mole of water the amounts of heat required in terms of enthalpy change (ΔH) is 285.9 kJ. (16). However, if the process is at low temperatures, electricity must be supplied from external source to generate the majority the heat required while the rest can be applied in the form of thermal energy. Generally the energy required to make electrolytic

decomposition of water molecules possible can be stated from the enthalpy changes as follows:

$$\Delta H=\Delta G+T\Delta S \quad (3)$$

The equation for Gibbs free energy change (ΔG) can be determined from entropy change (ΔS) and temperature (T). In addition following relationship can also be employed to calculate ΔG from reversible voltage (E_{rev}), which is also identified as equilibrium voltage (E_{eq}):

$$\Delta G=nFE_{rev} \quad (4)$$

While "n" stands for the number of moles of electrons transferred in the reaction and F for Faradays constant with a value of 2 and (9.65x 10^4C/mol) respectively. The absolute minimum voltages required in order to produce hydrogen and oxygen by splitting water is designated as E_{rev}.

E_{rev} can be calculated from the sum of anodic and cathodic reversible voltages as shown in eq (5);

$$E_{rev} = E_{revO2} + E_{revH2} \quad (5)$$

The reversible voltage of hydrogen and oxygen evolution at standard conditions are given as 1.229 V and 0.000 V respectively, which the resulting sum of these gives E_{rev} of 1.229 V (17). Therefore, Erev of 1.229 volts must be applied between the anode and cathode for in eq. (1) to take place in the water electrolysis cell. Replacing this value of the reversible voltage into eq. (4) it gives Gibbs free energy of ΔG = 237.2 kJ/mol. On the other hand to calculate electrical potential needed for water electrolysis, Nernst equation is employed for conditions out of STP as stated below (18)

$$E_{T,P} = E_{rev} + \frac{RT}{nF}\ln\left(\frac{P_{H_2}P_{O_2}^{1/2}}{P_{H2O}}\right) \quad (6)$$

"R" stands for the gas constant with value (8.3144621 J/ mol K),while P_{O2}, P_{H2O}, and P_{H2} stands for the partial pressure of oxygen, partial pressure of water and partial pressure of hydrogen respectively. If the assumption is partial pressure is constant over the whole system, eq. (5) can be adjusted as:

$$E_{TP} = E_{rev} + \frac{RT}{nF}\ln\left(P^{1/2}\right) \quad (7)$$

The above equation can be interpreted as, for pressurized systems, Reversible voltage at different conditions other than STP is greater than reversible voltage at standard condition ($E_{revTP} > E_{rev}$). This implies that higher electrical energy is consumed in order to reaction in

eq. (1) to proceed. However, if the process is at low temperature, the voltage rise due to temperature increase is minimal. Let's take an example of an electrolysis cell at pressure and temperature of 100atm, 70°C respectively. Calculating E_{rev} it gives a difference of 0.034 V. This can be interpreted; as the system gets pressurized it reduces the ionic resistivity in the cell. After calculating the Gibbs free energy and enthalpy required for the process the additional thermal energy required is calculated using eq(3). At STP the amount of additional heat in the form of enthalpy or thermal energy is given as:

243.759 kJ/mol - 237.2 kJ/ mol = 6.32 kJ/mol (8)

Sometimes this amount of additional heat cannot be supplied from external heat source; therefore shortage of energy should be compensated from electrical source. This necessitates applying voltage more than E_{rev} = 1.229 volt difference at the anode and cathode for the electrolysis process to proceed. E lectrochemical reactions can proceed without absorption and generation of heat at specific voltage. This electrical energy required for to maintain the reaction at STP is defined as thermo neutral voltage (E_{th}).

The enthalpy of thermoneutral voltage which is minimum amount of energy needed for the reaction can be defined as:

$$E_{tn} = \frac{\Delta H}{nF} = \frac{\Delta G}{nF} + \frac{T\Delta S}{nF} \quad (9)$$

At standard temperature and pressure the minimum amount of energy needed is calculated to be 1.481 V (19)

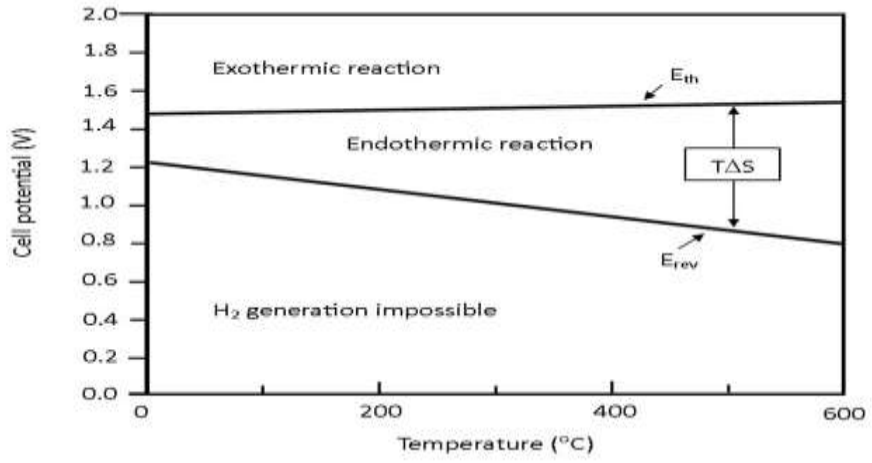

Figure 3: Cell potential for hydrogen production by water electrolysis as a function of temperature.

As shown in figure-3 the process above E_{th} is exothermic generating heat in effect while the process between E_{th} and E_{rev} is endothermic- external heat is applied so as the process to

proceed. On the other hand at a process below E_{rev} hydrogen generation is thermodynamically impossible. In addition Fig. 3 displays, though the greater proportion of the process energy to be supplied as heat, it reduces the cell voltage needed. Consequently, if inexpensive thermal energy is accessible, the cost of producing hydrogen through water electrolysis can be reduced. Conversely, though water can be directly broken down thermodynamically temperatures greater than 2000°C, direct heating at high temperatures without catalysts requires higher energy demand.

Naturally water is decomposes spontaneously at temperatures above 4100°C. Research showed that at temperature of 2000°C only 1vol% of water is decomposed (18). On the other hand 5000K directs heating is needed for full decomposition of water and 2500 K for partial decomposition.

2.1.2. The resistance in the electrolysis cell

Beyond the minimum energy theoretically needed for electrolysis, additional electrical resistance within the system must be overcome for the process to occur. Therefore, the actual voltage applied to the cell during electrolysis will always be higher than the theoretical voltage calculated based purely on thermodynamic principles. This extra voltage, necessary to overcome these resistances, is termed overpotential or overvoltage. These overpotentials in an electrolysis cell can be categorized into three types (20).

- Resistance overpotential (η_r)
- Concentration overpotential (η_c)
- Activation overpotential (η_a)

Resistance overpotential arises from the electrical resistance within the cell, including external wiring, electrode connections, and the electrodes themselves. This can typically be minimized by using highly conductive materials for wires and connections and ensuring adequate cross-sectional areas. However, the resistance within the electrodes can be substantial, particularly if they contain passivated oxides or isolating impurities, which should therefore be avoided.

Concentration overpotential results from resistance to ionic transport within the electrolyte between the anode and cathode. This type of overpotential is influenced by the electrolyte's ionic conductivity, the distance between the electrodes, the diaphragm's conductivity, and the presence of gas bubbles in the electrolyte.

In water electrolysis, hydrogen and oxygen gases are produced at the electrodes. These gases initially form as tiny bubbles that lack the buoyancy to detach immediately. The bubbles must

merge and grow sufficiently large before they can detach from the electrodes and rise into the electrolyte. This process of bubble formation, accumulation on the electrode surfaces, and dispersal within the electrolyte is known as the bubble phenomenon in alkaline water electrolysis. Gas bubbles adhering to the electrode surfaces reduce the available area for the electrochemical reaction, thus impeding its progress. Additionally, bubbles within the electrolyte increase its resistance to ion flow. These combined effects result in a substantial voltage drop (Ohmic drop) during operation and are the primary contributors to concentration overpotential in alkaline water electrolysis cells (21). To mitigate this overpotential, strategies like increasing the pressure within the electrolysis system, promoting efficient electrolyte flow, and using zero-gap cell designs can be implemented. The activation overpotential, which reflects the energy needed to initiate the electrochemical reactions at the anode and cathode, exhibits a logarithmic increase with rising current density (22). Concentration and activation overpotentials are the primary contributors to overall overpotential during electrolysis. Consequently, the total cell voltage for a water electrolysis cell (assuming adiabatic conditions) can be expressed as follows:

$$E_{cell} = E_{th} + E_r + E_a + E_c \quad (10)$$

Both the concentration overpotential and the resistance overpotential cause heat generation in the system. Some of the generated heat can, in well isolated electrolysis system, be used for heating up the electrolyte.

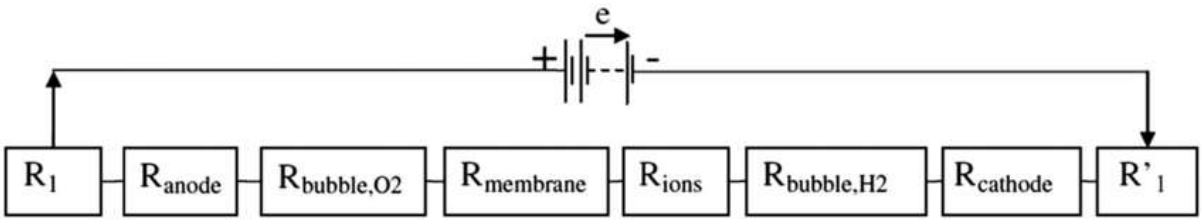

Figure 4: An electrical circuit analogy of resistances in the water electrolysis system (15).

In that way the efficiency loss from the overpotentials can be minimized. For the ease of identifying every single overpotential source in an electrolysis cell, the overall cell resistance can be expressed by the electrical circuit analogy as displayed in Fig. 4. Here R_1 & R_2 represents the electrical resistance from wiring and connections to the electrodes. R_{anode} and $R_{cathode}$ are the overpotentials required to overcome the activation energies of the oxygen and hydrogen formation, respectively. R_{O2} and R_{H2} is the resistance from the oxygen and hydrogen bubbles

in the electrolyte and on the electrodes surfaces. R_{ions} represents the resistance in the electrolyte and $R_{membrane}$ is the diaphragm resistance.

2.2.Efficiency of water electrolysis

The efficiency of water electrolysis can be evaluated and expressed in numerous ways, depending on the specific criteria used for assessment and comparison. This variety of approaches can sometimes lead to confusion and inconsistencies in published research. For the purpose of electrode development, cell efficiency is the most critical metric. Cell efficiency (η) is determined by comparing the measured cell voltage to either the reversible voltage or the thermoneutral voltage, as demonstrated in the following equation: (11) and (12):

$$\eta_{rev} = \frac{E_{rev}}{E_{cell}} \quad (11)$$

$$\eta_{th} = \frac{E_{th}}{E_{cell}} \quad (12)$$

As an example, an electrolysis cell operating at standard temperature and pressure (STP) with a cell potential of 1.48 V yields a calculated efficiency of 100% using equation (12), but only 83% when using equation (11). This stark difference highlights the critical importance of specifying the calculation method when reporting electrolysis efficiency; without this context, the reported value is essentially meaningless.

The theoretical efficiency (η_{th}), also known as the higher heating value (HHV) efficiency, is commonly found in the literature. While the reversible efficiency (η_{rev}) is inherently limited to values below 1 (since hydrogen production necessitates a potential at least equal to E_{rev}, η_{th} can surpass 1 because the reaction has the capacity to absorb heat from its surroundings), Efficiency can also be determined by comparing the energy supplied to the electrolysis system with the rate at which hydrogen is produced. This comparison can be made using the following method:

$$\eta_{H2} = \frac{V_{H2}}{UIt} \quad (13)$$

Commercial electrolysis systems commonly assess efficiency by measuring the total energy input required to generate one cubic meter of hydrogen. In this context, where V is the volumetric hydrogen production rate, U is cell voltage, I is current, and t is time, a 100% efficiency rating equates to an energy consumption of 3.54 kWh per normal cubic meter (Nm^3). Consequently, the theoretical production of roughly 11,000 liters of hydrogen at standard temperature and pressure necessitates 39 kWh of electricity and 8.9 liters of water (23). On the

other hand, currently the most efficient commercial electrolyzer systems have a maximum efficiency of 82% equivalent to 4.3 kWh/Nm^3.

2.2. An overview of water electrolysis technologies for hydrogen production

Water electrolysis is not a recent discovery; it was developed over two centuries ago by Nicholson and Carlisle. As of today, three primary water electrolysis technologies exist: alkaline electrolysis, polymer electrolyte membrane electrolysis, and solid oxide electrolysis cell. While AEM and PEM electrolysis are commercially available, SOECs are still under development.

Commercial alkaline and PEM electrolyzers typically operate below 100°C, whereas SOECs operate in a gaseous phase at much higher temperatures, between 800 and 1,000°C (24). Operating water electrolysis systems at high temperatures significantly reduces the electrical energy needed for hydrogen production, making it a key focus for developers. However, these higher temperatures present significant material challenges, leading to corrosion of metals and degradation of polymeric seals, which are major obstacles hindering the commercialization of solid oxide electrolysis cells.

At high current densities proton exchange membrane electrolyzer offer superior efficiency compared to other commercially available electrolyzers. Furthermore, material durability of alkaline electrolysis has greater lifetime than PEM. The lifetime of commercial alkaline electrolyzers is said to be higher than for PEM electrolyzers (25). Table 1 summarizes the principal technologies of electrolysis;

	Alkaline	AEM	PEM	SOE
Electrolyte	KOH/NaOH(5M)	DVB polymer support with 1 MKOH/NaOH	Solid polymer electrolyte (PFSA)	Yttria stabilized Zirconia (YSZ)
Gas diffusion layer	Nickel mesh	Nickel foam/carbon cloth	Titanium mesh/carbon cloth	Nickel mesh/foam
Bipolar plates	Stainless-steel/Nickel coated stainless-steel	Stainless-steel/Nickel coated stainless-steel	Platinum/Gold-coated Titanium or Titanium	Cobalt coated stainless-steel
Nominal current density	0.2–0.8A/cm^2	0.2–2A/cm^2	1–2A/cm^2	0.3–1A/cm^2
Voltage range	1.4–3V	1.4–2.0V	1.4–2.5V	1.0–1.5V
Temperature	70–90◦C	40–60^{O}C	50–80^{O}C	700–850^{O}C
Cell Pressure	<30bar	<35bar	<70bar	1bar
H_2 Purity	99.5–99.9998%	99.9–99.9999%	99.9-99.9999%	99.9–99.9%
Efficiency	50%–78%	57%–59%		
Lifetime	60000h	>30000h	50000–80000h	20000h
Development status	Mature	R&D	Commercial	R&D
Electrode area	10000–30000cm^2	<300cm^2	1500cm^2	200cm^2
Capital cost per 1MW	USD270/kW	Unknown	USD400/kW	>USD2000/kW
Capital cost per 10MW	USD500–1000/kW	Unknown	USD700–1400/kW	Unknown

Table 1: Comparison of typical water electrolysis technologies **(26)**

2.2.1. Alkaline Water Electrolysis

Alkaline electrolysis is a well-established and highly developed method for splitting water. In fact, William Nicholson and Anthony Carlisle first demonstrated this process, breaking water down into hydrogen and oxygen in 1800 (27). Alkaline water electrolysis uses electricity to split water electrochemically. The electrolysis cell contains two electrodes—an anode and a cathode—separated by an ion-conducting diaphragm. This diaphragm also prevents the hydrogen and oxygen gases produced from mixing. Figure 1 illustrates the operational principle of AWE. Applying a current between the electrodes decomposes water molecules near the cathode into hydrogen (H_2) and hydroxyl ions (OH^-). These negatively charged hydroxyl ions then migrate through the diaphragm to the positively charged anode, where they react to form water and oxygen.

Specifically, at the cathode, two moles of the alkaline solution are reduced, yielding one mole of hydrogen (H_2) gas and two moles of hydroxyl ions. The generated H_2 is released from the cathode surface, and the remaining hydroxyl ions are transported through the electric circuit from the cathode to the anode. This electrochemical water splitting process involves two distinct half-cell reactions. At the anode, the hydroxyl ions are discharged, producing half a molecule of oxygen and one molecule of water, as shown in eq (16)

$$2H_2O + 2e^- \rightarrow H_2\,(g) + 2OH^- \quad (15)$$

$$2OH^-\,(aq) \rightarrow \frac{1}{2}O_2\,(g) + H_2O\,(l) + 2e^- \quad (16)$$

Hydrogen is formed at the cathode where water is reduced according to eq (15). Hydroxide anions circulate across the diaphragm to the anode. Hydrogen purity level can reach between 99.5–99.9998 % (28). Moreover typical commercial alkaline electrolyzers operate with an efficiency of 60-75%, while the best small-scale systems achieve efficiencies of 80-85% (29) . The primary sources of efficiency loss in these systems stem from the activation energies required for hydrogen and oxygen evolution, the presence of gas bubbles within the electrolyte, and bubble accumulation on the electrode surfaces. These overpotentials are all influenced by the current density.

Consequently, the ohmic resistance within the electrolysis cell rises sharply with increasing current density. Hence, to achieve moderate efficiencies of up to 82% based on the higher heating value (HHV), the current density must be maintained at a relatively low level (30). Most of the times commercial alkaline electrolyzers are typically operated in a liquid electrolyte alkaline electrolyzers typically operate with a 25-40% alkaline solution at temperatures between 60 and 90°C. These electrolyzers are typically operated at 1-30bars, depending on their application. High pressure operation can reduce the ionic resistance caused by gas bubbles in the electrolyte, due to shrinkages of bubbles, and save the cost of compressing hydrogen after production.

While alkaline water electrolysis is a relatively mature technology, there's still room for improvement, particularly in increasing current density and minimizing gas crossover. Addressing these challenges requires developing advanced electrode materials and separators. In addition integrating alkaline water electrolyzers with renewable energy sources offers a promising pathway to reduce capital costs.

2.2.2. High temp alkaline water electrolysis

High-Temperature Alkaline Water Electrolysis (HTAWE) is essentially taking traditional Alkaline Water Electrolysis (AWE) and running it at much hotter temperatures. This increase in operating temperature significantly boosts the efficiency of the process. Consequently, a lot of research and development has focused on perfecting AWE techniques at temperatures exceeding 120°C.(31). Recent studies have demonstrated the performance of an alkaline electrolysis cell operating at temperatures as high as 250°C and a pressure of

42 bars. This cell achieved operating potentials of just 1.5 V and 1.75 V at current densities of 1 A/cm² and 2 A/cm², respectively. These results correspond to efficiencies of 85-99% based on the higher heating value, significantly exceeding previously reported efficiencies for alkaline systems at comparable current densities (32). Increasing the current density to 1 A/cm^2 resulted in a cell voltage of 1.9 V and 1.8 V for 90 and 120°C respectively. However, it is important to remember that the energy needed in order to heat and pressurise the cell is not taken into account for the efficiency measurements made. The developers for such techniques usually assume availability of low cost heat sources when and if used in practice. High-temperature alkaline water electrolyzers are still primarily in the research and development phase. A major challenge hindering HTAWE development is the limited stability of materials at the high operating temperatures.

2.2.3. Anion exchange water electrolysis

Anion exchange water electrolysis is a method of splitting water using electricity and a special membrane called an anion exchange membrane. This process involves two main reactions: one that produces hydrogen (hydrogen evolution reaction) and another that produces oxygen (oxygen evolution reaction). The first step happens at the cathode, where water molecules gain two electrons, breaking down into hydrogen gas and hydroxyl ions.

After hydrogen is produced and released at the cathode. Meanwhile, the hydroxyl ions move through the ion exchange membrane towards the anode, attracted by its positive charge. The electrons travel separately through an external circuit to the anode. At the anode, these hydroxyl ions combine, release their electrons, and form water molecules and oxygen, which is then released from the anode.

The core components of this electrochemical cell are the membrane (which acts as a separator), the electrode materials, current collectors (also known as gas diffusion layers), separator plates (bipolar plates), and end plates.

Specifically, the anion exchange membrane is typically made from quaternary ammonium ion exchange materials. For the anode and cathode, common electrode materials are transition metal-based electrocatalysts, with Nickel and NiFeCo alloy materials being frequently used. To manage gas flow and current collection, nickel foam or porous nickel mesh is employed for the anode's gas diffusion layer, while carbon cloth is used for the cathode's gas diffusion layer.

Anion exchange membrane (AEM) water electrolysis is an emerging technology for producing green hydrogen. It's gaining significant attention from research organizations and institutions because it offers lower costs and higher performance compared to traditional electrolysis methods. The earliest academic publication about Anion Exchange Membranes (AEMs) was a journal article by We and Scott in 2011. Since then, many other researchers have contributed to advancing AEM technology (33). The AEM water electrolysis technology is similar to conventional alkaline water electrolysis (34). The key distinction between traditional alkaline water electrolysis and Anion Exchange Membrane (AEM) water electrolysis lies in their separator.

While conventional alkaline systems historically used diaphragms, AEM electrolysis replaces these with a specialized anion exchange membrane, typically made of quaternary ammonium ion exchange materials. This membrane specifically allows hydroxyl ions to pass through, offering advantages over the less selective diaphragms used in older alkaline setups. AEM water electrolysis offers several advantages such as cost-effective transition metal catalysts being used instead of noble metal catalysts, distilled water/low concentrated alkaline solution (1M KOH) can be used as electrolyte instead of high concentrated (5 KOH solution) (35). Despite the significant advantages, AEMWE still required further investigations/improvements towards the stability and cell efficiency which are more essential for large-scale or commercial applications.

2.2.4. Polymer Electrolyte Membrane electrolysis

2.2.4.1.Working principle of PEM water electrolysis

In Proton Exchange Membrane (PEM) water electrolysis, electricity is used to split water into hydrogen and oxygen. The process begins at the anode, where water molecules break down, producing oxygen gas (O2), protons (H+), and electrons (e-). The oxygen is released from the anode's surface.

The generated protons then travel through a special proton-conducting membrane to the cathode. Simultaneously, the electrons move through an external electrical circuit to the cathode. At the cathode, these protons and electrons recombine to form hydrogen gas (H2).The specific chemical reactions that occur at the anode and cathode in PEM electrolysis are:

$$H_2O\ (l) \rightarrow 1/2O_2\ (g) + 2H^+\ (aq) + 2e^- \quad (19)$$

$$2H^+\ (aq) + 2e^- \rightarrow H_2\ (g) \quad (20)$$

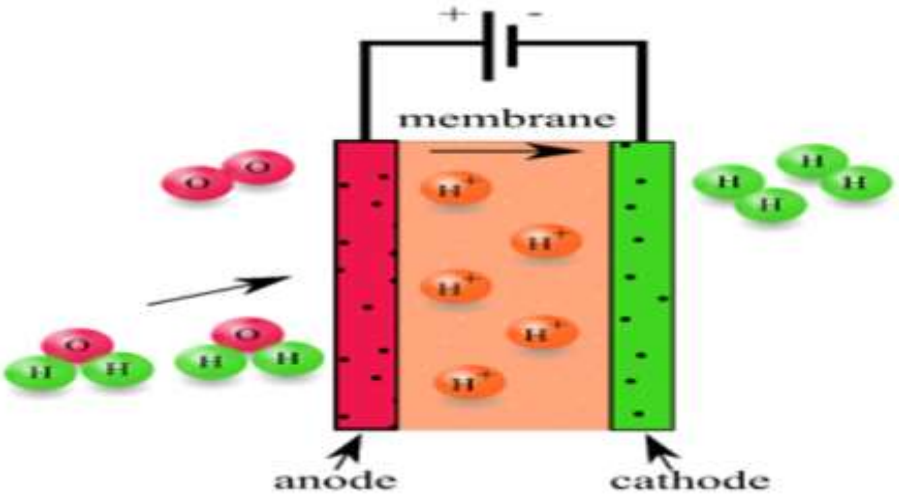

Figure 5: schematic representation of proton exchange membrane electrolyzer (36).

The primary components of a Proton Exchange Membrane (PEM) electrolyzer are the membrane electrode assembly (MEA), which combines the membrane with the anode and cathode electrode materials these, includes the gas diffusion layer, separator plates, and end plates. For the oxygen evolution reaction (OER), the most advanced anode electrocatalysts are currently iridium oxide (IrO2). Conversely, for the hydrogen evolution reaction (HER) at the cathode, the leading electrocatalyst is carbon-supported platinum. (37). These noble metals are more expensive, and iridium is scarcer than platinum. Porous titanium/titanium mesh and carbon cloth are used as anode and cathode gas diffusion layers.

Bipolar plates, which act as separators and end plates, are typically made of titanium and coated with platinum or gold. While various flow field designs exist for these plates, straight parallel flow fields have demonstrated superior performance, particularly in PEM water electrolyzers. Though, these separator plates are expensive and are responsible for 48% of the overall cell cost (38). However, PEM water electrolysis technology is similar to the PEM fuel cell technology; here the sulfonated polymer membrane can be used as an electrolyte.

The essential function of the electrochemical reaction is facilitated by protons (H+), which act as the ionic charge carriers as they move through the proton-conducting membrane. Typically, PEM water electrolysis operates at lower temperatures (30–80 ◦C) with higher current and produces high purity (99.999%) of gases (39). Because the kinetics of hydrogen evolution reaction in PEM water electrolysis is faster than alkaline water electrolysis due to the highly active area of the metal surface of platinum (Pt) electrodes and lower pH of the electrolyte. In addition PEM (Proton Exchange Membrane) water electrolysis offers a significant safety

advantage over traditional alkaline water electrolysis. This is primarily because PEM systems don't use corrosive liquid electrolytes, and they also require a smaller physical space.

Because of these benefits, many water electrolyzer manufacturers around the world are developing large-scale PEM electrolyzers (up to megawatt capacities) for use in various industries and for transportation. These systems have demonstrated impressive durability, with reported stability of up to 60,000 hours of operation with minimal performance degradation. The long-term goal is to achieve 100,000 hours of stability.

The primary distinction between Anion Exchange Membrane (AEM) electrolysis and Proton Exchange Membrane (PEM) electrolysis lies in the electrolyte. PEM electrolysis utilizes a solid, acidic membrane, commonly Nafion, instead of the liquid alkaline electrolyte found in AEM systems. The Nafion membrane offers several benefits, including excellent proton conductivity, the ability to operate at high current densities, and superior mechanical strength and chemical stability. Furthermore, this membrane also serves as a gas separator during hydrogen and oxygen production.

When current is applied across the cell the deionized water present at the anode decomposes into oxygen and hydrogen ions (protons).

Due to the presence of the sulfonic acid (-SO3H) groups in the membrane the protons formed at the anode are able to migrate to the cathode and form hydrogen (40).

While PEM electrolyzers operate under similar conditions to alkaline electrolyzers, a key difference is that PEM systems can achieve much higher current densities, up to 2,000mA/cm2, without a significant drop in efficiency, unlike alkaline systems. (41). This enhanced performance is attributed to the compact design of the membrane electrode assembly (MEA), where the electrodes and membrane are tightly integrated.

Developing catalysts for PEM (Proton Exchange Membrane) electrolyzers is incredibly difficult because of their acidic environment. As a result, the electrodes in these systems typically rely on expensive noble metals like platinum or iridium, which significantly contribute to the overall high cost of the electrolyzer. Currently PEM electrolysers only exist in small scales with maximum hydrogen productivity of 30Nm^3/h (42).

In Proton Exchange Membrane (PEM) electrolysis, the concentration overvoltage can become a more prominent issue compared to typical alkaline electrolyzers because PEM systems operate at higher current densities. The concentration overvoltage can be calculated according

to the Nernst equation (43).

$$U_{\text{con}} = \frac{RT}{zF}\ln\left(\frac{C1}{Co}\right) \quad (17)$$

Where C_0 is the starting concentration of the reagent at the electrode, and C_1 the changed concentration due to mass transfer (mol/m^3). The concentration overvoltage can also be expressed as (44).

$$U_{\text{con}} = -\frac{RT}{zF}\ln\left(1 - \frac{i_d}{i_{lim,d}}\right) \quad (18)$$

Where i_d is the diffusion current density, and $i_{lim,d}$ is the limiting diffusion current density that is directly proportional to the concentration of the reagent. The concentration overvoltage is negligible when the operating current density is below 1 A/cm2. Concentration overpotential would be significant only at very high current densities and would therefore be hardly seen in commercial PEM water electrolysers.

2.2.5. Solid Oxide Electrolysis Cell (SOEC)

A solid-oxide water electrolysis cell (SOEC) is an electrochemical device that transforms electrical energy into chemical energy. These cells typically function at high temperatures, using steam as their water source to produce green hydrogen and oxygen.

In solid-oxide water electrolysis, the process begins at the cathode, where water molecules gain two electrons, splitting into hydrogen gas (H_2) and oxide ions (O^{2-}). The hydrogen is released from the cathode's surface. The remaining oxide ions (O2−) then travel through the ion exchange membrane to the anode. At the anode, these oxide ions lose electrons, forming oxygen gas (O_2), which is then released from the anode's surface. The electrons, meanwhile, move through an external electrical circuit back to the cathode, attracted by its positive charge.

The solid ceramic material that makes up the electrolyte in Solid-Oxide Electrolysis Cells (SOECs) is designed to conduct oxygen ions. These cells need high operating temperatures to ensure the ceramic membrane achieves the necessary ionic conductivity. Beyond its role as an ionic conductor, this membrane also functions as a gas separator within the system. To achieve sufficient ion flow through the ceramic membrane, high operating temperatures are essential. Meanwhile being the ionic conductor for the system, the membrane also acts as a gas separator. As mentioned before, SOECs operate at the vapor-phase, in the range of 800-1000C°, allowing a greater portion of the required energy to come from heat instead of electricity (45). In addition operating at high temperatures necessitates using costly materials and manufacturing techniques.

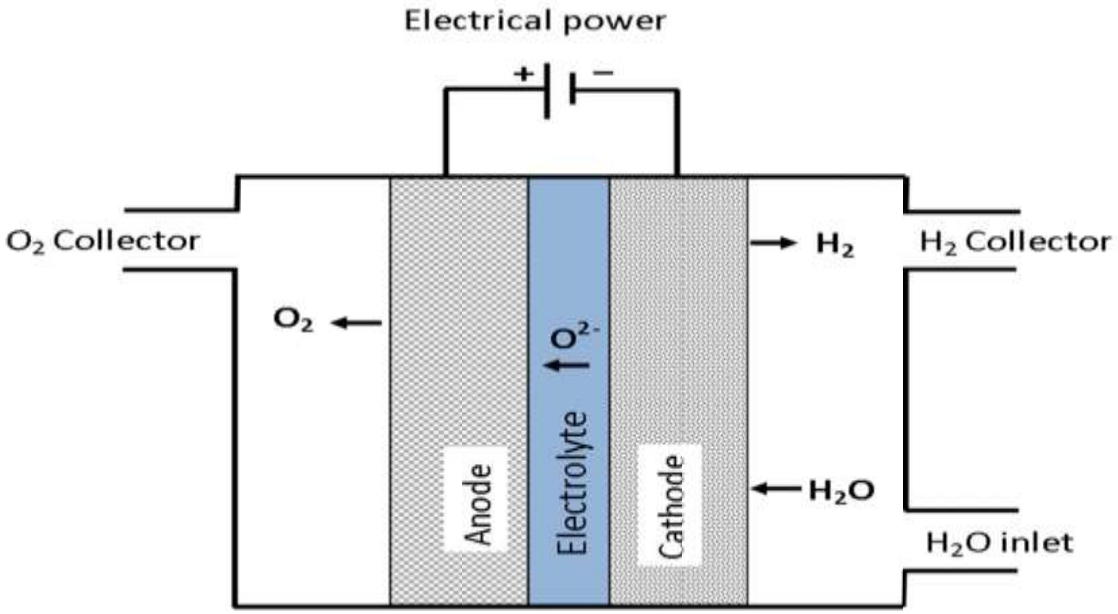

Figure 5: Outline of the operating principle of a SOEC (46)**.**

Chemical reactions taking place in SOE electrolysis at cathode and anode are (47).

$$H_2O\ (g) + 2e^- \rightarrow H_2\ (g) + O_2^- \quad (21)$$

$$O_2^- \rightarrow \frac{1}{2}O_2\ (g) + 2e^- \quad (22)$$

During operation, steam is fed into the cathode. Here, the water is converted into hydrogen gas (H_2) and oxide ions (O_2^-). These anions then move through the porous solid oxide to the anode, where they form oxygen gas and release electrons.

Typically, the solid oxide water electrolyzer operates with water in the form of steam at high temperatures (500–850 ◦C) can drastically reduce the power consumption to split the water into hydrogen and oxygen consequently increase the energy efficiency (48) An electrolysis cell can achieve an efficiency of 90% if you only consider the energy directly used for the splitting reaction, ignoring the energy required to heat the cell. However, once you factor in the efficiency losses from providing that heating, the overall efficiency drops to around 60% (49). Electricity is the biggest expense in producing hydrogen through electrolysis, making the process more energy-efficient can significantly lower the overall cost of hydrogen. Apart from that, Solid oxide water electrolysis (SOEC) has two main advantages over other electrolysis technologies. Firstly, its high operating temperature creates ideal conditions for the chemical reactions, leading to exceptional conversion efficiencies. The second one is that solid oxide water electrolysis can be thermally integrated easily with downstream chemical

synthesis i.e., the production of methanol, dimethyl ether, and ammonia (50). Additionally, solid oxide water electrolysis is appealing because it doesn't need expensive noble metal electrocatalysts and offers high conversion efficiency. However, its widespread commercial use has been hampered by a lack of long-term stability.

A solid oxide water electrolysis cell is made up of three primary parts: two porous electrodes (an anode and a cathode) and a dense ceramic electrolyte that conducts oxide ions ($O2^-$). The most common electrolyte material used in high-temperature solid oxide electrolysis is yttria-stabilized zirconia. In studies regarding electrolyte materials for SOFC, Scandia-stabilized zirconia is known to exhibit highest conductivity (51). Precious metals are used for thin electrical contact layers. The most commonly used electrolyte is yttria-stabilized zirconia, specifically a dense zirconium oxide-based ceramic material doped with 8 mol% yttria. This is because yttria-stabilized zirconia electrolytes demonstrate stable and excellent performance at elevated temperatures ranging from 700–850∘C. Moreover, YSZ electrolyte having high ionic conductivity is associated with good chemical and thermal stability.

In Solid-Oxide Electrolysis Cells (SOECs), the anode materials are usually composite electrodes made of yttria-stabilized zirconia (YSZ) combined with perovskite-type mixed oxides, such as lanthanum strontium cobalt ferrite, which are also used in solid oxide fuel cells (SOFCs). The cathode materials are generally a blend of Nickel (Ni) and ion-conducting particles, similar to the electrolyte material. Currently, solid oxide electrolyzers are still in the research and development phase.

2.4. Factors affecting Alkaline Water Electrolysis

2.4.1. Role of electrolyte concentration in hydrogen production on alkaline water electrolysis

Electrolysis is affected both by the concentration of electrolyte and the type of electrolyte. Potassium hydroxide (KOH) is widely favored as an electrolyte in water electrolysis systems. This preference stems from its excellent electrical conductivity and its less corrosive nature compared to acids, which makes it a practical and safer choice for these applications (52). Moreover the ability of an alkaline electrolyte to conduct electricity is influenced by its temperature. To reduce the electrolyte's resistance in an electrolysis system as much as possible, it's crucial to identify the concentration that offers the best conductivity at the specific operating temperature. In 1997, See and White published a comprehensive study on the conductivity of potassium hydroxide (KOH) solutions. Their research covered

temperatures from -15 to 100 degrees Celsius and KOH concentrations ranging from 15% to 45% by weight. The outcome showed that highest conductivity for aqueous KOH in the range of 80-100°C is reached between 30 and 40 wt. % KOH. (53). The more hydroxide ions in a solution, the higher the pH of the electrolyte (54)

$$P^{H} = 14 - P^{OH-} \quad (23)$$

$$P^{OH-} = -\log [OH-] \quad (24)$$

Increasing the concentration of potassium hydroxide (KOH) makes the solution more alkaline and, consequently, more corrosive. This corrosive effect is further intensified by higher temperatures. To make electrolyzer parts like electrodes and diaphragms last longer, it's often more beneficial to run the system at KOH concentrations and temperatures that are a bit lower than what would give peak performance.

Separately, a newer area of research in Anion Exchange Water Electrolysis (AWE) involves adding ionic activators to the electrolyte. The goal here is to reduce the energy needed for reactions at both the anode and cathode.

The common approach for creating these electrodes involves depositing metal directly onto their surfaces during the process (in-situ deposition). This method is generally preferred because it results in better catalytic performance compared to depositing the metals beforehand or separately (ex-situ processes). For the hydrogen reaction these can be ethylenediamine-based metal chloride complexes (55). Moreover, there's a significant disparity in research efforts when it comes to Anion Exchange Water Electrolysis (AWE) systems: while a great deal of work focuses on electrodes and diaphragms, very little has been done in electrolyte development. This means the field of electrolytes for AWE is largely unexplored, and we can anticipate new discoveries. For example, adding surfactants to the electrolyte that make electrodes more hydrophobic could help reduce the number of gas bubbles clinging to electrode surfaces during operation, potentially improving efficiency.

2.4.2. Impact of different alkaline electrolytes (KOH, NaOH)

To fully understand how electrolyte concentration affects hydrogen production, research has examined to electrolytes as shown in the figure below which represents the increase of hydrogen production rate with increase in electrolyte concentration at different applied DC voltages at constant 32°C operating temperature (56).

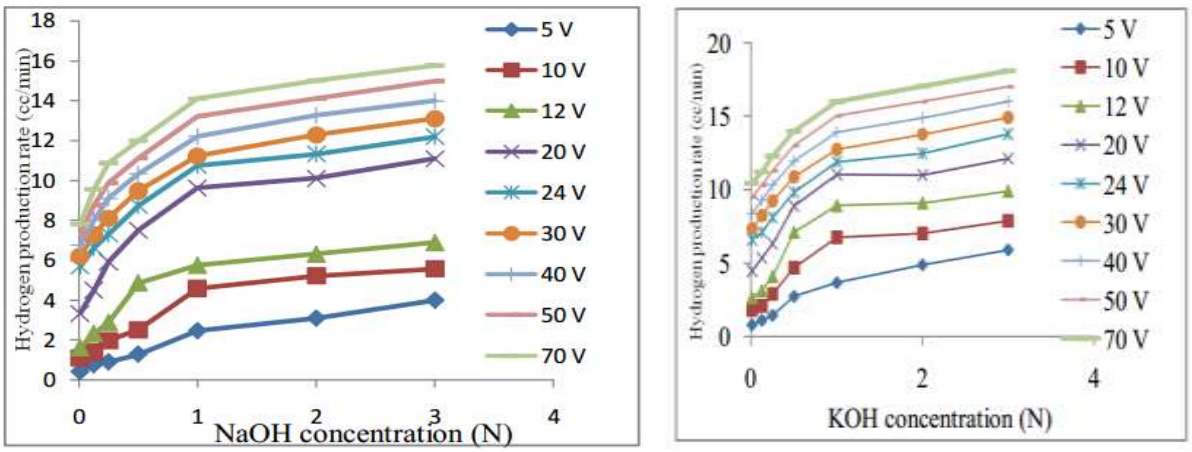

Figure 6: Hydrogen production rate vs. KOH and NaOH solution concentration for different DC **(56).**

Research reveals that the increase in hydrogen production rate is quite small, only 73.7%, when the highest DC voltage of 70 V is applied, across electrolyte concentrations from 0.01 N to 3.0 N. However, the study shows much more significant improvements in hydrogen production at lower DC voltages: And a remarkable 646.84% improvement at just 5 V DC (56). The improvement trend in hydrogen production rate clearly indicates that electrolysis depends not only on the number of ions in the solution, but also on their mobility. While a gradual increase in potassium hydroxide (KOH) electrolyte concentration certainly boosts the number of ions, it subsequently decreases ion mobility due to hindrance.

This report also indicates that electrolyte concentration has a more significant impact on hydrogen production at lower applied voltages, particularly at 5V DC. A greater improvement of 39.24% is observed when the concentration changes between 0.01 N and 0.125 N, also at 5V DC. In contrast, at 70V DC, hydrogen production is significantly less sensitive to concentration changes. At this higher voltage, the improvement is only 6.28% when changing the concentration from 2.0 N to 3.0 N, and just 7.58% for a change between 0.01 N and 0.125 N (56). The data clearly indicates that electrolysis performs better in terms of hydrogen production enhancement at low electrolyte concentrations and low applied voltages. This suggests that at higher electrolyte concentrations, the increased number of ions in the aqueous medium, coupled with irregular interactions between cations and anions (and among like-charged ions), restricts the free movement of ions across the electrodes. Logically, higher applied voltages and higher electrolyte concentrations also lead to greater overpotential loss and a more significant rise in electrolyte temperature compared to operating at lower voltages.

Hence, dilute electrolyte solutions and low applied DC voltages are considered the better option (57) In a similar manner the effect of electrolyte concentration (NaOH solution) on rate hydrogen production was observed to follow the same trend as with variable KOH concentrations but the magnitudes were comparatively smaller.

In recent findings, the rate of hydrogen production showed a minimal increase of 101.92% at the highest DC voltage applied. Conversely, a substantial 852.38% improvement in hydrogen production was observed at the lowest applied DC voltage (5 V DC), using the same range of electrolyte concentrations. Furthermore, the study reported significant enhancements in hydrogen production rates at other applied voltages: These improvements at various voltages fall within the spectrum of the maximum and minimum enhancements observed at the highest and lowest applied voltages.

A comparison of potassium hydroxide (KOH) and sodium hydroxide (NaOH) electrolytic solutions reveals that KOH consistently yields higher hydrogen production rates under all conditions. Conversely, NaOH shows a greater enhancement in its production rate.

This difference might be due to KOH's higher solubility (121g/100ml water for KOH versus 100g/100ml for NaOH) and its higher dissociation constant. Additionally, the water molecules in the hydration shell of sodium ions are more ordered than those around potassium ions, which could contribute to this difference.

In general, both KOH and NaOH solutions increase hydrogen production rates as their concentration and the applied voltage increase. However, the effect of concentration on electrolysis is more pronounced with NaOH solutions compared to KOH solutions.

2.4.3. Electrode Materials

The anode and cathode, positioned on either side of the diaphragm in an electrochemical cell, must remain stable without corroding. Simultaneously, they need to be effective catalysts for the electrochemical reactions occurring on their surfaces. Platinum stands out as an excellent choice because it's stable in alkaline environments and is recognized as the top electrocatalyst for water electrolysis, particularly for hydrogen production. However, due to its high price, other less expensive materials have replaced platinum electrodes in AWE systems (58). Among non-noble metals, nickel is recognized as one of the most stable options when exposed to strong alkaline solutions (59). Nickel is a fairly good catalyst for both hydrogen and oxygen

production. That's why electrodes in Anion Exchange Water Electrolysis (AWE) systems typically use nickel or nickel-plated materials as their core (60). Nickel electrodes are usually enhanced by either adding sulfur to their coatings or by creating a Raney nickel structure on their surface.

It is reported that the improved catalytic efficiency of Ni-S structures, compared to pure nickel electrodes, comes from the formation of strongly absorbed hydrogen within the Ni-S. For Raney nickel catalysts, the process involves selectively removing aluminum or zinc from a NiAl or NiZn alloy. This leaching creates lattice vacancies, resulting in a large surface area and many lattice defects. These defects then serve as active sites for the electrocatalytic reaction (61). Since then, a common strategy to boost the activity of hydrogen electrocatalysts has been to increase their surface area and change their electrocatalytic structure. This is often done by selectively removing one or more elements from metal alloys (62). Besides activating nickel cathodes with Raney nickel and nickel sulfide, researchers have also tried improving their electrocatalytic performance by doping them with active substances like iron (Fe), cobalt (Co), and molybdenum (Mo). However, the stability of these dopants during operation is questionable, and there have been report of Ni-Mo electrocatalysts losing their effectiveness over time. (63). Mixed oxides like, LaNiO3, NiCo2O4, and Co3O4, alongside Raney nickel and Raney cobalt all exhibit high activity as oxygen electrocatalysts for Anion Exchange Water Electrolysis (AWE).

Despite extensive efforts to develop ideal oxygen electrodes, very few of the electrocatalysts created so far possess the necessary durability or are cost-effective enough to be practical for industrial electrolyzers (64). For many of the recently developed electrocatalysts, durability data is often missing. Furthermore, very few of these published electrocatalysts have actually been tested in real-world electrolysis systems or at the relevant high current densities required for practical use.

Two hydrogen research programs from the 1980s and 1990s produced some of the best-performing electrolysis cells ever recorded in an electrolysis stack (65). Previous studies documented a notable cell voltage of 1.6 V when operating at 90°C and a current density of 0.2 A/cm^2. These setups featured perforated nickel electrodes: specifically, the cathode was treated with Ni-S, and the anode utilized spinel oxides containing either nickel or cobalt, or both. A subsequent research initiative at the German Aerospace Center (DLR) advanced this by incorporating vacuum plasma sprayed electrodes.

In these works, the cathode comprised Raney nickel with molybdenum content, while the anode consisted of Raney nickel/cobalt spinel oxides. The reported cell voltages ranged from 1.6 to 1.65 V, measured under conditions of 80°C and 0.3 A/cm^2. Crucially, both of these research initiatives implemented a zero-gap cell structure. This literature further implies that today's cutting-edge industrial electrolyzers commonly feature sulfur- or Raney-activated nickel or nickel-coated steel as their preferred electrode materials for both anode and cathode.

2.5. Wastewater Treatment and Hydrogen Production

2.5.1. Sources and Composition of wastewater

Wastewater's makeup varies depending on its origin, with the primary sources being domestic, industrial, and storm water. Understanding the nature of wastewater is crucial for effective management and for designing appropriate treatment systems.

Traditional wastewater treatment methods primarily aim to remove pollutants. However, these methods often demand extra resources like energy and chemicals. It's important to recognize that wastewater also frequently contains valuable components such as organics with high energy potential, precious minerals, acids, bases, and salts. Therefore, the reuse of treated water and the recovery of these valuable resources play a significant role in achieving sustainable development. Today, wastewaters are generated from various sources and industries. Iron and steel, pulp and paper, mining, petrochemicals, distilleries, food processing, tanneries, dairies, etc., are the top contributors to global wastewater generation

To protect the environment, industries need to put their wastewater through rigorous treatment processes before disposal. Currently, about 380 billion cubic meters of wastewater are generated worldwide every year, and this amount is predicted to jump by 51% by 2050, highlighting a growing environmental challenge.(66). Modern wastewater treatment faces significant hurdles if reuse and recovery aren't incorporated. This challenge stems from the stringent legislation and regulations governments have enacted to safeguard water sources. As a result, numerous treatment methods and strategies for diverse wastewater types are discussed in scientific literature. Conventional methods of wastewater treatment are generally aimed to make it compliant with the government discharge standards (67). In addition, governments have started to charge the industry for the use of water resources along with discharge standards to prevent the pollution and depletion of water basins and groundwater.

This has pushed industries to adopt alternative treatment methods that prioritize reuse and recovery as a way to manage rising water costs. Consequently, several studies have focused on

product recovery, particularly within water-intensive sectors like the textile, food, and metal industries. These wastewaters often contain heavy metals such as zinc, copper, mercury, and cadmium, along with chemicals like cyanide, which can mobilize these metals.

Typically, the pharmaceutical industry generates wastewater with high chemical and biological oxygen demands (COD/BOD). Furthermore, the presence of antibiotics in this wastewater complicates biological treatment processes. Similarly, dye, textile and food industries also produce wastewater with a high COD/BOD ratio, which varies depending on the specific dyes and processes used. Because of this wide array of pollutants, effectively treating these wastewaters often necessitates combining multiple treatment methods.

2.5.2. Impact of wastewater composition on electrolysis performance

The type of feedstock significantly influences electrolysis performance. The "strength" of wastewater, which indicates its contamination level, determines the required treatment time, reactor size, energy needs, and overall production. While high-strength wastewater possesses a greater organic load, allowing for more energy recovery through treatment, it typically demands longer treatment durations, resulting in extended hydraulic retention times. Ultimately, the organic loading rate is directly impacted by the varied organic content from different wastewater sources.

Previous research indicates that bio-electrochemical systems can treat wastewater, achieving specific chemical oxygen demand (COD) values. With advancements in the cost-effectiveness of electrolysis cells, an effective organic loading rate for such a system typically falls between 800 and 1400 mg/L. This range suggests the technology could be applied to urban wastewater, which is considered low-strength with a COD value of 300-500 mg/L (68)**.**

It's unlikely that a single solution will effectively treat all organic waste streams, and some simply aren't suitable for electrolysis. For instance, treating cheese whey with electrolysis yielded no gas, indicating the microorganisms couldn't break down the organic material. This might be because the feedstock inhibits methanogens, which are crucial for the process (69)**.** Further research into the performance of electrolysis for the treatment of different wastewater streams will provide insights in evaluating industry suitability.

Gil-Carrera et al. discovered that an electrolyzer designed for hydrogen production, with an oxygen loading rate exceeding 1000–2000 mg COD/L/day, could compete effectively with activated sludge treatment (70)**.** For oxygen loading rates of >2000 mg/L/day, the also indicates

that it is possible to increase the hydraulic retention rate and still be competitive against activated sludge treatment.

The energy demands for traditional wastewater treatment are high, especially for activated sludge treatment, equating to 60% of the total energy needed. Using microbial cell for wastewater treatment has resulted in a 1.7 times higher energy production and an accelerated substrate removal (71). Microbial cell electrolysis could drastically cut the energy needed for wastewater treatment globally. Research is growing to understand how well it treats various waste streams, offering insights into how different microbial communities interact with specific substances. For microbial cell electrolysis to be commercially successful, it'll need to out-compete existing technologies like anaerobic digestion (AD) systems (which focus on energy production) or activated sludge systems (which prioritize removing nutrients and chemical oxygen demand). Although many studies have looked at treating urban wastewater, their hydraulic retention times (HRT) typically fall between 8 to 48 hours, which is much longer than the recommended 5–9 hours. Generally, researchers should assess the economic viability of treatment of particular wastes and compare microbial cell electrolysis performance to the state-of-the-art technology used by industry.

2.6. Coupled Wastewater Treatment and Hydrogen Production:

The Wastewater Electrolysis Cell, shown in the figure below, offers a practical way to achieve self-sustaining, on-site wastewater treatment while also producing hydrogen in a decentralized manner. Naturally abundant chloride ions in saline wastewater provide electrical conductivity. When this wastewater is used as an electrolyte for electrochemical water splitting, these chloride ions are oxidized into reactive chlorine species, which then eliminate pollutants like organics, ammonium ions, and bacteria. (72).

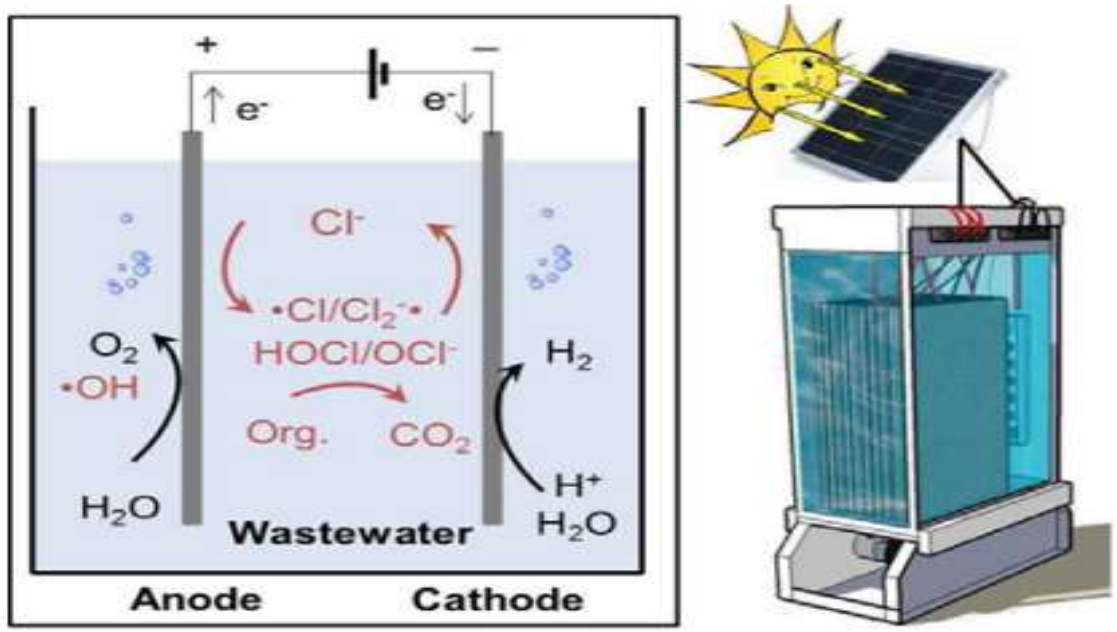

Figure 7: Schematic diagram of photovoltaic-powered wastewater electrolysis cell for on-site collection and treatment of wastewater. Adopted from (73)

This eco-friendly chemical process doesn't need external chemicals and can run entirely on photovoltaic (PV) panels, making it independent of traditional infrastructure. As wastewater is treated, the system can also store solar energy by producing hydrogen at the cathode (74). This hydrogen can then serve as a secondary energy source for the Wastewater Electrolysis Cell itself or for other household needs.

By this dual-functioning approach, one can reduce the cost of electrolytic H_2 production, particularly the cost for electrolyte and electricity, by saving energy for water treatment. Recent statistics suggest that handling water and wastewater occupied up to 4% of total electricity consumption in industries.

2.6.1. Electrochemical Degradation of Environmental Pollutants

Electrochemical systems are frequently employed to treat high-salinity wastewaters, including those from the textile dyeing, landfill leachate, olive mill, tannery, and livestock industries, as well as reverse osmosis concentrates, among other applications. Furthermore, the oxidative degradation of persistent or non-biodegradable pollutants—like pesticides, pharmaceuticals, personal care products, polycyclic aromatic hydrocarbons, and various aliphatic/phenolic compounds—has been extensively studied in electrochemical cells. These investigations

typically use electrolytes prepared in the laboratory, with the pollutants of interest added to a background electrolyte.

More thorough reviews on the electrochemical wastewater treatment can be found in recent report by Martinez and Ferro (75). Electrochemical wastewater treatment systems with a wide array of electrodes have conventionally categorized the electro-catalytic anodes into the following two groups: active electrodes and non-active anodes.

While "non-active" anodes do facilitate the heterogeneous oxidation of pollutants, this term actually refers to their electrocatalytic activity for the oxygen evolution reaction. Essentially, common non-active anodes like SnO_2, PbO_2, and boron-doped diamond are characterized by their relatively high overpotential for oxygen evolution.

Conversely, mixed metal oxide electrodes containing IrO_2or RuO_2 are known as active anodes in electrochemical wastewater treatment because they have the lowest oxygen evolution reaction overpotential. From a mechanistic perspective, electrochemical pollutant oxidation is categorized as either direct or indirect. While researchers have slightly different definitions, direct oxidation generally means pollutants are oxidized heterogeneously on the anode's surface. Indirect oxidation, on the other hand, involves the homogeneous oxidation of pollutants, often with the help of reactive oxygen species (ROS) and reactive chlorine species (RCS).

Despite the potential for direct electron transfer from pollutants to the anode, surface-bound reactive oxygen species, particularly the hydroxyl radical (OH^-), are almost always credited with heterogeneous direct oxidation. When an energized anode releases free reactive oxygen species (like OH^-, H_2O_2, and O_3) into the solution—either as intermediate steps or side products of oxygen evolution—indirect oxidation can happen.

Yet, differentiating between direct oxidation by these surface species and indirect oxidation by their free counterparts is challenging due to the fleeting nature and low concentration of free reactive oxygen species. This is why reactive chlorine species, formed when chloride is in the electrolyte, are recognized as the primary agents for indirect oxidation.

2.6.2. Molecular H_2 Production from Electrolysis of Wastewater

A wastewater electrolysis cell could be a promising way to produce hydrogen locally while treating wastewater right where it's generated. Hoffmann and co-workers (76)have investigated the electrolytic H_2 production from model organics containing solutions or real wastewater (domestic or industrial wastewater) in circum-neutral pH. In these reports, inexpensive stainless steel was employed as a cathode. The cathodic current efficiency and energy efficiency of H_2 production have been reported to vary widely depending on the electrolyte composition cell configuration (distance between anode and cathode, geometric area), and operating condition (applied cell voltage). The reported values ranged from 40 to 80% for current efficiency and from 15 - 60% for energy efficiency.

The major scavenging reactions for H_2 production include reduction of reactive chlorine species in NaCl background and reduction of oxygen. The current efficiency for H_2 production was usually higher in the $NaSO_4$ background than in the NaCl background. In the NaCl background solution, a presence of electron donating substrates enhances the current and energy efficiency of H_2 production. For example, batch injections of small amounts of organics including urea, phenol, and other oxidizable organics sharply enhanced the production of H_2.

The reactive chlorine species competing with water and proton to accept electrons can be readily quenched by the organics to enhance the H_2 production. The energy efficiency of H_2 production decreased as the electrical conductivity decreased or applied cell voltage (or current) increased, primarily owing to the increase in Ohmic energy loss. Park et al (77) would be the first to attempt electrolytic H_2 production from dilute aqueous solutions, which was solely powered by photovoltaic panels. In these reports, the sequential energy losses in the PV panel and the electrolysis cell resulted in net energy conversion efficiency about 1% (2.5% energy efficiency for solar light to direct current and 30~60% energy efficiency for the direct current to H_2).

2.7. Challenges and Future Directions

2.7.1. Technical challenges in scaling up the process

Future aspects of hydrogen production from wastewaters However, considering that industrial hydrogen production prices will decrease, methods should be studied and researched extensively to catch industrial progress.

The European Union projects that the cost of low-carbon hydrogen derived from fossil fuels will be between $2.3 and $2.9 per kilogram in 2030, and $1.29 to $2.81 per kilogram in 2050 (EC, 2020). While hydrogen production using fossil fuels can be as inexpensive as $1 per kilogram, the cost for clean hydrogen produced via renewable resources and sustainable methods ranges from $2 to $10 per kilogram (78).

The hydrogen production costs for the methods discussed in the article are significantly higher than current industrial hydrogen prices (79). These methods don't seem to be commercially viable when viewed in isolation. Yet, because industries must adhere to stringent wastewater discharge standards—which their treatment systems are designed to meet—evaluating these new methods requires a multi-criteria approach. This includes assessing:

- The effectiveness of the wastewater treatment.
- The quantity of hydrogen produced from the treated wastewater.
- The costs that can be offset by hydrogen generation.
- The practicality of implementing the methods in industrial environments.

Due to variations in industry type and size, the capital and operating costs of wastewater treatment plants are highly variable, preventing a definitive statement on overall treatment expenses. For this reason, a critical aspect of cost analysis is examining the cost associated with treating a unit volume of wastewater, in addition to the cost of hydrogen production.

Though hydrogen production from wastewaters is still a developing area, focusing on the production of hydrogen from wastewaters to recede fossil fuel consumption to produce renewable and clean hydrogen will reduce the environmental impact of human activities and create an alternative to fossil fuels. For hydrogen generation from wastewaters to become a practical and competitive option, the unit wastewater treatment costs, offset by the value of the generated hydrogen, must be more economical than current hydrogen production methods.

One of these methods, photonic hydrogen production methods currently suffers from a very low solar-to-hydrogen conversion ratio. Despite ongoing extensive research into novel

photoactive materials, it will likely be a long time before these methods are commercially viable. However, advancements in nanotechnology are accelerating the development of new photoactive materials. Future progress in photonic hydrogen production will largely depend on innovations in reactor, electrode, and nanomaterial designs.

On the other hand, electrolysis is a mature technology for producing hydrogen, but its application in wastewater treatment is still developing. While it's capable of treating certain types of wastewater, more research and innovation are needed to advance its use in this area. In addition emerging technologies like reverse electrodialysis and microbial electrodialysis are particularly new and require extensive study and experimentation to become viable for waste water electrolysis

In general the primary drivers behind resource recovery research are government restrictions on groundwater use and pricing policies designed to protect water sources. This is particularly true in water-intensive industries, where rising water and discharge costs have pushed them towards adopting recovery technologies (80). While generating hydrogen from wastewaters is not a common method yet. Given the significant carbon footprint of conventional treatment—due to its high energy, chemical, and cost requirements—it's essential to expand the use of more sustainable methods.

Chapter Three

3.0. Materials and Methodology

3.1.Sample and sampling

Alkaline waste water was collected from RBSC bottle washer room along 12 hour shift by the machine operator. Since this part of the production process uses sodium hydroxide solution (caustic soda) to clean impurities and kill microbes at relatively high temperature i took my

sample directly from this part for it was the source of the most alkaline solution by volume, almost all of it. This sampling technique was designed for its more than two benefits. Note that this research was solely conducted in one site-RBSC.

1. First the amount of impurities like organic maters, dissolved solids, microbes and other impurities are mostly removed in the prior stage of the washing process. So that this made taking a sample from the spot important by necessity.
2. Its second benefit was to minimize the pretreatment requirement of the alkaline waste water.

As it was the near final stage of the washing process the alkaline solution used at this stage will be mixed with the other waste waters streams from the factory and be discharged downstream. After the alkaline waste water was discharged dawn stream it would have needed complex pretreatment before make use of it for electrolysis. Therefore taking the sample, before the alkaline waste water was mixed with other waste water in downstream was appropriate by necessity as well as better for simplicity.

The sample collected was not immediately taken for laboratory for electrolysis. The sample was measured for its constituent elements and impurities. The main parameters measured were:

a. Concentration or pH
b. Total dissolved solids (TDS)
c. Total suspended solid (TSS)

A critical step before electrolysis was to thoroughly understand the composition of the wastewater the pH, TDS and TSS which were thoroughly measured.

Different measuring instruments were employed to measure and characterize the sample waste water before it was utilized for experimentation. Since electrolysis process was highly sensitive to electrolyte composition the sample waste was measured and characterized for constituent composition.

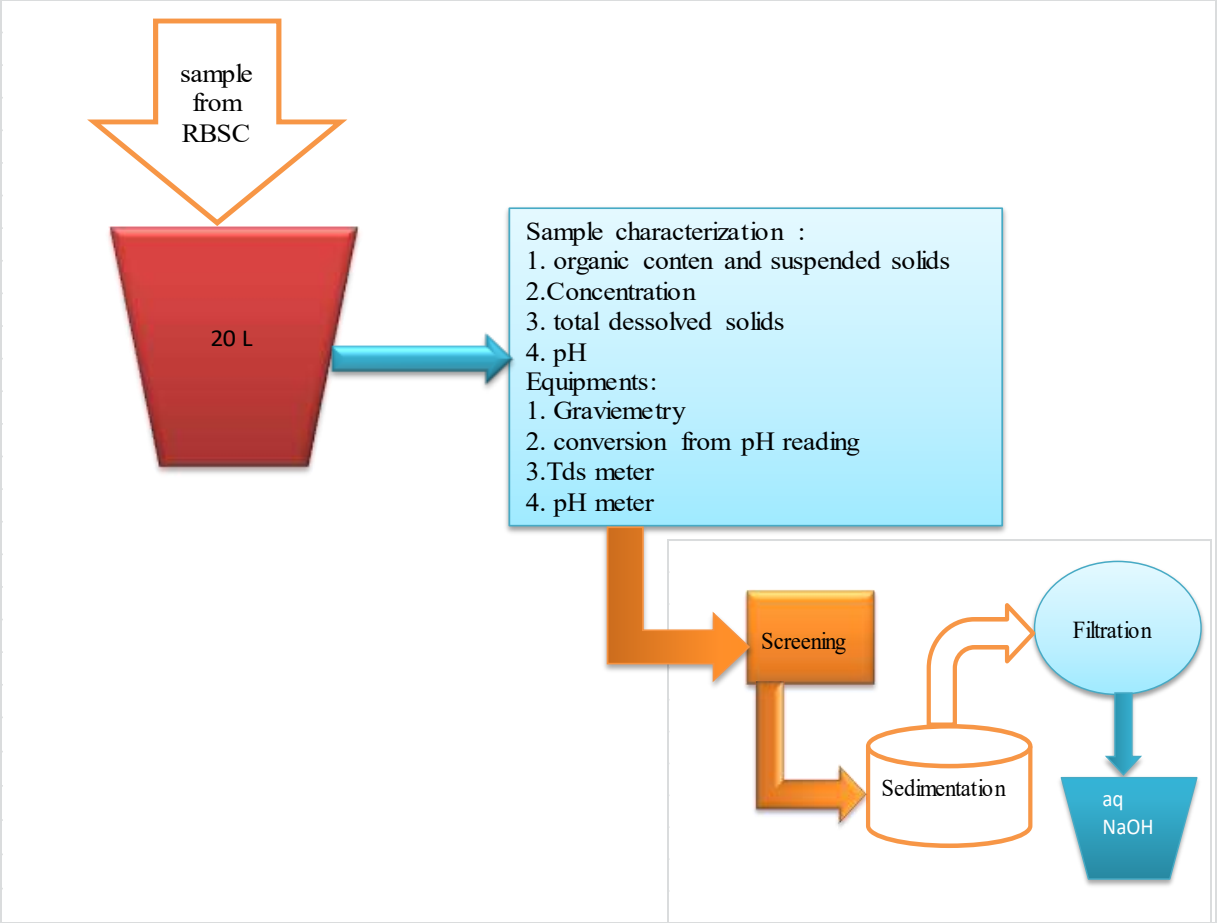

Figure 8: summary flow sheet; sample procurement, characterization and treatment.

3.1.1. Sample Characterization instruments

The sample was characterized for its constituent impurities – mainly – concentration and other impurities. The concentration was converted from pH reading of the digital meter whilst the TSS was measured using gravimeter. I used digital pH meter, TDS meter and gravimeter to measure concentration, dissolved solids and suspended solids. Some of the instruments gave indirect reading of the characteristic measured (i.e. pH was converted into concentration by conversion formula, mass of dried sample was estimated as TSS) whilst others gave direct reading like the TDS and thermometer.

a. **Digital pH Meter:** It consists of a probe that measures the electrical potential of the solution and converts it to a pH reading on a digital display. Using this instrument the reading displayed pH value of 13.398. Converting pH into concentration gives 10g/l of NaOH.
b. **TDS Measure:** This portable meter measures the electrical conductivity of the solution, which is proportional to the total concentration of dissolved ions and salts. Higher

conductivity indicates higher TDS. This method relies on the principle that the electrical conductivity of water increases with the concentration of dissolved ions.

A TDS meter is a portable instrument that measures the electrical conductivity of sample and converts it to an estimated TDS value. Using this instrument, it displayed 13.56g of dissolved solid per liter. One shortcoming of measuring the TDS was it does not show the constituents of the dissolved solids. This was later mitigated removing the dissolved solids by analytical methods

c. **Stirrer:** this instrument was used for two purposes. First in order to make accurate characterization of sample, sample content must be kept uniform throughout the container (i.e same impurities can settle to the bottom of container hindering accurate estimation of sample content). Second, to keep temperature constant throughout the electrolytes. I used inbuilt stirrer for this purpose.
d. **TSS measure:** TSS was measured using gravimetric method. Gravimetery works by removing the moisture - by evaporation of moisture - and measuring the remaining content. To make accurate estimate the sample suspended solid the moisture content was dried first by evaporator and then sundried until the moisture reading was 0%. For one liter of sample alkaline waste water 10g/l of TSS was observed.

3.2.Sample treatment

3.2.1. Technologies for sample treatment

To control experimental variables the sample was treated to remove impurities like dissolved solids, carbon content and suspended solids, in which some of them can be read in the form of TSS. This in effect required (1) effective removal of suspended solids from sample so that the effluent was clear; (2) the subnatant stream of sludge was removed and measured. Different technologies and methods can be employed to treat the sample. Some of them are;

a. **Centrifugation**: This method uses centrifugal force to separate particles of different densities. Heavier grit particles will settle at the bottom of the centrifuge tube, while the liquid sample remains on top for easy separation.
b. **Gravity Settling**: Using a settling container like a graduated cylinder. Allow the sample to sit undisturbed for some time, allowing the grit to settle at the bottom. Then the impurities can be carefully siphoned off.

c. **Coarse Mesh Screen**: Effective for removing larger debris and sand particles.
d. **Filter Paper**: Removes finer grit particles depending on the pore size.
e. **Membrane Filters**: Available in various pore sizes for highly efficient removal of even very fine grit.

Most of the time waste water treatment employs screening, grit removal and sedimentation as pretreatment methods before primary clarification. During screening, large solids and debris are removed by passing wastewater through a series of screens or mesh filters.

Screening, grit removal and sedimentation are used as pretreatment methods before primary clarification. The main purpose of these methods was to protect downstream equipment and improve the effectiveness of subsequent treatment stages by removing or grinding large solids and debris.

Sedimentation is a simple and common method for removing TSS from wastewater, which reduces BOD and COD (81). Sedimentation is used to remove TSS in wastewater from pulp and paper mills. Sedimentation can effectively remove more than 80% TSS (82).

In conventional waste water treatment plants sedimentation tanks, also known as primary settling tanks and primary clarifiers are placed after screening and grit removal and before secondary treatments.

Sedimentation is a highly effective method for removing suspended solids from industrial wastewater, commonly using rectangular or circular clarifiers. While nearly all domestic sewage plants utilize sedimentation, it's generally recommended for industrial wastewater only when it's mixed with domestic sewage or contains a significant amount of easily settleable solids.

Several factors influence the effectiveness of sedimentation tanks, including detention time; wastewater properties, tank depth, and particle size are some of the factors that affect sedimentation. The nature of the suspended solids is also crucial. Small, difficult-to-settle particles can be treated through flocculation, where they are attached to tiny air bubbles and float to the surface for removal by skimming.

For industrial wastes with larger, varied-sized suspended solids (like those from canneries, pulp and paper mills, or poultry processing), screening offers a cost-effective and efficient way to quickly separate these solids.

When it comes to selection of desired wastewater treatment equipment size of particles and level of removal must play into consideration. In consequence filtration is better for complete removal of finer particles, while sedimentation is suitable for larger particles.

In addition there are two types of sedimentation tanks based on their geometry of construction, rectangular and circular type. While rectangular tanks have a lower construction cost and can have a longer retention time but are less effective for treating wastewater with high TSS, whereas circular tanks have a lower maintenance cost and easier sludge collection but a shorter retention time. In addition the greater the tank depth, the greater the opportunity for contact among particles, and so sedimentation depends on the depth as well as on the properties of the fluid and the particles.

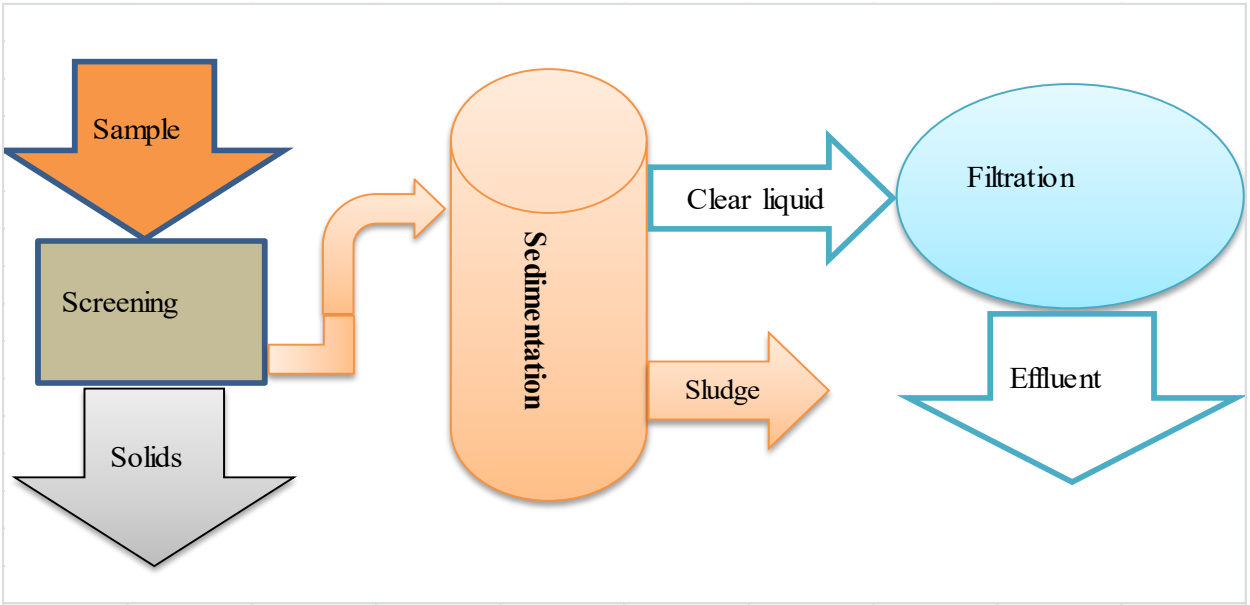

Figure 9: Schematic diagram of sample treatment by screening, sedimentation and filtration.

Based on the aforementioned criteria from literature and experience I chose screening, sedimentation and filtration for my lab case. In this experiment circular sedimentation tank of with height of 1.5M and diameter of 20cm was employed. In the sedimentation tank, the wastewater was made to remain still to cause reduction and removal of TSS, organic matter, and other particles settle to the bottom of the tank and form a layer of sludge. The sample was left for 24hr until the heavier particles settle and the clear liquid showed in the container.

After sedimentation the clarified effluent was collected near the top of the tank around the wastewater surface and it was further filtered with laboratory filtration for untraced solid particles. As mentioned in the literature filtration is a versatile option for removing both large and small particles. After this process the sample displayed substantial decrease in TSS. Final reading of TSS was below 1g/l and clear liquid of 15L was extracted.

3.3.Experimental procedure

3.3.1. General assumptions

While conducting the experiment some assumptions were made

1. The solubility of oxygen and hydrogen in the electrolyte is negligible; Even though oxygen and hydrogen are not soluble gases; experimental volume for each test run was minimal at 1L per run to make sure the gases did not remain trapped under the electrolyte.
2. All gases are assumed to exhibit ideal behavior.
3. Diffusion of hydrogen and oxygen across the electrolyte was negligible; when hydrogen and oxygen are extracted at the cathode and anode sides respectively, diffusion across electrolyte complicates hydrogen/product volume measurement. To eliminate diffusion the electrolyser was designed so that hydrogen was only removed through cathode and oxygen through anode eliminating the probability of product mixing on the electrolyte.
4. All experiments were at atmospheric pressure; to control the effect of pressure difference on the experimental results it was set at 1atm for all test-run.
5. The experimental variable (one variable at a time) was set to vary holding other variables constant.

3.3.2. Preparation of Electrolysis Cell:

- Laboratory-scale electrolysis cell with nickel and graphite for the anode and cathode was prepared.
- Plate heat was applied to control the experiment at different temperatures.

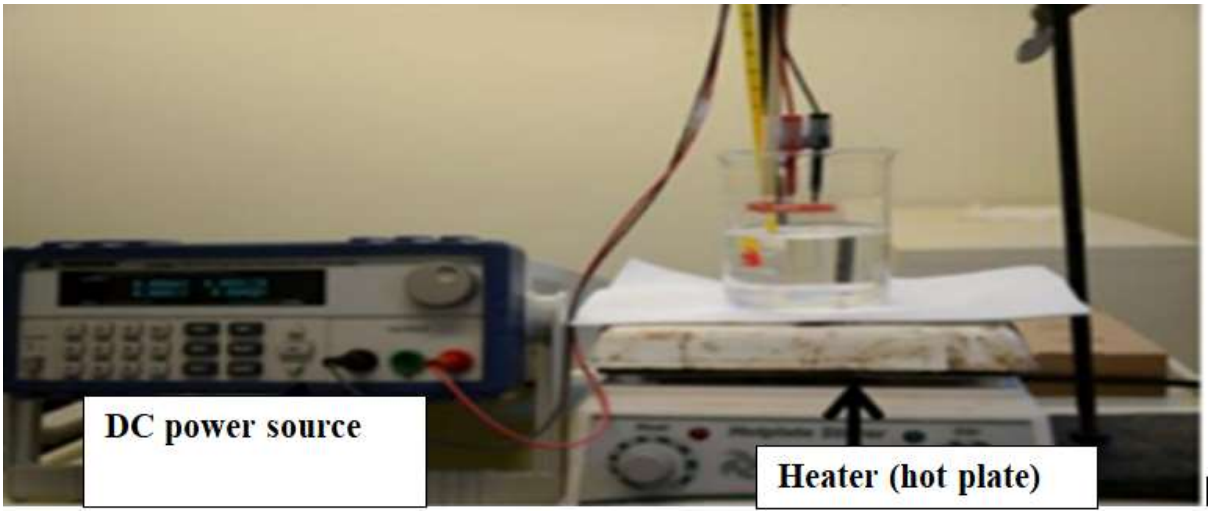

Figure 10: electrolysis process instrumentation.

3.3.3. Electrolyte Preparation: Using the treated alkaline waste water of 10g/l a 15 liter of aqueous NaOH;

a. By adding NaOH to further concentrate or adding pure water to reduce the concentration four sets of 1L solution was sampled for experimentation with concentration range 5g/l, 10g/l, 15g/l and 20g/l. temperature and current are set at a value 30^0C and 0.25A respectively.

b. To observe the effect of specific current six sets of 1L electrolyte was prepared (i.e. experimental test run were conducted at current 0.3, 0.4, 0.5, 0.6, 0.7 and 0.8 amps). This experiment was conducted at constant concentration of 10g/l and 40^0C.

c. To analyze effect of temperature on hydrogen evolution reaction rate five sets of 1L for the five trials (i.e. at temperature of 30, 40, 50, 60, and 70 ^{o}C). Concentration and

3.4.Hydrogen production experimental setup:

At the beginning of each experiment, the electrolyser was filled with 1L of alkaline waste water and the electrodes were immersed. The terminals of power supply were connected to the electrodes. One electrode was connected to the positive source of the power supply (anode) and the other electrode was connected to the negative source (cathode). These electrodes were held in the lower ends of two graduated tubes to collect gases produced. Each collecting tube was graduated from 0 to 40 ml. The distance between the electrodes had been set at approximately 1cm.

3.4.1. Stoichiometry

As electrical current was applied electrons flow from the external power source to the cathode and anode. This leads to the decomposition of water molecules into hydrogen and oxygen gases. Water molecules were reduced at the cathode, gaining electrons and splitting into hydrogen gas and hydroxide ions (OH^-) and Hydroxide ions from the electrolyte were oxidized at the anode, losing electrons and forming oxygen gas and water molecules.

Cathode (reduction): $2H_2O\ (l) + 2e^- \rightarrow H_2\ (g) + 2\ OH^-\ (aq)$ (25)

Anode (oxidation): $4OH^-\ (aq) \rightarrow O_2\ (g) + 2H_2O\ (l) + 4e^-$ (26)

Combining either half reaction pair yields the same overall decomposition of water into oxygen and hydrogen:

$2H_2O\ (l) \rightarrow 2H_2\ (g) + O_2\ (g)$ (27)

Producing one molecule of oxygen requires the transfer of 4 electrons compared to just 2 electrons for hydrogen. This means for a given amount of current passed through the system, twice the amount of hydrogen was produced compared to oxygen.

Generally the number of hydrogen molecules produced was thus twice the number of oxygen molecules. Assuming equal temperature and pressure for both gases, the produced hydrogen gas had therefore twice the volume of the produced oxygen gas. The number of electrons pushed through the water was twice the number of generated hydrogen molecules and four times the number of generated oxygen molecules.

3.4.2. Kinetic Considerations:

Since the volume of the container (electrolyser) was constant, the higher production rate of hydrogen molecules translates to a higher partial pressure of hydrogen gas compared to oxygen gas. The overall reaction for water electrolysis tells us the ratio of products formed: $2H_2O\ (l) \rightarrow 2H_2\ (g) + O_2\ (g)$. This means for every two molecules of hydrogen produced, only one molecule of oxygen was generated. In alkaline water electrolysis, hydrogen gas (H_2) has a higher partial pressure than oxygen gas (O_2) at the same temperature and pressure conditions within the electrolyser.

As the electrolysis starts the electrolyte inside the tubes was pushed out and the reading of the amount of hydrogen produced was taken from the cylinder reading.

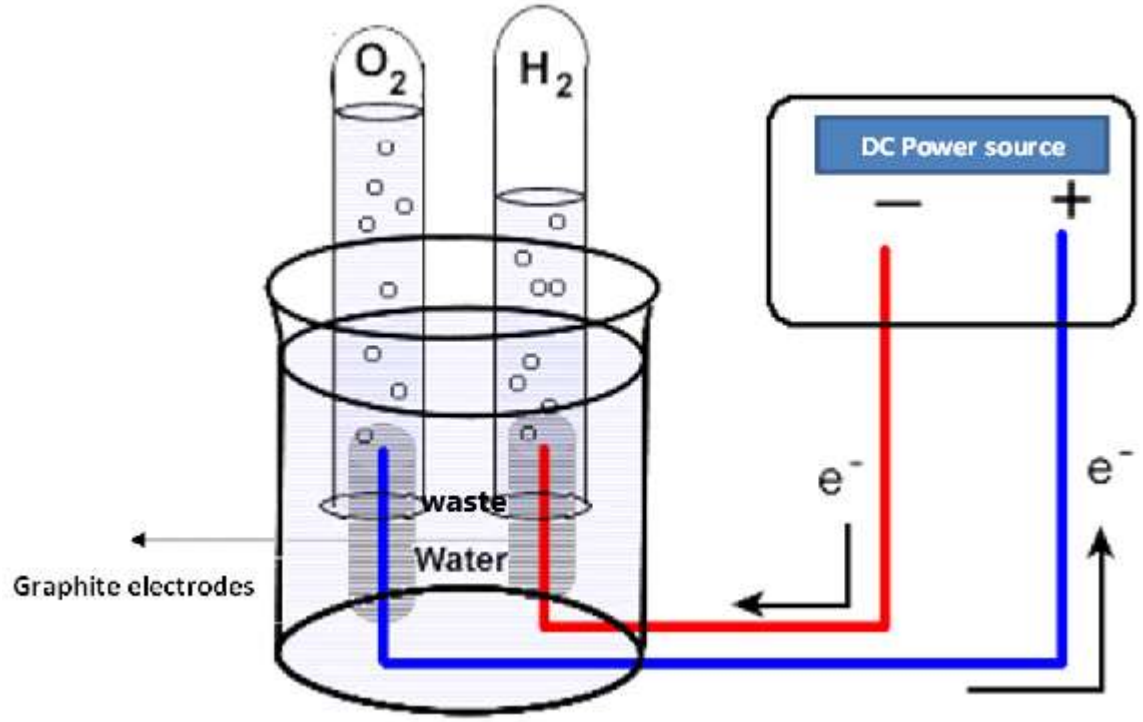

Figure 11: electrolysis of alkaline waste water using nickel anode and graphite cathode.

The above design helps to meet the need for flexibility in the choice of voltage and current. A conventional Power Supply Unit has a fixed current limit giving a power capability that reduces directly with the output voltage.

3.4.3. Instrumentation

Water electrolyser in Fig11 uses a cylindrical container with 40cm height, 5cm diameter and electrodes of pure grade nickel and graphite. The two electrodes in which each of them was 3 cm height, the gap between the electrodes was 1 cm. In addition graduated tube, thermometer, pH meter, stop watch, ammeter/voltmeter were used to measure volume of hydrogen, temperature, time lapse and current/voltage in the process.

- **Hydrogen measurement**: The volume of gas made in a reaction was measured using graduated tube. The gas was collected over the electrolyte using an upturned graduated cylinder. The graduated tube or measuring cylinder was filled with the electrolyte before the start of the reaction. As the reaction starts and gas bubbles form, it pushes the electrolyte out. The electrolyte level was then read off the scale on the measuring cylinder. This works well for insoluble gases, such as hydrogen, or gases that do not dissolve easily in water. The volume of gas was determined by reading the scale on the side reading of the graduated tube.

- **Working with variable Temperature**: while working with temperature the other variables were held constant. While other variables were constant the temperature of the alkaline waste water was made to vary.
- **Concentration**: initially the sample measured 10g/l concentration. This concentration was made to rise and decline its value by adding NaOH powder and by carefully adding and measuring each fraction of electrolyte consecutively. The measurement was made by pH meter then pH was converted to concentration.
- **Current**: similar method was employed while working with current. The other variables remained constant at certain value, concentration at 10g/l and temperature at 30^0C. Current was controlled by the DC meter and was regulated for different ranges for the experimentation.
- **Electrodes**:-I used Nickel as anode material. Nickels highly active and accessibility makes it easy to utilize as anode material. For cathode I used graphite. Graphite was selected to avoid the use of other noble metals. In addition graphite is cost effective and abundant.
- **DC generator:** converts alternative current that comes from power grids to direct current. For the electrical supply of the electrolysis cell, a DC generator was used. It gives variable combinations of current and voltage within a maximum power range, up to 5 Amps and 10 V. And thermo meter was used to measure the temperature.

3.5.Calculation of faradic Efficiency

3.5.1. Faradic efficiency

faradaic efficiency is a one of the quantitative analysis useful for to determine the how many electrons are transported in the external circuit to the surface of electrode for conducting the electrochemical reaction either oxygen evolution reaction (OER) or hydrogen evolution reaction (HER) and other electrochemical reactions in the electrolytes.

Therefore, the faradaic efficiency can be defined as the ratio between experimentally evolved volume of gas value (hydrogen or oxygen) and theoretically calculated volume of gas value, as shown in:

$$\eta = \frac{Vr}{Vi} * 100 \quad (28)$$

When the practical amount produced in the experiment can be measured by water-gas displacement method or gas chromatography analysis (83). Using faradays law of electrolysis the ideal volume of hydrogen is calculated as

$$V_i = \frac{I*Vm*t}{2*F} \quad (29)$$

Where I is the current, V_m is the molar volume of ideal gas under standard condition and F is faradays number (96485c/mol) .The real volume of hydrogen is calculated as:

$$V_r = V_e * \frac{Ts}{Te} \quad (30)$$

Whilst Ve is the volume obtained by observation or measurement, T_s is the standard temperature, and T_e is the measured temperature at the given period.

3.5.2. Factors affecting faradic efficiency

Two key factors affecting faradic efficiency are energy hydrogen Overvoltage and Ohmic Losses.

- **Hydrogen Overvoltage**: Hydrogen overvoltage refers to the extra electrical energy required beyond the theoretical thermodynamic potential to drive the hydrogen evolution reaction (HER) at the cathode. Even under ideal conditions, some additional energy is needed. Overcome the kinetic limitations associated with the HER process. These limitations include: activation energy and mass transfer limitation.
    - **Activation energy**: The initial energy required for the reaction to begin.
    - **Mass transfer limitations**: The movement of electrolyte to the electrode surface.

Generally higher hydrogen overvoltage translates to a higher cell voltage needed for electrolysis. This means more electrical energy is used to overcome the limitations at the cathode, reducing the overall efficiency of the process.

- **Ohmic Losses**: Ohmic losses refer to the energy wasted due to the electrical resistance within the electrolysis cell. This resistance can arise from several sources:
    - **Electrolyte resistance**: The resistance of the electrolyte solution to the flow of ions.
    - **Electrode resistance**: The resistance of the electrode materials themselves.

Ohmic losses contribute to a higher cell voltage required for electrolysis. The electrical energy used to overcome this resistance is not directly involved in the water splitting reaction, thus decreasing efficiency.

Chapter four

4. Results and Discussions

4.1. Effect of current variability

Among the factors that affect electrolysis the amount of current applied stands as the key parameter, because the whole principle of electrolysis depends on applying current to electrolyte to break the hydrogen bond and produce hydrogen and oxygen. The same is true in electrolysis of alkaline waste water. Hence to analyze the effect of current variability on alkaline waste water electrolysis I experimented with six sets of amperage at range of 0.3A up to 0.8A with range between tests was 0.1A.

The experimental results revealed that at constant temperature of 40^0C (313.15K) and 10 g/L of electrolyte, and the time required to reach different sets of volume were measured from the experiment. At amperage of 0.3A the reading from Table-3&Fig-13 indicates that to reach volume of hydrogen 2ml, 4ml, 6ml, 8ml and 10ml the time reading were, 132.98sec,265.95sec,398.93sec,531.91sec and 664.88sec respectively. From the same references Table-3&Fig-13 the results at different set of current to produce different ranges of volume indicated declining time pattern with rise of current. For example for 0.4A the volume versus time relation to reach volume of 2ml, 4ml, 6ml, 8ml and 10ml time was 93.97sec,187.95sec,281.92sec,375.89sec, and 469.87sec respectively. In addition at the last set of current 0.8A, to produce volume range of 2ml, 4ml, 6ml, 8ml and 10ml hydrogen the time needed to reach the respective volume was 48.84sec, 92.11sec, 138.21sec, 184.16sec and 230.08sec respectively. To extract 2ml of hydrogen at 0.3A, 0.4A and 0.8A it took 132.98sec, 93.97sec and 48.84sec respectively. These observations confirm that as applied amperage rises the time needed to reach each volume set of hydrogen declines.

In addition to reach 10 ml hydrogen by volume at the amperages of 0.3A, 0.4A, 0.5A, 0.6A, 0.7A and 0.8A 15 time set of 664.88sec, 469.87sec, 349.24sec, 271.66sec, 244.21 and 230.08sec respectively. From these data we can analyze that with rising amperage the time required to reach 10ml by volume of hydrogen significantly declined from 664.88sec at 0.3A to230.08sec at 0.8A. This is due to the rate of electrical charge conducted by the solution, the higher the current flow (in Amps), the more electrons are transfers per unit time – seconds- the reaction for producing hydrogen on the surface of the electrode become more noticeable. Using graphite as cathode a study displayed similar pattern of the effect of current density on

hydrogen production by electrolysis in which the conditions were 24% NaOH (84). These results display similar trend with the result analysis shown in figure 13.

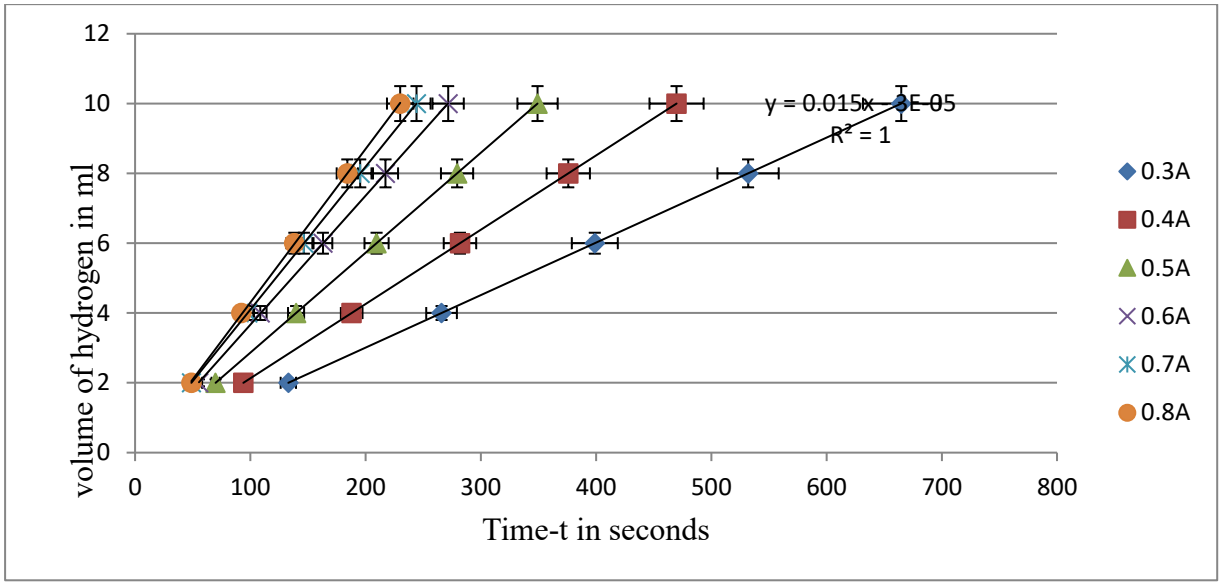

Figure 12: time required to reach 10 ml of hydrogen gas by different current densities

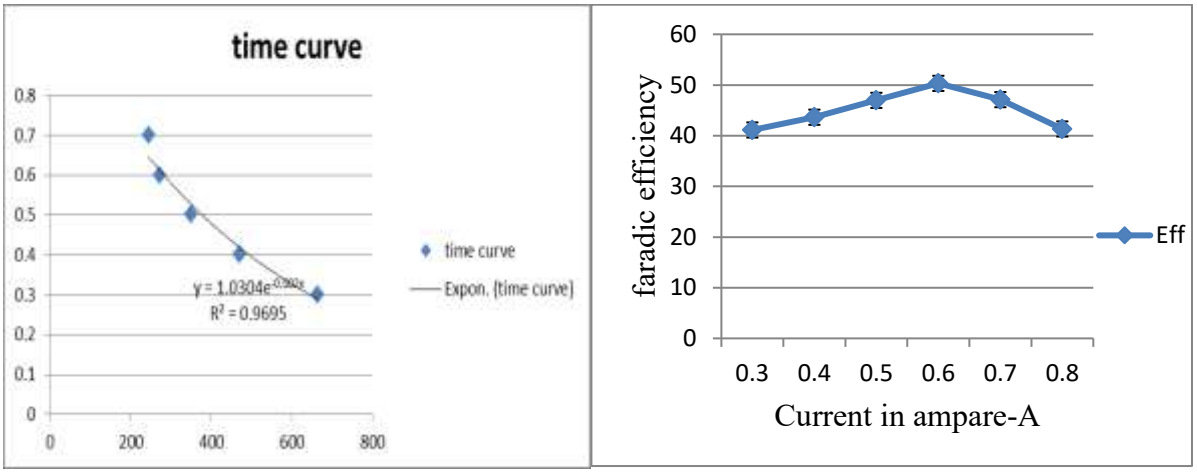

Figure 13:: (a) the efficiency at each current to reach 10 ml of hydrogen (efficiency curve), (b) time versus current to reach 10 ml of hydrogen at each given current (time Vs. current curve)

From observation of Fig14a As the current rises in linear manner the time required to reach the terminal volume decreases with decreasing exponential pattern. This implies with each increment of applied current the hydrogen produced increased exponentially. This was in agreement with the data from Table-4 as the applied current goes upward from 0.3- 0.8amps the terminal time needed declines from 664.88sec-244.21sec.

On the other hand deducing from Fig14b at amperage of 0.3A, 0.4A, 0.5A, 0.6A, 0.7A and 0.8A the respective faradic efficiency was 41.122%, 43.642%,46.973%, 50.322%,47.982% and 44.562%. Hence faradic efficiency exhibits an upward trend until 0.6A and further rise in current led to efficiency stagnation and finally efficiency drops. This implies as current increases electrolysis goes with ease and hydrogen is produced at shorter possible time. On the other hand as the applied current rises the efficiency of hydrogen yield grows at steady pace and then starts to decline. This can be attributed for the mass transfer effect and bubble formation phenomena.

With increasing current the reaction is active and the electrode starts to be covered by bubbles. This decreases the contact surface with implication of decreasing hydrogen produced at an increasing current due to bubble phenomena. Over the past three decades, AWE researchers have become aware of the substantial energy losses linked to gas bubbles in the electrolyte. Consequently, there's been a greater push to analyze and find solutions for this bubble phenomenon (85). Already few solutions are in place for decreasing the Ohmic drop caused by the gas bubbles. Firstly, an electrolyte flow is usually applied to endorse bubble separation from the electrode surfaces during operation. Secondly, the electrolysis cells are pressurized in order to reduce the gas bubble volume. These solutions do, nonetheless, only eliminate the high Ohmic resistance caused by the bubble phenomena to a limited extent (86). Recent reports showed higher reduction in the ohmic drop can be achieved by applying an external field such as magnetic, ultrasonic and super gravity to the electrolysis cell (87). The external fields promote the detachment of bubbles from the electrode surface during electrolysis. As a consequence the current density is increased since more active sites are available for the processes to proceed.

4.2. Effect of temperature variation on hydrogen formation

Thermodynamics is another driving factor to design efficient electrolysis systems. To grasp better understanding of the effect of thermodynamics on hydrogen production i investigated the effect of temperature change on electrolysis. Hence to analyze the effect of temperature change on alkaline waste water electrolysis I experimented with five sets temperature at range of 303.15K (30^0C) up to 343.15K(70^0C) .

The time required to reach different sets of volume were measured at constant current of 0.6A and 10 g/L of electrolyte for the whole range of temperatures. At temperature of 303.15K (30^0C) to reach volume of 2ml, 4ml, 6ml, 8ml and 10ml it required, 62.52sec, 125.04sec,

187.57sec, 250.09sec, and 312.61sec respectively, For 323.15K the volume versus time relation to reach volume of 2ml, 4ml, 6ml, 8ml and 10ml was 46.8sec,93.58sec, 140.38secsec 187.17sec and 233.96sec respectively. In addition at the last temperature run of 343.15K(70^0C), to produce volume range of 2ml, 4ml, 6ml, 8ml and 10ml hydrogen the time needed to reach the each volume set was 36.84sec, 73.7 1sec,110.51sec, 147.35sec and 184.184sec respectively. These observations confirm that as temperature rose the time needed to reach each volume set of hydrogen declined.

In addition to reach 10 ml hydrogen by volume at the temperatures of 303.15K, 313.15K, 323.15K, 333.15K and 343.15K it required 312.61sec, 267.61sec, 233.96sec, 189.72sec, and 184.18sec respectively. From these data we can analyze that with as temperature rose the time required to reach 10ml by volume of hydrogen significantly declined from 312.61sec at 303.15K to 184.18sec at 343.15K. As presented in the Figure-15 when temperature varies the time needed to produce 10ml of hydrogen was considerably affected. Starting with 303.15K (30^oC) the time needed to reach 10ml of hydrogen was 321.61sec and declined to 184.18 at 343.15K (70^oC). This indicated hydrogen production rate grew steadily with the temperature. Since higher temperatures are difficult to sustain in terms of energy cost temperature range of 50-60^{o}C was chosen as favorable range for this study. These outcomes are in similar pattern with report that displayed increasing the operational temperature of AWE the efficiency can be significantly increased (88).It is observed that at high temperature less cell voltage was required because the overvoltage potentials, activation and ohomic voltages contributed to the overall cell voltage are reduced. In fact, energy efficiency at high temperature was much higher compared to low temperature. This was because the overall cell voltage was much closer to the actual minimum voltage (89). Heating the electrolyte provides the extra energy gain by the electrolyte and leads to lower the necessary Gibb's free energy, which makes the reaction to happen.

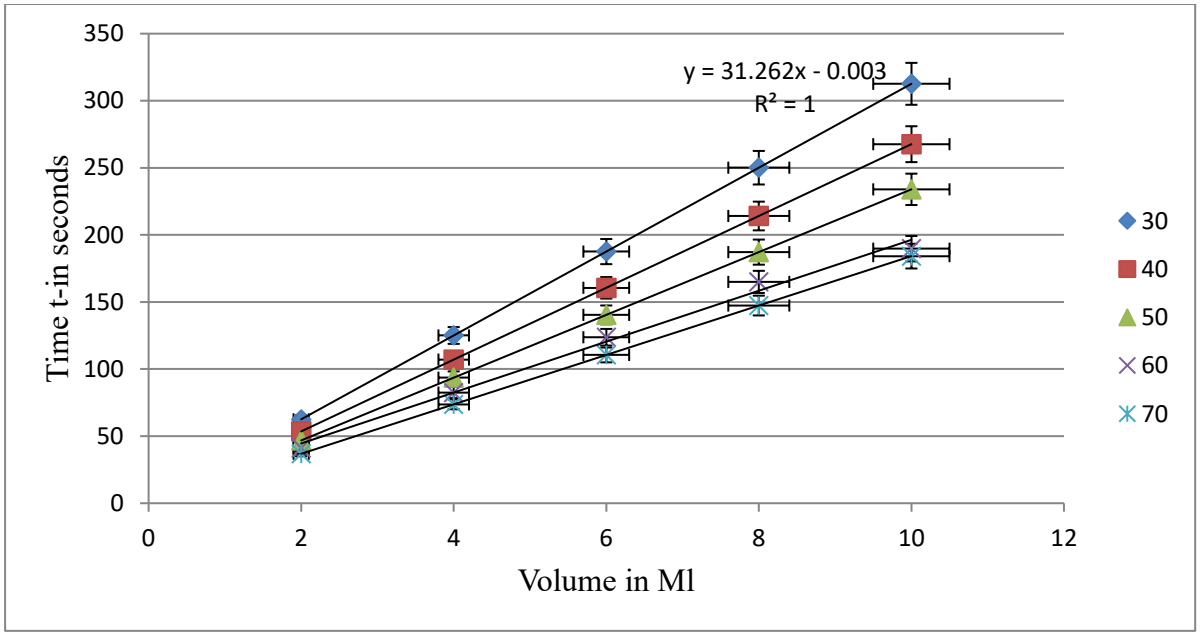

Figure 14: volumes of hydrogen produced at different temperatures.

In Figure-15 (based on data from Table-4) the linear trend lines indicates that with a rise of temperature at constant range the volume of hydrogen rose in linear fashion with temperature. This generalizes the assertion that with each higher temperature value the volume extracted from our system rises persistently.

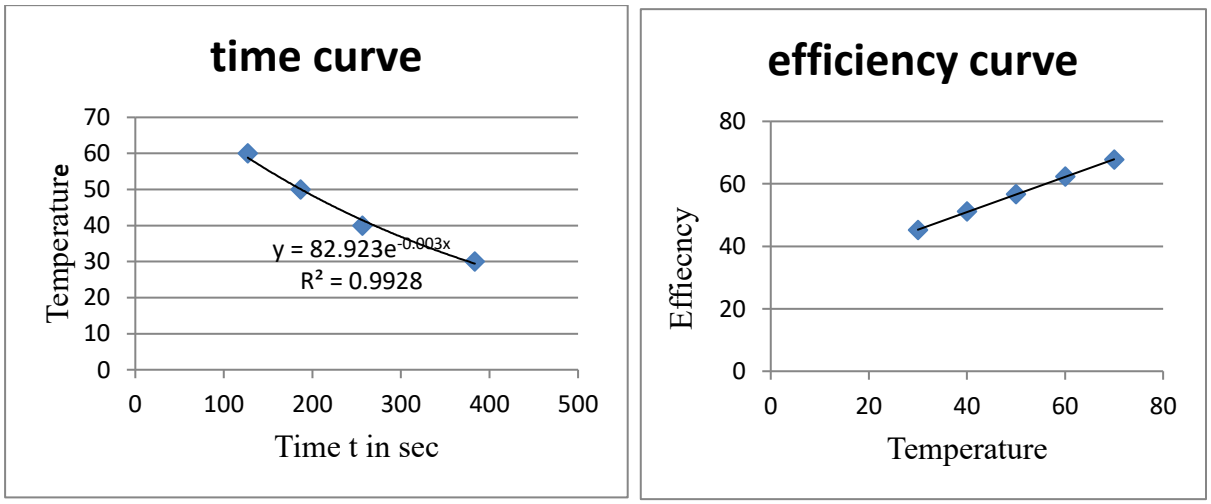

Figure 15: (a) Efficiency curve to liberate 10 ml of hydrogen at variable temperature (b) times for production of 10ml of hydrogen at variable temperature.

As presented in the curve in Figure-16b it shows that more feasible way of improving electrolysis efficiency is increasing the temperature, which lowers the voltage required to electrolyze the water. In addition the efficiency of electrolysis rises in direct proportion with

temperature while the time of reaction declines. However increasing temperature indefinitely will be power consuming; also it may lead to system degradation and material corrosion. Moreover the electrolyte has traces of alcohol and other carbohydrates that will start vaporizing at higher temperature greater than 343.13K (70ºC). Therefore temperature should better be limited between 50ºC-60ºC for hydrogen production.

4.3.Effect of concentration difference

One of the factors that affect rate of hydrogen production is electrolyte concentration. To analyze the effect of this parameter on the hydrogen yield from alkaline waste water a range of experiments and observations were made. At constant amperage of 0.25 Amps for all ranges of concentration tested: 5 g/l; 10 g/l; 15 g/l and 20 g/l NaOH, and the time needed to reach different sets of volume are measured from the experiment. At 5g/l the outcome showed that to reach volume of hydrogen 2ml, 4ml, 6ml, 8ml and 10ml the time were, 68.32sec, 136.63sec, 204.95sec, 273.26sec and 341.58sec respectively. From the same references Table5&Fig17 the results at different set of concentration to produce different ranges of volume indicates decreasing pattern with rising concentration. For example for 15g/l the volume versus time relation exhibited that to reach volume of 2ml, 4ml, 6ml, 8ml and 10ml time was 47.24sec, 94.47sec, 141.71sec, 188.94sec, and 236.18sec respectively. In addition at 20g/l, to produce volume range of 2ml, 4ml, 6ml, 8ml and 10ml hydrogen the time required to reach the respective volume are 41.91sec, 83.83sec, and 125.74sec, 167.65sec, and 209.57sec respectively. In general as concentration rises the time needed to reach each volume set of hydrogen declines.

In addition to reach volume of 10 ml hydrogen for 5 g/l; 10 g/l; 15 g/l and 20 g/l the time needed at each concentration set were 341.58sec, 276sec, 236.18sec and 209.57sec respectively. From these data patterns we can deduce that with upward increment in concentration the time needed to reach the terminal volume displayed significant diminishing trend. This was explained as increasing electrolyte concentration leads to rise in the electrical conductivity of the solution and consequently which promote more of reaction for producing hydrogen. This remark was in agreement with results obtained by Guy Bertrand Noumi&etal (90) that used graphite as material for cathode.

To summarize electrolyte concentration has strong effect on volume of hydrogen, it is clear that the production of hydrogen at a minimum duration has been higher with increasing electrolyte concentration.

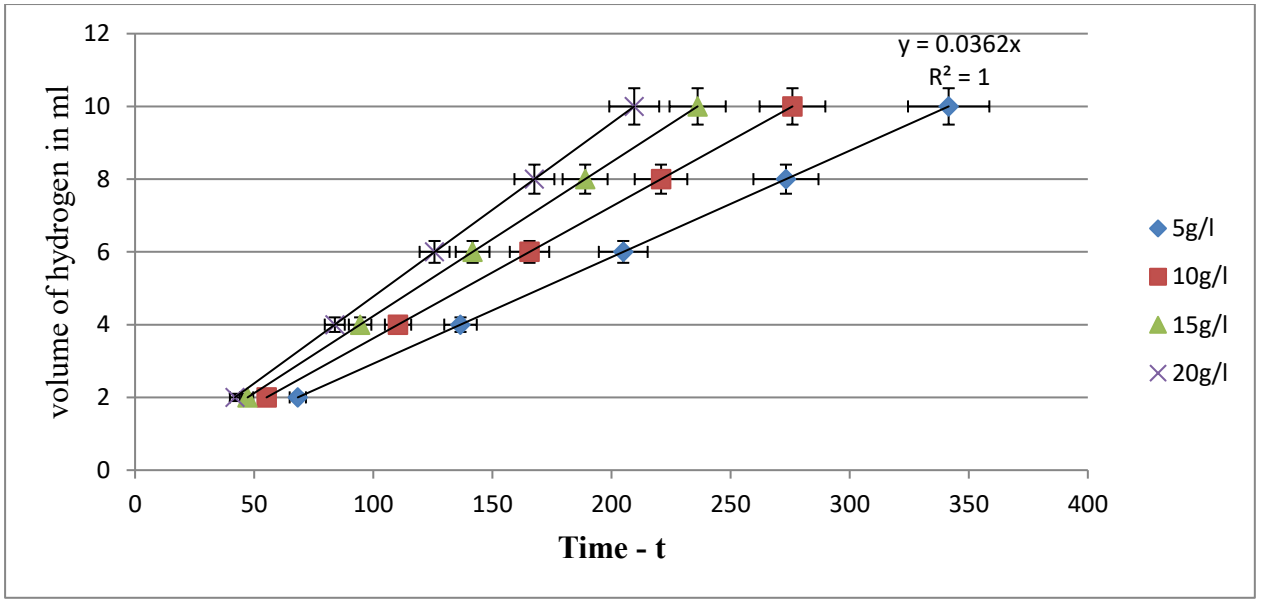

Figure 16: volumes of hydrogen produced at different concentrations

The above Figure-17 (based on data from Table-5) resembles the general picture of the effect of concentration on hydrogen production rate. At the range 5g/l -20g/l the trend line is further from the Y-axis initially and gets closer to the Y-axis with each added concentration making stiffer slope. This implies more volume of hydrogen is produced at each additional concentration lowering the time required to reach the volume sets. This is in agreement with the data from Table-5; for 5g/l – 20g/l of alkaline concentration it takes 341.58 sec down to 209.57sec to produce 10ml of hydrogen by volume.

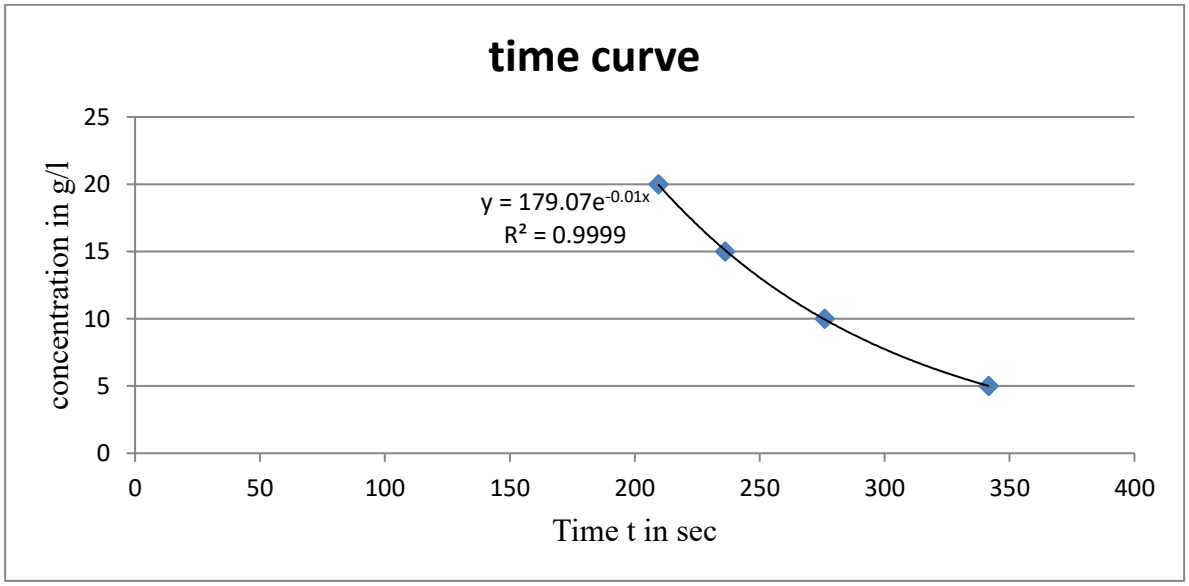

Figure 17: times to produce 10 ml of hydrogen at different concentrations.

From Figure-18 I one deduce that with each added concentration the time necessary to extract 10ml of hydrogen dropped in declining exponential pattern. As observed in the experimental range of 5g/l-20g/l of NaOH solution it displayed that the time decreased in declining exponential. Similar pattern was observed in Table-5; as the electrolyte concentration goes upward from 5g/l to 20g/l the time required to reach 10 ml of hydrogen decrease from 341.58sec -209.57sec.

Generally the ohmic performance of water electrolysis strongly depends on concentration of the electrolyte solution. The higher concentration means that higher number of available reactant at the electrode surface area for reaction at same power input which leads to increase in hydrogen production efficiency. This shows that electrolyte concentration has strong effect on cell performance. This behavior is attributed to the increase in the electrical conductivity of the solution due to the increase in the concentration of the sodium hydroxide. Increasing the electrical conductivity leads to an increase in the electrical current passing through the solution and consequently to a decrease of requirement of less voltage at same current density operation.

Note: all graphs are drawn based on the data tables in appendix A and B.

Chapter five

5.1. Conclusions

This study aimed at determining the effect of electrolyte concentration, temperature and current on the efficiency of hydrogen production from alkaline waste water electrolysis using graphite and nickel as electrodes.

In conclusion the parameters like the influence of temperature, electrolyte concentration and current on the performance of alkaline electrolyzers on hydrogen production are investigated.

The findings indicate that the electrolysis performance is significantly influenced by these parameters. Specifically the volume of hydrogen produced increases continuously with rising amperage. At the tested currents of 0.3A to 0.8A with 0.1A range difference the time needed to reach 10ml of hydrogen declined from 664.88s - 230.08s. In addition efficiency improved with each added current, from 41.122% to 44.562%. While higher applied currents initially enhance yield. However, beyond a certain threshold, further increases in current lead to a decline in efficiency. This is most likely due to limitations in mass transport and bubble formation within and around the cell.

The other variables tested were effects of temperature and electrolyte concentration on rate of hydrogen production. The test showed that as the at temperature increase of 10K interval; from 303.15K - 343.15K the time needed to reach 10ml by volume of hydrogen reduced in higher order of magnitude; 312.61s to 184.18s. In addition the efficiency of hydrogen production rate improved with each added temperature; from 45.173% to 67.735% consecutively. Though high temperature improves efficiency, it is not favored due to the higher operating costs and evaporation of electrolyte after certain point.

The effect of electrolyte concentration also showed certain significance in the hydrogen rate. At current of 0.25 Amps for all ranges of concentration tested: From 5 g/l to 20 g/l NaOH at arrange of 5g/l the time(s) to reach 10ml by volume of hydrogen declined from 341.58s to 209.57s. This is due to higher ionic mobility with an increased concentration of electrolyte.

To summarize the volume of hydrogen produced increases continuously with increasing current applied. This is due mainly to the rate of electrical charge conducted by the solution, because the higher the current flow (in Amps), the more electrons are transferred per unit time - seconds.

In similar manner the rise in the electrolyte temperature of 30 °C to 70 ° C greatly cuts the time required to reach 10ml of hydrogen. This is due to the increment in activity of electrolyte molecules which in another case increases the contact of hydroxide and hydrogen ions with the electrode. For cost effective and –in terms of the calculated faradic efficiency - hydrogen production temperature of 50-60ºC is more favorable zone.

In addition the electrolyte concentration has strong effect on volume of hydrogen produced, it is clear that the production of hydrogen at a minimum duration has been higher with increasing electrolyte concentration. This is explained because increasing electrolyte concentration leads to increase the electrical conductivity of the solution and consequently which promote more the reaction for producing hydrogen.

As observed in the results hydrogen yield reached its pic value at a current 0.6A with faradic efficiency 50.32%. On the other hand with each additional temperature the efficiency rises in linear manner. Furthermore to mitigate economic cost, power consumption and minimize evaporation temperature of 323.15 K was selected as favorable point. In addition our electrolyte was at 10 g/LNaOH from its primary source. Connecting these points the experiment displayed hydrogen yield approximately 57% efficiency.

5.2. Recommendations

- The current data shows peak efficiency around 0.7A (50.322%), but a slight dip at 0.8A. Maximizing hydrogen yield per unit of electrical energy input is crucial for operational cost-effectiveness. Pinpointing this optimal current range will allow for immediate operational improvements. This can be achieved by conducting further focused experiments to precisely identify the "threshold" current beyond which efficiency declines due to mass transport and bubble formation limitations.
- While higher temperatures improve efficiency, a detailed cost-benefit analysis is needed. Short-term studies should quantify the energy input required to maintain higher temperatures against the increased hydrogen production efficiency gained. This can be overcome by modeling the energy consumption for heating the electrolyte to different temperatures and compare it with the energy savings from improved electrolysis efficiency at those temperatures, considering energy prices.
- As mentioned in the outcome of the research limitations in mass transport and bubble formation cause for efficiency decline at higher currents. Addressing this directly could

yield immediate improvements. This can be addressed for the identified optimal current densities, initiate preliminary investigations into simple bubble management techniques - cell design modifications, agitation, electrode spacing adjustments-to mitigate mass transport limitations.

- Long-term stability of electrodes in wastewater electrolysis is a critical factor for sustained, reliable operation. This can be implemented by setting- up pilot-scale experiments with continuous wastewater feed and periodic electrode characterization for prolonged continuous operation tests to assess the long-term stability and durability of electrodes in actual alkaline wastewater.
- Hydrogen production from wastewater offers a pathway to sustainable energy storage, directly supporting grid stability for renewable energy. To achieve such objectives future research can be conducted by Simulation or building a small-scale system powered by renewables and investigates control algorithms for optimal performance under variable power conditions to explore the integration of the alkaline wastewater electrolysis system directly with intermittent renewable energy sources (e.g., solar panels or wind turbines). This would involve studying dynamic operating strategies to match fluctuating power inputs.
- For large scale production of hydrogen gravity settling and filtration are time-consuming and costly. Instead of these pretreatment methods screen filtration and sand filtration can be utilized to maximize output and minimize cost.
- In a large scale hydrogen plants stainless steel, zinc and copper alloys are much better in terms of durability. These alloys can be exploited for their durability of extreme conditions.
- After production of hydrogen other byproducts will remain in the form of sodium ion and other unwanted byproducts. Though sodium can be further purified as useful byproduct, it better can be exploited by concentrating it and recycling it back as an electrolyte material.

5.3. Area for Further Study

- Since this research is exclusively limited on identifying effect of some parameters' on

hydrogen production. Future research can be conducted on designing large scale design of alkaline electrolyser. These researches will also identify the potential for production of hydrogen from alkaline waste water on other industries.

- Another broad area of research is also integration of electrolysis with intermittent energy sources like wind and solar power. By integrating the electrolysis cells to intermittent energy resources they can be used as energy storage. So that future researchers can investigate the potential feasibility as well as applicability.
- This research is also limited by the laboratory capability to study hydrogen purity level. But future researchers can identify and maximize the hydrogen purity level.
- Exploring the impact of different electrode materials (anode and cathode) on electrolysis performance will also be an area for future investigation. Different alloys will be tested for their efficiency, rust resistance and durability. This could involve investigating materials with higher activity, improved stability in wastewater, or lower cost.
- Wastewater composition might contribute to electrode fouling; exploring strategies to mitigate it can be another area of study. This could involve developing anti-fouling electrode materials or optimizing operating conditions to minimize contaminant buildup.
- Investigation of the potential impact of microorganisms in the wastewater on the electrolysis process might give better oversight of alkaline waste electrolysis. Some microbes might be beneficial (promoting hydrogen evolution), while others could be detrimental (reducing efficiency). Therefore future researchers can investigate the effect of microbes on electrolysis.
- Investigation of hydrogen production efficiency by different types of alloy for electrode material can also contribute in designing and implementing large scale electrolysers.

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Appendix A

Experimental data sets for variation of current, concentration and temperature from experiment

sets of current(amps) &time (s)		volume of hydrogen gas (ml)				
		2	4	6	8	10
	0.3	132.98	265.95	398.93	531.91	664.88
	0.4	93.97	187.95	281.92	375.89	469.87
	0.5	69.85	139.7	209.55	279.4	349.24
	0.6	55.43	108.66	163	217.32	271.66
	0.7	48.84	97.68	146.52	195.37	244.21
	0.8	48.84	92.11	138.21	184.16	230.08

Table 2: time vs. volume of hydrogen at different current densities

temperature(k) & time(s	T(k)	**volume of hydrogen(ml)**				
		2	4	6	8	10
	303.15	62.52	125.04	187.57	250.09	312.61
	313.15	53.52	107.05	160.57	214.09	267.61
	323.15	46.8	93.58	140.38	187.17	233.96
	333.15	41.24	82.5	123.73	164.98	189.72
	343.15	36.84	73.7	110.51	147.35	184.18

Table 3: times to produce the volumes of hydrogen at different temperatures

volume(ml) & Time(s)		**concentration (g/L)**			
		5	10	15	20
	2	68.32	55.2	47.24	41.91
	4	136.63	110.4	94.47	83.83

	6	204.95	165.6	141.71	125.74
	8	273.26	220.8	188.94	167.65
	10	341.58	276	236.18	209.57

Table 4: time to produce volumes of hydrogen at different concentrations

Appendix B

Faradic efficiency calculation: From the empirical calculation efficiency of alkaline electrolyser was calculated using eq (26&27)

K(nf/Vm)	**Ve(ml)**	**Ts(k)**	**Te(k)**	**t (s)**	**I**	**Η**

At constant temperature (T = 313.15K or 40ºC) and concentration (10g/l) of sodium hydroxide versus variable current						
8.615	10	298.15	313.15	664.88	0.3	0.41122
8.615	10	298.15	313.15	469.87	0.4	0.43642
8.615	10	298.15	313.15	349.24	0.5	0.46973
8.615	10	298.15	313.15	271.66	0.6	0.50322
8.615	10	298.15	313.15	244.21	0.7	0.47982
8.615	10	298.15	313.15	230.08	0.8	0.44562
Constant concentration, and current versus variable temperature						
8.615	10	298.15	303.15	312.61	0.6	0.45173
8.615	10	298.15	313.15	267.61	0.6	0.51084
8.615	10	298.15	323.15	233.96	0.6	0.56623
8.615	10	298.15	333.15	189.72	0.6	0.6231
8.615	10	298.15	343.15	184.18	0.6	0.67735

Table 5: calculated efficiencies to produce 10 ml of hydrogen at varying current and temperature from top to bottom respectively

Note that:

V_m is the molar volume of perfect gas at STP which is 22.4mol/liter

f is faraday constant which is 96485 c/s

n is the molecules of hydrogen equals to 2.